

Supporting information for:

A Multipolymer Reaction System for Selective Aerobic Alcohol

Oxidation: Simultaneous Use of Multiple Different Polymer-Supported

Ligands

Cecilia Wan Ying Chung and Patrick H. Toy*

Department of Chemistry, The University of Hong Kong, Pokfulam Road, Hong Kong, People's Republic of China

phtoy@hku.hk

Contents:

¹ H- and ¹³ C-NMR spectra for 1-8	S2-S8
¹ H-NMR data for 10a-r	S9-S11

General Procedures. Commercially available reagents were used as received. Tetrahydrofuran was purified using a Solv-Tek purification system employing activated Al₂O₃. All reactions were monitored by TLC analysis using GF₂₅₄ silica gel coated plates. Column chromatography was carried out using silica gel (300-400 mesh) at increased pressure. ¹H- and ¹³C-NMR spectra were recorded in CDCl₃ on a Bruker DRX-300 or DRX-400 spectrometer operating at 300 or 400 MHz for ¹H analysis and 100 or 75 MHz for ¹³C analysis. Chemical shift data is expressed in ppm with reference to TMS. HR EI-MS data was recorded on a Finnigan MAT 96 mass spectrometer.

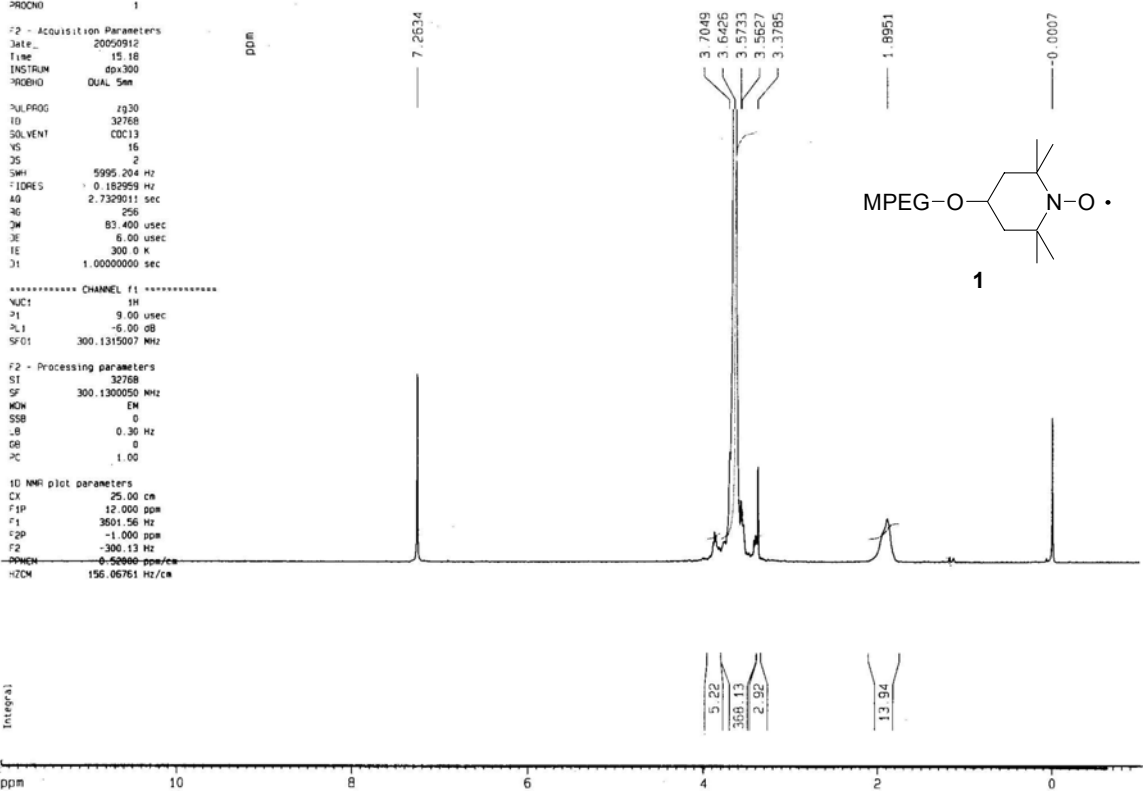
Current Data Parameters
 NAME cec1-b2-171
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20050912
 Time 15.18
 INSTRUM dpx300
 PROBHD DUAL 5mm
 PULPROG zg30
 ID 32768
 SOLVENT CDCl3
 VS 16
 ZS 2
 SFO 5995.204 Hz
 FIDRES 0.182959 Hz
 AQ 2.7329011 sec
 RG 256
 SW 83.400 usec
 SE 8.00 usec
 IE 300.0 K
 SI 1.0000000 sec

***** CHANNEL f1 *****
 NUC1 1H
 P1 9.00 usec
 PL1 -6.00 dB
 SFO1 300.1315007 MHz

F2 - Processing parameters
 SI 32768
 SF 300.1300050 MHz
 WDW EM
 SSB 0
 B 0.30 Hz
 GB 0
 PC 1.00

ID NMR plot parameters
 CX 25.00 cm
 F1P 12.000 ppm
 F1 3601.56 Hz
 F2P -1.000 ppm
 F2 -300.13 Hz
 PRMCH 0.50000 ppm/cm
 HZCM 156.06761 Hz/cm



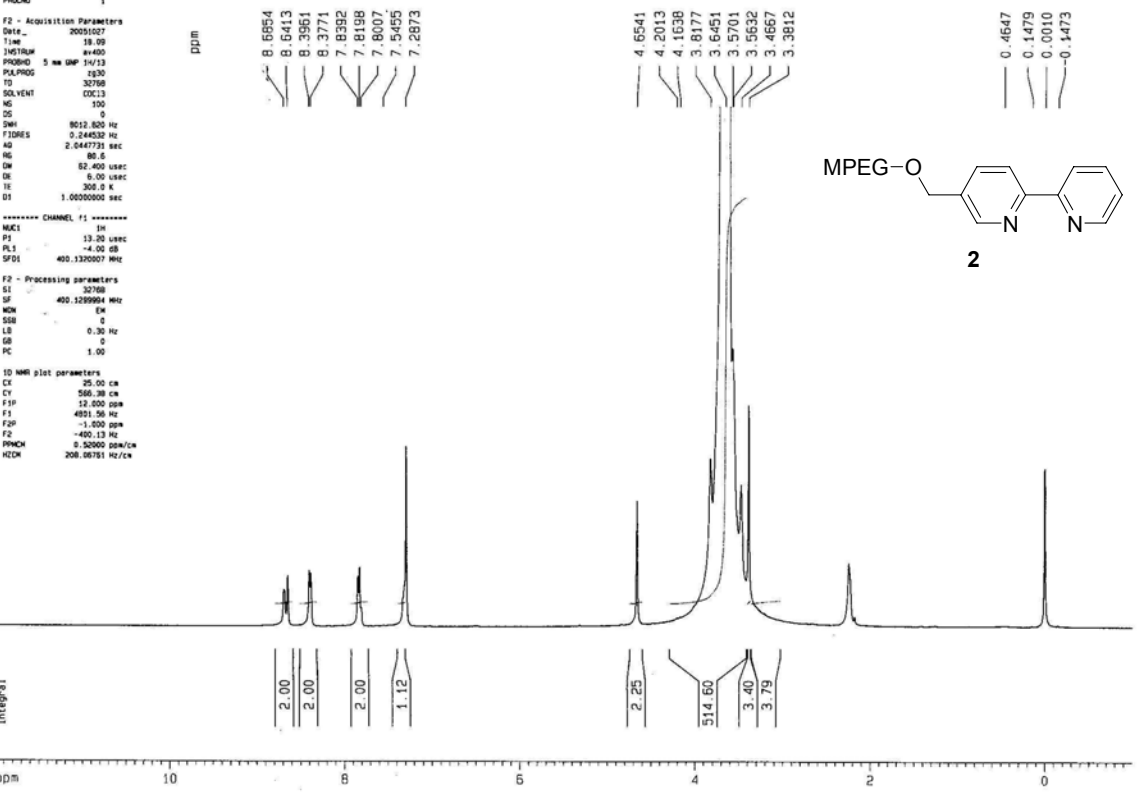
Current Data Parameters
 NAME cec1-b2-185
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20051027
 Time 18.00
 INSTRUM av400
 PROBHD 5 mm SVP 1H/13
 PULPROG zgpg30
 ID 32768
 SOLVENT CDCl3
 NS 300
 DS 0
 SW 802.800 Hz
 FIDRES 0.244532 Hz
 AQ 2.0447731 sec
 RG 66.5
 SW 82.400 usec
 SE 8.00 usec
 IE 300.0 K
 SI 1.0000000 sec

***** CHANNEL f1 *****
 NUC1 1H
 P1 13.00 usec
 PL1 -4.00 dB
 SFO1 400.1300007 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1299994 MHz
 WDW EM
 SSB 0
 B 0.30 Hz
 GB 0
 PC 1.00

ID NMR plot parameters
 CX 25.00 cm
 CY 566.30 cm
 F1P 12.000 ppm
 F1 4001.30 Hz
 F2P -1.000 ppm
 F2 -400.13 Hz
 PRMCH 0.50000 ppm/cm
 HZCM 208.06761 Hz/cm



```

Current Data Parameters
NAME      cec1-93-98
EXPNO    1
PROCNO   1

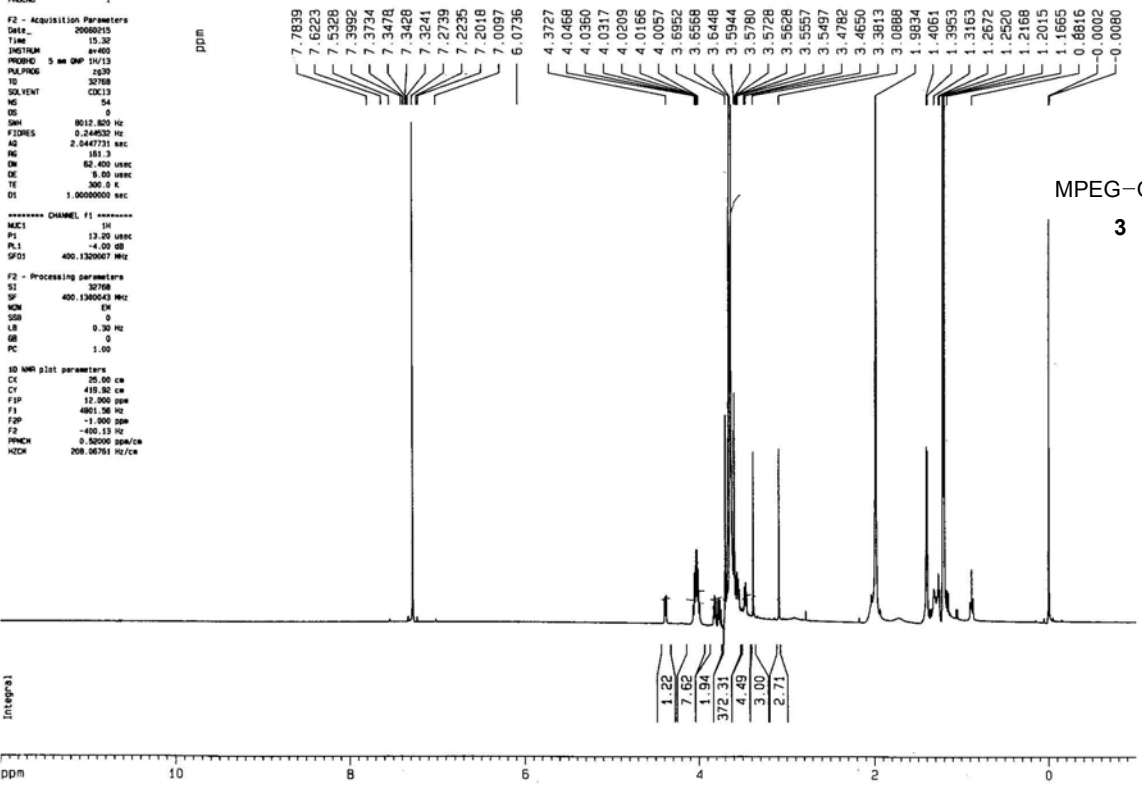
F2 - Acquisition Parameters
Date_    20080915
Time     15.30
INSTRUM  av400
PROBHD   5 mm QNP 1H/13
PULPROG  zgpg30
TD        32768
SOLVENT  CDCl3
NS        64
DS        0
SWH       8032.820 Hz
FIDRES    0.244832 Hz
AQ        2.0447731 sec
RG         381.3
DN        62.400 usec
DE         9.00 usec
TE        300.2 K
D1        1.00000000 sec

----- CHANNEL f1 -----
NUC1      13C
P1        13.20 usec
PL1       -4.00 dB
SFO1     400.1320007 MHz

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

ID NMR plot parameters
CX        25.00 cm
CY        419.50 cm
F1P       12.000 ppm
F1        4001.56 Hz
F2P       -1.000 ppm
F2        -400.13 Hz
P1NCH     0.52000 ppm/cx
HZCX      208.96761 Hz/cx

```



Current Data Parameters
 NAME cec1-b2-146-2
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20050721
 Time 11:29
 INSTRUM gxi300
 PROBHD DUAL 5mm

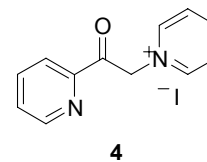
PULPROG zg30
 TD 32768
 SOLVENT DMSO
 NS 24
 DS 2
 SWH 5995.204 Hz
 FIDRES 0.182958 Hz
 AQ 2.7329011 sec
 RG 362
 DM 83.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.0000000 sec

----- CHANNEL f1 -----
 NUC1 1H
 P1 9.00 usec
 PL1 -6.00 dB
 SFO1 300.1315007 MHz

F2 - Processing parameters
 SI 32768
 SF 300.1300015 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 25.00 cm
 F1P 12.000 ppm
 F1 3601.56 Hz
 F2P -1.000 ppm
 F2 -300.13 Hz
 SFO1 300.1315007 MHz
 HZCM 156.06761 Hz/cm

ppm
 9.01987
 9.00072
 8.88175
 8.86661
 8.73281
 8.29943
 8.27589
 8.25295
 8.13944
 8.11652
 8.11413
 8.08713
 7.83520
 6.51075



Integrals

2.0553
 0.9815
 1.0252
 2.0629
 2.0483
 1.0000
 2.0251



F2 - Acquisition Parameters
 Date_ 20050606
 Time 10:43
 INSTRUM gxi300
 PROBHD DUAL 5mm

PULPROG zgpg30
 TD 32768
 SOLVENT DMSO
 NS 230
 DS 1
 SWH 18832.393 Hz
 FIDRES 0.574719 Hz
 AQ 0.8700404 sec
 RG 4096
 DM 26.550 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.5000000 sec
 D11 0.0300000 sec

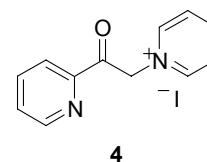
----- CHANNEL f1 -----
 NUC1 13C
 P1 7.00 usec
 PL1 -6.00 dB
 SFO1 75.4760204 MHz

----- CHANNEL f2 -----
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 100.00 usec
 PL2 120.00 dB
 PL12 18.00 dB
 SFO2 300.1312005 MHz

F2 - Processing parameters
 SI 32768
 SF 75.4677160 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 25.00 cm
 F1P 234.811 ppm
 F1 17720.64 Hz
 F2P -14.731 ppm
 F2 -1111.75 Hz
 SFO1 75.4677160 MHz
 HZCM 753.25959 Hz/cm

ppm
 192.375
 151.370
 150.476
 147.258
 147.221
 139.085
 130.050
 128.631
 122.964
 67.565
 41.293
 41.013
 40.737
 40.460
 40.182
 39.907
 39.640



Current Data Parameters
 NAME cec1-b2-138
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20050805
 Time 13.47
 INSTRUM dp300
 PROBHD DUAL 5mm

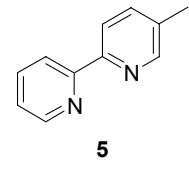
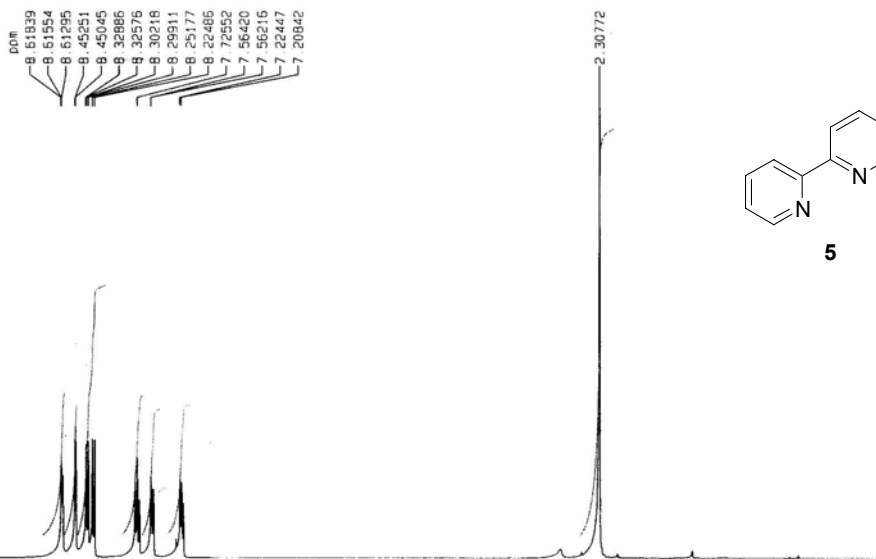
PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 5995.204 Hz
 FIDRES 0.182959 Hz
 AQ .2.7329011 sec
 RG 64
 DM 83.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.0000000 sec

----- CHANNEL f1 -----
 NUC1 1H
 P1 9.00 usec
 PL1 -6.00 dB
 SFO1 300.1315007 MHz

F2 - Processing parameters
 SI 32768
 SF 300.1300035 MHz
 MDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

ID NMR plot parameters
 CX 25.00 cm
 F1P 12.000 ppm
 F1 3601.56 Hz
 F2P -1.000 ppm
 F2 -300.13 Hz
 PRMCH 0.52660 ppm/cm
 HZCX 156.08761 Hz/cm

ppm
 8.61539
 8.61554
 8.61295
 8.45231
 8.45045
 8.32886
 8.32575
 8.30218
 8.29911
 8.25177
 8.22466
 7.72552
 7.56420
 7.56216
 7.22447
 7.20842



Integral

1.0458
 0.9570
 1.8411
 1.0362
 0.9293
 0.9581

3.0000

ppm 10 8 6 4 2 0

Current Data Parameters
 NAME cec1-b2-138
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20050805
 Time 13.57
 INSTRUM dp300
 PROBHD DUAL 5mm

PULPROG zgpc
 TD 32768
 SOLVENT CDCl3
 NS 101
 DS 1
 SWH 18632.393 Hz
 FIDRES 0.574719 Hz
 AQ 0.8700404 sec
 RG 8192
 DM 26.550 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.5000000 sec
 d11 0.0300000 sec

----- CHANNEL f1 -----
 NUC1 13C
 P1 7.00 usec
 PL1 -6.00 dB
 SFO1 75.476204 MHz

----- CHANNEL f2 -----
 CPDPRG2 waltz16
 NUC2 1H
 PPRD2 100.00 usec
 PL2 120.00 dB
 PL12 18.00 dB
 SFO2 300.1312005 MHz

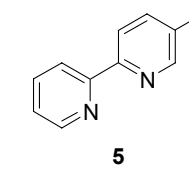
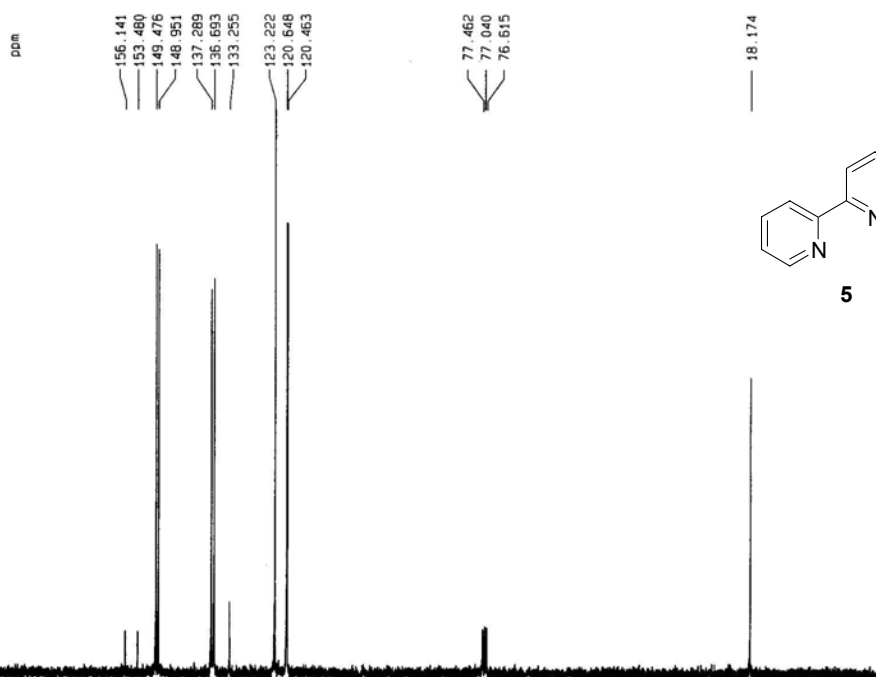
F2 - Processing parameters
 SI 32768
 SF 75.4677564 MHz
 MDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

ID NMR plot parameters
 CX 25.00 cm
 F1P 234.765 ppm
 F1 17717.20 Hz
 F2P -14.777 ppm
 F2 -1115.20 Hz
 PRMCH 9.98169 ppm/cm
 HZCX 753.29508 Hz/cm

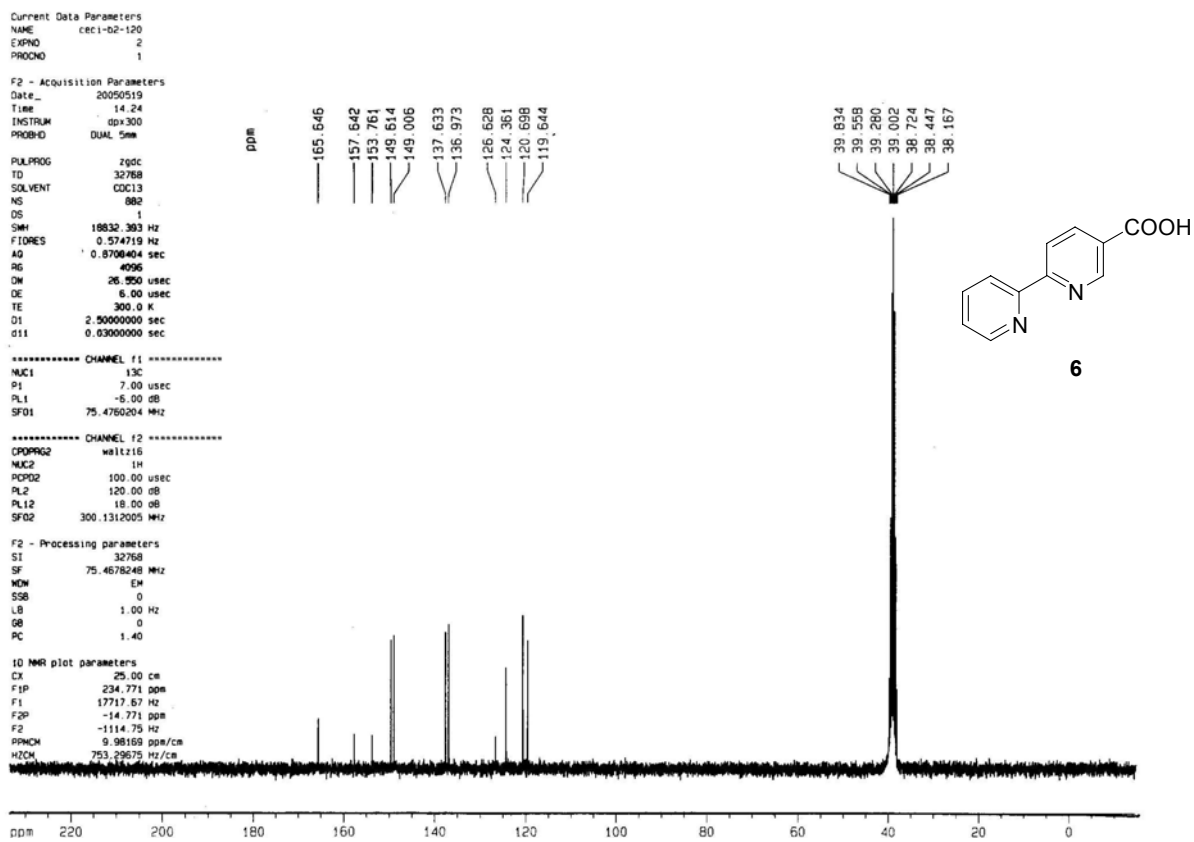
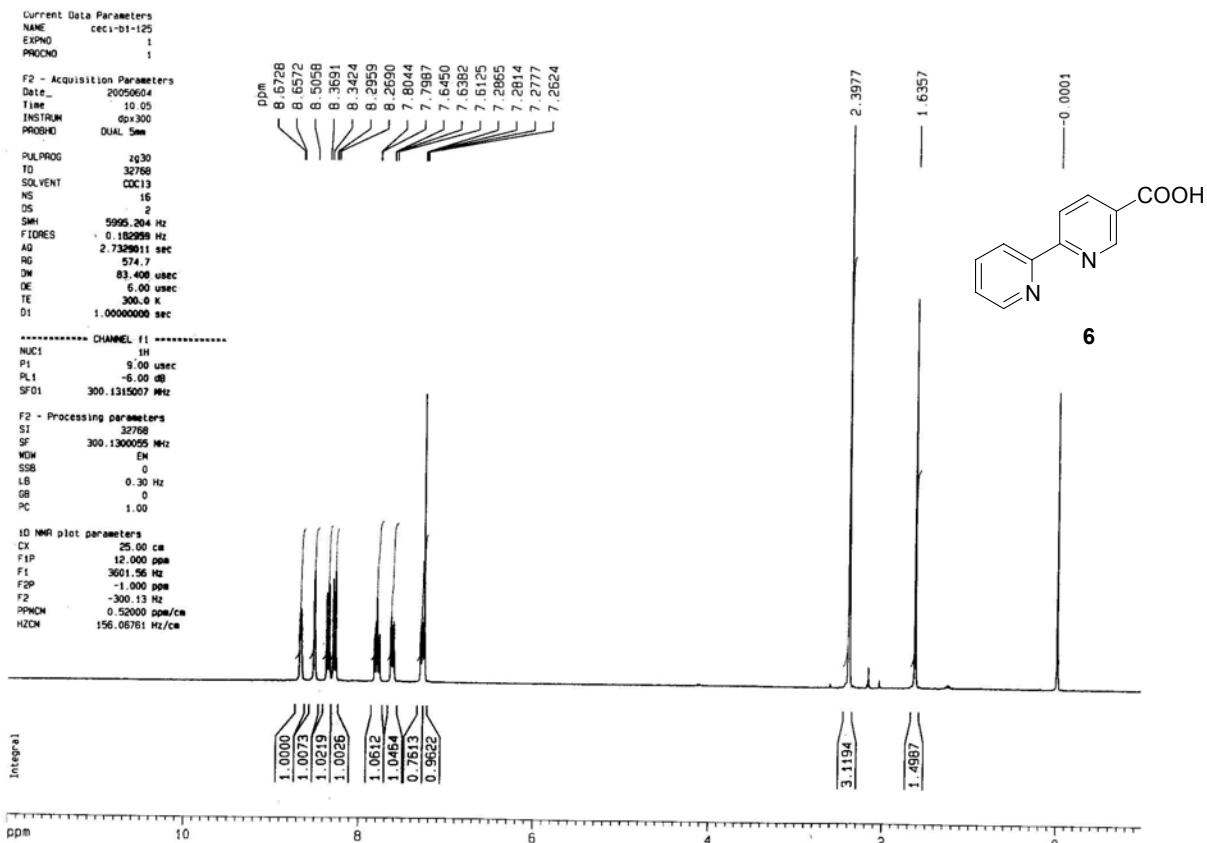
ppm
 156.141
 153.480
 149.476
 148.951
 137.289
 136.693
 133.255
 123.222
 120.648
 120.463

77.462
 77.040
 76.615

18.174



ppm 220 200 180 160 140 120 100 80 60 40 20 0



```

Current Data Parameters
NAME cec1-02-137
EXPNO 1
PROCNO 1

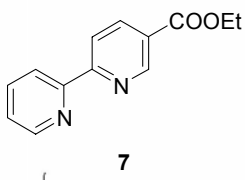
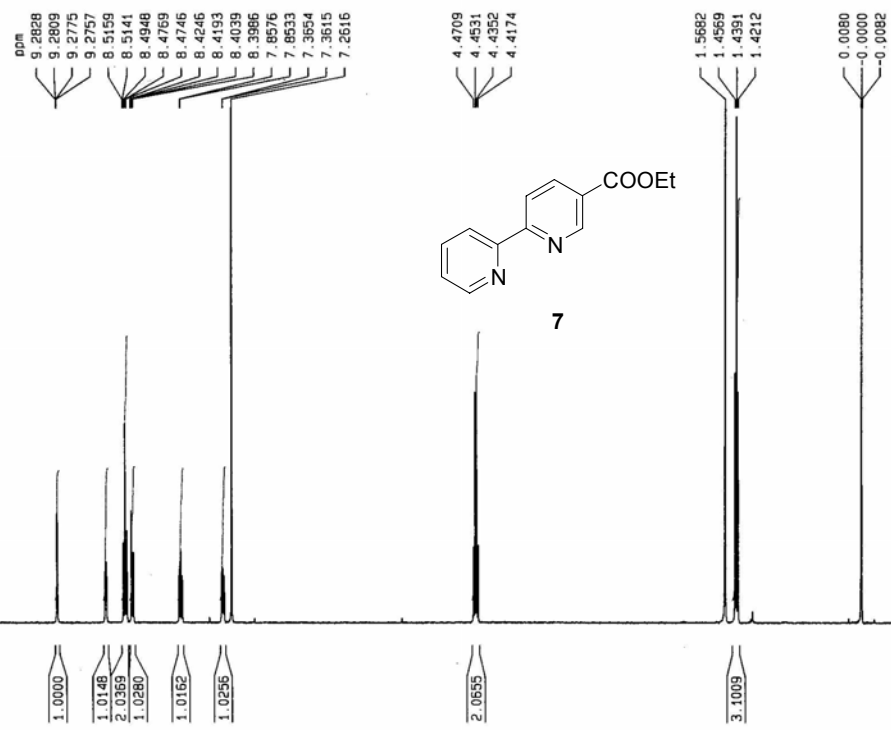
F2 - Acquisition Parameters
Date_ 20050626
Time 19.30
INSTRUM mv600
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 80
DS 4
SWH 801.800 Hz
FIDRES 0.344530 Hz
AQ 2.6447731 sec
RG 2500.3
DM 80.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.0000000 sec

----- CHANNEL f1 -----
NUC1 13C
P1 13.20 usec
PL1 -4.00 dB
SFO1 400.1320007 MHz

F2 - Processing parameters
SI 32768
SF 400.1320000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

ID NMR plot parameters
CX 25.00 cm
CY 24.00 cm
F1P 12.000 ppm
F1 400.136 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PCHCK 0.52000 ppm/cm
KDCW 508.00751 Hz/cm

```



```

Current Data Parameters
NAME cec1-02-145
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050728
Time 17.05
INSTRUM dpx300
PROBHD DUAL 5mm
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 500
DS 1
SWH 18832.393 Hz
FIDRES 0.574719 Hz
AQ 0.8700404 sec
RG 4997.6
DM 26.550 usec
DE 6.00 usec
TE 300.0 K
D1 2.5000000 sec
d11 0.0300000 sec

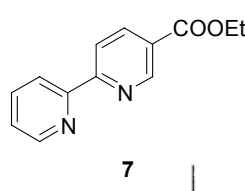
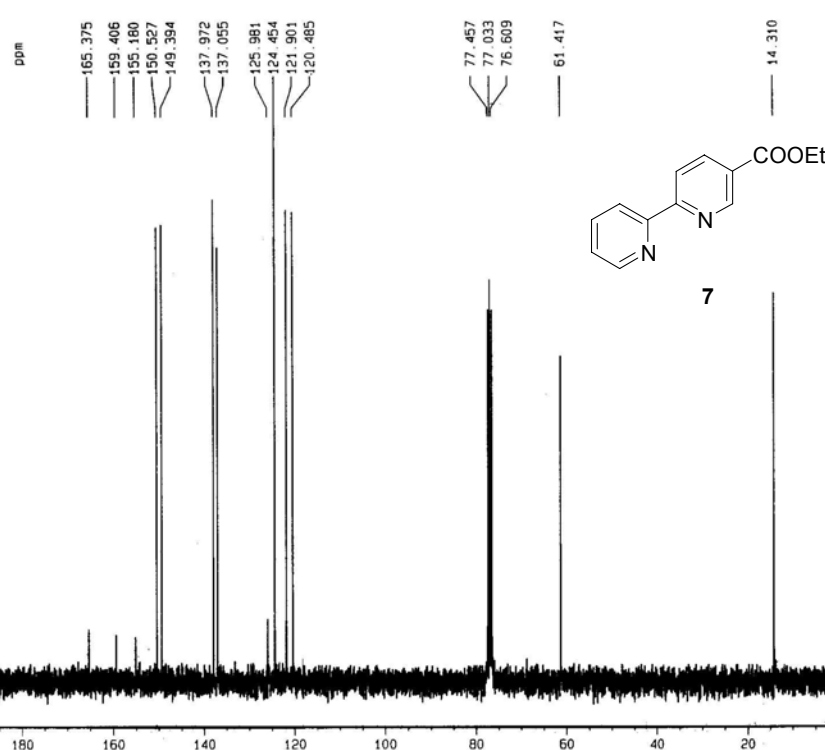
----- CHANNEL f1 -----
NUC1 13C
P1 7.00 usec
PL1 -6.00 dB
SFO1 75.4760204 MHz

----- CHANNEL f2 -----
CHOPPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 18.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677472 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

ID NMR plot parameters
CX 25.00 cm
CY 234.398 ppm
F1P 17689.46 Hz
F1 17689.46 Hz
F2P -15.145 ppm
F2 -1542.04 Hz
PCHCK 9.98169 ppm/cm
KDCW 178.2338 Hz/cm

```

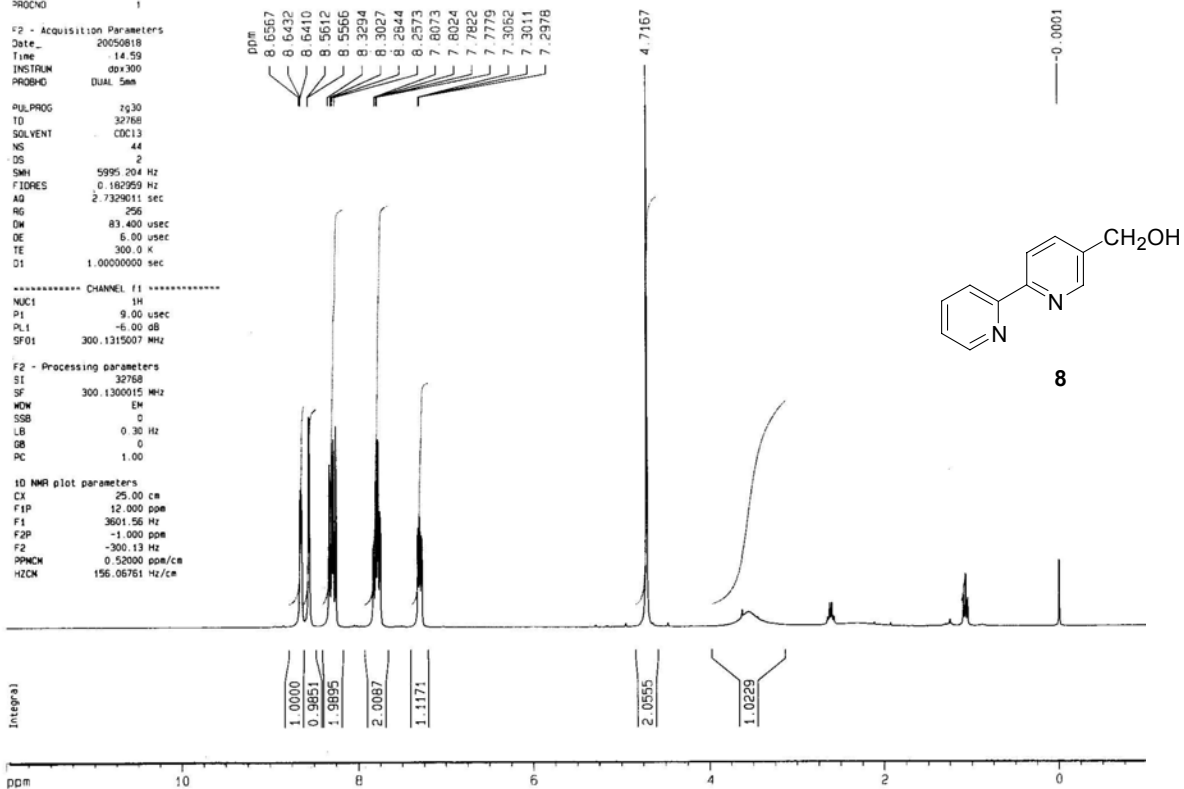


Current Data Parameters
 NAME cec1-02-153
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20050818
 Time 14.59
 INSTRUM dp300
 PROBHD DUAL 5mm
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 44
 DS 2
 SSI 5995.204 Hz
 FIDRES 0.182959 Hz
 AQ 2.7329011 sec
 RG 296
 DM 83.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.0000000 sec

----- CHANNEL f1 -----
 NUC1 1H
 P1 9.00 usec
 PL1 -6.00 dB
 SFO1 300.1315007 MHz
 F2 - Processing parameters
 SI 32768
 SF 300.1300015 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 25.00 cm
 F1P 12.000 ppm
 F1 3601.56 Hz
 F2P -1.000 ppm
 F2 -300.13 Hz
 PPMCH 0.52000 ppm/cm
 HZCN 156.06761 Hz/cm



Current Data Parameters
 NAME yd-1-30
 EXPNO 2
 PROCNO 2

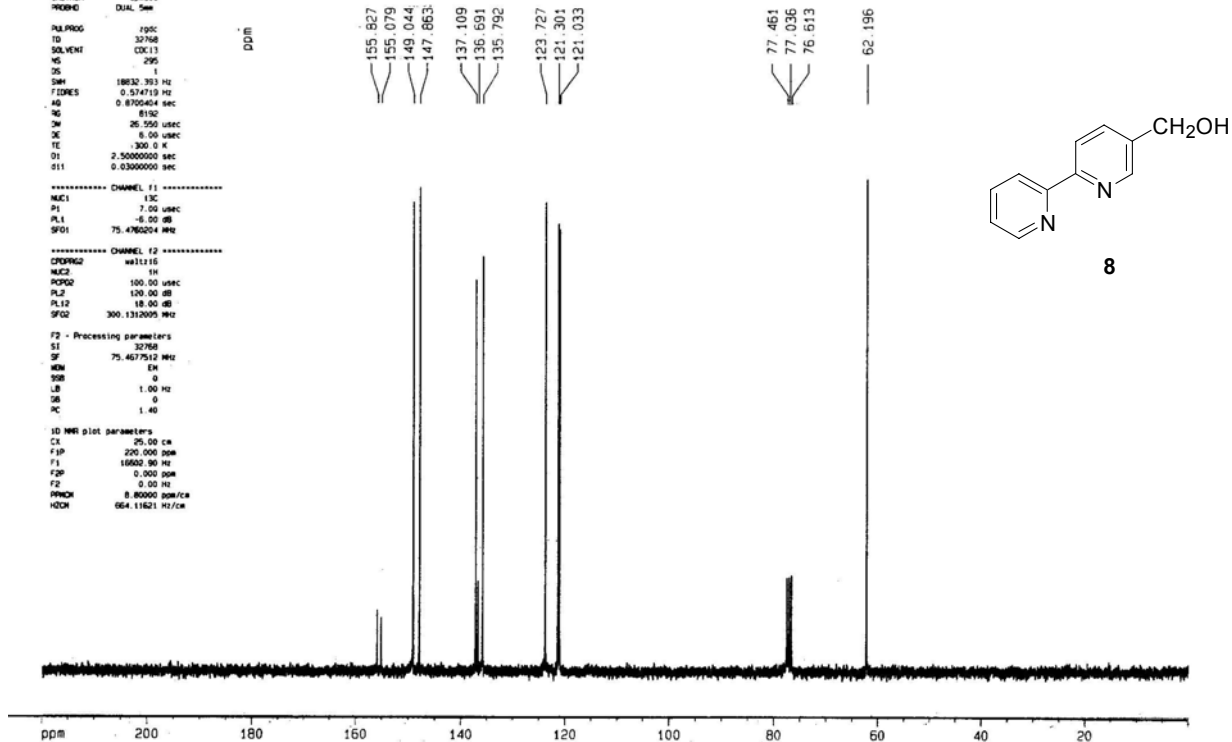
F2 - Acquisition Parameters
 Date_ 20060308
 Time 13.32
 INSTRUM dp300
 PROBHD DUAL 5mm
 PULPROG zgpg
 TD 32768
 SOLVENT CDCl3
 NS 295
 DS 1
 SSI 18833.353 Hz
 FIDRES 0.574719 Hz
 AQ 0.870044 sec
 RG 8192
 DM 26.550 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.5000000 sec
 D11 0.0300000 sec

----- CHANNEL f1 -----
 NUC1 13C
 P1 7.00 usec
 PL1 -6.00 dB
 SFO1 75.4762024 MHz

----- CHANNEL f2 -----
 CPDPRG2 waltz16
 NUC2 1H
 P2P2 100.00 usec
 PL2 120.00 dB
 PL12 18.00 dB
 SFO2 300.1312005 MHz

F2 - Processing parameters
 SI 32768
 SF 75.4677512 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 25.00 cm
 F1P 200.000 ppm
 F1 18862.80 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPMCH 8.80000 ppm/cm
 HZCN 664.11621 Hz/cm



Characterization data for 10a-r:

Benzaldehyde (10a). $^1\text{H-NMR}$ (CHCl_3 , 300 MHz): δ 7.51-7.67 (3H, m), 7.87-7.91 (2H, m), 10.03 (1H, s).

4-Bromobenzaldehyde (10b). $^1\text{H-NMR}$ (CHCl_3 , 300 MHz): δ 7.69 (2H, d, $J = 8.5$ Hz), 7.76 (2H, d, $J = 8.2$ Hz), 9.98 (1H, s).

4-Nitrobenzaldehyde (10c). $^1\text{H-NMR}$ (CHCl_3 , 300 MHz): δ 8.08 (2H, d, $J = 8.3$ Hz), 8.40 (2H, d, $J = 8.6$ Hz), 10.16 (1H, s).

4-Methylbenzaldehyde (10d). $^1\text{H-NMR}$ (CHCl_3 , 400 MHz): δ 2.44 (3H, s), 7.33 (2H, d, $J = 8.0$ Hz), 7.78 (2H, d, $J = 8.1$ Hz), 9.97 (1H, s).

4-Methoxybenzaldehyde (10e). $^1\text{H-NMR}$ (CHCl_3 , 300 MHz): δ 3.90 (3H, s), 7.01 (2H, d, $J = 8.6$ Hz), 7.85 (2H, d, $J = 8.4$ Hz), 9.89 (1H, s).

Piperonal (10f). $^1\text{H-NMR}$ (CHCl_3 , 300 MHz): δ 6.08 (2H, s), 6.94 (1H, d, $J = 7.9$ Hz), 7.34 (1H, d, $J = 1.5$ Hz), 7.42 (1H, dd, $J = 7.9, 1.5$ Hz), 9.82 (1H, s).

2-Bromobenzaldehyde (10g). $^1\text{H-NMR}$ (CHCl_3 , 400 MHz): δ 7.43-7.47 (2H, m), 7.64-7.67 (1H, m), 7.91-7.93 (1H, m), 10.37 (1H, s).

2-Nitrobenzaldehyde (10h). $^1\text{H-NMR}$ (CHCl_3 , 400 MHz): δ 7.75-7.83 (2H, m), 7.96 (1H, dd, $J = 7.3, 1.9$ Hz), 8.13 (1H, dd, $J = 7.7, 1.3$ Hz), 10.43 (1H, s).

2-Methylbenzaldehyde (10i). $^1\text{H-NMR}$ (CHCl_3 , 300 MHz): δ 2.68 (3H, s), 7.26 (1H, d, $J = 7.5$ Hz), 7.36 (1H, t, $J = 7.3$ Hz), 7.48 (1H, td, $J = 7.6, 1.5$ Hz), 7.80 (1H, dd, $J = 7.6, 1.4$ Hz), 10.28 (1H, s).

2-Methoxybenzaldehyde (10j). $^1\text{H-NMR}$ (CHCl_3 , 400 MHz): δ 3.93 (3H, s), 6.98-7.05 (2H, m), 7.56 (1H, td, $J = 7.9, 1.8$ Hz), 7.83 (1H, dd, $J = 7.7, 1.8$ Hz), 10.48 (1H, s).

2,4-Dimethoxybenzaldehyde (10k). $^1\text{H-NMR}$ (CHCl_3 , 300 MHz): δ 3.88 (3H, s), 3.91 (3H, s), 6.45 (1H, d, $J = 2.2$ Hz), 6.55 (1H, dd, $J = 8.9, 2.2$ Hz), 7.82 (1H, d $J = 8.7$ Hz), 10.29 (1H, s).

2,4,6-Trimethylbenzaldehyde (10l). $^1\text{H-NMR}$ (CHCl_3 , 400 MHz): δ 2.32 (3H, s), 2.58 (6H, s), 6.90 (2H, s), 10.57 (1H, s).

2,6-Dichlorobenzaldehyde (10m). $^1\text{H-NMR}$ (CHCl_3 , 400 MHz): δ 7.40 (3H, s), 10.37 (1H, s).

2,4,6-Trichlorobenzaldehyde (10n).¹ $^1\text{H-NMR}$ (CHCl_3 , 300 MHz): δ 7.42 (2H, s), 10.43 (1H, s).

Thiophene-2-carbaldehyde (10o). $^1\text{H-NMR}$ (CHCl_3 , 300 MHz): δ 7.22 (1H, dd, $J = 4.8, 3.8$ Hz), 7.76-7.80 (2H, m), 9.95 (1H, d, $J = 1.2$ Hz).

Pyridine-3-carbaldehyde (10p). $^1\text{H-NMR}$ (CHCl_3 , 300 MHz): δ 7.50 (1H, dd, $J = 7.8, 5.2$ Hz), 8.19 (1H, d, $J = 6.6$ Hz), 8.86 (1H, d, $J = 4.7$ Hz), 9.09 (1H, s), 10.13 (1H, s).

Cinnamaldehyde (10q). $^1\text{H-NMR}$ (CHCl_3 , 400 MHz): δ 6.73 (1H, dd, $J = 15.9, 7.7$ Hz), 7.44-7.51 (4H, m), 7.57-7.59 (2H, m), 9.72 (1H, d, $J = 7.7$ Hz).

Geranial (10r). $^1\text{H-NMR}$ (CHCl_3 , 400 MHz): δ 1.61 (3H, s), 1.69 (3H, s), 2.17 (3H, d, $J = 1.2$ Hz), 2.19-2.26 (4H, m), 5.05-5.08 (1H, m), 5.89 (1H, d, $J = 8.1$ Hz), 10.00 (1H, d, $J = 8.1$ Hz).

Citronellal (10s). $^1\text{H-NMR}$ (CHCl_3 , 400 MHz): δ 0.97 (3H, d, $J = 6.7$ Hz), 1.21-1.42 (2H, m), 1.60 (3H, s), 1.67 (3H, s), 1.98-2.12 (3H, m), 2.20-2.26 (1H, m), 2.38-2.46 (1H, m), 5.09 (1H, t, $J = 6.4$ Hz), 9.76 (1H, s).

Benzaldehyde (10a) from 9t. $^1\text{H-NMR}$ (CHCl_3 , 300 MHz): δ 7.51-7.64 (3H, m), 7.87-7.90 (2H, m), 10.03 (1H, s).

Reference:

1. Giurg, M.; Mlochowski, J. *Synth. Commun.* **1999**, *29*, 4307-4313.