Zirconium Oxychloride Hydrate: An Efficient and Reusable Catalyst for Retro-Claisen Condensation of Alcohols with 1,3-Diketones

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1. Experimental section

1.1 General information.

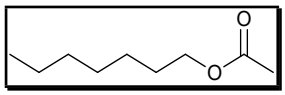
All reactions were performed in oven-dried glassware flushed with nitrogen. The GC analysis was carried out using GC Shimadzu (GC-2030) gas chromatograph equipped with FID detector and capillary column (SH-Rtx-1, length 30 meter, inner diameter 0.32 mm, film 0.25 mm). ^{1}H NMR and ^{13}C NMR spectra were recorded in a Bruker Avanceneo 600 spectrometer using $CDCl_3$ as solvent. The chemical shifts (δ) are reported in ppm and coupling constants (*J*) in hertz (Hz). The GC-mass spectrometric analysis was carried out using Shimadzu GC-MS QP 2020 equipped with FID detector and capillary column (SH-Rtx-1701, length 60 meter, inner diameter 0.25 mm, film 0.25 mm). TLC inspections were performed on Silica gel 60 F_{254} plates. Column chromatography was performed on silica gel (60-120 mesh) using ethyl acetate/hexanes as eluent.

1.2 General procedure

In a typical procedure, Zirconium oxychloride hydrate ($ZrOCl_2.8H_2O$) was added to the well stirred solution of alcohol (2 mmol) and 1,3 diketone (2.2 mmol) in a 25 mL sealed tube and the reaction mixture was stirred at 100 ^{o}C for 16 h. After completion of the reaction (as monitored by TLC using $KMnO_4$ stain) the reaction mixture was cooled to room temperature and diluted with dichloromethane (20 mL). The solid catalyst was separated by centrifugation and solvent was removed under reduced pressure to yield the crude product. The crude product was purified by flash column chromatography on silica gel (60-120 mesh) using ethyl acetate/hexanes as the eluent. All the products were identified on the basis of ^{1}H, ^{13}C NMR and mass spectral data and compared to references reported previously.

2. Spectroscopic data

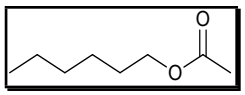
1-Heptyl acetate (Table 1, 3a)[7]



^{1}H NMR (600 MHz, $CDCl_3$): δ (ppm) = 4.05 (t, *J* = 6.8 Hz, 2H), 2.04 (s, 3H), 1.64-1.59 (m, 2H), 1.34-1.28 (m, 8H), 0.88 (t, *J* = 7.3 Hz, 3H);

^{13}C NMR (150 MHz, $CDCl_3$): δ (ppm) = 171.16, 64.62, 31.69, 28.89, 28.60, 25.85, 22.54, 20.94, 13.99. NMR data were in accordance with those reported in the literature.[7]

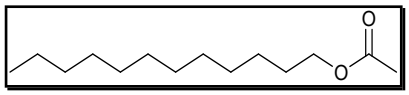
1-Hexyl acetate (Table 2, 3b)[1]



^{1}H NMR (600 MHz, $CDCl_3$): δ (ppm) = 4.06 (t, *J* = 6.8 Hz, 2H), 2.05 (s, 3H), 1.64-1.60 (m, 2H), 1.36-1.26 (m, 6H), 0.89 (t, *J* = 7.1 Hz, 3H);

^{13}C NMR (150 MHz, $CDCl_3$): δ (ppm) = 171.20, 64.65, 31.43, 28.57, 25.57, 22.51, 20.97, 13.95. NMR data were in accordance with those reported in the literature.[1]

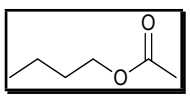
1-Dodecyl acetate (Table 2, 3c)[5]



^{1}H NMR (600 MHz, $CDCl_3$): δ (ppm) = 4.06 (t, *J* = 6.8 Hz, 2H), 2.05 (s, 3H), 1.65-1.60 (m, 2H), 1.35-1.25 (m, 18H), 0.89 (t, *J* = 7.2 Hz, 3H);

^{13}C NMR (150 MHz, $CDCl_3$): δ (ppm) = 171.18, 64.65, 31.90, 29.62, 29.60, 29.55, 29.50, 29.32, 29.34, 28.61, 25.90, 22.66, 20.97, 14.07. NMR data were in accordance with those reported in the literature.[5]

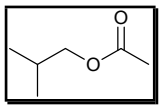
1-Butyl acetate (Table 2, 3d)[1]



^{1}H NMR (600 MHz, $CDCl_3$): δ (ppm) = 4.07 (t, *J* = 6.7 Hz, 2H), 2.04 (s, 3H), 1.62-1.60 (m, 2H), 1.40-1.38 (m, 2H), 0.94 (t, *J* = 7.5 Hz, 3H);

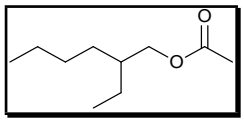
^{13}C NMR (150 MHz, $CDCl_3$): δ (ppm) = 171.23, 64.35, 30.65, 28.89, 20.96, 19.11, 13.66. NMR data were in accordance with those reported in the literature.[1]

Isobutyl acetate (Table 2, 3e)[1]



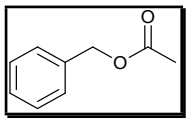
^{1}H NMR (600 MHz, $CDCl_3$): δ (ppm) = 3.84 (d, *J* = 6.8 Hz, 2H), 2.05 (s, 3H), 1.96-1.88 (m, 1H), 0.93 (d, *J* = 7.0 Hz, 6H);
^{13}C NMR (150 MHz, $CDCl_3$): δ (ppm) = 171.24, 70.62, 27.66, 20.89, 19.04. NMR data were in accordance with those reported in the literature.[1]

2-Ethylhexyl acetate (Table 2, 3f)[11]



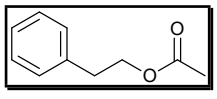
^{1}H NMR (600 MHz, $CDCl_3$): δ (ppm) = 4.00 (d, *J* = 7.3 Hz, 2H), 2.05 (s, 3H), 1.58-1.50 (m, 1H), 1.37-1.29 (m, 8H), 0.90-0.80 (m, 6H);
^{13}C NMR (150 MHz, $CDCl_3$): δ (ppm) = 171.32 66.99, 38.74, 30.40, 28.92, 23.77, 22.94, 20.95, 13.99, 10.95. NMR data were in accordance with those reported in the literature.[11]

Benzyl acetate (Table 2, 3g)[1]



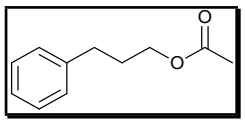
^{1}H NMR (600 MHz, $CDCl_3$): δ (ppm) = 7.40-7.35 (m, 5H), 5.13 (s, 2H) 2.13 (s, 3H);
^{13}C NMR (150 MHz, $CDCl_3$): δ (ppm) = 170.86, 135.97, 128.56, 128.40, 128.24, 66.30, 20.98. NMR data were in accordance with those reported in the literature.[1]

2-Phenethyl acetate (Table 2, 3h)[1]



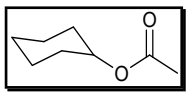
^{1}H NMR (600 MHz, $CDCl_3$): δ (ppm) = 7.33-7.28 (m, 2H), 7.26-7.22 (m, 3H), 4.29 (t, *J* = 7.2 Hz, 2H) 2.95 (t, *J* = 7.2 Hz, 2H) 2.05 (s, 3H);
^{13}C NMR (150 MHz, $CDCl_3$): δ (ppm) = 170.99, 137.83, 128.87, 128.49, 126.56, 64.91, 35.11, 20.93. NMR data were in accordance with those reported in the literature.[1]

3-Phenylpropyl acetate (Table 2, 3i)[1]



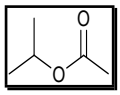
^{1}H NMR (600 MHz, $CDCl_3$): δ (ppm) = 7.31-7.27 (m, 2H), 7.22-7.18 (m, 3H), 4.09 (t, *J* = 6.8 Hz, 2H) 2.70 (t, *J* = 6.8 Hz, 2H) 2.06 (s, 3H), 1.99-1.94 (m, 2H);
^{13}C NMR (150 MHz, $CDCl_3$): δ (ppm) = 171.12, 141.21, 128.43, 128.38, 126.00, 63.83, 32.19, 30.18, 20.93. NMR data were in accordance with those reported in the literature.[1]

Cyclohexyl acetate (Table 2, 3j)[2]



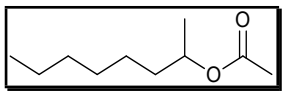
^{1}H NMR (600 MHz, $CDCl_3$): δ (ppm) = 4.75-4.72 (m, 1H), 2.03 (s, 3H), 1.87-1.84 (m, 2H), 1.74-1.71 (m, 2H), 1.57-1.53 (m, 1H), 1.41-1.34 (m, 4H), 1.27-1.23 (m, 1H);
^{13}C NMR (150 MHz, $CDCl_3$): δ (ppm) = 170.57, 72.67, 31.67, 25.39, 23.80, 21.42. NMR data were in accordance with those reported in the literature.[2]

Isopropyl acetate (Table 2, 3k)[7]



^{1}H NMR (600 MHz, $CDCl_3$): δ (ppm) = 4.98-4.95 (m, 1H), 1.99 (s, 3H), 1.21 (d, *J* = 6.8 Hz, 6H);
^{13}C NMR (150 MHz, $CDCl_3$): δ (ppm) = 170.52, 67.53, 21.74, 21.32. NMR data were in accordance with those reported in the literature.[7]

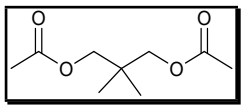
2-Octyl acetate (Table 2, 3l)[5]



^{1}H NMR (600 MHz, $CDCl_3$): δ (ppm) = 4.91-4.87 (m, 1H), 2.03 (s, 3H), 1.60-1.45 (m, 2H), 1.31-1.25 (m, 8H), 1.20 (d, *J* = 7.6 Hz, 3H), 0.88 (t, *J* = 6.9 Hz, 3H);

^{13}C NMR (150 MHz, $CDCl_3$): δ (ppm) = 170.78, 71.08, 35.94, 31.73, 29.11, 25.35, 22.56, 21.36, 19.94, 14.02. NMR data were in accordance with those reported in the literature.[5]

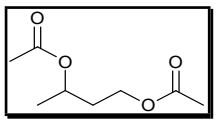
2,2-Dimethylpropane-1,3-diyl diacetate (Table 2, 3m)[3]



^{1}H NMR (600 MHz, $CDCl_3$): δ (ppm) = 3.86 (s, 4H), 2.04 (s, 6H), 0.95 (s, 6H);

^{13}C NMR (150 MHz, $CDCl_3$): δ (ppm) = 170.93, 69.18, 34.52, 21.67, 20.76. NMR data were in accordance with those reported in the literature.[3]

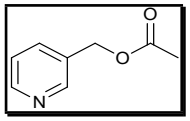
Butane-1,3-diyl diacetate (Table 2, 3n)[10]



^{1}H NMR (600 MHz, $CDCl_3$): δ (ppm) = 5.01-5.03 (m, 1H), 4.12 (t, *J* = 6.3 Hz, 2H), 2.04-2.06 (m, 6H), 1.85-1.94 (m, 2H), 1.27 (d, *J* = 6.8 Hz, 3H);

^{13}C NMR (150 MHz, $CDCl_3$): δ (ppm) = 170.98, 170.54, 67.87, 60.79, 34.78, 21.21, 20.87, 20.04. NMR data were in accordance with those reported in the literature.[10]

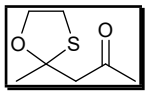
Pyridin-3-ylmethyl acetate (Table 2, 3o)[8]



^{1}H NMR (600 MHz, $CDCl_3$): δ (ppm) = 8.64 (m, 1H), 8.59 (m, 1H), 7.72 (m, 1H), 7.34-7.27 (m, 1H), 5.14 (s, 2H) 2.12 (s, 3H);

^{13}C NMR (150 MHz, $CDCl_3$): δ (ppm) = 170.64, 149.41, 149.35, 136.21, 131.75, 123.51, 63.64, 20.81. NMR data were in accordance with those reported in the literature.[8]

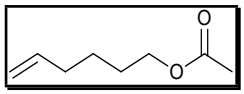
1-(2-Methyl-1,3-oxathiolan-2-yl)propan-2-one (Table 2, 3r[l])[12]



^{1}H NMR (600 MHz, $CDCl_3$): δ (ppm) = 4.21-4.12 (m, 2), 3.11-2.97 (m, 4H), 2.21 (s, 3H), 1.69 (s, 3H);

^{13}C NMR (150 MHz, $CDCl_3$): δ (ppm) = 205.59, 91.25, 69.97, 56.30, 33.67, 31.25, 29.29. NMR data were in accordance with those reported in the literature.[12]

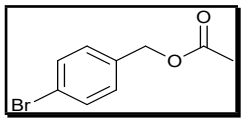
Hex-5-en-1-yl acetate (Table 3, 3w)[4]



^{1}H NMR (600 MHz, $CDCl_3$): δ (ppm) = 5.82-5.77 (m, 1), 5.04-4.95 (dd, *J* = 15.9, 6.9 Hz, 2H), 4.07 (t, *J* = 6.8 Hz, 2H), 2.11-2.08 (m, 2H), 2.05, (s, 3H), 1.65-1.60 (m, 2H), 1.47-1.45 (m, 2H);

^{13}C NMR (150 MHz, $CDCl_3$): δ (ppm) = 171.17, 138.33, 114.80, 64.39, 33.26, 28.04, 25.18, 20.96. NMR data were in accordance with those reported in the literature.[4]

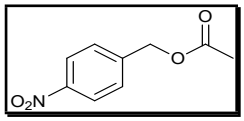
4-Bromobenzyl acetate (Table 3, 3x)[6]



^{1}H NMR (600 MHz, $CDCl_3$): δ (ppm) = 7.51 (d, *J* = 8.2 Hz, 2H), 7.25 (d, *J* = 8.2 Hz, 2H), 5.07 (s, 2H) 2.12 (s, 3H);

^{13}C NMR (150 MHz, $CDCl_3$): δ (ppm) = 170.7, 134.99, 131.72, 129.91, 122.28, 65.47, 20.92. NMR data were in accordance with those reported in the literature.[6]

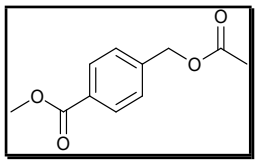
4-Nitrobenzyl acetate (Table 3, 3y)[2]



^{1}H NMR (600 MHz, $CDCl_3$): δ (ppm) =8.24 (d, *J* = 8.4 Hz, 2H), 7.53 (d, *J* = 8.4 Hz, 2H), 5.21 (s, 2H) 2.16 (s, 3H);

^{13}C NMR (150 MHz, $CDCl_3$): δ (ppm) = 170.48, 147.75, 143.20, 128.38, 123.80, 64.77, 20.80. NMR data were in accordance with those reported in the literature.[2]

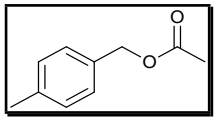
Methyl 4-(acetoxymethyl) benzoate (Table 3, 3z)[9]



^{1}H NMR (600 MHz, $CDCl_3$): δ (ppm) = 8.05 (d, *J* = 8.1 Hz, 2H), 7.46 (d, *J* = 8.5 Hz, 2H), 5.18 (s, 2H), 3.94 (s, 3H), 2.15 (s, 3H);

^{13}C NMR (150 MHz, CDCl3): δ (ppm) = 170.79 166.92, 141.06, 130.05, 130.03, 127.78, 65.71, 52.34, 21.05. NMR data were in accordance with those reported in the literature.[9]

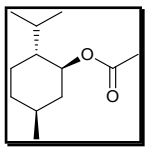
4-Methylbenzyl acetate (Table 3, 3ab)[14]



^{1}H NMR (600 MHz, $CDCl_3$): δ (ppm) = 7.28-7.25 (d, *J* = 7.1 Hz, 2H), 7.22-7.19 (d, J = 7.1 Hz, 2H), 5.09 (s, 2H) 2.38 (s, 3H), 2.11 (s, 3H);

^{13}C NMR (150 MHz, $CDCl_3$): δ (ppm) = 170.91, 138.11, 132.98, 129.26, 128.44, 66.26, 21.19, 21.03. NMR data were in accordance with those reported in the literature.[14]

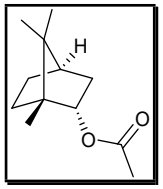
Menthyl acetate (Table 4, Entry 1)[6]



^{1}H NMR (600 MHz, $CDCl_3$): δ (ppm) = 4.69 (m, 1H), 2.05 (s, 3H), 2.02-1.98 (m, 1H), 1.90-1.81 (m, 1H), 1.74-1.61 (m, 2H), 1.52-1.46 (m, 1H), 1.39-1.36 (m, 1H), 1.08 (m, 3H), 0.91 (d, *J* = 7.3 Hz, 6H), 0.78 (d, *J* = 7.6 Hz, 3H);

^{13}C NMR (150 MHz, $CDCl_3$): δ (ppm) = 170.67, 74.18, 47.03, 40.95, 34.28, 31.37, 26.36, 23.55, 21.99, 21.31, 20.71, 16.41. NMR data were in accordance with those reported in the literature.[6]

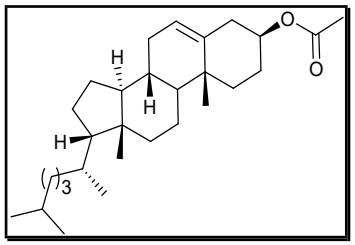
Borenyl acetate (Table 4, Entry 2)[13]



^{1}H NMR (600 MHz, $CDCl_3$): δ (ppm) = 4.88 (t, *J* = 7.6 Hz, 1H), 2.38-2.34 (m, 1H), 2.06 (s, 3H), 1.96-1.91 (m, 1H), 1.76-1.66 (m, 1H), 1.33-1.22 (m, 3H), 0.92 (m, 1H), 0.90 (s, 3H), 0.87 (s, 3H), 0.83 (s, 3H);

^{13}C NMR (150 MHz, $CDCl_3$): δ (ppm) = 171.55, 79.97, 48.78, 47.86, 45.00, 36.85, 28.12, 27.16, 21.38, 19.79, 18.91, 13.55. NMR data were in accordance with those reported in the literature.[13]

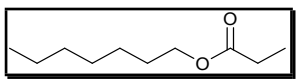
Cholestaryl acetate (Table 4, Entry 3)[10]



^{1}H NMR (600 MHz, $CDCl_3$): δ (ppm) = 5.38 (m, 1H), 4.60 (m, 1H), 2.33-2.28 (m, 2H), 2.03 (s, 3H), 2.00-1.95 (m, 2H), 1.88-1.84 (m, 3H), 1.61-1.32 (m, 12H), 1.28-1.09 (m, 7H), 1.07-1.01- (m, 9H), 0.99-0.87, (d, *J* = 6.7 Hz, 6H), 0.69-0.67, (d, *J* = 6.7 Hz, 3H);

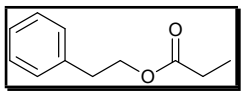
^{13}C NMR (150 MHz, $CDCl_3$): δ (ppm) = 170.50, 139.68, 122.64, 73.99, 56.71, 56.17, 50.06, 42.33, 39.75, 39.53, 38.14, 37.01, 36.60, 36.20, 35.79, 31.91, 31.88, 28.22, 28.01, 27.79, 24.29, 23.84, 22.80, 22.55, 21.41, 21.04, 19.31, 18.72, 11.86. NMR data were in accordance with those reported in the literature.[10]

Heptyl propionate (Table 2, Entry 21)[15]



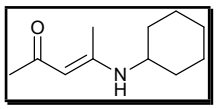
^{1}H NMR (600 MHz, $CDCl_3$): δ (ppm) = 4.06 (t, *J* = 6.72 Hz, 2H), 2.36-2.28 (m, 2H), 1.68-1.55 (m, 2H), 1.38-1.25 (m, 8H), 1.14 (t, *J* = 7.47 Hz, 3H), 0.88, (t, *J* = 6.72 Hz, 3H). NMR data were in accordance with those reported in the literature.[15]

Phenethyl propionate (Table 2, Entry 22)[16]



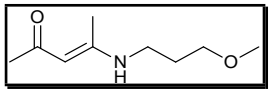
^{1}H NMR (600 MHz, $CDCl_3$): δ (ppm) = 7.31 (t, *J* = 7.78 Hz, 2H), 7.24 (t, *J* = 7.17 Hz, 3H), 4.30 (t, *J* = 7.01 Hz, 2H), 2.95 (t, *J* = 7.01 Hz, 2H), 2.36-2.28 (m, 2H), 1.13 (t, *J* = 7.62 Hz, 3H). NMR data were in accordance with those reported in the literature.[16]

(E)-4-(Cyclohexylamino)pent-3-en-2-one[17]



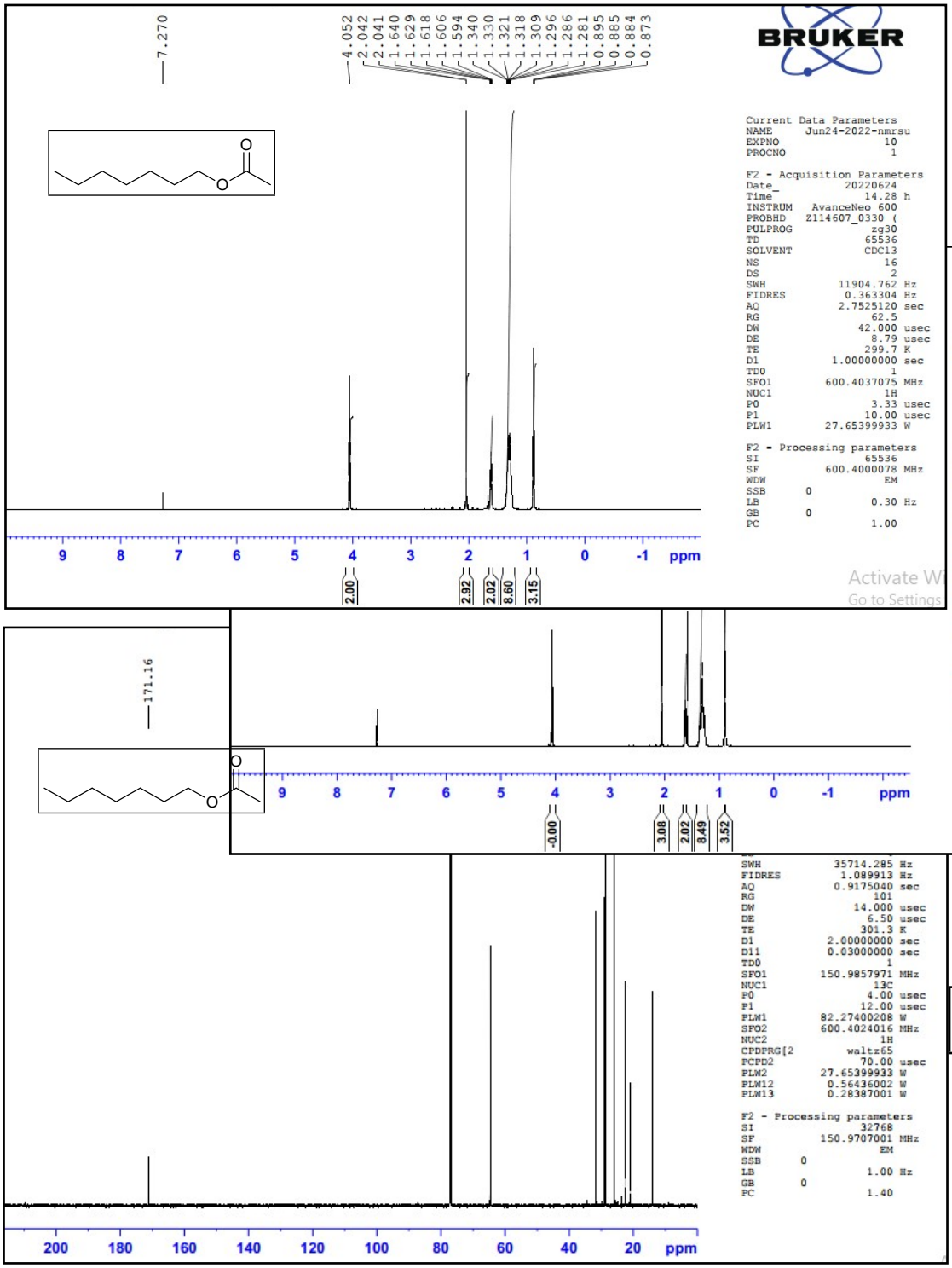
^{1}H NMR (600 MHz, $CDCl_3$): δ (ppm) = 10.98 (s, 1H), 4.90 (s, 1H), 3.36 (m, 1H), 1.98 (s, 3H), 1.93 (s, 3H), 1.83-1.81 (m, 2H), 1.80-1.73 (m, 2H), 1.62-1.55 (m, 1H), 1.39-1.24 (m, 6H). NMR data were in accordance with those reported in the literature.[17]

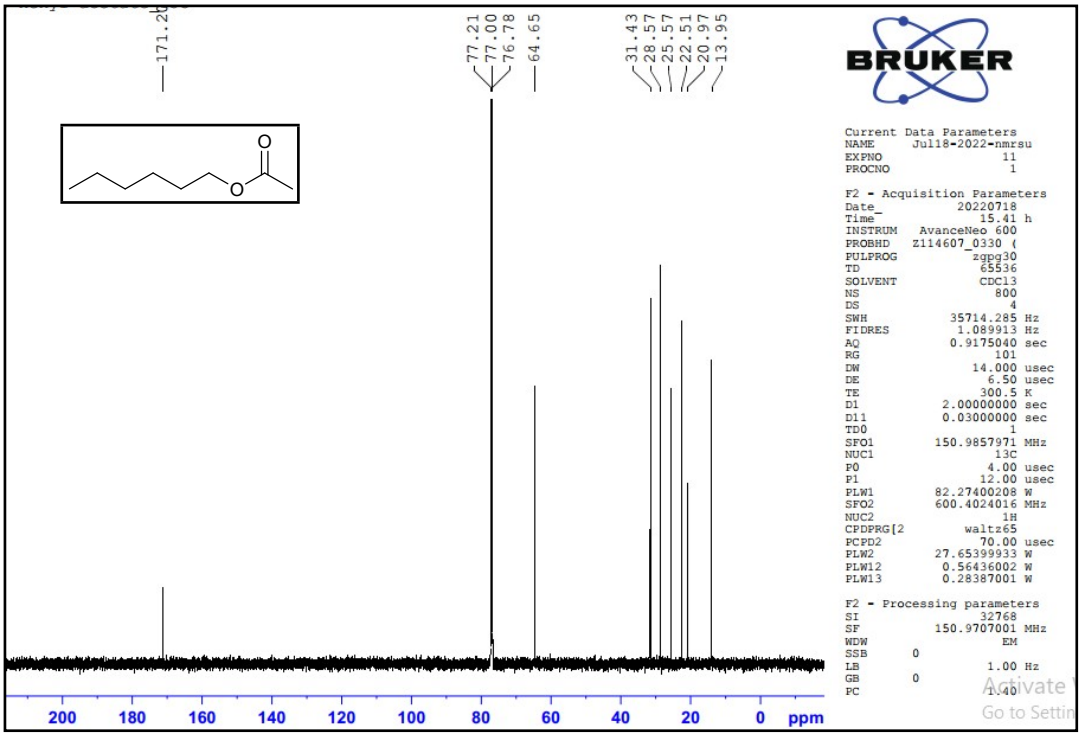
(E)-4-((3-Methoxypropyl)amino)pent-3-en-2-one



^{1}H NMR (600 MHz, $CDCl_3$): δ (ppm) = 10.83 (s, 1H), 4.95 (s, 1H), 3.43 (t, *J* = 5.95 Hz, 2H), 3.38-3.30 (m, 6H), 1.99 (s, 3H), 1.92 (s, 3H), 1.86-1.70 (m, 2H). NMR data were in accordance with those reported in the literature.

3. Copies of ^{1}H and ^{13}C NMR spectra of products





BRUKER

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FIDRES         1.089913 Hz
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RG                  101
DW               14.000 usec
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TE                300.5 K
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D11          0.03000000 sec
TD0                   1
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NUC1                13C
P0                4.00 usec
P1               12.00 usec
PLW1         82.27400208 W
SFO2         600.4024016 MHz
NUC2                1H
CPDPRG[2        waltz65
PCPD2            70.00 usec
PLW2         27.65399933 W
PLW12         0.56436002 W
PLW13         0.28387001 W

F2 - Processing parameters
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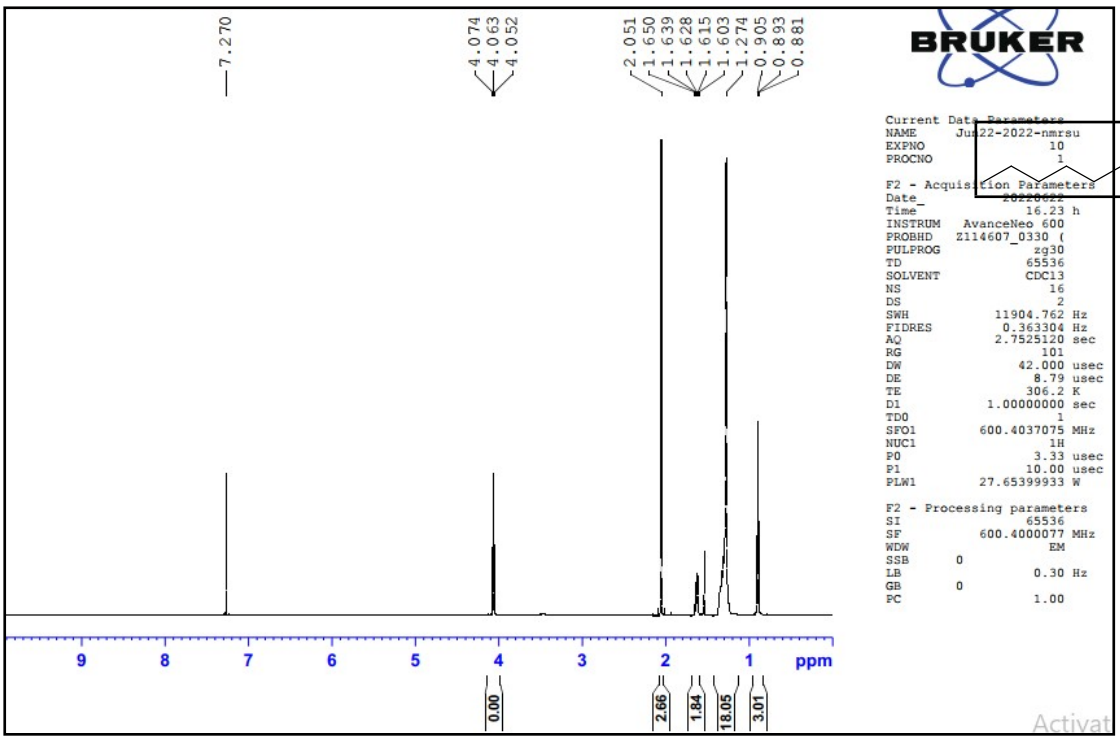

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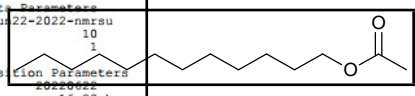


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PROCNO               1

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PULPROG             zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           11904.762 Hz
FIDRES         0.363304 Hz
AQ            2.7525120 sec
RG                  101
DW               42.000 usec
DE                8.79 usec
TE                306.2 K
D1           1.00000000 sec
TD0                   1
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NUC1                1H
P0                3.33 usec
P1               10.00 usec
PLW1         27.65399933 W

F2 - Processing parameters
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SF          600.4000077 MHz
WDW                 EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
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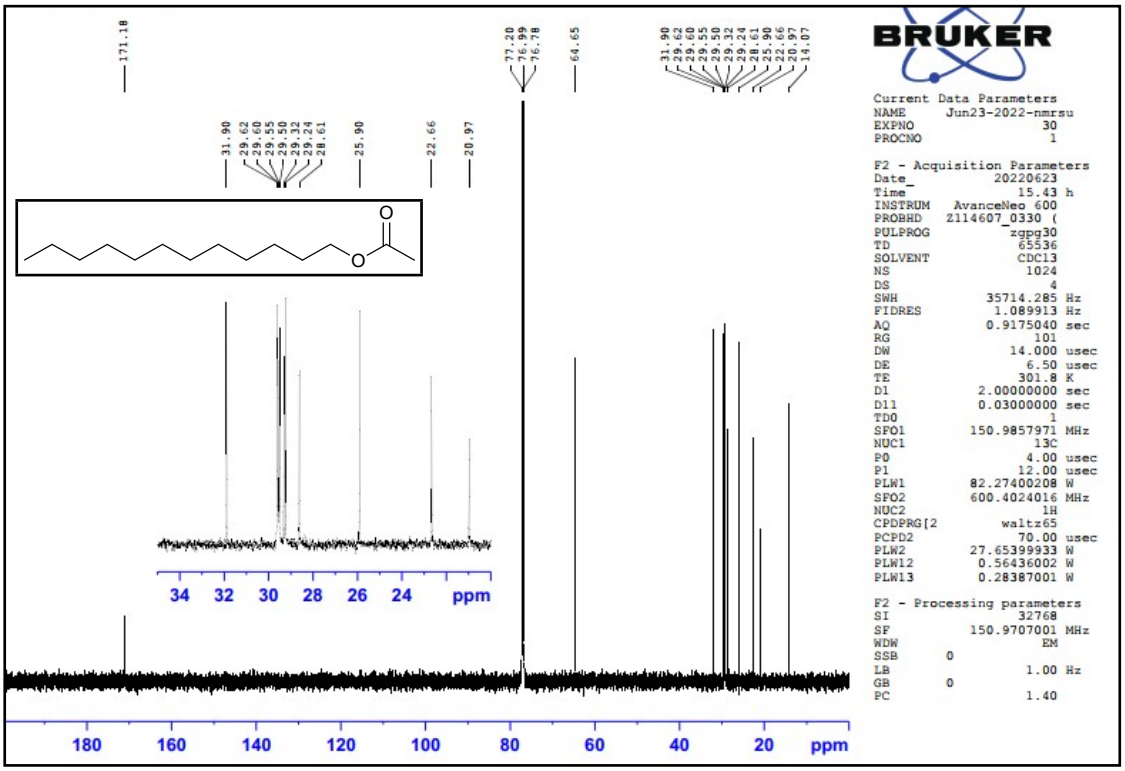
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31.90
29.62
29.60
29.55
29.50
29.32
29.24
28.61
25.90
22.66
20.97
14.07
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O
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180 160 140 120 100 80 60 40 20 ppm
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PROCNO 1
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Time 15.43 h
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DS 4
SWH 35714.285 Hz
FIDRES 1.089913 Hz
AQ 0.9175040 sec
RG 101
DW 14.000 usec
DE 6.50 usec
TE 301.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 150.9857971 MHz
NUC1 13C
P0 4.00 usec
P1 12.00 usec
PLW1 82.27400208 W
SFO2 600.4024016 MHz
NUC2 1H
CPDPRG[2 waltz65
PCPD2 70.00 usec
PLW2 27.65399933 W
PLW12 0.56436002 W
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F2 - Processing parameters
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WDW EM
SSB 0
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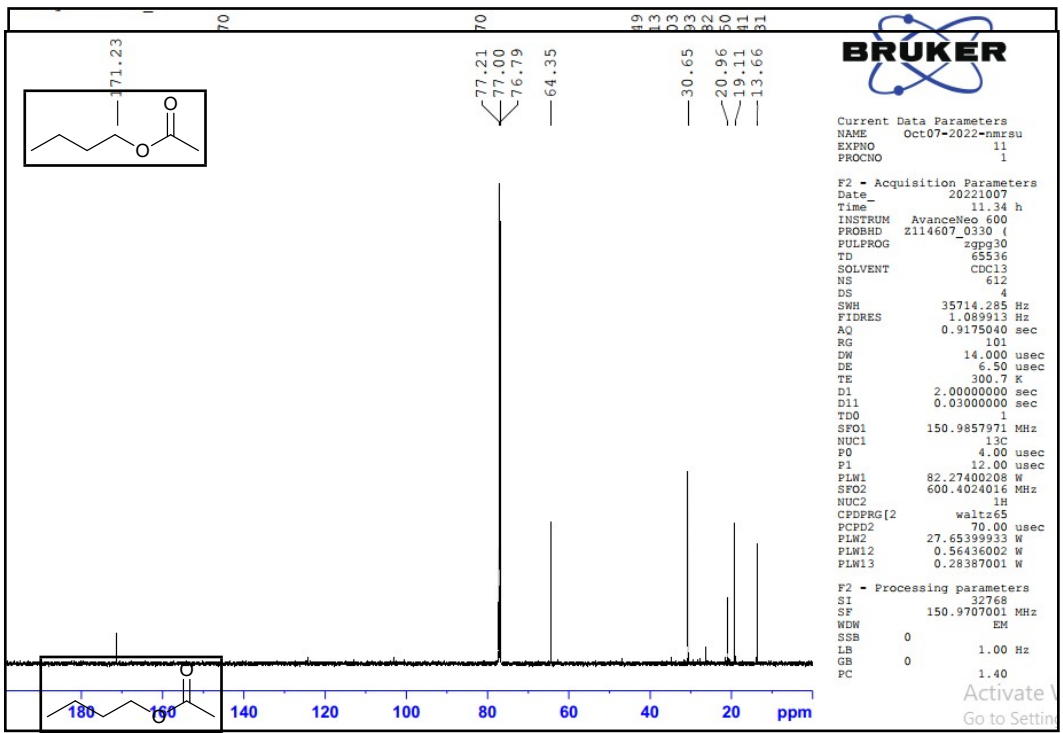
GB 0
PC 1.40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76.79
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30.65
20.96
19.11
13.66
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O
180 160 140 120 100 80 60 40 20 ppm
BRUKER
Current Data Parameters
NAME Oct07-2022-nmrsu
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
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Time 11.34 h
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PROBHD Z114607_0330 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 612
DS 4
SWH 35714.285 Hz
FIDRES 1.089913 Hz
AQ 0.9175040 sec
RG 101
DW 14.000 usec
DE 6.50 usec
TE 300.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 150.9857971 MHz
NUC1 13C
P0 4.00 usec
P1 12.00 usec
PLW1 82.27400208 W
SFO2 600.4024016 MHz
NUC2 1H
CPDPRG[2 waltz65
PCPD2 70.00 usec
PLW2 27.65399933 W
PLW12 0.56436002 W
PLW13 0.28387001 W
F2 - Processing parameters
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SF 150.9707001 MHz
WDW EM
SSB 0
LB 1.00 Hz
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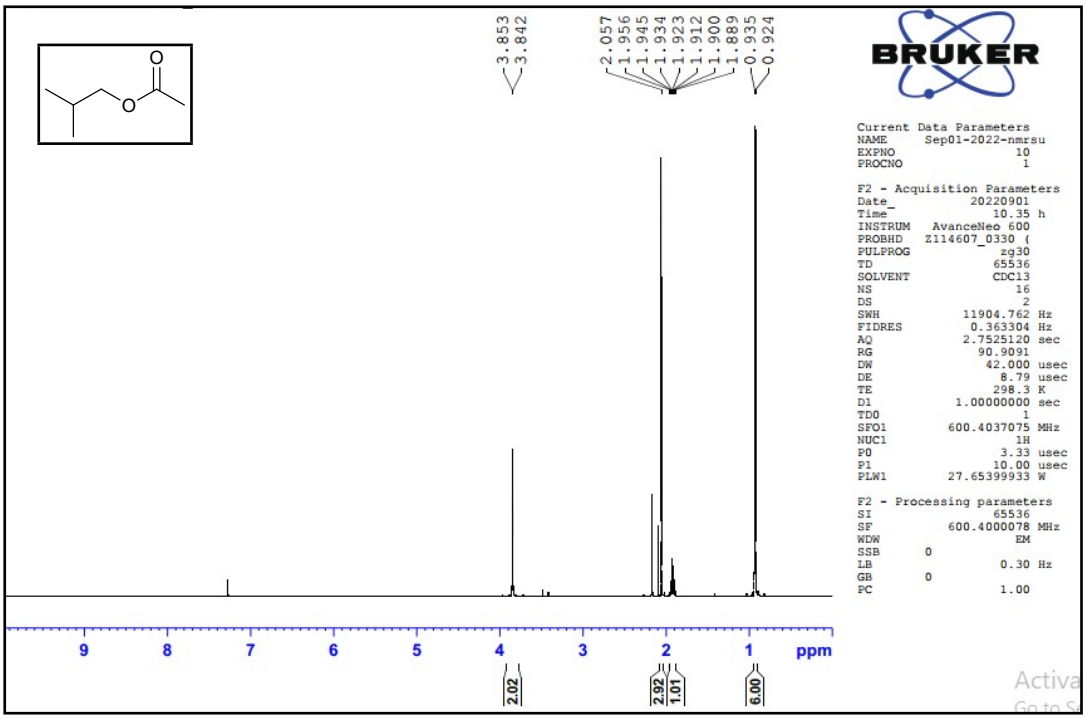
PC 1.40

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PROCNO                   1

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PROBHD   Z114607_0330 (
PULPROG              zg30
TD                  65536
SOLVENT              CDC13
NS                      16
DS                       2
SWH             11904.762 Hz
FIDRES           0.363304 Hz
AQ               2.7525120 sec
RG                  90.9091
DW                  42.000 usec
DE                   8.79 usec
TE                  298.3 K
D1               1.00000000 sec
TD0                      1
SFO1            600.4037075 MHz
NUC1                    1H
P0                   3.33 usec
P1                  10.00 usec
PLW1            27.65399933 W

F2 - Processing parameters
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SF              600.4000078 MHz
WDW                     EM
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LB                   0.30 Hz
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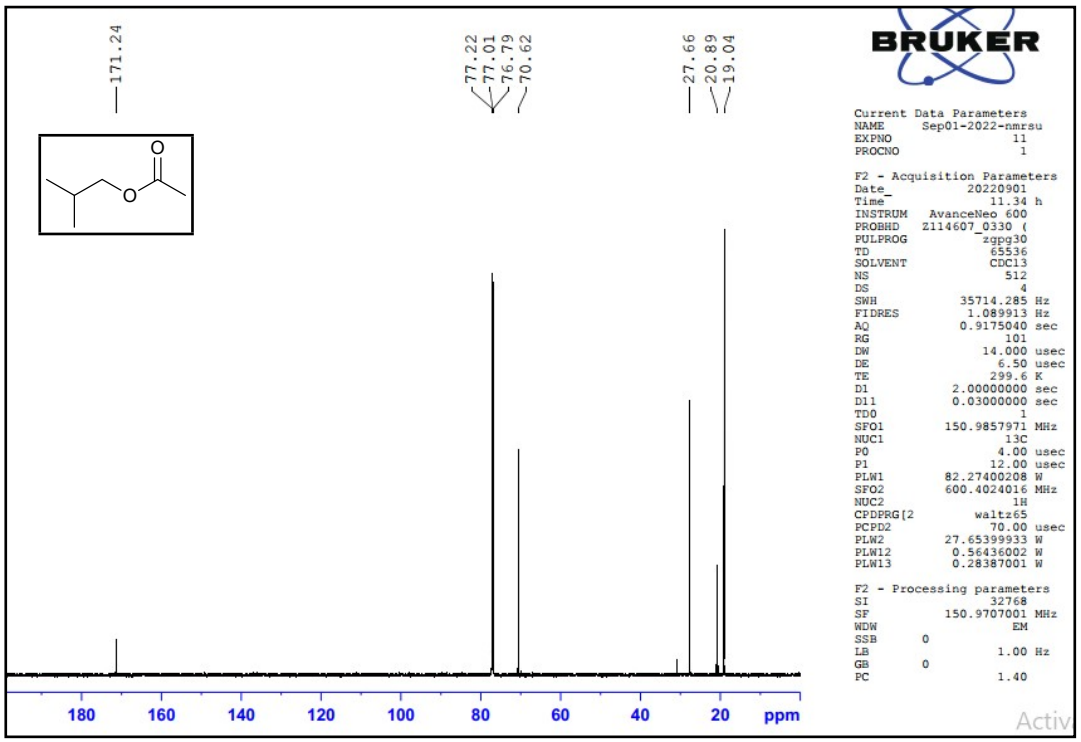


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EXPNO                   11
PROCNO                   1

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PULPROG            zgpg30
TD                  65536
SOLVENT              CDC13
NS                     512
DS                       4
SWH             35714.285 Hz
FIDRES           1.089913 Hz
AQ               0.9175040 sec
RG                     101
DW                  14.000 usec
DE                   6.50 usec
TE                  299.6 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                      1
SFO1            150.9857971 MHz
NUC1                    13C
P0                   4.00 usec
P1                  12.00 usec
PLW1            82.27400208 W
SFO2            600.4024016 MHz
NUC2                    1H
CPDPRG[2          waltz65
PCPD2               70.00 usec
PLW2            27.65399933 W
PLW12            0.56436002 W
PLW13            0.28387001 W

F2 - Processing parameters
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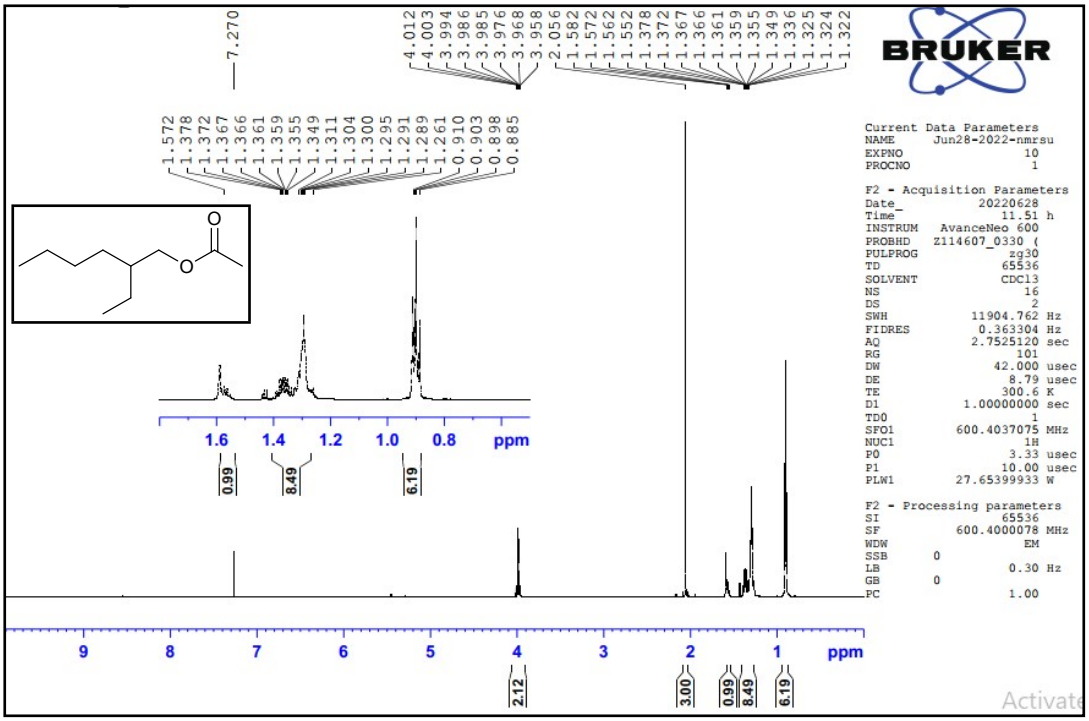
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1.562
1.552
1.378
1.372
1.367
1.366
1.361
1.359
1.355
1.349
1.336
1.325
1.324
1.322
1.572
1.378
1.372
1.367
1.366
1.361
1.359
1.355
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1.304
1.300
1.295
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1.261
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5
4
3
2
1
ppm
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8.49
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BRUKER
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EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
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Time 11.51 h
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PROBHD Z114607_0330 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 11904.762 Hz
FIDRES 0.363304 Hz
AQ 2.7525120 sec
RG 101
DW 42.000 usec
DE 8.79 usec
TE 300.6 K
D1 1.00000000 sec
TD0 1
SFO1 600.4037075 MHz
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P0 3.33 usec
P1 10.00 usec
PLW1 27.65399933 W
F2 - Processing parameters
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WDW EM
SSB 0
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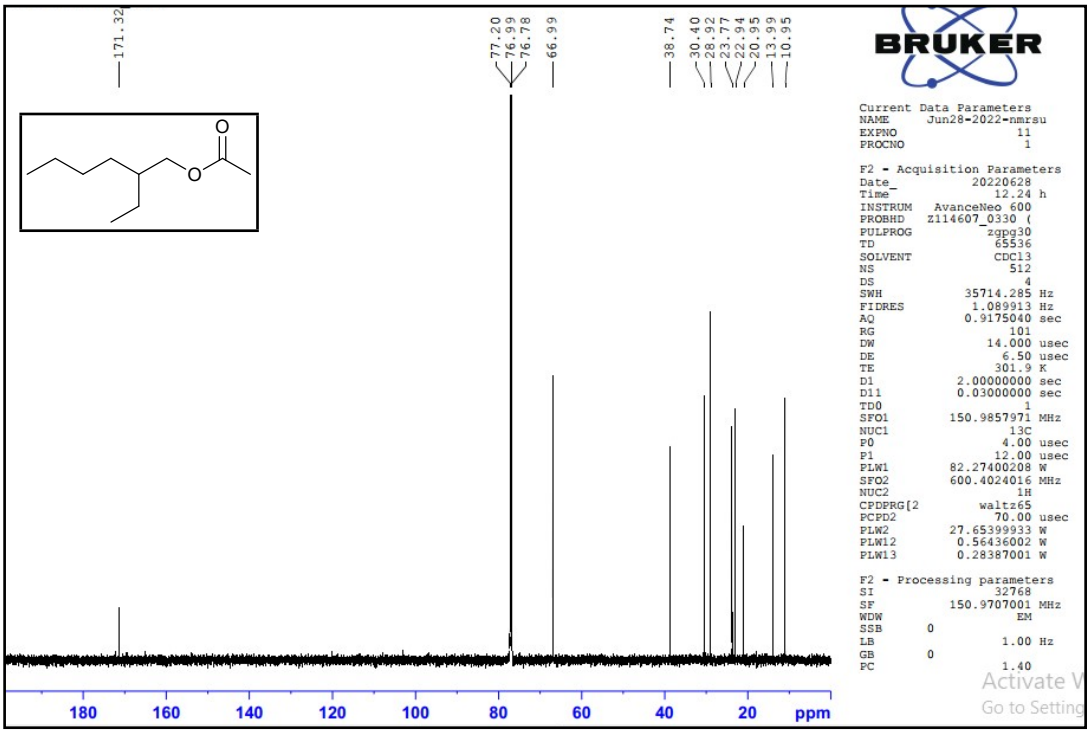
GB 0
PC 1.00
Activate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160
140
120
100
80
60
40
20
ppm
BRUKER
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NAME Jun28-2022-nmrsu
EXPNO 11
PROCNO 1
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PULPROG zgpg30
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SOLVENT CDCl3
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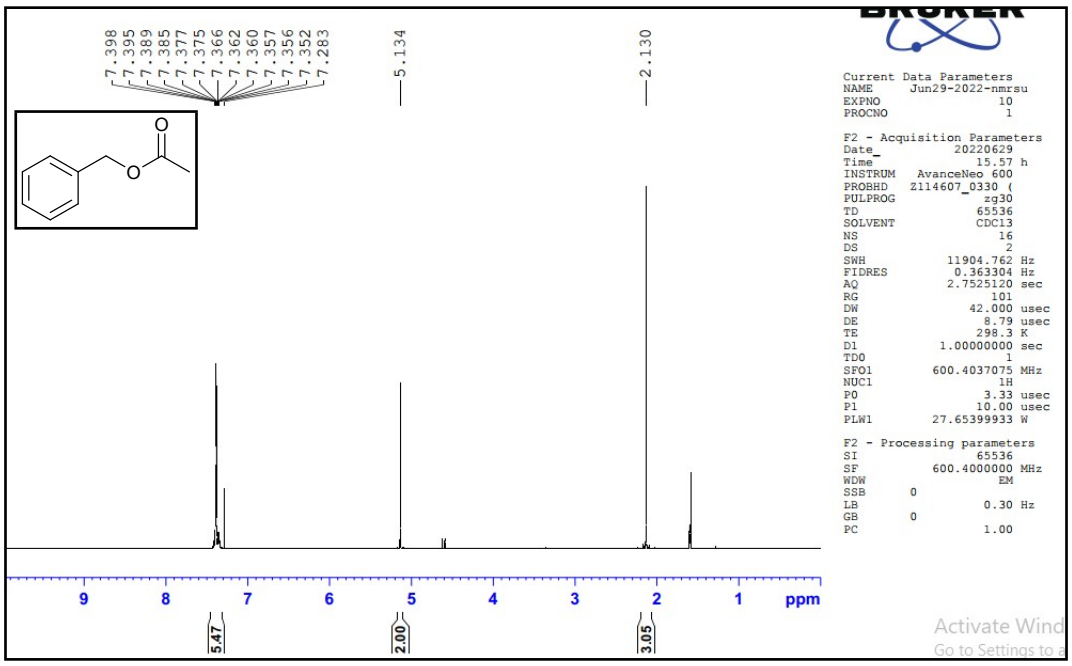
FIDRES 1.089913 Hz
AQ 0.9175040 sec
RG 101
DW 14.000 usec
DE 6.50 usec
TE 301.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 150.9857971 MHz
NUC1 13C
P0 4.00 usec
P1 12.00 usec
PLW1 82.27400208 W
SFO2 600.4024016 MHz
NUC2 1H
CPDPRG[2 waltz65
PCPD2 70.00 usec
PLW2 27.65399933 W
PLW12 0.56436002 W
PLW13 0.28387001 W
F2 - Processing parameters
SI 32768
SF 150.9707001 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Activate W
Go to Setting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PULPROG zg30
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SWH 11904.762 Hz
FIDRES 0.363304 Hz
AQ 2.7525120 sec
RG 101
DW 42.000 usec
DE 8.79 usec
TE 298.3 K
D1 1.00000000 sec
TD0 1
SFO1 600.4037075 MHz
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P0 3.33 usec
P1 10.00 usec
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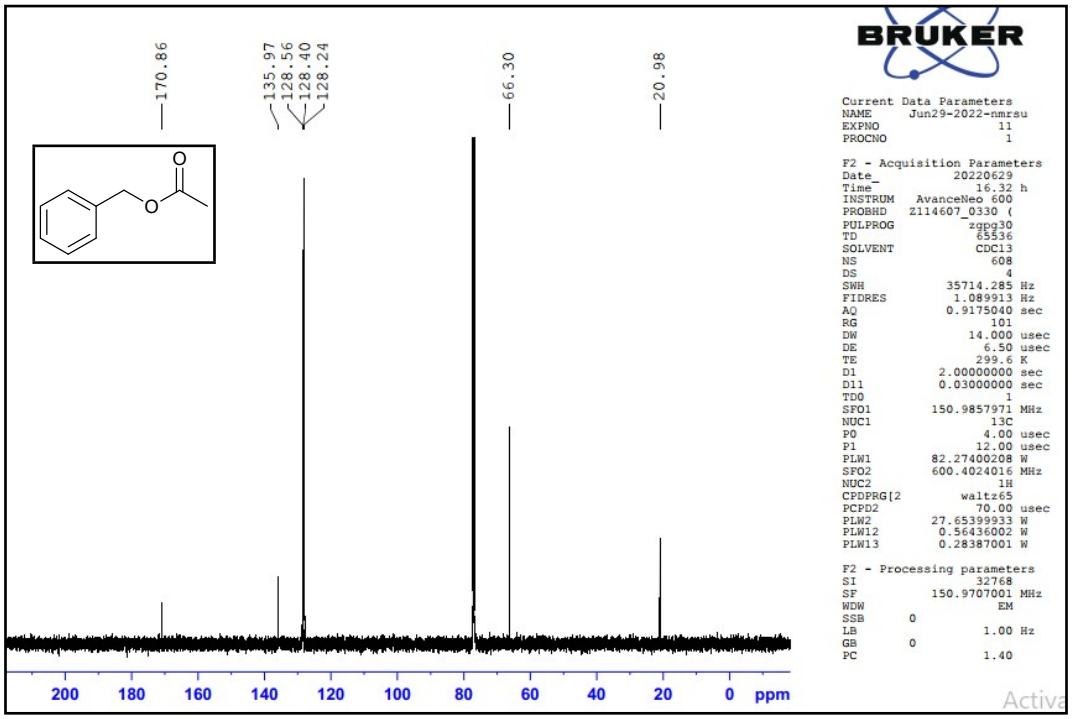
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60
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20
0
ppm
BRUKER
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PROCNO 1
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Time 16.32 h
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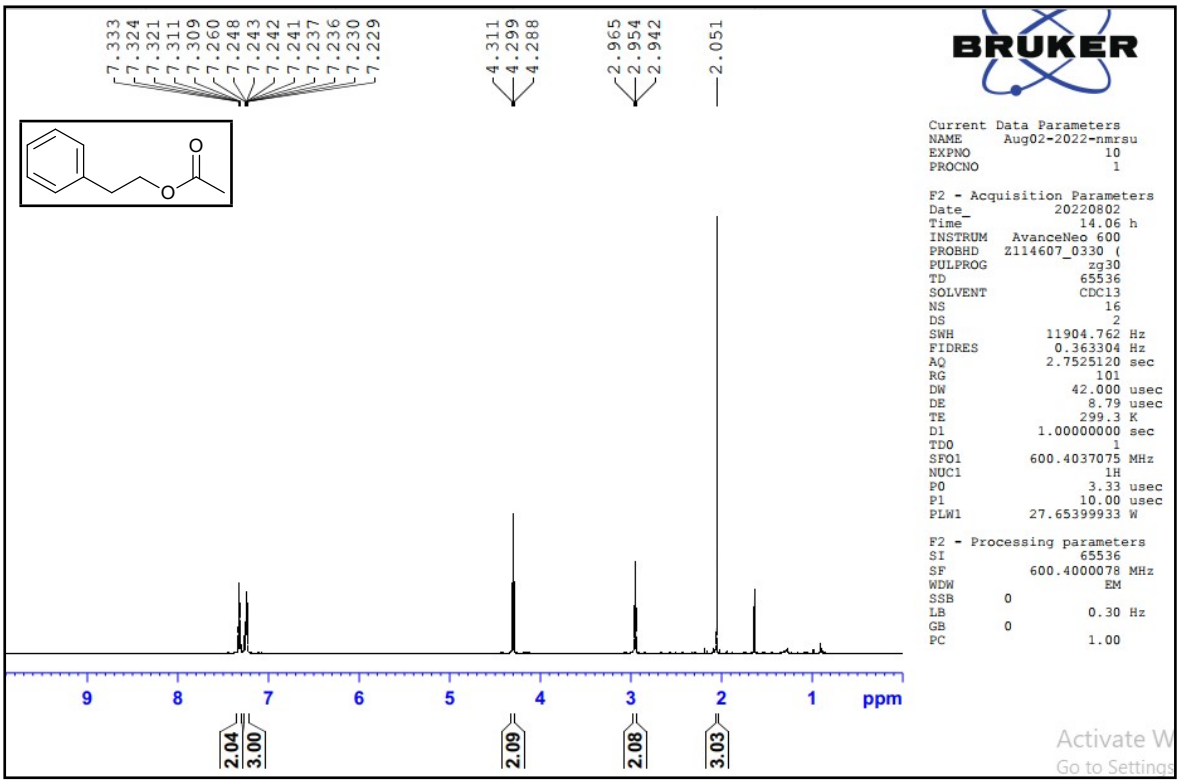
TD 65536
SOLVENT CDCl3
NS 608
DS 4
SWH 35714.285 Hz
FIDRES 1.089913 Hz
AQ 0.9175040 sec
RG 101
DW 14.000 usec
DE 6.50 usec
TE 299.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 150.9857971 MHz
NUC1 13C
P0 4.00 usec
P1 12.00 usec
PLW1 82.27400208 W
SFO2 600.4024016 MHz
NUC2 1H
CPDPRG[2 waltz65
PCPD2 70.00 usec
PLW2 27.65399933 W
PLW12 0.56436002 W
PLW13 0.28387001 W
F2 - Processing parameters
SI 32768
SF 150.9707001 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40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ppm
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2.09
2.08
3.03
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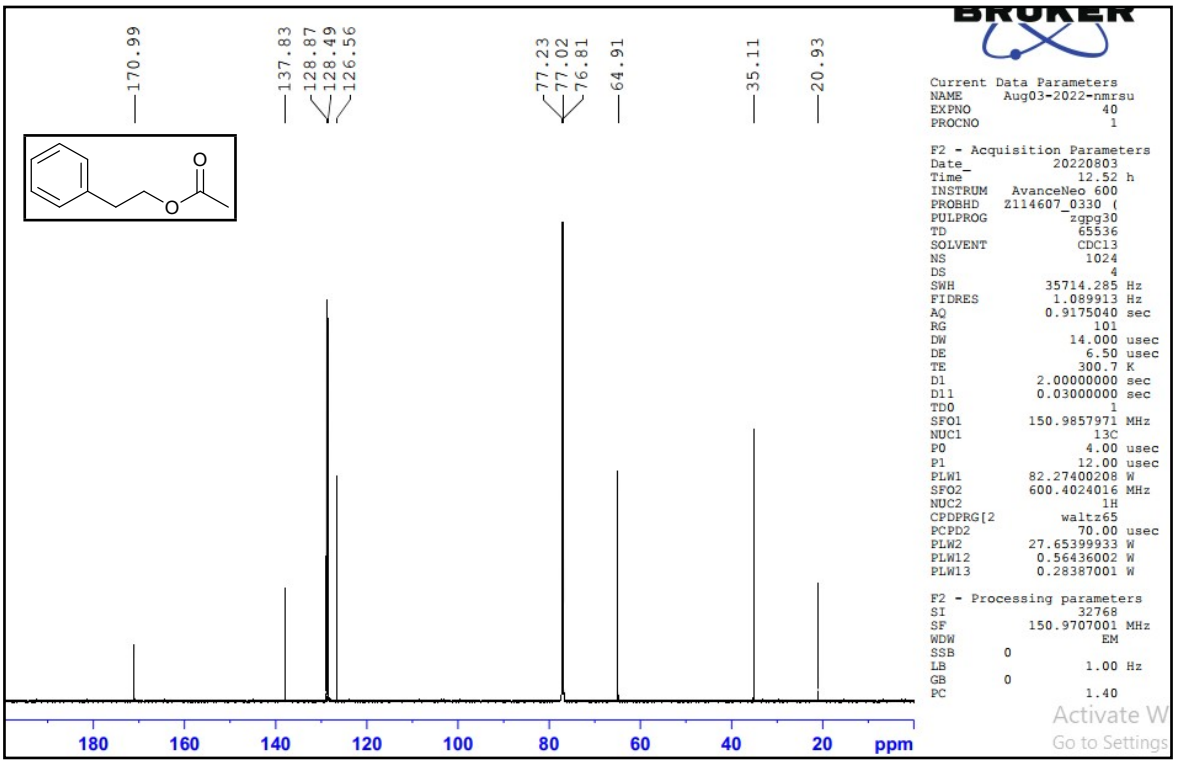
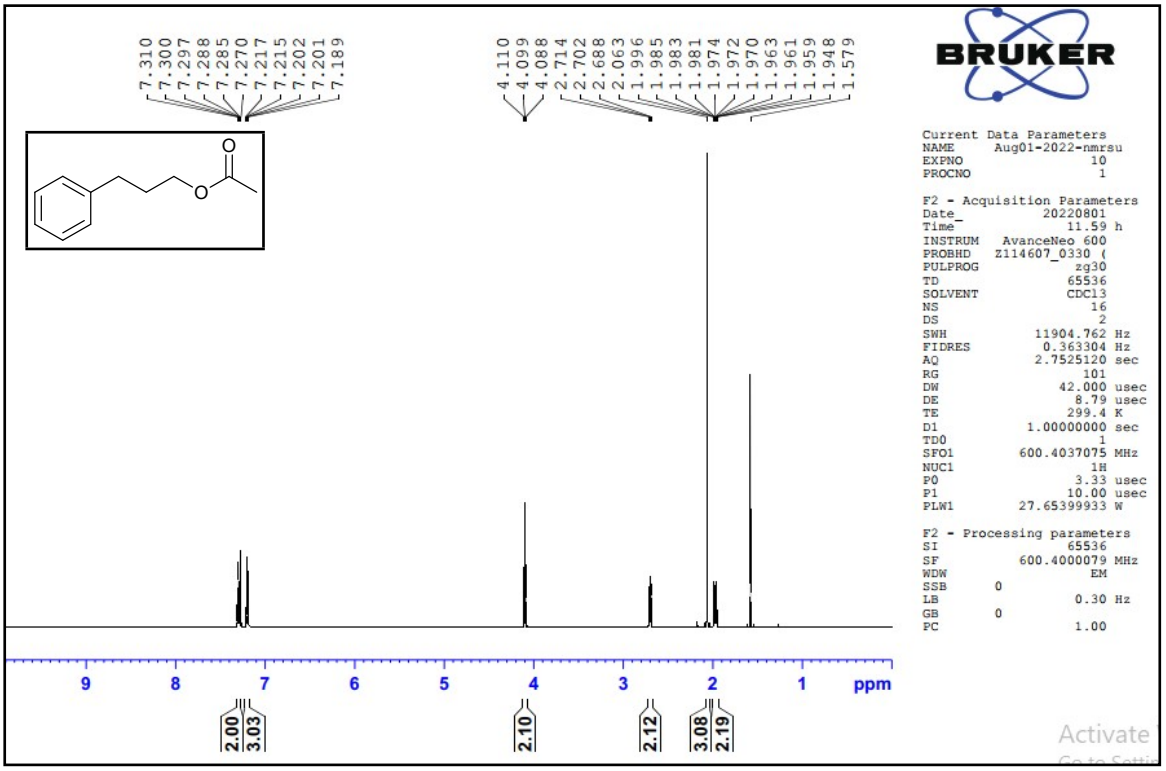
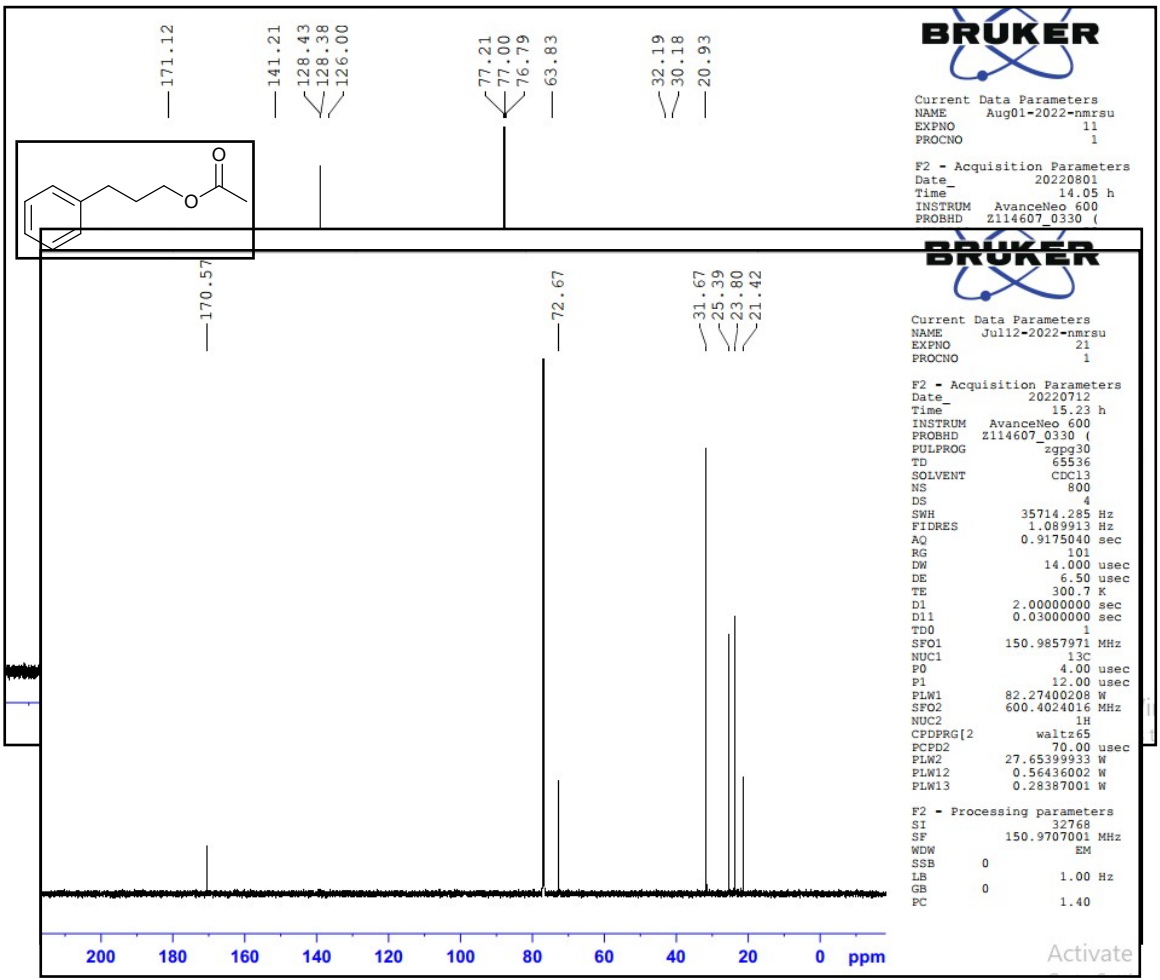
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 11904.762 Hz
FIDRES 0.363304 Hz
AQ 2.7525120 sec
RG 101
DW 42.000 usec
DE 8.79 usec
TE 299.3 K
D1 1.00000000 sec
TD0 1
SFO1 600.4037075 MHz
NUC1 1H
P0 3.33 usec
P1 10.00 usec
PLW1 27.65399933 W
F2 - Processing parameters
SI 65536
SF 600.4000078 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Activate W
Go to Settings


170.99
137.83
128.87
128.49
126.56
77.23
77.02
76.81
64.91
35.11
20.93
O
O
180
160
140
120
100
80
60
40
20
ppm
BRUKER
Current Data Parameters
NAME Aug03-2022-nmrsu
EXPNO 40
PROCNO 1
F2 - Acquisition Parameters
Date_ 20220803
Time 12.52 h
INSTRUM AvanceNeo 600
PROBHD Z114607_0330 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 35714.285 Hz
FIDRES 1.089913 Hz
AQ 0.9175040 sec
RG 101
DW 14.000 usec
DE 6.50 usec
TE 300.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 150.9857971 MHz
NUC1 13C
P0 4.00 usec
P1 12.00 usec
PLW1 82.27400208 W
SFO2 600.4024016 MHz
NUC2 1H
CPDPRG[2 waltz65
PCPD2 70.00 usec
PLW2 27.65399933 W
PLW12 0.56436002 W
PLW13 0.28387001 W
F2 - Processing parameters
SI 32768
SF 150.9707001 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Activate W
Go to Settings



BRUKER

```
Current Data Parameters
NAME      Aug01-2022-nmrsu
EXPNO                   10
PROCNO                   1

F2 - Acquisition Parameters
Date_           20220801
Time              11.59 h
INSTRUM   AvanceNeo 600
PROBHD   Z114607_0330 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           11904.762 Hz
FIDRES         0.363304 Hz
AQ             2.7525120 sec
RG                   101
DW                42.000 usec
DE                 8.79 usec
TE                299.4 K
D1           1.00000000 sec
TD0                    1
SFO1         600.4037075 MHz
NUC1                  1H
P0                 3.33 usec
P1                10.00 usec
PLW1         27.65399933 W

F2 - Processing parameters
SI                 65536
SF           600.4000079 MHz
WDW                   EM
SSB        0
LB                 0.30 Hz
GB         0
PC                 1.00
```

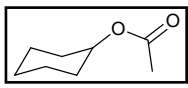


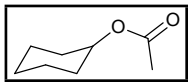
BRUKER

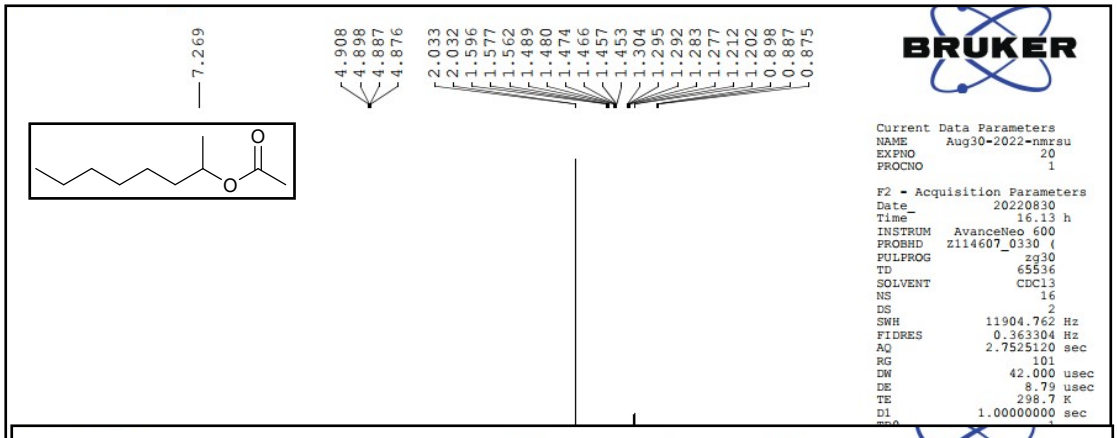
```
Current Data Parameters
NAME      Aug01-2022-nmrsu
EXPNO                   11
PROCNO                   1

F2 - Acquisition Parameters
Date_           20220801
Time              14.05 h
INSTRUM   AvanceNeo 600
PROBHD   Z114607_0330 (
```

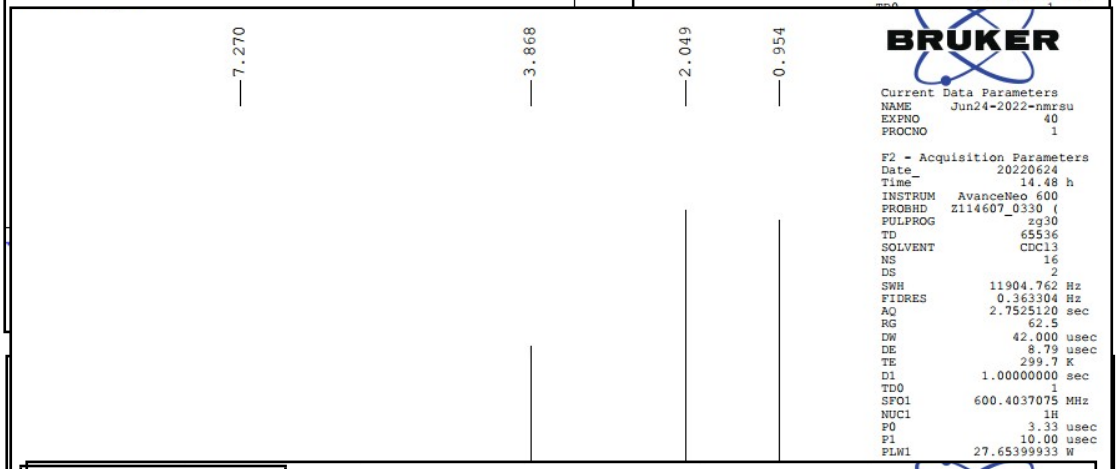
BRUKER

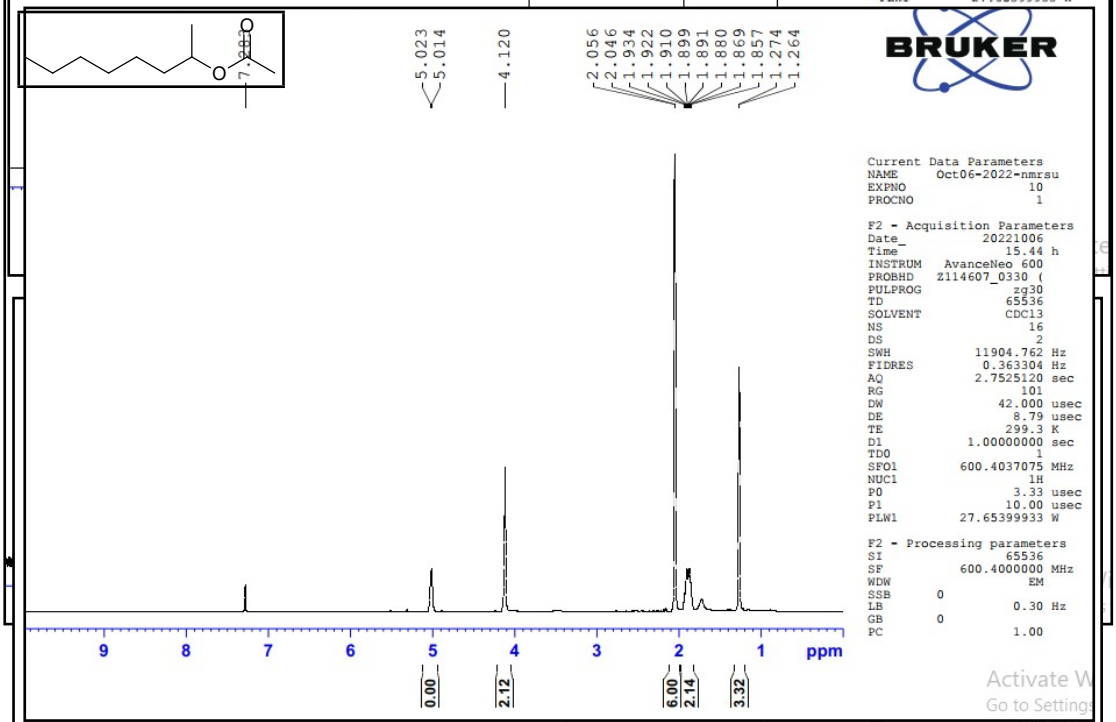
```
Current Data Parameters
NAME      Jul12-2022-nmrsu
EXPNO                   21
PROCNO                   1

F2 - Acquisition Parameters
Date_           20220712
Time              15.23 h
INSTRUM   AvanceNeo 600
PROBHD   Z114607_0330 (
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   800
DS                     4
SWH           35714.285 Hz
FIDRES         1.089913 Hz
AQ             0.9175040 sec
RG                   101
DW                14.000 usec
DE                 6.50 usec
TE                300.7 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1
SFO1         150.9857971 MHz
NUC1                  13C
P0                 4.00 usec
P1                12.00 usec
PLW1         82.27400208 W
SFO2         600.4024016 MHz
NUC2                  1H
CPDPRG[2          waltz65
PCPD2             70.00 usec
PLW2         27.65399933 W
PLW12         0.56436002 W
PLW13         0.28387001 W

F2 - Processing parameters
SI                 32768
SF           150.9707001 MHz
WDW                   EM
SSB        0
LB                 1.00 Hz
GB         0
PC                 1.40
```

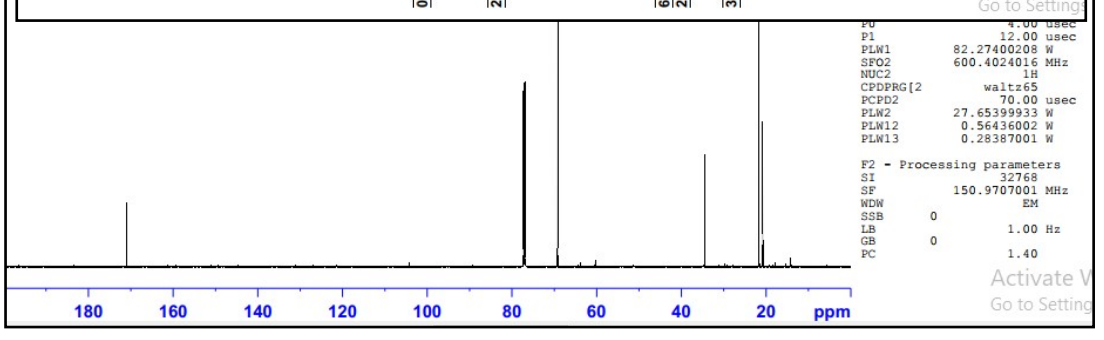


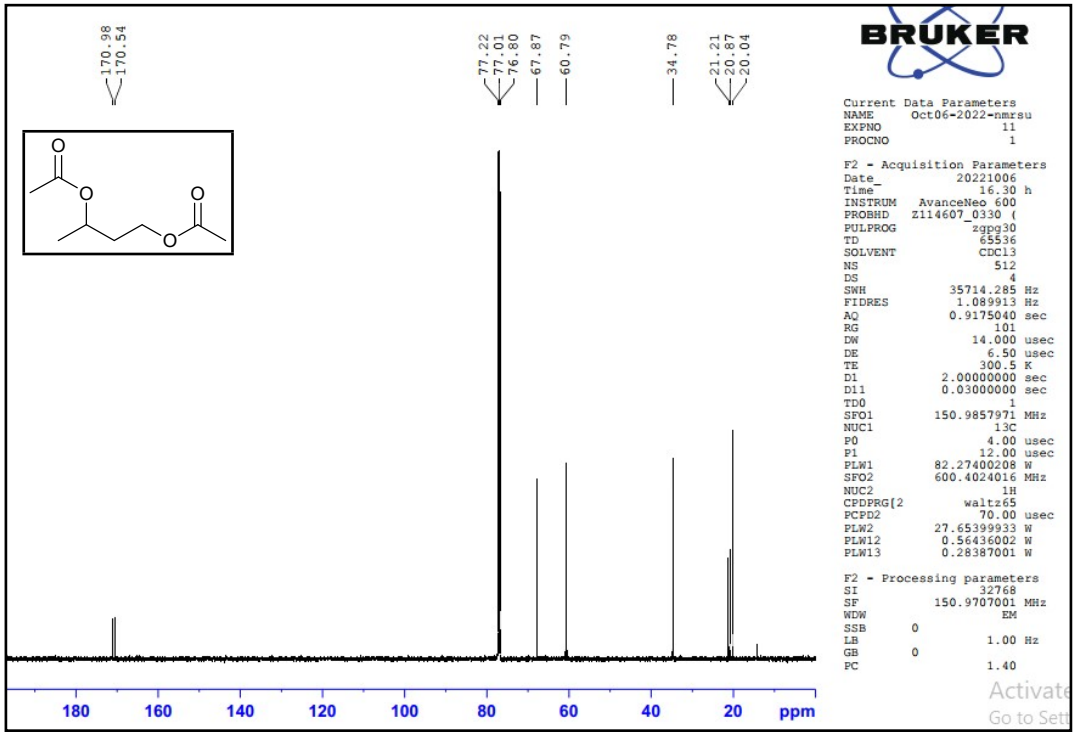



7.269
4.908
4.898
4.887
4.876
2.033
2.032
1.596
1.577
1.562
1.489
1.480
1.474
1.466
1.457
1.453
1.304
1.295
1.292
1.283
1.277
1.212
1.202
0.898
0.887
0.875
BRUKER
Current Data Parameters
NAME Aug30-2022-nmrsu
EXPNO 20
PROCNO 1
F2 - Acquisition Parameters
Date_ 20220830
Time 16.13 h
INSTRUM AvanceNeo 600
PROBHD Z114607_0330 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 11904.762 Hz
FIDRES 0.363304 Hz
AQ 2.7525120 sec
RG 101
DW 42.000 usec
DE 8.79 usec
TE 298.7 K
D1 1.00000000 sec


7.270
3.868
2.049
0.954
BRUKER
Current Data Parameters
NAME Jun24-2022-nmrsu
EXPNO 40
PROCNO 1
F2 - Acquisition Parameters
Date_ 20220624
Time 14.48 h
INSTRUM AvanceNeo 600
PROBHD Z114607_0330 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 11904.762 Hz
FIDRES 0.363304 Hz
AQ 2.7525120 sec
RG 62.5
DW 42.000 usec
DE 8.79 usec
TE 299.7 K
D1 1.00000000 sec
TD0 1
SFO1 600.4037075 MHz
NUC1 1H
P0 3.33 usec
P1 10.00 usec
PLW1 27.65399933 W

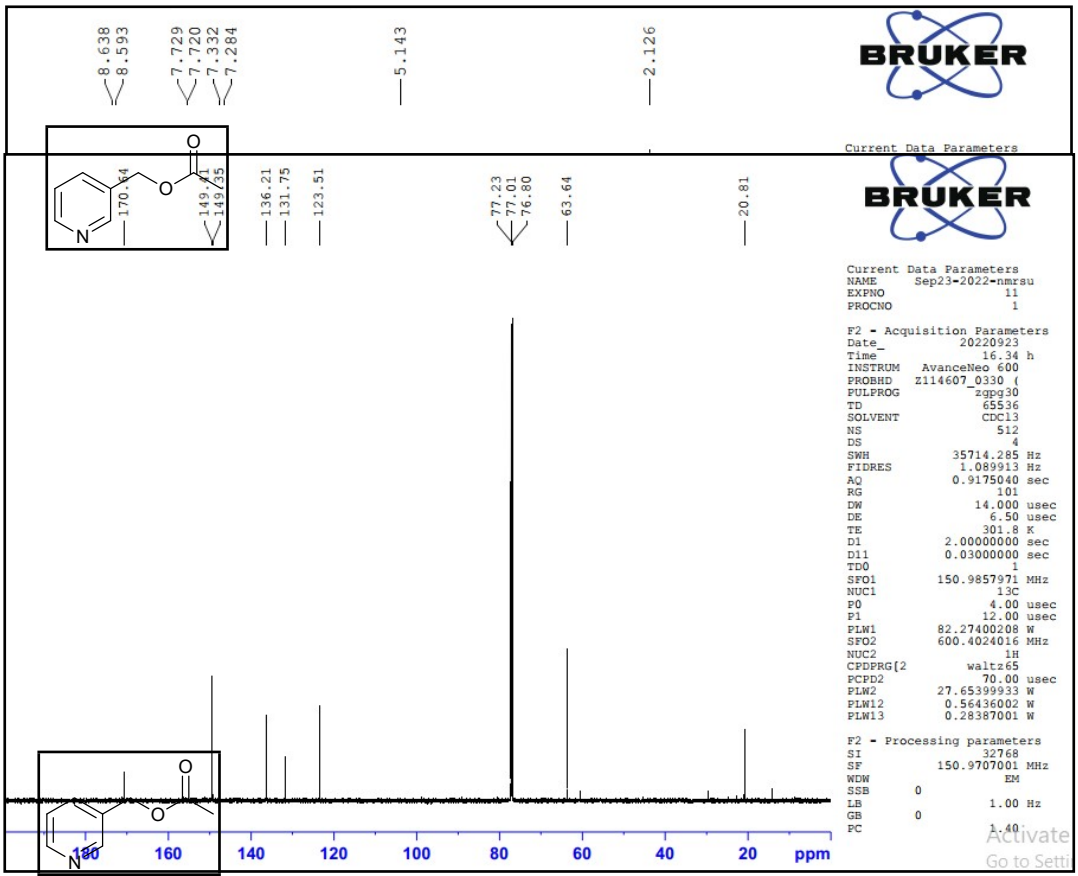

5.023
5.014
4.120
2.056
2.046
1.934
1.922
1.910
1.899
1.891
1.880
1.869
1.857
1.274
1.264
BRUKER
Current Data Parameters
NAME Oct06-2022-nmrsu
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20221006
Time 15.44 h
INSTRUM AvanceNeo 600
PROBHD Z114607_0330 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 11904.762 Hz
FIDRES 0.363304 Hz
AQ 2.7525120 sec
RG 101
DW 42.000 usec
DE 8.79 usec
TE 299.3 K
D1 1.00000000 sec
TD0 1
SFO1 600.4037075 MHz
NUC1 1H
P0 3.33 usec
P1 10.00 usec
PLW1 27.65399933 W
F2 - Processing parameters
SI 65536
SF 600.4000000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9 8 7 6 5 4 3 2 1 ppm
0.00 2.12 6.00 2.14 3.32


P0 4.00 usec
P1 12.00 usec
PLW1 82.27400208 W
SFO2 600.4024016 MHz
NUC2 1H
CPDPRG[2 waltz65
PCPD2 70.00 usec
PLW2 27.65399933 W
PLW12 0.56436002 W
PLW13 0.28387001 W
F2 - Processing parameters
SI 32768
SF 150.9707001 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
180 160 140 120 100 80 60 40 20 ppm



BRUKER

```
Current Data Parameters
NAME      Oct06-2022-nmrsu
EXPNO                   11
PROCNO                   1

F2 - Acquisition Parameters
Date_           20221006
Time              16.30 h
INSTRUM   AvanceNeo 600
PROBHD   Z114607_0330 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  512
DS                    4
SWH           35714.285 Hz
FIDRES         1.089913 Hz
AQ            0.9175040 sec
RG                  101
DW               14.000 usec
DE                6.50 usec
TE                300.5 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        150.9857971 MHz
NUC1                13C
P0                4.00 usec
P1               12.00 usec
PLW1        82.27400208 W
SFO2        600.4024016 MHz
NUC2                1H
CPDPRG[2        waltz65
PCPD2            70.00 usec
PLW2        27.65399933 W
PLW12        0.56436002 W
PLW13        0.28387001 W

F2 - Processing parameters
SI                32768
SF          150.9707001 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

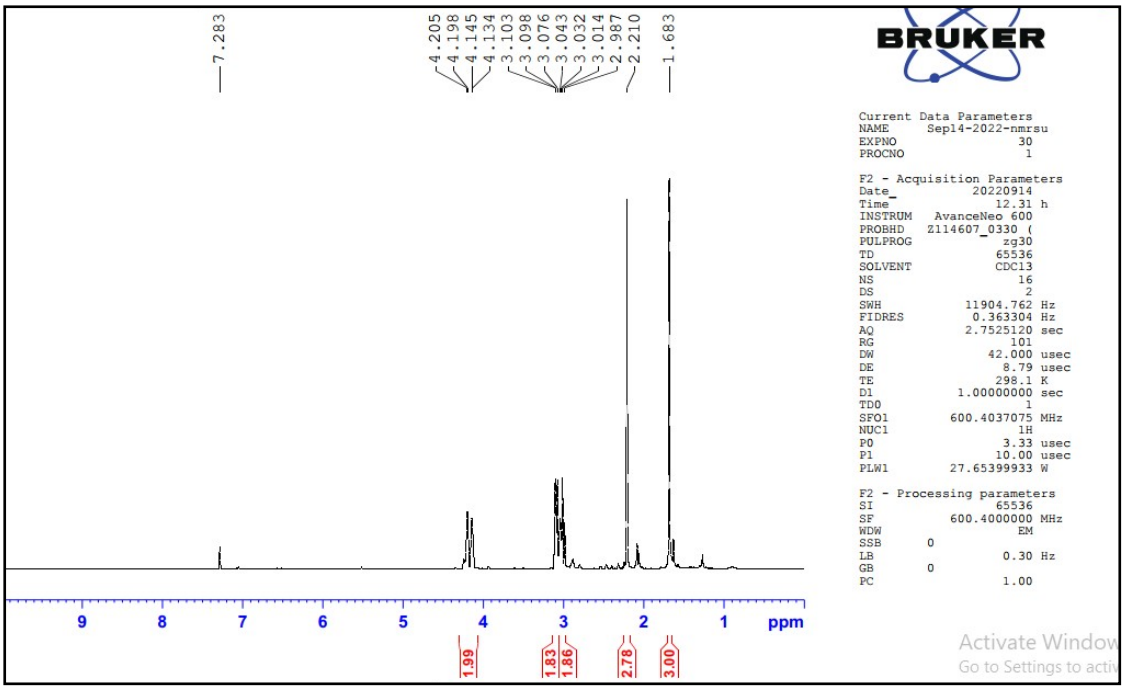



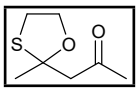
BRUKER

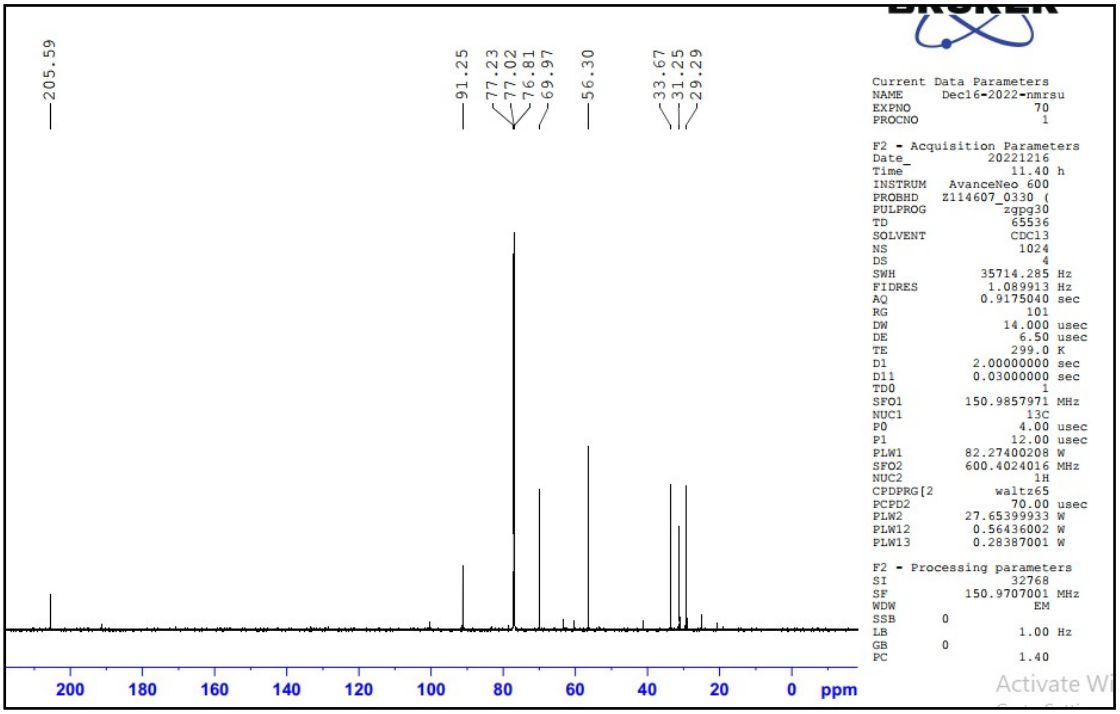
Current Data Parameters

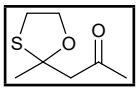
BRUKER

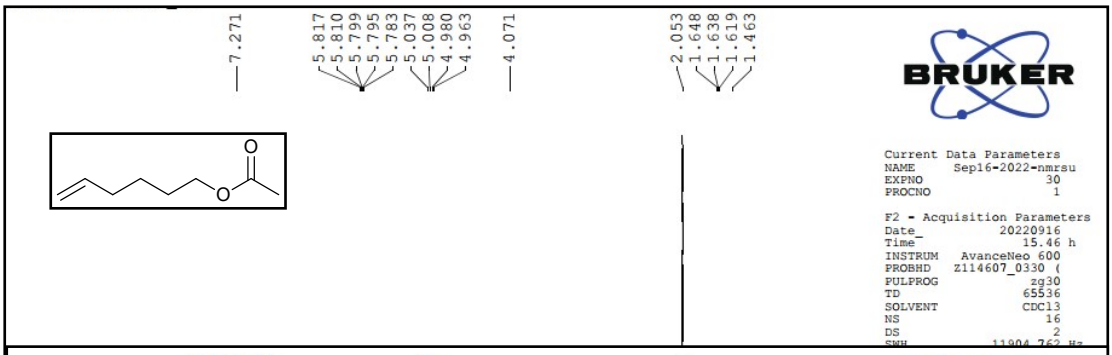
```
Current Data Parameters
NAME      Sep23-2022-nmrsu
EXPNO                   11
PROCNO                   1

F2 - Acquisition Parameters
Date_           20220923
Time              16.34 h
INSTRUM   AvanceNeo 600
PROBHD   Z114607_0330 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  512
DS                    4
SWH           35714.285 Hz
FIDRES         1.089913 Hz
AQ            0.9175040 sec
RG                  101
DW               14.000 usec
DE                6.50 usec
TE                301.8 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        150.9857971 MHz
NUC1                13C
P0                4.00 usec
P1               12.00 usec
PLW1        82.27400208 W
SFO2        600.4024016 MHz
NUC2                1H
CPDPRG[2        waltz65
PCPD2            70.00 usec
PLW2        27.65399933 W
PLW12        0.56436002 W
PLW13        0.28387001 W

F2 - Processing parameters
SI                32768
SF          150.9707001 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

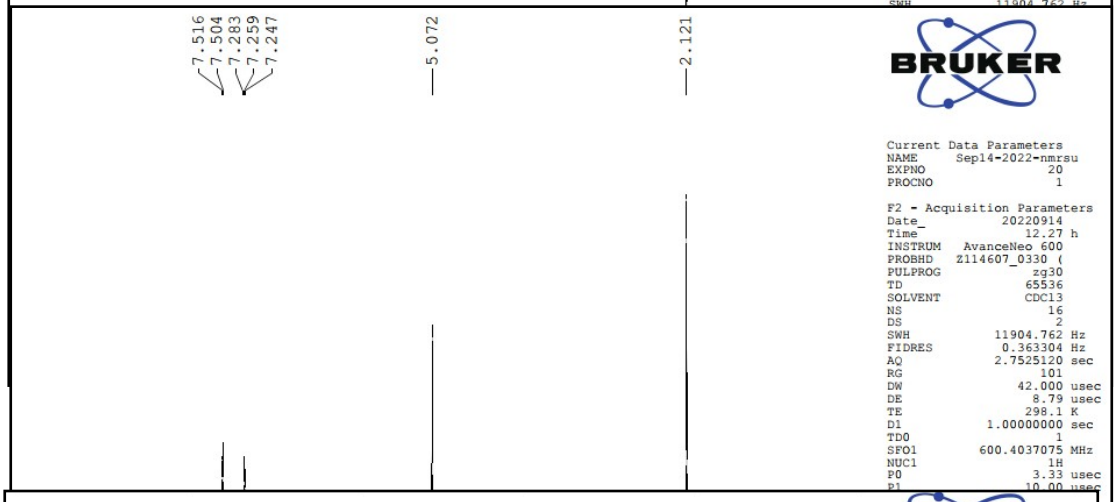

7.283
4.205
4.198
4.145
4.134
3.103
3.098
3.076
3.043
3.032
3.014
2.987
2.210
1.683
BRUKER
Current Data Parameters
NAME Sep14-2022-nmrsu
EXPNO 30
PROCNO 1
F2 - Acquisition Parameters
Date_ 20220914
Time 12.31 h
INSTRUM AvanceNeo 600
PROBHD Z114607_0330 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 11904.762 Hz
FIDRES 0.363304 Hz
AQ 2.7525120 sec
RG 101
DW 42.000 usec
DE 8.79 usec
TE 298.1 K
D1 1.00000000 sec
TD0 1
SFO1 600.4037075 MHz
NUC1 1H
P0 3.33 usec
P1 10.00 usec
PLW1 27.65399933 W
F2 - Processing parameters
SI 65536
SF 600.4000000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9 8 7 6 5 4 3 2 1 ppm
1.99
1.83
1.86
2.78
3.00
Activate Window
Go to Settings to acti

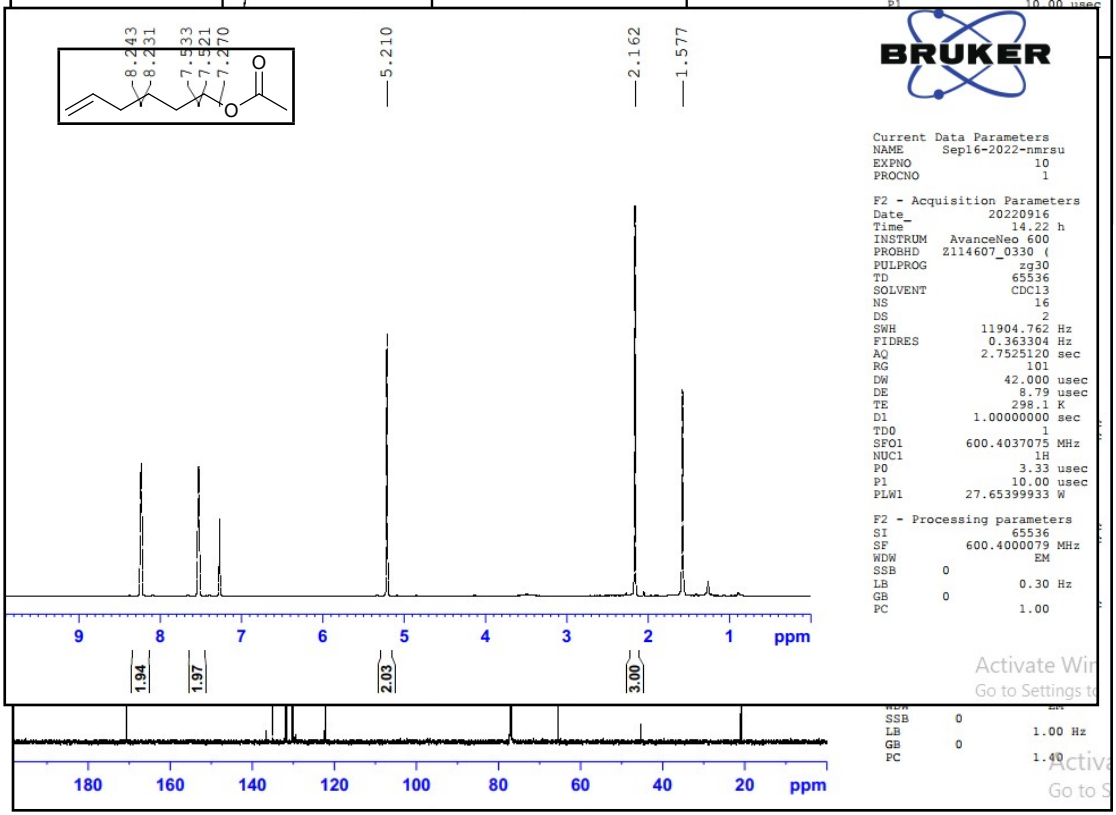

S
O
O

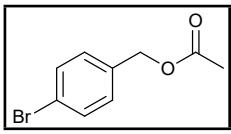

205.59
91.25
77.23
77.02
76.81
69.97
56.30
33.67
31.25
29.29
Current Data Parameters
NAME Dec16-2022-nmrsu
EXPNO 70
PROCNO 1
F2 - Acquisition Parameters
Date_ 20221216
Time 11.40 h
INSTRUM AvanceNeo 600
PROBHD Z114607_0330 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 35714.285 Hz
FIDRES 1.089913 Hz
AQ 0.9175040 sec
RG 101
DW 14.000 usec
DE 6.50 usec
TE 299.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 150.9857971 MHz
NUC1 13C
P0 4.00 usec
P1 12.00 usec
PLW1 82.27400208 W
SFO2 600.4024016 MHz
NUC2 1H
CPDPRG[2 waltz65
PCPD2 70.00 usec
PLW2 27.65399933 W
PLW12 0.56436002 W
PLW13 0.28387001 W
F2 - Processing parameters
SI 32768
SF 150.9707001 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 0 ppm
Activate Wi

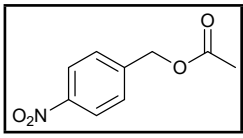

S
O
O

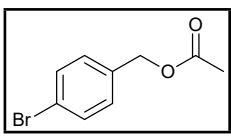

7.271
5.817
5.810
5.799
5.795
5.783
5.037
5.008
4.980
4.963
4.071
2.053
1.648
1.638
1.619
1.463
BRUKER
Current Data Parameters
NAME Sep16-2022-nmrsu
EXPNO 30
PROCNO 1
F2 - Acquisition Parameters
Date_ 20220916
Time 15.46 h
INSTRUM AvanceNeo 600
PROBHD Z114607_0330 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2

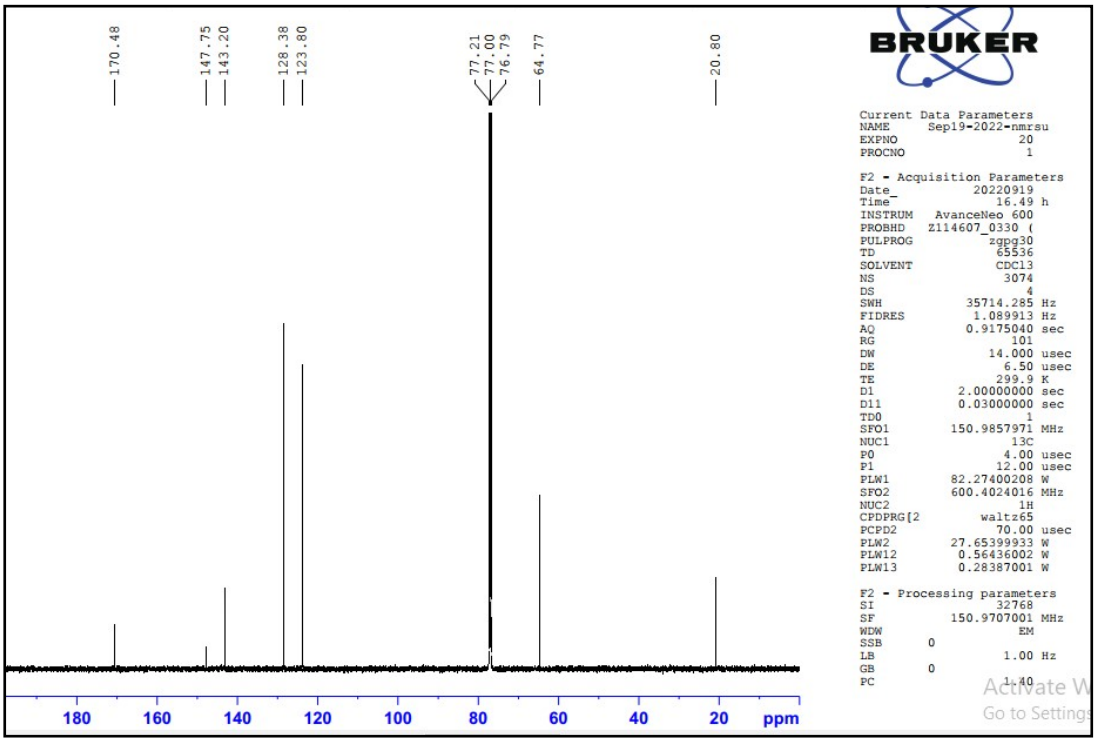

7.516
7.504
7.283
7.259
7.247
5.072
2.121
BRUKER
Current Data Parameters
NAME Sep14-2022-nmrsu
EXPNO 20
PROCNO 1
F2 - Acquisition Parameters
Date_ 20220914
Time 12.27 h
INSTRUM AvanceNeo 600
PROBHD Z114607_0330 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 11904.762 Hz
FIDRES 0.363304 Hz
AQ 2.7525120 sec
RG 101
DW 42.000 usec
DE 8.79 usec
TE 298.1 K
D1 1.00000000 sec
TD0 1
SFO1 600.4037075 MHz
NUC1 1H
P0 3.33 usec


8.243
8.231
7.533
7.521
7.270
5.210
2.162
1.577
BRUKER
Current Data Parameters
NAME Sep16-2022-nmrsu
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20220916
Time 14.22 h
INSTRUM AvanceNeo 600
PROBHD Z114607_0330 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 11904.762 Hz
FIDRES 0.363304 Hz
AQ 2.7525120 sec
RG 101
DW 42.000 usec
DE 8.79 usec
TE 298.1 K
D1 1.00000000 sec
TD0 1
SFO1 600.4037075 MHz
NUC1 1H
P0 3.33 usec
P1 10.00 usec
PLW1 27.65399933 W
F2 - Processing parameters
SI 65536
SF 600.4000079 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9 8 7 6 5 4 3 2 1 ppm
1.94 1.97 2.03 3.00
180 160 140 120 100 80 60 40 20 ppm
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


Br

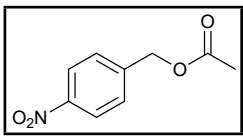

O2N

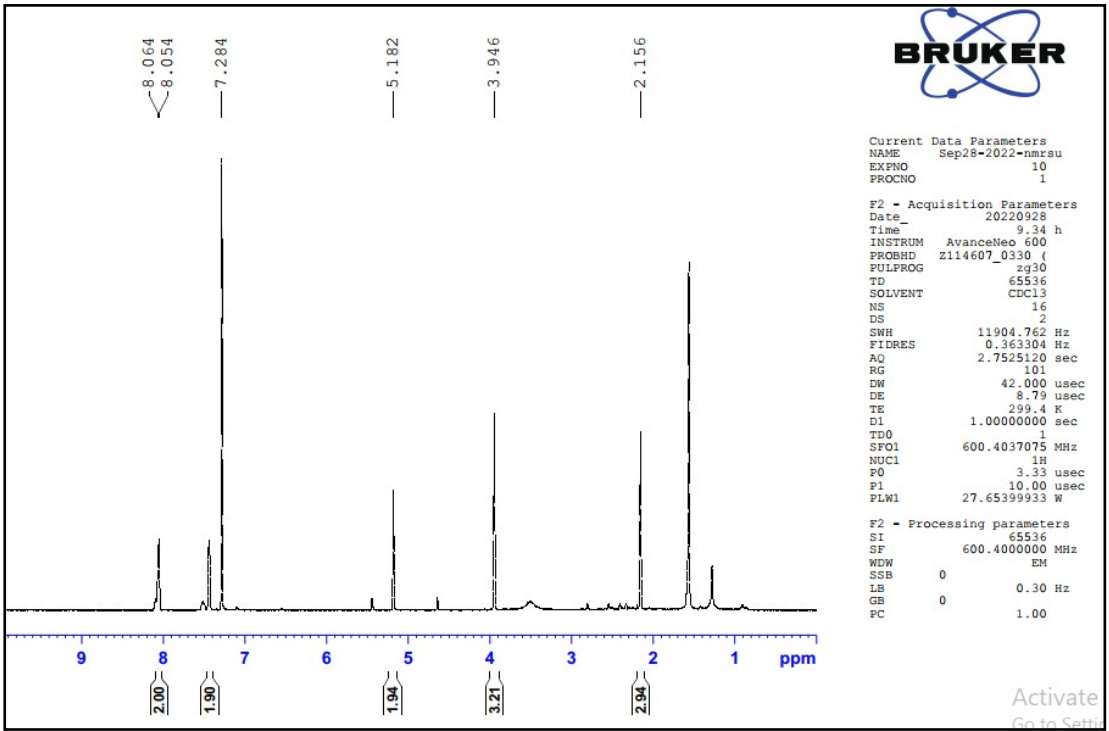

Br



BRUKER

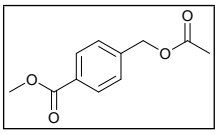
```
Current Data Parameters
NAME      Sep19-2022-nmrsu
EXPNO                   20
PROCNO                   1

F2 - Acquisition Parameters
Date_           20220919
Time              16.49 h
INSTRUM   AvanceNeo 600
PROBHD   Z114607_0330 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 3074
DS                    4
SWH           35714.285 Hz
FIDRES         1.089913 Hz
AQ            0.9175040 sec
RG                  101
DW               14.000 usec
DE                6.50 usec
TE               299.9 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        150.9857971 MHz
NUC1               13C
P0                4.00 usec
P1               12.00 usec
PLW1        82.27400208 W
SFO2        600.4024016 MHz
NUC2               1H
CPDPRG[2        waltz65
PCPD2            70.00 usec
PLW2        27.65399933 W
PLW12        0.56436002 W
PLW13        0.28387001 W

F2 - Processing parameters
SI                32768
SF          150.9707001 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

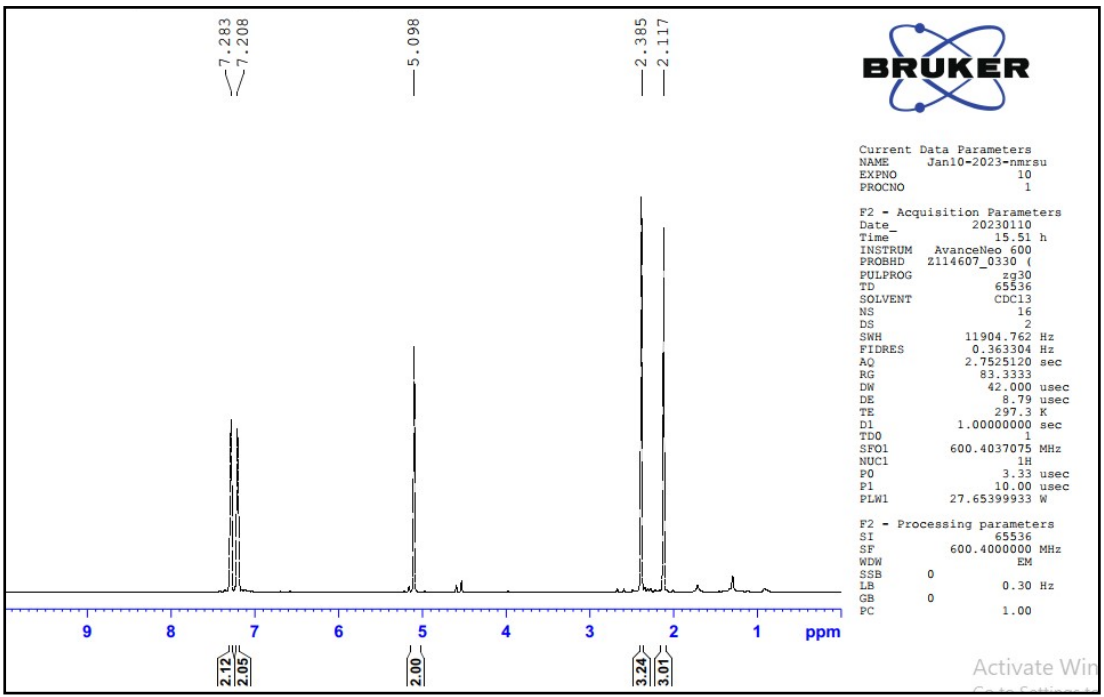


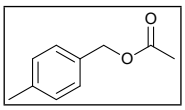


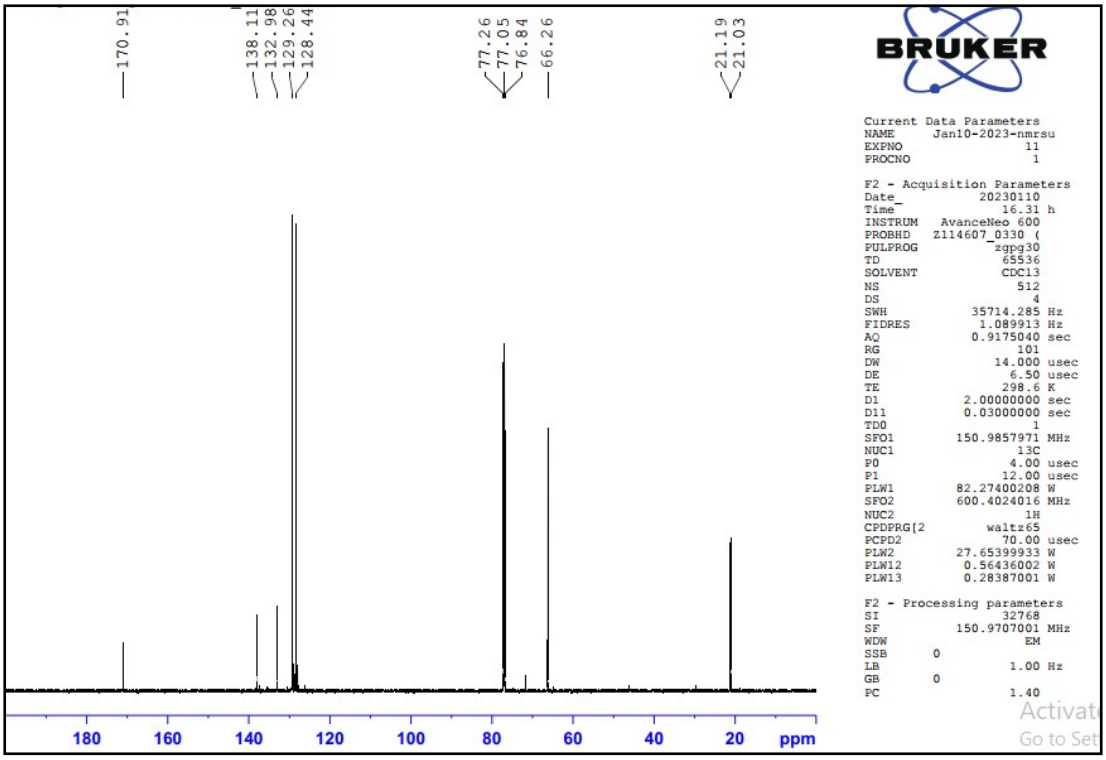
BRUKER

```
Current Data Parameters
NAME      Sep28-2022-nmrsu
EXPNO                   10
PROCNO                   1

F2 - Acquisition Parameters
Date_           20220928
Time               9.34 h
INSTRUM   AvanceNeo 600
PROBHD   Z114607_0330 (
PULPROG             zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           11904.762 Hz
FIDRES         0.363304 Hz
AQ            2.7525120 sec
RG                  101
DW               42.000 usec
DE                8.79 usec
TE               299.4 K
D1           1.00000000 sec
TD0                   1
SFO1        600.4037075 MHz
NUC1               1H
P0                3.33 usec
P1               10.00 usec
PLW1        27.65399933 W

F2 - Processing parameters
SI                65536
SF          600.4000000 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```




7.283
7.208
5.098
2.385
2.117
BRUKER
Current Data Parameters
NAME Jan10-2023-nmrsu
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20230110
Time 15.51 h
INSTRUM AvanceNeo 600
PROBHD Z114607_0330 (
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 2
SWH 11904.762 Hz
FIDRES 0.363304 Hz
AQ 2.7525120 sec
RG 83.3333
DW 42.000 usec
DE 8.79 usec
TE 297.3 K
D1 1.00000000 sec
TD0 1
SFO1 600.4037075 MHz
NUC1 1H
P0 3.33 usec
P1 10.00 usec
PLW1 27.65399933 W
F2 - Processing parameters
SI 65536
SF 600.4000000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
ppm
2.12
2.05
2.00
3.24
3.01

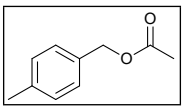


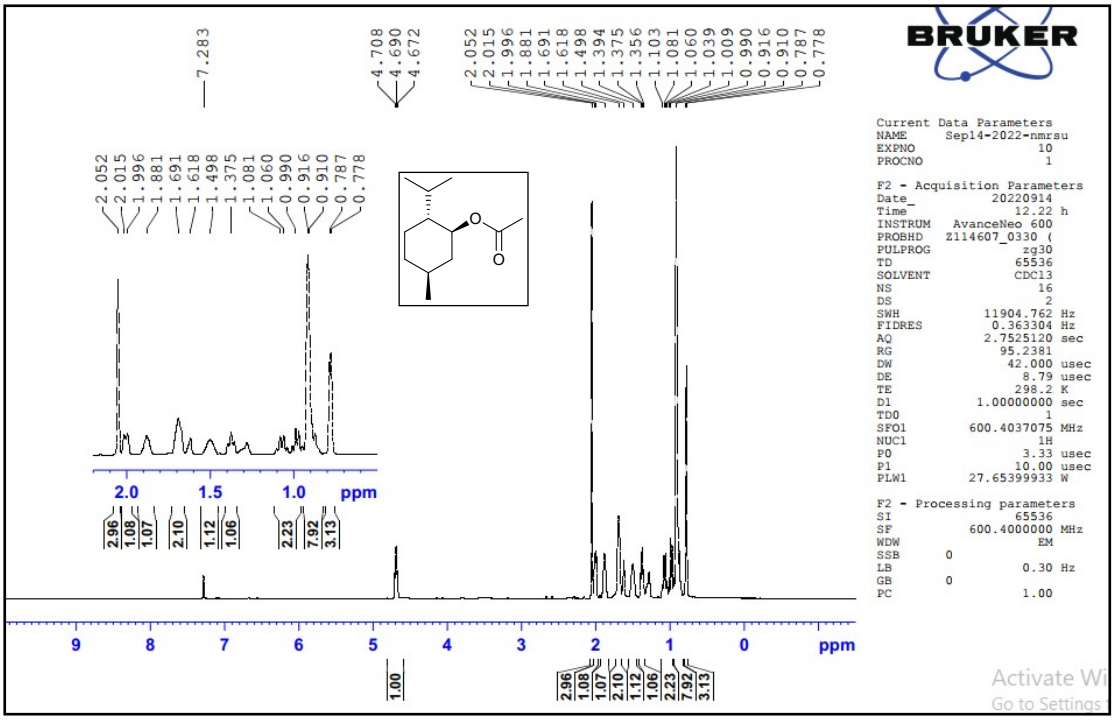


Current Data Parameters
NAME Jan10-2023-nmrsu
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20230110
Time 16.31 h
INSTRUM AvanceNeo 600
PROBHD Z114607_0330 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 35714.285 Hz
FIDRES 1.089913 Hz
AQ 0.9175040 sec
RG 101
DW 14.000 usec
DE 6.50 usec
TE 298.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 150.9857971 MHz
NUC1 13C
P0 4.00 usec
P1 12.00 usec
PLW1 82.27400208 W
SFO2 600.4024016 MHz
NUC2 1H
CPDPRG[2 waltz65
PCPD2 70.00 usec
PLW2 27.65399933 W
PLW12 0.56436002 W
PLW13 0.28387001 W

F2 - Processing parameters
SI 32768
SF 150.9707001 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

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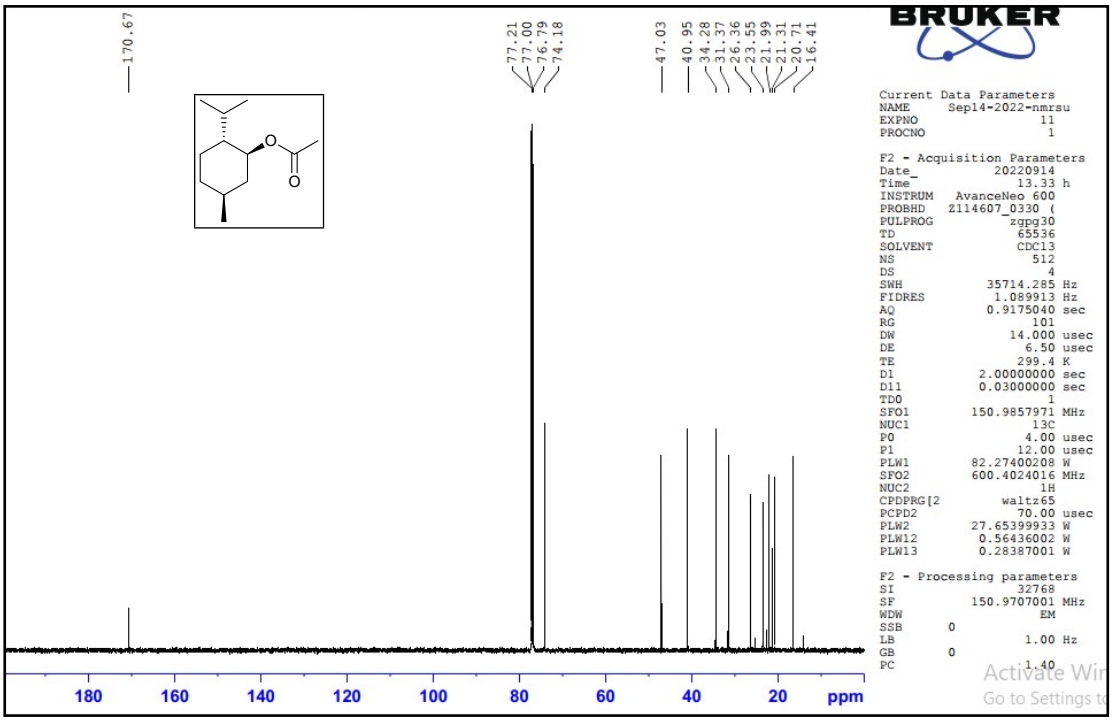
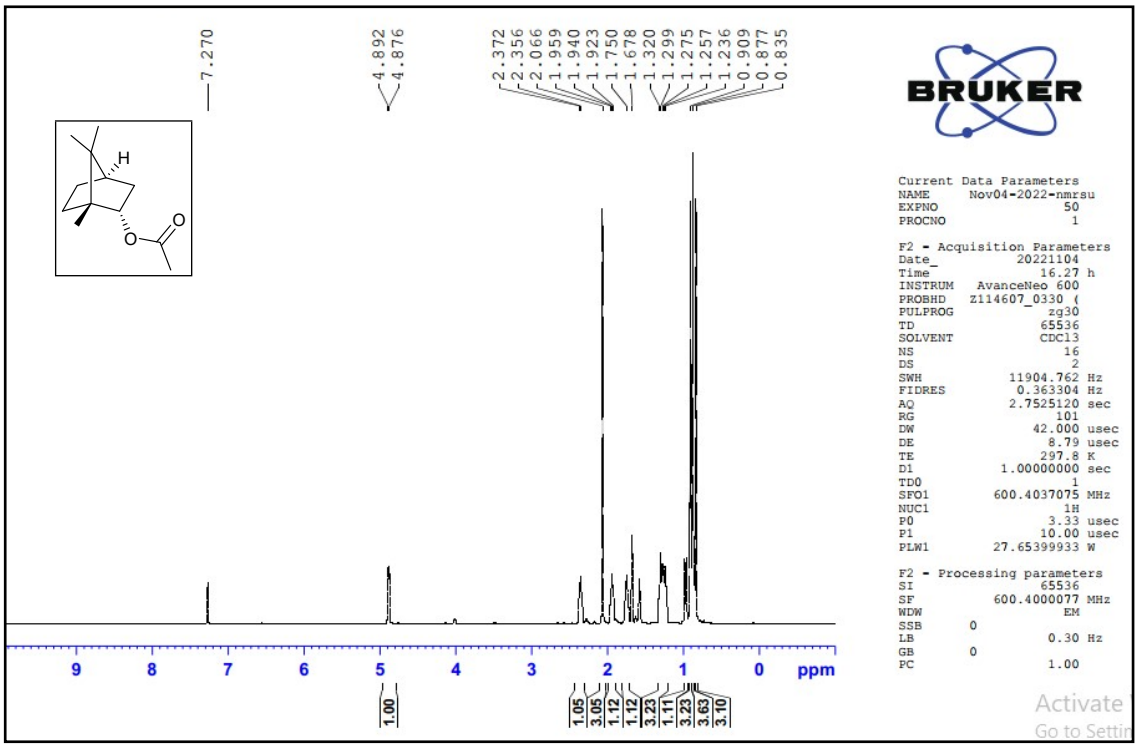
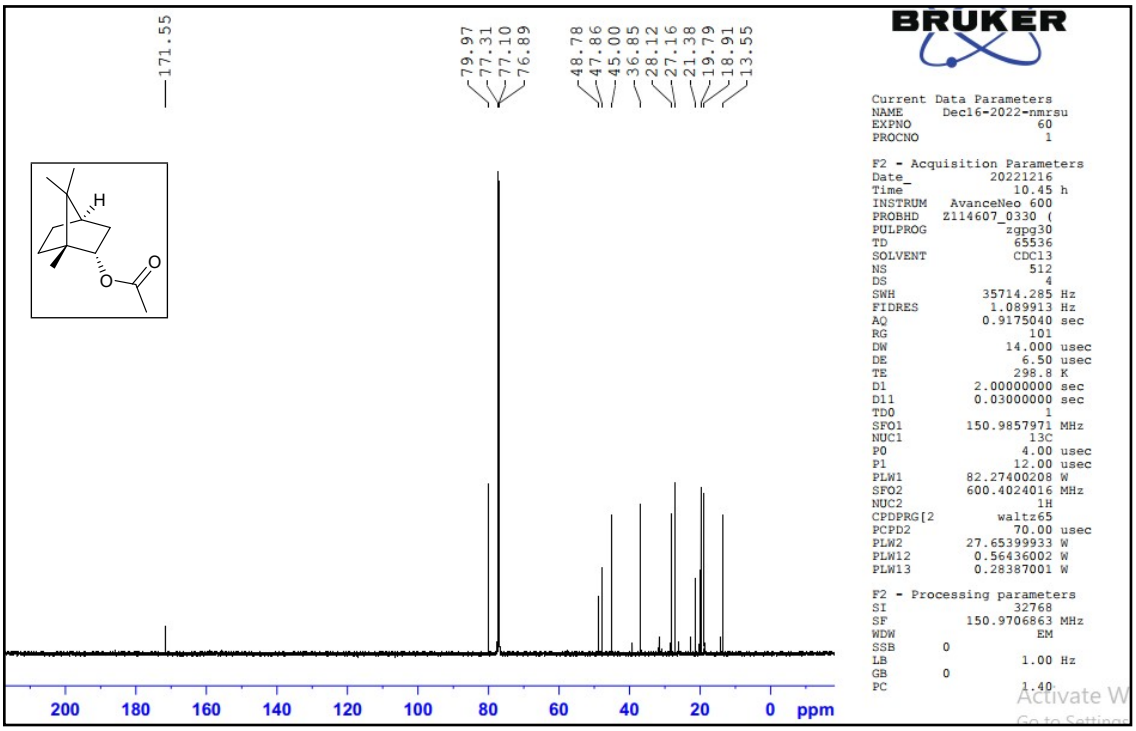
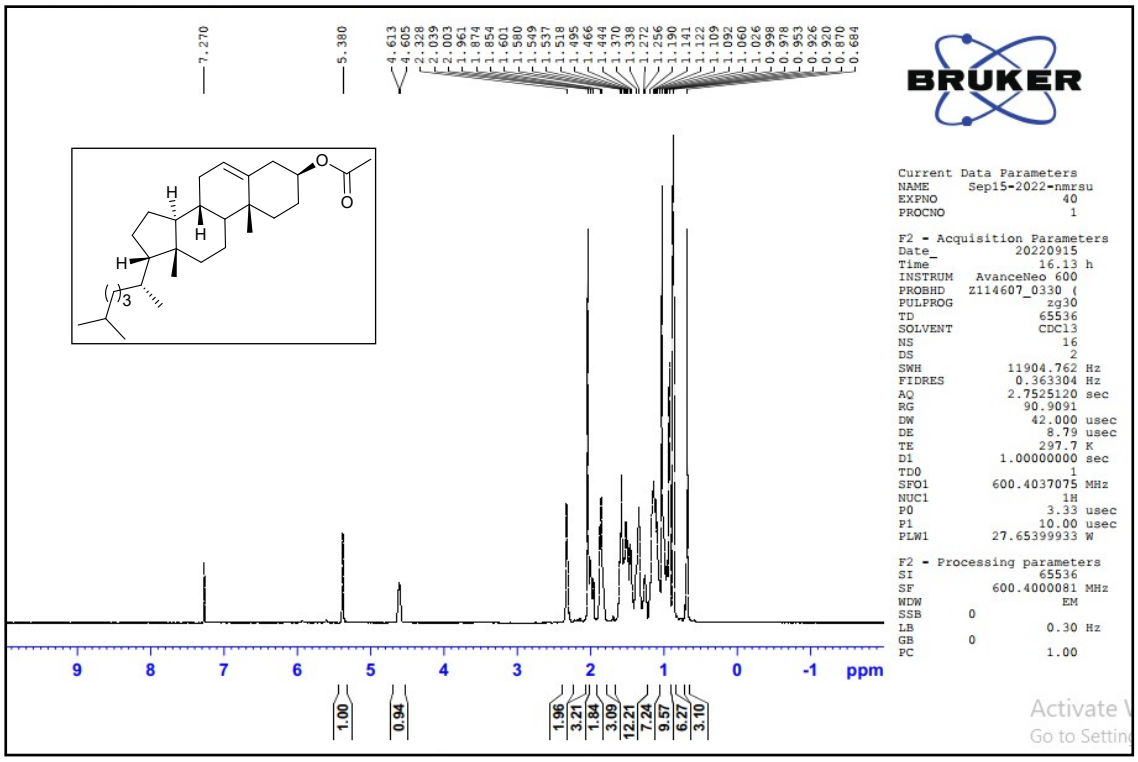
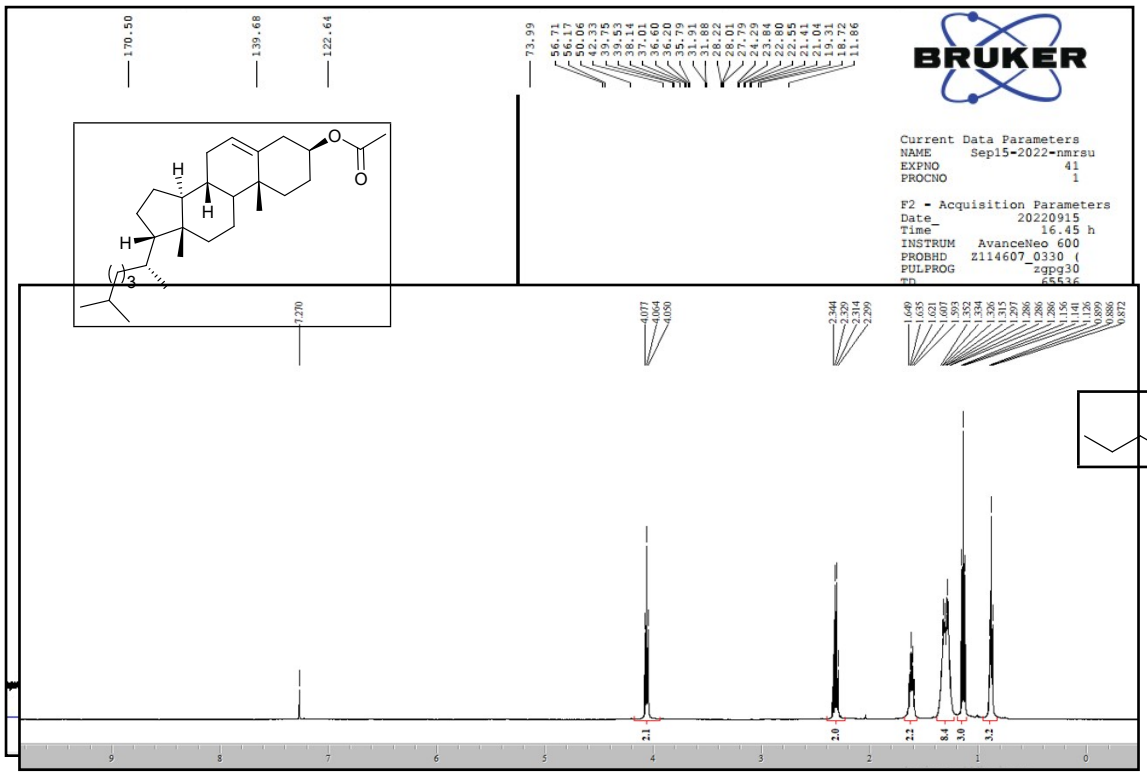
4.708
4.690
4.672
2.052
2.015
1.996
1.881
1.691
1.618
1.498
1.394
1.375
1.356
1.103
1.081
1.060
1.039
1.009
0.990
0.916
0.910
0.787
0.778
2.0
1.5
1.0
ppm
2.96
1.08
1.07
2.10
1.12
1.06
2.23
7.92
3.13
9
8
7
6
5
4
3
2
1
0
ppm
1.00
2.96
1.08
1.07
2.10
1.12
1.06
2.23
7.92
3.13
BRUKER
Current Data Parameters
NAME Sep14-2022-nmrsu
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20220914
Time 12.22 h
INSTRUM AvanceNeo 600
PROBHD Z114607_0330 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 11904.762 Hz
FIDRES 0.363304 Hz
AQ 2.7525120 sec
RG 95.2381
DW 42.000 usec
DE 8.79 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
SFO1 600.4037075 MHz
NUC1 1H
P0 3.33 usec
P1 10.00 usec
PLW1 27.65399933 W
F2 - Processing parameters
SI 65536
SF 600.4000000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

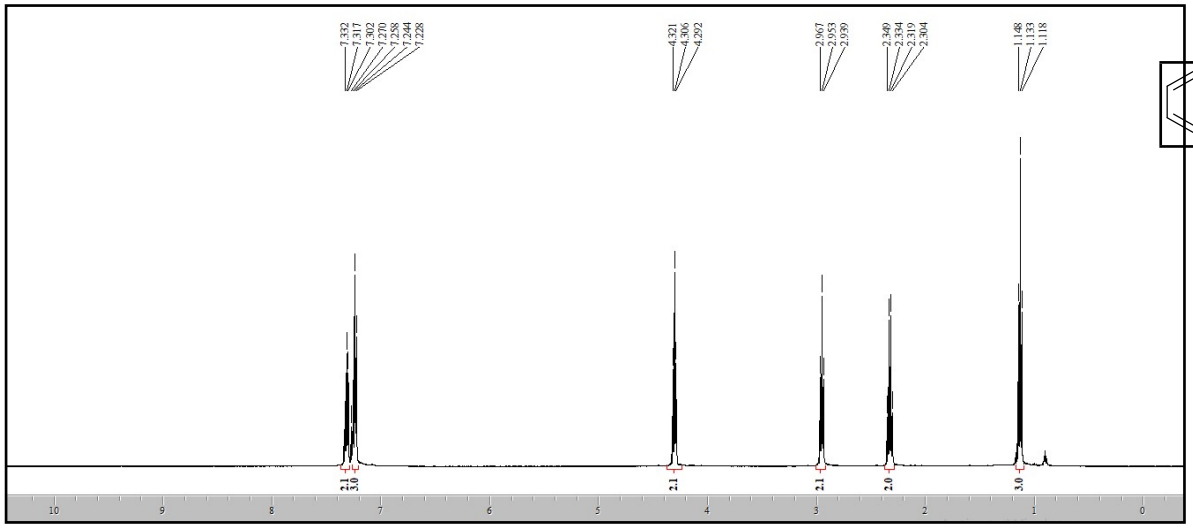

170.67
77.21
77.00
76.79
74.18
47.03
40.95
34.28
31.37
26.36
23.55
21.99
21.31
20.71
16.41
180
160
140
120
100
80
60
40
20
ppm
BRUKER
Current Data Parameters
NAME Sep14-2022-nmrsu
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20220914
Time 13.33 h
INSTRUM AvanceNeo 600
PROBHD Z114607_0330 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 35714.285 Hz
FIDRES 1.089913 Hz
AQ 0.9175040 sec
RG 101
DW 14.000 usec
DE 6.50 usec
TE 299.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 150.9857971 MHz
NUC1 13C
P0 4.00 usec
P1 12.00 usec
PLW1 82.27400208 W
SFO2 600.4024016 MHz
NUC2 1H
CPDPRG[2 waltz65
PCPD2 70.00 usec
PLW2 27.65399933 W
PLW12 0.56436002 W
PLW13 0.28387001 W
F2 - Processing parameters
SI 32768
SF 150.9707001 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

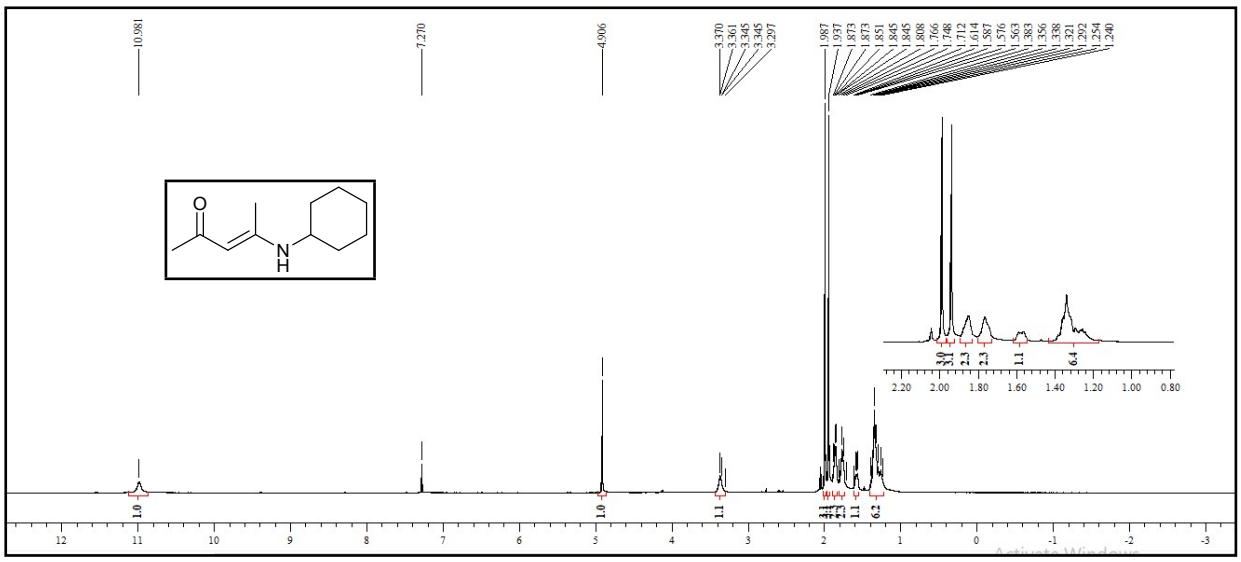

7.270
4.892
4.876
2.372
2.356
2.066
1.959
1.940
1.923
1.750
1.678
1.320
1.299
1.275
1.257
1.236
0.909
0.877
0.835
H
O
O
9
8
7
6
5
4
3
2
1
0
ppm
1.00
1.05
3.05
1.12
1.12
3.23
1.11
3.23
3.63
3.10
BRUKER
Current Data Parameters
NAME Nov04-2022-nmrsu
EXPNO 50
PROCNO 1
F2 - Acquisition Parameters
Date_ 20221104
Time 16.27 h
INSTRUM AvanceNeo 600
PROBHD Z114607_0330 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 11904.762 Hz
FIDRES 0.363304 Hz
AQ 2.7525120 sec
RG 101
DW 42.000 usec
DE 8.79 usec
TE 297.8 K
D1 1.00000000 sec
TD0 1
SFO1 600.4037075 MHz
NUC1 1H
P0 3.33 usec
P1 10.00 usec
PLW1 27.65399933 W
F2 - Processing parameters
SI 65536
SF 600.4000077 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

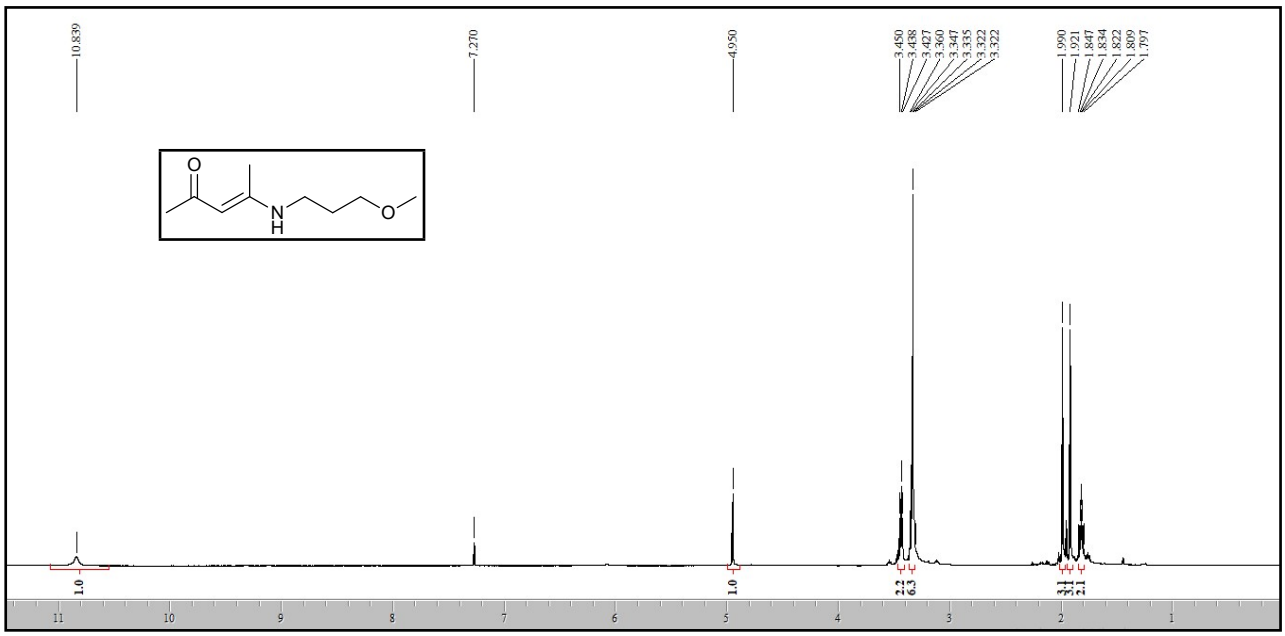

171.55
79.97
77.31
77.10
76.89
48.78
47.86
45.00
36.85
28.12
27.16
21.38
19.79
18.91
13.55
H
O
O
200
180
160
140
120
100
80
60
40
20
0
ppm
BRUKER
Current Data Parameters
NAME Dec16-2022-nmrsu
EXPNO 60
PROCNO 1
F2 - Acquisition Parameters
Date_ 20221216
Time 10.45 h
INSTRUM AvanceNeo 600
PROBHD Z114607_0330 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 35714.285 Hz
FIDRES 1.089913 Hz
AQ 0.9175040 sec
RG 101
DW 14.000 usec
DE 6.50 usec
TE 298.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 150.9857971 MHz
NUC1 13C
P0 4.00 usec
P1 12.00 usec
PLW1 82.27400208 W
SFO2 600.4024016 MHz
NUC2 1H
CPDPRG[2 waltz65
PCPD2 70.00 usec
PLW2 27.65399933 W
PLW12 0.56436002 W
PLW13 0.28387001 W
F2 - Processing parameters
SI 32768
SF 150.9706863 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


7.270
5.380
4.613
4.605
2.328
2.039
2.003
1.961
1.874
1.854
1.601
1.580
1.549
1.537
1.518
1.495
1.466
1.444
1.370
1.338
1.272
1.256
1.190
1.141
1.122
1.109
1.092
1.060
1.026
0.998
0.978
0.953
0.926
0.920
0.870
0.684
BRUKER
Current Data Parameters
NAME Sep15-2022-nmrsu
EXPNO 40
PROCNO 1
F2 - Acquisition Parameters
Date_ 20220915
Time 16.13 h
INSTRUM AvanceNeo 600
PROBHD Z114607_0330 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 11904.762 Hz
FIDRES 0.363304 Hz
AQ 2.7525120 sec
RG 90.9091
DW 42.000 usec
DE 8.79 usec
TE 297.7 K
D1 1.00000000 sec
TD0 1
SFO1 600.4037075 MHz
NUC1 1H
P0 3.33 usec
P1 10.00 usec
PLW1 27.65399933 W
F2 - Processing parameters
SI 65536
SF 600.4000081 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9 8 7 6 5 4 3 2 1 0 -1 ppm
1.00
0.94
1.96
3.21
1.84
3.09
12.21
7.24
9.57
6.27
3.10


170.50
139.68
122.64
73.99
56.71
56.17
50.06
42.33
39.75
39.53
38.14
37.01
36.60
36.20
35.79
31.91
31.88
28.22
28.01
27.79
24.29
23.84
22.80
22.55
21.41
21.04
19.31
18.72
11.86
BRUKER
Current Data Parameters
NAME Sep15-2022-nmrsu
EXPNO 41
PROCNO 1
F2 - Acquisition Parameters
Date_ 20220915
Time 16.45 h
INSTRUM AvanceNeo 600
PROBHD Z114607_0330 (
PULPROG zgpg30
7.270
4.077
4.064
4.050
2.344
2.329
2.314
2.299
2.1
2.0
2.2
8.4
3.0
3.2


7.332
7.317
7.302
7.270
7.258
7.244
7.228
4.321
4.306
4.292
2.967
2.953
2.939
2.349
2.334
2.319
2.304
1.148
1.133
1.118
2.1
3.0
2.1
2.1
2.0
3.0
10
9
8
7
6
5
4
3
2
1
0
O
O


10.981
7.270
4.906
3.370
3.361
3.345
3.345
3.297
1.987
1.937
1.873
1.873
1.851
1.845
1.845
1.808
1.766
1.748
1.712
1.614
1.587
1.576
1.563
1.383
1.356
1.338
1.321
1.292
1.254
1.240
O
N
H
1.0
1.0
1.1
3.1
2.5
1.1
6.2
3.0
2.3
2.3
1.1
6.4


10.839
7.270
4.950
3.450
3.438
3.427
3.360
3.347
3.335
3.322
3.322
1.990
1.971
1.847
1.834
1.822
1.809
1.797
O
N
H
O
1.0
1.0
2.2
6.3
3.1
3.1
2.1
11
10
9
8
7
6
5
4
3
2
1

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