Supplementary Material (ESI) for Chemical Communications
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Electronic Supplementary Information (ESI) for:

Hantzsch reaction on free nano-Fe_2O_3 catalyst: Excellent reactivity combined with facile catalyst recovery and recyclability

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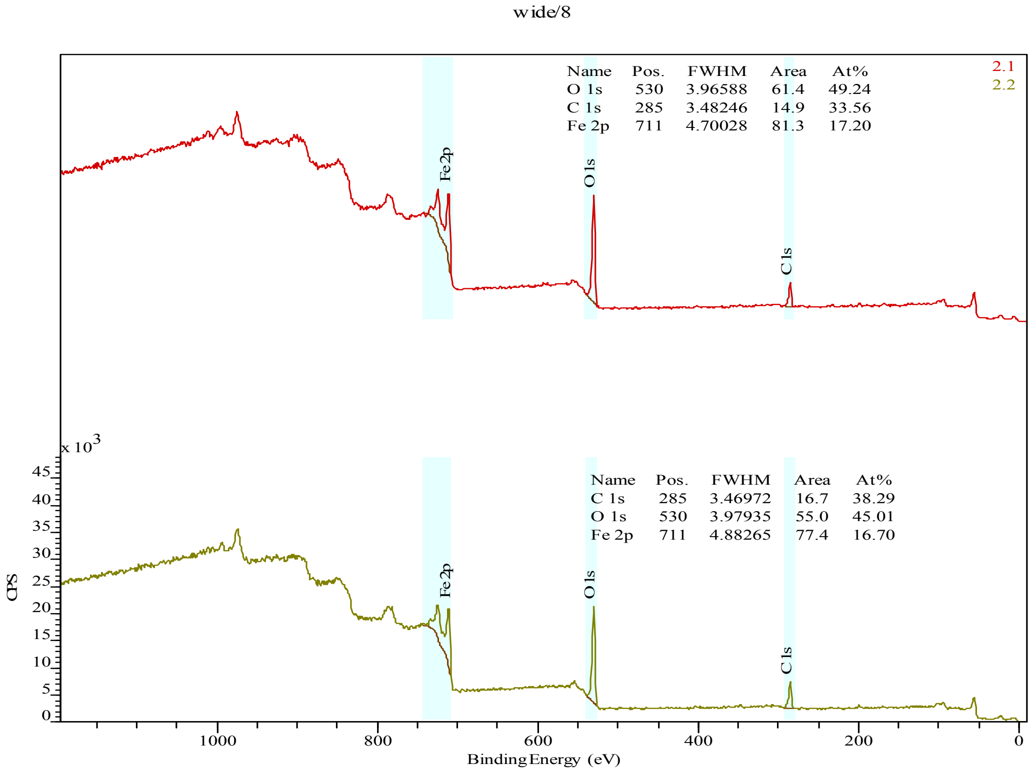
Instrumentation, Analysis and Starting Materials:

Experimental:

General: All chemicals were purchased from Merck, Fluka or Acros companies and used without any further purification. Nano-γ-Fe_2O_3 was prepared with the reported method.[1-2] NMR spectra were recorded with a Bruker Avance 300 spectrometer (^{1}HNMR 300 MHz and ^{13}CNMR 75 MHz) in pure deuteriated chloroform, acetone and dimethyl sulfoxide with tetramethylsilane (TMS) as the internal standard. X-ray data was measured on a STOE IPDS-II two circle diffractometer at 120 K, using graphite monochromated Mo Ka X-ray radiation (k = 0.7107 Å), Suitable crystal was obtained from ethanol solution by the slow evaporation of the solvent over several days. Scanning electron microscope (SEM, Philips, XL30, Netherlands) was used for preparation of SEM images. Transmission electron microscope, TEM (Philips CM10) was also used to obtain TEM images. The crystal structure of synthesized materials was determined by an X-ray diffractometer (XRD) (38066 RIVA, d/G. Via M. Misone, 11/D (TN), Italy) at ambient temperature. XPS spectra were acquired using a Kratos Axis ULTRA “DLD” X-ray photoelectron spectrometer equipped with a monochromatic Al-KR X-ray source. The pass energies were 160eV for the survey scans and 20 eV for the high resolution scans. The instrument was operated in electrostatic mode as the samples were magnetic. The magnetic measurement was carried out in a vibrating sample magnetometer (VSM) (4 inch, Daghigh Meghnatis Kashan Co., Kashan, Iran) at room temperature.

General procedure for the synthesis of nano-γ-Fe_2O_3:

Magnetite (Fe_3O_4) nanoparticles were prepared by partial reduction coprecipitation method based on the procedure of S.C. Qu, H.B. Yang, S.H. Kan, et al.[2] 3 mL $FeCl_3$ (2 mol L^{-1} dissolved in 2 mol L^{-1} HCl) was added to 10.33 mL double distilled water, and 2 mL Na_2SO_3 (1 mol L^{-1}) was added to the former solution dropwise in 1min under magnetic stirring. When the color of the solution turned back from red to light yellow, the solution was added to 80 mL $NH_3 \cdot H_2O$ solution (0.85 mol L^{-1}) under vigorous stirring. After 30 min, the magnetite precipitates were washed to pH<7.5 by deoxygenated water. The black precipitate obtained above was diluted to a volume of 168 mL with a mass concentration of 6 mg mL^{-1}. The pH of the suspension was adjusted to 3.0 with HCl (0.1 mol L^{-1}) and the pH was kept stable for 5min. The temperature of the suspension was raised to 90 °C in 5min, and was stirred under aeration (with air) for 60 min at about 100 °C. The reddish-brown solution was washed with water by magnetic decantation four times.[1]



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Fe 2p	711	4.70028	81.3	17.20

Name	Pos.	FWHM	Area	At%
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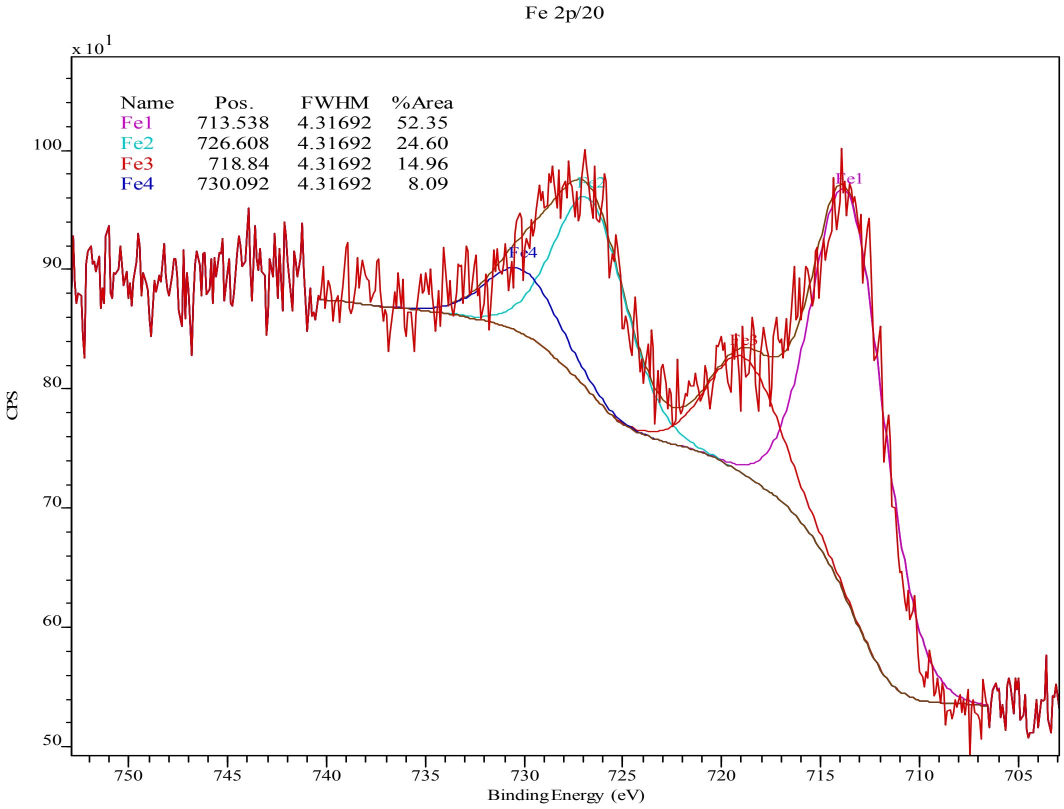
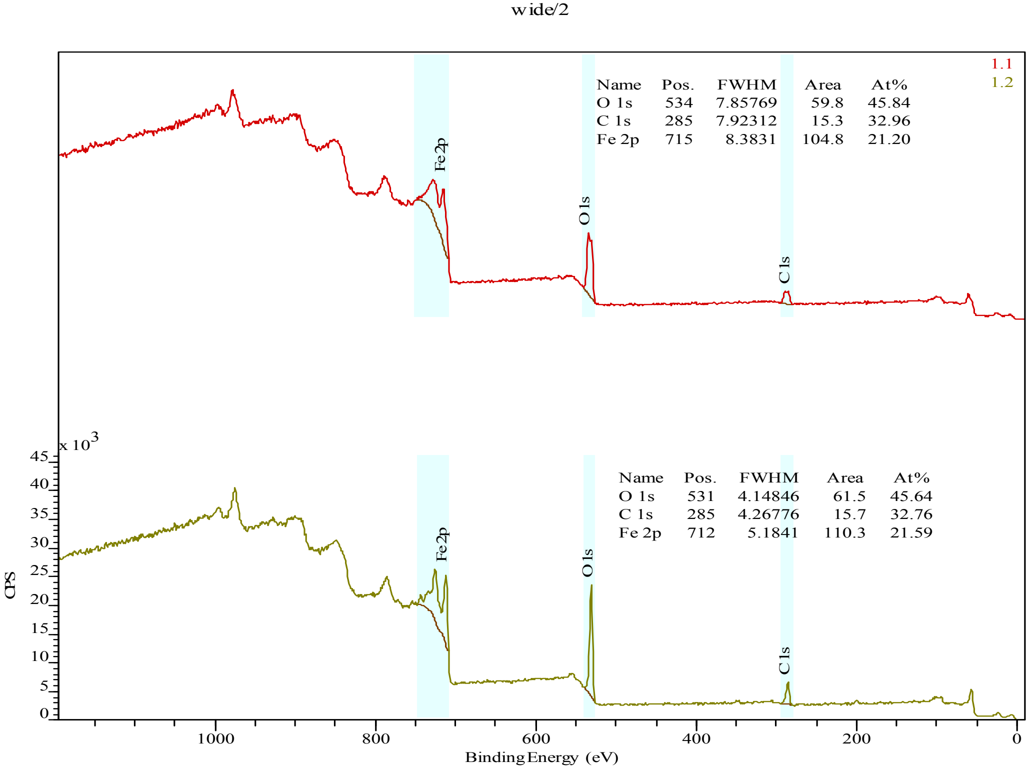


Name	Pos.	FWHM	%Area
Fe1	713.538	4.31692	52.35
Fe2	726.608	4.31692	24.60
Fe3	718.84	4.31692	14.96
Fe4	730.092	4.31692	8.09

Figure S1. XPS spectrum of nano Fe_3O_4.



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Name	Pos.	FWHM	Area	At%
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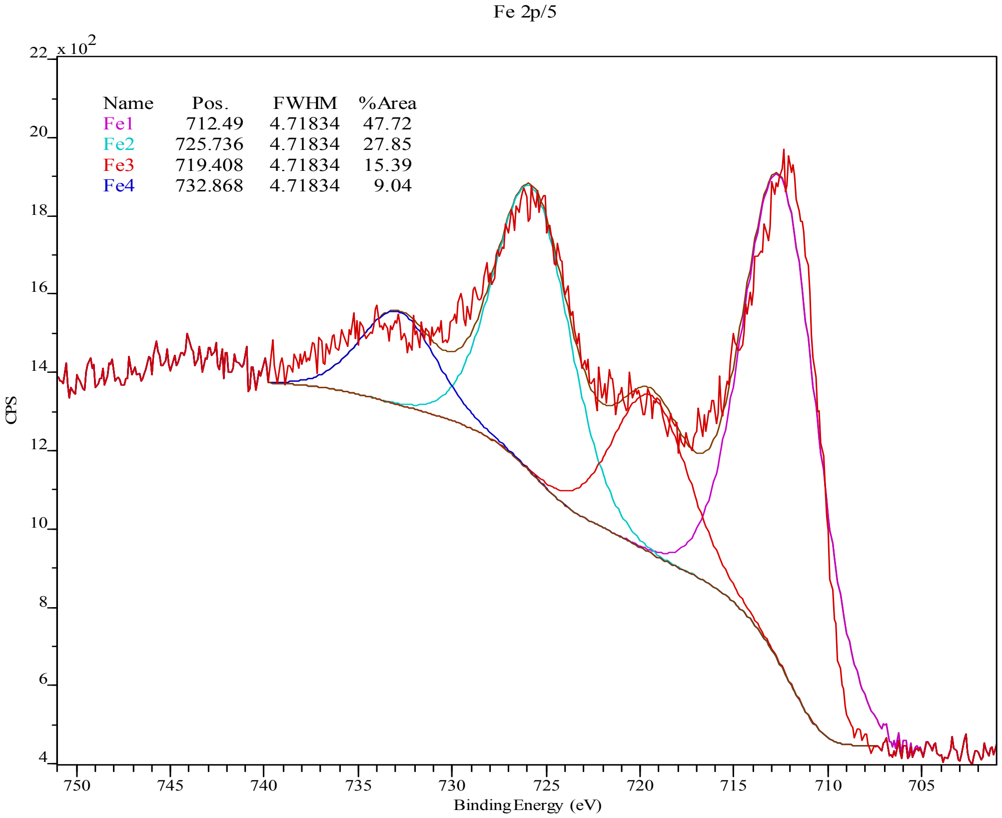
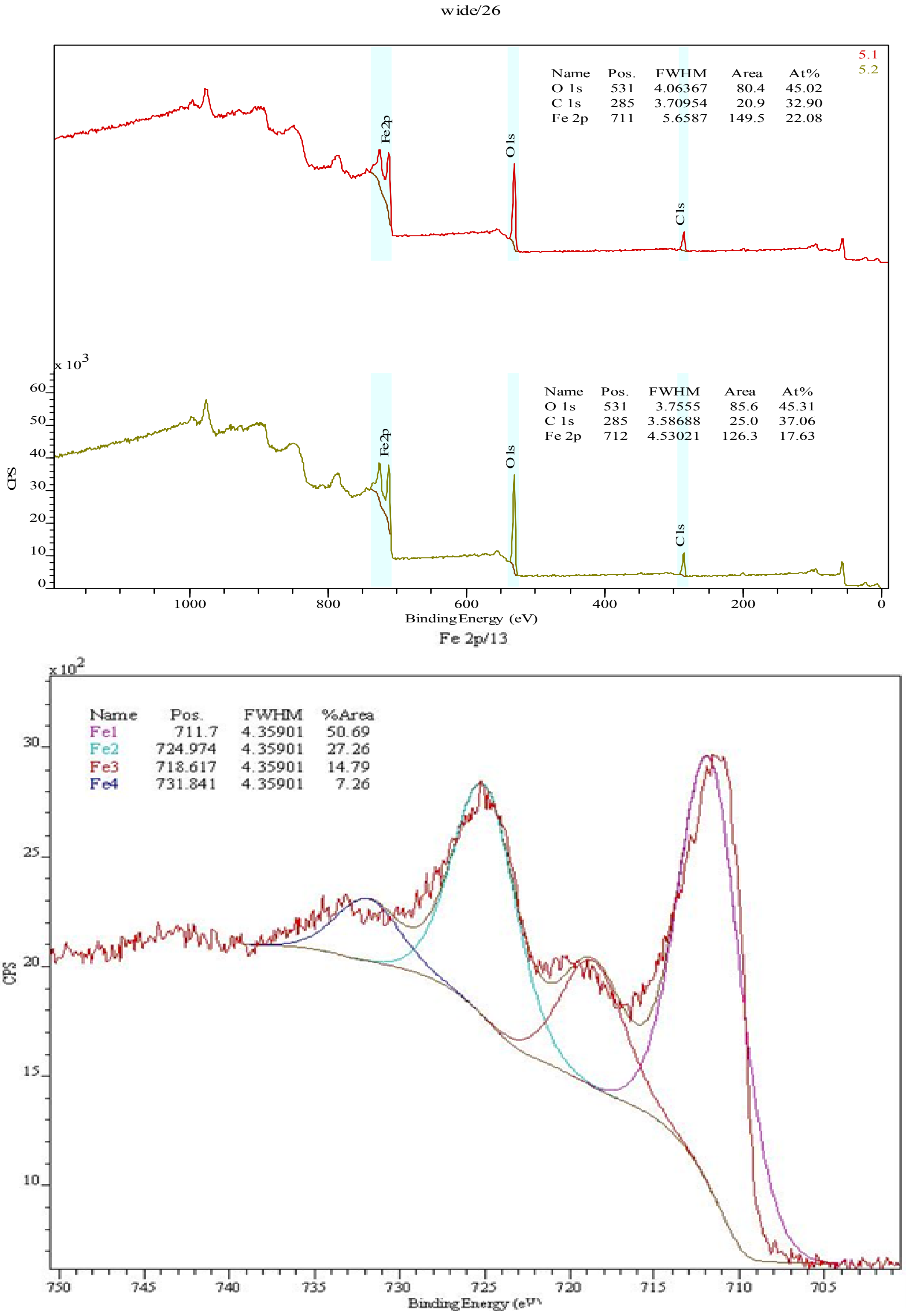


Name	Pos.	FWHM	%Area
Fe1	712.49	4.71834	47.72
Fe2	725.736	4.71834	27.85
Fe3	719.408	4.71834	15.39
Fe4	732.868	4.71834	9.04

Figure S2. XPS spectrum of nano-γ-Fe_2O_3.[3]



wide/26

Name	Pos.	FWHM	Area	At%
O 1s	531	4.06367	80.4	45.02
C 1s	285	3.70954	20.9	32.90
Fe 2p	711	5.6587	149.5	22.08

Name	Pos.	FWHM	Area	At%
O 1s	531	3.7555	85.6	45.31
C 1s	285	3.58688	25.0	37.06
Fe 2p	712	4.53021	126.3	17.63

Fe 2p/13

Name	Pos.	FWHM	%Area
Fe1	711.7	4.35901	50.69
Fe2	724.974	4.35901	27.26
Fe3	718.617	4.35901	14.79
Fe4	731.841	4.35901	7.26

Figure S3. XPS spectrum of nano-γ-Fe_2O_3 after reuse five times.

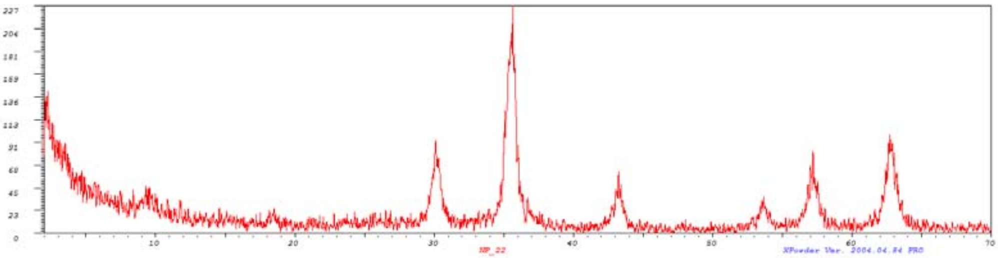


Figure S4. XRD patterns of the nano-γ-Fe_2O_3.[4-5]

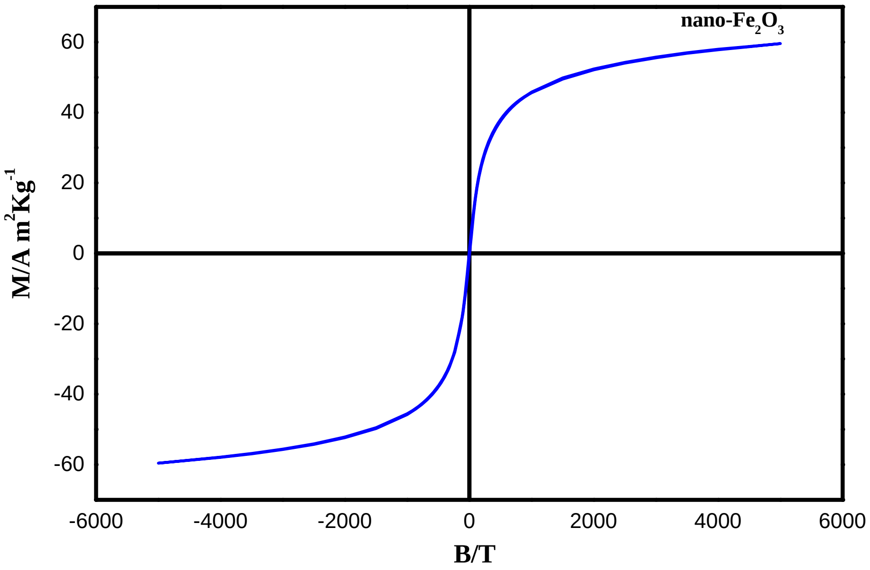


Figure S5: Magnetization curve for the Fe_2O_3 nanoparticles at room temperature.

The room-temperature magnetization curve of the magnetic nanoparticles proves that the magnetic nanoparticles are superparamagnetic. The saturation magnetization of ~60 emu/g was determined for the fine Fe_2O_3 nanoparticles.

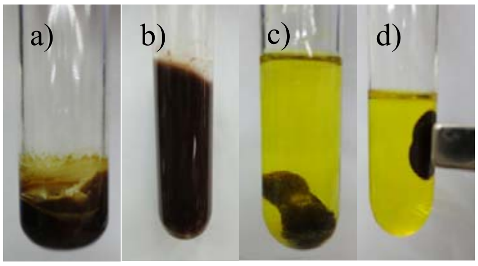


Figure S6: Photographs of suspension of nano-γ-Fe_2O_3 before (a, b) and after (c, d) magnetic capture.

The particles suspended smoothly by eccentricity force after the magnet bar start to stir, so that, the suspended Fe_2O_3 particles turned the reaction color into smooth brown during the reaction. Because of the reaction carried out in solvent free condition and the product is in viscose form, in order to make separation easier, at the end of the reaction, the mixture was triturated with ethyl acetate and after 10 s suspended particles moved on to the stirrer bar. Using an external magnet can make the separation of Nps easier during the cycling.

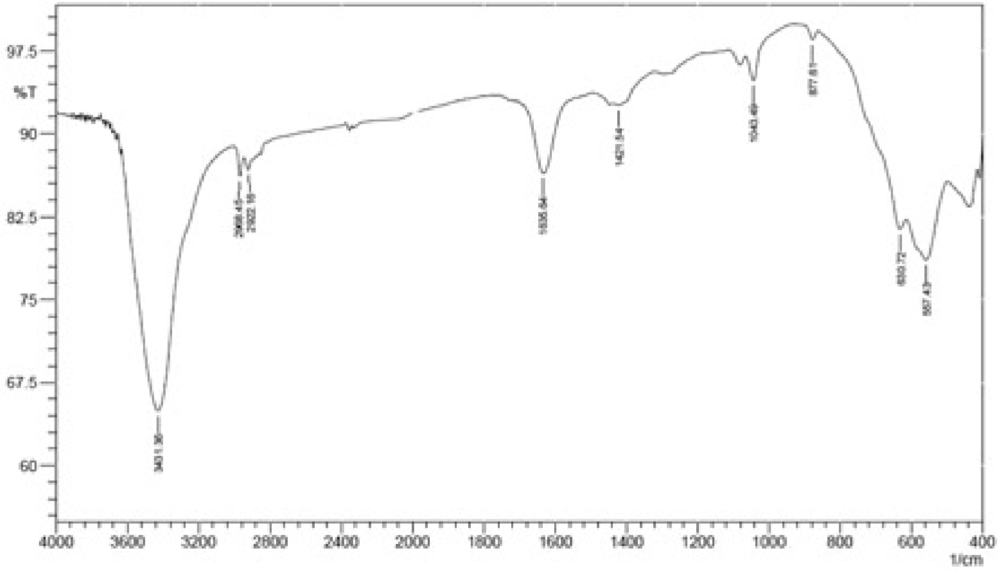


Figure S7: FTIR spectra of γ-Fe_2O_3 nanoparticles.[6]

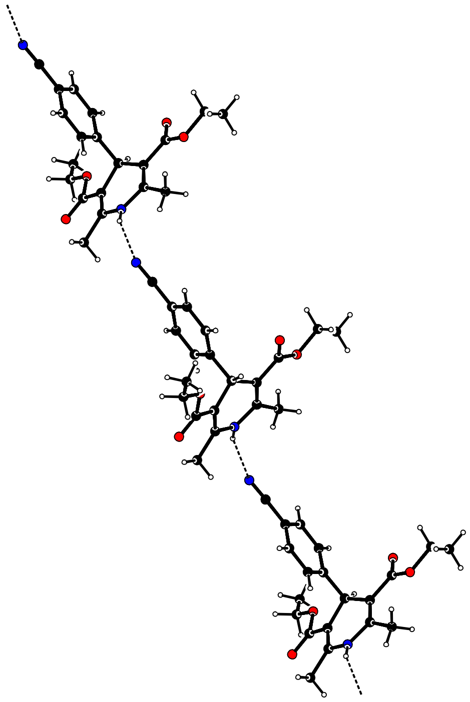
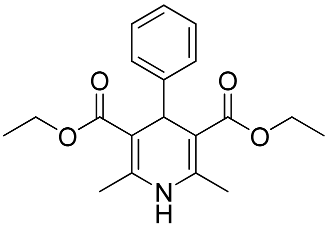


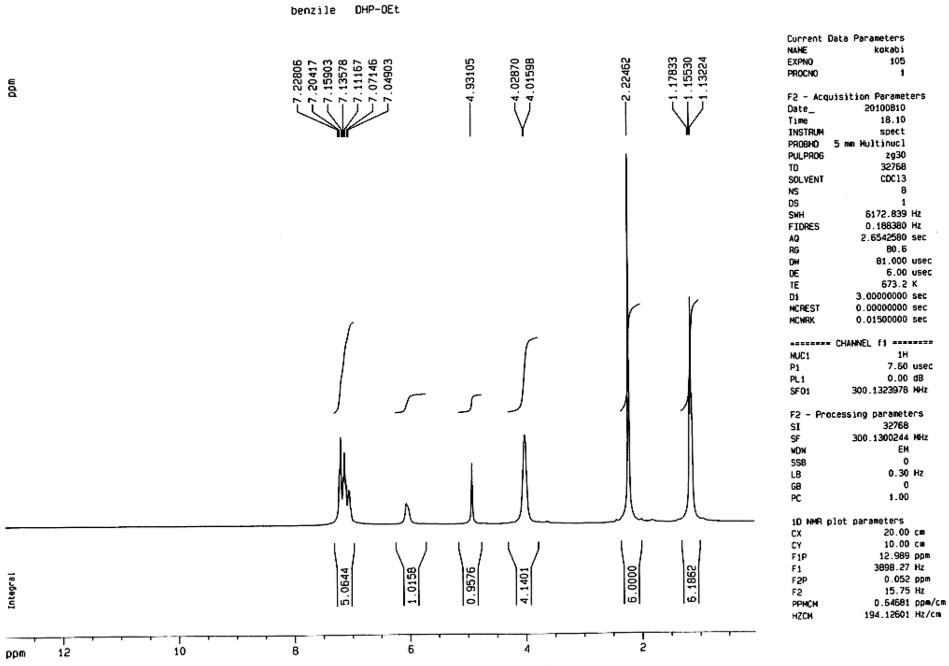
Figure S8. X-ray structure of 4g.

Spectral data:

Diethyl 2,6-dimethyl-4-phenyl-1,4-dihydropyridine-3,5-dicarboxylate[7]



Mp: 157-159 °C; ^{1}H NMR ($CDCl_3$, 300 MHz): δ (ppm): 1.15 (t, 6 H), 2.22 (s, 6 H), 4.0 (q, 4 H), 4.93 (s, 1 H), 6.2 (b, 1 H), 7.04-7.22 (m, 5 H); ^{13}C NMR ($CDCl_3$, 75 MHz): δ (ppm): 14.26, 19.40, 39.63, 59.74, 103.93, 126.1, 127.84, 127.98, 144.23, 147.85, 167.81; Elemental Analysis: Calcd. C: 69.28, H: 7.04, N: 4.25, O: 19.43, Found. C: 69.12, H: 7.00, N: 4.21, O: 19.39.



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TE                 673.2 K
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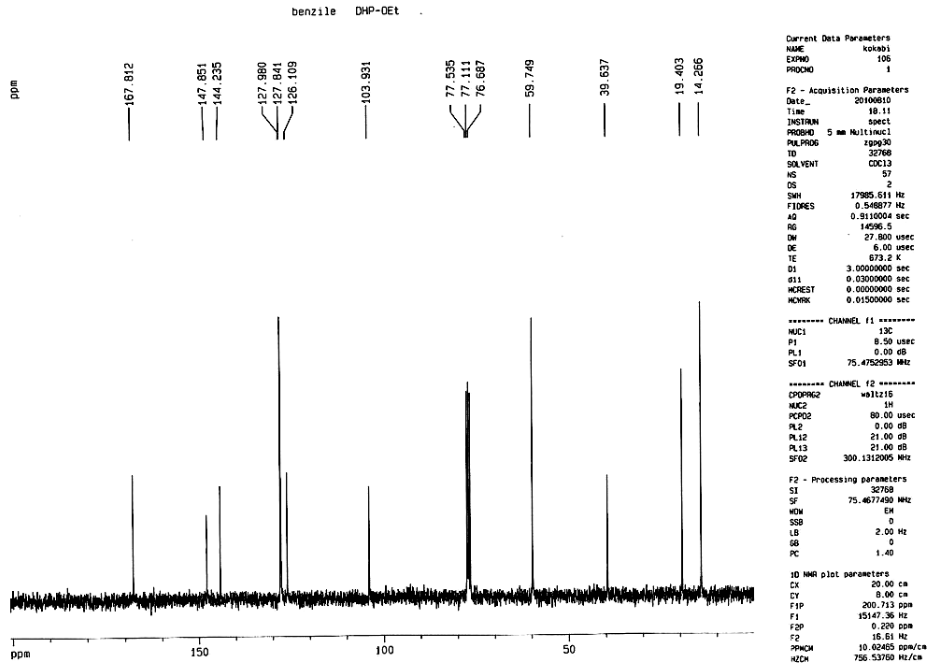
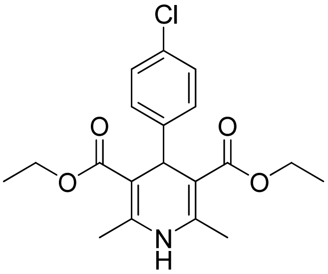
^{1}H NMR of 4a

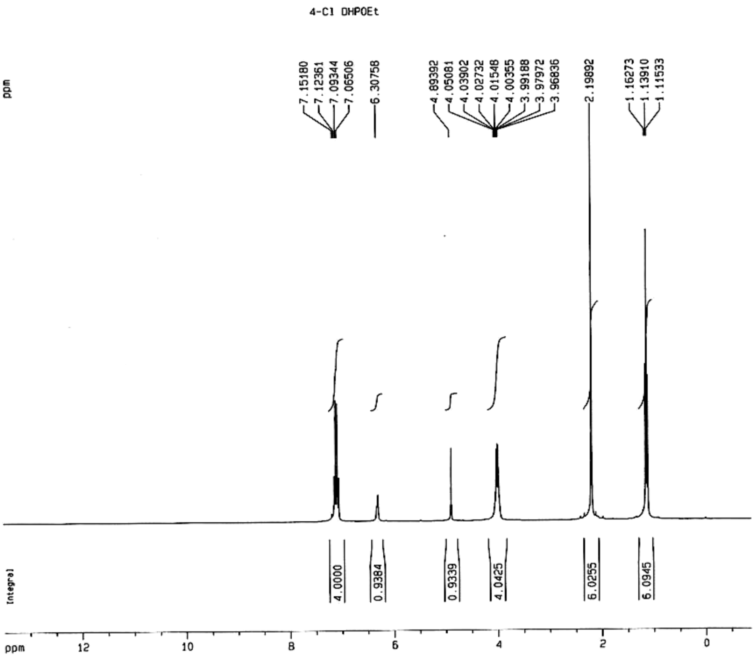


^{13}C NMR of 4a

Diethyl 4-(4-chlorophenyl)-2,6-dimethyl-1,4-dihydropyridine-3,5-dicarboxylate[7]



Mp: 144-146 °C; ^{1}H NMR ($CDCl_3$, 300 MHz): δ (ppm): 1.16-1.11 (t, 6 H), 2.19 (s, 6 H), 4.03-5.96 (q, 4 H), 4.89 (s, 1 H), 6.30 (s, 1 H), 7.15-7.06 (q, 4 H); ^{13}C NMR ($CDCl_3$, 75 MHz): δ (ppm): 14.27, 19.31, 39.24, 59.85, 103.50, 127.92, 129.36, 131.66, 144.56, 146.45, 167.68; Elemental Analysis: Calcd. C: 62.72, H: 6.09, N: 3.85, O: 17.59, Found. C: 62.46, H: 6.02, N: 3.80, O: 17.48.



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AQ             2.6542580 sec
RG                  50.8
DW                81.000 usec
DE                  6.00 usec
TE                 673.2 K
D1            3.00000000 sec
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MCWRK         0.01500000 sec

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1D NMR plot parameters
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F1               4051.36 Hz
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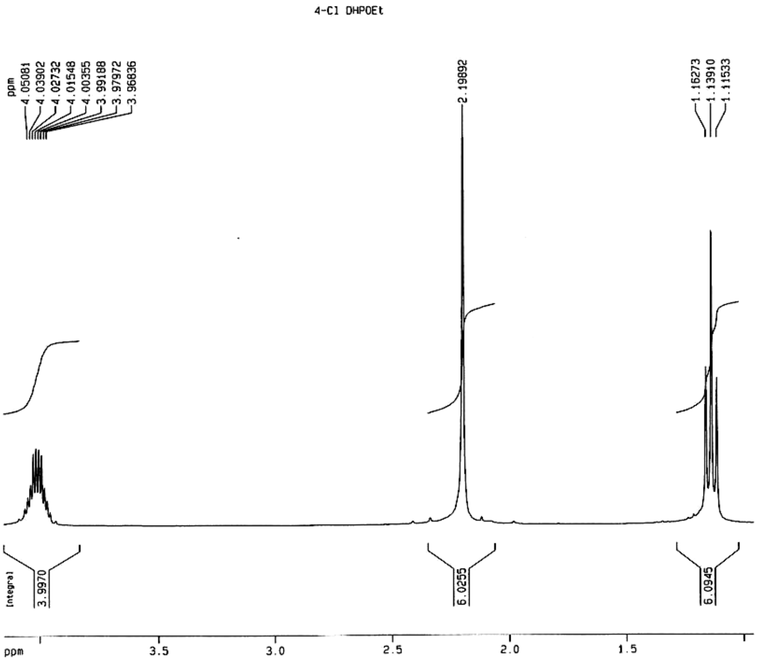
^{1}**H NMR of 4c**



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NS                     8
DS                     1
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FIDRES          0.188380 Hz
AQ             2.6542580 sec
RG                  50.8
DW                81.000 usec
DE                  6.00 usec
TE                 673.2 K
D1            3.00000000 sec
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MCWRK         0.01500000 sec

======== CHANNEL f1 ========
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P1                  7.60 usec
PL1                 0.00 dB
SFO1         300.1323978 MHz

F2 - Processing parameters
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WDW                   EM
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1D NMR plot parameters
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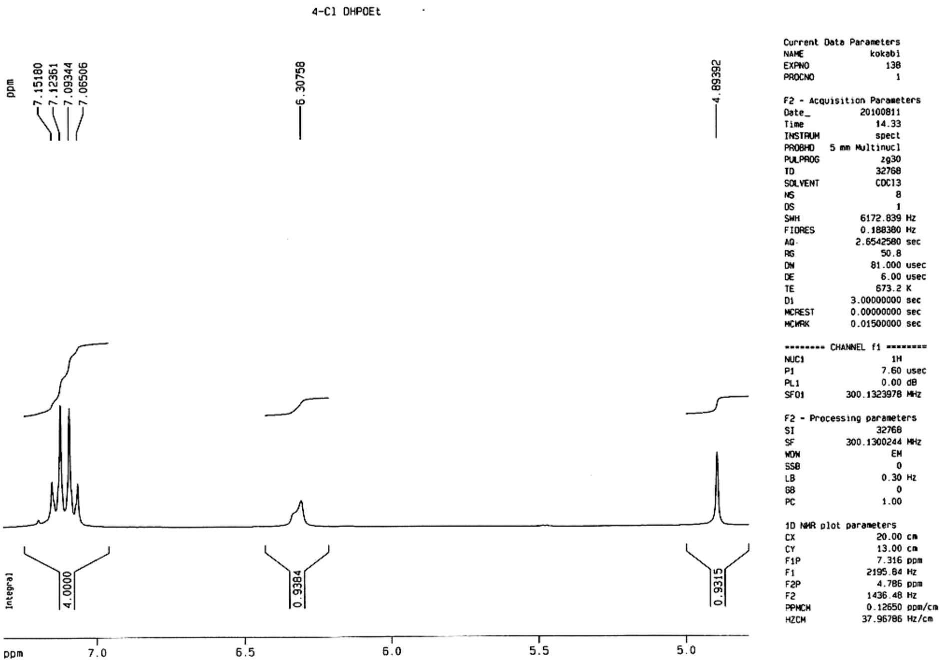
Expanded^{1}H NMR of 4c



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TD                 32768
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DS                     1
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RG                  50.8
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TE                 673.2 K
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PL1                 0.00 dB
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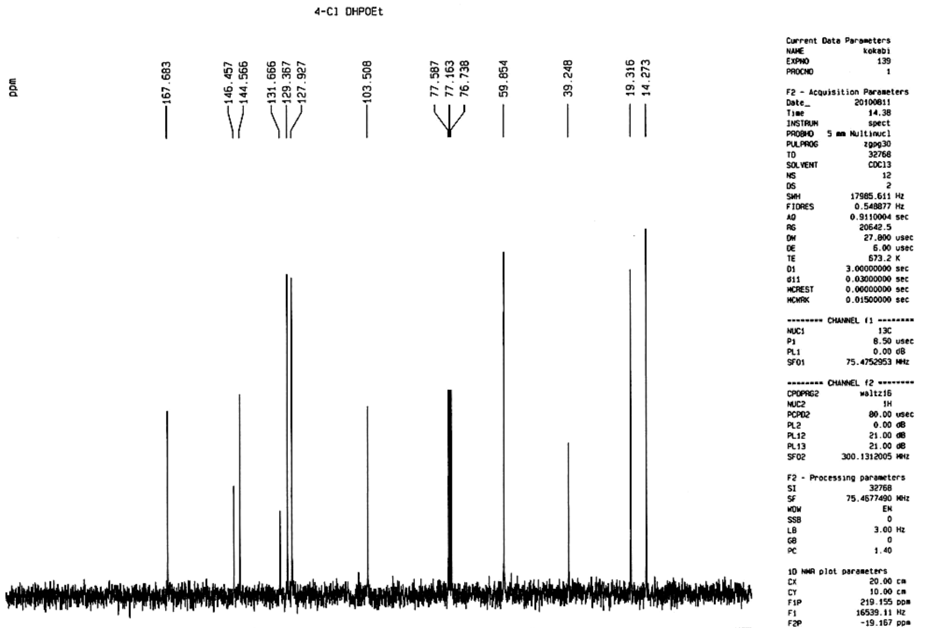
Expanded ^{1}H NMR of 4c



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F2              -1446.51 Hz
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HZCM           899.28058 Hz/cm
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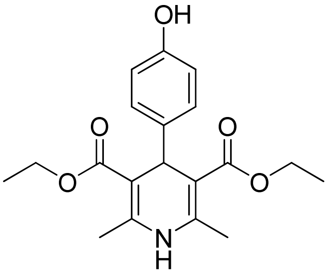
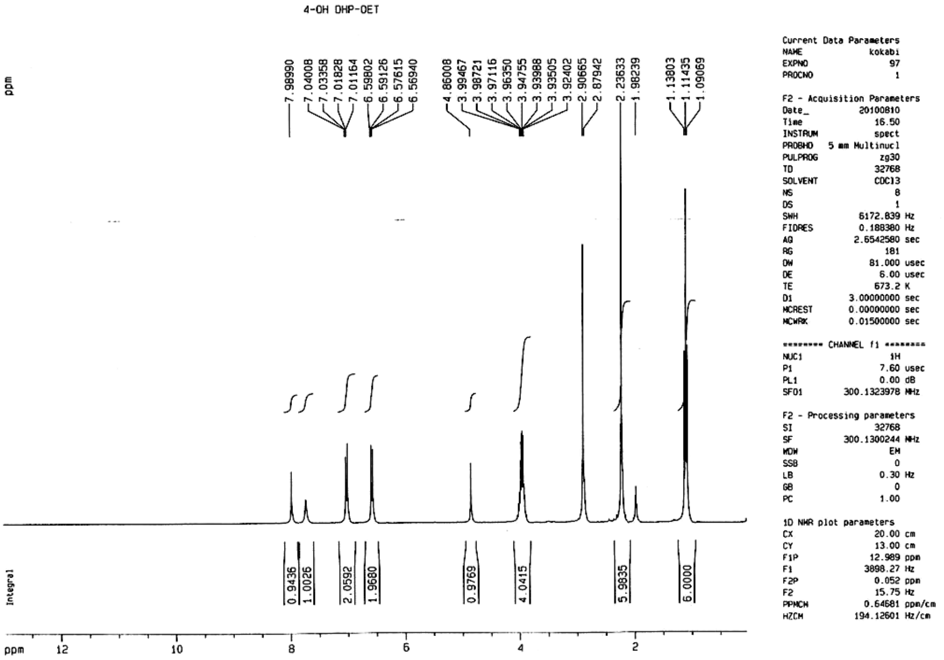
^{13}C NMR of 4c

Diethyl 4-(4-hydroxyphenyl)-2,6-dimethyl-1,4-dihydropyridine-3,5-dicarboxylate[8]



Mp: 230-231 °C; ^{1}H NMR ($CDCl_3$, 300 MHz): δ (ppm): 1.13-1.9(t, 6 H), 2.23 (s, 6 H), 3.99-3.93 (q, 4 H), 4.86 (s, 1 H), 7.04-6.56 (m, 4 H), 7.90(s, 1H), 7.98(s,1H); ^{13}C NMR (Acetone D, 75 MHz): δ (ppm): 13.77, 17.87, 38.54, 58.87, 103.39, 114.40, 128.83, 139.76, 144.38, 155.54, 167.23; Elemental Analysis: Calcd. C: 66.07, H: 6.71, N: 4.06, O: 23.16, Found. C: 65.77, H: 6.63, N: 3.95, O: 23.10.



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RG                     181
DW                  81.000 usec
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TE                   673.2 K
D1             3.00000000 sec
MCREST         0.00000000 sec
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======== CHANNEL f1 ========
NUC1                    1H
P1                    7.60 usec
PL1                   0.00 dB
SFO1           300.1323978 MHz

F2 - Processing parameters
SI                   32768
SF             300.1300244 MHz
WDW                     EM
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LB                    0.30 Hz
GB                       0
PC                    1.00

1D NMR plot parameters
CX                   20.00 cm
CY                   13.00 cm
F1P                 12.989 ppm
F1                 3898.27 Hz
F2P                  0.052 ppm
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HZCM             194.12601 Hz/cm
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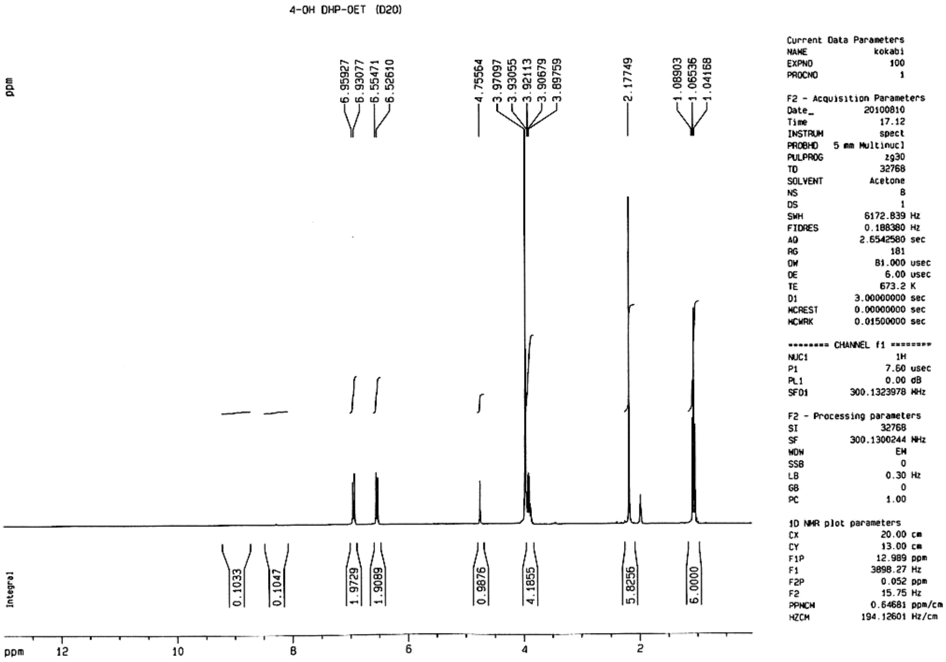
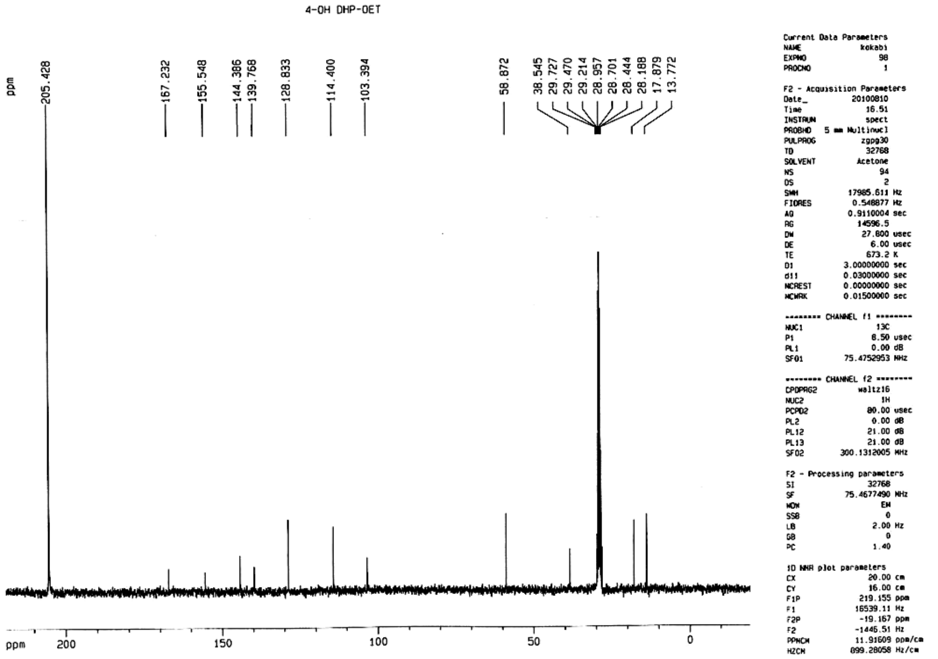
^{1}H NMR of 4d

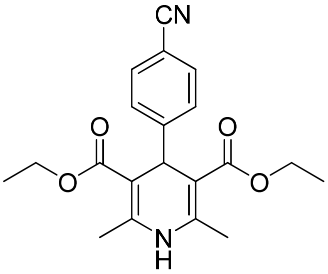


^{1}H NMR of 4d

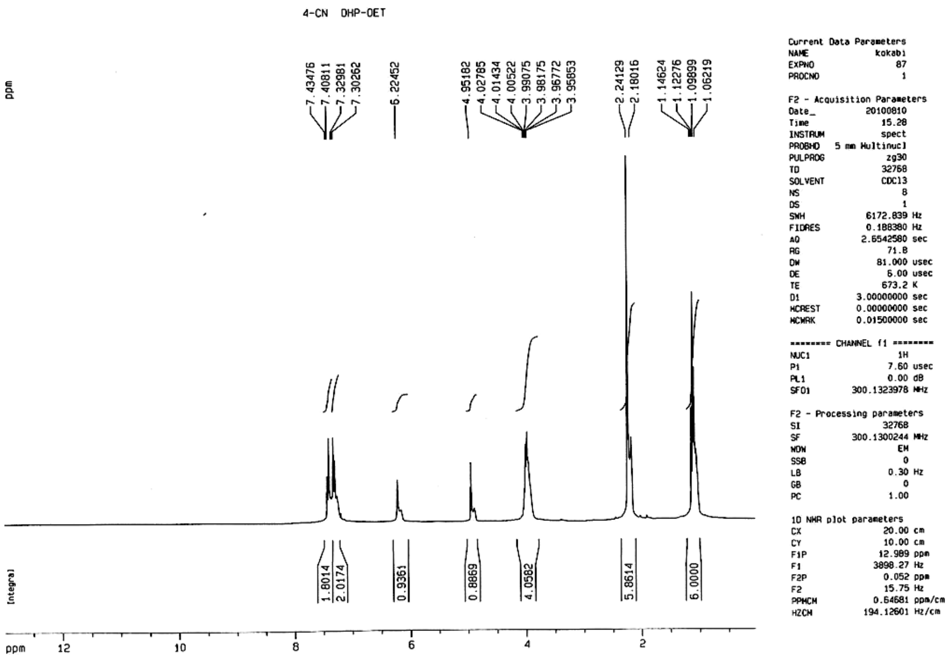


^{13}C NMR of 4d

Diethyl 4-(4-cyanophenyl)-2,6-dimethyl-1,4-dihydropyridine-3,5-dicarboxylate



Mp: 165-167 °C; ^{1}H NMR ($CDCl_3$, 300 MHz): δ (ppm): 1.10 (t, 6 H), 2.20 (s, 6 H), 4.0 (q, 4 H), 4.95 (s, 1 H), 6.22 (b, 1 H), 7.30-7.43 (m, 4 H); ^{13}C NMR ($CDCl_3$, 75 MHz): δ (ppm): 14.25, 19.36, 40.4, 59.93, 102.93, 109.52, 119.35, 128.87, 131.87, 145.03, 163.28, 167.27; Elemental Analysis: Calcd. C: 67.78, H: 6.26, N: 7.90, O: 18.06, Found. C: 67.71, H: 6.22, N: 7.89, O: 18.01.



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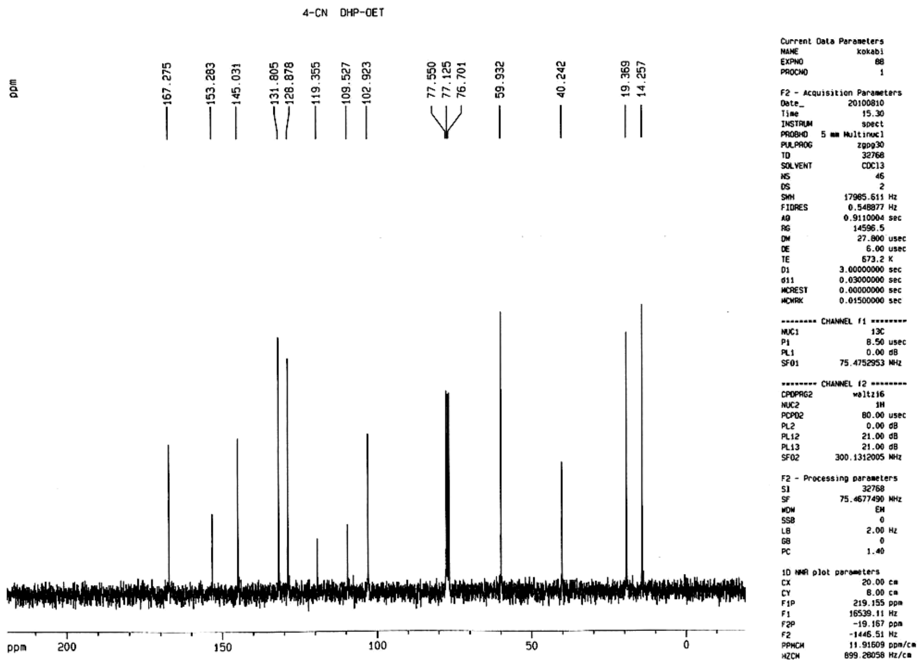
PROBHD   5 mm Multinucl
PULPROG           zg30
TD               32768
SOLVENT          CDCl3
NS                   8
DS                   1
SWH           6172.839 Hz
FIDRES        0.188380 Hz
AQ           2.6542580 sec
RG                71.8
DW              81.000 usec
DE                6.00 usec
TE               673.2 K
D1          3.00000000 sec
HCREST      0.00000000 sec
HCWRK       0.01500000 sec

======== CHANNEL f1 ========
NUC1                1H
P1                7.60 usec
PL1               0.00 dB
SFO1       300.1323978 MHz

F2 - Processing parameters
SI               32768
SF         300.1300244 MHz
WDW                 EM
SSB                  0
LB                0.30 Hz
GB                   0
PC                1.00

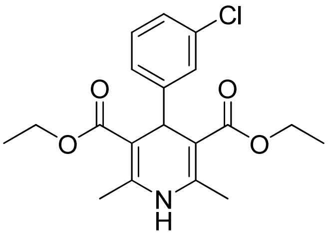
1D NMR plot parameters
CX               20.00 cm
CY               10.00 cm
F1P             12.989 ppm
F1             3898.27 Hz
F2P              0.052 ppm
F2               15.75 Hz
PPMCM          0.64681 ppm/cm
HZCM         194.12601 Hz/cm
```

^{1}H NMR of 4h

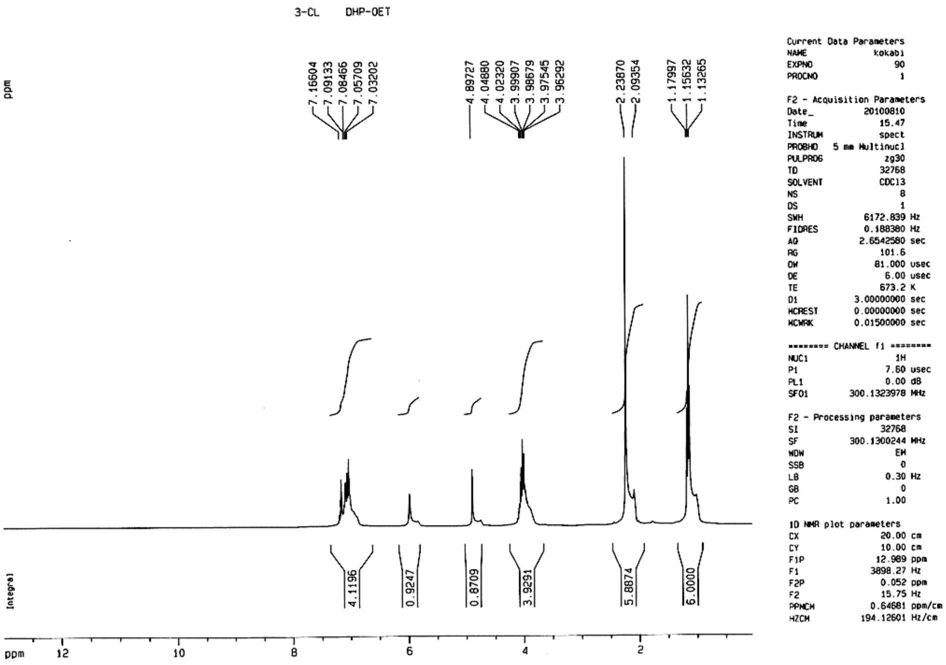


^{13}C NMR of 4h

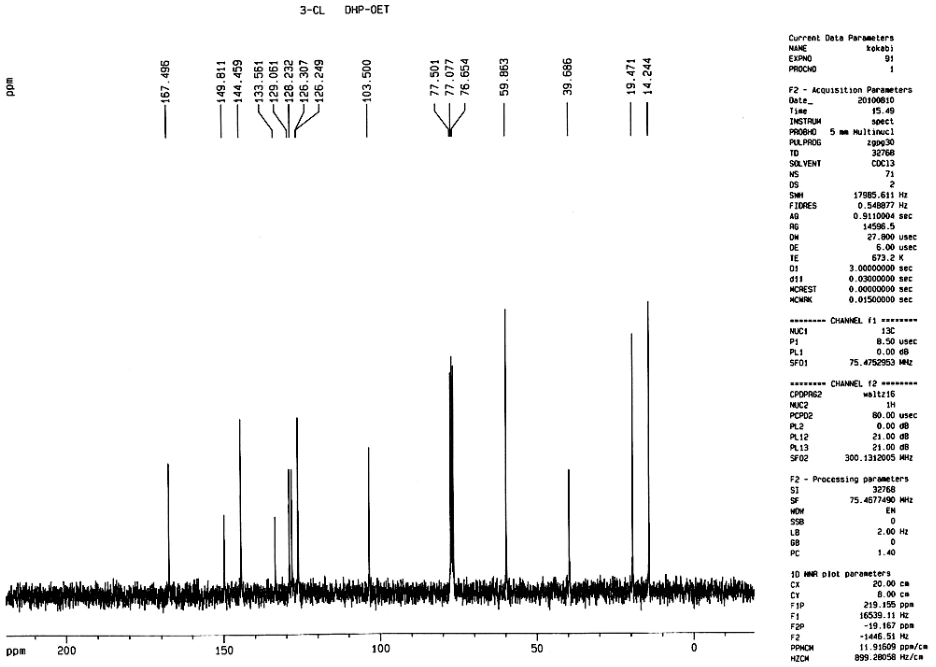
Diethyl 4-(3-chlorophenyl)-2,6-dimethyl-1,4-dihydropyridine-3,5-dicarboxylate[7]



Mp: 141-142 °C; ^{1}H NMR (CDCl$_3$, 300 MHz): δ (ppm): 1.15 (t, 6 H), 2.23-2.09(s, 6 H), 4.04-3.96 (q, 4 H), 4.89 (s, 1 H), 6.2 (s, 1 H), 7.16-7.03 (m, 4 H); ^{13}C NMR (CDCl$_3$, 75 MHz): δ (ppm): 14.21, 19.47, 39.68, 59.86, 103.50, 126.24, 126.30, 128.23, 129.06, 133.56, 144.45, 149.81, 167.49; Elemental Analysis: Calcd. C: 62.72, H: 6.09, N: 3.85, O: 17.59, Found. C: 62.68, H: 6.01, N: 3.78, O: 17.52.

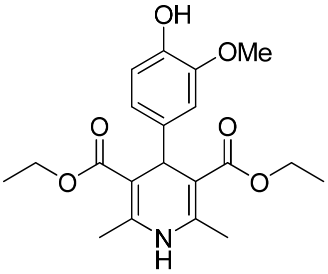


^{1}H NMR of 4k

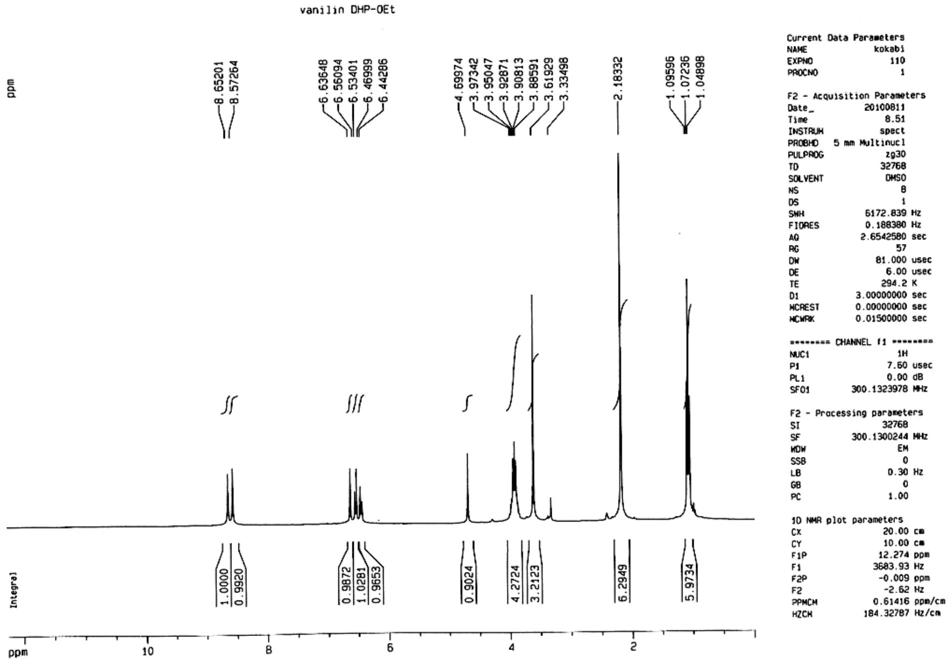


^{13}C NMR of 4k

Diethyl 4-(4-hydroxy-3-methoxyphenyl)-2,6-dimethyl-1,4-dihydropyridine-3,5-dicarboxylate



Mp: 159-161°C; ^{1}H NMR (DMSO, 300 MHz): δ (ppm): 1.07 (t, 6 H), 2.18 (s, 6 H), 3.61 (s, 3 H), 3.88-3.95 (q, 1 H), 4.69 (s, 1 H), 6.44-6.56 (dd, 2 H), 6.63(s ,1 H), 8.57(s ,1H), 8.65(S,1H); ^{13}C NMR (DMSO, 75 MHz): δ (ppm): 14.68, 18.65, 38.64, 55.84, 59.13, 102.64, 112.24, 115.24, 120.09, 140.03, 145.16, 145.32, 147.15, 167.58; Elemental Analysis: Calcd. C: 63.99, H: 6.71, N: 3.73, O: 25.57, Found. C: 63.90, H: 6.70, N: 3.71, O: 25.50.



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Current Data Parameters
NAME               kokabi
EXPNO                 110
PROCNO                  1

F2 - Acquisition Parameters
Date_            20100811
Time                 8.51
INSTRUM             spect
PROBHD   5 mm Multinucl
PULPROG              zg30
TD                  32768
SOLVENT              DMSO
NS                      8
DS                      1
SWH              6172.839 Hz
FIDRES           0.188380 Hz
AQ              2.6542580 sec
RG                     57
DW                 81.000 usec
DE                   6.00 usec
TE                  294.2 K
D1             3.00000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                   7.60 usec
PL1                  0.00 dB
SFO1          300.1323978 MHz

F2 - Processing parameters
SI                  32768
SF            300.1300244 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00

1D NMR plot parameters
CX                  20.00 cm
CY                  10.00 cm
F1P                12.274 ppm
F1                3683.93 Hz
F2P                -0.009 ppm
F2                  -2.62 Hz
PPMCM             0.61416 ppm/cm
HZCM            184.32787 Hz/cm
```
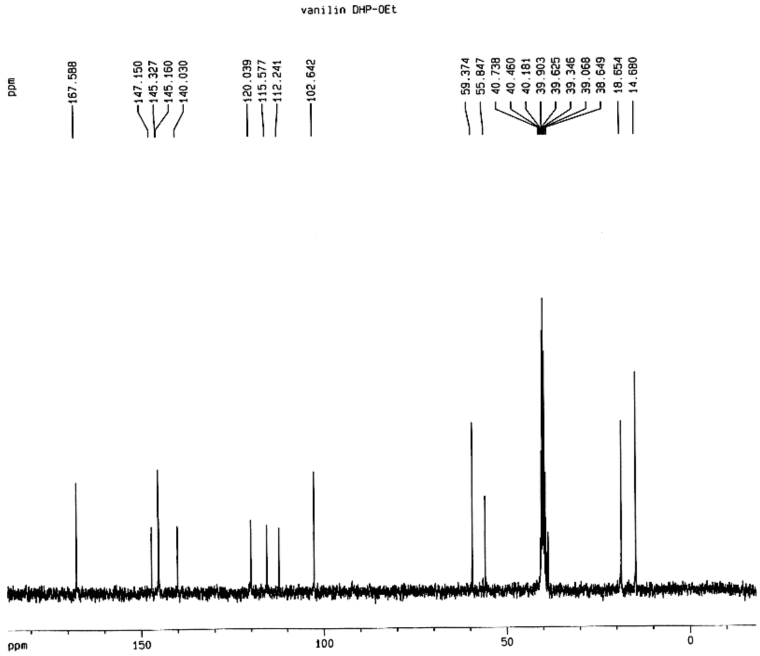

^{1}H NMR of 4l



```
Current Data Parameters
NAME             kokabi
EXPNO               111
PROCNO                1

F2 - Acquisition Parameters
Date_          20100811
Time               8.55
INSTRUM           spect
PROBHD   5 mm Multinucl
PULPROG         zgpg30
TD                32768
SOLVENT            DMSO
NS                   31
DS                    2
SWH           17985.611 Hz
FIDRES         0.548877 Hz
AQ            0.9110004 sec
RG              14596.5
DW               27.800 usec
DE                 6.00 usec
TE                294.2 K
D1           3.00000000 sec
d11          0.03000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                13C
P1                 8.50 usec
PL1                0.00 dB
SFO1         75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2                0.00 dB
PL12              21.00 dB
PL13              21.00 dB
SFO2        300.1312005 MHz

F2 - Processing parameters
SI                32768
SF           75.4677490 MHz
WDW                  EM
SSB                   0
LB                 2.00 Hz
GB                    0
PC                 1.40

1D NMR plot parameters
CX                20.00 cm
CY                 8.00 cm
F1P             186.291 ppm
F1             14058.95 Hz
F2P             -17.985 ppm
F2             -1357.29 Hz
PPMCM          10.21379 ppm/cm
HZCM          770.81195 Hz/cm
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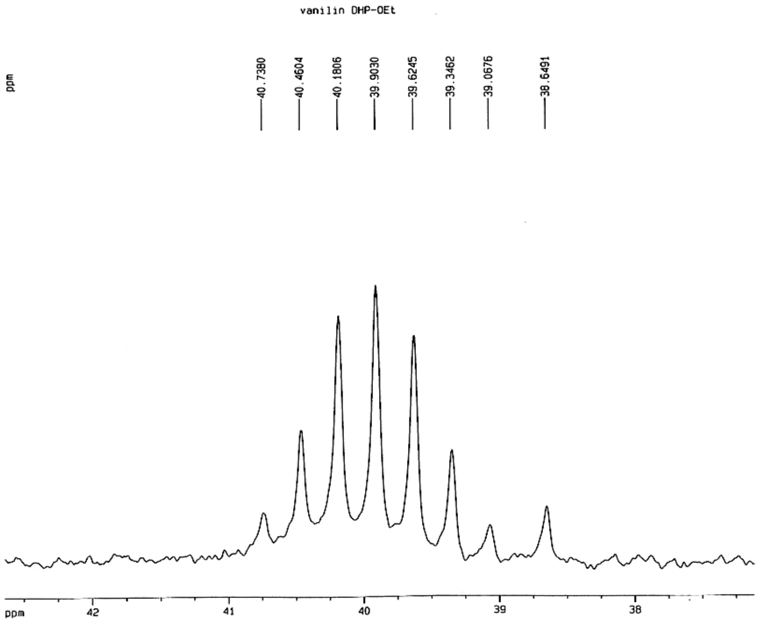
^{13}C NMR of 4l



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Current Data Parameters
NAME             kokabi
EXPNO               111
PROCNO                1

F2 - Acquisition Parameters
Date_          20100811
Time               8.55
INSTRUM           spect
PROBHD   5 mm Multinucl
PULPROG         zgpg30
TD                32768
SOLVENT            DMSO
NS                   31
DS                    2
SWH           17985.611 Hz
FIDRES         0.548877 Hz
AQ            0.9110004 sec
RG              14596.5
DW               27.800 usec
DE                 6.00 usec
TE                294.2 K
D1           3.00000000 sec
d11          0.03000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                13C
P1                 8.50 usec
PL1                0.00 dB
SFO1         75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2                0.00 dB
PL12              21.00 dB
PL13              21.00 dB
SFO2        300.1312005 MHz

F2 - Processing parameters
SI                32768
SF           75.4677490 MHz
WDW                  EM
SSB                   0
LB                 2.00 Hz
GB                    0
PC                 1.40

1D NMR plot parameters
CX                20.00 cm
CY                 8.00 cm
F1P              42.642 ppm
F1              3218.13 Hz
F2P              37.118 ppm
F2              2801.23 Hz
PPMCM           0.27621 ppm/cm
HZCM           20.84501 Hz/cm
```
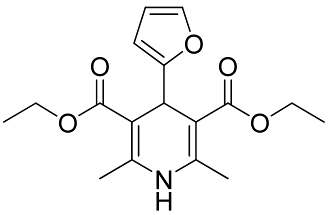
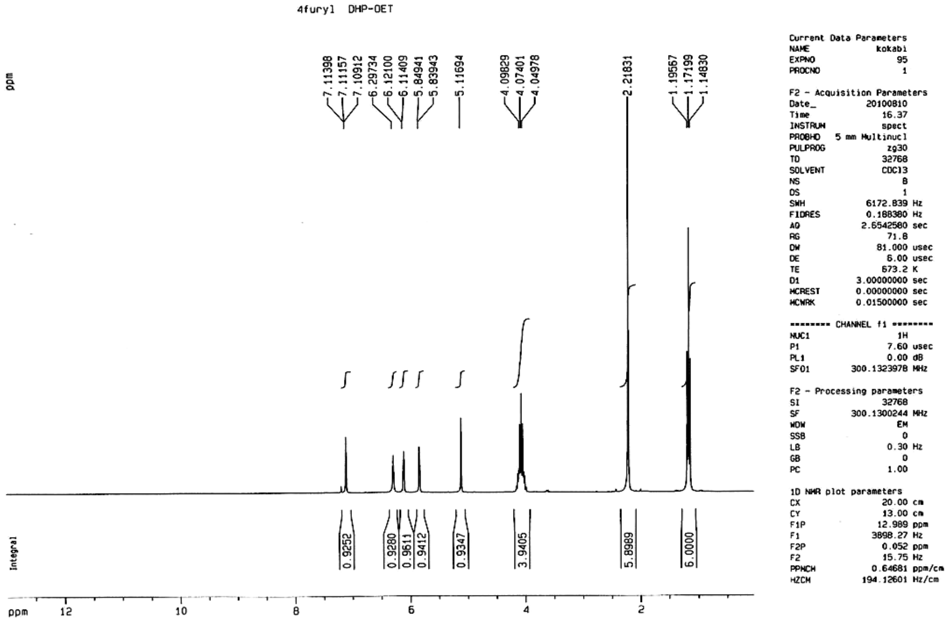

Expanded ^{13}C NMR of 4l

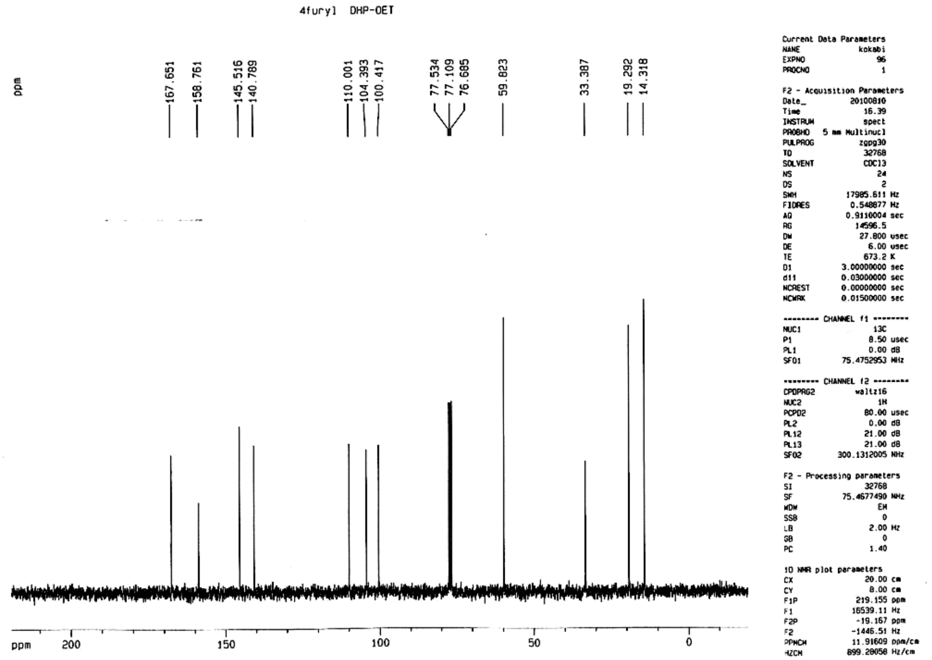
Diethyl 4-(furan-2-yl)-2,6-dimethyl-1,4-dihydropyridine-3,5-dicarboxylate[7]



Mp: 161-162 °C; ^{1}H NMR (CDCl$_3$, 300 MHz): δ (ppm): 1.17 (t, 6 H), 2.21 (s, 6 H), 4.07 (q, 4 H), 5.11 (s, 1 H), 5.83-6.12 (m , 2H), 6.29 (s,1H), 7.10-7.11 (t, 1 H); ^{13}C NMR (CDCl$_3$, 75 MHz): δ (ppm): 143.31, 19.29, 33.38, 59.82, 100.41, 104.39, 110.00, 140.78, 145.51, 158.76, 167.65; Elemental Analysis: Calcd. C: 63.94, H: 6.63, N: 4.39, O: 25.05, Found. C: 63.91, H: 6.59, N: 4.32, O: 25.01.



^{1}H NMR of 4n



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Current Data Parameters
NANE                kokabi
EXPNO                   96
PROCNO                   1

F2 - Acquisition Parameters
Date_             20100810
Time                 16.39
INSTRUM              spect
PROBHD   5 mm Multinucl
PULPROG            zgpg30
TD                   32768
SOLVENT              CDCl3
NS                      24
DS                       2
SWH              17985.611 Hz
FIDRES            0.548877 Hz
AQ               0.9110004 sec
RG                 14596.5
DW                  27.800 usec
DE                    6.00 usec
TE                   673.2 K
D1              3.00000000 sec
d11             0.03000000 sec
NCREST          0.00000000 sec
NCWRK           0.01500000 sec

======== CHANNEL f1 ========
NUC1                   13C
P1                    8.50 usec
PL1                   0.00 dB
SFO1            75.4752953 MHz

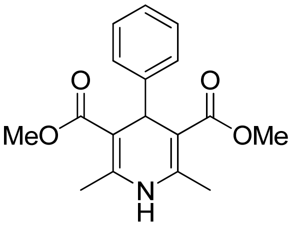
======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2                80.00 usec
PL2                   0.00 dB
PL12                 21.00 dB
PL13                 21.00 dB
SFO2           300.1312005 MHz

F2 - Processing parameters
SI                   32768
SF              75.4677490 MHz
WDW                     EM
SSB                      0
LB                    2.00 Hz
GB                       0
PC                    1.40

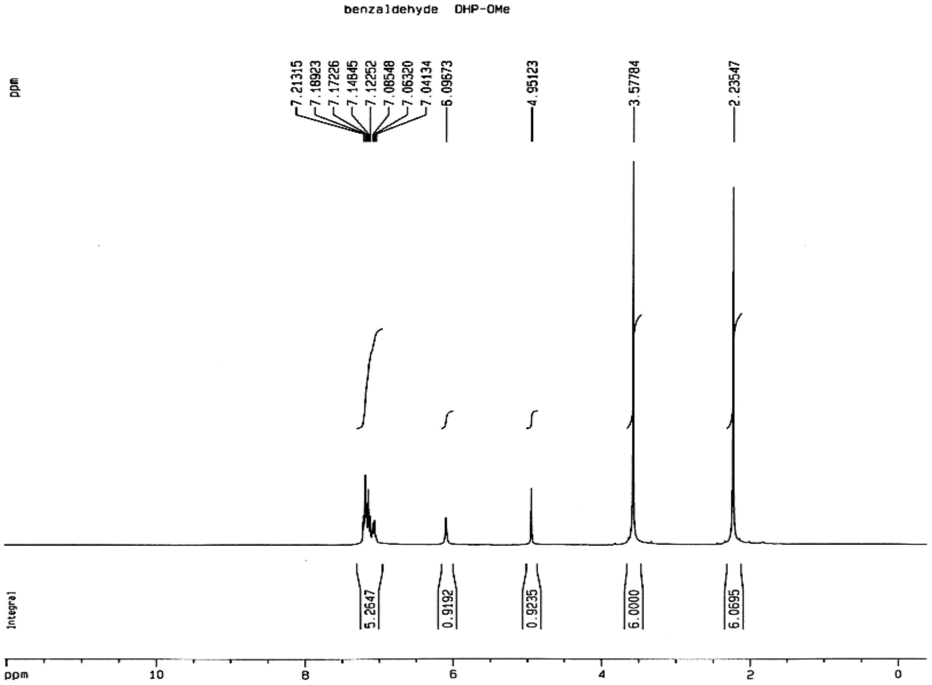
1D NMR plot parameters
CX                   20.00 cm
CY                    8.00 cm
F1P                219.155 ppm
F1                16539.11 Hz
F2P                -19.167 ppm
F2                -1446.51 Hz
PPMCM             11.91609 ppm/cm
HZCM             899.28058 Hz/cm
```

^{13}C NMR of 4n

Dimethyl 2,6-dimethyl-4-phenyl-1,4-dihydropyridine-3,5-dicarboxylate[9]



Mp: 196–197 °C; ^{1}H NMR ($CDCl_3$, 300 MHz): δ (ppm): 2.23 (s, 6 H), 3.75 (s, 6 H), 4.95 (s, 1 H), 6.09 (b, 1 H), 7.14-7.21 (m, 4 H); ^{13}C NMR ($CDCl_3$, 75 MHz): δ (ppm): 19.41, 38.28, 51.03, 103.64, 126.23, 127.60, 128.05, 144.61, 147.48, 168.22; Elemental Analysis: Calcd. C: 67.76, H: 6.36, N: 4.65, O: 21.24, Found. C: 67.43, H: 6.31, N: 4.56, O: 21.20.



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Current Data Parameters
NAME                kokabi
EXPNO                   77
PROCNO                   1

F2 - Acquisition Parameters
Date_             20100810
Time                 13.46
INSTRUM              spect
PROBHD   5 mm Multinucl
PULPROG               zg30
TD                   32768
SOLVENT              CDCl3
NS                       8
DS                       1
SWH             6172.839 Hz
FIDRES          0.188380 Hz
AQ             2.6542580 sec
RG                    90.5
DW                81.000 usec
DE                  6.00 usec
TE                 673.2 K
D1            3.00000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                    1H
P1                  7.60 usec
PL1                 0.00 dB
SFO1        300.1323978 MHz

F2 - Processing parameters
SI                   32768
SF          300.1300244 MHz
WDW                     EM
SSB                      0
LB                  0.30 Hz
GB                       0
PC                    1.00

1D NMR plot parameters
CX                  20.00 cm
CY                  10.00 cm
F1P                12.030 ppm
F1                3610.45 Hz
F2P                -0.376 ppm
F2                -112.85 Hz
PPMCM             0.62028 ppm/cm
HZCM            186.16499 Hz/cm
```
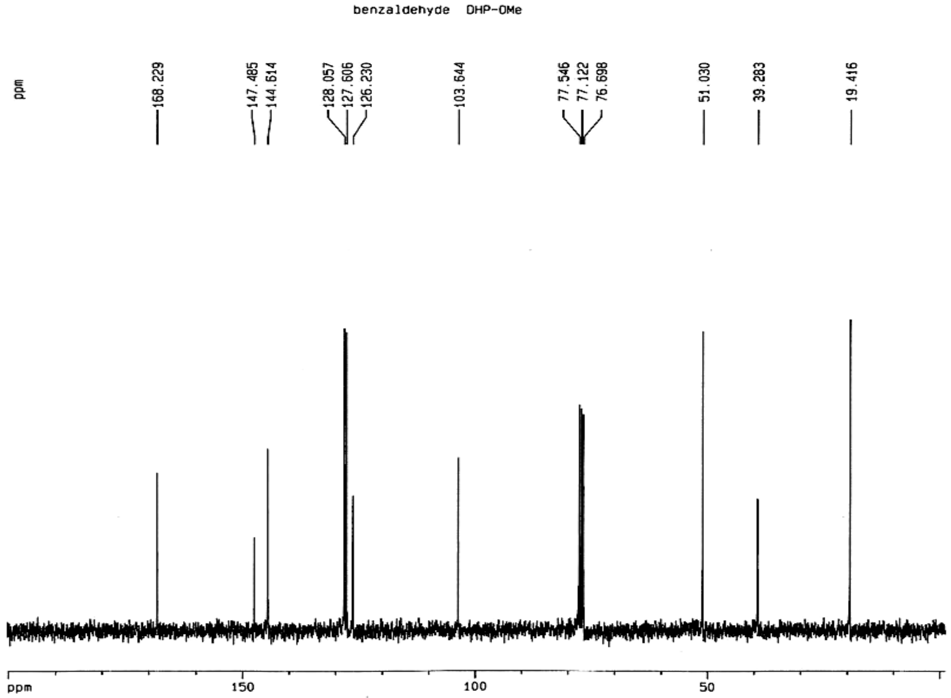

^{1}H NMR of 5a



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Current Data Parameters
NAME                kokabi
EXPNO                   78
PROCNO                   1

F2 - Acquisition Parameters
Date_             20100810
Time                 13.47
INSTRUM              spect
PROBHD   5 mm Multinucl
PULPROG             zgpg30
TD                   32768
SOLVENT              CDCl3
NS                      32
DS                       2
SWH            17985.611 Hz
FIDRES          0.548877 Hz
AQ             0.9110004 sec
RG                  9195.2
DW                27.800 usec
DE                  6.00 usec
TE                 673.2 K
D1            3.00000000 sec
d11           0.03000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                   13C
P1                  8.50 usec
PL1                 0.00 dB
SFO1         75.4752953 MHz

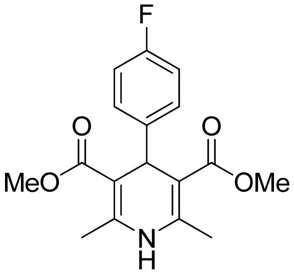
======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2              80.00 usec
PL2                 0.00 dB
PL12               21.00 dB
PL13               21.00 dB
SFO2        300.1312005 MHz

F2 - Processing parameters
SI                   32768
SF           75.4677490 MHz
WDW                     EM
SSB                      0
LB                  2.00 Hz
GB                       0
PC                    1.40

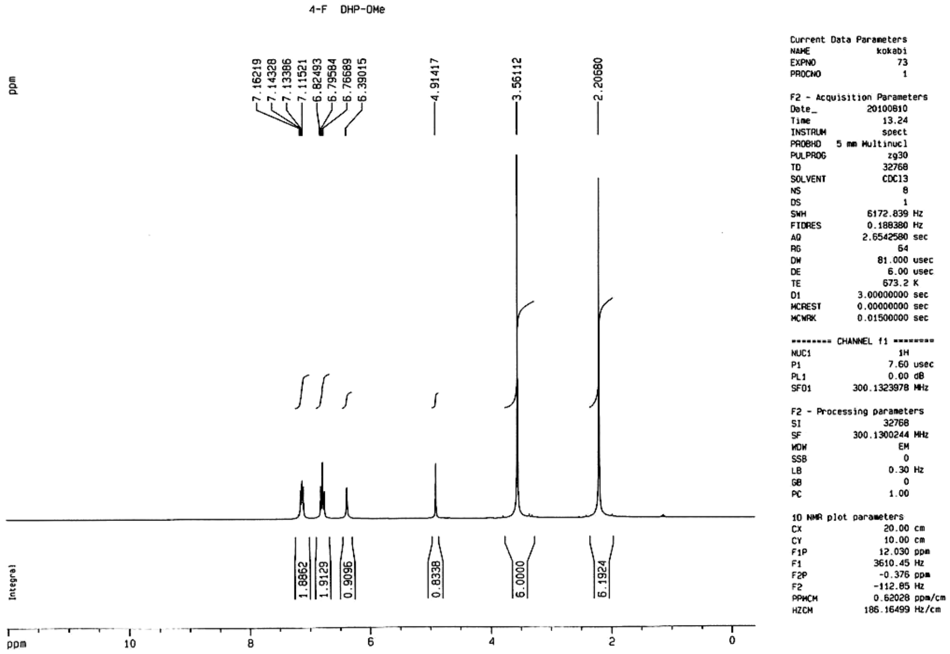
1D NMR plot parameters
CX                  20.00 cm
CY                   8.00 cm
F1P               200.240 ppm
F1               15111.68 Hz
F2P                -1.198 ppm
F2                 -90.45 Hz
PPMCM            10.07193 ppm/cm
HZCM            760.10620 Hz/cm
```

^{13}C NMR of 5a

Dimethyl 4-(4-fluorophenyl)-2,6-dimethyl-1,4-dihydropyridine-3,5-dicarboxylate[10]



Mp: 171-172 °C; ^{1}H NMR ($CDCl_3$, 300 MHz): δ (ppm): 2.20 (s, 6 H), 3.56 (s, 6 H), 4.91 (s, 1 H), 6.39 (s, 1 H), 7.16-6.76 (m, 4 H); ^{13}C NMR ($CDCl_3$, 75 MHz): δ (ppm): 19.28, 38.71, 51.03, 103.49, 114.55, 114.83, 129.01, 129.12, 143.49, 144.78, 159/75, 162.98, 168.21; Elemental Analysis: Calcd. C: 63.94, H: 5.68, N: 4.39, O: 20.04, Found. C: 63.73, H: 5.65, N: 4.33, O: 20.02.



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Current Data Parameters
NAME              kokabi
EXPNO                 73
PROCNO                 1

F2 - Acquisition Parameters
Date_           20100810
Time               13.24
INSTRUM            spect
PROBHD   5 mm Multinucl
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                     8
DS                     1
SWH             6172.839 Hz
FIDRES          0.188380 Hz
AQ             2.6542580 sec
RG                    64
DW                81.000 usec
DE                  6.00 usec
TE                 673.2 K
D1            3.00000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                  7.60 usec
PL1                 0.00 dB
SFO1         300.1323978 MHz

F2 - Processing parameters
SI                 32768
SF           300.1300244 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX                 20.00 cm
CY                 10.00 cm
F1P               12.030 ppm
F1               3610.45 Hz
F2P               -0.376 ppm
F2               -112.85 Hz
PPMCM            0.62028 ppm/cm
HZCM           186.16499 Hz/cm
```
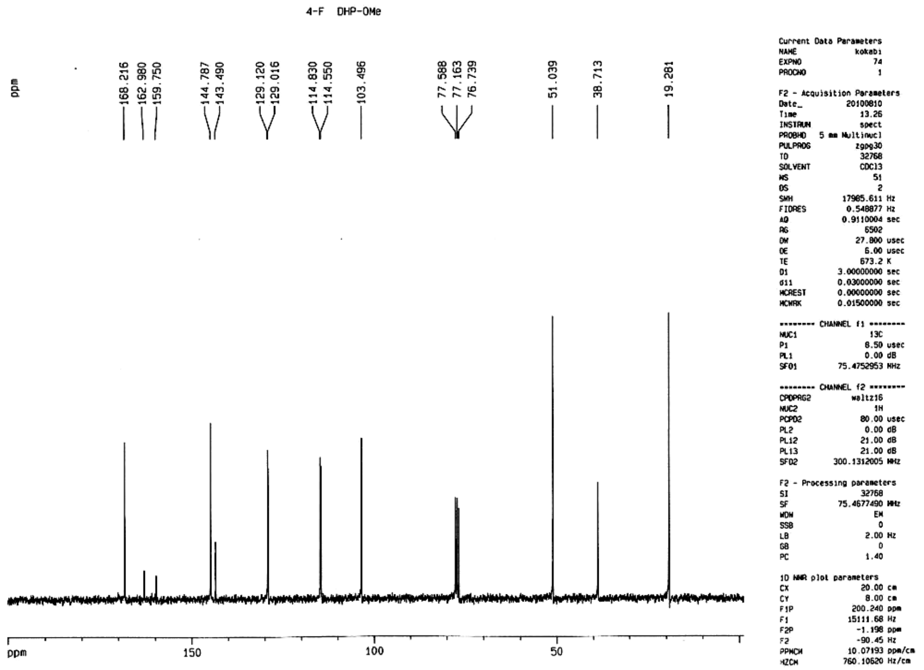

^{1}**H NMR of 5h**



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Current Data Parameters
NAME              kokabi
EXPNO                 74
PROCNO                 1

F2 - Acquisition Parameters
Date_           20100810
Time               13.26
INSTRUM            spect
PROBHD   5 mm Multinucl
PULPROG          zgpg30
TD                 32768
SOLVENT            CDCl3
NS                    51
DS                     2
SWH           17985.611 Hz
FIDRES         0.548877 Hz
AQ            0.9110004 sec
RG                  6502
DW               27.800 usec
DE                 6.00 usec
TE                673.2 K
D1           3.00000000 sec
d11          0.03000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                  8.50 usec
PL1                 0.00 dB
SFO1          75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                 0.00 dB
PL12               21.00 dB
PL13               21.00 dB
SFO2         300.1312005 MHz

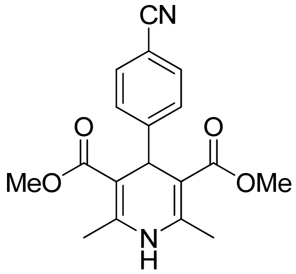
F2 - Processing parameters
SI                 32768
SF            75.4677490 MHz
WDW                   EM
SSB                    0
LB                  2.00 Hz
GB                     0
PC                  1.40

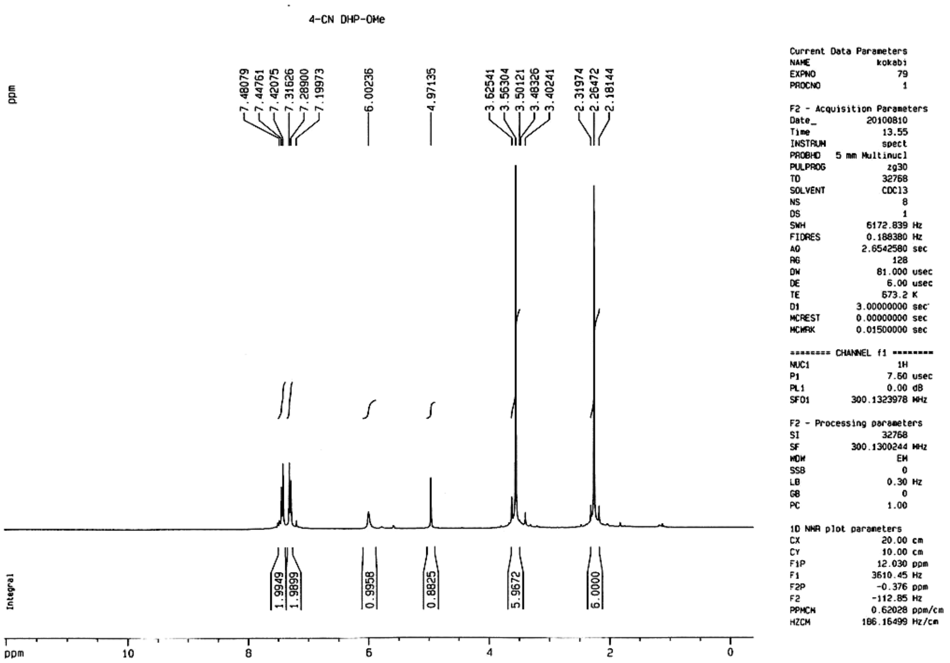
1D NMR plot parameters
CX                 20.00 cm
CY                  8.00 cm
F1P              200.240 ppm
F1              15111.68 Hz
F2P               -1.198 ppm
F2                -90.45 Hz
PPMCM           10.07193 ppm/cm
HZCM           760.10620 Hz/cm
```

^{13}C NMR of 5h

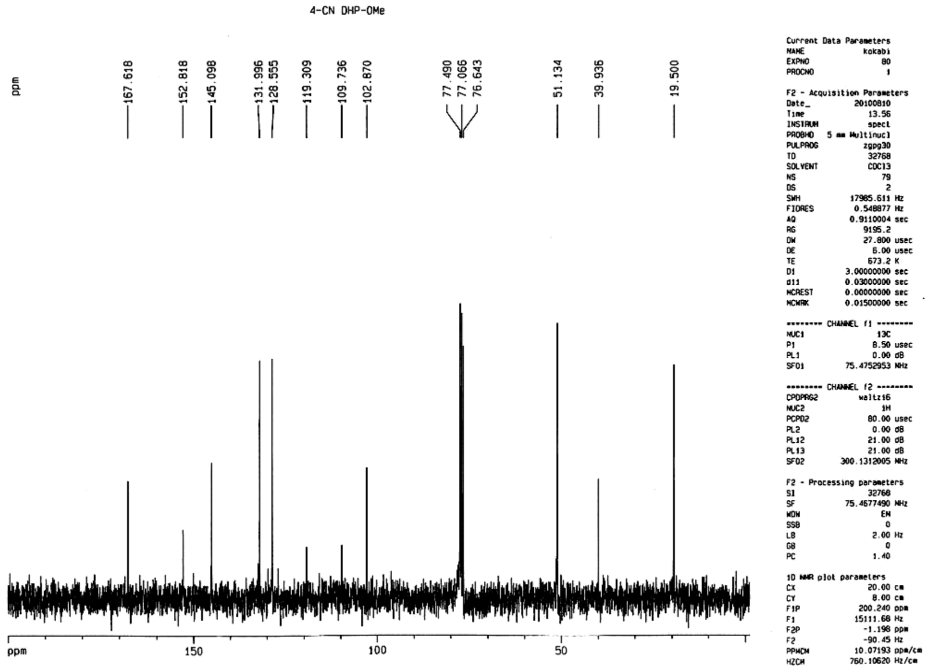
Dimethyl 4-(4-cyanophenyl)-2,6-dimethyl-1,4-dihydropyridine-3,5-dicarboxylate

Mp: 225-227 °C; ^{1}H NMR ($CDCl_3$, 300 MHz): δ (ppm): 2.26 (s, 6 H), 3.50 (s, 6 H), 4.97 (s, 1 H), 6.00 (s, 1 H), 7.48-7.19 (m, 4 H); ^{13}C NMR ($CDCl_3$, 75 MHz): δ (ppm): 19.50, 39.93, 51.13, 102.87, 109.73, 119.30, 128.55, 131.99, 145.09, 152.81, 167.61; Elemental Analysis: Calcd. C: 66.25, H: 5.56, N: 8.58, O: 19.61, Found. C: 66.18, H: 5.55, N: 8.54, O: 19.58.





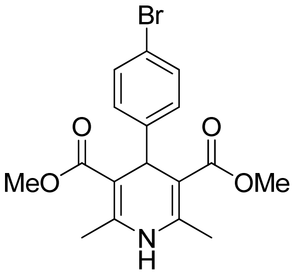
^{1}H NMR of 5i

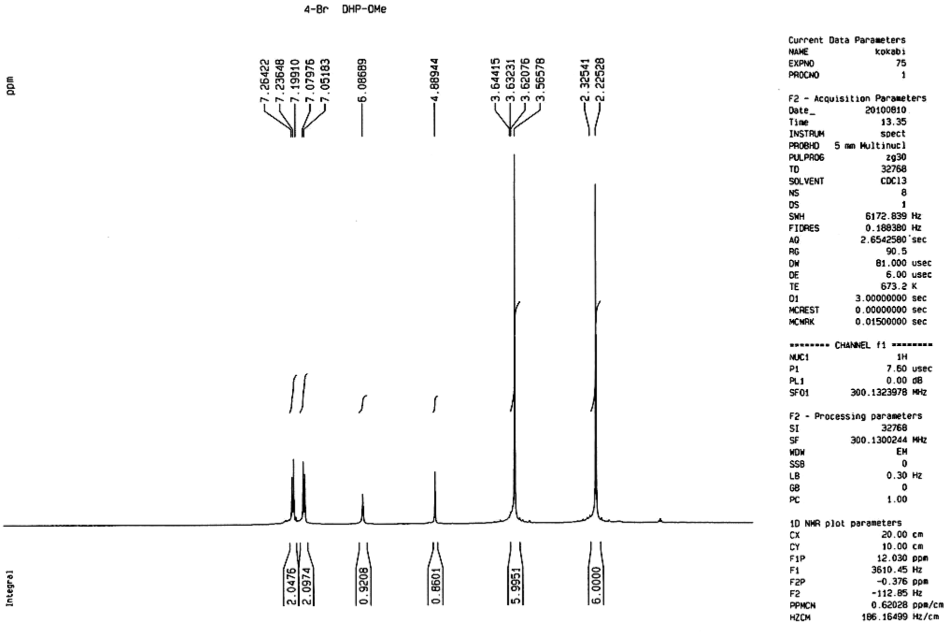


^{13}C NMR of 5i

Dimethyl 4-(4-bromophenyl)-2,6-dimethyl-1,4-dihydropyridine-3,5-dicarboxylate[11]

Mp: 200-201 °C; ^{1}H NMR ($CDCl_3$, 300 MHz): δ (ppm): 2.32 (s, 6 H), 3.63 (s, 6 H), 4.88 (s, 1 H), 6.08 (s, 1 H), 7.05-7.26 (m, 4 H); ^{13}C NMR ($CDCl_3$, 75 MHz): δ (ppm): 19.45, 20.88, 39.01, 51.08, 103.33, 119.98, 129.48, 131.08, 144.69, 146.56, 167.97; Elemental Analysis: Calcd. C: 53.70, H: 4.77, N: 3.68, O: 16.83, Found. C: 53.40, H: 4.71, N: 3.62, O: 16.82.





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Current Data Parameters
NAME             kokabi
EXPNO                75
PROCNO                1

F2 - Acquisition Parameters
Date_          20100810
Time              13.35
INSTRUM           spect
PROBHD   5 mm Multinucl
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                    8
DS                    1
SWH            6172.839 Hz
FIDRES         0.188380 Hz
AQ            2.6542580 sec
RG                 90.5
DW               81.000 usec
DE                 6.00 usec
TE                673.2 K
D1           3.00000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                 7.60 usec
PL1                0.00 dB
SFO1        300.1323978 MHz

F2 - Processing parameters
SI                32768
SF          300.1300244 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX                20.00 cm
CY                10.00 cm
F1P              12.030 ppm
F1              3610.45 Hz
F2P              -0.376 ppm
F2              -112.85 Hz
PPMCM           0.62028 ppm/cm
HZCM          186.16499 Hz/cm
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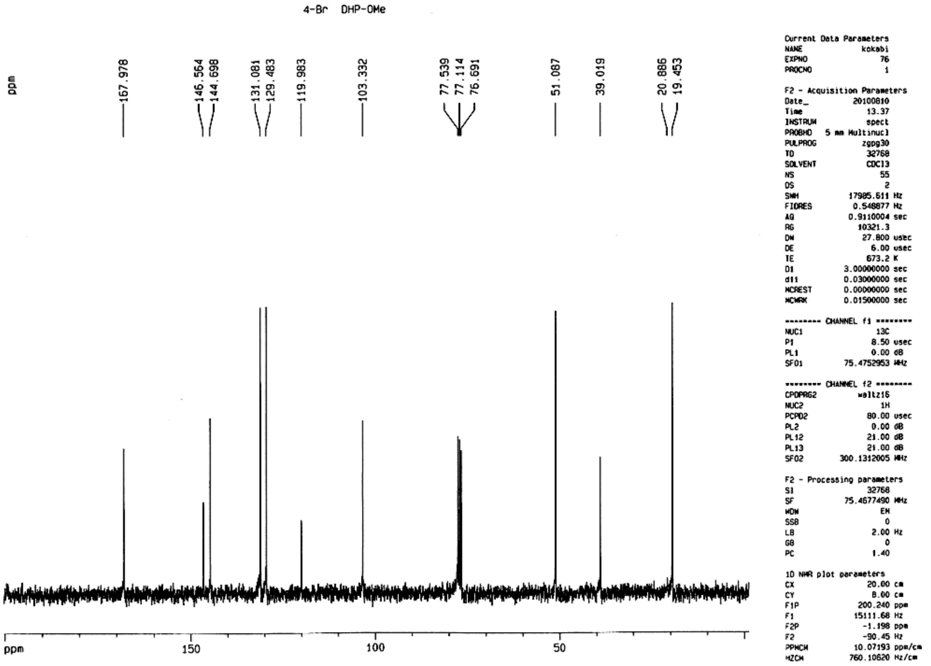
^{1}H NMR of 5j



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Current Data Parameters
NAME              kokabi
EXPNO                 76
PROCNO                 1

F2 - Acquisition Parameters
Date_           20100810
Time               13.37
INSTRUM            spect
PROBHD   5 mm Multinucl
PULPROG          zgpg30
TD                 32768
SOLVENT            CDCl3
NS                    55
DS                     2
SWH          17985.611 Hz
FIDRES        0.548877 Hz
AQ           0.9110004 sec
RG             10321.3
DW              27.800 usec
DE                6.00 usec
TE               673.2 K
D1          3.00000000 sec
d11         0.03000000 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                  8.50 usec
PL1                 0.00 dB
SFO1         75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                 0.00 dB
PL12               21.00 dB
PL13               21.00 dB
SFO2        300.1312005 MHz

F2 - Processing parameters
SI                 32768
SF           75.4677490 MHz
WDW                   EM
SSB                    0
LB                  2.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 20.00 cm
CY                  8.00 cm
F1P              200.240 ppm
F1              15111.68 Hz
F2P               -1.198 ppm
F2                -90.45 Hz
PPMCM           10.07193 ppm/cm
HZCM           760.10620 Hz/cm
```
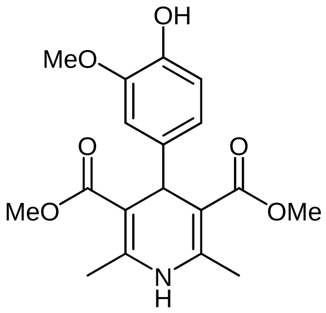
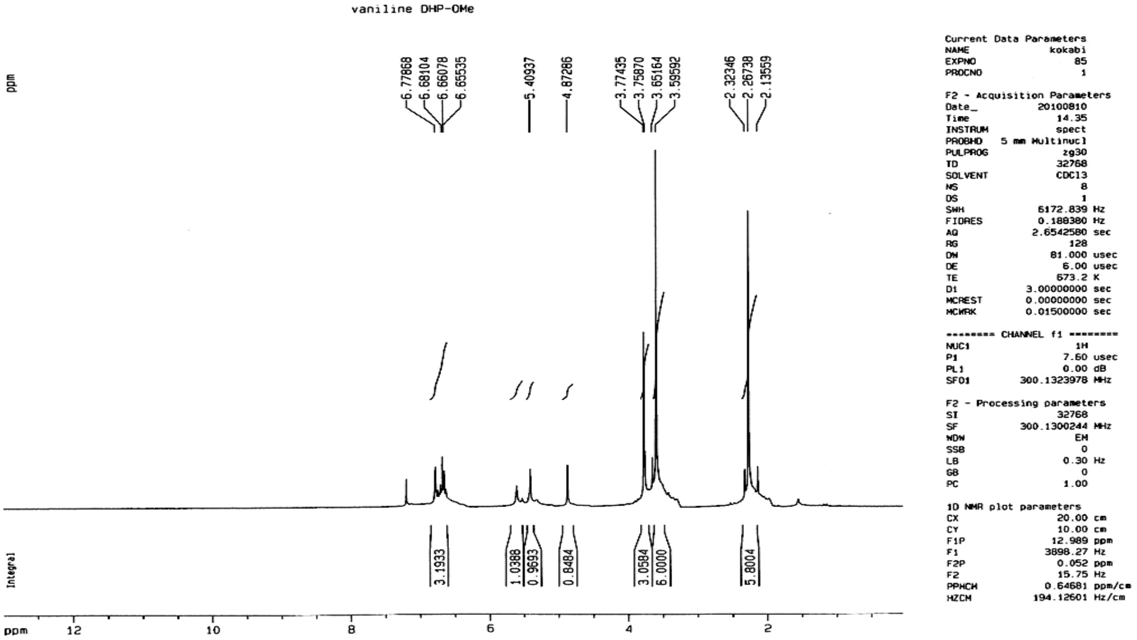

^{13}C NMR of 5j

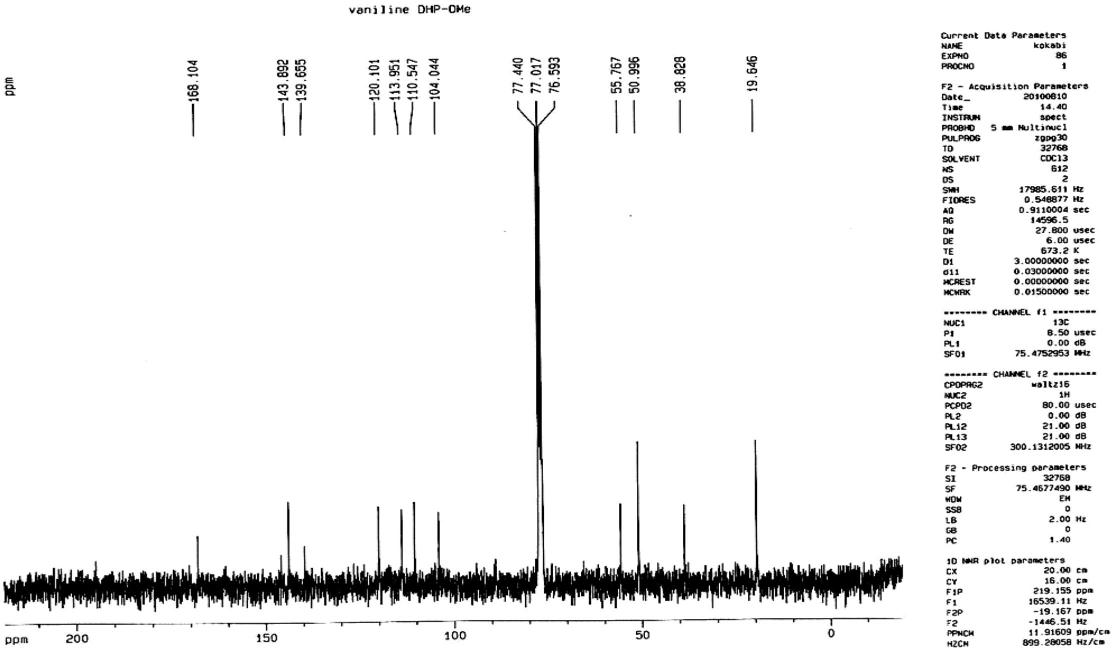
Dimethyl 4-(4-hydroxy-3-methoxyphenyl)-2,6-dimethyl-1,4-dihydropyridine-3,5-dicarboxylate



Mp: 227-228 °C; ^{1}H NMR (CDCl$_3$, 300 MHz): δ (ppm): 2.26 (s, 6 H), 3.65 (s, 6 H), 3.77 (s, 3 H), 4.87 (s, 1 H), 5.30 (s, 1H), 5.40(s, 1H), 6.77-6.65 (m, 3 H); ^{13}C NMR (CDCl$_3$, 75 MHz): δ (ppm): 19.64, 38.82, 50.99, 55.76, 104.04, 110.54, 113.95, 120.10, 139.65, 143.89, 168.10; Elemental Analysis: Calcd. C: 62.24, H: 6.09, N: 4.03, O: 27.64, Found. C: 62.01, H: 6.04, N: 4.01, O: 27.59.

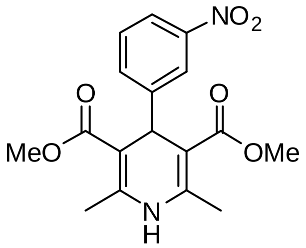


^{1}H NMR of 5l

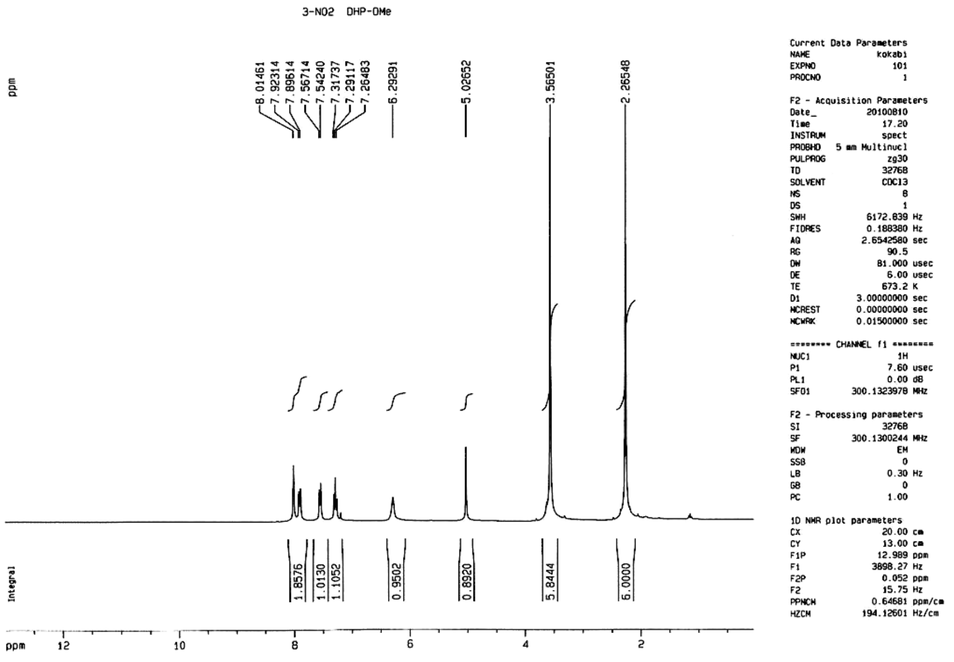


^{13}C NMR of 5l

Dimethyl 2,6-dimethyl-4-(3-nitrophenyl)-1,4-dihydropyridine-3,5-dicarboxylate[9]



Mp: 187-190 °C; ^{1}H NMR (CDCl$_3$, 300 MHz): δ (ppm): 2.26 (s, 6 H), 3.56(s, 6 H), 5.02 (s, 1 H), 6.29 (s, 1 H), 8.01-7.26 (m, 4 H); ^{13}C NMR (CDCl$_3$, 75 MHz): δ (ppm): 19.44, 39.60, 51.16, 102.88, 121.39, 122.69, 128.76, 134.24, 145.37, 148.31, 149.65, 167.67; Elemental Analysis: Calcd. C: 58.96, H: 5.24, N: 8.09, O: 27.72, Found. C: 58.66, H: 5.21, N: 8.01, O: 27.70.



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Current Data Parameters
NAME               kokabi
EXPNO                 101
PROCNO                  1

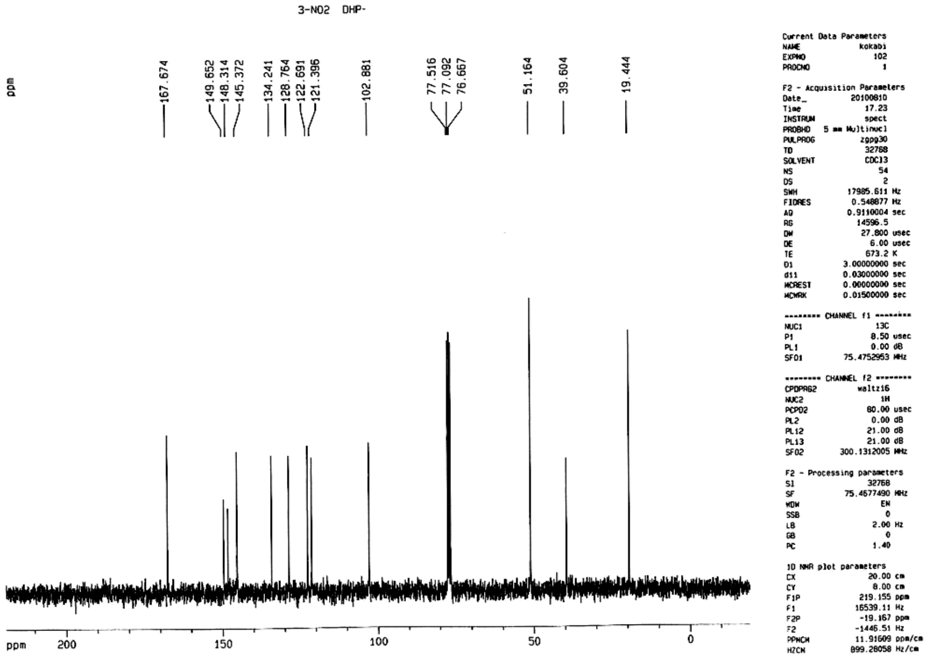
F2 - Acquisition Parameters
Date_            20100810
Time                17.20
INSTRUM             spect
PROBHD   5 mm Multinucl
PULPROG              zg30
TD                  32768
SOLVENT             CDC13
NS                      8
DS                      1
SWH             6172.839 Hz
FIDRES          0.188380 Hz
AQ             2.6542580 sec
RG                   90.5
DW                 81.000 usec
DE                   6.00 usec
TE                  673.2 K
D1            3.00000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                   7.60 usec
PL1                  0.00 dB
SFO1         300.1323978 MHz

F2 - Processing parameters
SI                  32768
SF           300.1300244 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00

1D NMR plot parameters
CX                  20.00 cm
CY                  13.00 cm
F1P                12.989 ppm
F1                3898.27 Hz
F2P                 0.052 ppm
F2                  15.75 Hz
PPMCM             0.64681 ppm/cm
HZCM            194.12601 Hz/cm
```

^{1}H NMR of 5o



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Current Data Parameters
NAME              kokabi
EXPNO                102
PROCNO                 1

F2 - Acquisition Parameters
Date_           20100810
Time               17.23
INSTRUM            spect
PROBHD   5 mm Multinucl
PULPROG           zgpg30
TD                 32768
SOLVENT            CDCl3
NS                    54
DS                     2
SWH            17985.611 Hz
FIDRES          0.548877 Hz
AQ             0.9110004 sec
RG               14596.5
DW                27.800 usec
DE                  6.00 usec
TE                 673.2 K
D1            3.00000000 sec
d11           0.03000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                  8.50 usec
PL1                 0.00 dB
SFO1          75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                 0.00 dB
PL12               21.00 dB
PL13               21.00 dB
SFO2         300.1312005 MHz

F2 - Processing parameters
SI                 32768
SF            75.4677490 MHz
WDW                   EM
SSB                    0
LB                  2.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 20.00 cm
CY                  8.00 cm
F1P              219.155 ppm
F1              16539.11 Hz
F2P              -19.167 ppm
F2              -1446.51 Hz
PPMCM           11.91609 ppm/cm
HZCM           899.28058 Hz/cm
```

^{13}C NMR of 5o

References:

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