Supplementary data

Alkaline earth metal-based metal-organic framework: Hydrothermal synthesis, X-ray structure and heterogeneously catalyzed Claisen–Schmidt reaction

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Table S1 Hydrogen bonding interactions for complexes **1** and **2** [Å, °]

D—H···A	*d*(D—H)	*d*(H...A)	*d*(D...A)	<(DHA)	Symmetry transform
N1—H1···O1[a]	0.837(18)	2.344(19)	2.6529(15)	102.4(14)	-
N1—H1···O3[a]	0.837(18)	2.253(19)	2.6235(14)	107.0(15)	1+x, y, -1+z
N1—H1···O1[a]	0.837(18)	2.387(17)	3.0215(15)	133.1(16)	-x, 1-y, 3-z
O7—H4···O4[a]	0.84(3)	1.93(3)	2.6683(17)	146(3)	-
O7—H5···O6[a]	0.81(3)	2.09(3)	2.7414(14)	138(2)	1+x, y, z
O6—H6···O4[a]	0.89(2)	1.80(2)	2.6920(15)	178(2)	x, 3/2-y, 1/2+z
O6—H7···O2[a]	0.78(2)	2.00(2)	2.7657(14)	170(3)	1+x, y, z
N1—H1···O2[b]	0.8600	2.2300	2.603(2)	106.00	-
N1—H1···O4[b]	0.8600	2.2500	2.624(2)	106.00	-1/2+x, -1/2+y, z

a: Compound **1**, b: Compound **2**

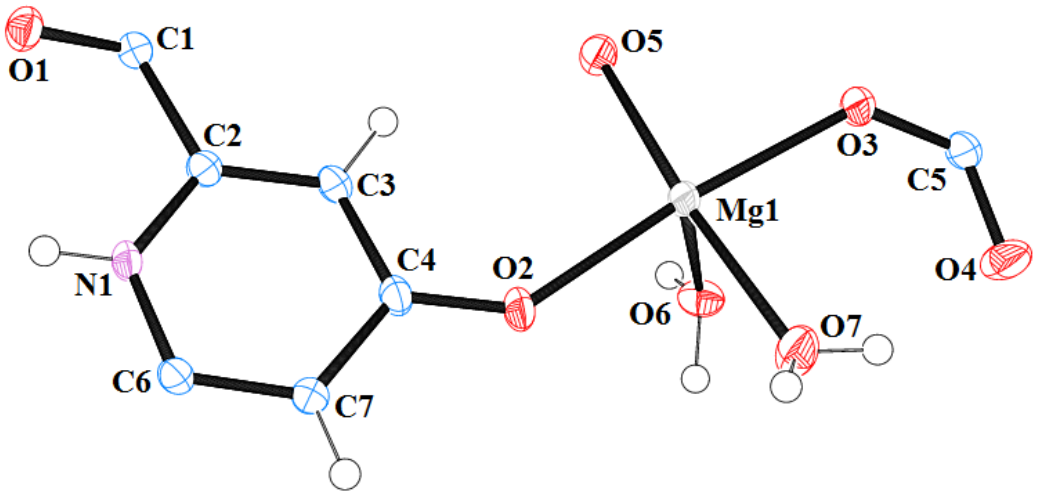


Fig. S1. ORTEP diagram of compound **1** with 40% ellipsoid probability**.**

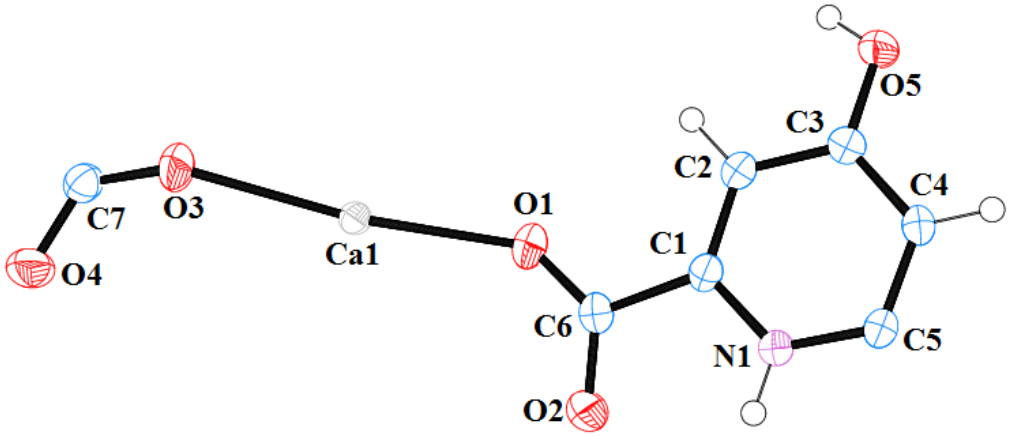


Fig. S2. ORTEP diagram of compound **2** with 40% ellipsoid probability**.**

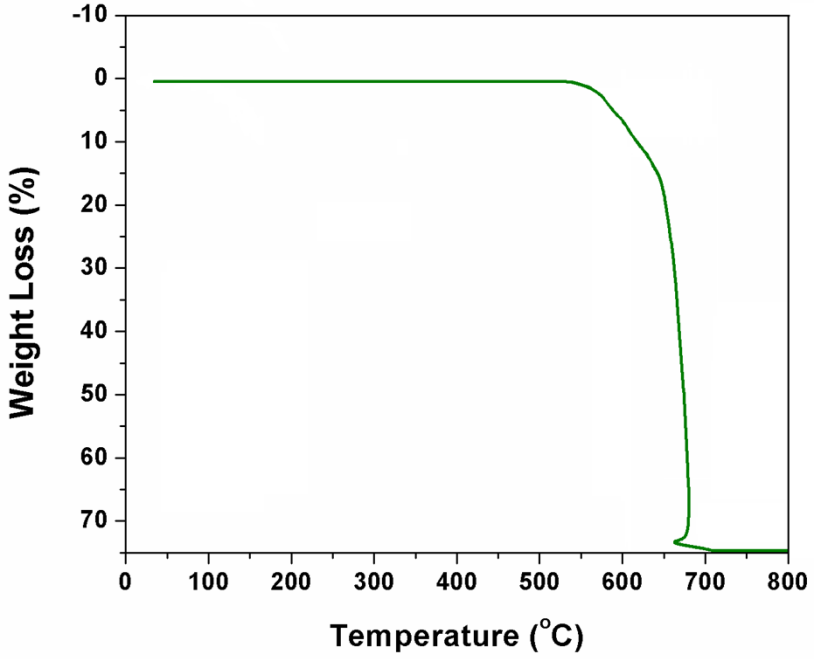


Fig. S3. TGA of compound **2.**

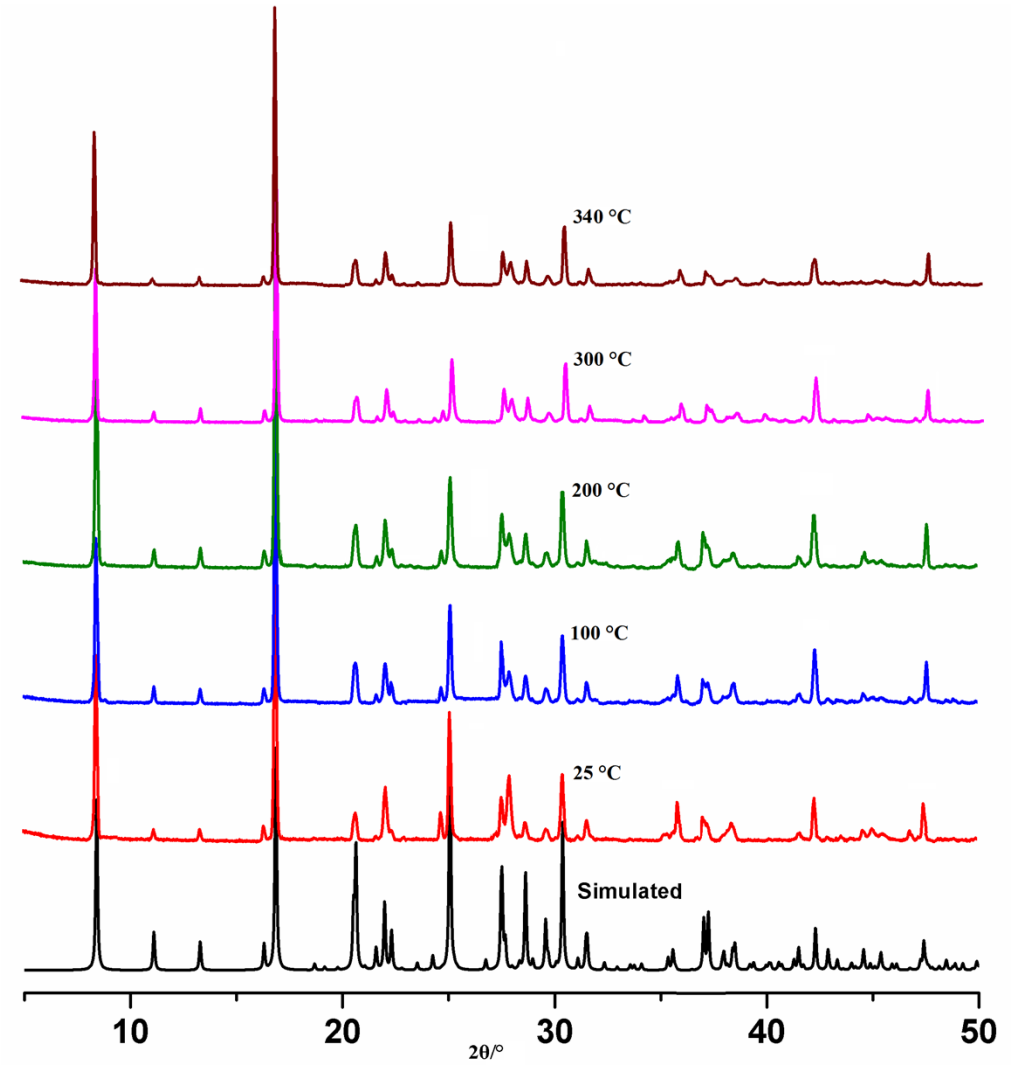


Fig. S4. In-situ PXRD pattern of **1.**

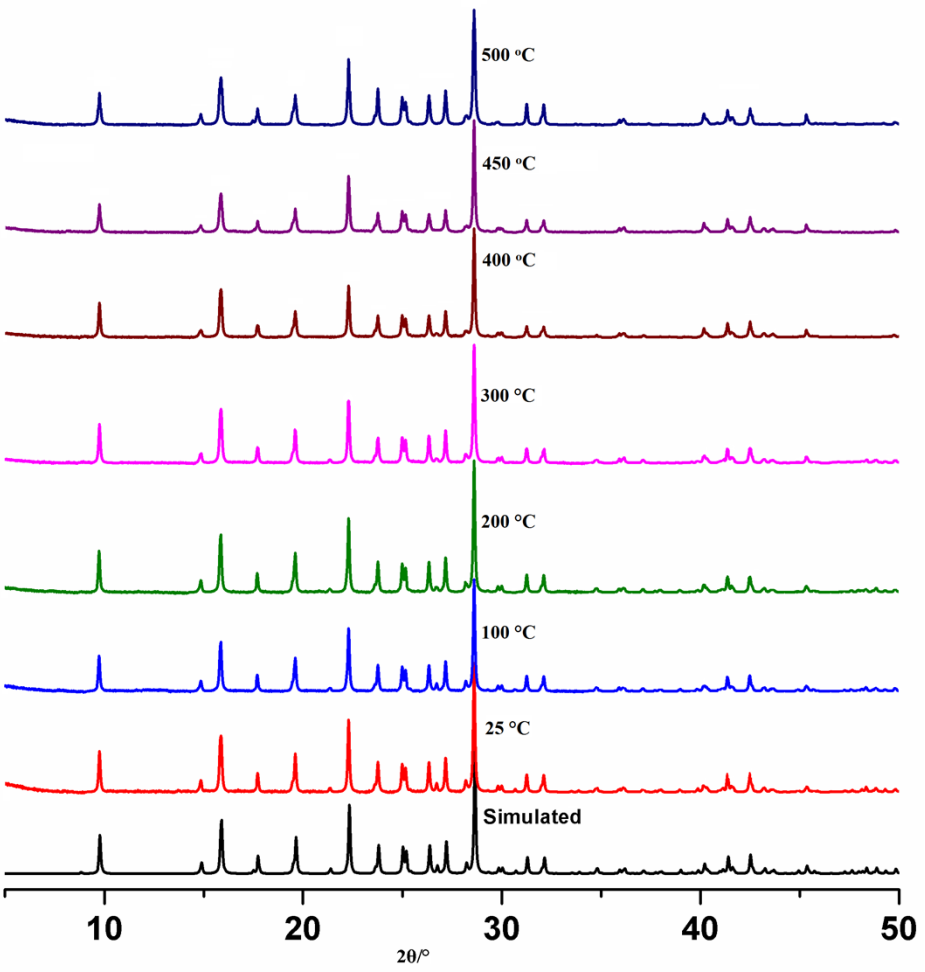


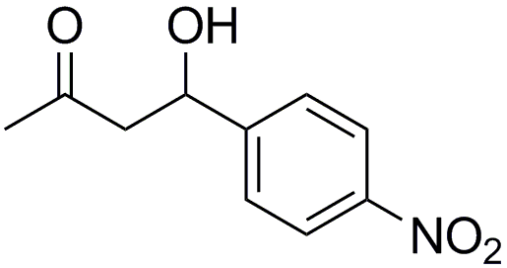
Fig. S5. In-situ PXRD pattern of **2.**

General Information

Column chromatography was performed over silica gel (mesh 60-120) and hexane/ethyl acetate combination was used as the eluent. Melting points were recorded using a Büchi Melting point apparatus M-560. ^{1}H NMR and ^{13}C NMR spectra were recorded at ambient temperature in $CDCl_3$. The chemical shifts (δ) and coupling constants (*J*) were expressed in ppm and Hz respectively, on Bruker Avance 300 instrument. High resolution mass spectra were obtained on a Xevo G2 QTof mass spectrometer. Ionization employed was the electronic impact mode (potential of ionization: 70 eV). Fourier transformed infrared spectra of KBr pellets were measured using a Perkin-Elmer RX I FT-IR spectrometer. Elemental analysis of the products was performed by using Perkin-Elmer 240C elemental analyzer.

Characterization of Products

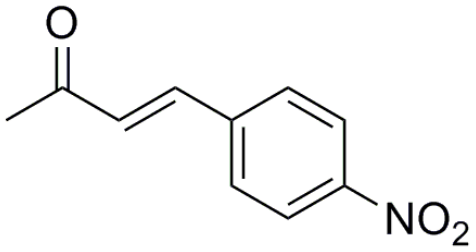
4-hydroxy-4-(4-nitrophenyl)-butan-2-one (Table 4, entry 1):



Yellow Oil; ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm): 8.07 (d, *J* = 8.8 Hz, 2H), 7.47 (d, *J* = 8.5 Hz, 2H), 5.20 (m, 1H), 3.57 (br s, 1H), 2.81 (m, 2H), 2.15 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm): 208.46, 149.99, 147.26, 126.38, 123.71, 68.85, 51.47, 30.66; HRMS (ESI): calcd. for $[M+K]^+$ ($C_{10}H_{11}NO_4$) requires m/z 248.0325, found 248.0335; IR peaks (Neat, ν, cm^{-1}): 3447, 3079, 2919, 1704, 1597, 1509, 1344, 1072, 857, 750, 691;

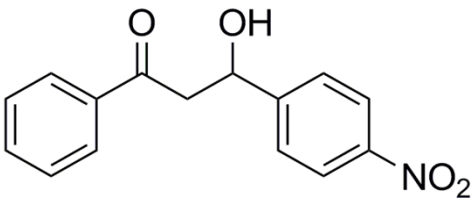
Anal. Calcd. for $C_{10}H_{11}NO_4$: C, 57.41%; H, 5.30%; N, 6.69%. Found: C, 57.45%; H, 5.27%; N, 6.67%.

4-(4-nitrophenyl)but-3-en-2-one (Table 4, entry 2):



Yellow solid; mp 104-105 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm): 8.26 (d, *J* = 8.6 Hz, 2H), 7.70 (d, *J* = 8.6 Hz, 2H), 7.54 (d, *J* = 16.2 Hz, 1H), 6.82 (d, *J* = 17.5 Hz, 1H), 2.42 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm): 197.58, 148.57, 140.68, 140.09, 130.40, 128.82, 124.20, 28.03; HRMS (ESI): calcd. for $[M+H]^+$ ($C_{10}H_9NO_3$) requires m/z 192.0661, found 192.0650; IR peaks (KBr disk, ν, cm^{-1}): 3111, 2919, 2851, 1669, 1508, 1338, 967, 817, 737; Anal. Calcd. for $C_{10}H_9NO_3$: C, 62.82%; H, 4.47%; N, 7.33%. Found: C, 62.84%; H, 4.46%; N, 7.32%.

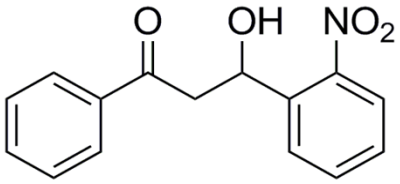
3-hydroxy-3-(4-nitrophenyl)-1-phenylpropane-1-one (Table 4, entry 3):



White soid; mp 113-114 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm): 8.24 (d, *J* = 8.8 Hz, 2H), 7.95 (d, *J* = 8.1 Hz, 2H), 7.64-7.59 (m, 3H), 7.5-7.45 (m,2H), 5.46 (d,8.6 Hz, 1H), 3.84 (d, 3.1 Hz, 1H), 3.39-3.37 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm): 199.50,

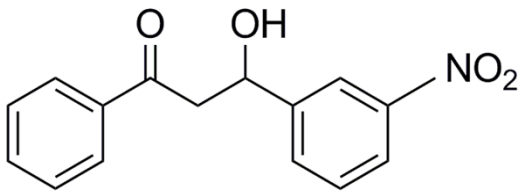
150.23, 147.40, 136.21, 134.02, 128.84, 128.16, 126.57, 123.81, 69.24, 46.98; HRMS (ESI): calcd. for $[M+Na]^+$ ($C_{15}H_{13}NO_4$) requires m/z 294.0742, found 294.0734; IR peaks (KBr disk, ν, cm^{-1}): 3500, 3065; 2905; 1669, 1598, 1508, 1338, 1208, 1078, 837, 747, 697; Anal. Calcd. for $C_{15}H_{13}NO_4$: C, 66.41%; H, 4.83%; N, 5.16%. Found: C, 66.42%; H, 4.82%; N, 5.17%.

3-hydroxy-3-(2-nitrophenyl)-1-phenylpropane-1-one (Table 4, entry 4):



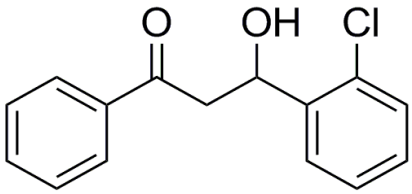
White solid; mp 108-109 °C; ^1H NMR (300 MHz, $CDCl_3$): δ (ppm): 8.00-7.96 (m, 4H), 7.7 (t, *J* = 7.6 Hz, 1H), 7.6 (t, *J* = 7.4Hz, 1H), 7.5-7.45 (m,3H), 5.86 (d, *J* = 9.3 Hz, 1H), 4 (d, *J* = 3 Hz, 1H), 3.77-3.71 (dd, *J* = 18.6 Hz, 2.1 Hz, 1H), 3.25-3.16 (dd, *J* = 17.7 Hz, 9.3 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm): 199.92, 147.34, 138.57, 136.36, 133.82, 128.75, 128.43, 128.31, 128.24, 124.43, 65.97, 46.45; HRMS (ESI): calcd. for $[M+Na]^+$ ($C_{15}H_{13}NO_4$) requires m/z 294.0742, found 294.0735; IR peaks (KBr disk, ν, cm^{-1}): 3502, 3057, 2919, 1659, 1589, 1528, 1348, 1208, 1078, 998, 747, 687, 557; Anal. Calcd. for $C_{15}H_{13}NO_4$: C, 66.41%; H, 4.83%; N, 5.16%. Found: C, 66.44%; H, 4.80%; N, 5.14%.

3-hydroxy-3-(3-nitrophenyl)-1-phenylpropane-1-one (Table 4, entry 5):



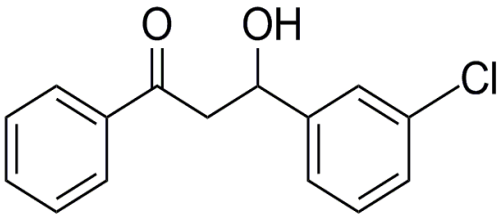
White solid; mp 85-86 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm): 8.32 (s, 1H), 8.16 (d, *J* = 9.4 Hz, 1H), 7.95 (d, *J* = 8.4 Hz, 2H), 7.79 (d, *J* = 8.3 Hz, 1H), 7.64-7.58 (m,2H), 7.53-7.46 (m, 2H), 5.48-5.43 (m, 1H), 3.88-3.87 (m, 1H), 3.42-3.38 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm): 199.44, 148.32, 145.41, 136.29, 133.88, 132.09, 129.48, 128.77, 128.19, 122.47, 120.88, 69.04, 47.06; HRMS (ESI): calcd. for $[M+Na]^+$ ($C_{15}H_{13}NO_4$) requires m/z 294.0742, found 294.0740; IR peaks (KBr disk, ν, cm^{-1}): 3508, 3095, 2875, 1669, 1518, 1348, 1208, 1078, 757, 677, 557; Anal. Calcd. for $C_{15}H_{13}NO_4$: C, 66.41%; H, 4.83%; N, 5.16%. Found: C, 66.43%; H, 4.85%; N, 5.17%.

3-hydroxy-3-(2-chlorophenyl)-1-phenylpropane-1-one (Table 4, entry 6):



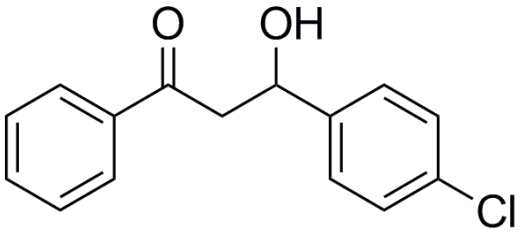
White solid; mp 78-80 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm): 7.97 (d, *J* = 7.6 Hz, 2H), 7.72 (d, *J* = 7.9 Hz, 1H), 7.58 (d, *J* = 7.5Hz, 1H), 7.47 (t, *J* = 7.4 Hz, 2H), 7.37-7.24 (m,3H), 5.69 (d, *J* = 9.1 Hz, 1H), 3.83 (d, *J* = 3 Hz, 1H), 3.61-3.54 (dd, *J* = 17.7 Hz, 2.1 Hz, 1H), 3.19-3.10 (dd, *J* = 17.8 Hz, 9.6 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm): 200.14, 140.54, 136.52, 133.70, 131.21, 129.36, 128.73, 128.60, 128.25, 127.33, 66.85, 45.47; HRMS (ESI): calcd. for $[M+Na]^+$ ($C_{15}H_{13}ClO_2$) requires m/z 283.0502, found 283.0504; IR peaks (KBr disk, ν, cm^{-1}): 3522, 3057, 2900, 1659, 1579, 1438, 1388, 1208, 747, 687; Anal. Calcd. for $C_{15}H_{13}ClO_2$: C, 69.10%; H, 5.03%. Found: C, 69.11%; H, 5.01%.

3-hydroxy-3-(3-chlorophenyl)-1-phenylpropane-1-one (Table 4, entry 7):



Colourless oil; ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm): 7.98-7.24 (m, 9H), 5.35-5.26 (m, 1H), 3.71 (d, J = 3 Hz, 1H), 3.48-3.43 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm): 199.69, 145.34, 136.50, 134.43, 133.76, 129.86, 128.74, 128.21, 127.71, 126.08, 124.01, 69.40, 47.28; HRMS (ESI): calcd. for $[M+Na]^+$ ($C_{15}H_{13}ClO_2$) requires m/z 283.0502, found 283.0517; IR peaks (Neat, ν, cm^{-1}): 3463, 3061, 2900, 1679, 1589, 1358, 1288, 1208, 1067, 887, 787, 757,687, 577; Anal. Calcd. for $C_{15}H_{13}ClO_2$: C, 69.10%; H, 5.03%. Found: C, 69.12%; H, 5.03%.

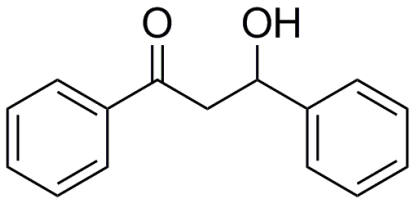
3-hydroxy-3-(4-chlorophenyl)-1-phenylpropane-1-one (Table 4, entry 8):



White solid; mp 99-100 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm): 7.96-7.35 (m, 9H), 5.39 (d, J = 9.2 Hz, 1H), 3.65 (d, J = 3 Hz, 1H), 3.50-3.19 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm): 199.78, 141.71, 136.52, 133.73, 133.26, 128.75, 128.67, 128.19, 127.25, 69.40, 47.29; HRMS (ESI): calcd. for $[M+Na]^+$ ($C_{15}H_{13}ClO_2$) requires m/z 283.0502, found 283.0505; IR peaks (KBr disk, ν, cm^{-1}): 3462, 3051, 2941, 1659, 1579,

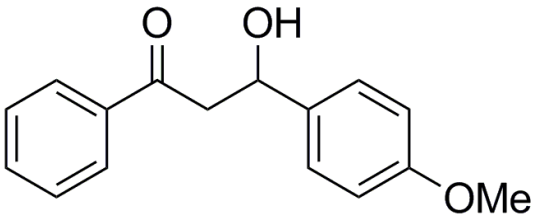
1489, 1378, 1278, 1208, 1017, 828, 747, 677, 537; Anal. Calcd. for $C_{15}H_{13}ClO_2$: C, 69.10%; H, 5.03%. Found: C, 69.07%; H, 5.04%.

3-hydroxy-1,3-biphenylpropane-1-one (Table 4, entry 9):



Colourless oil; ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm): 7.92 (d, J = 7.4 Hz, 2H), 7.55-7.27 (m, 8H), 5.30-5.26 (m, 1H), 3.67 (s, 1H), 3.42-3.29 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm): 200.09, 143.16, 136.68, 133.63, 128.72, 128.59, 128.23, 127.68, 125.84, 70.07, 47.47; Anal. HRMS (ESI): calcd. for $[M+Na]^+$ ($C_{15}H_{14}O_2$) requires m/z 249.0891, found 249.0855; IR peaks (Neat, ν, cm^{-1}): 3466, 3048, 2902, 1675, 1587, 1451, 1353, 1208, 1022, 750, 691; Calcd. for $C_{15}H_{14}O_2$: C, 79.62%; H, 6.24%. Found: C, 79.59%; H, 6.23%.

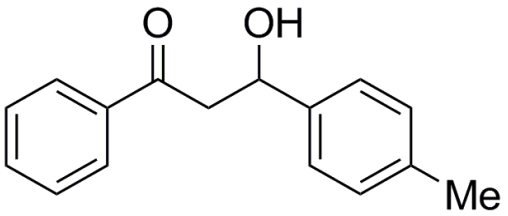
3-hydroxy-3-(4-methoxyphenyl)-1-phenylpropane-1-one (Table 4, entry 10):



White solid; mp 100-112 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm): 7.92 (d, J = 7.4 Hz, 2H), 7.43-7.24 (m, 5H), 6.92-6.88 (m, 2H), 5.32-5.28 (m, 1H), 3.80 (s, 3H), 3.5 (br s, 1H), 3.37-3.34 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm): 200.22, 159.17, 136.69, 135.22, 133.59, 128.70, 128.17, 127.05, 113.98, 69.72, 55.32, 47.36; HRMS (ESI): calcd.

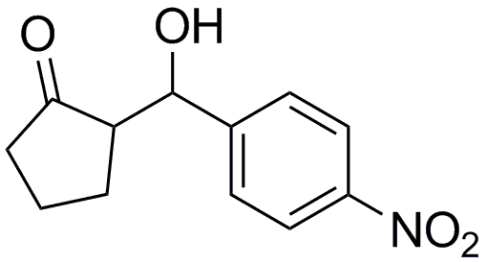
for $[M+Na]^+$ ($C_{16}H_{16}O_3$) requires m/z 279.0997, found 249.0960; IR peaks (KBr disk, ν, cm^{-1}): 3472, 2921, 1679, 1598, 1508, 1449, 1248, 1028, 828, 757, 687, 567; Anal. Calcd. for $C_{16}H_{16}O_3$: C, 74.98%; H, 6.29%. Found: C, 74.97%; H, 6.27%.

3-hydroxy-3-(4-tolyl)-1-phenylpropane-1-one (Table 4, entry 11):



Colourless oil; ^1H NMR (300 MHz, $CDCl_3$): δ (ppm): 7.96-7.93 (m, 2H), 7.52-7.18 (m, 7H), 5.31-5.28 (m, 1H), 3.52 (s, 1H), 3.37-3.30 (m, 2H), 2.38 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm): 200.20, 140.06, 137.36, 136.69, 133.59, 129.24, 128.69, 128.16, 125.71, 69.94, 47.41, 21.11; HRMS (ESI): calcd. for $[M+Na]^+$ ($C_{16}H_{16}O_2$) requires m/z 263.1048, found 263.1010; IR peaks (Neat, ν, cm^{-1}): 3466, 2912, 1685, 1597, 1578, 1509, 1451, 1208, 1072, 1003, 818, 760, 691, 546; Anal. Calcd. for $C_{16}H_{16}O_2$: C, 79.97%; H, 6.71%. Found: C, 79.98%; H, 6.69%.

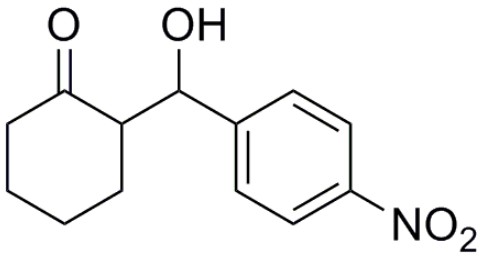
2-(hydroxy-(4-nitrophenyl)-methyl)-cyclopentanone (Table 4, entry 12):



Yellow solid, mp 88-90 °C; ^1H NMR (300 MHz, $CDCl_3$): δ (ppm): 8.24 (d, J = 8.8 Hz, 2H), 7.55-7.5 (m, 2H), 4.86 (d, J = 9.2 Hz, 1H), 3.84 (d, J = 3.1 Hz, 1H), 2.51-1.69 (m,

7H); ^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm): 222.24, 219.50, 150.14, 148.63, 147.19, 127.36, 126.37, 123.73, 74.44, 70.50, 56.08, 55.09, 38.93, 38.60, 26.85, 22.43, 20.38, 20.33; HRMS (ESI): calcd. for $[M+Na]^+$ ($C_{12}H_{13}NO_4$) requires m/z 258.0742, found 258.0727; IR peaks (KBr disk, ν, cm^{-1}): 3441, 2950, 2881, 1729, 1598, 1518, 1338, 1158, 1098, 1028, 857, 737, 687, 557; Anal. Calcd. for $C_{12}H_{13}NO_4$: C, 61.27%; H, 5.57%; N, 5.95%. Found: C, 61.29%; H, 5.55%; N, 5.96%.

2-(hydroxy-(4-nitrophenyl)-methyl)-cyclohexanone (Table 4, entry 13):



Yellow solid; mp 118-120 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm): 8.21 (d, J = 9 Hz, 2H), 7.5 (d, J = 8.7 Hz, 2H), 4.94 (d, J = 8.2 Hz, 1H), 3.96 (br, s, 1H), 2.8-2.75 (m, 1H); 2.49-2.36 (m, 2H), 2.14-2.07 (m,1H), 1.88-1.59 (m, 5H); ^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm): 214.70, 213.98, 149.10, 148.35, 147.76, 146.97, 127.82, 126.56, 123.50, 123.39, 73.93, 70.04, 57.12, 56.73, 42.61, 42.54, 30.70, 27.78, 27.59, 25.84, 24.70, 24.62; HRMS (ESI): calcd. for $[M+Na]^+$ ($C_{13}H_{15}NO_4$) requires m/z 272.0889, found 272.0868; IR peaks (KBr disk, ν, cm^{-1}): 3451, 3081, 2921, 2851, 1699, 1669, 1608, 1528, 1348, 1258, 977, 897, 856; 817, 797, 727, 667, 517; Anal. Calcd. for $C_{13}H_{15}NO_4$: C, 62.64%; H, 6.07%; N, 5.62%. Found: C, 62.67%; H, 6.06%; N, 5.62%.

References

[1] S. Hu, T. Jiang, Z. Zhang, A. Zhu, B. Han, J. Song, Y. Xie, W. Li, *Tetrahedron Lett.* 2007, **48**, 5613.

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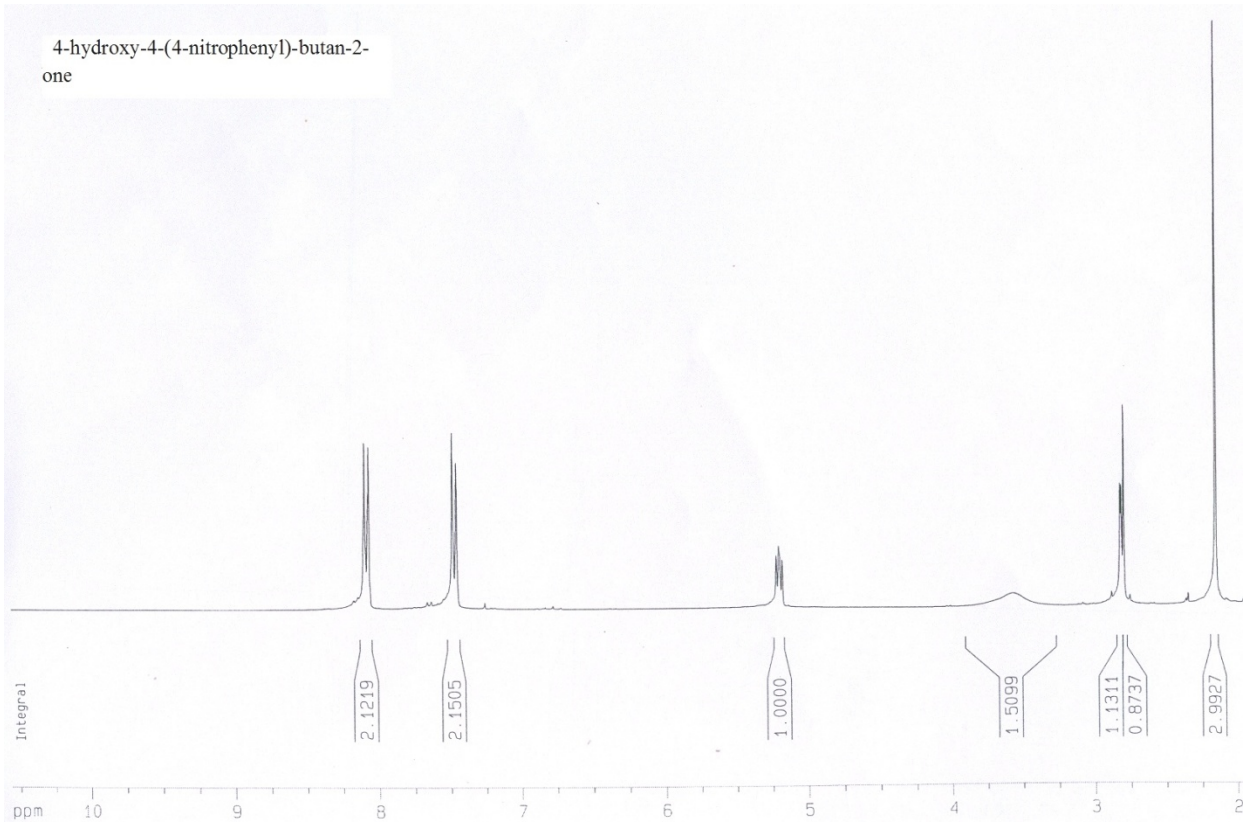
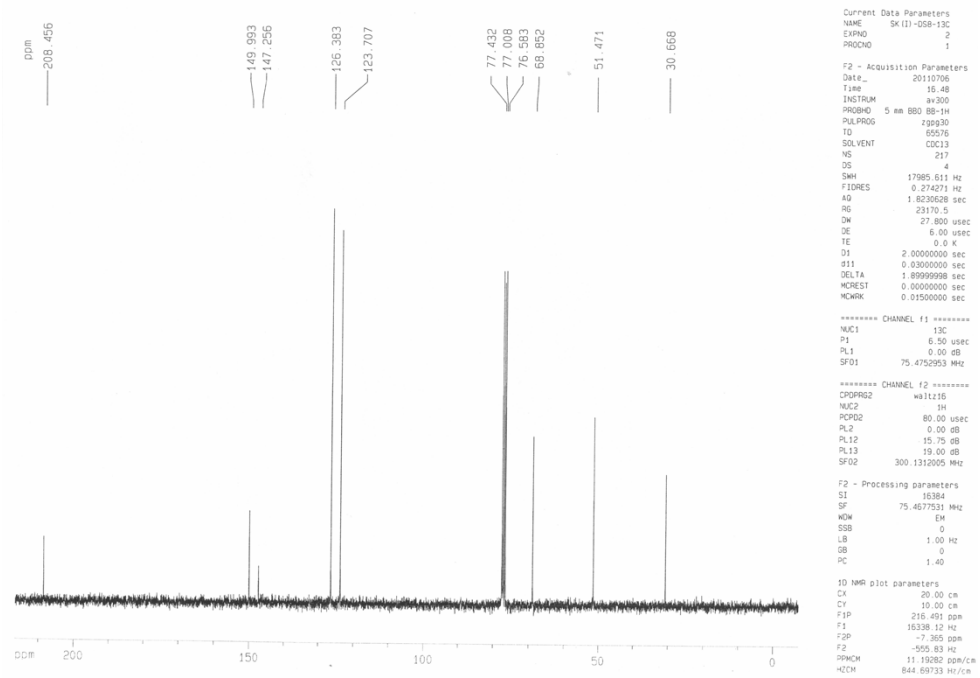
Entry 1





Current Data Parameters
NAME SK(I)-DSB-13C
EXPNO 2
PROCNO 1

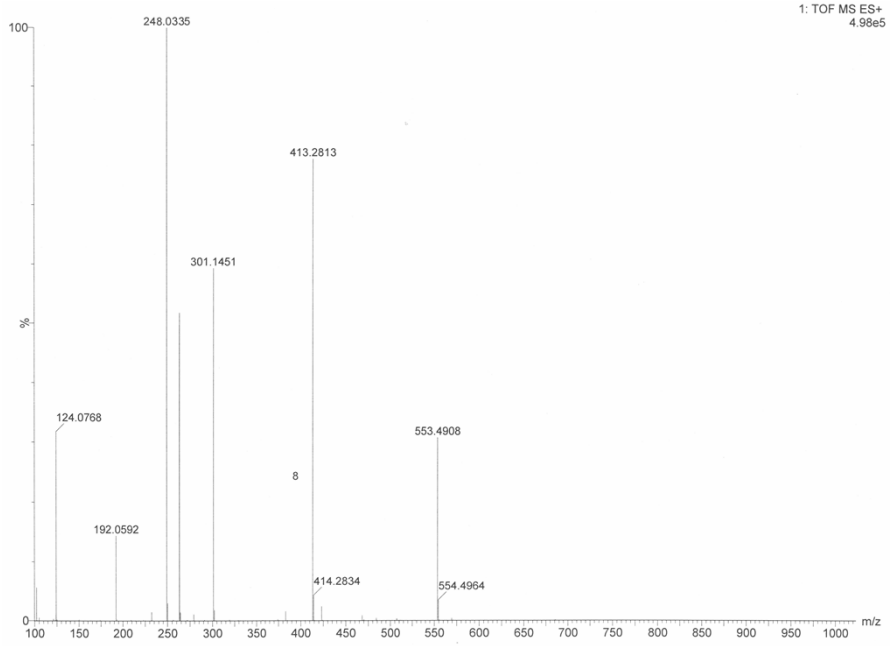
F2 - Acquisition Parameters
Date_ 20110706
Time 16.48
INSTRUM av300
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65576
SOLVENT CDCl3
NS 217
DS 4
SWH 17985.611 Hz
FIDRES 0.274271 Hz
AQ 1.8230628 sec
RG 23170.5
DW 27.800 usec
DE 6.00 usec
TE 0.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

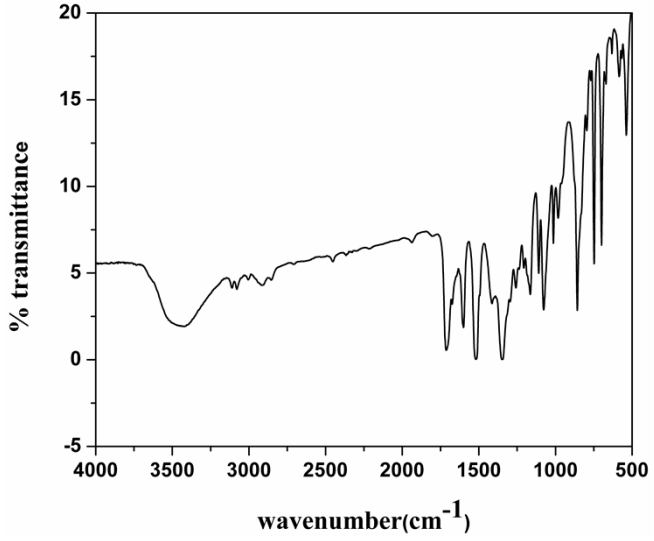
======== CHANNEL f1 ========
NUC1 13C
P1 6.50 usec
PL1 0.00 dB
SFO1 75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 15.75 dB
PL13 19.00 dB
SFO2 300.1312005 MHz

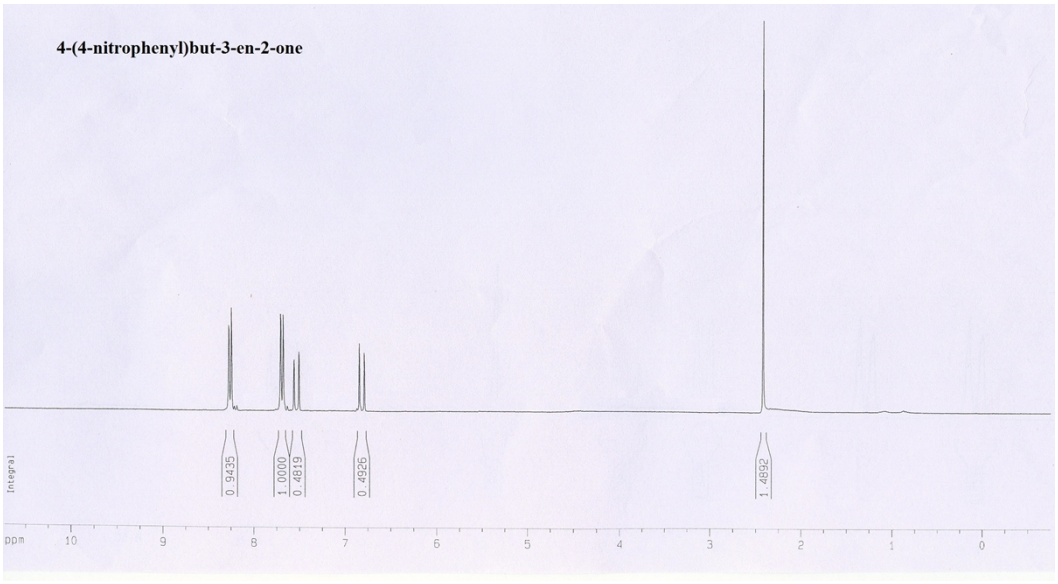
F2 - Processing parameters
SI 16384
SF 75.4677531 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

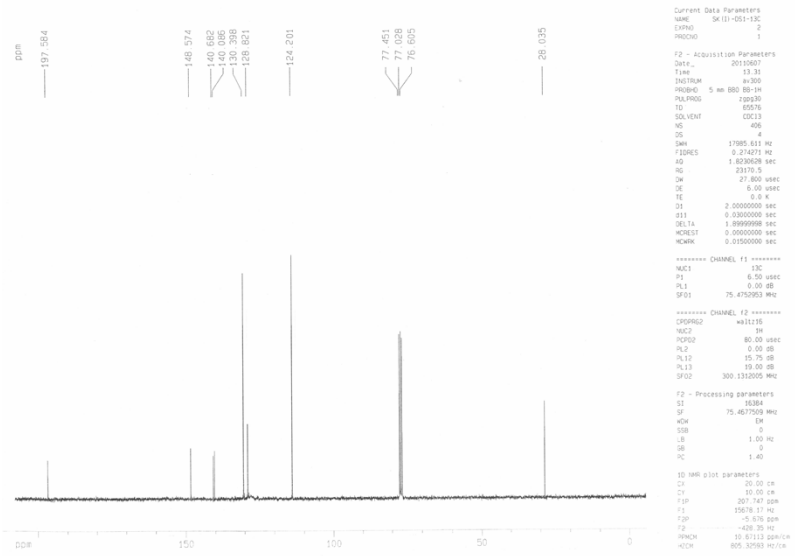
1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
F1P 216.491 ppm
F1 16338.12 Hz
F2P -7.365 ppm
F2 -555.83 Hz
PPMCM 11.19282 ppm/cm
HZCM 844.69733 Hz/cm

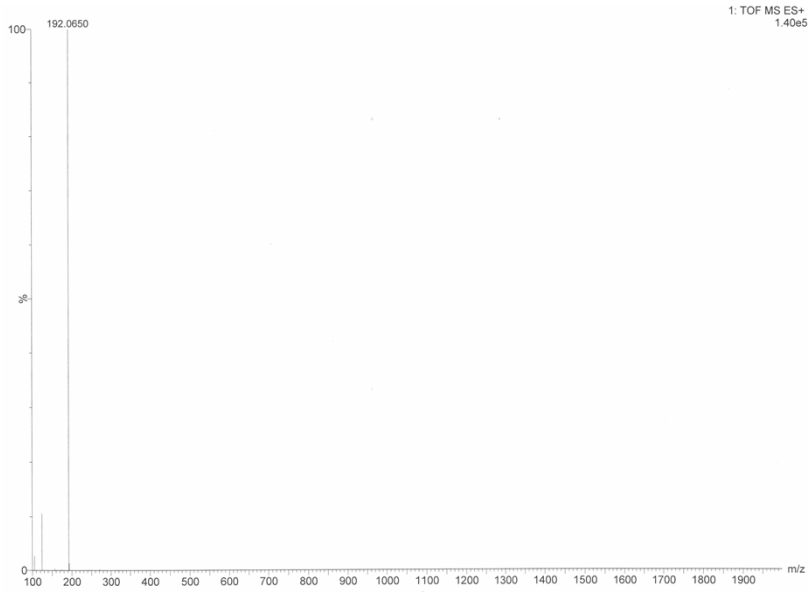

1: TOF MS ES+
4.98e5
248.0335
413.2813
301.1451
124.0768
553.4908
192.0592
414.2834
554.4964
%
m/z

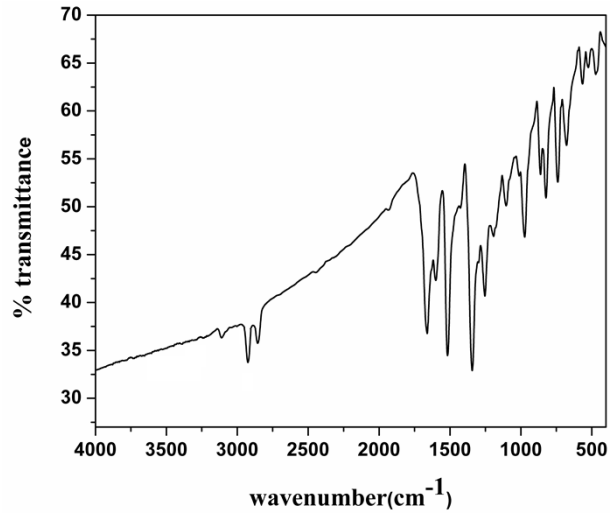

% transmittance
wavenumber(cm-1)

Entry 2

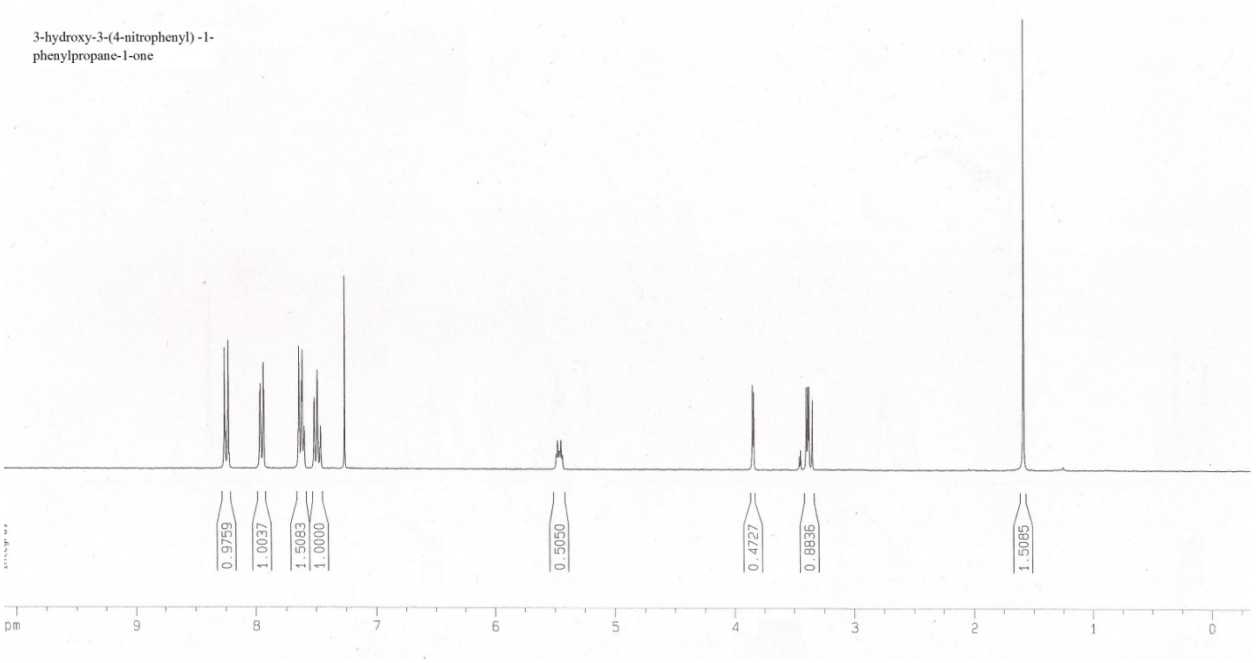

4-(4-nitrophenyl)but-3-en-2-one
Integral
0.9435
1.0000
0.4819
0.4926
1.4892
ppm
10
9
8
7
6
5
4
3
2
1
0

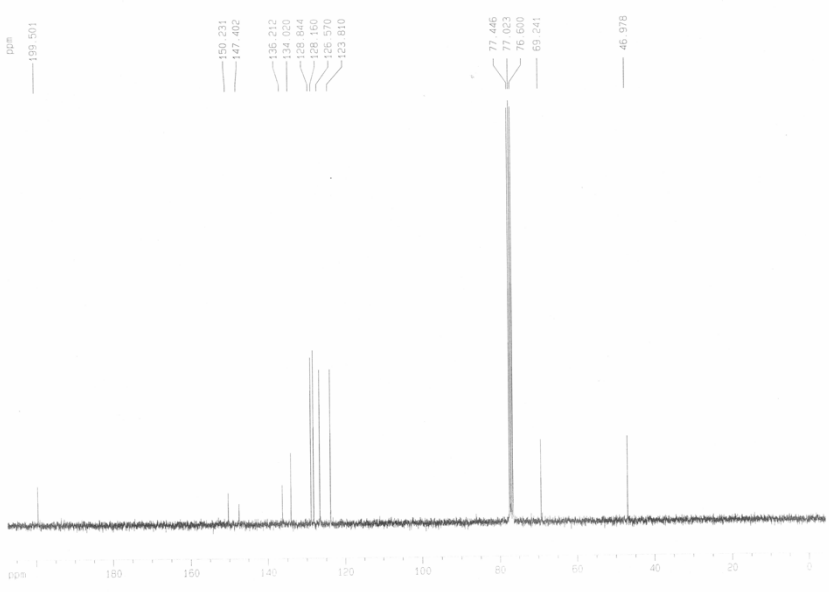

ppm
197.584
148.574
140.682
140.086
130.398
128.821
124.201
77.451
77.028
76.605
28.035
150
100
50
0


1: TOF MS ES+
1.40e5
192.0650
100
%
0
100 200 300 400 500 600 700 800 900 1000 1100 1200 1300 1400 1500 1600 1700 1800 1900
m/z

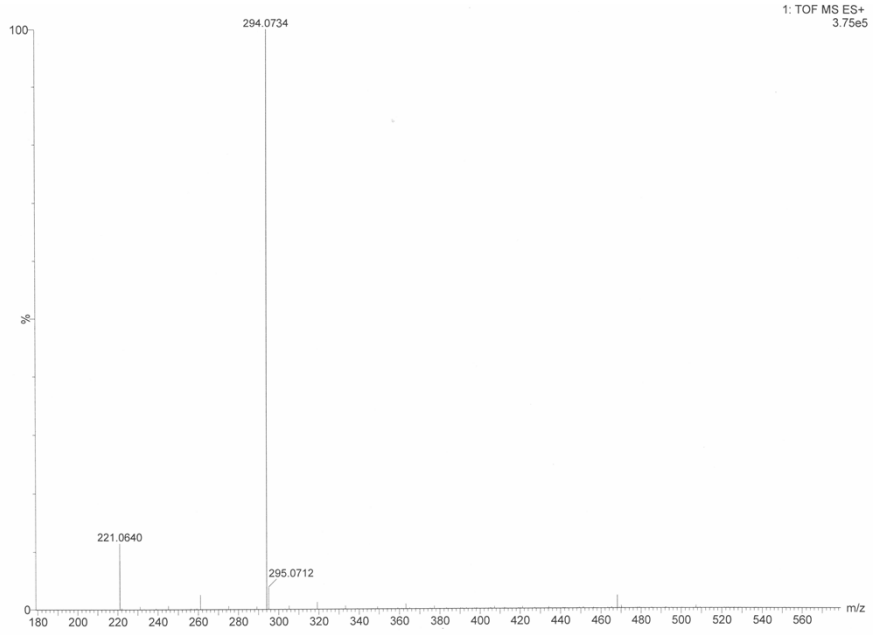

% transmittance
70 65 60 55 50 45 40 35 30
4000 3500 3000 2500 2000 1500 1000 500
wavenumber(cm-1)

Entry 3


3-hydroxy-3-(4-nitrophenyl) -1-
phenylpropane-1-one
0.9759
1.0037
1.5083
1.0000
0.5050
0.4727
0.8836
1.5085
pm
9
8
7
6
5
4
3
2
1
0


ppm
199.501
150.231
147.402
136.212
134.020
128.844
128.160
126.570
123.810
77.446
77.023
76.600
69.241
46.978
ppm
180
160
140
120
100
80
60
40
20
0


Current Data Parameters
NAME SK(I)-DSPH1-13C
EXPNO 1
PROCNO 2
F2 - Acquisition Parameters
Date_ 20140417
Time 15.01
INSTRUM av300
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 354
DS 4
SWH 16556.291 Hz
FIDRES 0.252629 Hz
AQ 1.9792372 sec
RG 18390.4
DW 30.200 usec
DE 6.00 usec
TE 0.0 K
D1 2.00000000 sec
D11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 11.00 usec
PL1 -1.00 dB
SFO1 75.4752958 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 13.00 dB
PL13 17.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 24.00 cm
CY 9.00 cm
F1P 207.251 ppm
F1 15640.76 Hz
F2P -4.322 ppm
F2 -326.23 Hz
PPMCM 8.61556 ppm/cm
HZCM 665.29034 Hz/cm


1: TOF MS ES+
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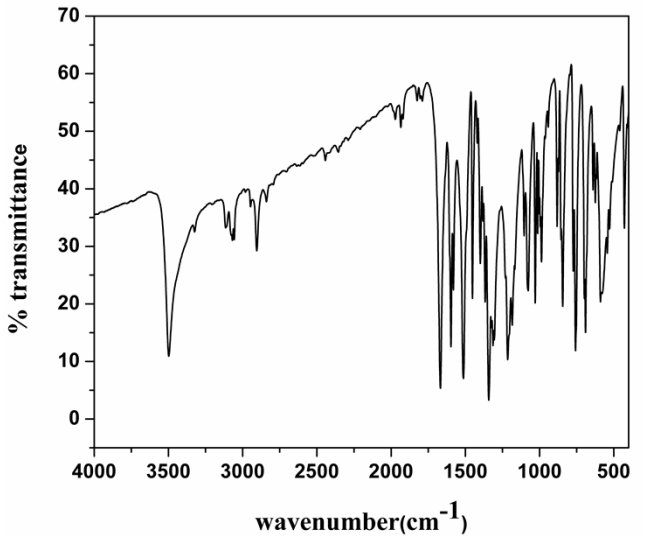
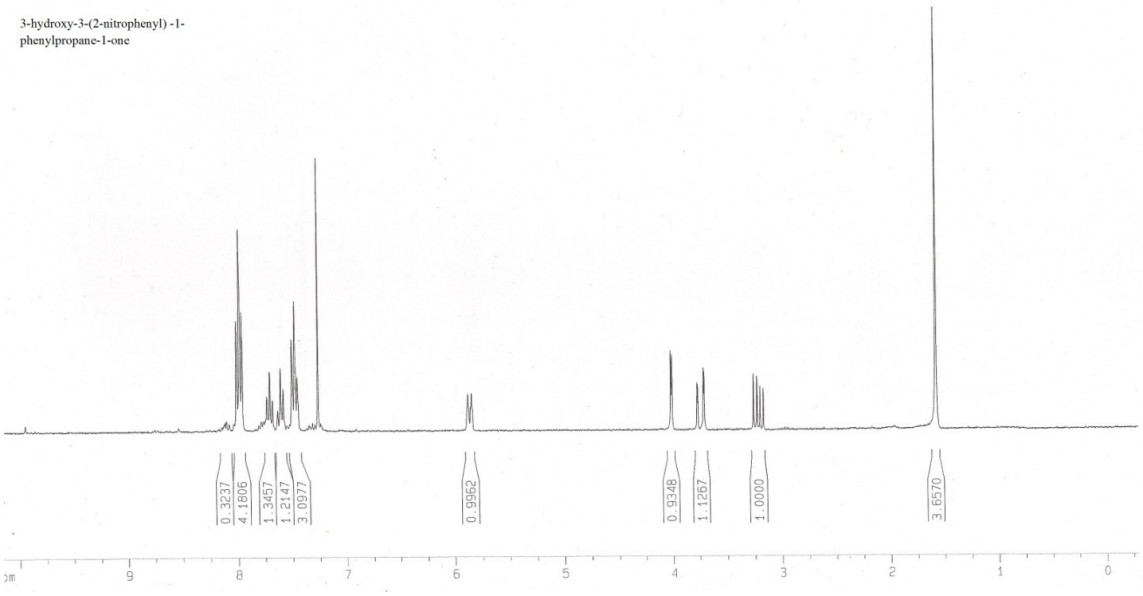
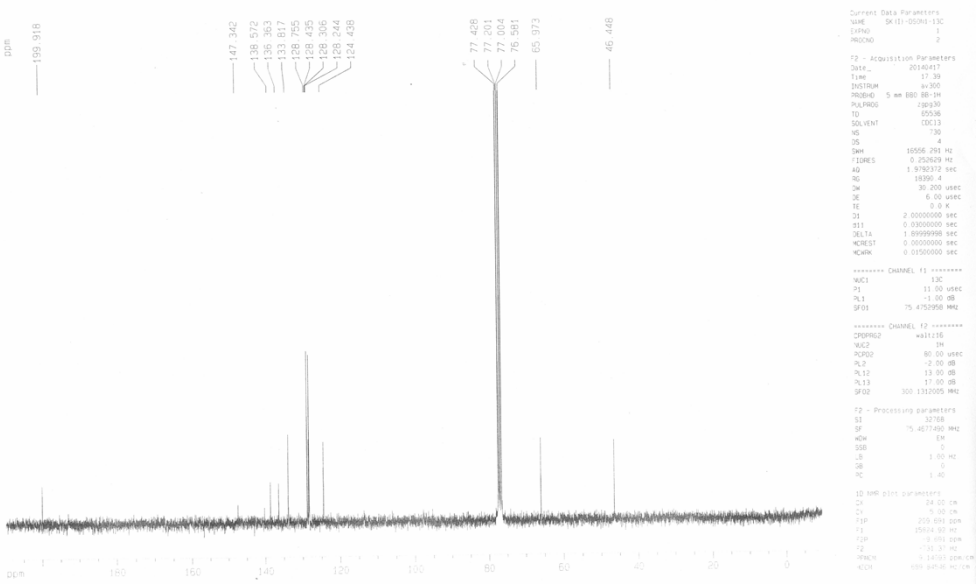
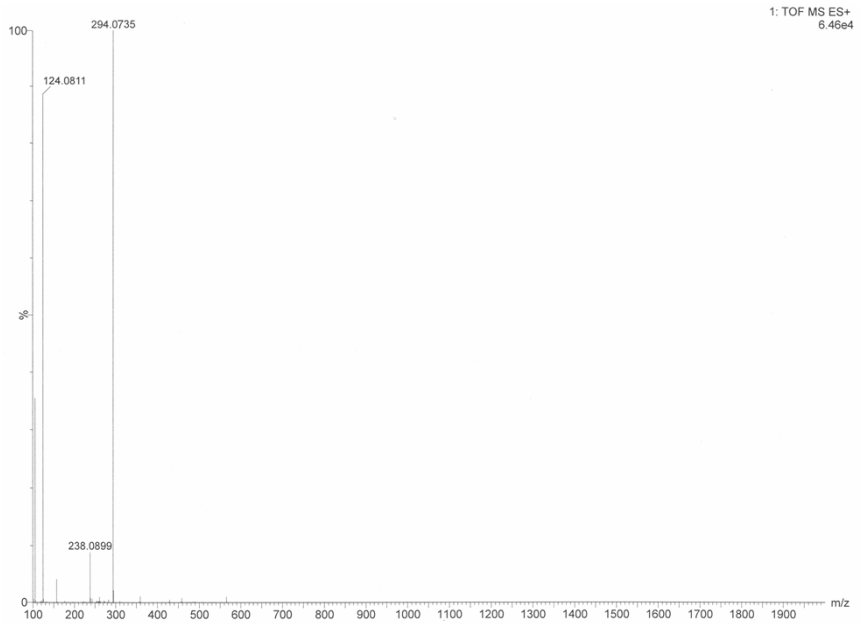
294.0734
100
%
221.0640
295.0712
0
180 200 220 240 260 280 300 320 340 360 380 400 420 440 460 480 500 520 540 560
m/z

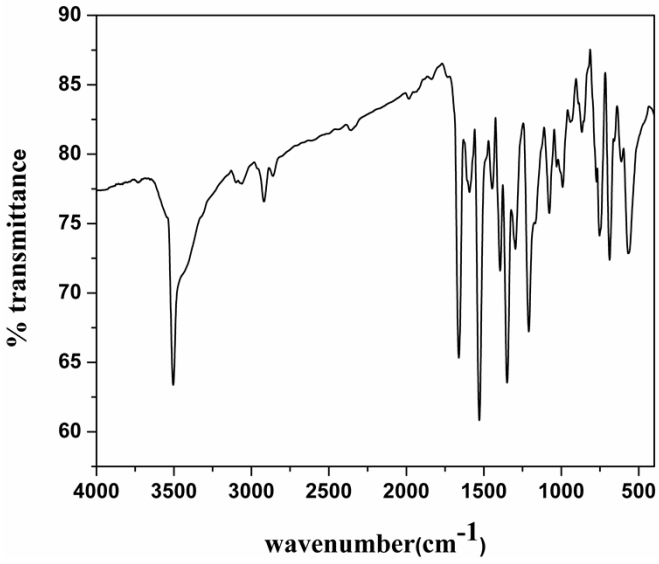

% transmittance
70
60
50
40
30
20
10
0
4000 3500 3000 2500 2000 1500 1000 500
wavenumber(cm-1)

Entry 4

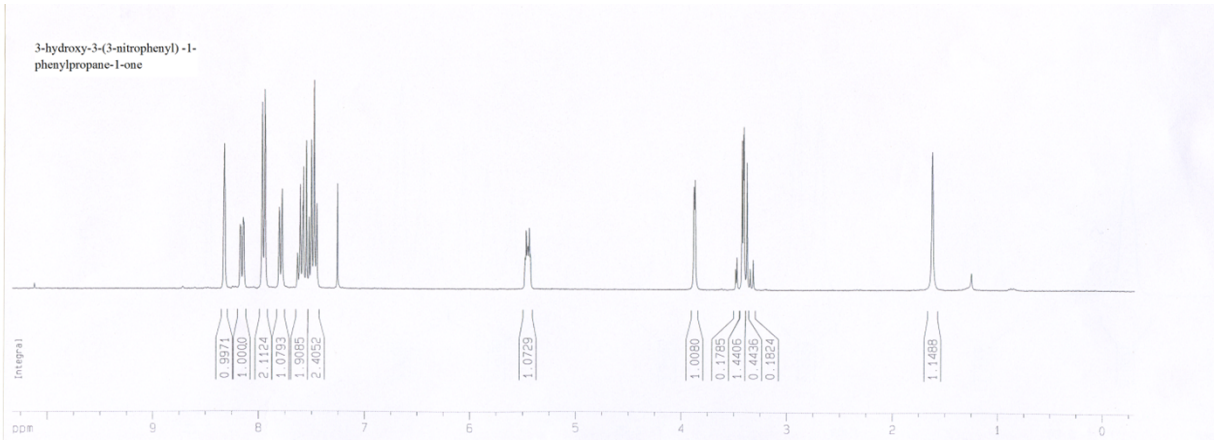

3-hydroxy-3-(2-nitrophenyl) -1-phenylpropane-1-one
0.3237
4.1606
1.3457
1.2147
3.0977
0.9962
0.9348
1.1267
1.0000
3.6570
ppm
9
8
7
6
5
4
3
2
1
0

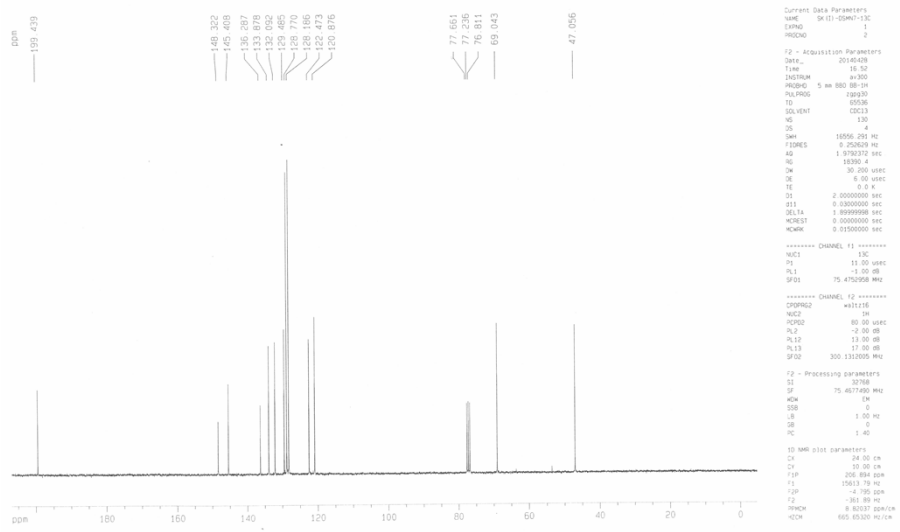

ppm
199.918
147.342
138.572
135.363
133.817
128.756
128.435
128.306
128.244
124.438
77.428
77.201
77.004
76.581
65.973
46.446
ppm
180
160
140
120
100
80
60
40
20
0

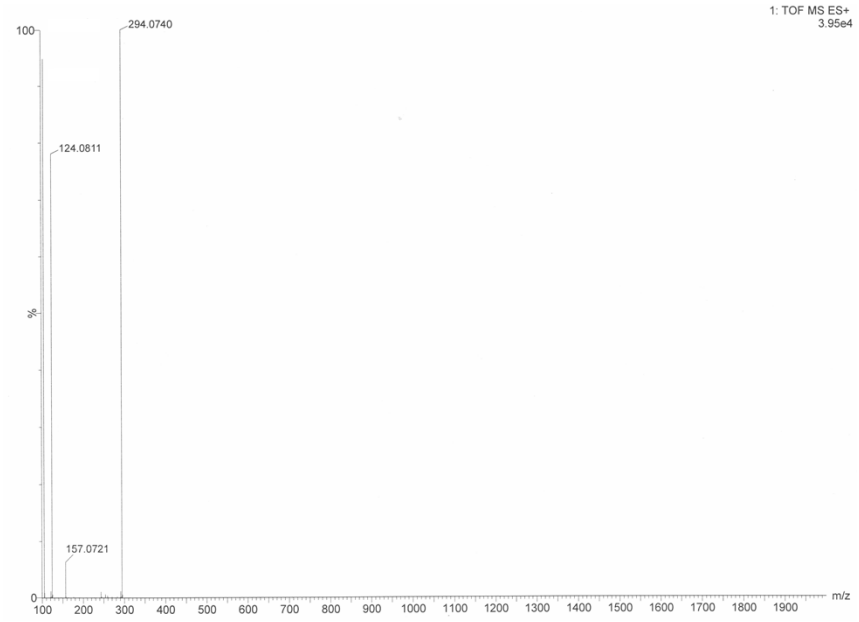

1: TOF MS ES+
6.46e4
294.0735
124.0811
238.0899
100
%
0
m/z
100 200 300 400 500 600 700 800 900 1000 1100 1200 1300 1400 1500 1600 1700 1800 1900


% transmittance
wavenumber(cm-1)
90
85
80
75
70
65
60
4000 3500 3000 2500 2000 1500 1000 500

Entry 5


3-hydroxy-3-(3-nitrophenyl) -1-phenylpropane-1-one
Integral
0.9971
1.0000
2.1124
1.0793
1.9085
2.4052
1.0729
1.0080
0.1785
1.4406
0.4436
0.1824
1.1488
ppm


ppm
199.439
148.322
145.408
136.287
133.878
132.052
129.465
128.770
125.155
122.473
120.876
77.661
77.236
76.811
69.043
47.056
ppm


1: TOF MS ES+
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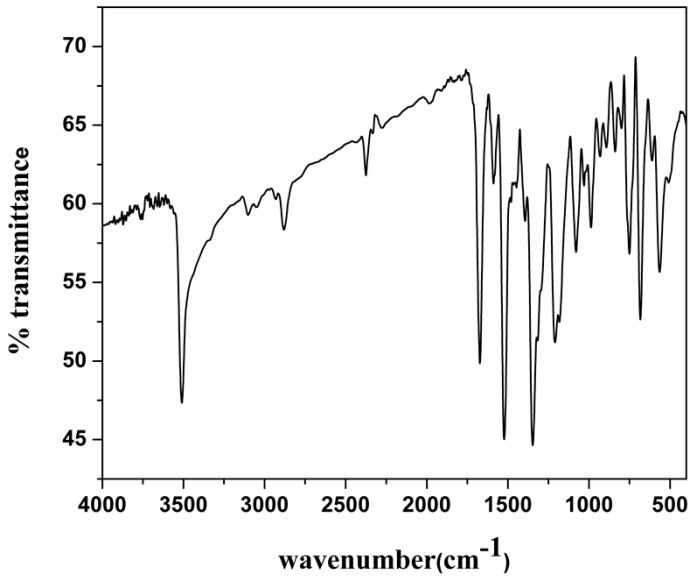
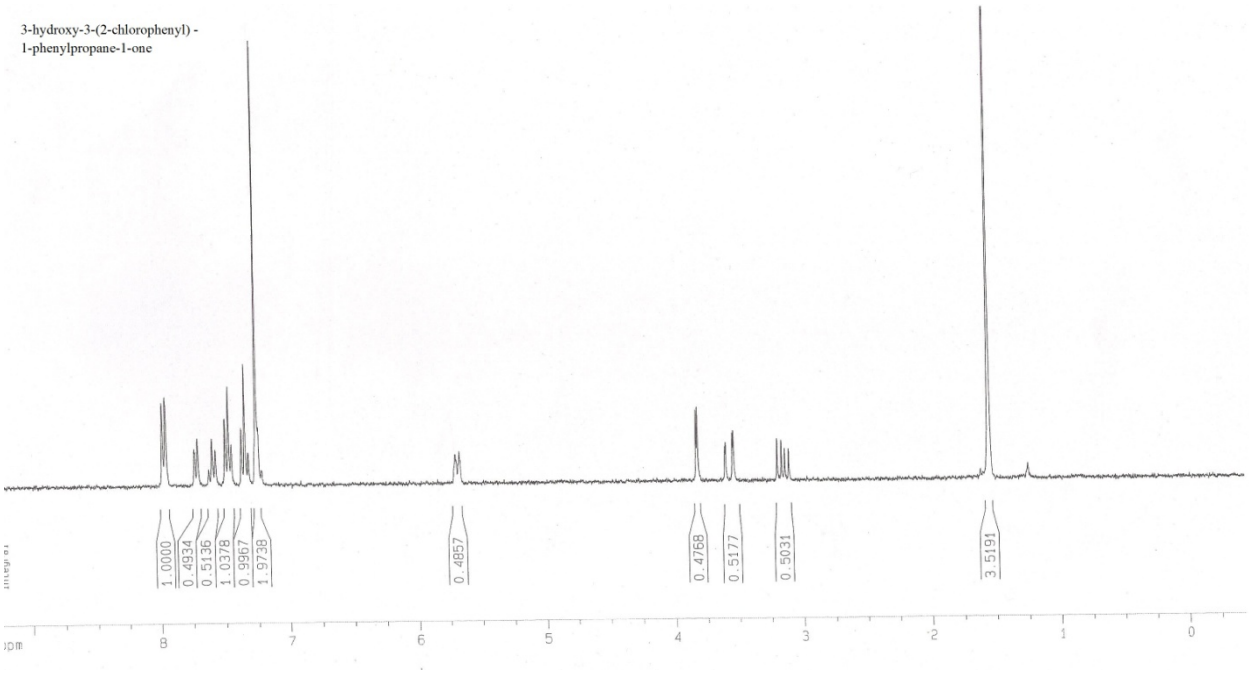
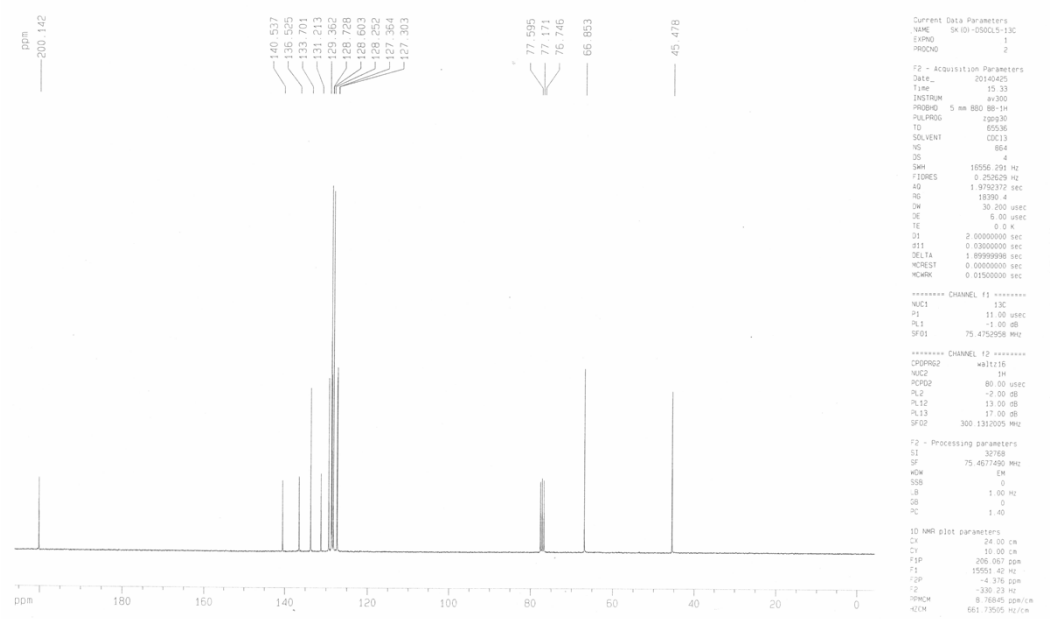
294.0740
124.0811
157.0721
100
%
0
m/z
100 200 300 400 500 600 700 800 900 1000 1100 1200 1300 1400 1500 1600 1700 1800 1900


% transmittance
70
65
60
55
50
45
4000 3500 3000 2500 2000 1500 1000 500
wavenumber(cm-1)

Entry 6





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EXPNO 1
PROCNO 2

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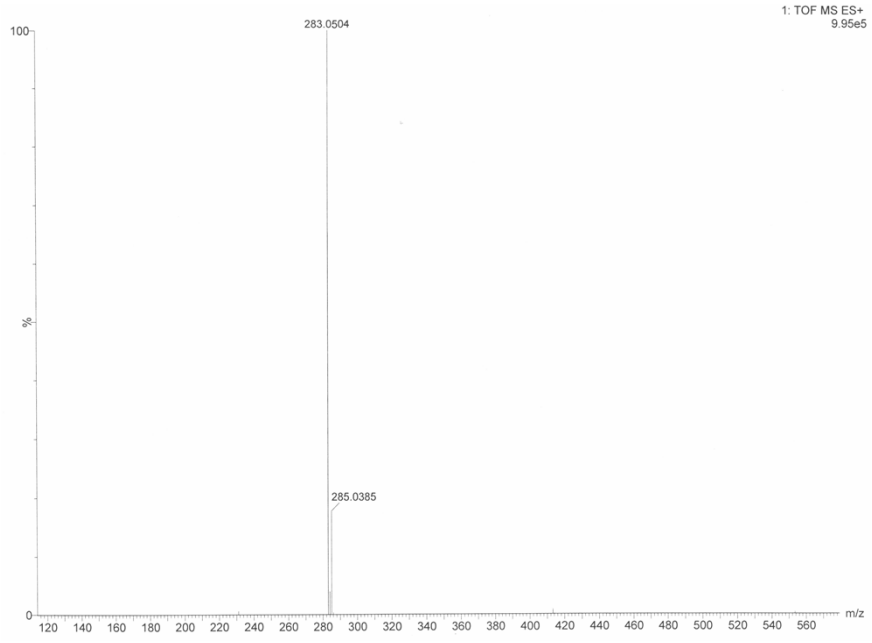
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 864
DS 4
SWH 16556.291 Hz
FIDRES 0.252629 Hz
AQ 1.9792372 sec
RG 18390.4
DW 30.200 usec
DE 6.00 usec
TE 0.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

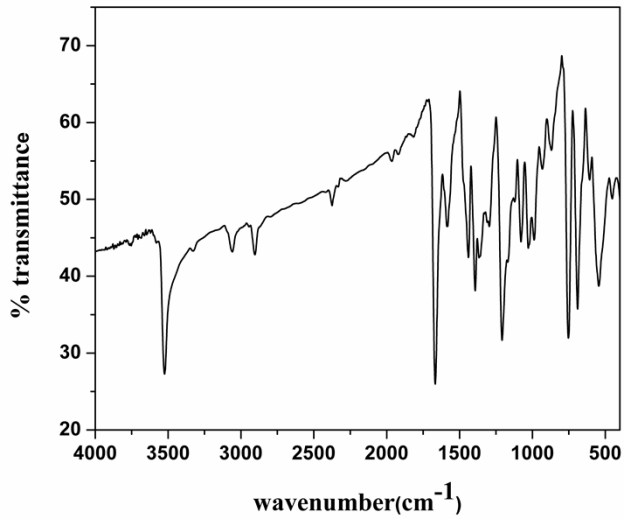
======== CHANNEL f1 ========
NUC1 13C
P1 11.00 usec
PL1 -1.00 dB
SFO1 75.4752958 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 13.00 dB
PL13 17.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

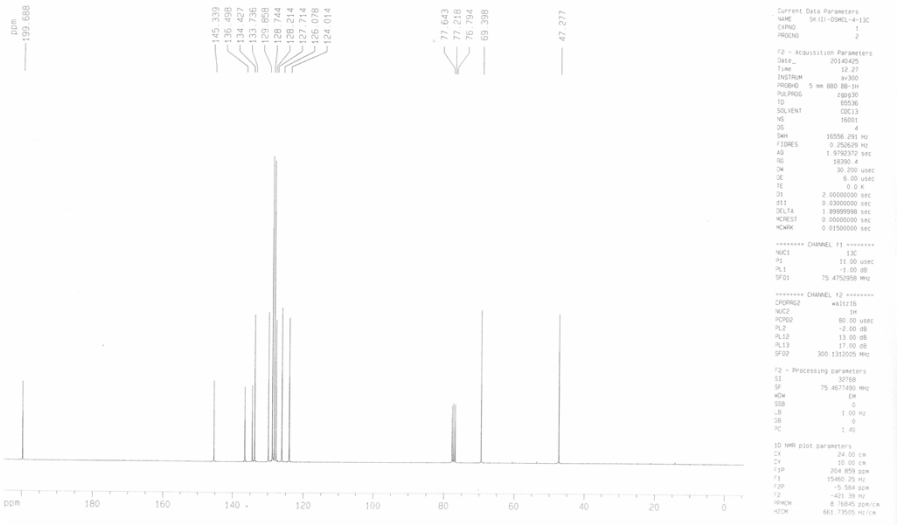
1D NMR plot parameters
CX 24.00 cm
CY 10.00 cm
F1P 206.067 ppm
F1 15551.42 Hz
F2P -4.376 ppm
F2 -330.23 Hz
PPMCM 8.76845 ppm/cm
HZCM 661.73595 Hz/cm

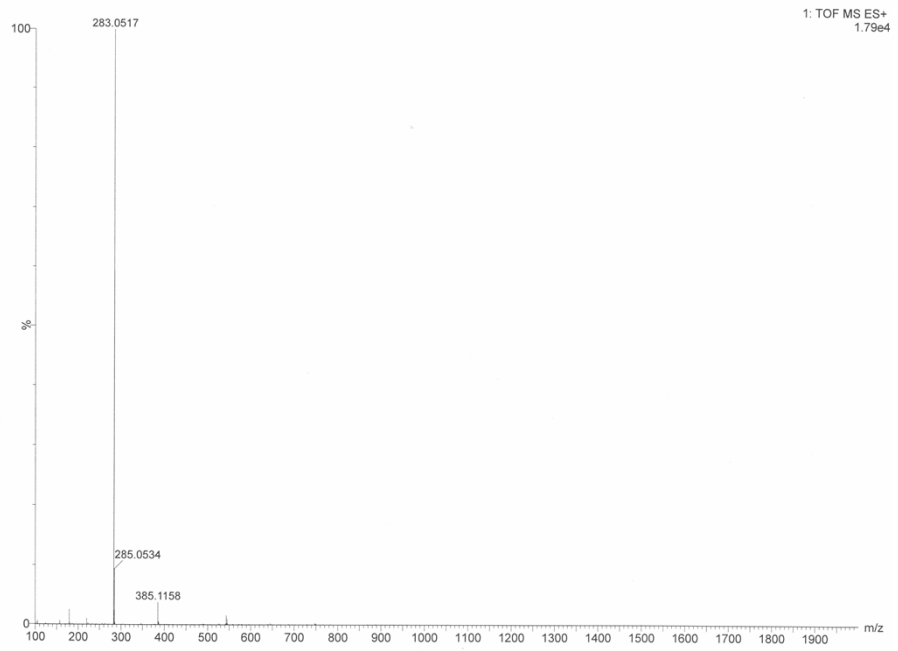

1: TOF MS ES+
9.95e5
283.0504
285.0385
100
%
0
120 140 160 180 200 220 240 260 280 300 320 340 360 380 400 420 440 460 480 500 520 540 560
m/z

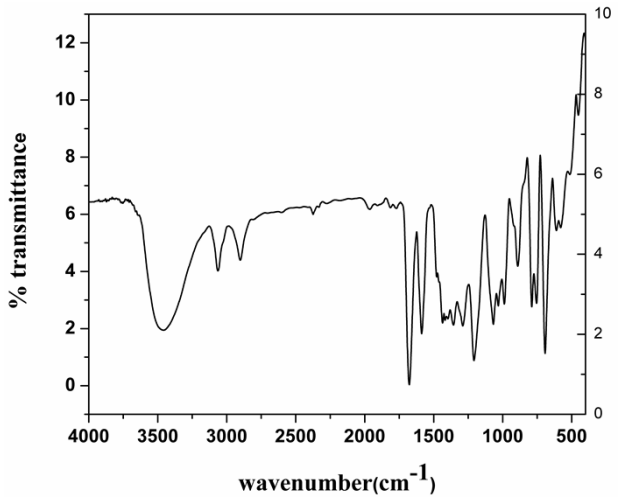

% transmittance
70
60
50
40
30
20
4000 3500 3000 2500 2000 1500 1000 500
wavenumber(cm-1)

Entry 7

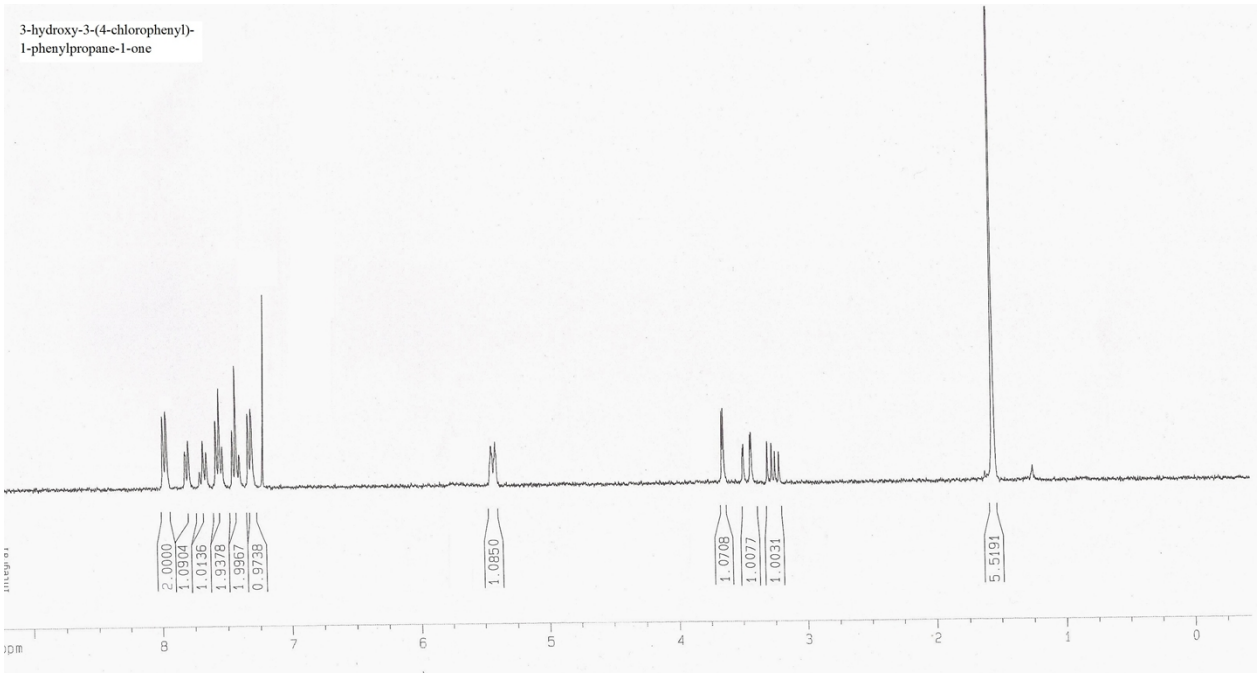

3-hydroxy-3-(3-chlorophenyl) -
1-phenylpropane-1-one
1.9967
1.0378
2.0000
1.0272
2.0227
1.9030
1.0807
1.4768
0.4170
0.3001
0.0031
3.2191
ppm
8
7
6
5
4
3
2
1
0

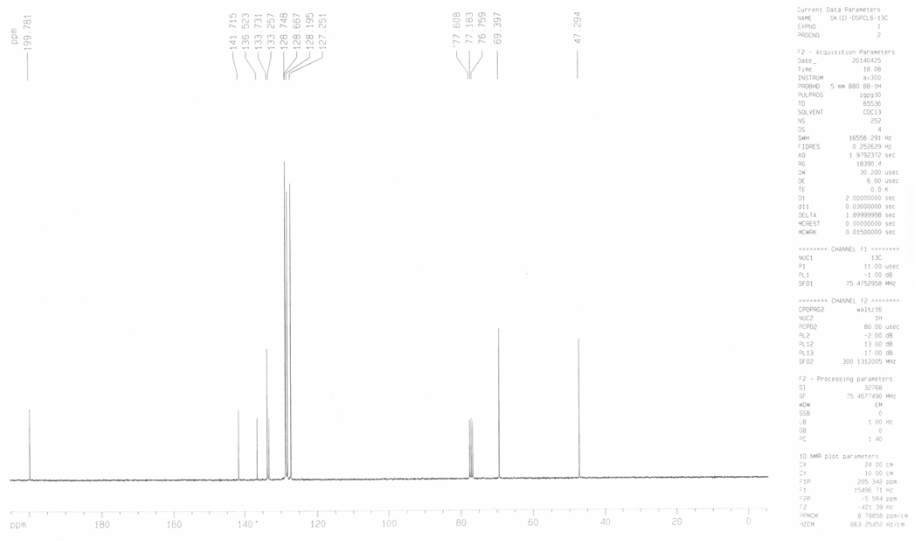

ppm
199.688
145.339
136.498
134.427
133.736
129.868
128.744
128.244
127.714
126.078
124.014
77.643
77.218
76.794
69.398
47.277
ppm
180
160
140
120
100
80
60
40
20
0

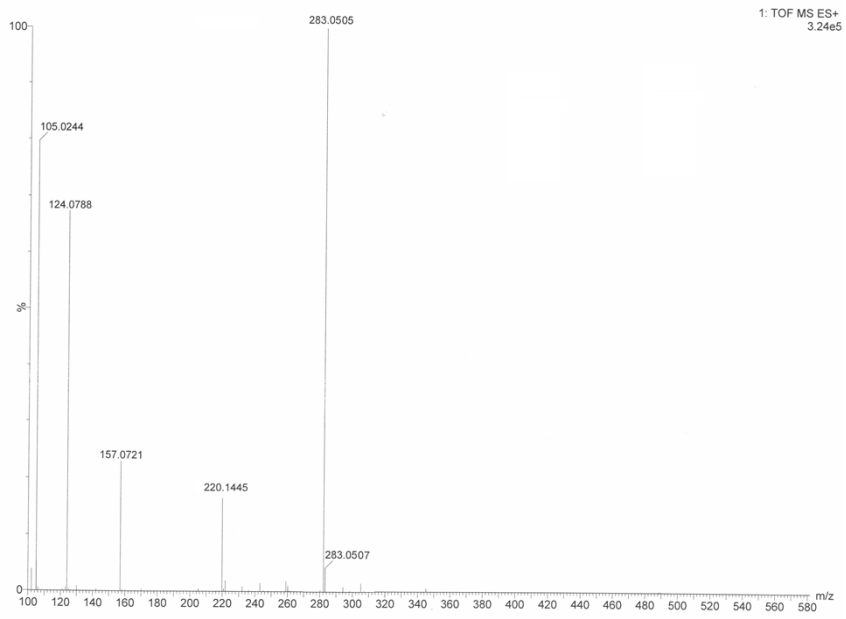

1: TOF MS ES+
1.79e4
283.0517
285.0534
385.1158
%
m/z

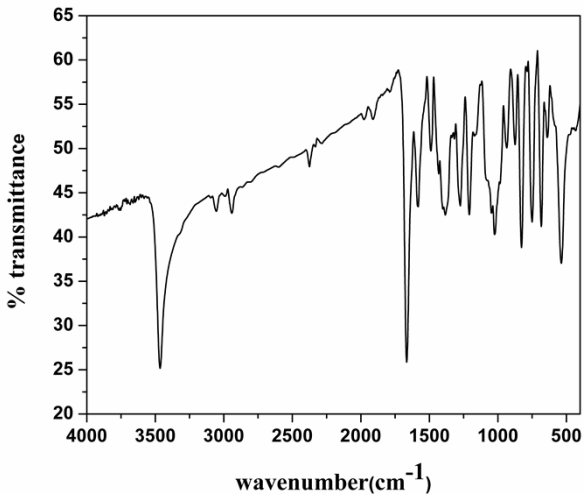

% transmittance
wavenumber(cm-1)

Entry 8

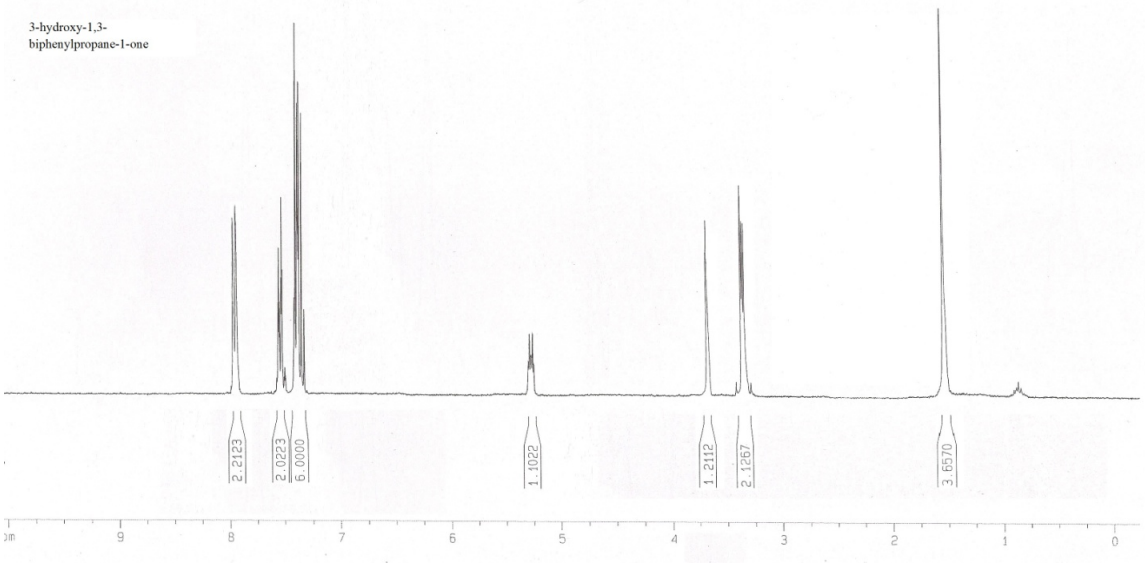

3-hydroxy-3-(4-chlorophenyl)-
1-phenylpropane-1-one
2.0000
1.0904
1.0136
1.9378
1.9967
0.9738
1.0850
1.0708
1.0077
1.0031
5.5191
ppm
8
7
6
5
4
3
2
1
0

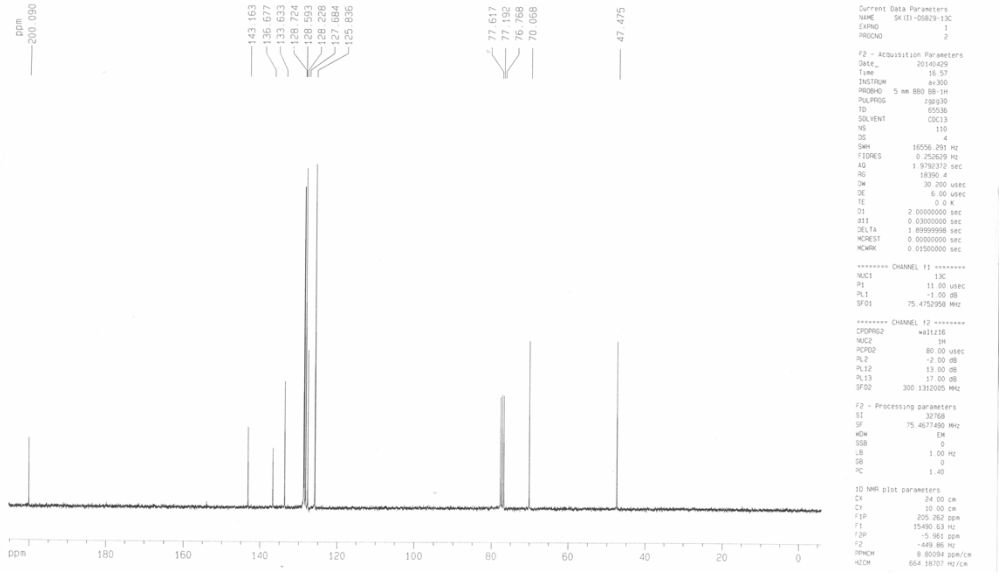

ppm
199.781
141.715
136.523
133.731
129.297
128.748
128.667
128.195
127.251
77.608
77.183
76.759
69.397
47.264
ppm
180
160
140
120
100
80
60
40
20
0


1: TOF MS ES+
3.24e5
283.0505
105.0244
124.0788
157.0721
220.1445
283.0507
%
m/z


% transmittance
wavenumber(cm-1)

Entry 9





Current Data Parameters
NAME SK(1)-05829-13C
EXPNO 1
PROCNO 2

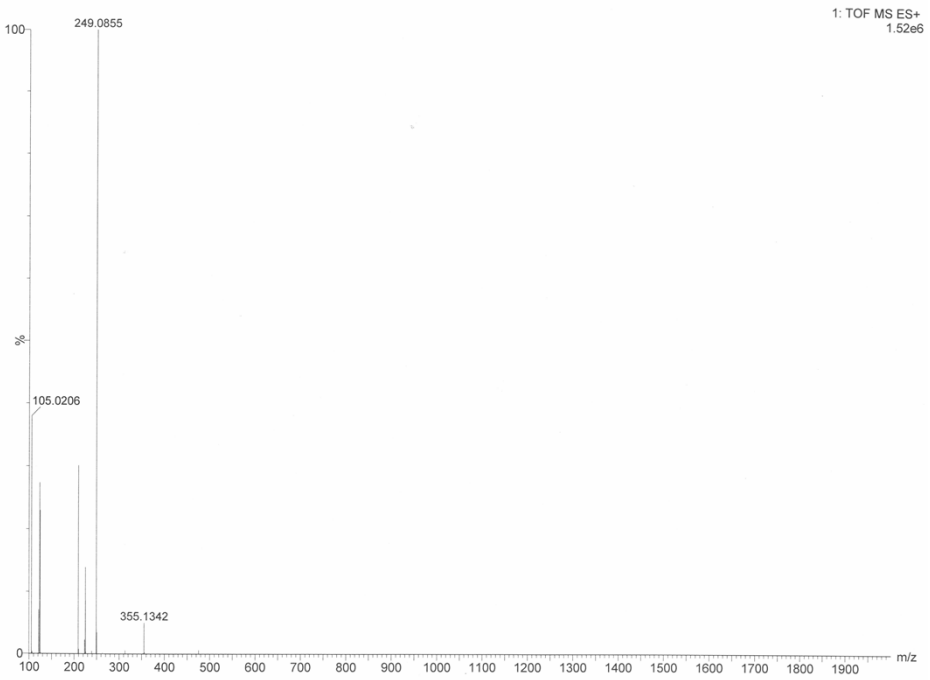
F2 - Acquisition Parameters
Date_ 20140429
Time 16.57
INSTRUM av300
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 110
DS 4
SWH 16556.291 Hz
FIDRES 0.252629 Hz
AQ 1.9792372 sec
RG 16390.4
DW 30.200 usec
DE 6.00 usec
TE 0.0 K
D1 2.00000000 sec
D11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

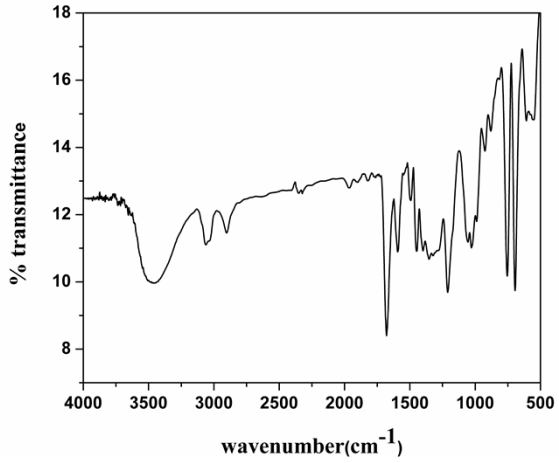
======== CHANNEL f1 ========
NUC1 13C
P1 11.00 usec
PL1 -1.00 dB
SFO1 75.4752958 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 13.00 dB
PL13 17.00 dB
SFO2 300.1312005 MHz

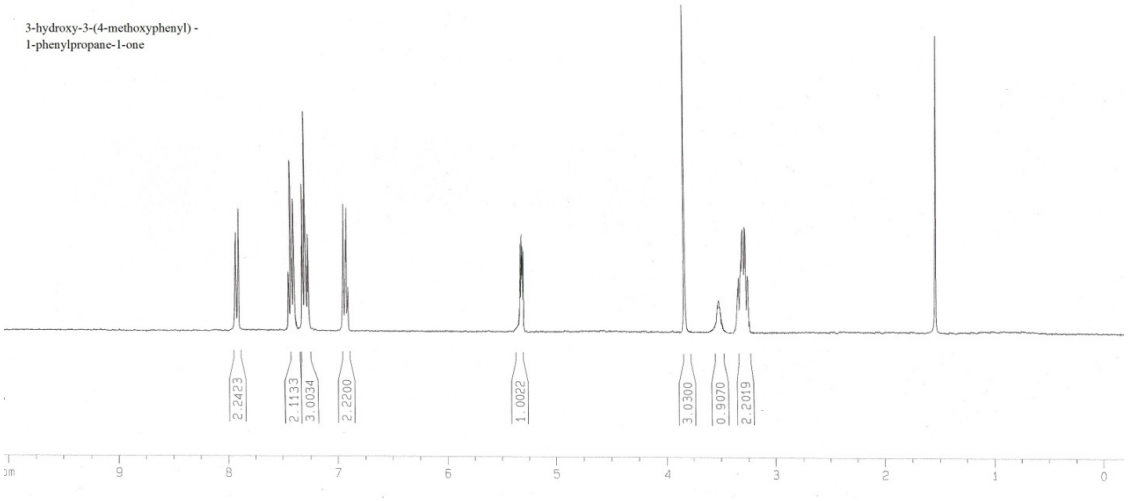
F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

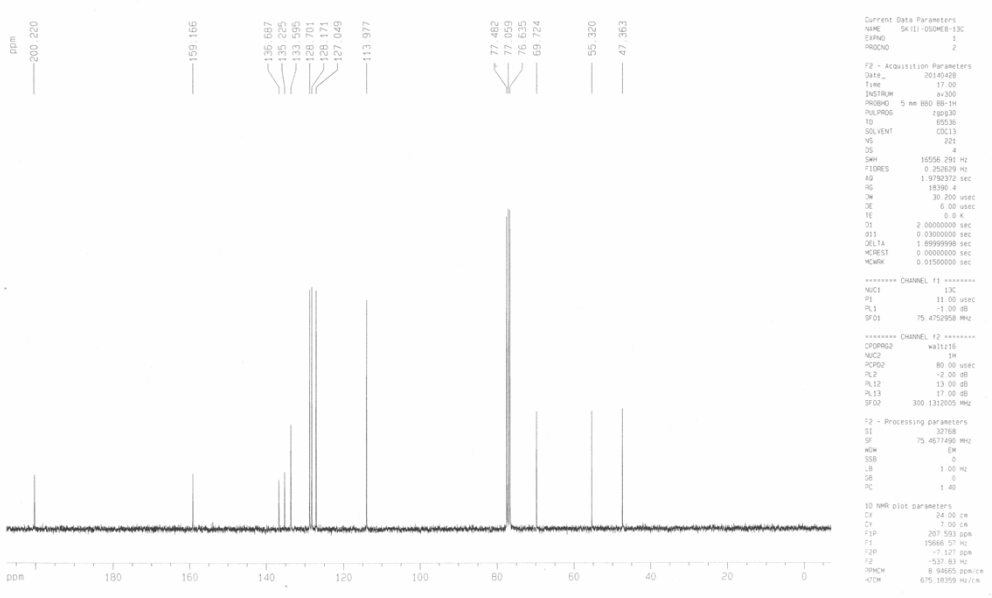
1D NMR plot parameters
CX 24.00 cm
CY 10.00 cm
F1P 205.262 ppm
F1 15490.63 Hz
F2P -5.961 ppm
F2 -449.86 Hz
PPMCM 8.80094 ppm/cm
HZCM 664.38707 Hz/cm

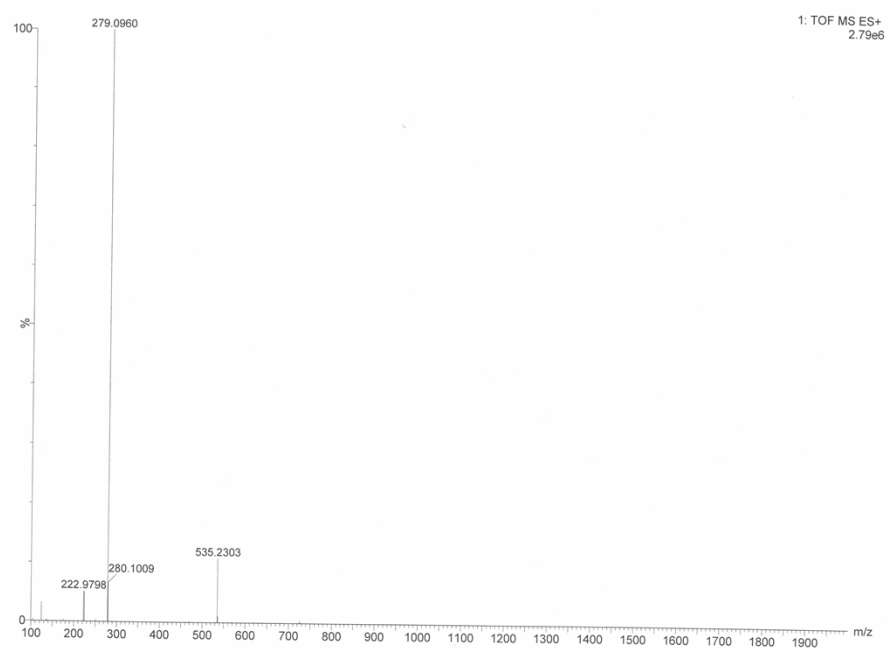

1: TOF MS ES+
1.52e6
249.0855
105.0206
355.1342
%
m/z
100
200
300
400
500
600
700
800
900
1000
1100
1200
1300
1400
1500
1600
1700
1800
1900

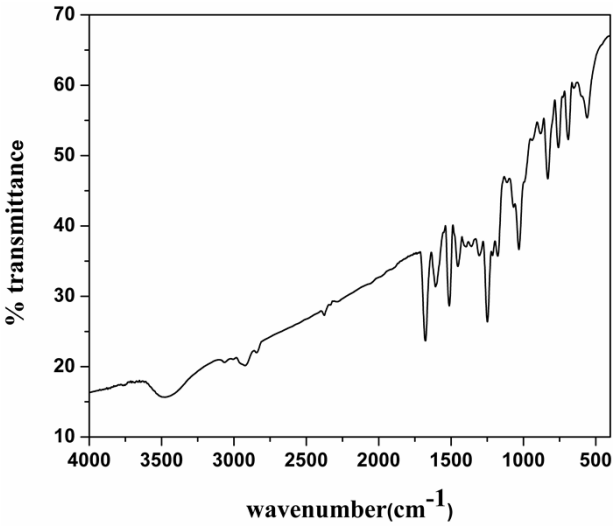

% transmittance
wavenumber(cm-1)
18
16
14
12
10
8
4000
3500
3000
2500
2000
1500
1000
500

Entry 10

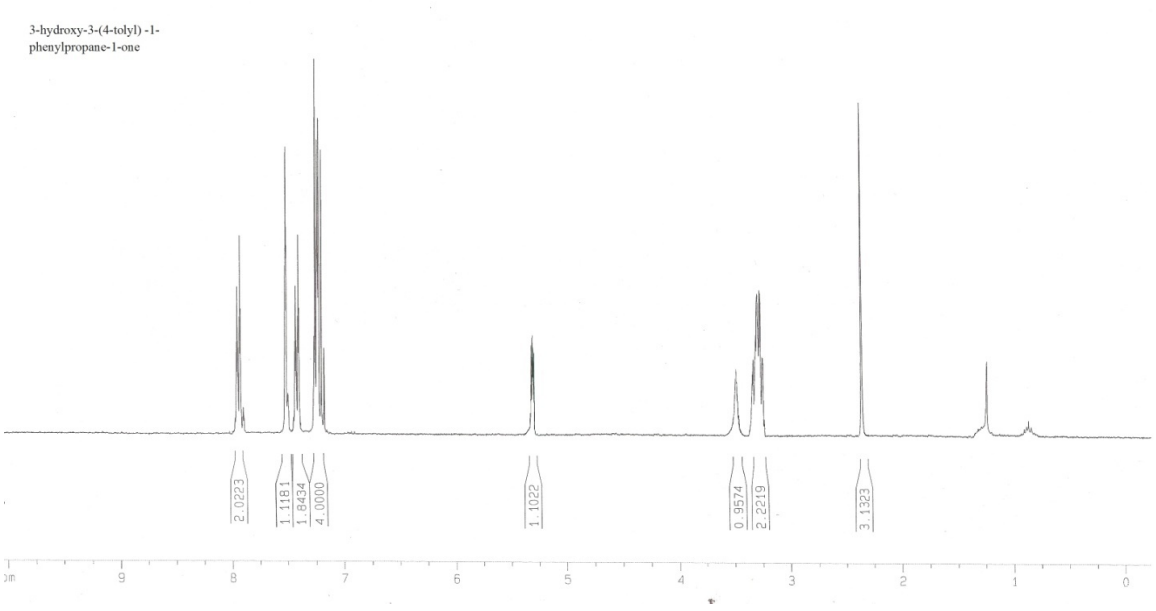

3-hydroxy-3-(4-methoxyphenyl) -
1-phenylpropane-1-one
2.2423
2.1133
3.0034
2.2200
1.0022
3.0300
0.9070
2.019
ppm
9
8
7
6
5
4
3
2
1
0

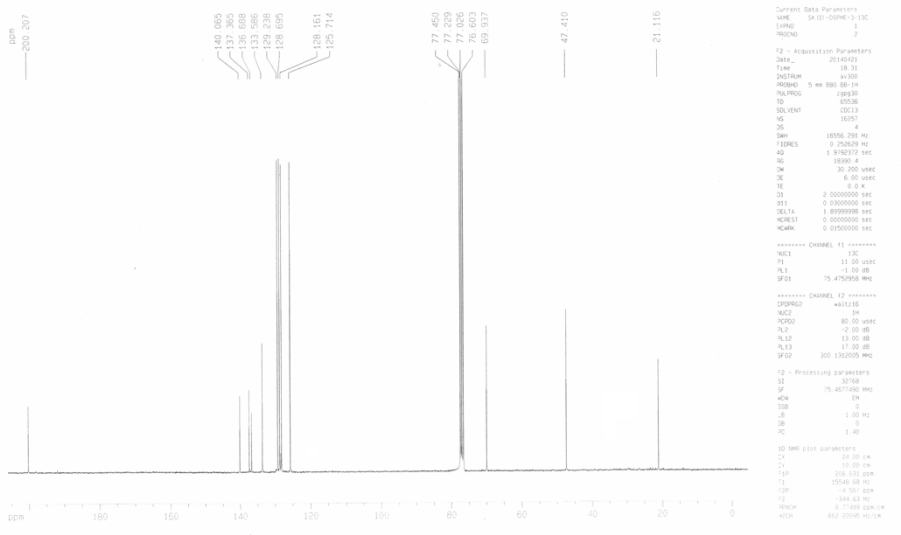

ppm
200.220
159.166
136.687
135.225
133.595
128.701
128.171
127.049
113.977
77.482
77.059
76.635
69.724
55.320
47.363
ppm
180
160
140
120
100
80
60
40
20
0
Current Data Parameters
NAME SK(I)-DSOMEB-13C
EXPNO 1
PROCNO 2
F2 - Acquisition Parameters
Date_ 20140428
Time 17.00
INSTRUM av300
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 221
DS 4
SWH 16556.291 Hz
FIDRES 0.252629 Hz
AQ 1.9792372 sec
RG 18390.4
DW 30.200 usec
DE 6.00 usec
TE 0.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 11.00 usec
PL1 -1.00 dB
SF01 75.4752958 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 13.00 dB
PL13 17.00 dB
SF02 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 24.00 cm
CY 7.00 cm
F1P 207.593 ppm
F1 15666.57 Hz
F2P -7.127 ppm
F2 -537.83 Hz
PPMCM 8.94665 ppm/cm
HZCM 675.18359 Hz/cm

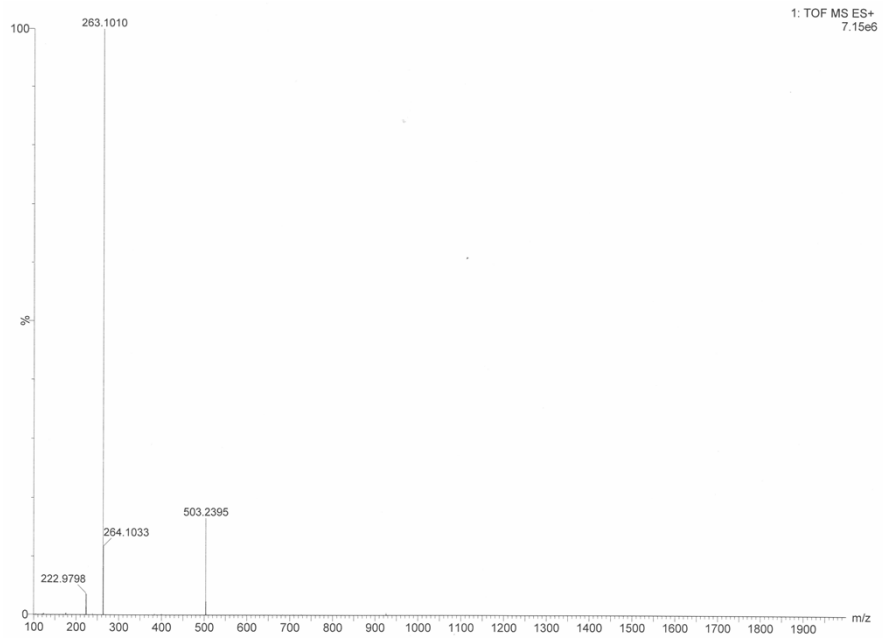

1: TOF MS ES+
2.79e6
279.0960
535.2303
280.1009
222.9798
100
%
0
100 200 300 400 500 600 700 800 900 1000 1100 1200 1300 1400 1500 1600 1700 1800 1900
m/z

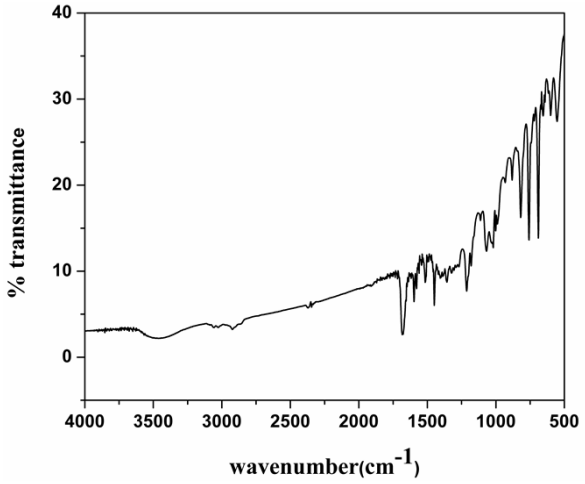

% transmittance
70
60
50
40
30
20
10
4000 3500 3000 2500 2000 1500 1000 500
wavenumber(cm-1)

Entry 11

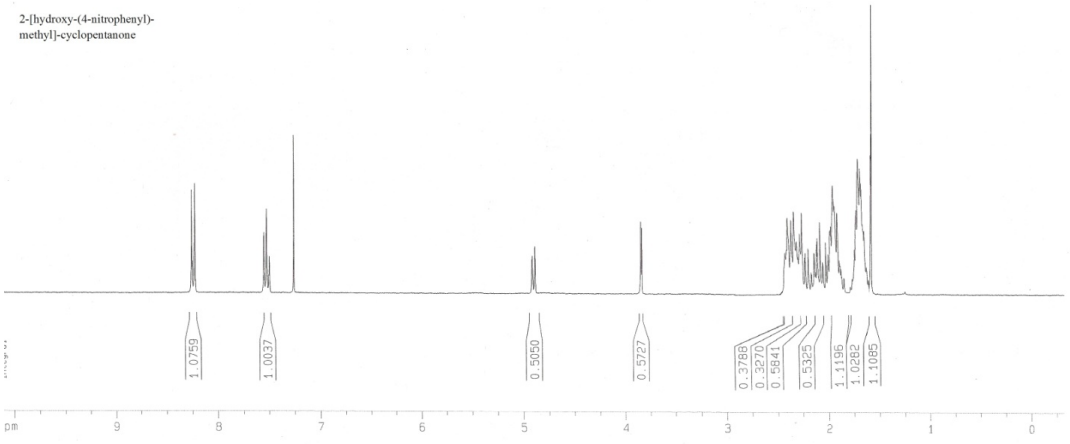

3-hydroxy-3-(4-tolyl) -1-
phenylpropane-1-one
2.0223
1.1181
1.8434
4.0000
1.1022
0.9574
2.2219
3.1323
9
8
7
6
5
4
3
2
1
0

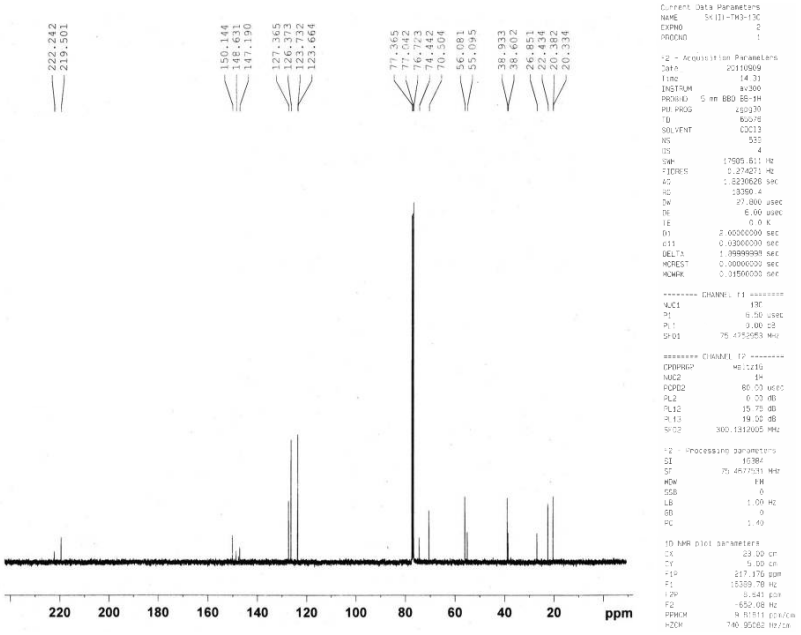

ppm
200.207
140.065
137.365
136.688
133.586
129.238
128.695
128.161
125.714
77.459
77.228
77.005
76.603
69.937
47.410
21.116
ppm
180
160
140
120
100
80
60
40
20
0


1: TOF MS ES+
7.15e6
100
%
0
263.1010
264.1033
503.2395
222.9798
100 200 300 400 500 600 700 800 900 1000 1100 1200 1300 1400 1500 1600 1700 1800 1900
m/z


% transmittance
40
30
20
10
0
4000 3500 3000 2500 2000 1500 1000 500
wavenumber(cm-1)

Entry 12





Current Data Parameters
NAME SKIII-TM3-13C
EXPNO 2
PROCNO 1

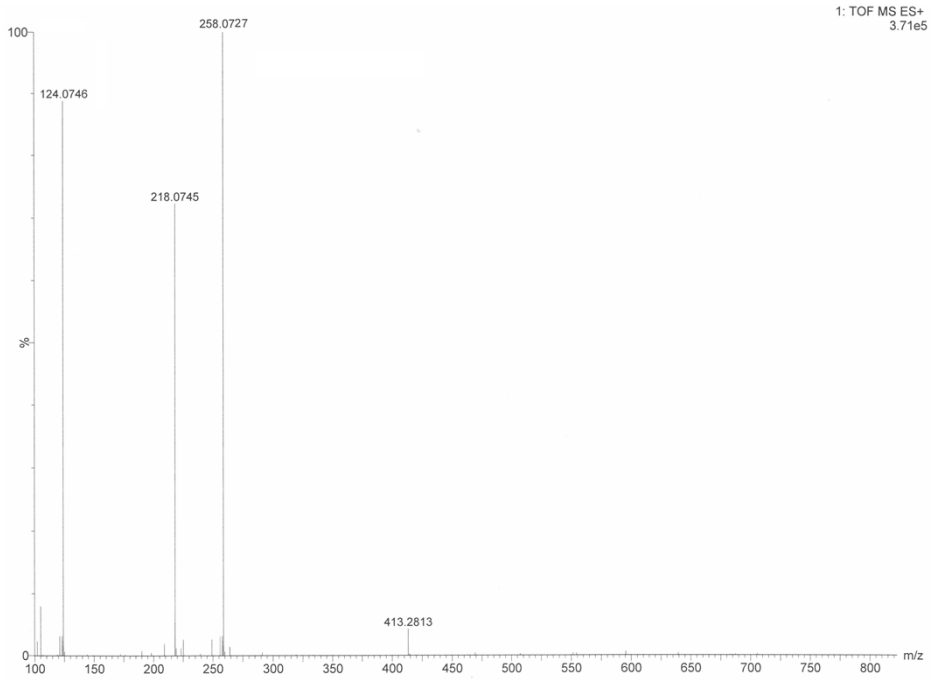
F2 - Acquisition Parameters
Date 20110909
Time 14.31
INSTRUM av300
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 532
DS 4
SWH 17605.611 Hz
FIDRES 0.274071 Hz
AQ 1.8230626 sec
RG 18350.4
DW 27.800 usec
DE 6.00 usec
TE 0.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.99999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

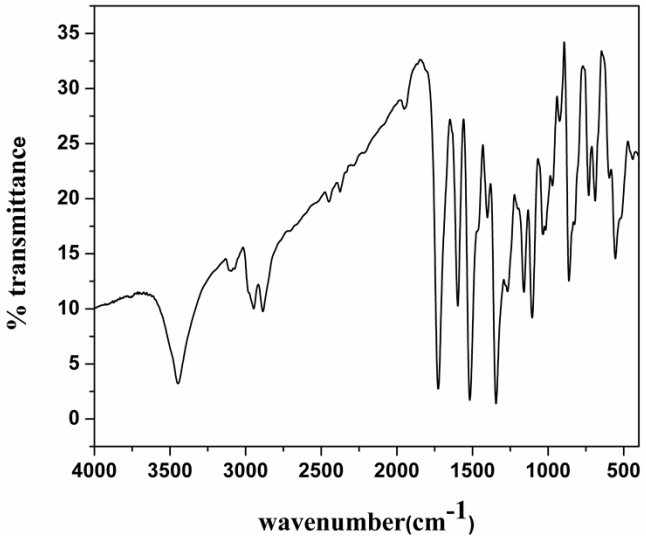
======== CHANNEL f1 ========
NUC1 13C
P1 6.50 usec
PL1 0.00 dB
SFO1 75.4753953 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 15.75 dB
PL13 19.00 dB
SFO2 300.1312005 MHz

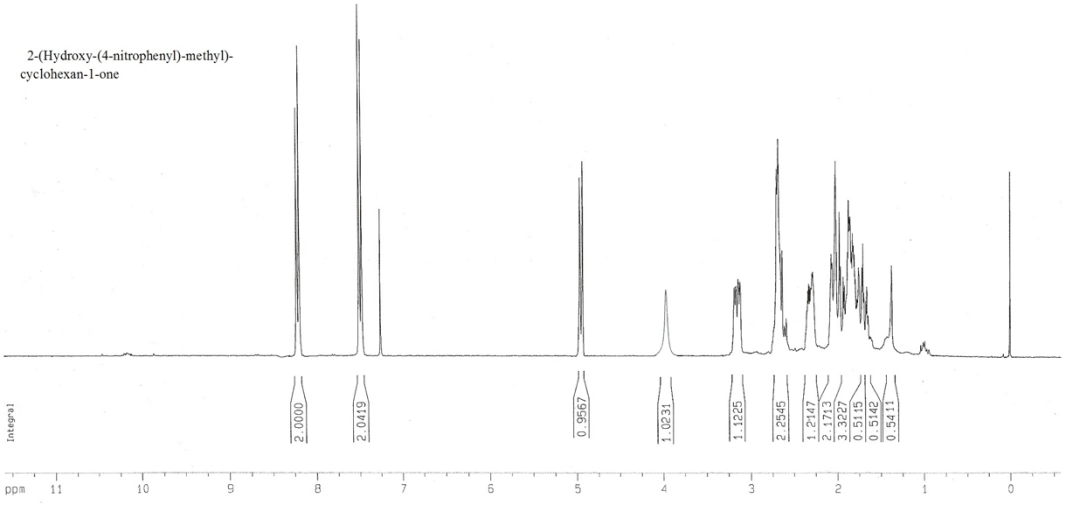
F2 - Processing parameters
SI 16384
SF 75.4677531 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

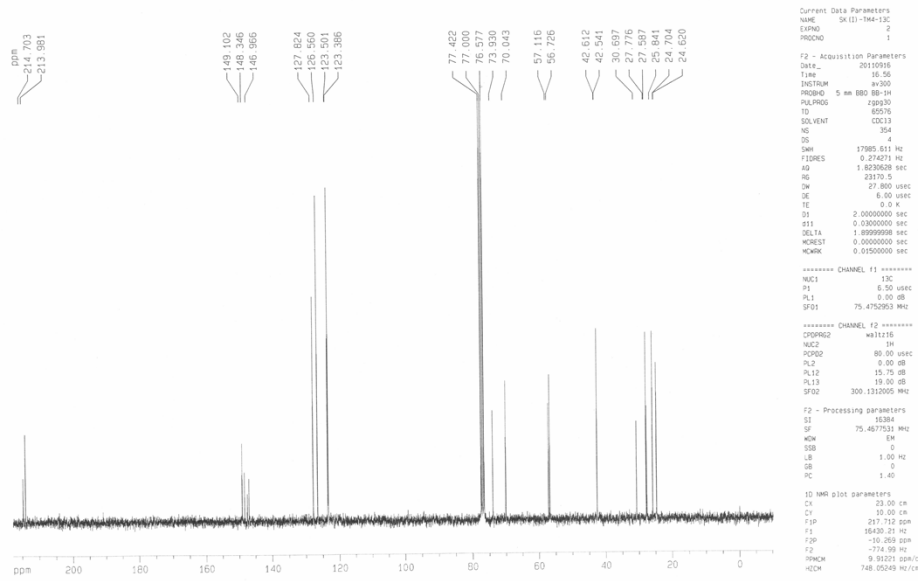
1D NMR plot parameters
CX 23.00 cm
CY 15.00 cm
F1P 217.176 ppm
F1 16389.78 Hz
F2P -8.641 ppm
F2 -652.08 Hz
PPMCM 9.81811 ppm/cm
HZCM 740.95062 Hz/cm


1: TOF MS ES+
3.71e5
258.0727
124.0746
218.0745
413.2813
%
m/z
100
150
200
250
300
350
400
450
500
550
600
650
700
750
800

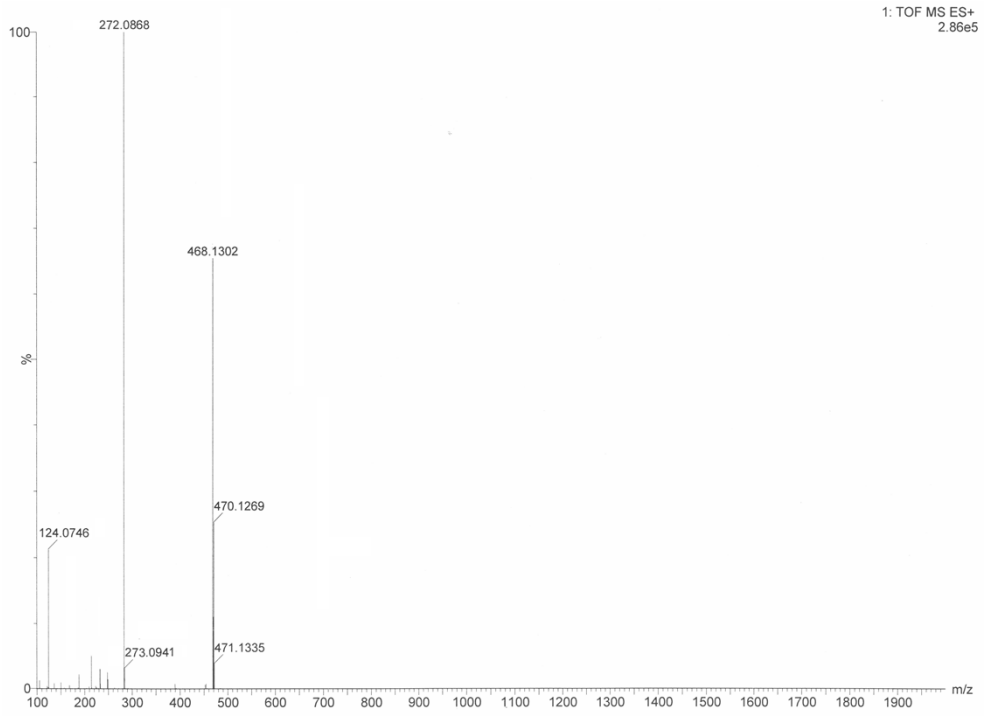

% transmittance
wavenumber(cm-1)
35
30
25
20
15
10
5
0
4000
3500
3000
2500
2000
1500
1000
500

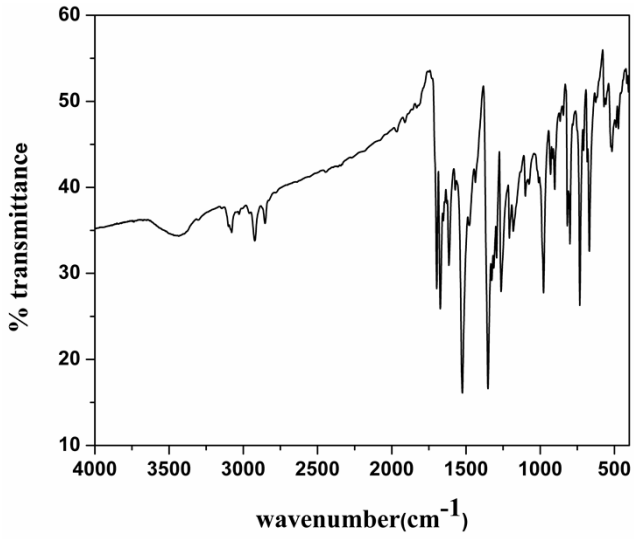
Entry 13






```
Current Data Parameters
NAME          SK(I)-TM4-13C
EXPNO                     2
PROCNO                    1

F2 - Acquisition Parameters
Date_          20110916
Time              16.56
INSTRUM           av300
PROBHD  5 mm BBO BB-1H
PULPROG         zgpg30
TD               65576
SOLVENT          CDCl3
NS                 354
DS                   4
SWH           17985.611 Hz
FIDRES        0.274271 Hz
AQ            1.8236628 sec
RG               2370.5
DW              27.800 usec
DE               6.00 usec
TE                0.0 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1               13C
P1                6.50 usec
PL1               0.00 dB
SFO1        75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2               1H
PCPD2            80.00 usec
PL2               0.00 dB
PL12             15.75 dB
PL13             19.00 dB
SFO2       300.1312005 MHz

F2 - Processing parameters
SI                16384
SF          75.4677531 MHz
WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC                1.40

1D NMR plot parameters
CX               23.00 cm
CY               10.00 cm
F1P             217.712 ppm
F1              16430.21 Hz
F2P             -10.269 ppm
F2              -774.99 Hz
PPMCM          9.91021 ppm/c
HZCM          748.05249 Hz/cm
```


1: TOF MS ES+
2.86e5
272.0868
468.1302
470.1269
124.0746
273.0941
471.1335
%
m/z


% transmittance
wavenumber(cm-1)