Electronic Supplementary Information

A family of ligand and anion dependent structurally diverse Cu(II) Schiff-base complexes and their catalytic efficacy in O-arylation reaction in ethanolic medium

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Table S1 Selected bond lengths (Å) and angles (°) for compounds **1** and **2**

Cu2–O2	1.894(5)	Cu1–O1	1.909(2)
Cu2–O3	1.989(5)	Cu1–O3	1.981(2)
Cu2–O4	2.734(7)	Cu1–O4	2.654(3)
Cu2–O6	2.451(6)	Cu1–O6	2.494(4)
Cu2–N1	1.914(6)	Cu1–N1	2.061(3)
Cu2–N2	2.061(6)	Cu1–N3	1.918(3)
O2–Cu2–O3	86.9(2)	O1–Cu1–O3	87.25(10)
O2–Cu2–O4	91.80(19)	O1–Cu1–O4	93.34(9)
O2–Cu2–O6	86.9(2)	O1–Cu1–O6	87.33(11)
O2–Cu2–N1	94.5(2)	O1–Cu1–N1	169.39(11)
O2–Cu2–N2	169.8(2)	O1–Cu1–N3	94.42(10)
O3–Cu2–O4	51.7(2)	O3–Cu1–O4	53.20(10)
O3–Cu2–O6	101.9(2)	O3–Cu1–O6	99.74(12)
O3–Cu2–N1	171.1(2)	O3–Cu1–N1	93.22(11)
O3–Cu2–N2	94.6(2)	O3–Cu1–N3	171.58(11)
O4–Cu2–O6	153.6(2)	O4–Cu1–O6	152.79(12)
O4–Cu2–N1	119.4(2)	O4–Cu1–N1	95.37(10)
O4–Cu2–N2	97.0(2)	O4–Cu1–N3	118.42(11)
O6–Cu2–N1	86.9(2)	O6–Cu1–N1	82.13(12)
O6–Cu2–N2	82.9(2)	O6–Cu1–N3	88.66(11)
N1–Cu2–N2	85.6(3)	N1–Cu1–N3	86.66(11)

Table S2 Selected bond lengths (Å) and angles (°) for compounds **3, 4** and **5**

Cu–O1	1.8704(13)	Cu–O1	1.862(3)	Cu–O1	1.8704(13)
Cu–O22	2.0263(14)	Cu–O19	2.016(3)	Cu–O18	2.0263(14)
Cu–O23	2.5440(15)	Cu–O20	2.550(3)	Cu–O19	2.5440(15)
Cu–N13	1.9202(15)	Cu–N10	1.919(3)	Cu–N9	1.9202(15)
Cu–N16	2.0557(14)	Cu–N13	2.044(3)	Cu–N12	2.0557(14)
O1–Cu–O22	89.59(6)	O1–Cu–O19	89.00(10)	O1–Cu–O18	92.33(6)
O1–Cu–O23	94.14(5)	O1–Cu–O20	93.32(10)	O1–Cu–O19	88.46(6)
O1–Cu–N13	95.56(6)	O1–Cu–N10	95.68(12)	O1–Cu–N9	95.56(6)
O1–Cu–N16	165.23(6)	O1–Cu–N13	165.25(11)	O1–Cu–N12	167.42(6)
O22–Cu–O23	55.31(5)	O19–Cu–O20	55.04(9)	O18–Cu–O19	56.24(5)
O22–Cu–N13	169.41(6)	O19–Cu–N10	169.27(10)	O18–Cu–N9	118.87(5)
O22–Cu–N16	90.66(6)	O19–Cu–N13	90.55(10)	O18–Cu–N12	98.02(5)
O23–Cu–N13	114.92(6)	O20–Cu–N10	114.89(10)	O19–Cu–N9	173.91(6)
O23–Cu–N16	98.14(5)	O20–Cu–N13	98.50(10)	O19–Cu–N12	91.35(6)
N13–Cu–N16	86.71(6)	N10–Cu–N13	87.36(12)	N9–Cu–N12	85.73(6)

Table S3 Selected bond lengths (Å) and angles (°) for compound **6** and **7**

Cu1–O1	1.9901(18)	Cu1–O1	1.980(2)
Cu1–O41	1.9728(19)	Cu1–O3	2.640(3)
Cu1–O42	2.233(3)	Cu1–O4	1.915(3)
Cu1–N12	1.928(3)	Cu1–O8	2.484(3)
Cu1–N15	2.070(2)	Cu1–N2	1.910(3)
Cu2–O1	1.9875(18)	Cu1–N3	2.115(2)
Cu2–O8	2.378(3)		
Cu2–O21	1.927(2)		
Cu2–O41	2.493(2)		
Cu2–N32	1.921(3)		
Cu2–N35	2.122(2)		
O1–Cu1–O41	85.25(8)	O1–Cu1–O3	53.39(9)
O1–Cu1–O42	94.09(9)	O1–Cu1–O4	88.34(9)
O1–Cu1–N12	91.35(9)	O1–Cu1–O8	79.08(9)
O1–Cu1–N15	169.04(9)	O1–Cu1–N2	176.28(10)
O41–Cu1–O42	89.92(10)	O1–Cu1–N3	92.55(10)
O41–Cu1–N12	172.17(10)	O3–Cu1–O4	84.32(9)
O41–Cu1–N15	96.81(9)	O3–Cu1–O8	132.17(9)
O42–Cu1–N12	97.37(11)	O3–Cu1–N2	123.58(10)
O42–Cu1–N15	96.68(10)	O3–Cu1–N3	91.45(9)
N12–Cu1–N15	85.23(10)	O4–Cu1–O8	89.85(9)
O1–Cu2–O8	74.63(8)	O4–Cu1–N2	93.54(10)
O1–Cu2–O21	87.78(9)	O4–Cu1–N3	174.02(10)

O1–Cu2–O41	72.61(7)	O8–Cu1–N2	104.12(10)
O1–Cu2–N32	175.17(10)	O8–Cu1–N3	96.13(9)
O1–Cu2–N35	95.98(8)	N2–Cu1–N3	85.25(11)
O8–Cu2–O21	88.57(9)		
O8–Cu2–O41	146.80(7)		
O8–Cu2–N32	110.08(10)		
O8–Cu2–N35	93.19(9)		
O21–Cu2–N32	84.95(9)		
O21–Cu2–N35	91.24(11)		
O21–Cu2–N35	176.16(10)		
O41–Cu2–N15	102.59(9)		
O41–Cu2–N15	95.39(8)		
N32–Cu2–N15	84.95(10)		

Table S4 Hydrogen bond dimensions of complex **1, 2, 3, 4, 5, 6** and **7**

D–H···A (Å)	D–H (Å)	H···A (Å)	D···A (Å)	∠D–H···A (°)	Symmetry
N3–H3···O8	0.8600	2.4100	2.903(10)	117.00	-1+x,y,z
N3–H3···O7	0.8600	2.2200	2.773(8)	122.00	-1/2+x,-1/2+y,z
N3–H3···O1	0.8600	2.4300	3.013(9)	126.00	3/2-x,-1/2+y,1/2-z
N2–H2···O9	0.8600	2.5200	3.019(8)	118.00	-1+x,y,z
N2–H2···O7	0.8600	2.1800	2.773(5)	126.00	-1/2+x,-1/2+y,z
N19–H19A···O101	0.88(3)	2.18(3)	2.936(3)	144(2)	x,1/2-y,-1/2+z
N19–H19A···O103	0.88(3)	2.15(3)	2.986(3)	157(2)	x,1/2-y,-1/2+z
N19–H19B···O101	0.86(3)	1.91(3)	2.720(3)	157(2)	-x,-y,1-z
N16–H16A···O103	0.9900	1.8300	2.729(5)	149.00	–
N16–H16B···O102	0.9900	1.9900	2.933(4)	158.00	1-x,-1/2+y,1/2-z
N16–H16B···O103	0.9900	2.1700	2.964(6)	136.00	1-x,-1/2+y,1/2-z
N15–H15A···O103	0.9900	2.2100	2.992(3)	134.00	–
N15–H15B···O103	0.9900	1.8500	2.750(3)	150.00	-x,-1/2+y,1/2-z
O100–H10G···O102	0.83(4)	2.02(4)	2.807(5)	158(6)	1-x,1-y,1-z
O100–H10G···O302	0.83(4)	2.60(6)	3.120(12)	123(5)	1-x,1-y,1-z
O100–H10H···O203	0.827(17)	2.08(2)	2.884(5)	164(6)	-1+x,y,z
N18–H18A···O101	0.9900	2.2500	2.886(4)	121.00	–
N18–H18A···O201	0.9900	2.4200	3.012(4)	118.00	2-x,1-y,1-z
N18–H18A···O304	0.9900	2.4100	3.141(7)	130.00	1-x,2-y,1-z
N18–H18B···O201	0.9900	2.3000	3.156(4)	144.00	-1+x,y,z
N18–H18B···O101	0.9900	2.3800	3.148(4)	134.00	1-x,1-y,1-z

N38–H38A···O302	0.9900	2.2500	2.917(14)	124.00	1+x,-1+y,z
N38–H38A···O303	0.9900	2.3200	2.812(12)	110.00	2-x,1-y,1-z
N38–H38B···O203	0.9900	1.9500	2.881(4)	156.00	–
O41–H41A···O200	0.92(4)	1.76(5)	2.627(4)	158(4)	–
O41–H41B···O100	0.80(4)	1.80(5)	2.573(4)	163(5)	–
O42–H42A···O400	0.69(7)	2.33(7)	2.690(11)	115(6)	–
O42–H42A···O403	0.69(7)	2.45(7)	3.087(9)	155(7)	-1+x,y,z
O42–H42B···O21	0.69(7)	2.15(5)	2.741(4)	136(5)	–
O42–H42B···O28	0.69(7)	2.40(4)	3.100(5)	155(5)	–
N4–H4A···O9	0.90	2.44	2.862(4)	109	–
N4–H4A···O6	0.90	2.31	3.090(4)	146	-1/2+x,3/2-y,-1/2+z
N4–H4A···O7	0.90	2.33	2.985(4)	130	-1/2+x,3/2-y,-1/2+z
N4–H4B···O9	0.90	2.54	2.862(4)	101	–
N4–H4B···O7	0.90	2.46	3.055(4)	124	-x,1-y,-z
N4–H4B···O8	0.90	2.36	3.240(4)	165	-x,1-y,-z
O9–H9···O4	0.82	1.92	2.729(3)	169	-x,1-y,-z

Table S5 Comparison of copper complexes with other homogeneous catalysts[a]

Entry	Catalyst	Base	Solvent	Yield[b] (%)	TOF[c]
1	$Cu(OAc)_2.H_2O$	K_2CO_3	Ethanol	11	1
2	$Cu(SO_4)_2.5H_2O$	K_2CO_3	Ethanol	6	1
3	$Cu(ClO_4)_2.6H_2O$	K_2CO_3	Ethanol	8	1
4	$CuCl_2.2H_2O$	K_2CO_3	Ethanol	10	1
5	$CuBr_2$	K_2CO_3	Ethanol	9	1
6	$Cu(NO_3)_2.3H_2O$ + N-(2-ethylamino)piperazine	K_2CO_3	Ethanol	29	3
7	$Cu(ClO_4)_2.6H_2O$ + N-(2-ethylamino)piperazine	K_2CO_3	Ethanol	22	3
8	$[Cu(HL)NO_3]_n$ (L = 1-(N-ortho-hydroxyacetophenimine)-ethane-2-ol) (see reference 17c in the main text)	K_2CO_3	Ethanol	87	11

[a] Reaction condition: *p*–nitrobenzaldehyde (1.1 mmol), *p*–methylphenol (1.0 mmol), K_2CO_3 (1.2 mmol), catalyst (1 mol%), EtOH (3 mL) at 80 °C for 8 h. [b] Isolated yield. [c] Mol. Diaryl ether/mol. Cu h.

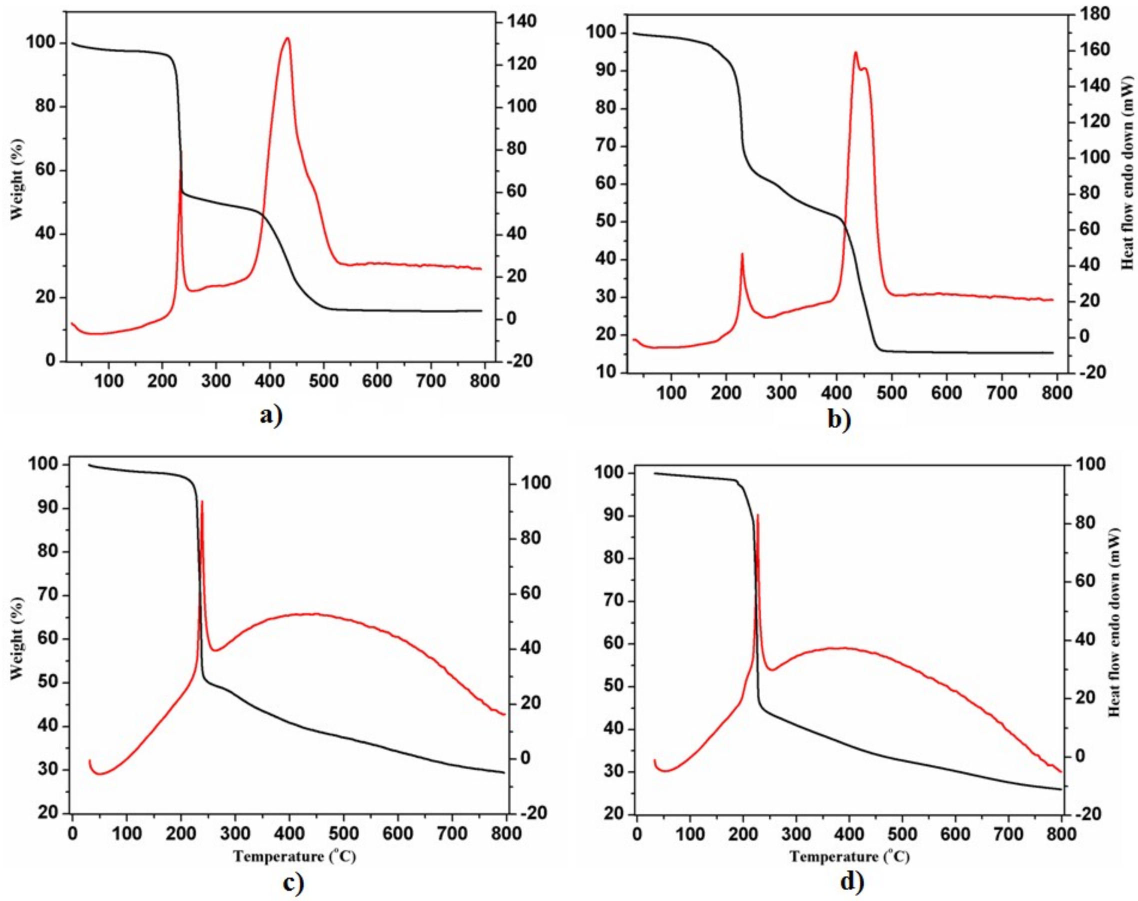


Figure S1. TG (black)/DTA (red) graphs of compounds a) **1**, b) **2**, c) **3** and d) **4**.

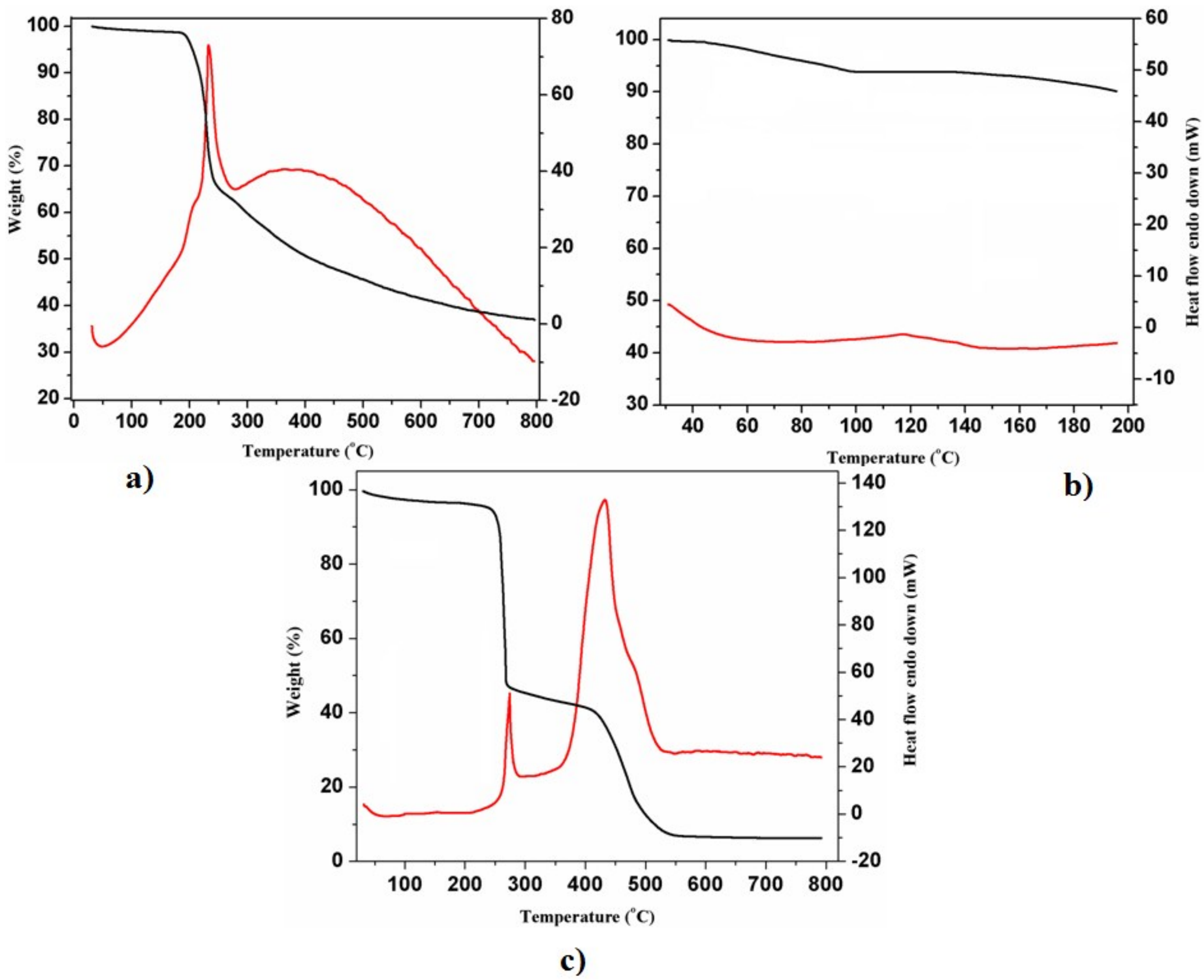


Figure S2. TG (black)/DTA (red) graph of compounds a) **5,** b) **6** and c) **7**.

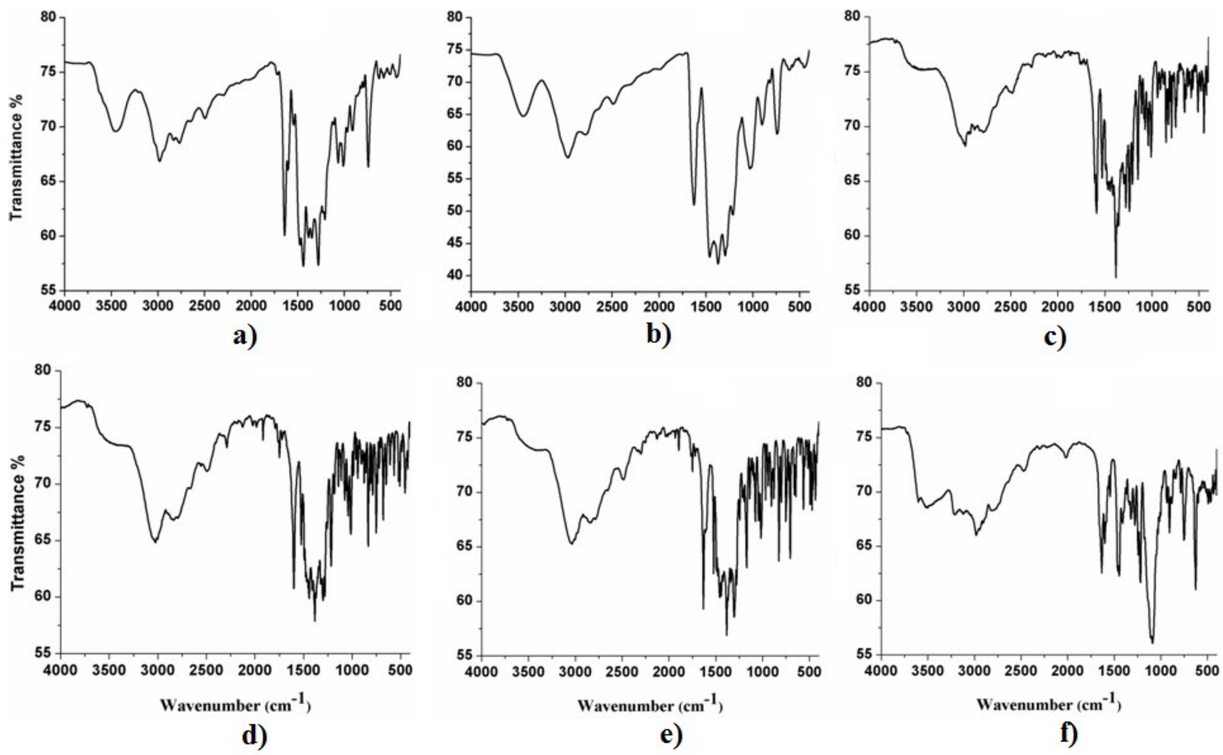


Figure S3. FTIR spectra of compounds a) **1**, b) **2**, c) **3**, d) **4**, e) **5** and f) **6** using KBr palate.

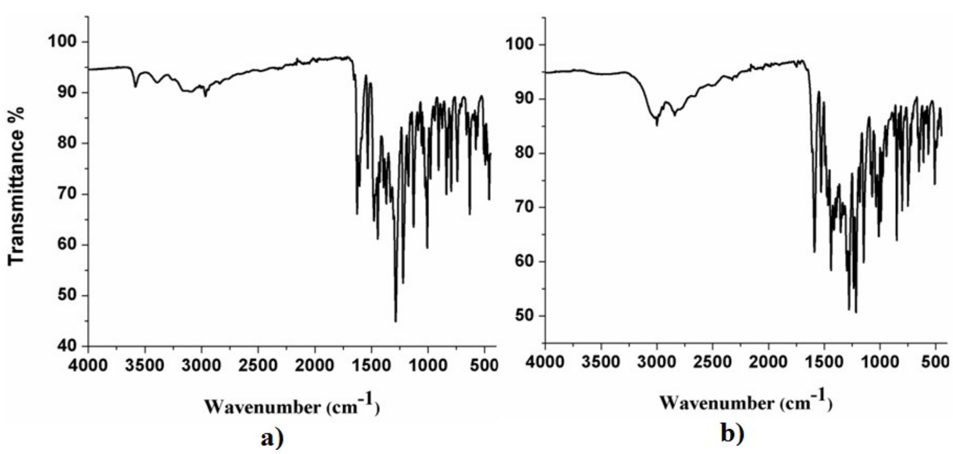


Figure S4. FTIR spectra of compounds a) **7** and b) **8** using KBr palate

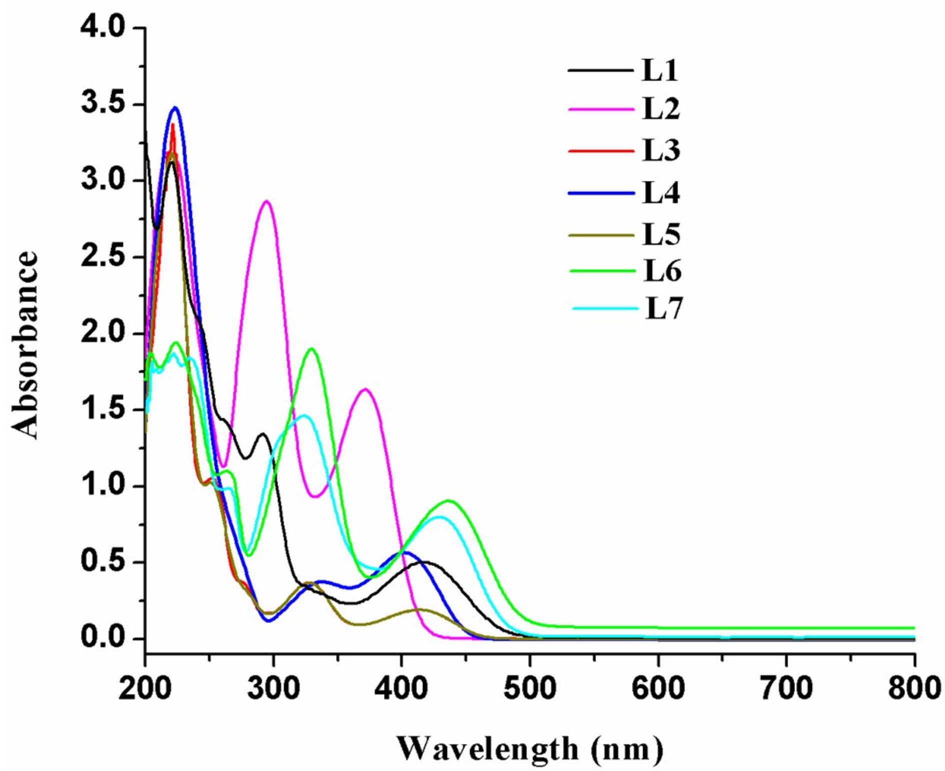


Figure S5. UV-Vis spectra of Schiff base ligands

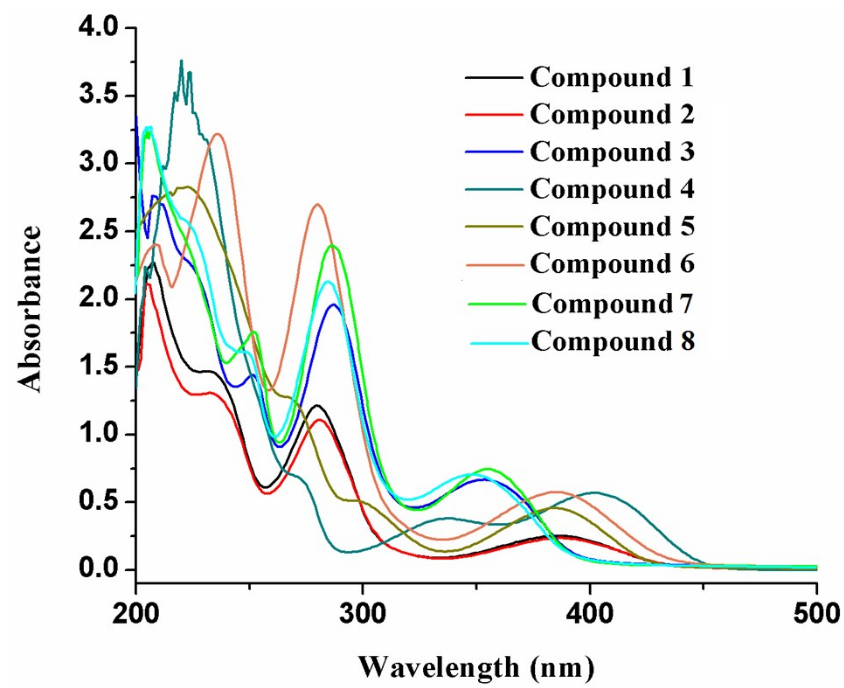


Figure S6. UV-Vis spectra of compounds **1-8** in the range 200-500 nm

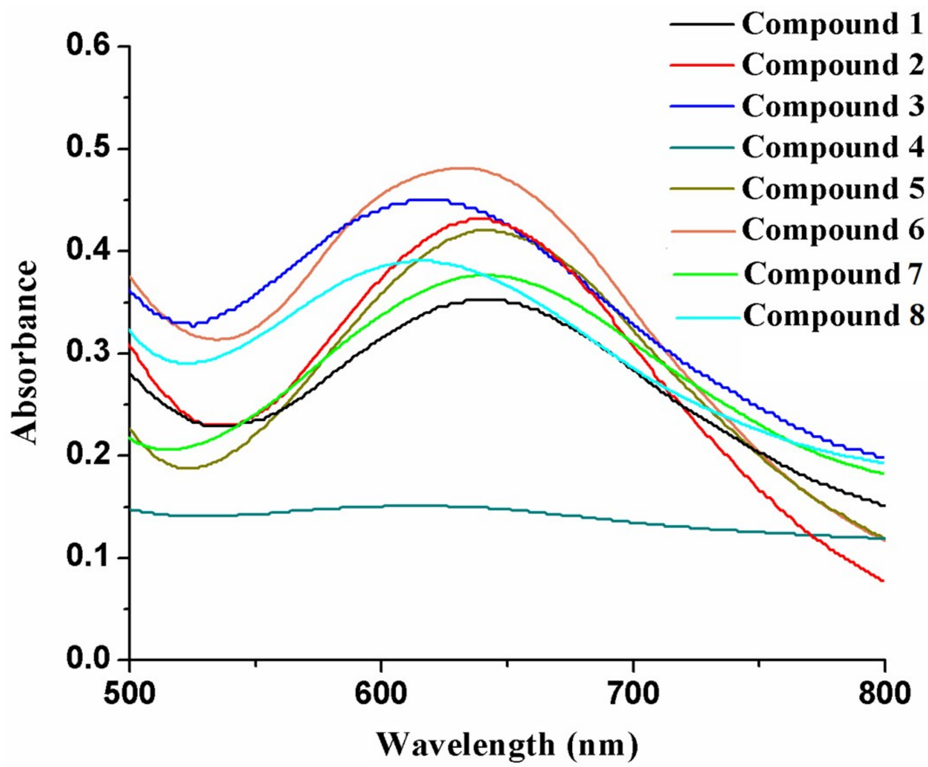


Figure S7. UV-Vis spectra of compounds **1**-**8** in the range 500-800 nm

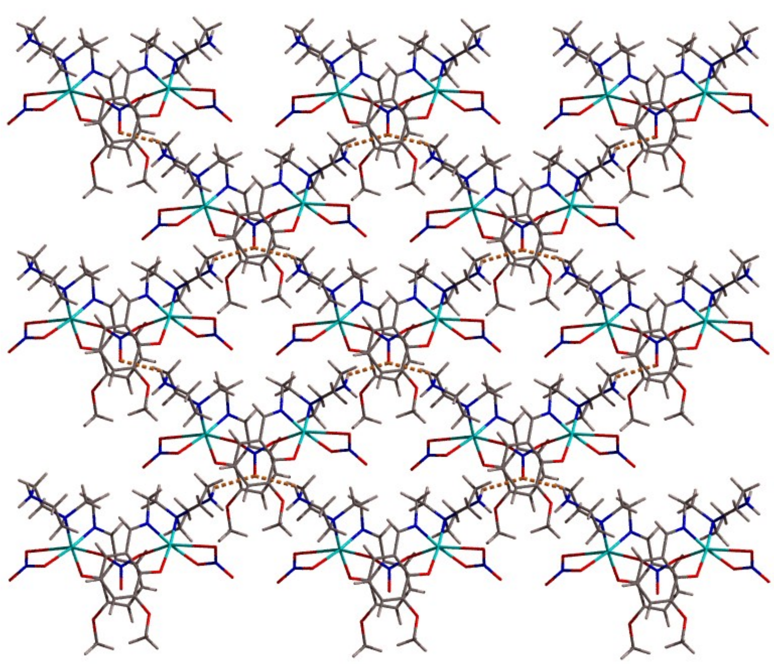


Figure S8. H-bonded structure of compound **1**

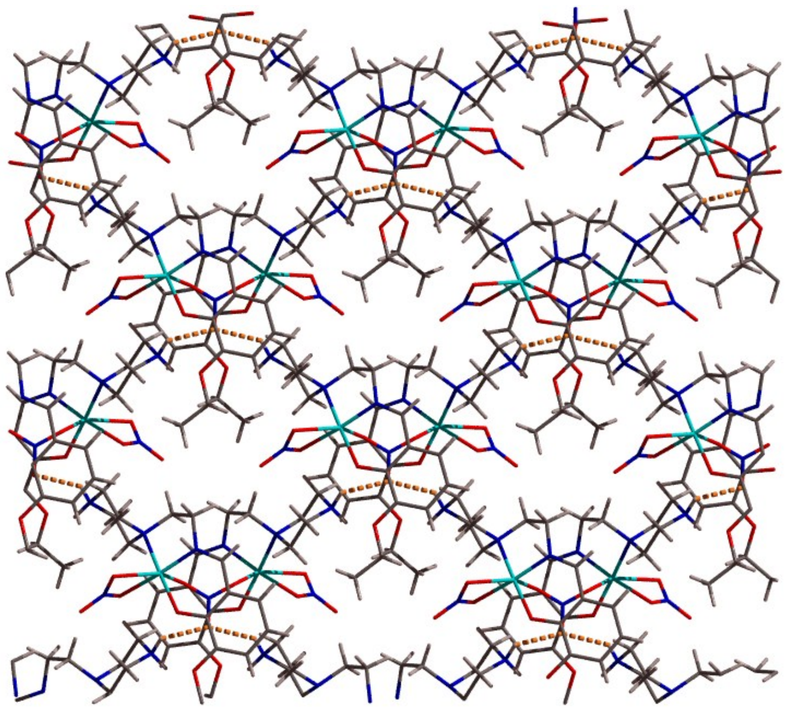


Figure S9. H-bonded 2D structure of compound **2**

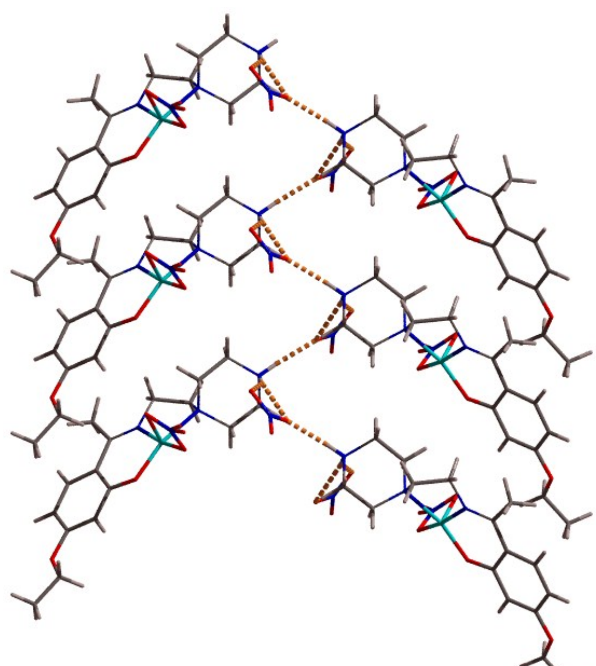


Figure S10. H-bonded 1D supramolecular structure of compound **3**

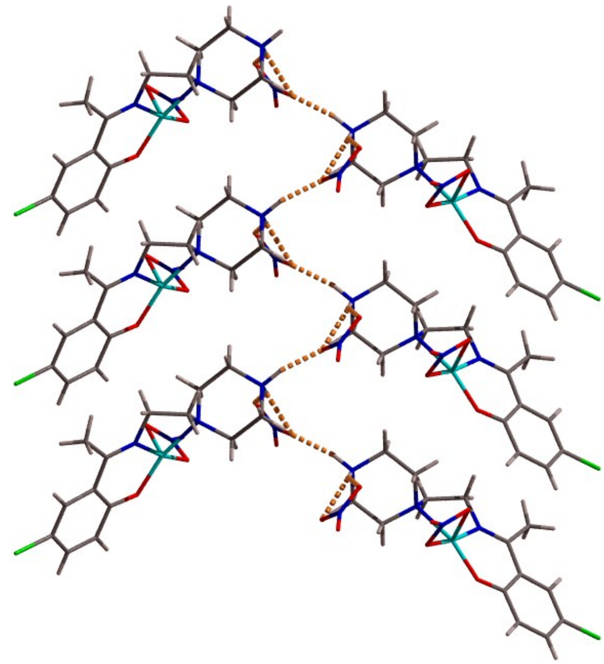


Figure S11. H-bonded 1D zigzag structure of compound **4**

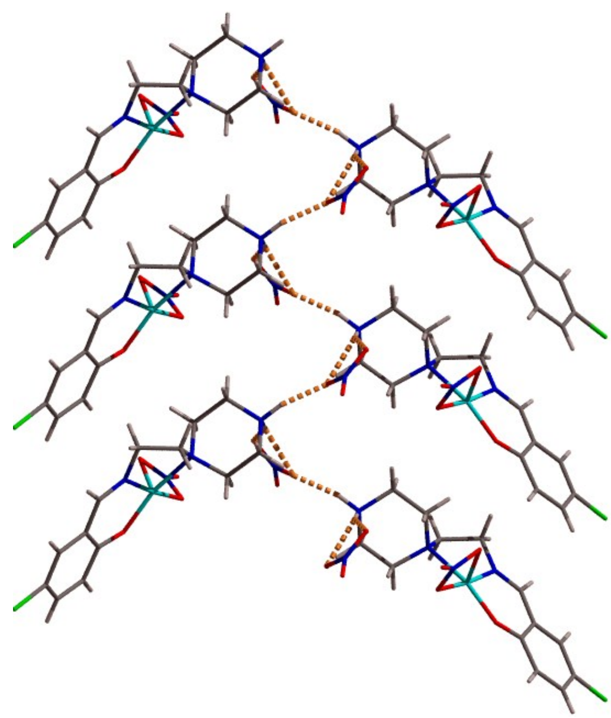


Figure S12. H-bonded 1D zigzag chain of compound **5**

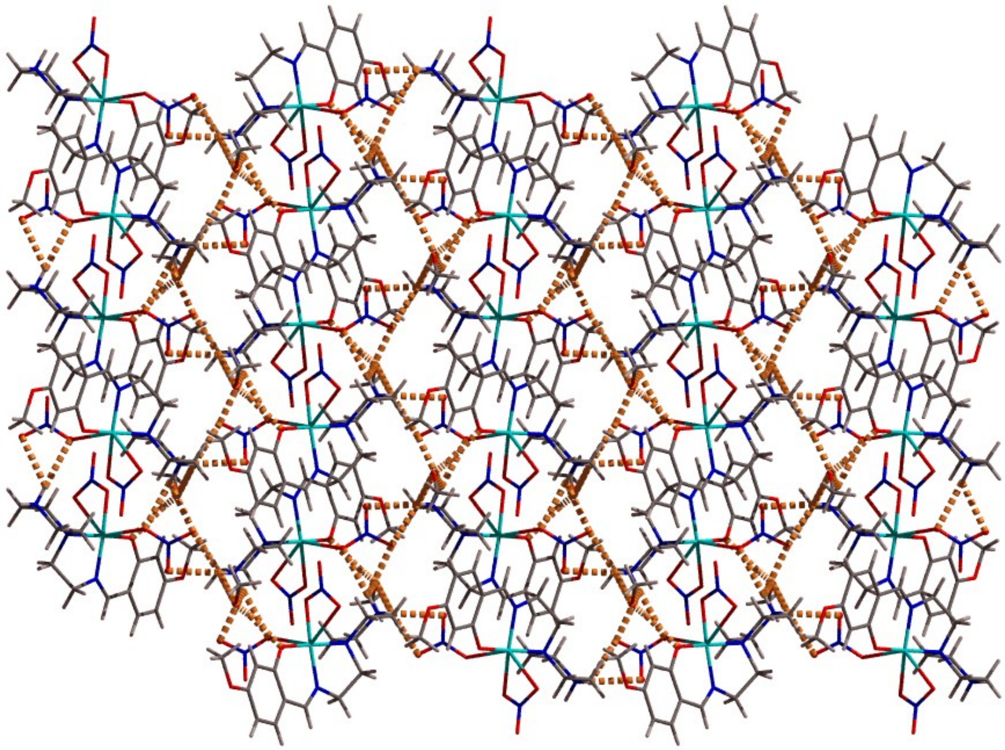


Figure S13. H-bonded 2D supramolecular structure of compound **7**

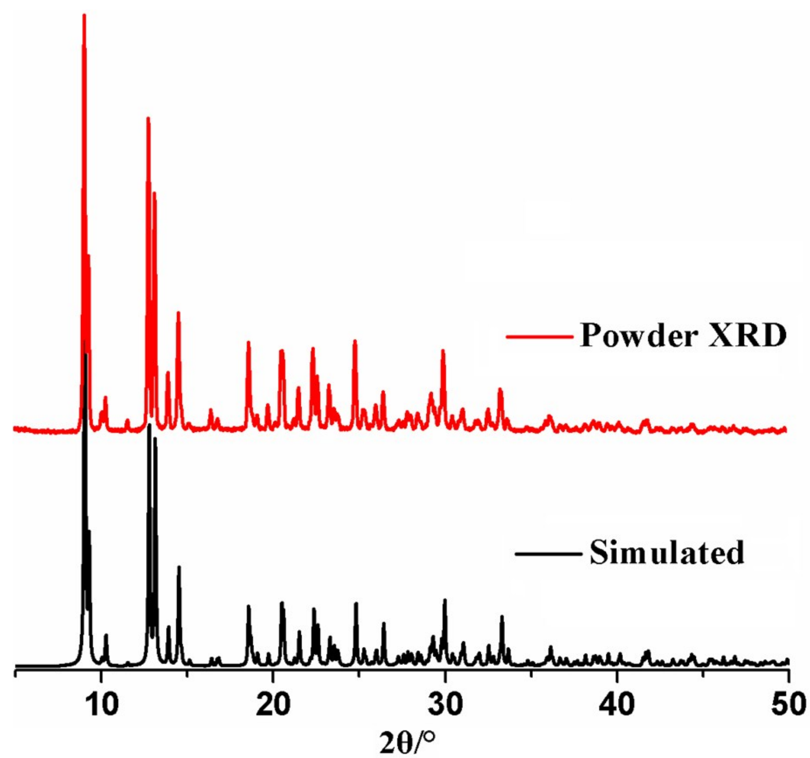


Figure S14. Powder-XRD patterns of **1**

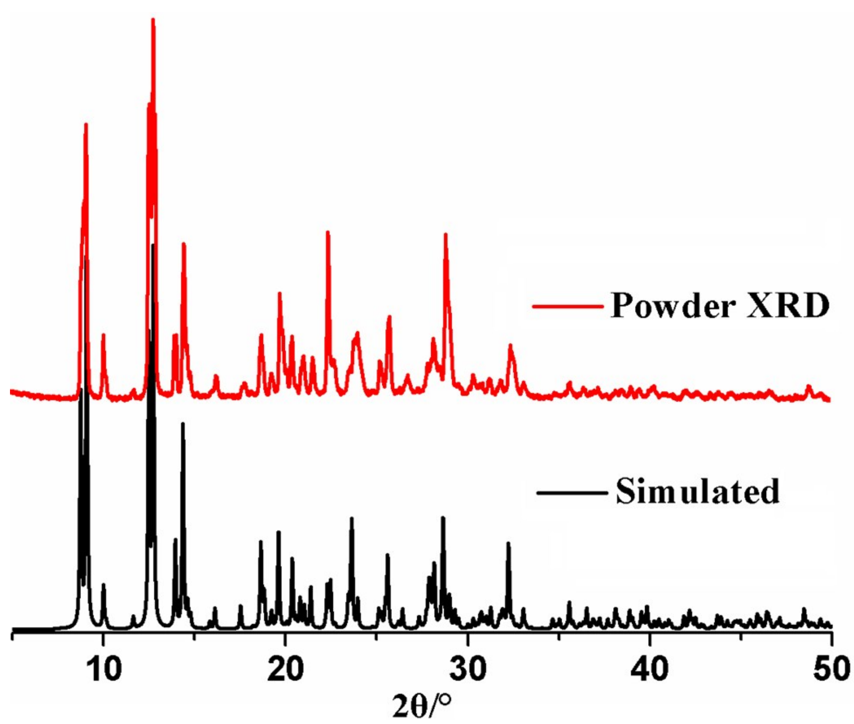


Figure S15. Powder-XRD patterns of **2**

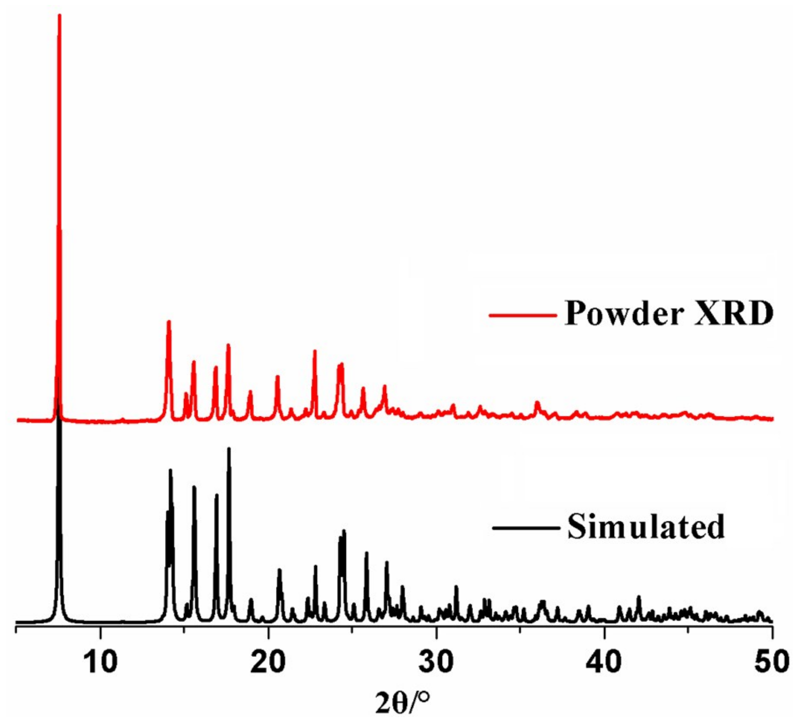


Figure S16. Powder-XRD patterns of **3**

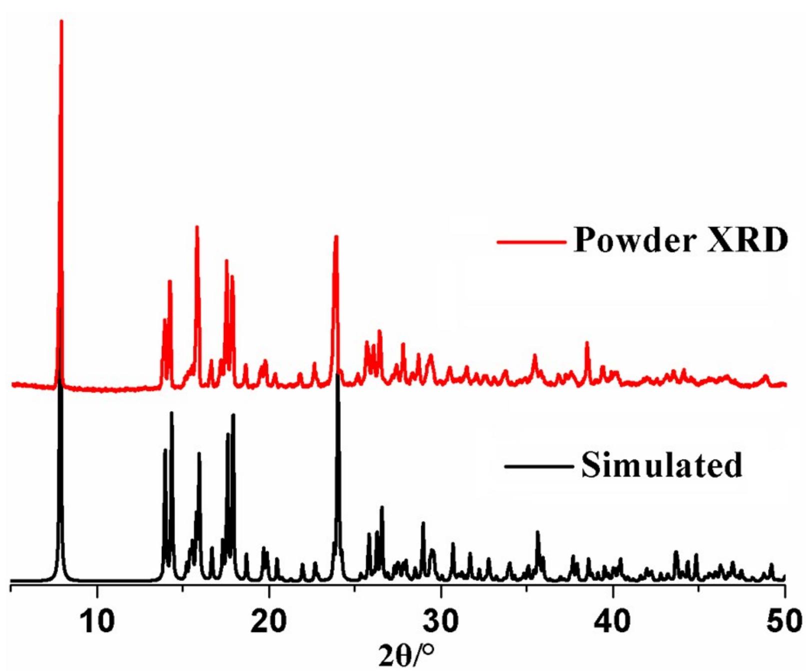


Figure S17. Powder-XRD patterns of **4**

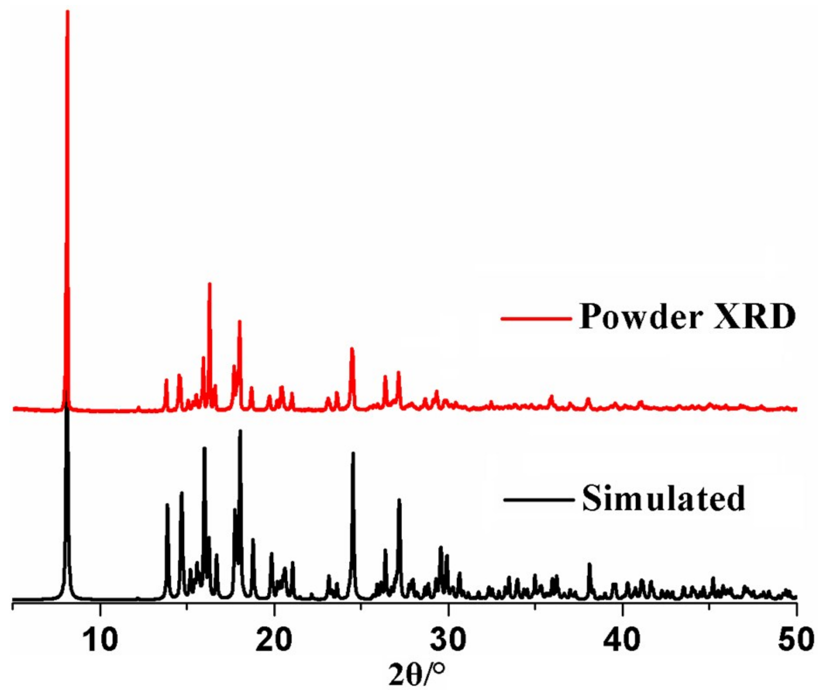


Figure S18. Powder-XRD patterns of **5**

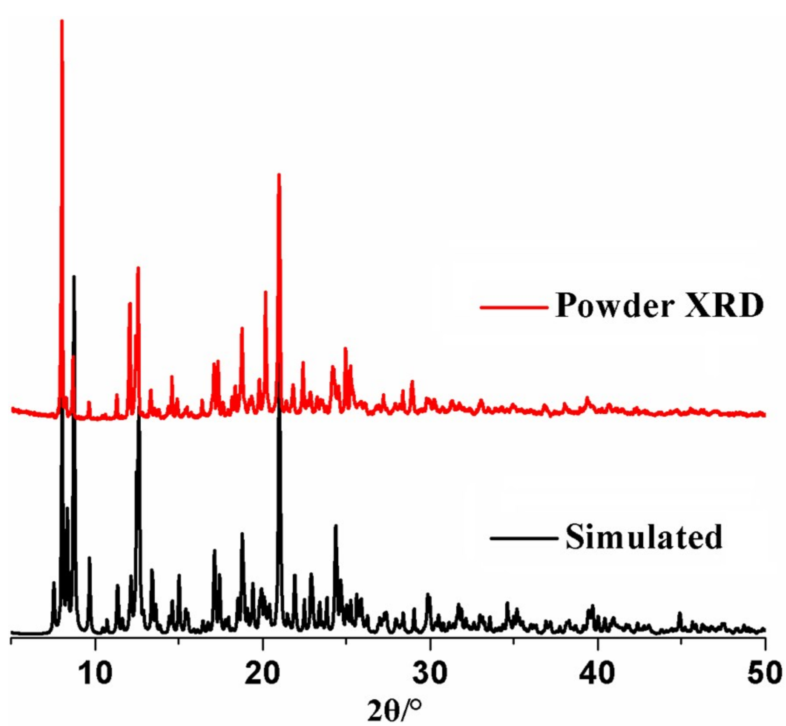


Figure S19. Powder-XRD patterns of **6**

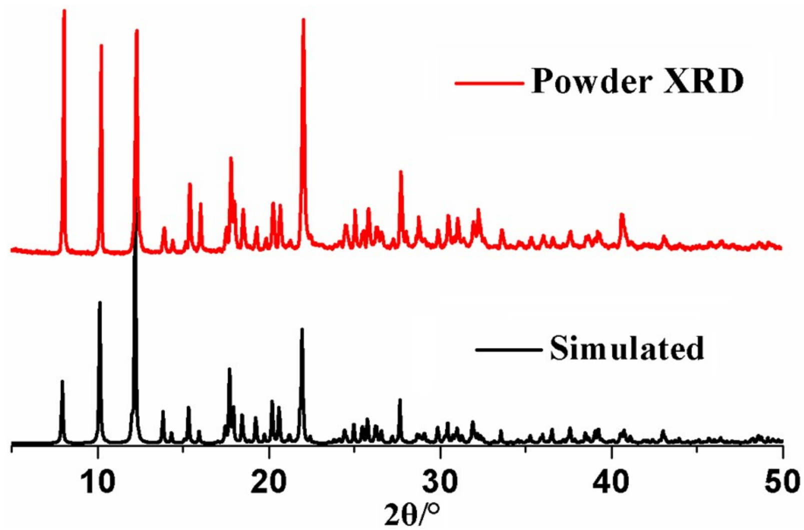


Figure S20. Powder-XRD patterns of **7**

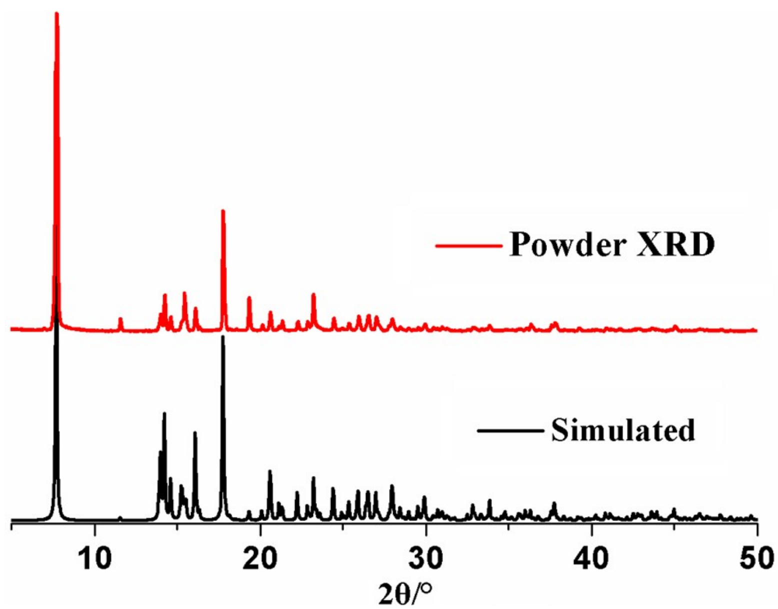
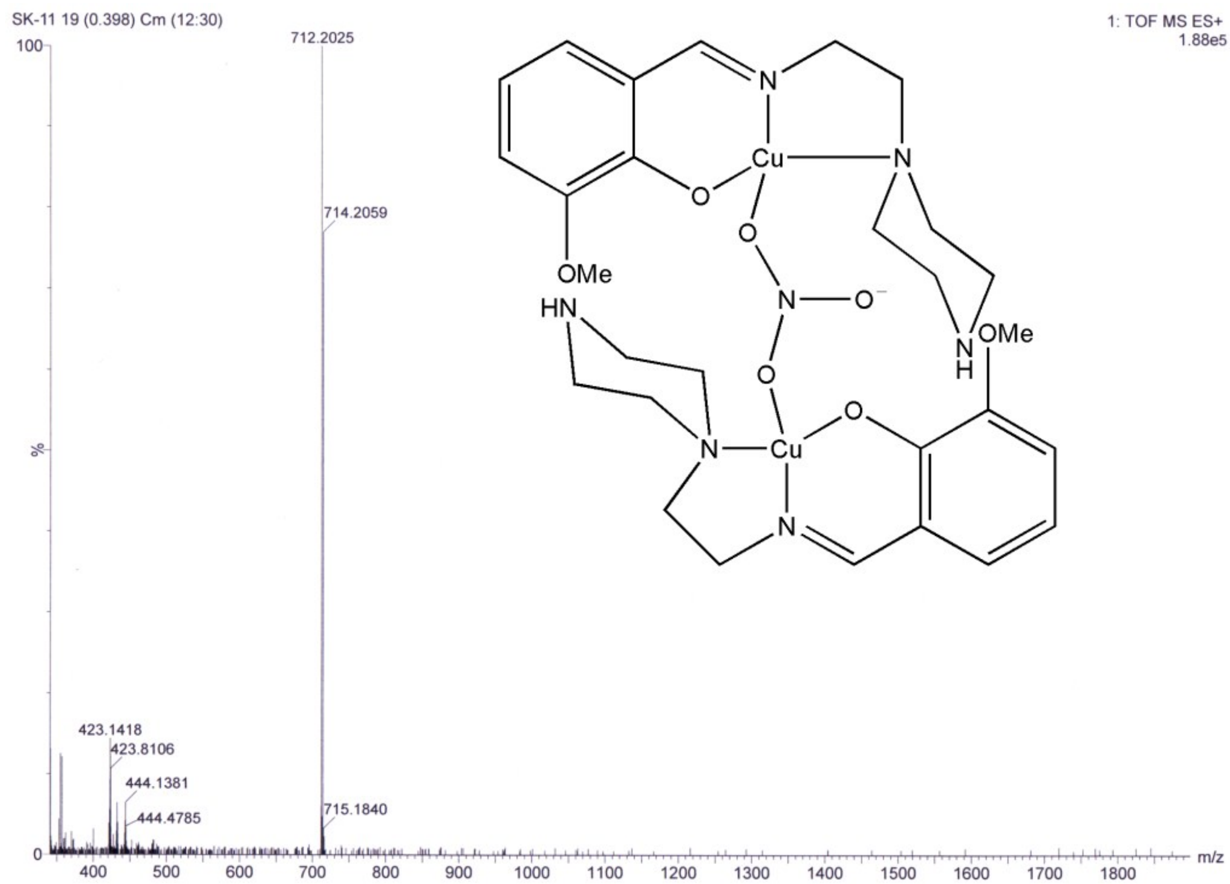


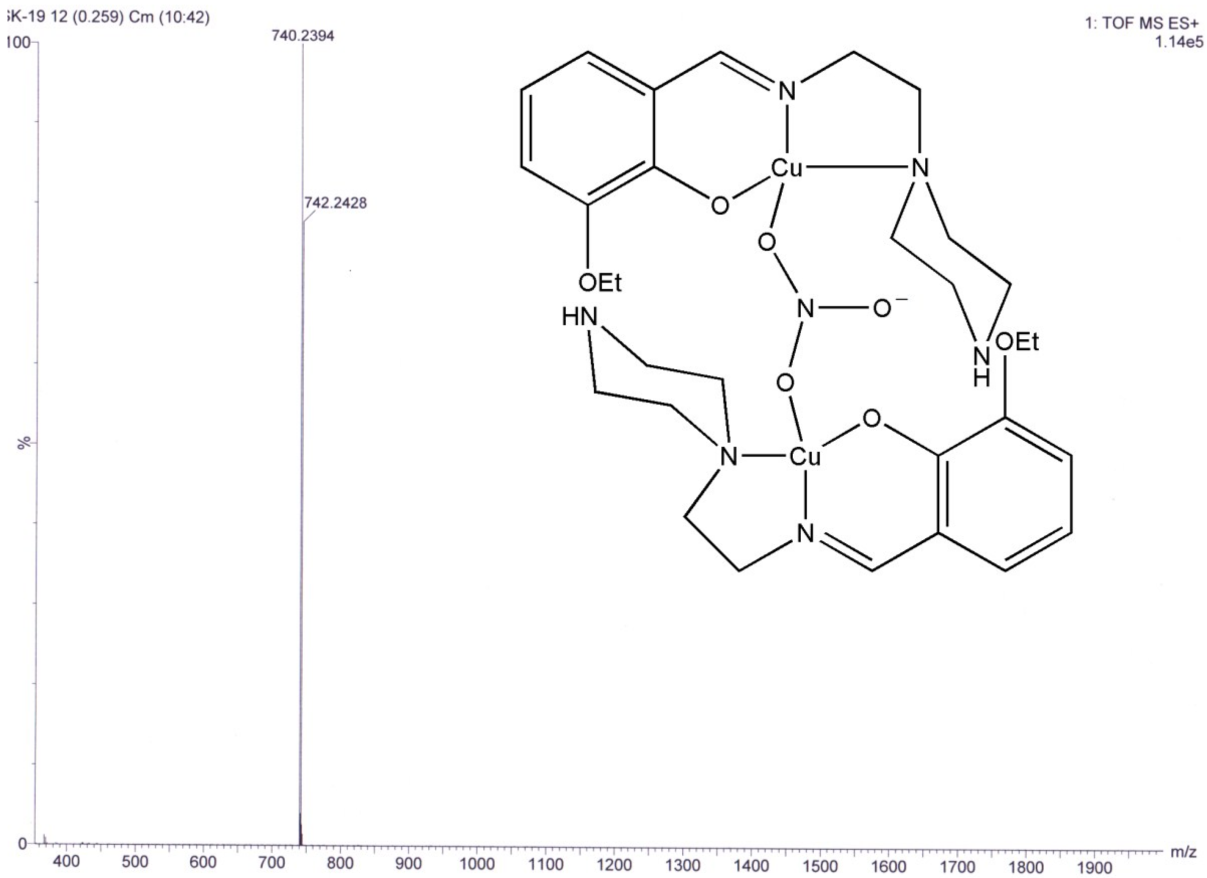
Figure S21. Powder-XRD patterns of **8**

Characterization of Compounds:

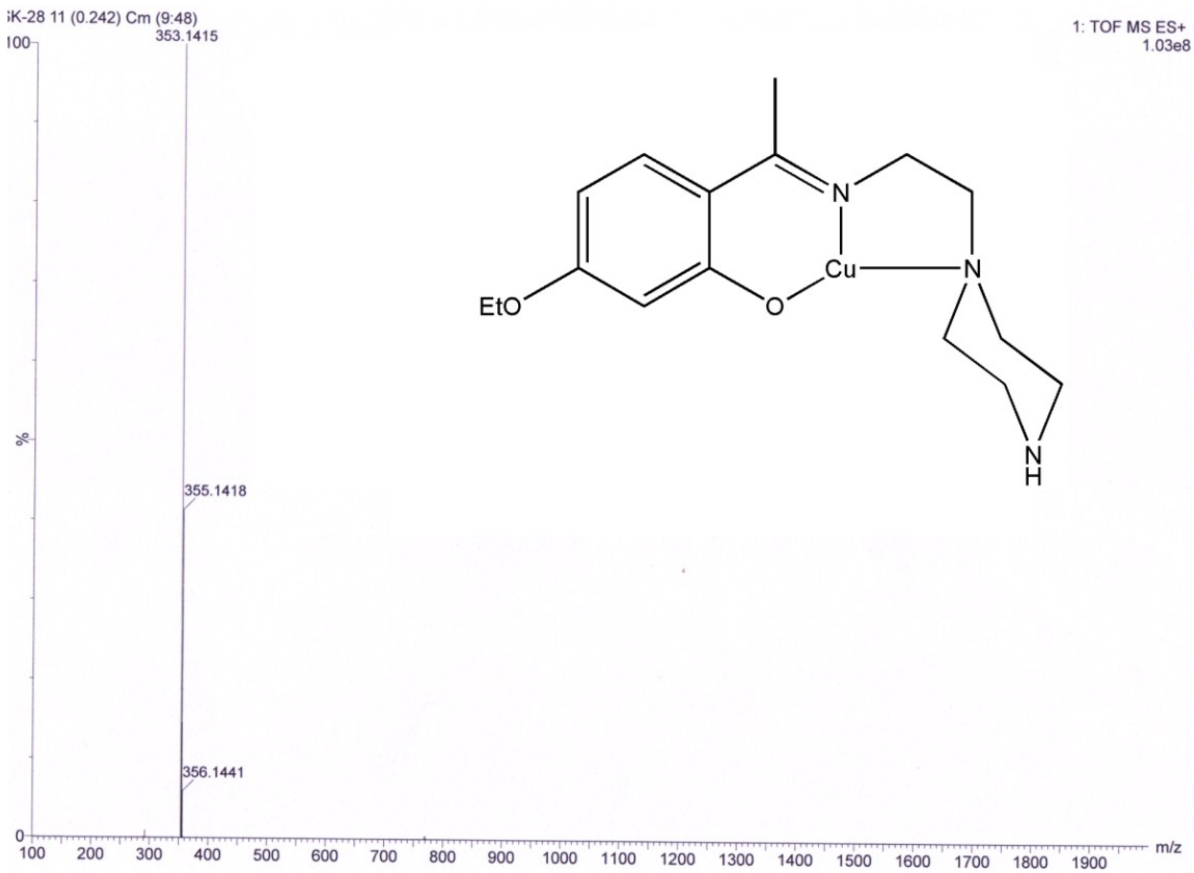
Compound **1;** HRMS in ethanol *m/z* 712.2025 [$(C_{28}H_{40}Cu_2N_7O_7)^+$, 100%].



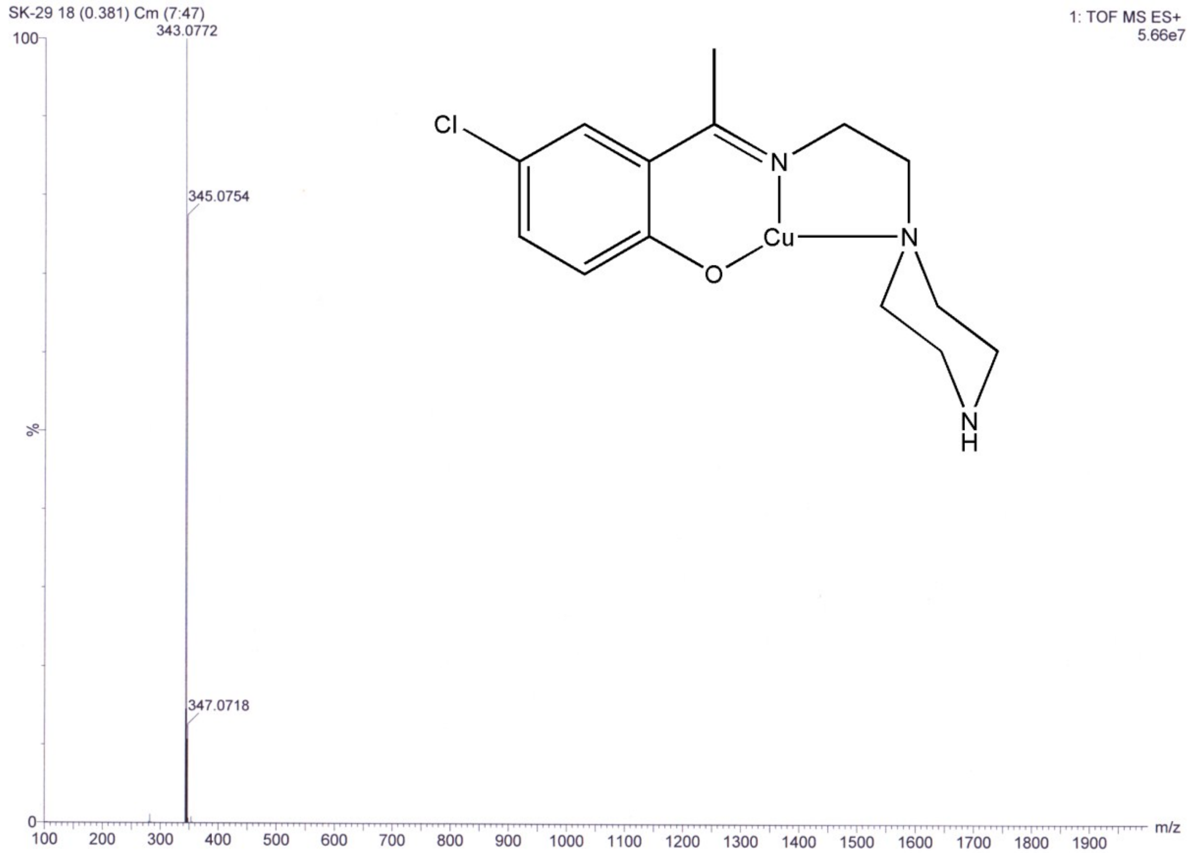
Compound **2;** HRMS in ethanol *m/z* 740.2394 [$(C_{30}H_{44}Cu_2N_7O_7)^+$, 100%].



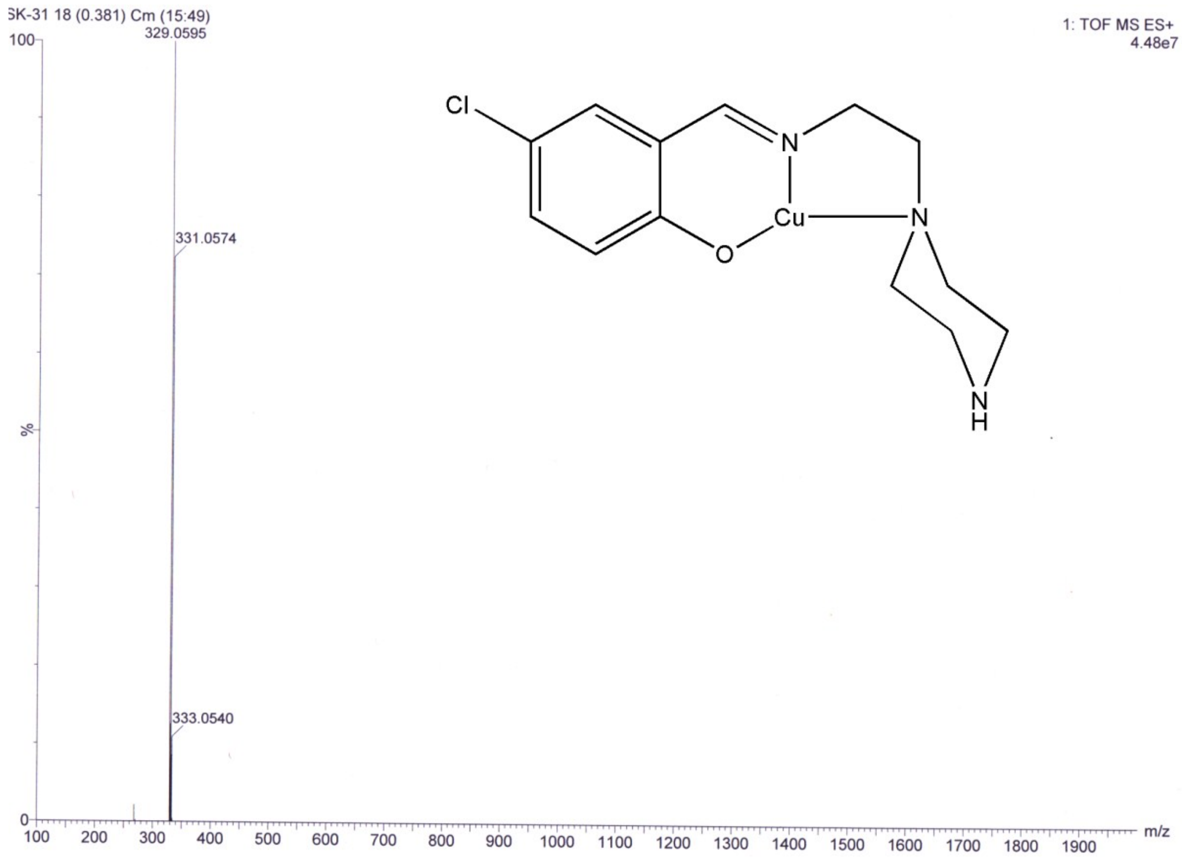
Compound **3;** HRMS in ethanol *m/z* 353.1415 [$(C_{16}H_{24}CuN_3O_2)^+$, 100%].



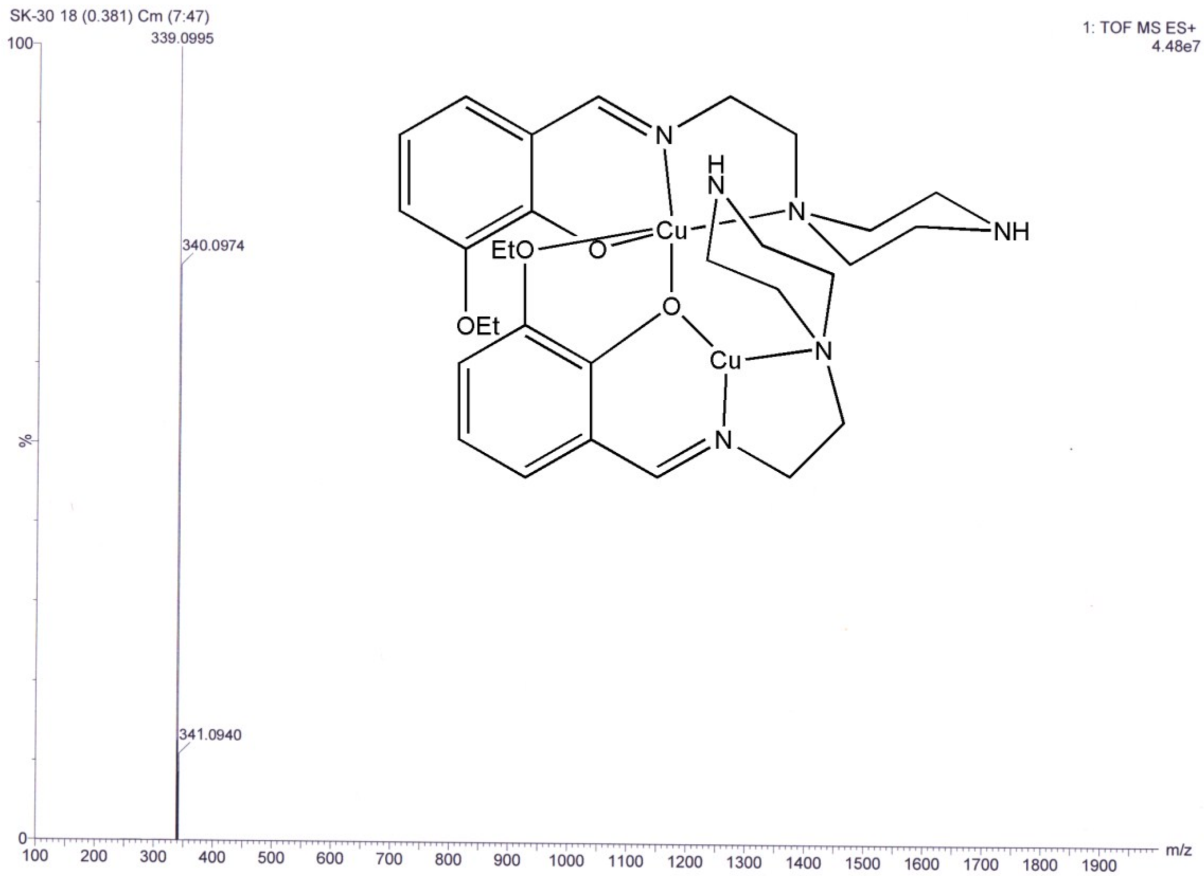
Compound **4;** HRMS in ethanol *m/z* 343.0772 [$(C_{14}H_{19}ClCuN_3O)^+$, 100%].



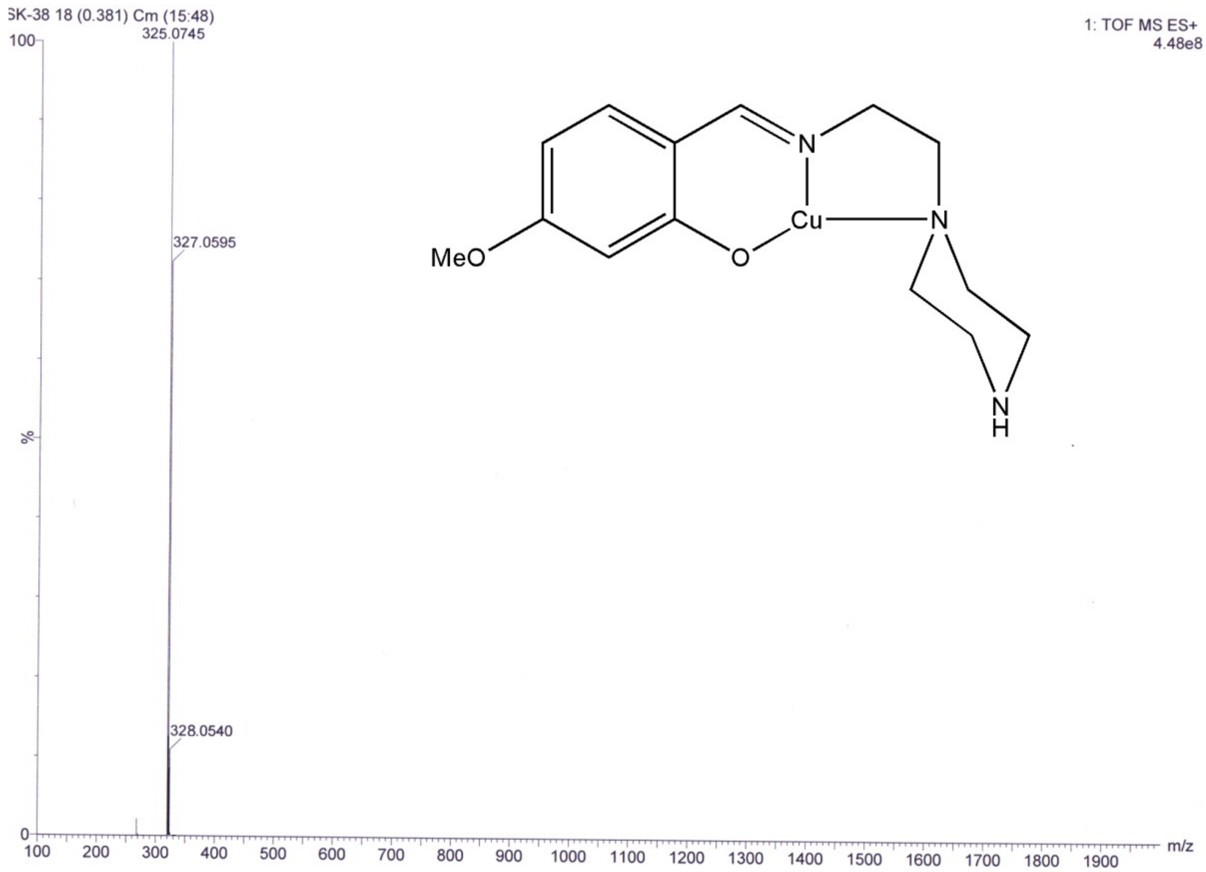
Compound **5;** HRMS in ethanol *m/z* 329.05995 [$(C_{13}H_{17}ClCuN_3O)^+$, 100%].



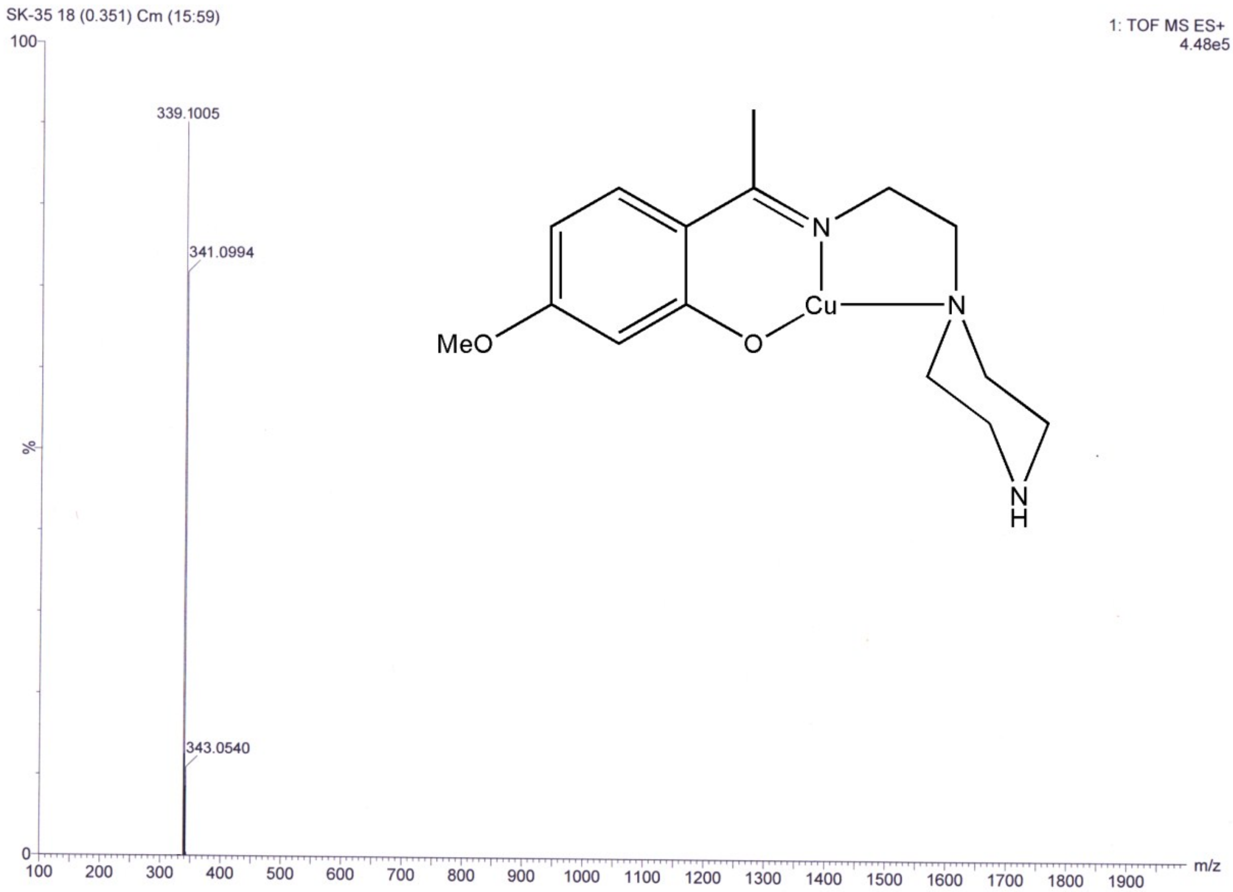
Compound **6;** HRMS in ethanol *m/z* 339.0995 [$(C_{30}H_{44}Cu_2N_6O_4)^{2+}$, 100%].



Compound **7;** HRMS in ethanol *m/z* 325.0745 [$(C_{14}H_{20}CuN_3O_2)^+$, 100%].

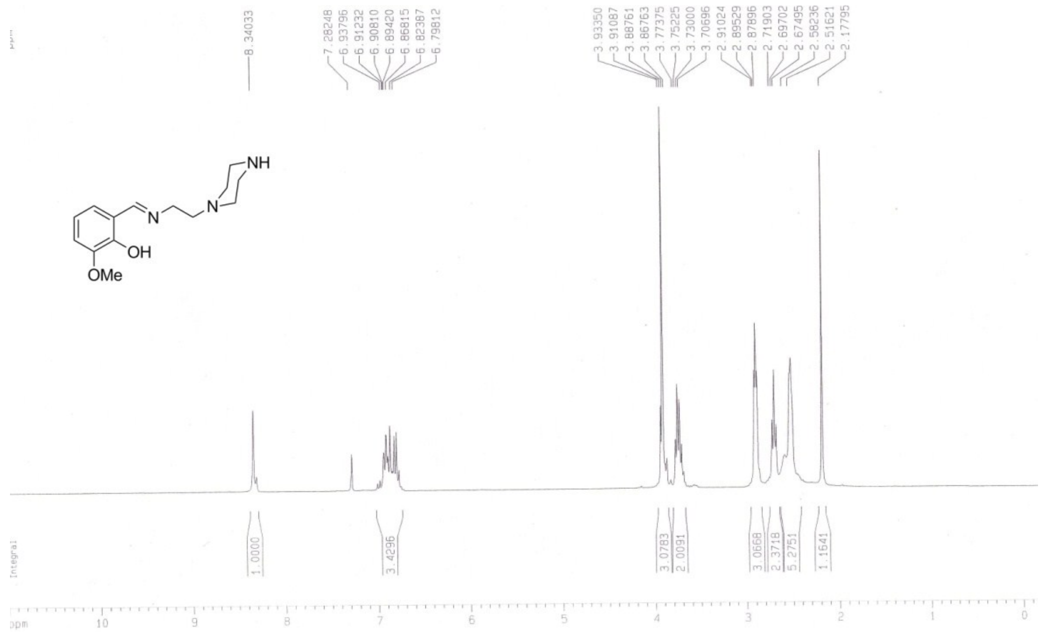


Compound **8;** HRMS in ethanol *m/z* 339.1005 [$(C_{15}H_{22}CuN_3O_2)^+$, 90%].



Characterization of Ligands:

L1; δ_H (300 MHz; $CDCl_3$) 2.18 (1H, s), 2.52-2.58 (5H, m), 2.69 (2H, t, *J* = 6.6 Hz), 2.89 (3H, t, *J* = 4.5 Hz), 3.71-3.77 (2H, m), 3.87-3.93 (3H, m), 6.79-6.94 (3H, m), 8.34 (1H, s); δ_C (75 MHz; $CDCl_3$) 46.09, 54.74, 56.07, 56.39, 59.22, 113.89, 117.66, 118.48, 122.86, 148.61, 152.43, 165.53; *m/z* 264.2008 [$(M+H)^+$, 100%]; Yield ca. 97% based on amine; Elemental analysis (found: C 63.51, H 8.02, N 15.93; for $C_{14}H_{21}N_3O_2$ requires: C 63.58, H 8.04, N 15.96%).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7.28248
6.93796
6.91232
6.90810
6.89420
6.86815
6.82387
6.79812
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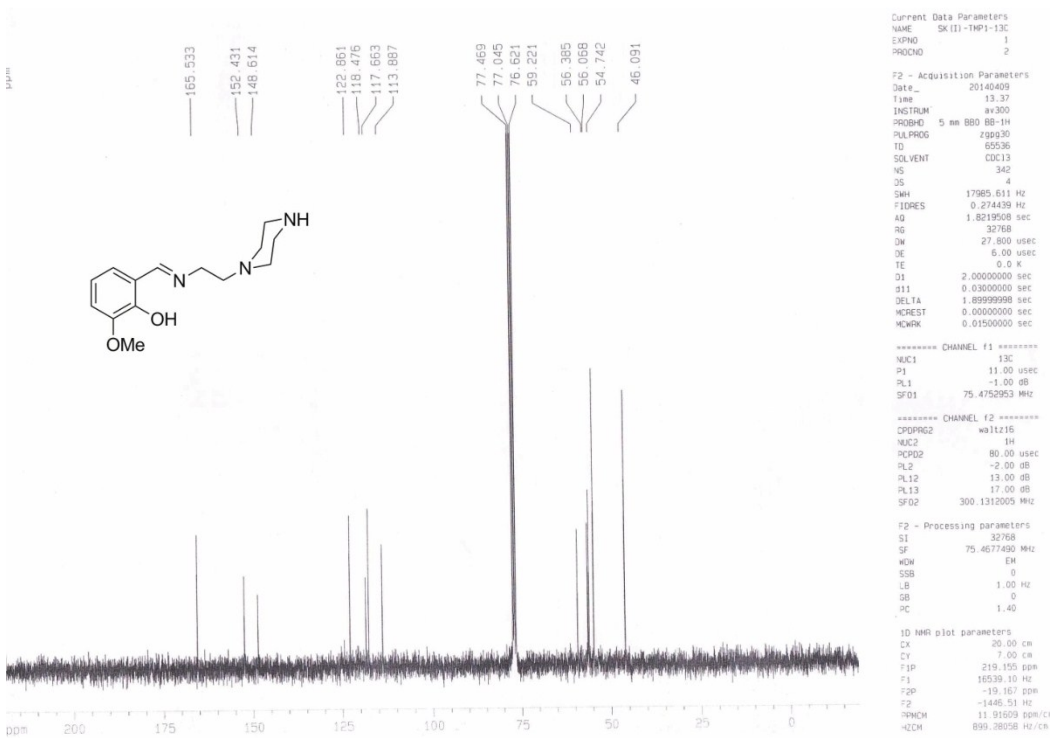
3.91087
3.88761
3.86763
3.77375
3.75225
3.73000
3.70696
2.91024
2.89529
2.87896
2.71903
2.69702
2.67495
2.58236
2.51621
2.17795
NH
N
N
OH
OMe
Integral
1.0000
3.4296
3.0783
2.0091
3.0668
2.3718
5.2751
1.1641
ppm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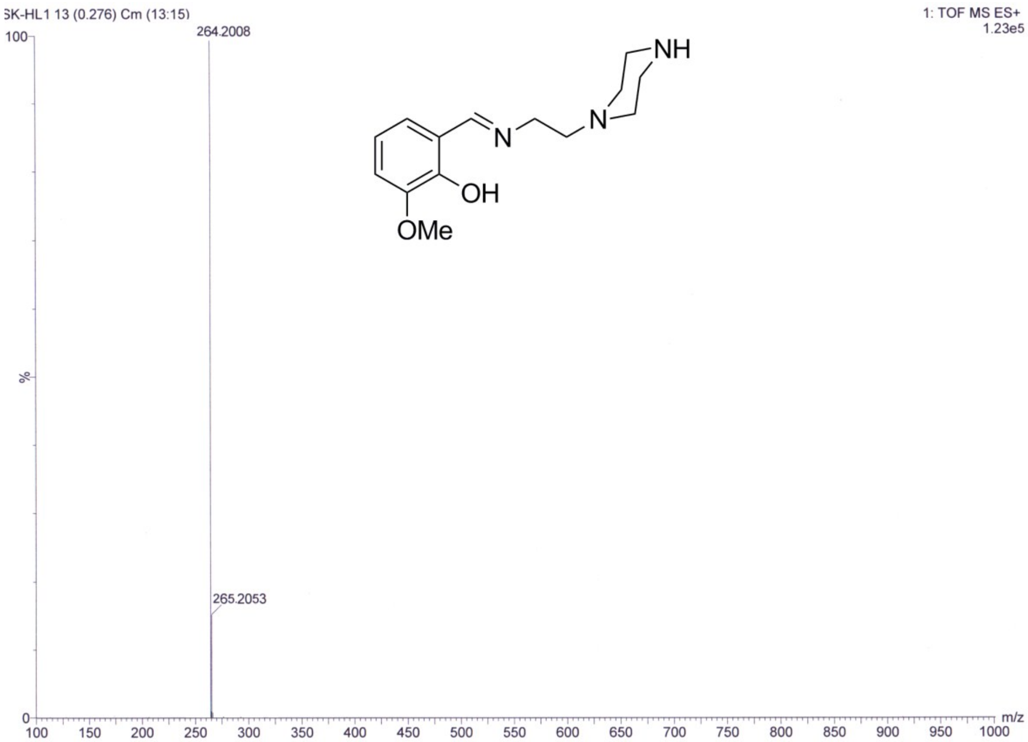
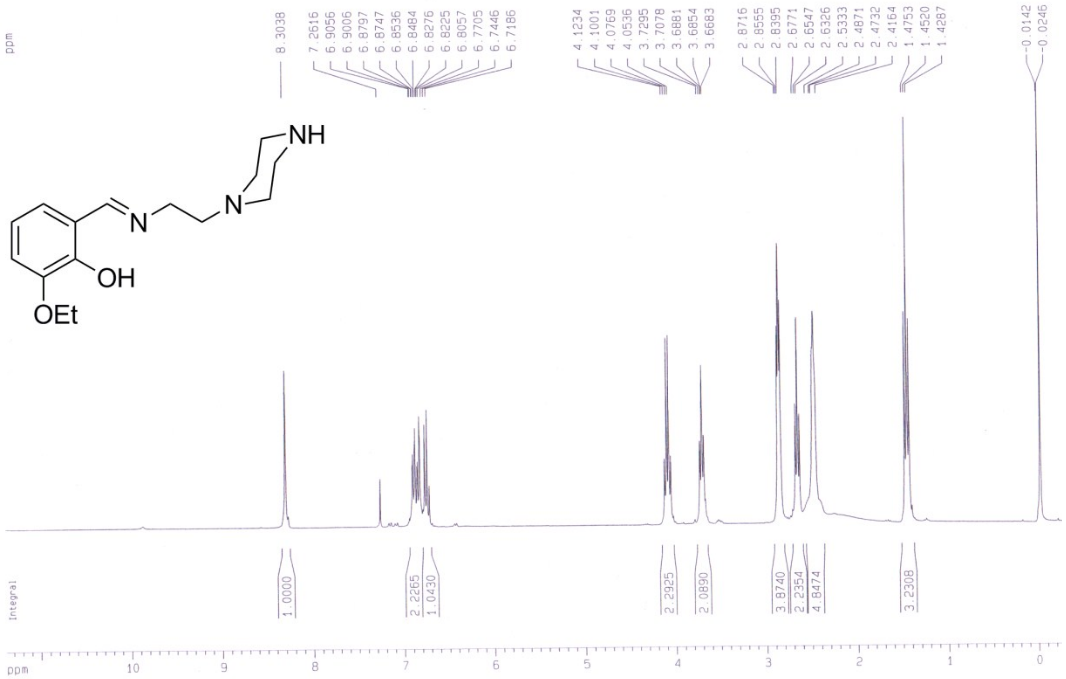
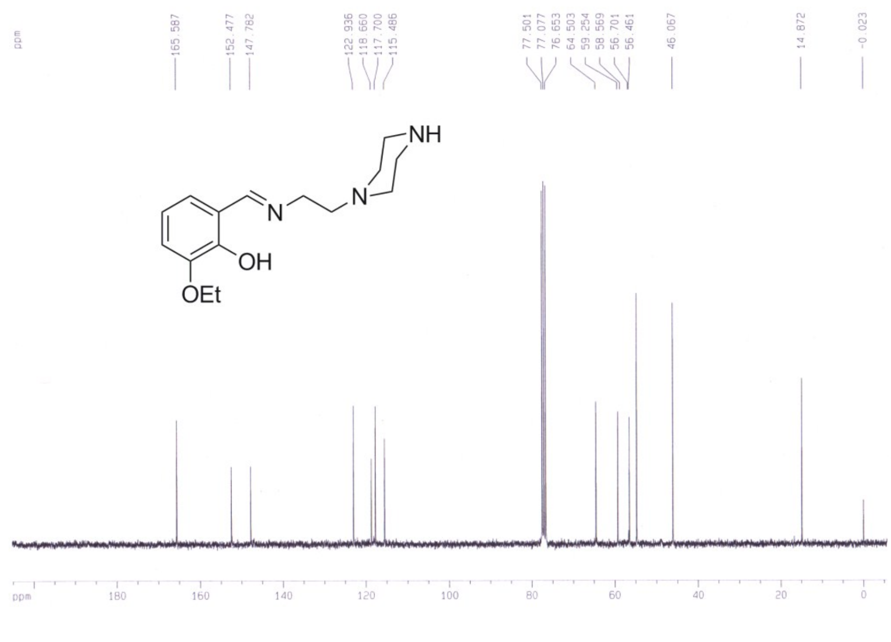
152.431
148.614
122.861
118.476
117.663
113.887
77.469
77.045
76.621
59.221
56.385
56.068
54.742
46.091
NH
N
N
OH
OMe
ppm
Current Data Parameters
NAME SK(I)-TMP1-13C
EXPNO 1
PROCNO 2
F2 - Acquisition Parameters
Date_ 20140409
Time 13.37
INSTRUM av300
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 342
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 32768
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 11.00 usec
PL1 -1.00 dB
SFO1 75.4752953 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 20.00 cm
CY 7.00 cm
F1P 219.155 ppm
F1 16539.10 Hz
F2P -19.167 ppm
F2 -1446.51 Hz
PPMCM 11.91609 ppm/cm
HZCM 899.28058 Hz/cm



L2; δ_H (300 MHz; $CDCl_3$, TMS) 1.45 (3H, t, J = 6.9 Hz), 2.42-2.53 (5H, m), 2.65 (2H, t, J = 6.6 Hz), 2.86 (4H, t, J = 4.8 Hz), 3.67-3.73 (2H, m), 4.05-4.12 (2H, m), 6.72-6.90 (3H, m), 8.30 (1H, s); δ_C (75 MHz; $CDCl_3$, TMS) 14.87, 46.07, 56.46, 56.70, 58.57, 59.25, 64.50, 115.49, 117.70, 118.66, 122.94, 147.78, 152.48, 165.59; *m/z* 278.1848 [$(M+H)^+$, 100%]; Yield ca. 97% based on amine; Elemental analysis (found: C 64.98, H 8.38, N 15.19; for $C_{15}H_{23}N_3O_2$ requires: C 64.95, H 8.36, N, 15.15%).





Current Data Parameters
NAME SK(I)-TMHL2-13C
EXPNO 1
PROCNO 2

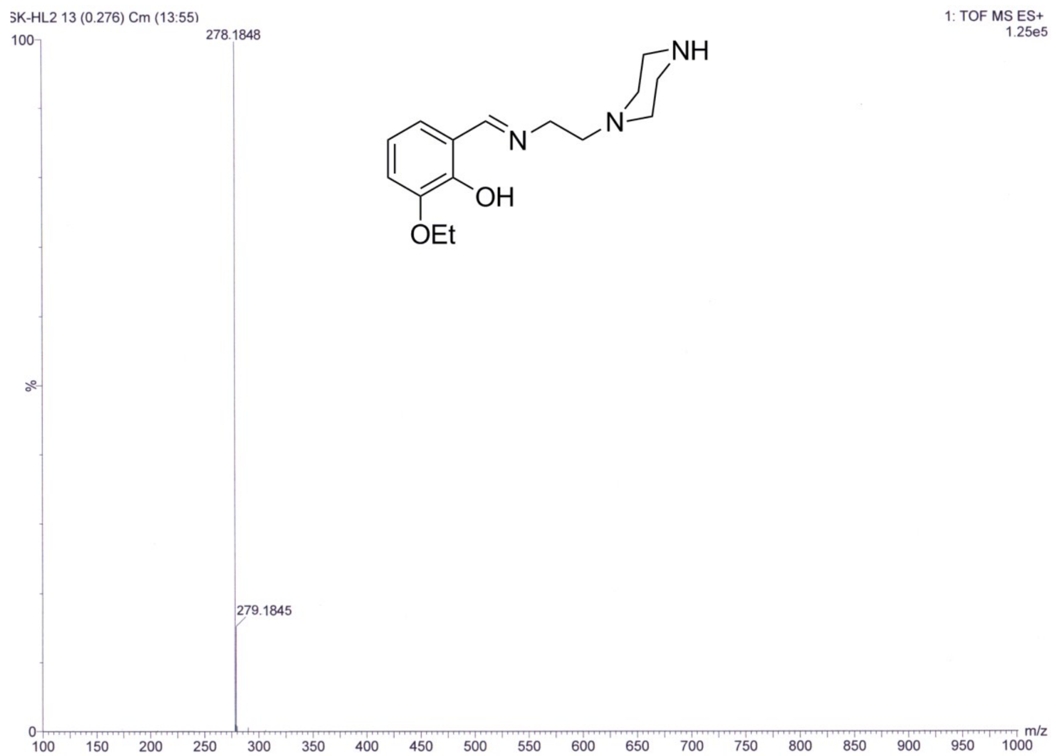
F2 - Acquisition Parameters
Date_ 20140825
Time 14.43
INSTRUM av300
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 577
DS 4
SWH 16556.291 Hz
FIDRES 0.252629 Hz
AQ 1.9792372 sec
RG 18390.4
DW 30.200 usec
DE 6.00 usec
TE 0.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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

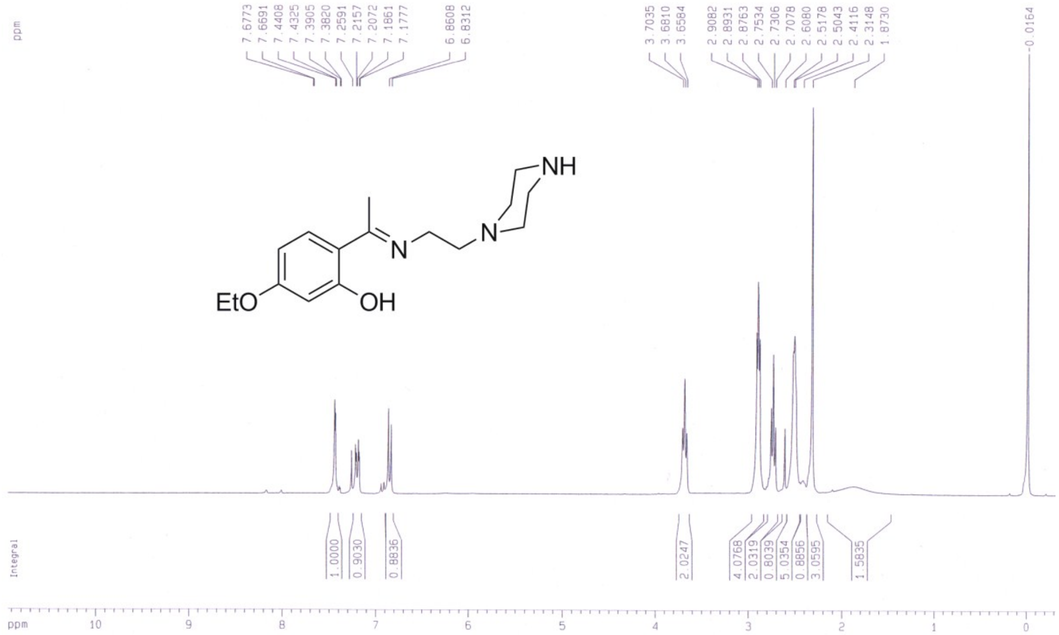
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 13.00 dB
PL13 17.00 dB
SFO2 300.1312005 MHz

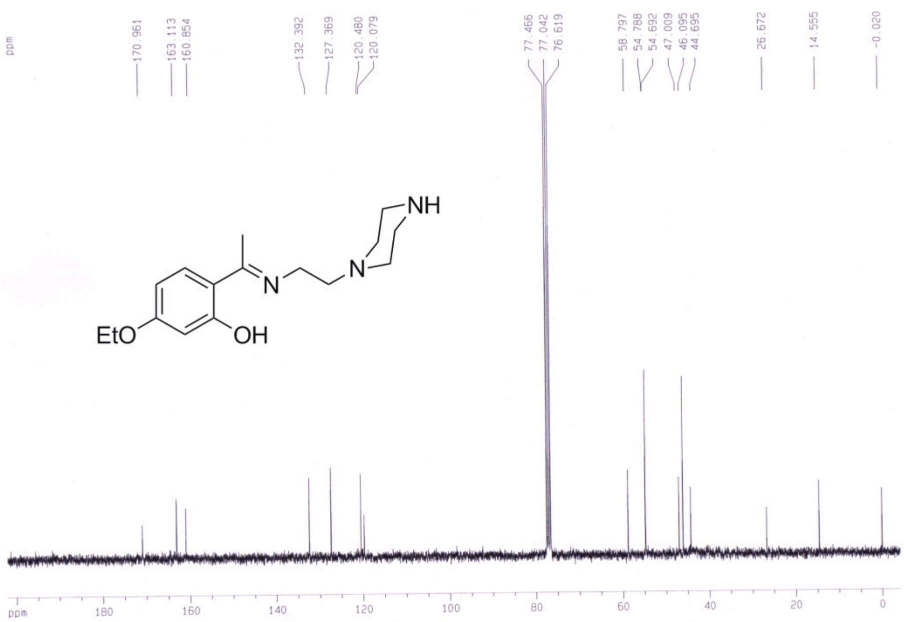
F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 24.00 cm
CY 7.00 cm
F1P 205.264 ppm
F1 15490.79 Hz
F2P -5.706 ppm
F2 -430.65 Hz
PPMCM 8.79042 ppm/cm
HZCM 663.39325 Hz/cm



L3; δ_H (300 MHz; $CDCl_3$, TMS) 1.87 (2H, br, s), 2.31 (3H, s), 2.41 (1H, s), 2.51 (5H, d, *J* = 4.1 Hz), 2.61 (1H, s), 2.73 (2H, t, *J* = 6.8 Hz), 2.89 (4H, t, *J* = 5 Hz), 3.68 (2H, t, *J* = 6.8 Hz), 6.85 (1H, d, *J* = 8.9 Hz), 7.38-7.44 (1H, m), 7.67 (1H, d, *J* = 2.5 Hz); δ_C (75 MHz; $CDCl_3$, TMS) 14.56, 26.67, 44.69, 46.09, 47.01, 54.69, 54.79, 58.79, 115.49, 120.08, 120.49, 127.37, 132.39, 160.85, 163.11, 170.96; *m/z* 292.2013 [$(M+H)^+$, 100%]; Yield ca. 95% based on amine; Elemental analysis (found: C 65.90, H 8.63, N 14.39; for $C_{16}H_{25}N_3O_2$ requires: C 65.95, H 8.65, N, 14.42%).



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Current Data Parameters
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EXPNO                   1
PROCNO                  2

F2 - Acquisition Parameters
Date_            20140826
Time                13.46
INSTRUM             av300
PROBHD   5 mm BBO BB-1H
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   836
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                   5.00 cm
F1P               202.164 ppm
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F2P                -4.157 ppm
F2                -313.70 Hz
PPMCM             8.59672 ppm/cm
HZCM            648.77496 Hz/cm
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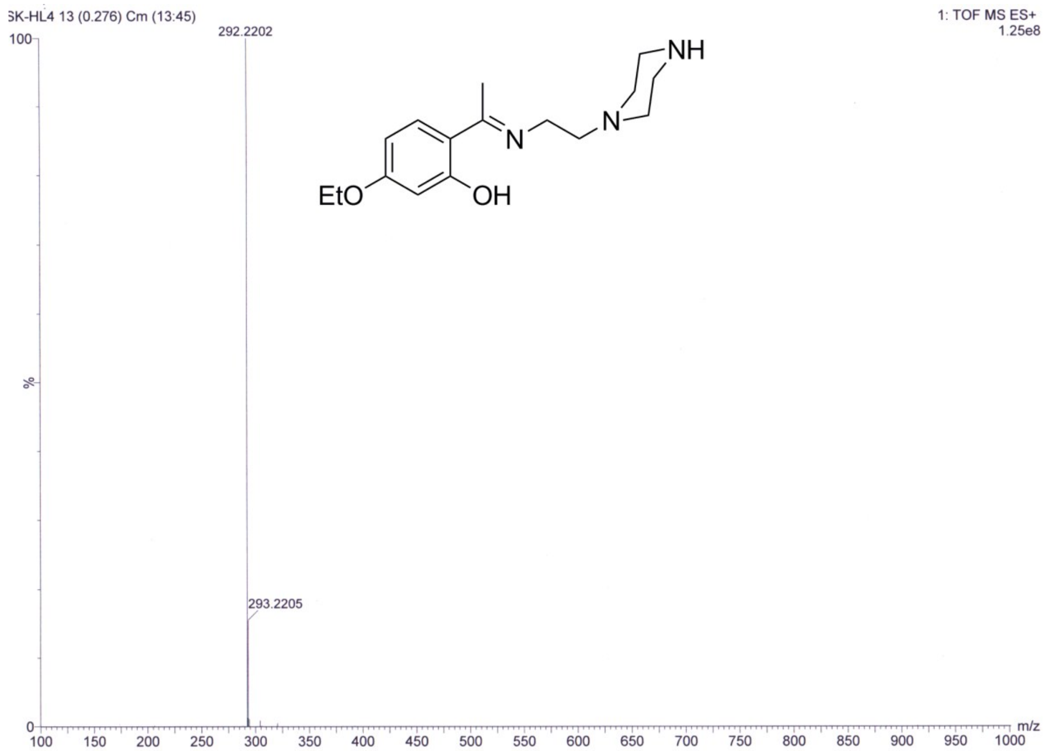
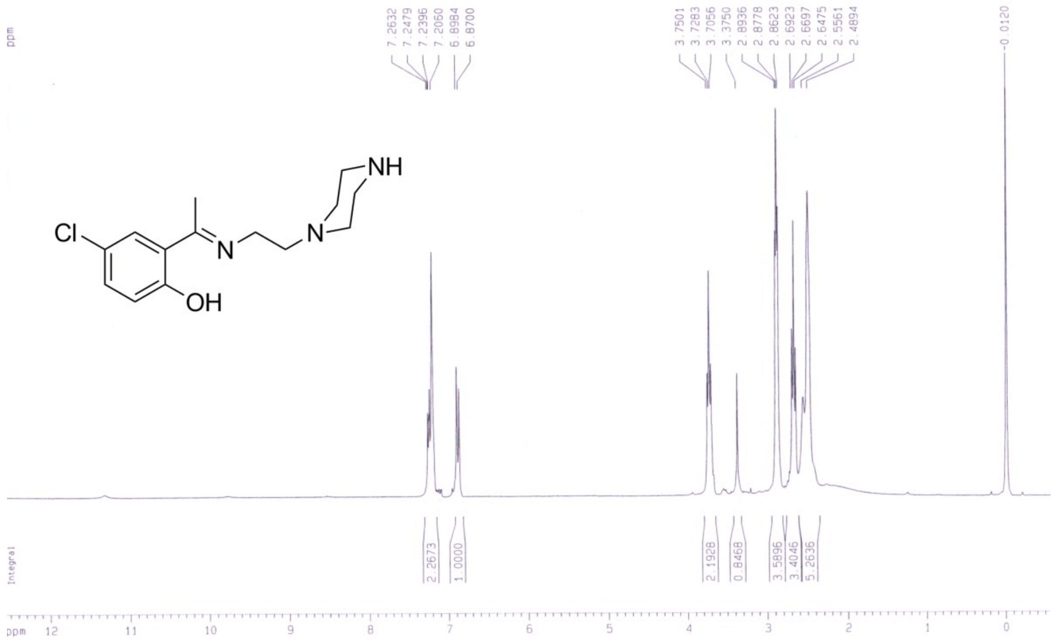


L4; δ_H (300 MHz; $CDCl_3$, TMS) 2.49-2.56 (5H, m), 2.67 (3H, t, J = 6.7 Hz), 2.88 (4H, t, J = 4.7 Hz), 3.38 (1H, s), 3.73 (2H, t, J = 8.2 Hz), 6.88 (1H, d, J = 8.5 Hz), 7.21-7.26 (2H, m); δ_C (75 MHz; $CDCl_3$, TMS) 19.06, 46.06, 53.41, 54.69, 56.70, 59.08, 118.65, 119.51, 123.03, 130.31, 132.05, 159.80, 159.94, 164.43; *m/z* 282.1319 [$(M+H)^+$, 100%]; Yield ca. 94% based on amine; Elemental analysis (found: C 59.64, H 7.15, N 14.88; for $C_{14}H_{20}ClN_3O_1$ requires: C 59.67, H 7.15, N, 14.91%).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7.2060
6.8984
6.8700
3.7501
3.7283
3.7056
3.3750
2.8936
2.8778
2.8623
2.6923
2.6697
2.6475
2.5561
2.4894
-0.0120
NH
Cl
N
N
OH
Integral
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2.1928
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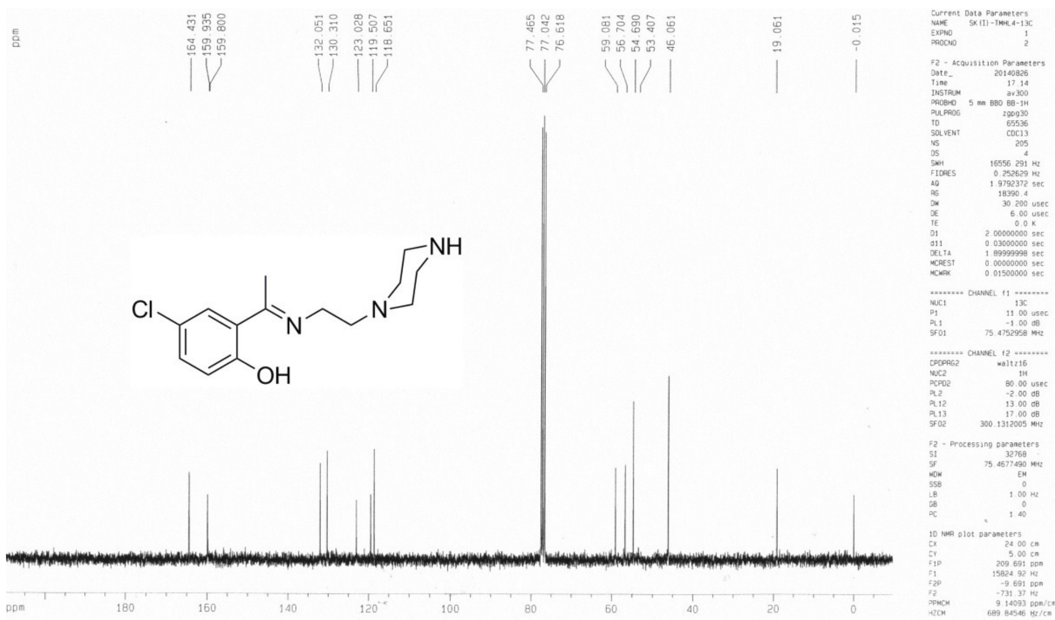
5.2636
ppm 12 11 10 9 8 7 6 5 4 3 2 1 0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130.310
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119.507
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77.042
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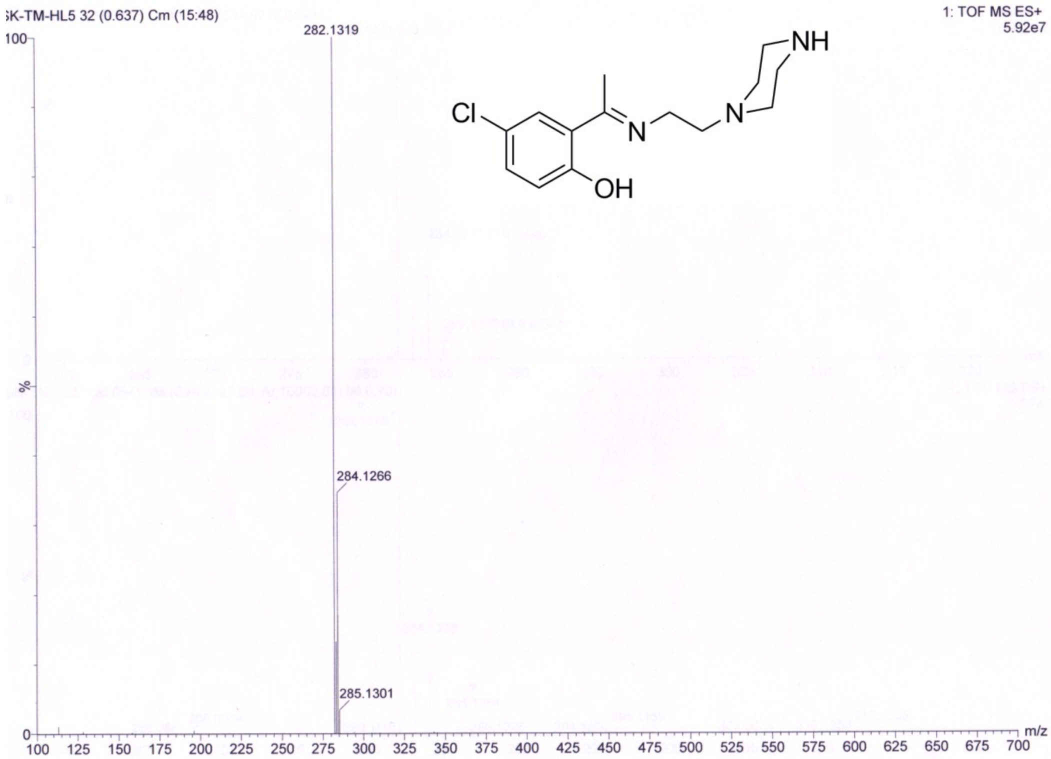
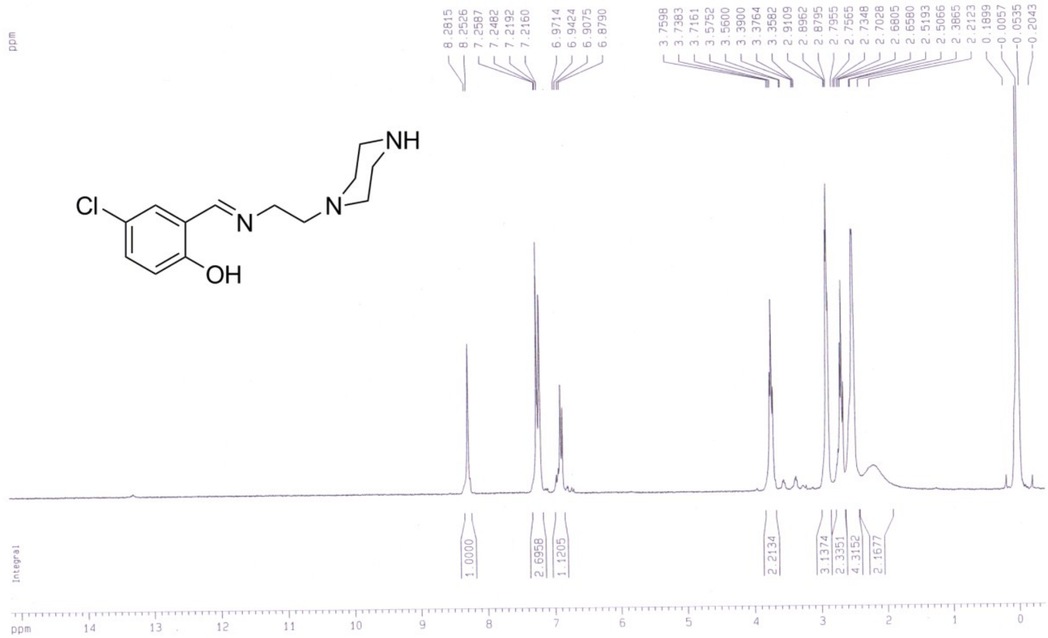
59.081
56.704
54.690
53.407
46.061
19.061
-0.015
NH
Cl
N
N
OH
ppm 180 160 140 120 100 80 60 40 20 0
Current Data Parameters
NAME SK(I)-TMHL4-13C
EXPNO 1
PROCNO 2
F2 - Acquisition Parameters
Date_ 20140826
Time 17.14
INSTRUM av300
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 205
DS 4
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 5.00 cm
F1P 209.691 ppm
F1 15824.92 Hz
F2P -9.691 ppm
F2 -731.37 Hz
PPMCM 9.14093 ppm/cm
HZCM 689.84546 Hz/cm



L5; δ_H (300 MHz; $CDCl_3$, TMS) 2.21 (2H, s), 2.51 (4H, d, J = 3.8 Hz), 2.67-2.79 (2H, m), 2.89 (3H, t, J = 5 Hz), 3.74 (2H, t, J = 6.7 Hz), 6.88-6.97 (1H, m), 7.22-7.26 (2H, m), 8.28 (1H, s); δ_C (75 MHz; $CDCl_3$, TMS) 46.01, 54.56, 56.74, 59.06, 118.66, 119.52, 123.06, 130.32, 132.07, 159.93, 164.44; *m/z* 268.1385 [$(M+H)^+$, 100%]; Yield ca. 95% based on amine; Elemental analysis (found: C 58.37, H 6.81, N 15.69; for $C_{13}H_{18}ClN_3O_1$ requires: C 58.31, H 6.78, N 15.69%).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3.7598 3.7383 3.7161 3.5752 3.5600 3.3900 3.3764 3.3582 2.9109 2.8962 2.8795 2.7955 2.7565 2.7348 2.7028 2.6805 2.6580 2.5193 2.5066 2.3865 2.2123 0.1899 -0.0057 -0.0535 -0.2043
NH
Cl
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OH
Integral
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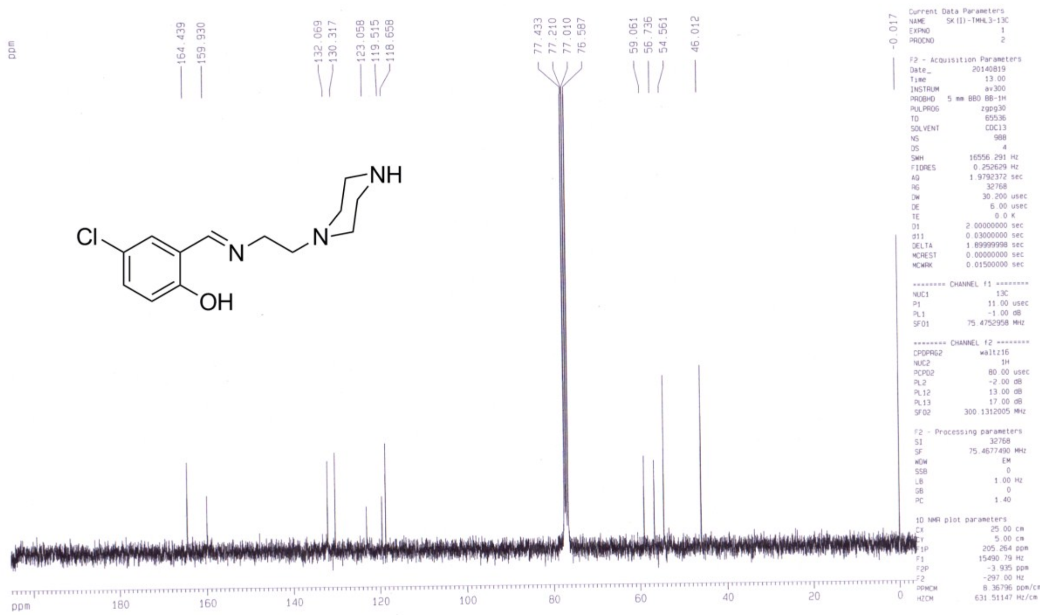
ppm
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NH
Cl
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N
OH
Current Data Parameters
NAME SK(I)-TMHL3-13C
EXPNO 1
PROCNO 2
F2 - Acquisition Parameters
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Time 13.00
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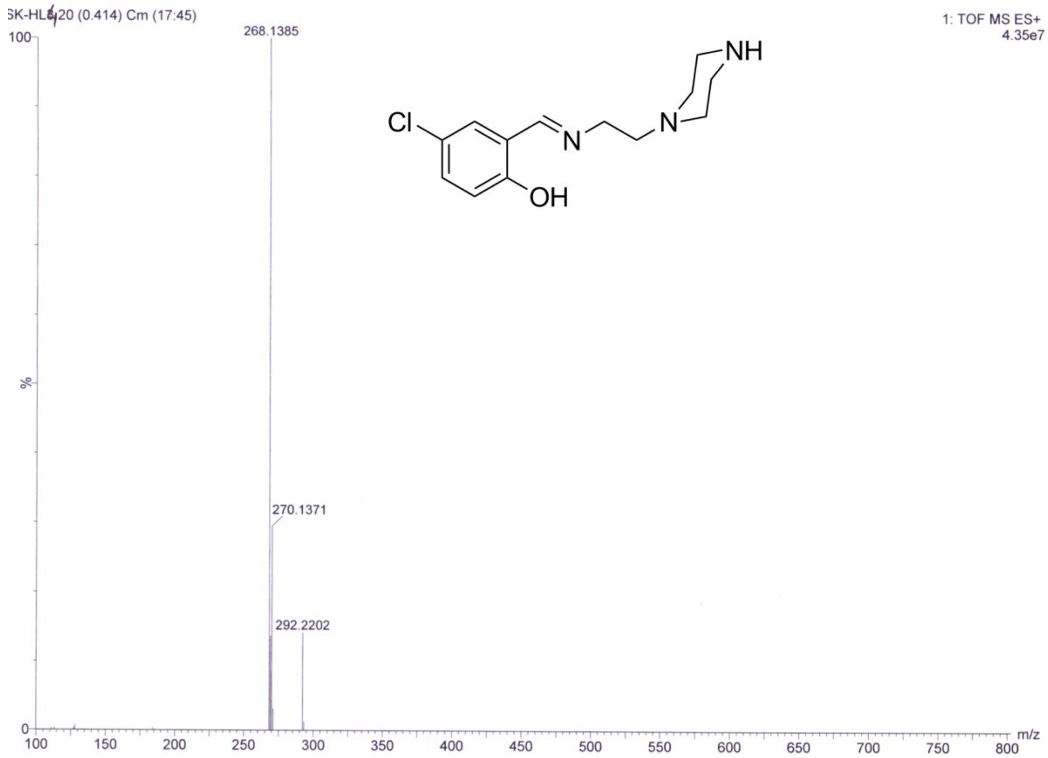
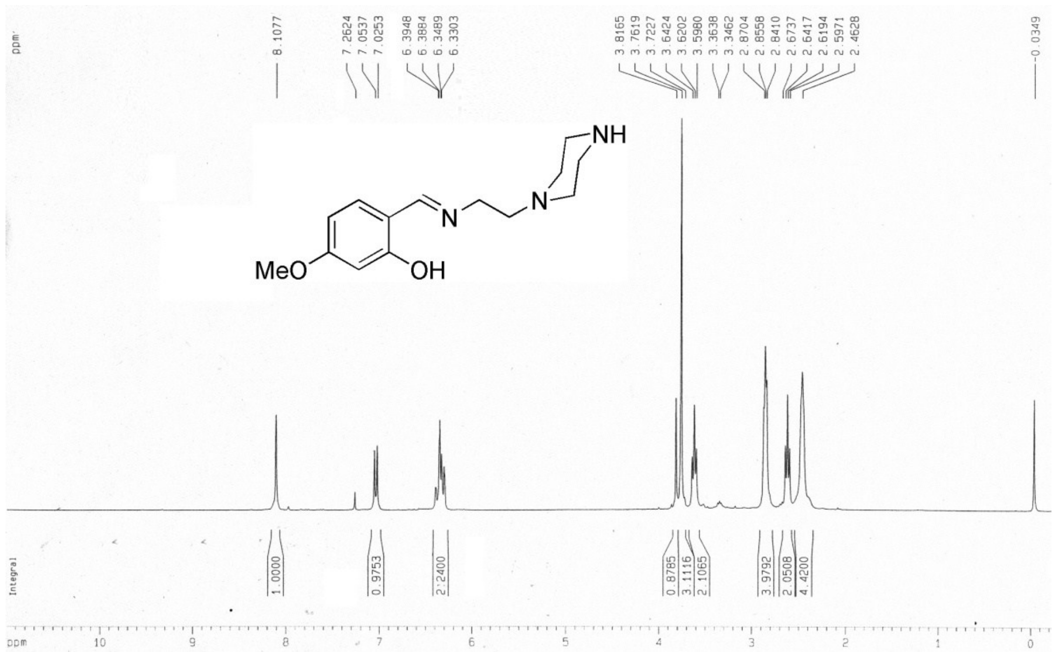
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 988
DS 4
SWH 16556.291 Hz
FIDRES 0.252629 Hz
AQ 1.9792372 sec
RG 32768
DW 30.200 usec
DE 6.00 usec
TE 0.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
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 25.00 cm
CY 5.00 cm
F1P 205.264 ppm
F1 15490.79 Hz
F2P -3.935 ppm
F2 -297.00 Hz
PPMCM 8.36796 ppm/cm
HZCM 631.51147 Hz/cm
ppm 180 160 140 120 100 80 60 40 20 0



L6; δ_H (300 MHz; $CDCl_3$, TMS) 2.46 (4H, s), 2.59-2.67 (2H, m), 2.84-2.87 (4H, m), 3.62 (2H, t, J = 6.7 Hz), 3.76 (3H, s), 3.81 (1H, s), 6.33-6.39 (2H, m), 7.04 (1H, d, J = 8.5 Hz), 8.10 (1H, s); δ_C (75 MHz; $CDCl_3$, TMS) 46.07, 50.48, 55.30, 55.69, 59.21, 101.40, 106.37, 112.14, 132.63, 163.92, 164.20, 166.88; *m/z* 263.1648 [$(M+H)^+$, 80%]; Yield ca. 97% based on amine; Elemental analysis (found: C 63.87, H 8.08, N 15.99; for $C_{14}H_{21}N_2O_3$ requires: C 63.85, H 8.04, N 15.96%).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2.6417
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2.5971
2.4628
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MeO
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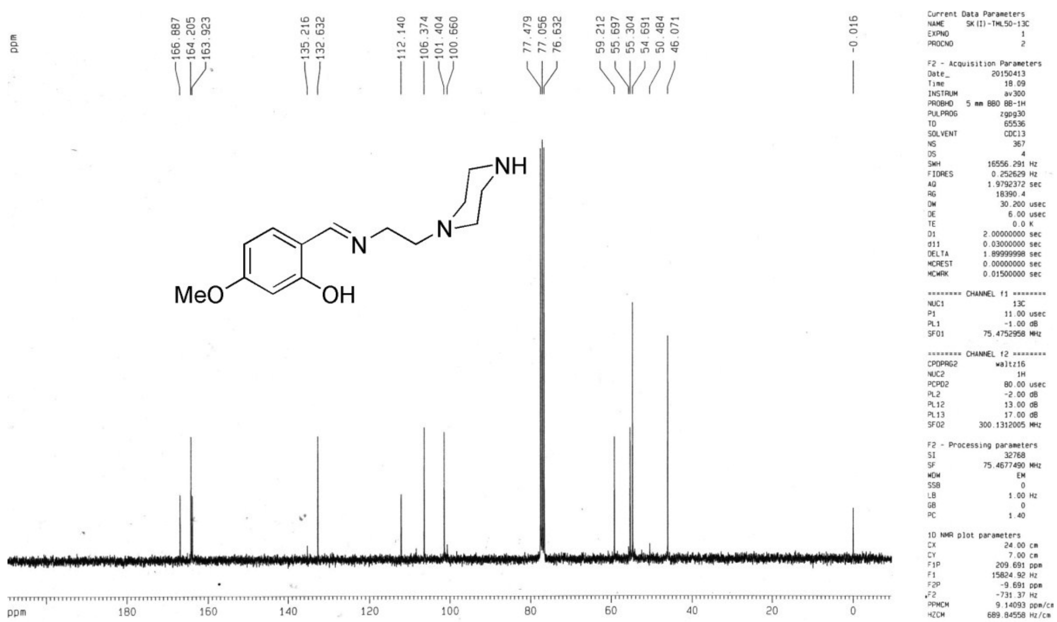
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50.484
46.071
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NH
N
N
MeO
OH
ppm
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160
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120
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80
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40
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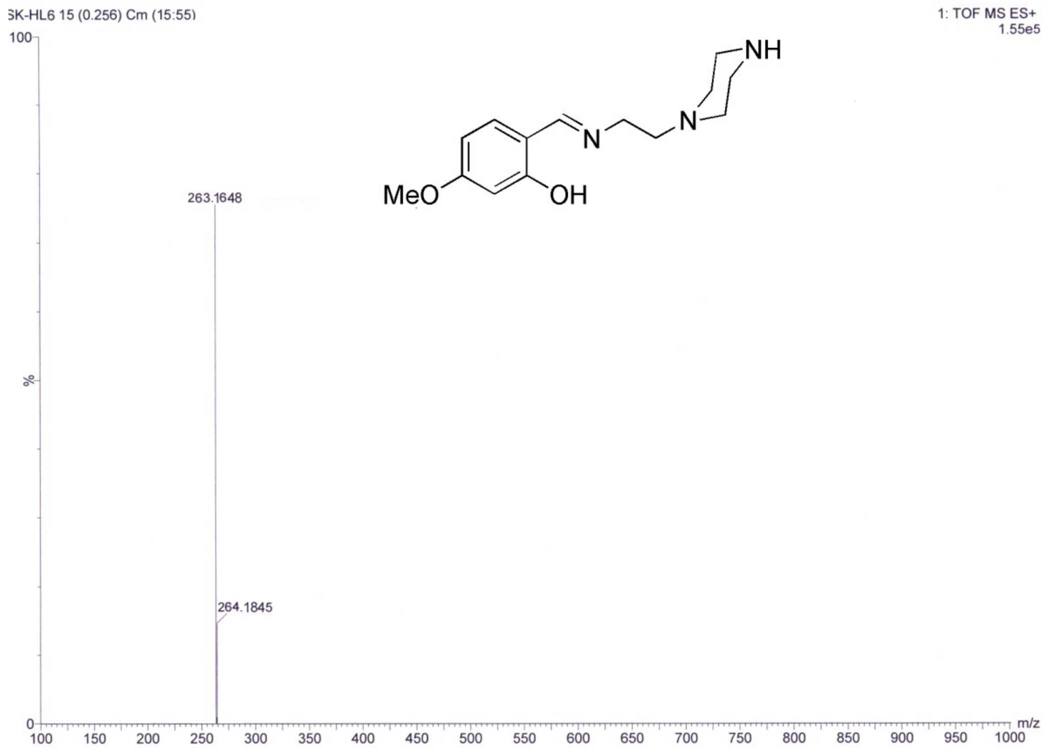
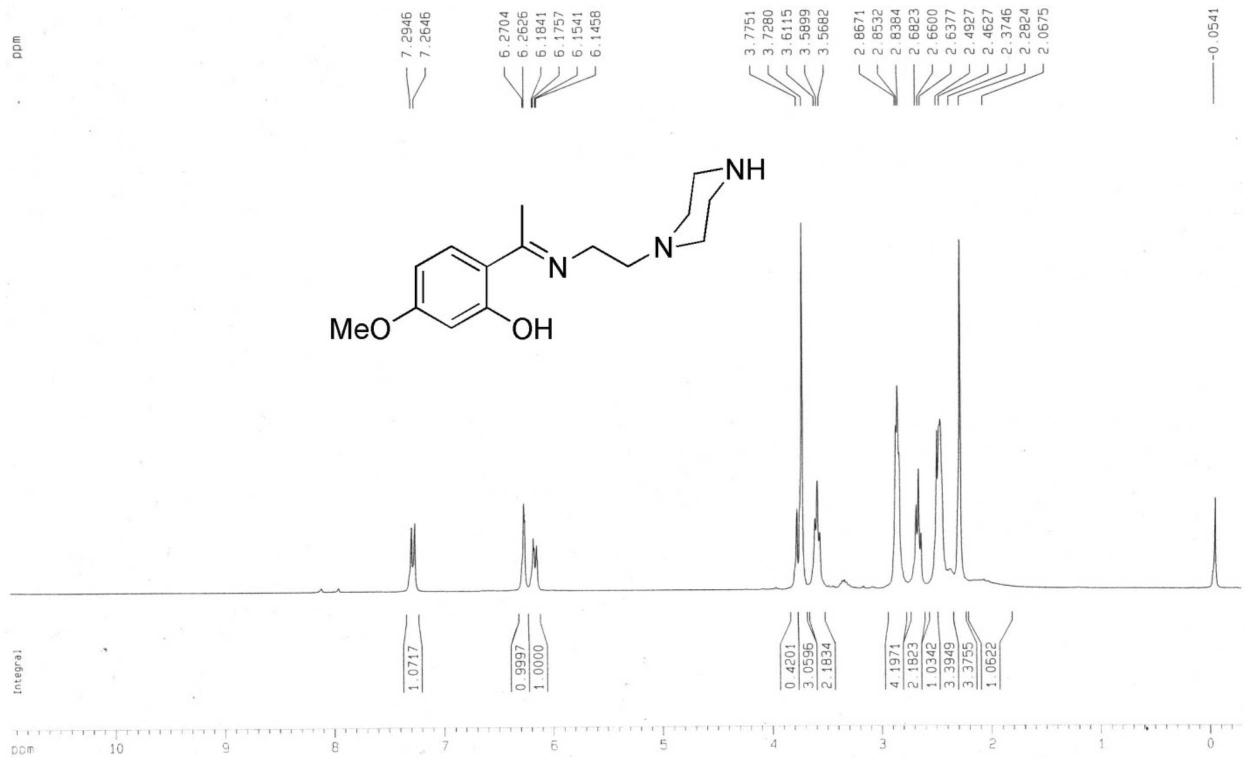
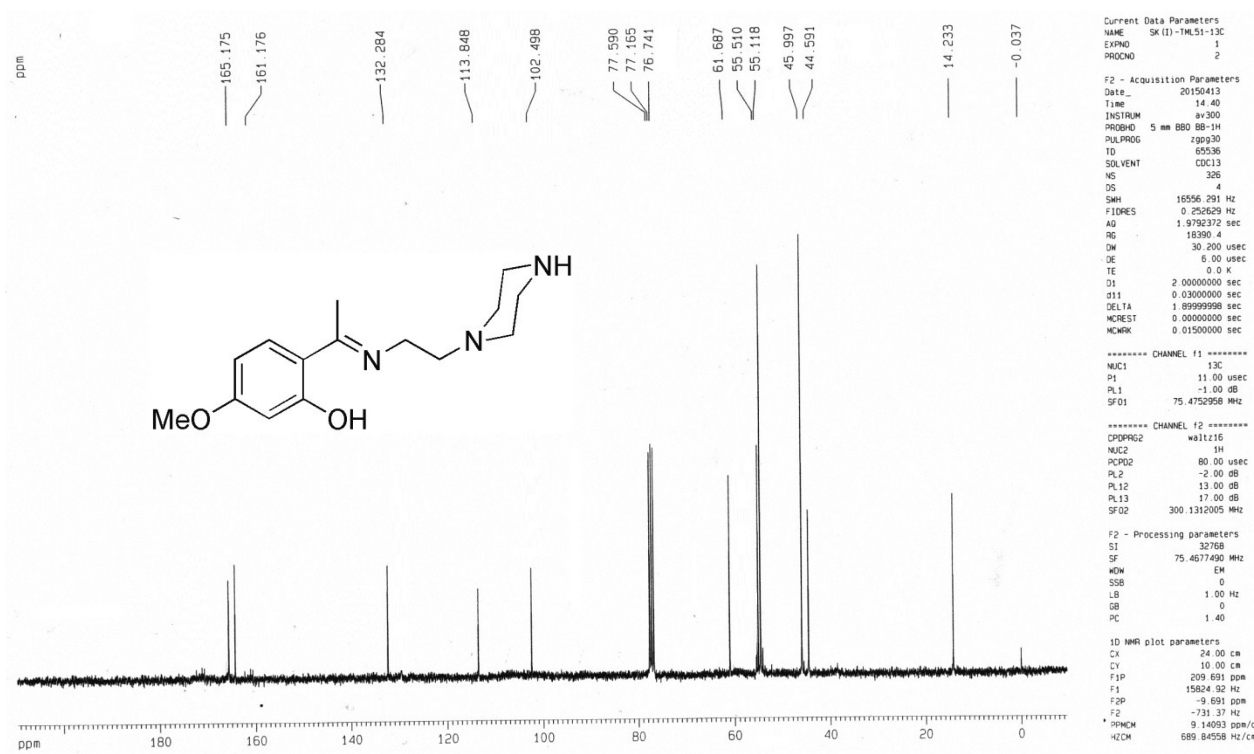
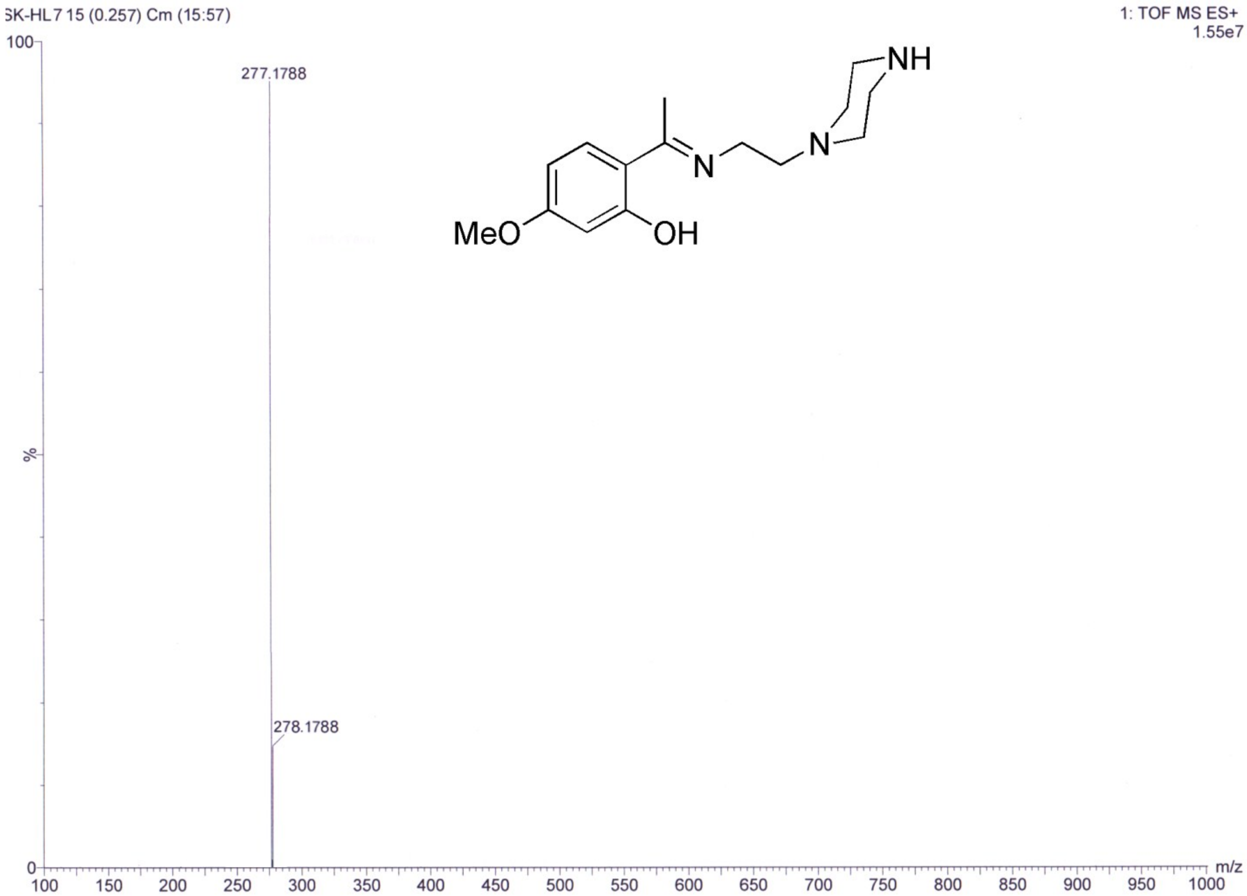
NAME SK(I)-TML50-13C
EXPNO 1
PROCNO 2
F2 - Acquisition Parameters
Date_ 20150413
Time 18.09
INSTRUM av300
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 367
DS 4
SWH 16556.291 Hz
FIDRES 0.252629 Hz
AQ 1.9792372 sec
RG 18390.4
DW 30.200 usec
DE 6.00 usec
TE 0.0 K
D1 2.00000000 sec
d11 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 7.00 cm
F1P 209.691 ppm
F1 15824.92 Hz
F2P -9.691 ppm
F2 -731.37 Hz
PPMCM 9.14093 ppm/cm
HZCM 689.84558 Hz/cm



L7; δ_H (300 MHz; $CDCl_3$, TMS) 2.06 (1H, s), 2.28 (3H, s), 2.37-2.49 (4H, m), 2.66 (2H, t, J = 6.7 Hz), 2.85 (4H, t, J = 4.4 Hz), 3.58 (2H, t, J = 6.5 Hz), 3.72 (3H, s), 6.14-6.27 (2H, m), 7.28 (1H, d, J = 9 Hz); δ_C (75 MHz; $CDCl_3$, TMS) 14.23, 44.59, 45.99, 55.12, 55.51, 61.69, 102.49, 113.85, 132.28, 161.18, 165.18; *m/z* 277.1788 [$(M+H)^+$, 100%]; Yield ca. 95% based on amine; Elemental analysis (found: C 64.97, H 8.35, N 15.19; for $C_{15}H_{23}N_3O_2$ requires: C 64.95, H 8.36, N 15.15%).

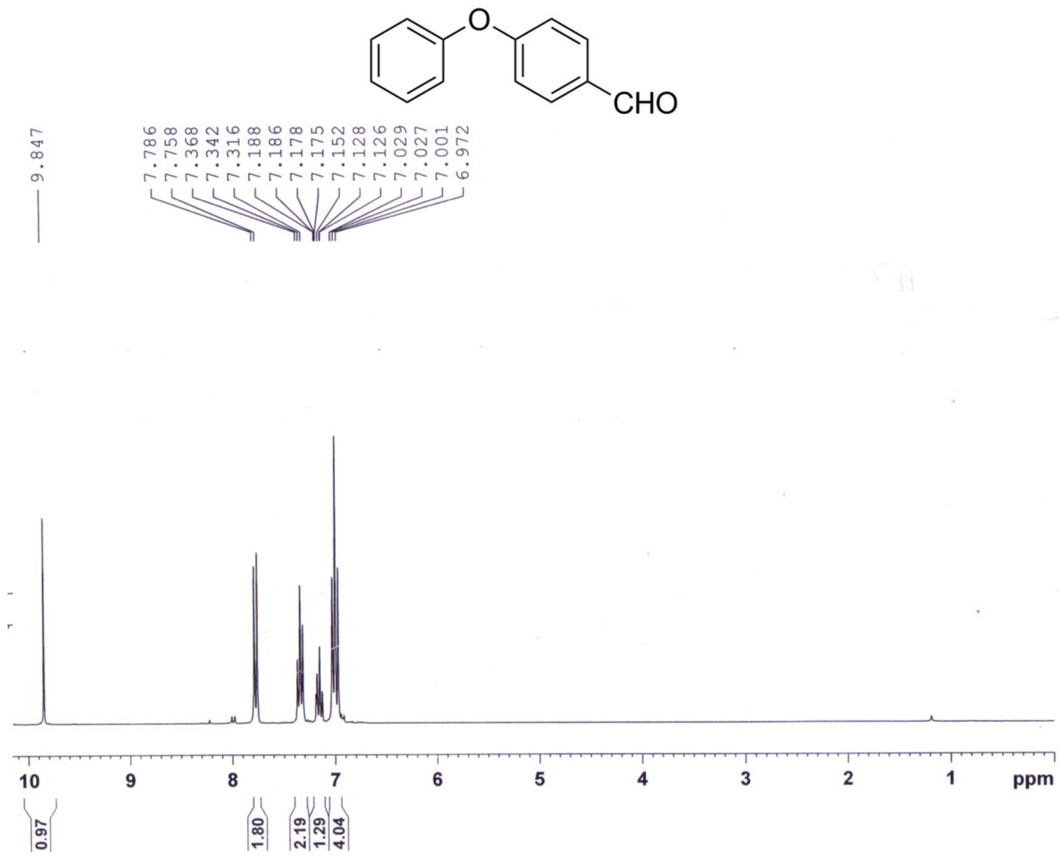

ppm
7.2946
7.2646
6.2704
6.2626
6.1841
6.1757
6.1541
6.1458
3.7751
3.7280
3.6115
3.5899
3.5682
2.8671
2.8532
2.8384
2.6823
2.6600
2.6377
2.4927
2.4627
2.3746
2.2824
2.0675
-0.0541
NH
N
N
MeO
OH
Integral
1.0717
0.9997
1.0000
0.4201
3.0596
2.1834
4.1971
2.1823
1.0342
3.3949
3.3755
1.0622
ppm
10
9
8
7
6
5
4
3
2
1
0

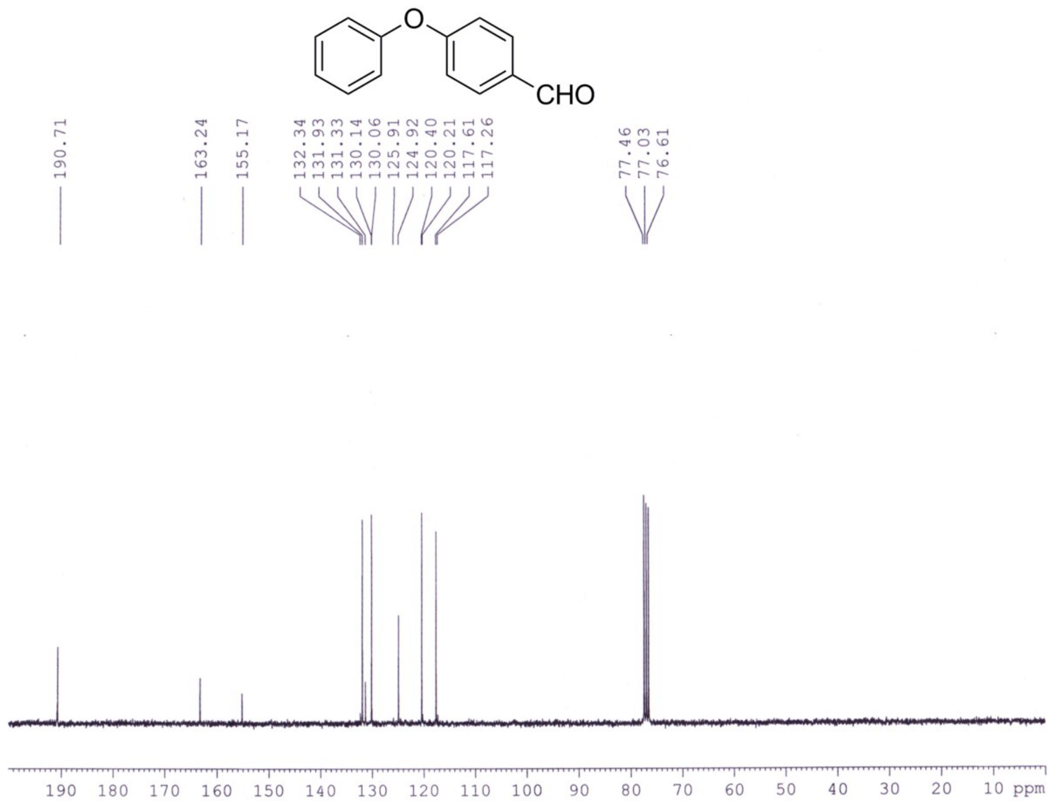

ppm
165.175
161.176
132.284
113.848
102.498
77.590
77.165
76.741
61.687
55.510
55.118
45.997
44.591
14.233
-0.037
NH
N
N
MeO
OH
ppm
180
160
140
120
100
80
60
40
20
0
Current Data Parameters
NAME SK(I)-TML51-13C
EXPNO 1
PROCNO 2
F2 - Acquisition Parameters
Date_ 20150413
Time 14.40
INSTRUM av300
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 326
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 209.691 ppm
F1 15824.92 Hz
F2P -9.691 ppm
F2 -731.37 Hz
PPMCM 9.14093 ppm/c
HZCM 689.84558 Hz/cm

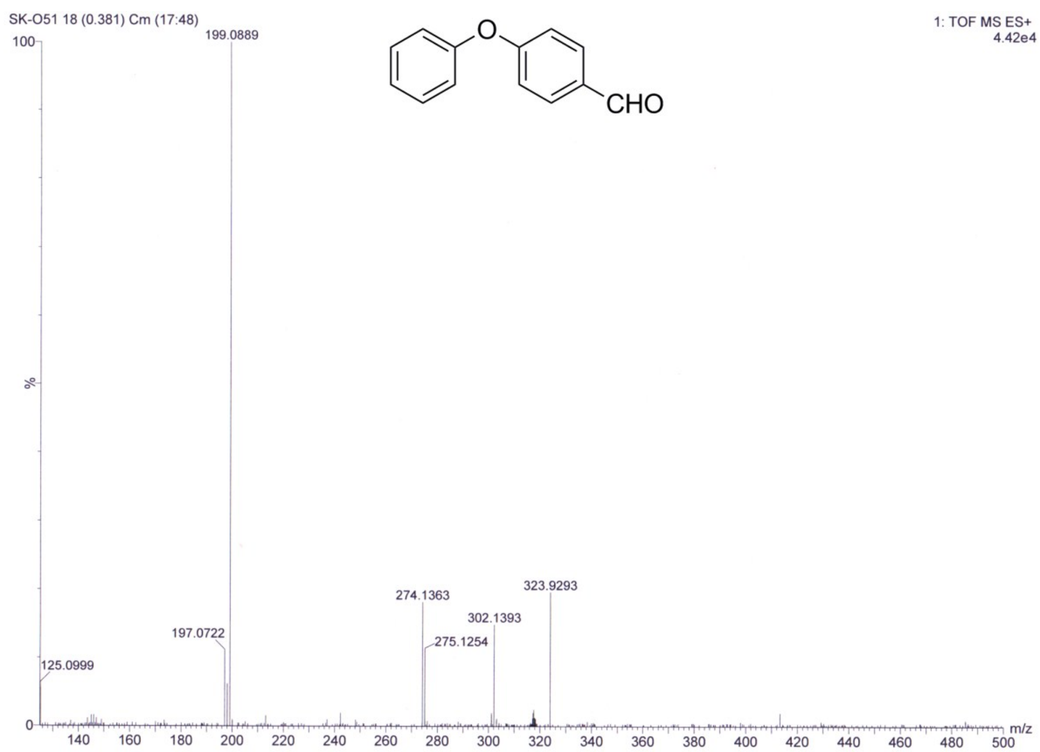


Characterization of Products

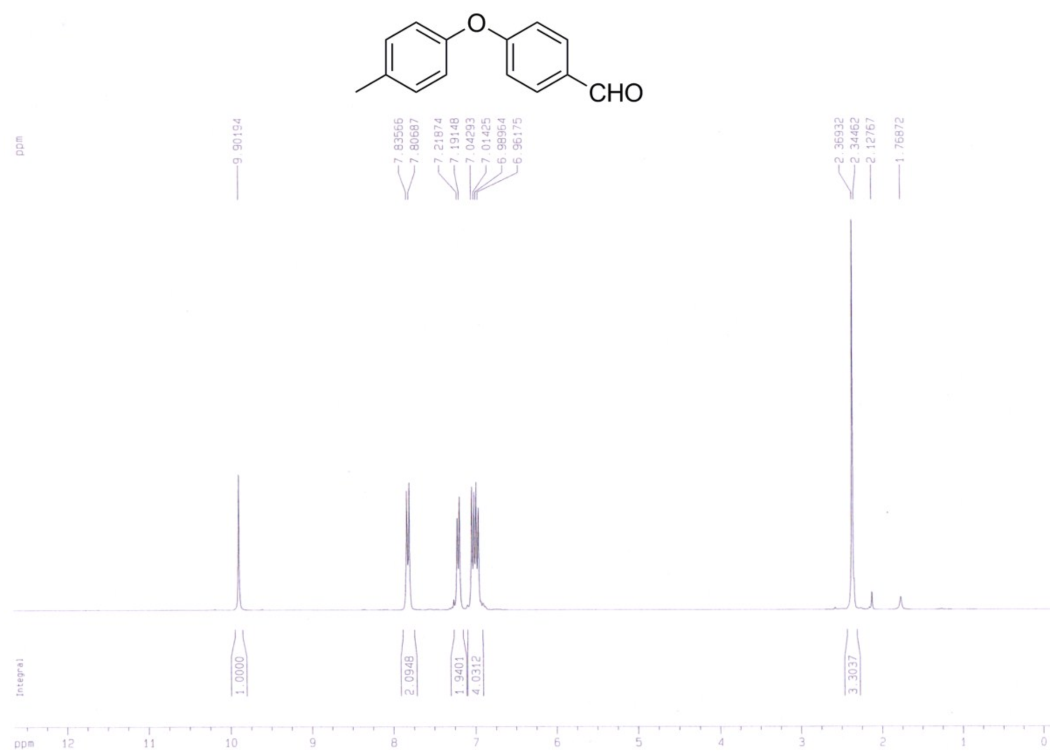
1; δ_H (300 MHz; $CDCl_3$) 6.97-7.03 (4H, m), 7.13-7.19 (1H, m), 7.34 (2H, t, J = 7.8 Hz), 7.77 (2H, d, J = 8.4 Hz), 9.85 (1H, s); δ_C (75 MHz; $CDCl_3$) 117.61, 120.40, 125.91, 130.14, 131.33, 131.93, 132.34, 155.17, 163.24, 190.71; *m/z* 199.0889 [$(M+H)^+$, 100%]; Yield ca. 93% based on amine; Elemental analysis (found: C 78.79, H 5.10; for $C_{13}H_{10}O_2$ requires: C 78.77, H 5.09%).

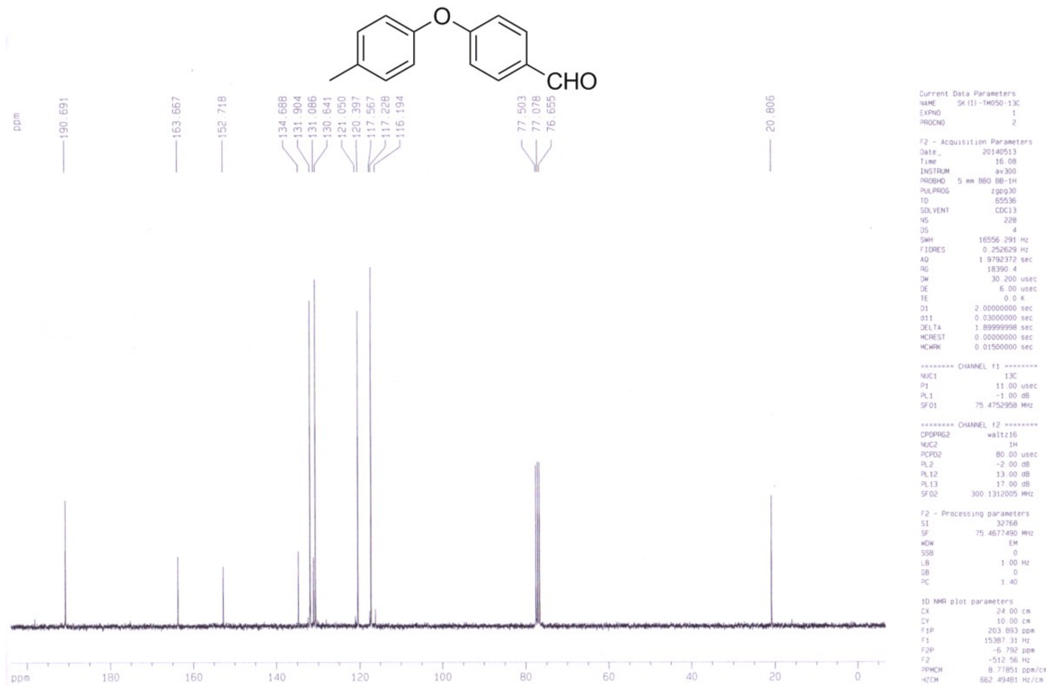

O
CHO
9.847
7.786
7.758
7.368
7.342
7.316
7.188
7.186
7.178
7.175
7.152
7.128
7.126
7.029
7.027
7.001
6.972
10 9 8 7 6 5 4 3 2 1 ppm
0.97
1.80
2.19
1.29
4.04

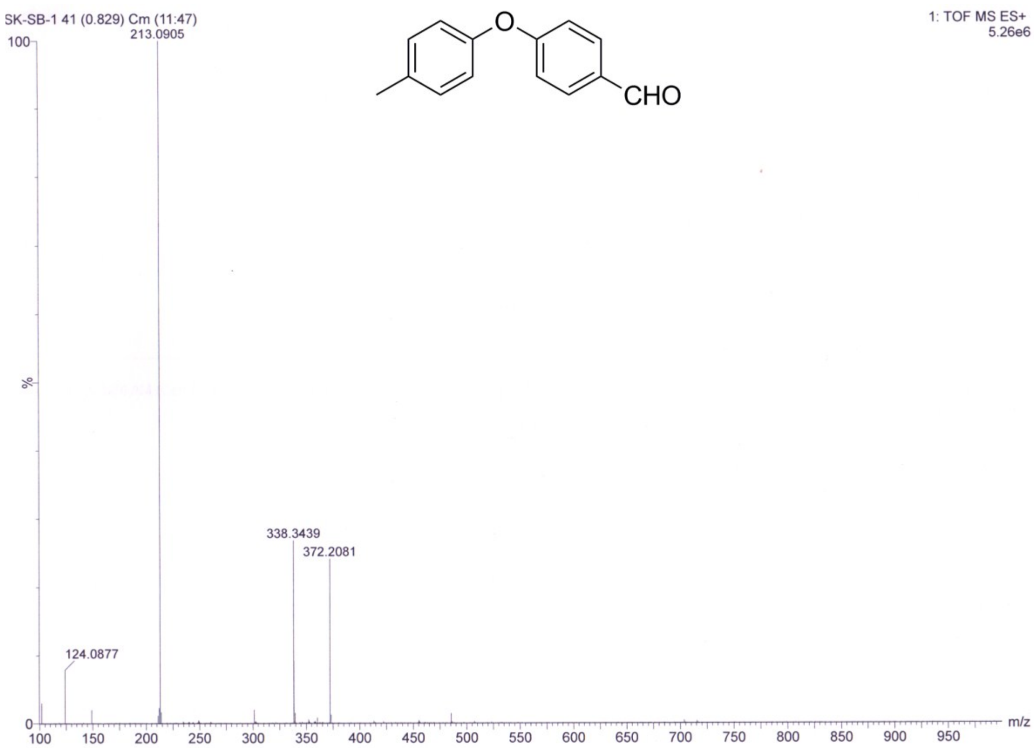

CHO
190.71
163.24
155.17
132.34
131.93
131.33
130.14
130.06
125.91
124.92
120.40
120.21
117.61
117.26
77.46
77.03
76.61
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


SK-O51 18 (0.381) Cm (17:48)
1: TOF MS ES+
4.42e4
CHO
100
%
0
199.0889
197.0722
125.0999
274.1363
275.1254
302.1393
323.9293
140 160 180 200 220 240 260 280 300 320 340 360 380 400 420 440 460 480 500
m/z

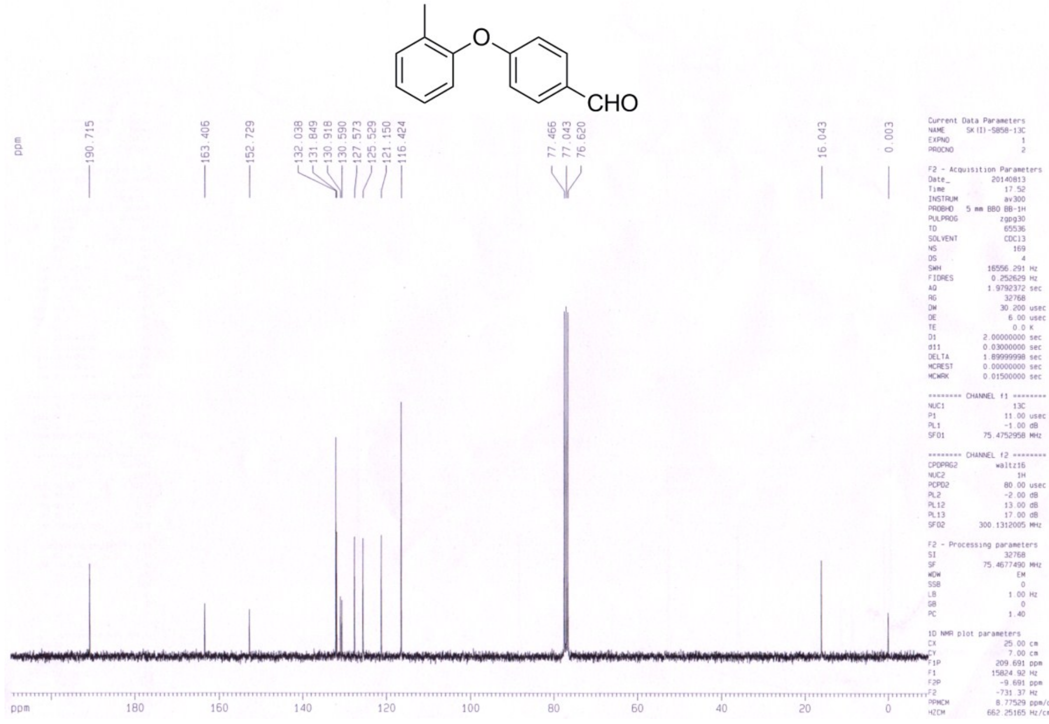
2; δ_H (300 MHz; $CDCl_3$) 2.37 (3H, s), 6.96-7.04 (4H, m), 7.21 (2H, d, J = 8.2 Hz), 7.82 (2H, d, J = 8.6 Hz), 9.90 (1H, s); δ_C (75 MHz; $CDCl_3$) 20.81, 116.19, 117.23, 117.57, 120.39, 121.05, 130.64, 131.09, 131.90, 134.69, 152.72, 163.67, 190.69; *m/z* 213.0905 [$(M+H)^+$, 100%]; Elemental analysis (found: C 79.25, H 5.72; for $C_{14}H_{12}O_2$ requires: C 79.22, H 5.70%).

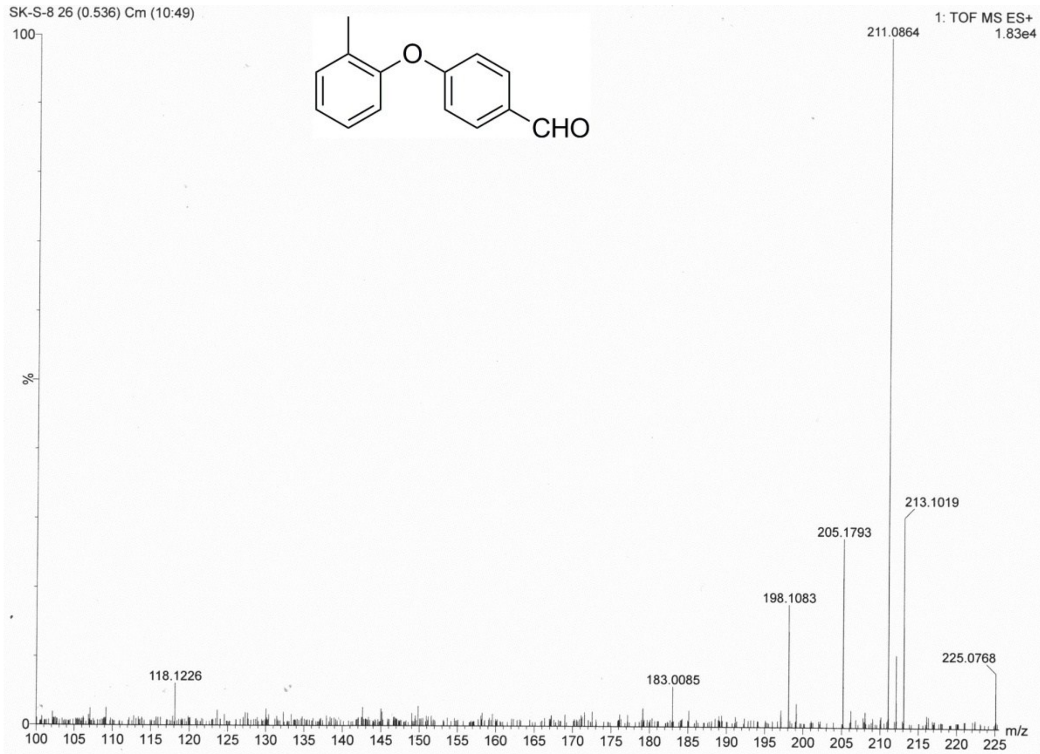




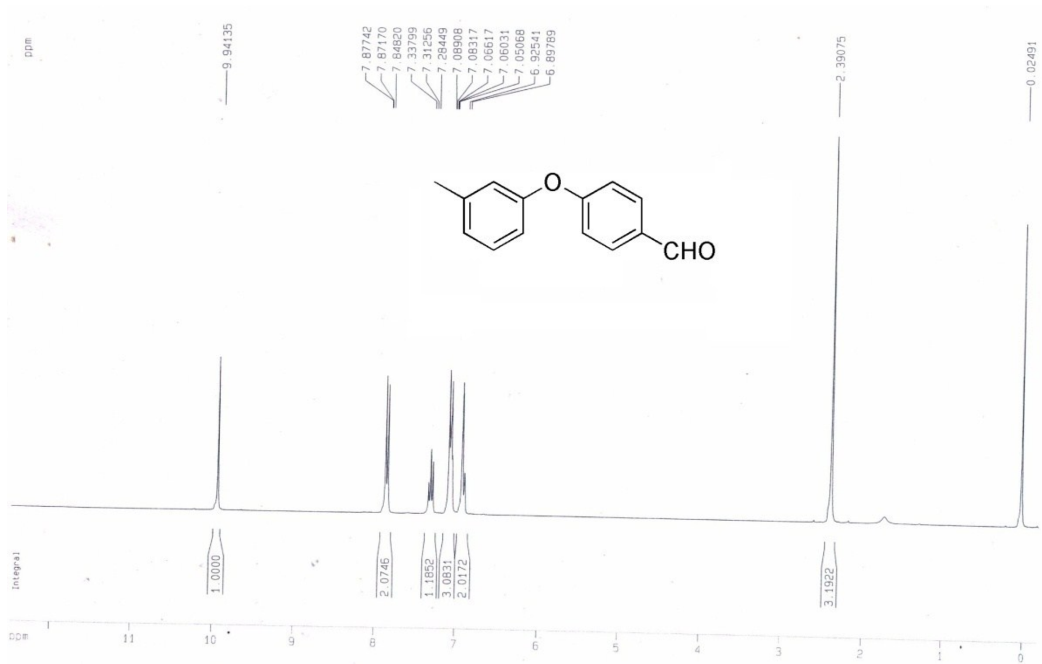


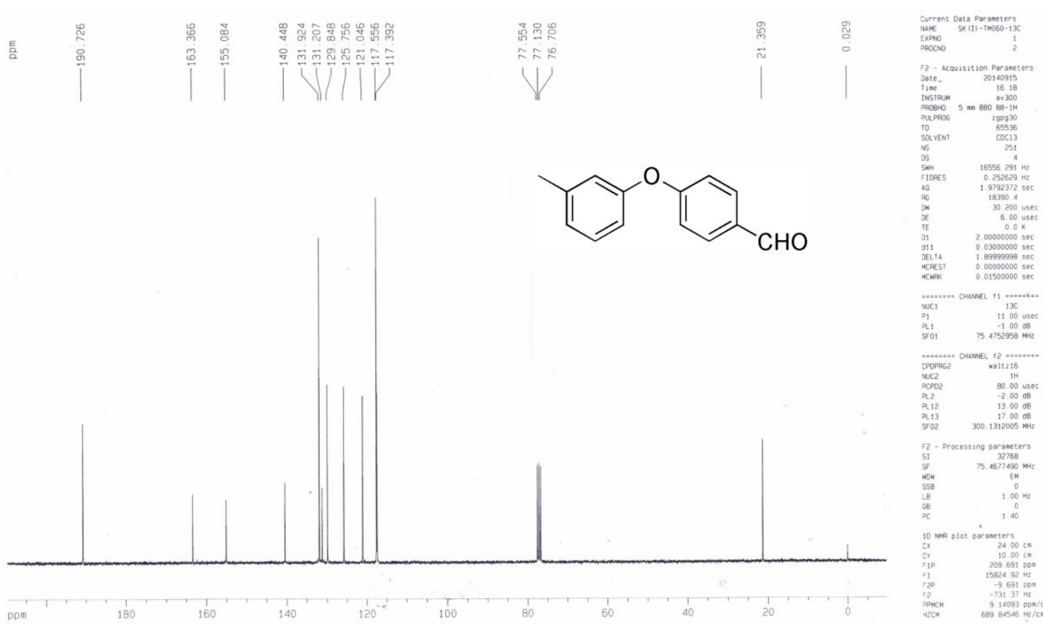
3; δ_H (300 MHz; $CDCl_3$, TMS) 2.19 (3H, s), 6.95-7.02 (3H, m), 7.17-7.31 (3H, m), 7.83 (2H, d, *J* = 8.7 Hz), 9.91 (1H, s); δ_C (75 MHz; $CDCl_3$, TMS) 16.04, 116.42, 121.15, 125.53, 127.57, 130.59, 130.92, 131.85, 132.04, 152.73, 163.41, 190.72; *m/z* 213.1019 [$(M+H)^+$, 30%]; Elemental analysis (found: C 79.23, H 5.69; for $C_{14}H_{12}O_2$ requires: C 79.22, H 5.70%).



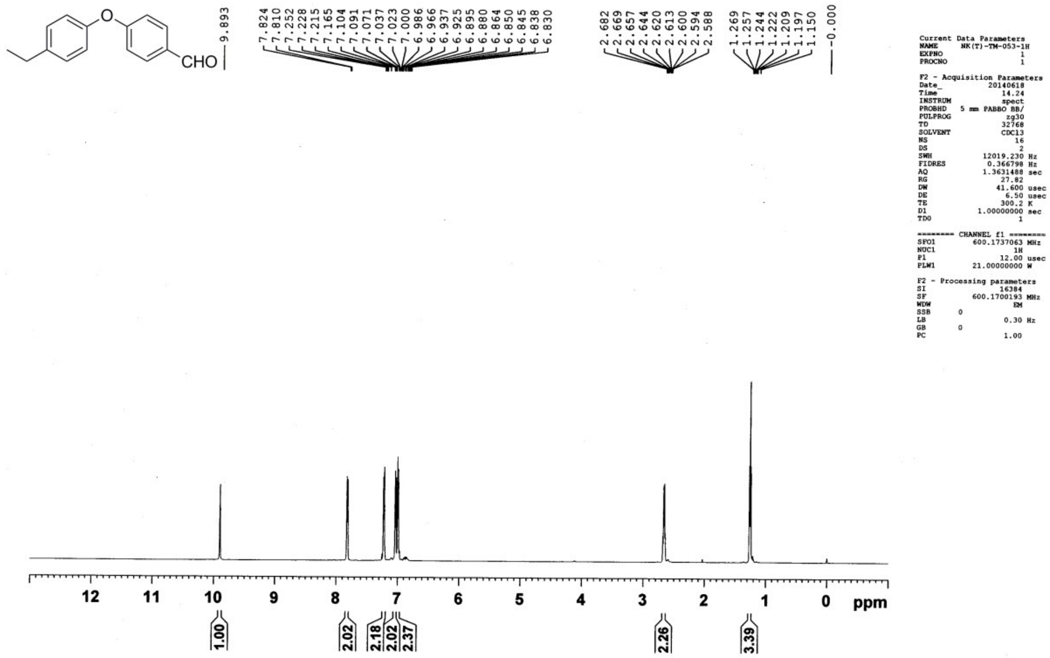


4; δ_H (300 MHz; $CDCl_3$, TMS) 2.39 (3H, s), 6.91 (2H, d, J = 8.3 Hz), 7.05-7.09 (3H, m), 7.31 (1H, t, J = 7.6 Hz), 7.85-7.88 (2H, m), 9.94 (1H, s); δ_C (75 MHz; $CDCl_3$, TMS) 21.36, 117.39, 117.56, 121.05, 125.76, 129.85, 131.21, 131.92, 140.45, 155.08, 163.37, 190.73; Elemental analysis (found: C 79.24, H 5.71; for $C_{14}H_{12}O_2$ requires: C 79.22, H 5.70%).

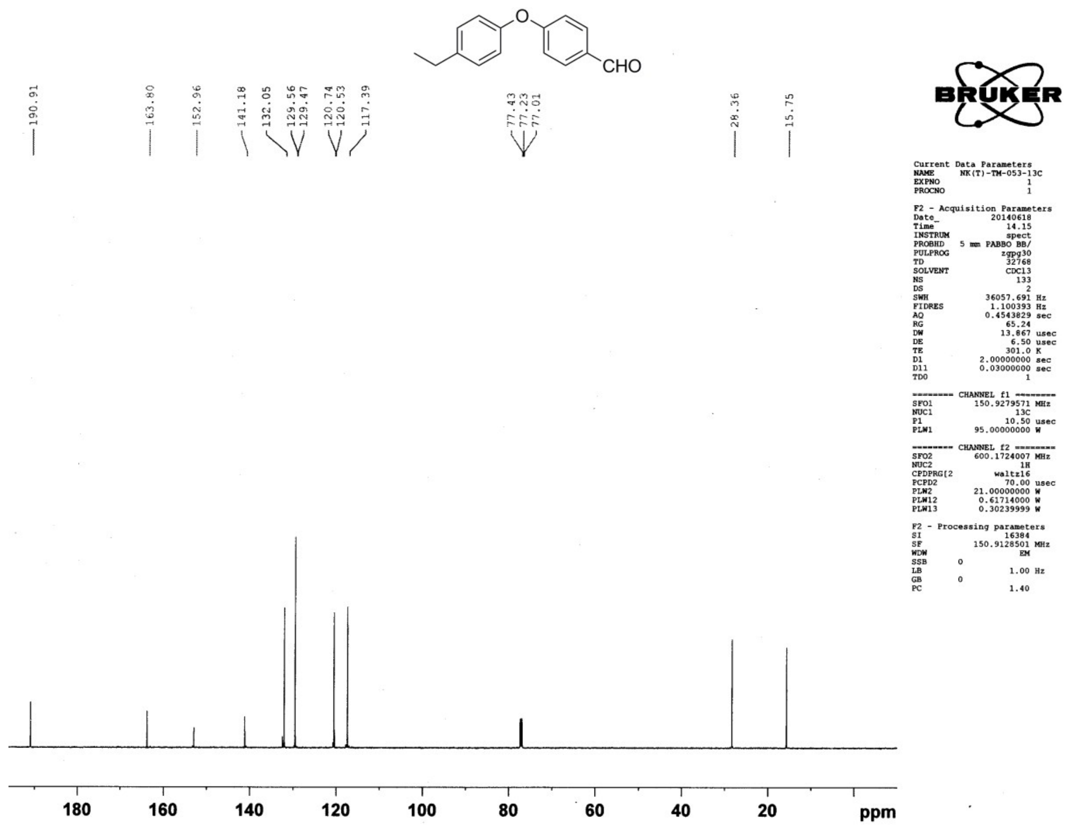




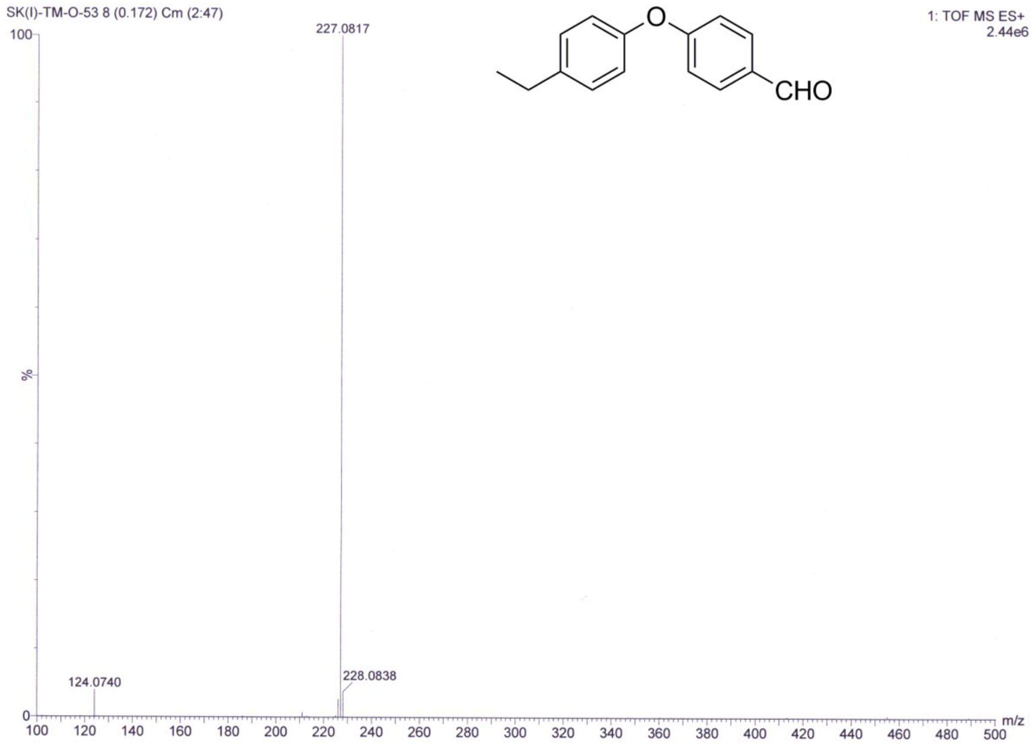
5; δ_H (600 MHz; $CDCl_3$, TMS) 1.15-1.27 (3H, m), 2.59-2.68 (2H, m), 6.93-7.10 (4H, m), 7.22-7.23 (2H, m), 7.82 (2H, d, J = 8.4 Hz), 9.89 (1H, s); δ_C (150 MHz; $CDCl_3$, TMS) 15.75, 28.36, 117.39, 120.53, 129.56, 132.05, 141.28, 152.96, 163.80, 190.91; *m/z* 227.0817 [$(M+H)^+$, 100%]; Elemental analysis (found: C 79.60, H 6.22; for $C_{15}H_{14}O_2$ requires: C 79.62, H 6.24%).



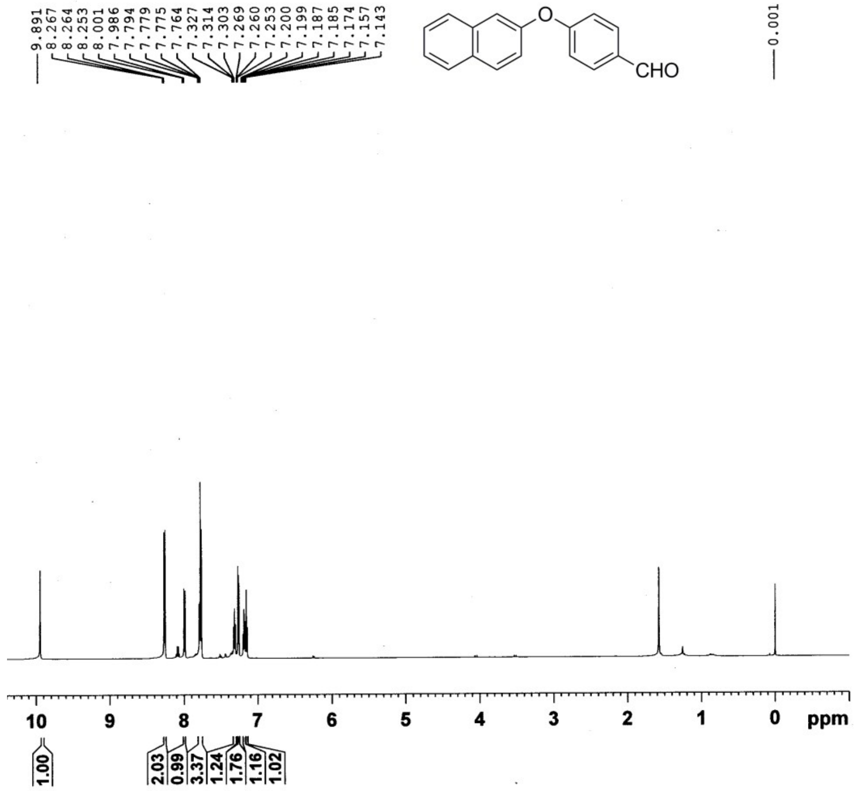
CHO
O
9.893
7.824
7.810
7.252
7.228
7.215
7.165
7.104
7.091
7.071
7.037
7.023
7.000
6.986
6.966
6.937
6.925
6.895
6.880
6.864
6.850
6.845
6.838
6.830
2.682
2.669
2.657
2.644
2.620
2.613
2.600
2.594
2.588
1.269
1.257
1.244
1.222
1.209
1.197
1.150
-0.000
Current Data Parameters
NAME NK(T)-TM-053-1H
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140618
Time 14.24
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 12019.230 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 27.82
DW 41.600 usec
DE 6.50 usec
TE 300.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 600.1737063 MHz
NUC1 1H
P1 12.00 usec
PLW1 21.00000000 W
F2 - Processing parameters
SI 16384
SF 600.1700193 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
12 11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.00
2.02
2.18
2.02
2.37
2.26
3.39



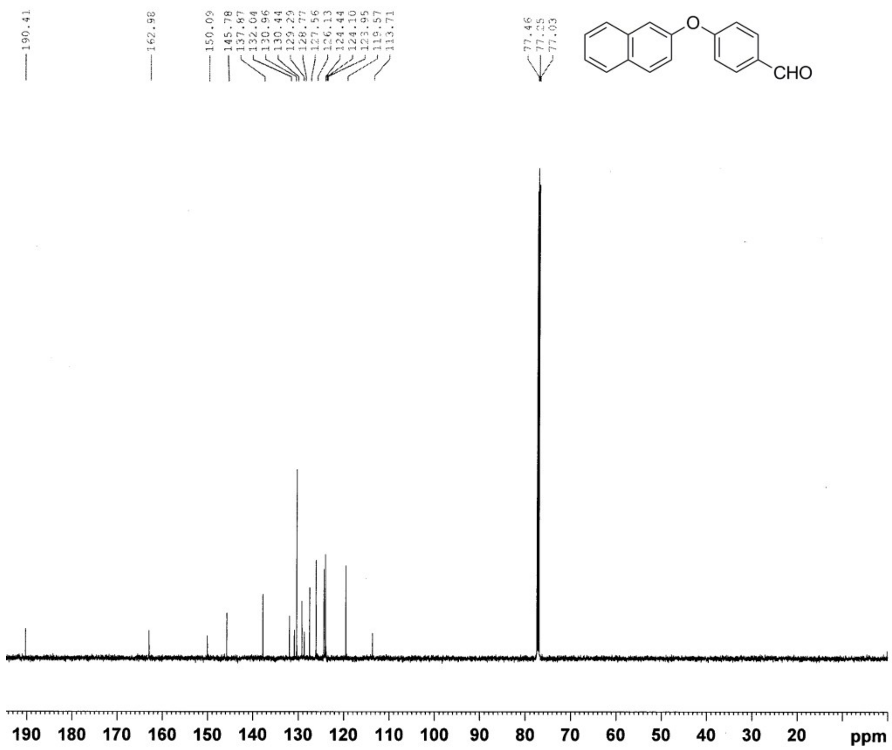
CHO
O
190.91
163.80
152.96
141.18
132.05
129.56
129.47
120.74
120.53
117.39
77.43
77.23
77.01
28.36
15.75
BRUKER
Current Data Parameters
NAME NK(T)-TM-053-13C
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140618
Time 14.15
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 133
DS 2
SWH 36057.691 Hz
FIDRES 1.100393 Hz
AQ 0.4543829 sec
RG 65.24
DW 13.867 usec
DE 6.50 usec
TE 301.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 150.9279571 MHz
NUC1 13C
P1 10.50 usec
PLW1 95.00000000 W
======== CHANNEL f2 ========
SFO2 600.1724007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 21.00000000 W
PLW12 0.61714000 W
PLW13 0.30239999 W
F2 - Processing parameters
SI 16384
SF 150.9128501 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
180 160 140 120 100 80 60 40 20 ppm



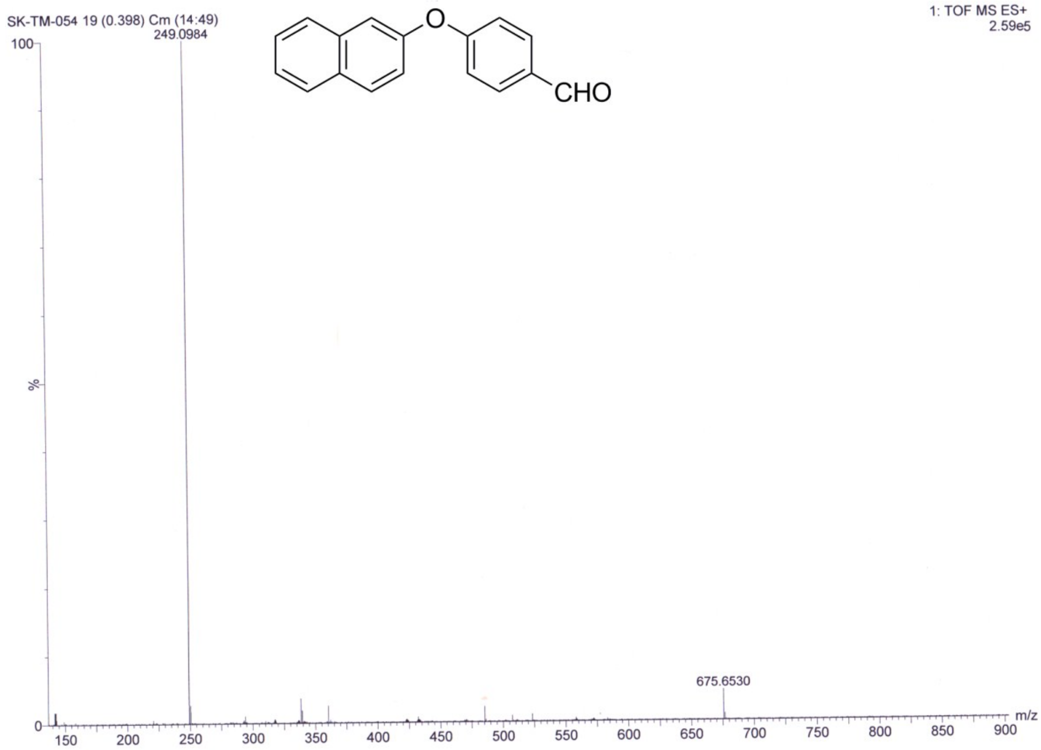
6; δ_H (600 MHz; $CDCl_3$, TMS) 7.14-7.33 (5H, m), 7.76-7.79 (3H, m), 7.99 (1H, d, J = 9 Hz), 8.25-8.27 (2H, m), 9.89 (1H, s); δ_C (150 MHz; $CDCl_3$, TMS) 113.71, 119.57, 123.95, 124.10, 124.44, 126.13, 127.56, 128.77, 129.29, 130.44, 130.96, 132.04, 137.87, 145.78, 150.09, 162.98, 190.41; *m/z* 249.0984 [$(M+H)^+$, 100%]; Elemental analysis (found: C 82.20, H 4.86; for $C_{14}H_{12}O_2$ requires: C 82.24, H 4.87%).



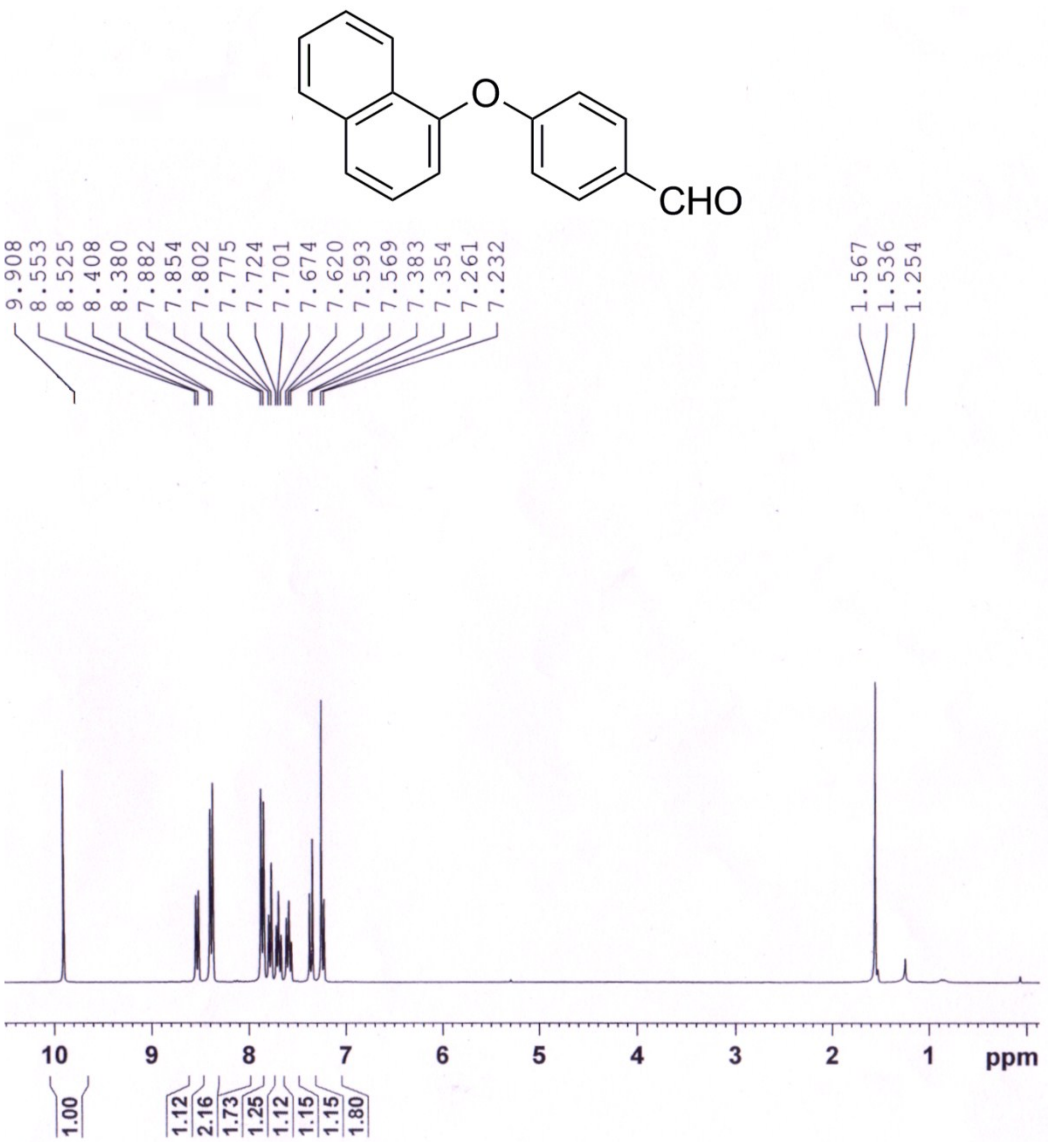

```
Current Data Parameters
NAME     NK(T)-TM-054-1H
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140618
Time               14.31
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     2
SWH            12019.230 Hz
FIDRES          0.366798 Hz
AQ             1.3631488 sec
RG                   113
DW                41.600 usec
DE                  6.50 usec
TE                 300.1 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         600.1737063 MHz
NUC1                  1H
P1                 12.00 usec
PLW1         21.00000000 W

F2 - Processing parameters
SI                 16384
SF           600.1700146 MHz
WDW                   EM
SSB      0
LB                  0.30 Hz
GB       0
PC                  1.00
```



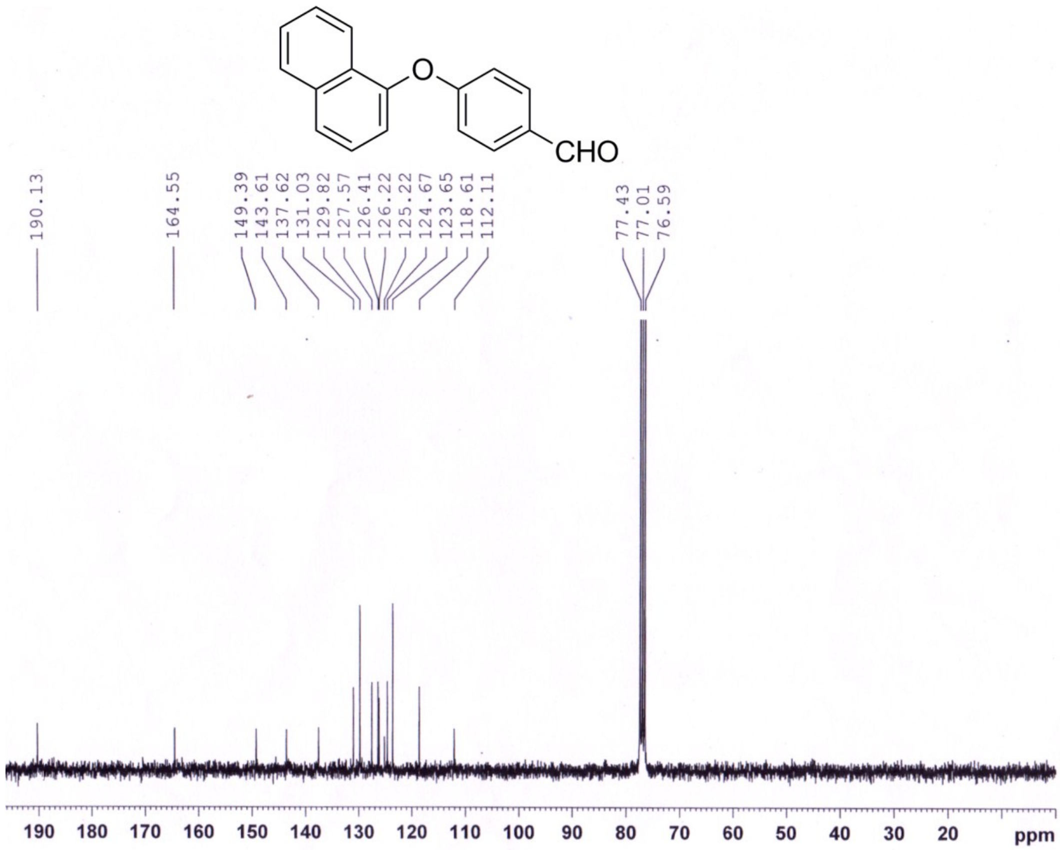

```
Current Data Parameters
NAME     NK(T)-TM-054-13C
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140618
Time               14.38
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG           zgpg30
TD                 32768
SOLVENT            CDCl3
NS                   307
DS                     2
SWH            36057.691 Hz
FIDRES          1.100393 Hz
AQ             0.4543829 sec
RG                 65.24
DW                13.867 usec
DE                  6.50 usec
TE                 300.9 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         150.9279571 MHz
NUC1                 13C
P1                 10.50 usec
PLW1         95.00000000 W

======== CHANNEL f2 ========
SFO2         600.1724007 MHz
NUC2                  1H
CPDPRG[2         waltz16
PCPD2              70.00 usec
PLW2         21.00000000 W
PLW12         0.61714000 W
PLW13         0.30239999 W

F2 - Processing parameters
SI                 16384
SF           150.9128316 MHz
WDW                   EM
SSB      0
LB                  1.00 Hz
GB       0
PC                  1.40
```

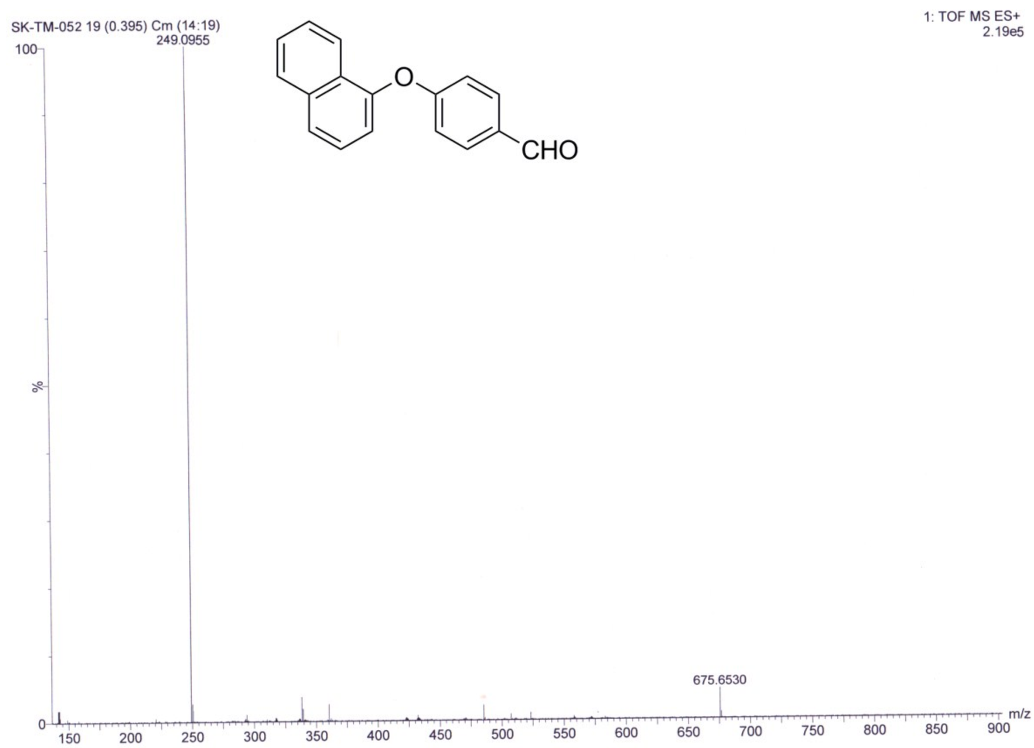


7; δ_H (300 MHz; $CDCl_3$) 7.25 (2H, d, *J* = 8.7 Hz), 7.37 (1H, d, *J* = 8.7 Hz), 7.59 (1H, t, *J* = 7.2 Hz), 7.70 (1H, t, *J* = 7.2 Hz), 7.79 (1H, d, *J* = 8.1 Hz), 7.87 (2H, d, *J* = 7.8 Hz), 8.39 (2H, d, *J* = 8.4 Hz), 8.54 (1H, d, *J* = 8.4 Hz), 9.91 (1H, s); δ_C (75 MHz; $CDCl_3$) 112.11, 118.61, 123.65, 124.67, 125.22, 126.22, 126.41, 127.57, 129.82, 131.03, 137.62, 143.61, 149.39, 164.55, 190.13; *m/z* 249.0955 [$(M+H)^+$, 100%]; Elemental analysis (found: C 82.25, H 4.85; for $C_{14}H_{12}O_2$ requires: C 82.24, H 4.87%).

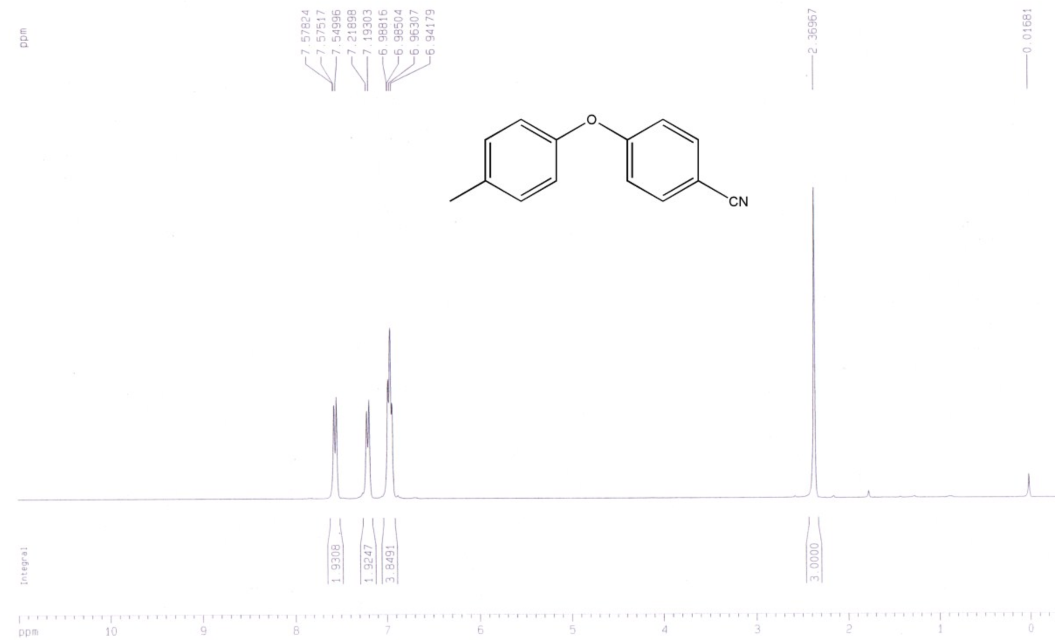


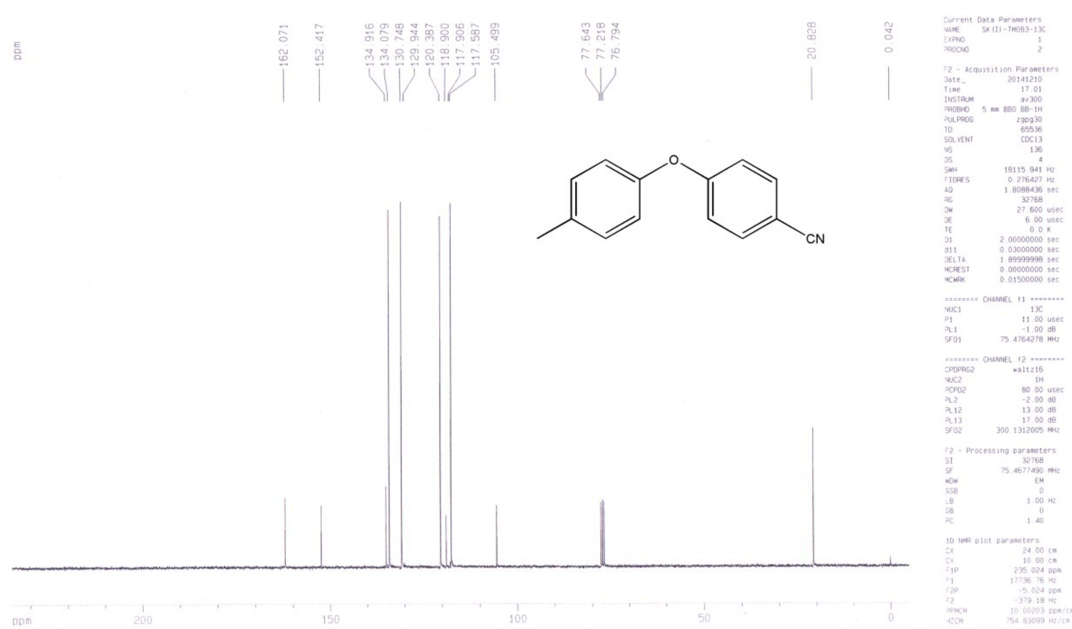
O
CHO
9.908
8.553
8.525
8.408
8.380
7.882
7.854
7.802
7.775
7.724
7.701
7.674
7.620
7.593
7.569
7.383
7.354
7.261
7.232
1.567
1.536
1.254
10
9
8
7
6
5
4
3
2
1
ppm
1.00
1.12
2.16
1.73
1.25
1.12
1.15
1.15
1.80

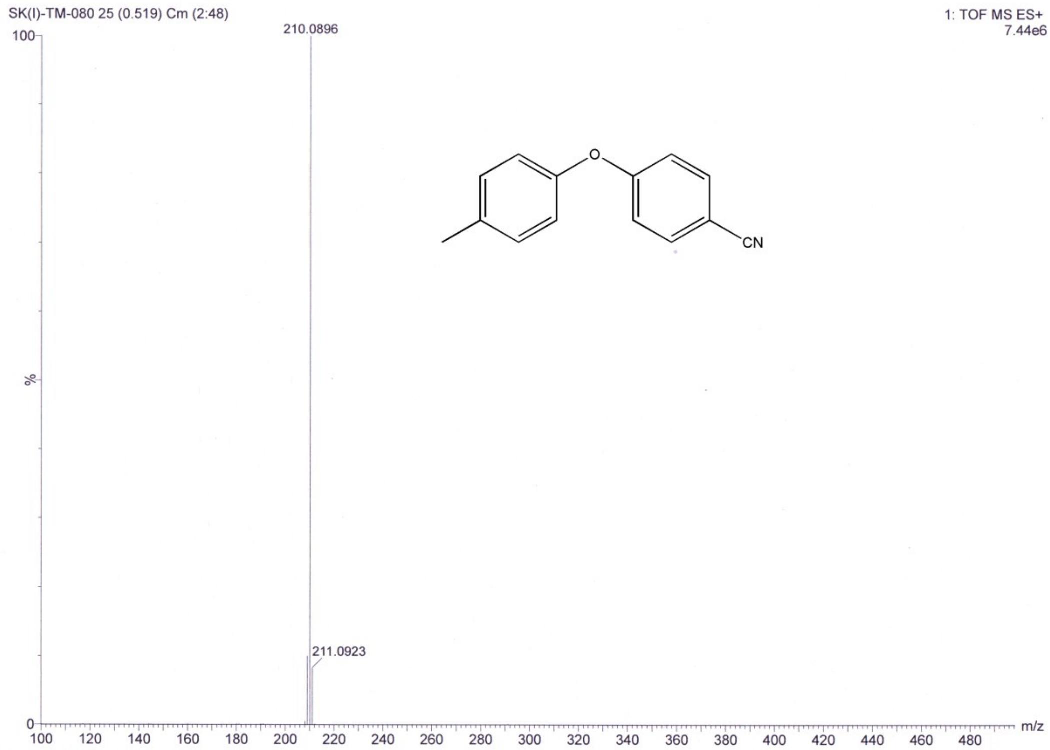

CHO
190.13
164.55
149.39
143.61
137.62
131.03
129.82
127.57
126.41
126.22
125.22
124.67
123.65
118.61
112.11
77.43
77.01
76.59
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm


SK-TM-052 19 (0.395) Cm (14:19)
1: TOF MS ES+
2.19e5
249.0955
CHO
100
%
0
675.6530
150 200 250 300 350 400 450 500 550 600 650 700 750 800 850 900
m/z

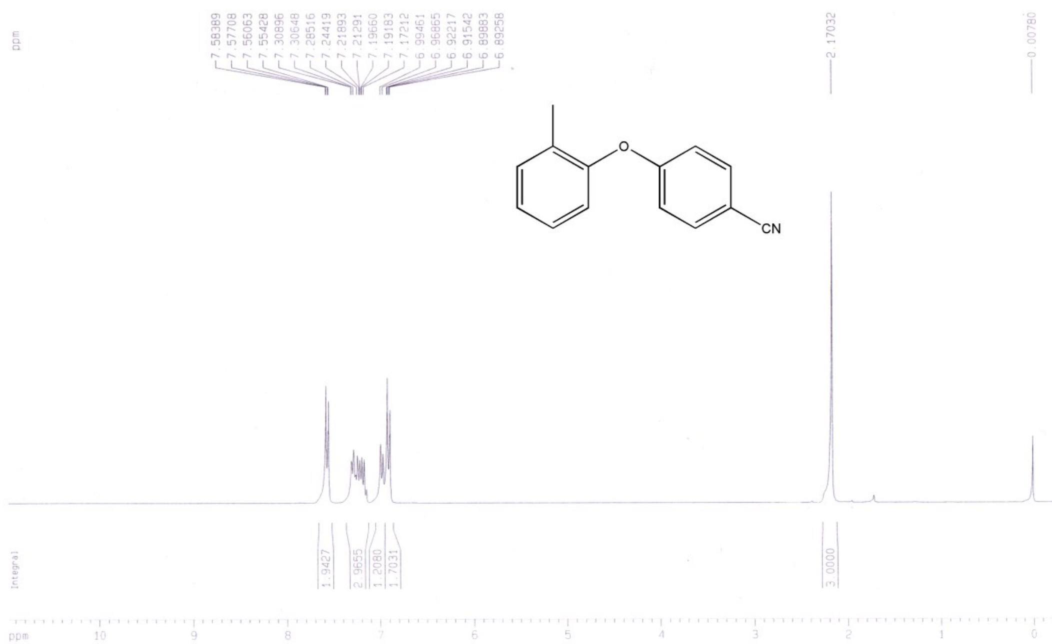
8; δ_H (300 MHz; $CDCl_3$) 2.37 (3H, s), 6.99-6.94 (4H, m), 7.21 (2H, d, J = 7.8 Hz), 7.56 (2H, d, J = 8.4 Hz); δ_C (75 MHz; $CDCl_3$) 20.83, 105.49, 117.59, 117.91, 118.90, 120.39, 129.94, 134.08, 134.92, 152.42, 162.07; *m/z* 210.0896 [$(M+H)^+$, 100%]; Elemental analysis (found: C 80.36, H 5.30, N 6.69; for $C_{14}H_{12}O_2$ requires: C 80.34, H 5.37, N 6.63%).

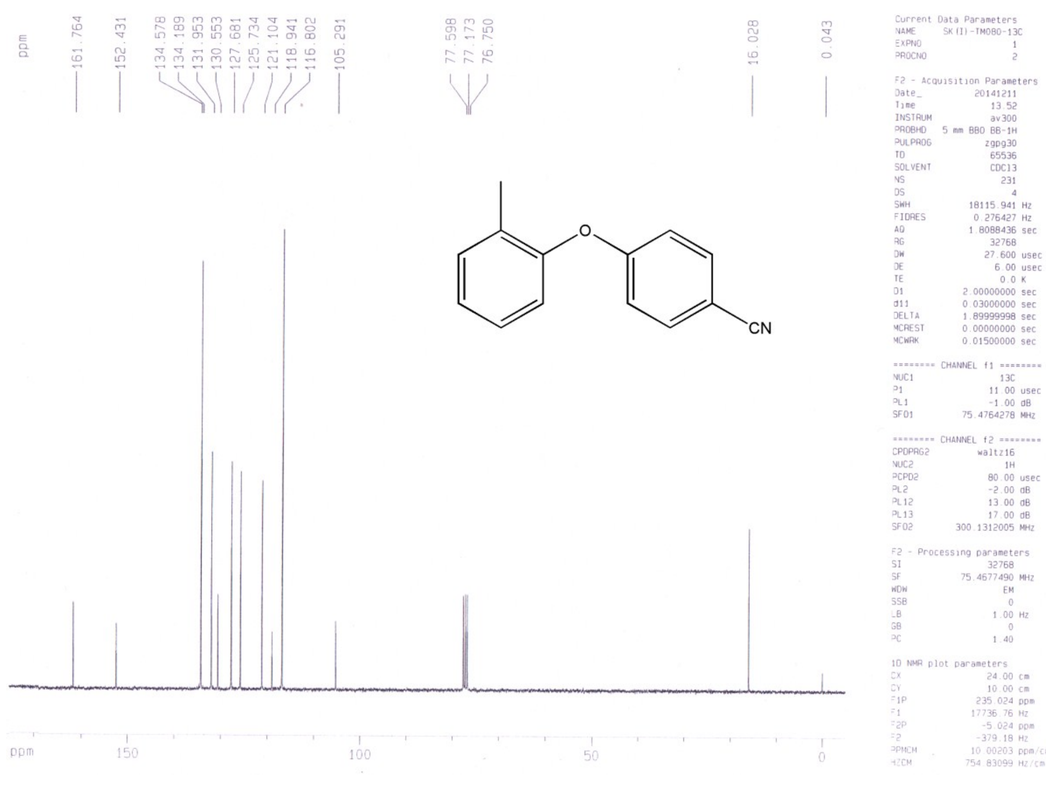




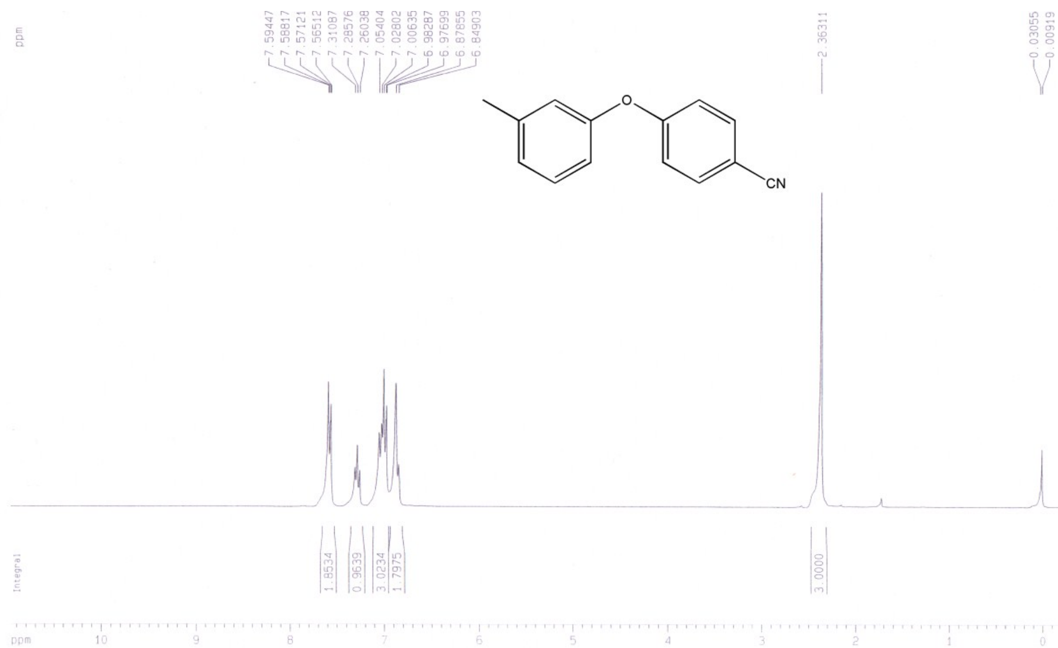


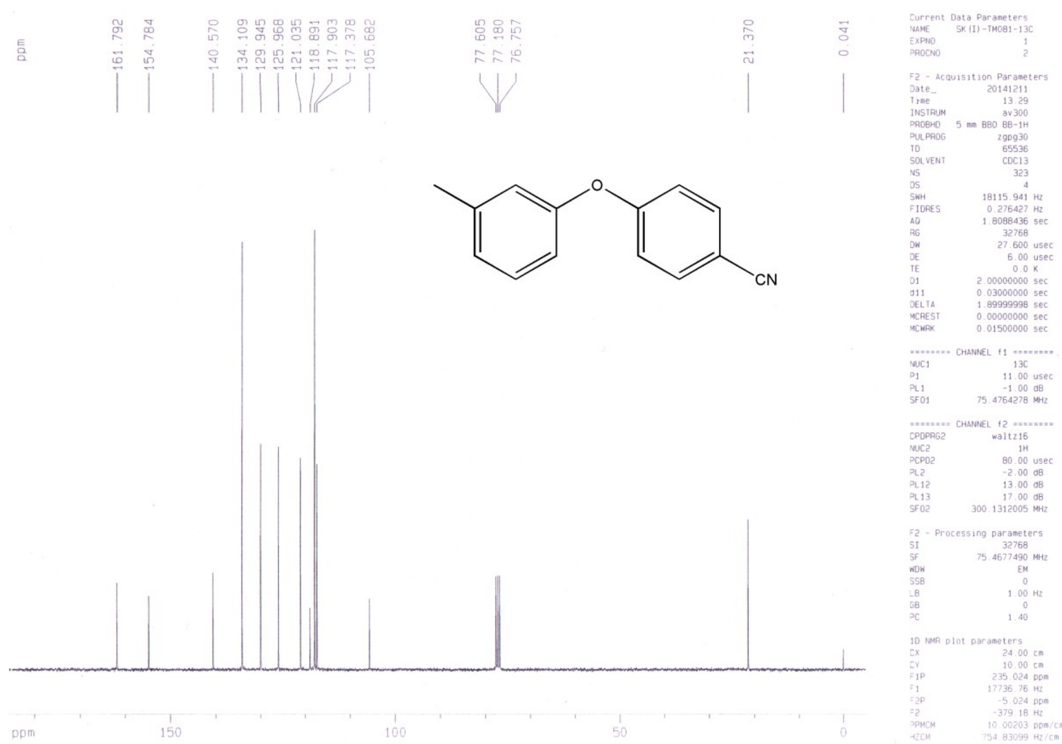
9; δ_H (300 MHz; $CDCl_3$) 2.17 (3H, s), 6.89-6.99 (3H, m), 7.17-7.30 (3H, m), 7.57 (2H, dd, J = 1.9 and J = 6.8 Hz); δ_C (75 MHz; $CDCl_3$) 16.02, 105.29, 116.80, 118.94, 121.10, 125.73, 127.68, 130.55, 131.95, 134.19, 134.58, 152.43, 161.76; Elemental analysis (found: C 80.36, H 5.30, N 6.69; for $C_{14}H_{12}O_2$ requires: C 80.39, H 5.34, N 6.72%).



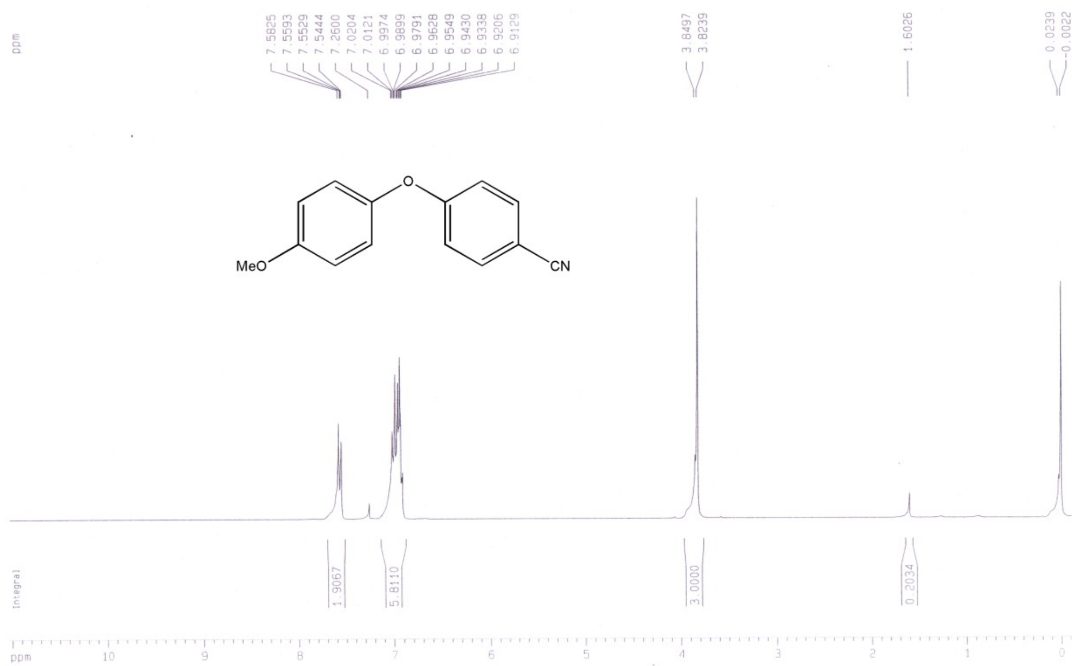


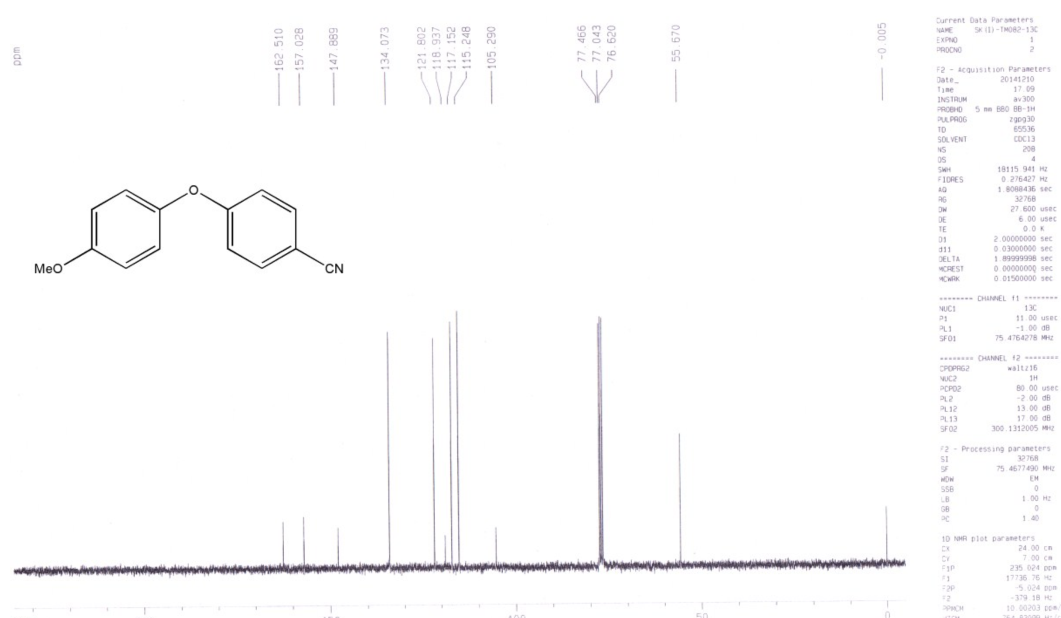
10; δ_H (300 MHz; $CDCl_3$) 2.36 (3H, s), 6.85-7.05 (5H, m), 7.29 (1H, t, J = 7.6 Hz), 7.57-7.59 (2H, m); δ_C (75 MHz; $CDCl_3$) 21.37, 105.68, 117.34, 117.90, 118.89, 121.04, 125.97, 129.95, 134.10, 140.57, 154.78, 161.79; Elemental analysis (found: C 80.36, H 5.30, N 6.69; for $C_{14}H_{12}O_2$ requires: C 80.32, H 5.28, N 6.62%).

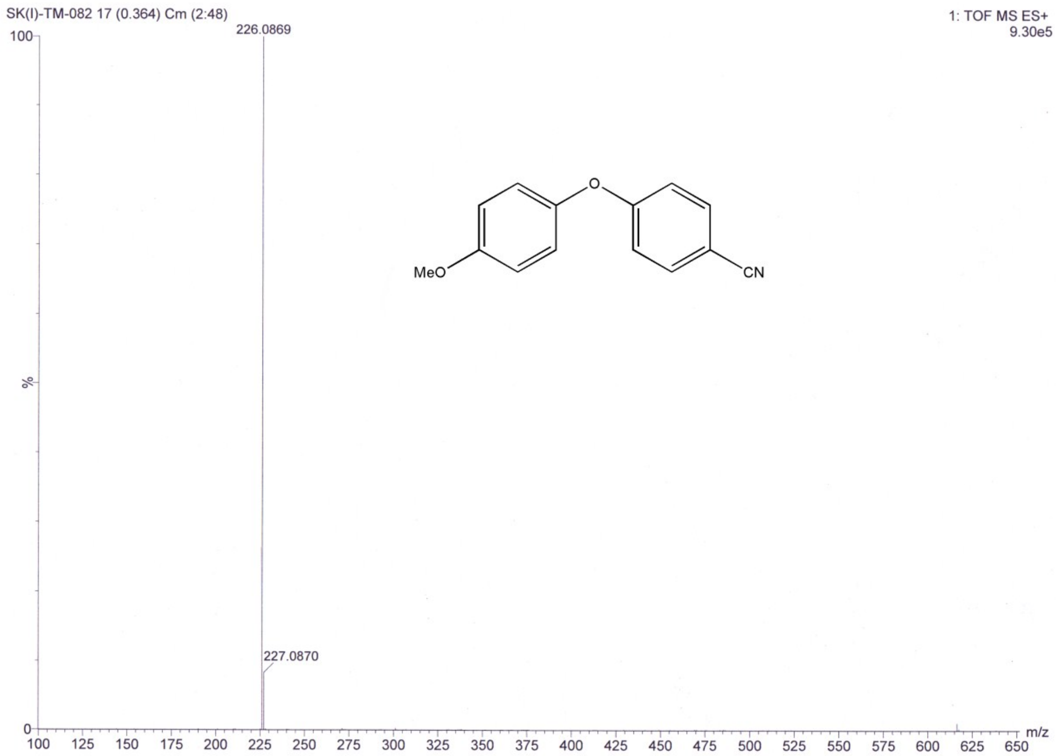




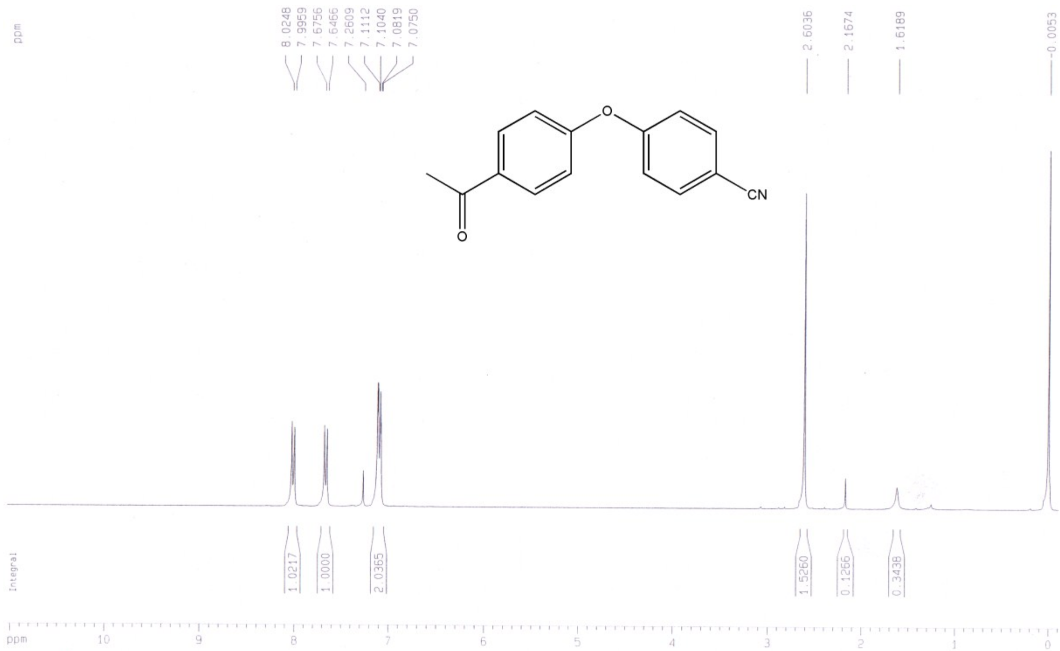
11; δ_H (300 MHz; $CDCl_3$) 3.83-3.85 (3H, m), 6.91-7.02 (6H, m), 7.54-7.58 (2H, m); δ_C (75 MHz; $CDCl_3$) 55.67, 105.29, 115.25, 117.15, 118.94, 121.80, 134.07, 147.89, 157.03, 162.51; *m/z* 226.0869 [$(M+H)^+$, 100%]; Elemental analysis (found: C 74.65, H 4.92, N 6.22; for $C_{14}H_{12}O_2$ requires: C 74.62, H 4.97, N 6.20%).







12; δ_H (300 MHz; $CDCl_3$) 2.60 (3H, s), 6.99-6.94 (4H, m), 7.09 (4H, dd, J = 2.1 and J = 8.7 Hz), 7.66 (2H, d, J = 8.7 Hz), 8.01 (2H, d, J = 8.7 Hz); δ_C (75 MHz; $CDCl_3$) 26.54, 107.32, 118.47, 119.24, 130.84, 133.65, 134.36, 159.36, 160.06, 196.57; *m/z* 238.0817 [$(M+H)^+$, 100%]; Elemental analysis (found: C 75.94, H 4.67, N 5.90; for $C_{14}H_{12}O_2$ requires: C 75.98, H 4.69, N 5.93%).



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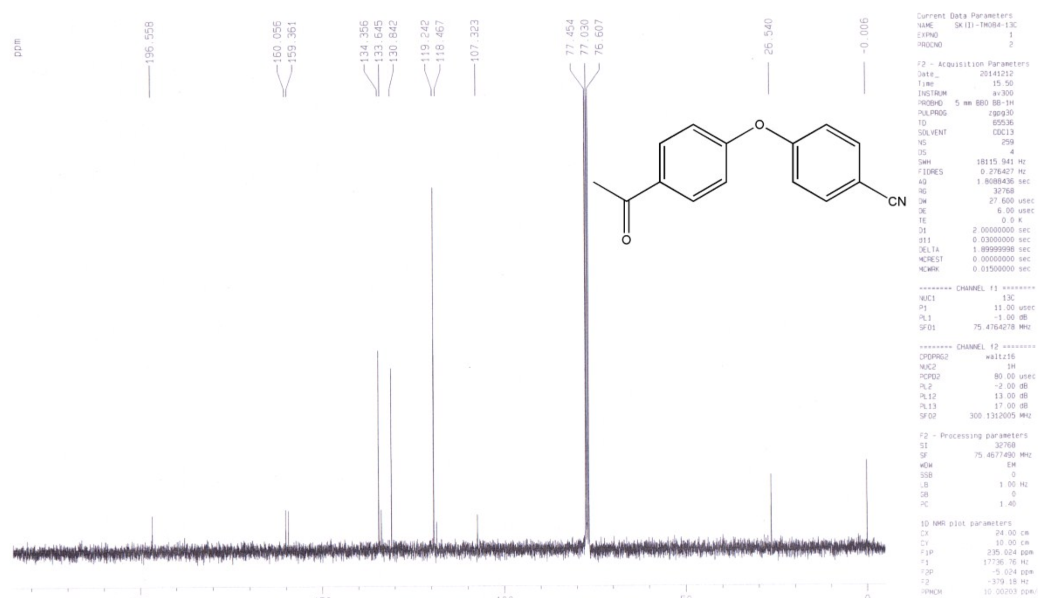
7.6756
7.6466
7.2609
7.1112
7.1040
7.0819
7.0750
2.6036
2.1674
1.6189
-0.0053
CN
O
Integral
1.0217
1.0000
2.0365
1.5260
0.1266
0.3438
ppm
10
9
8
7
6
5
4
3
2
1
0



ppm
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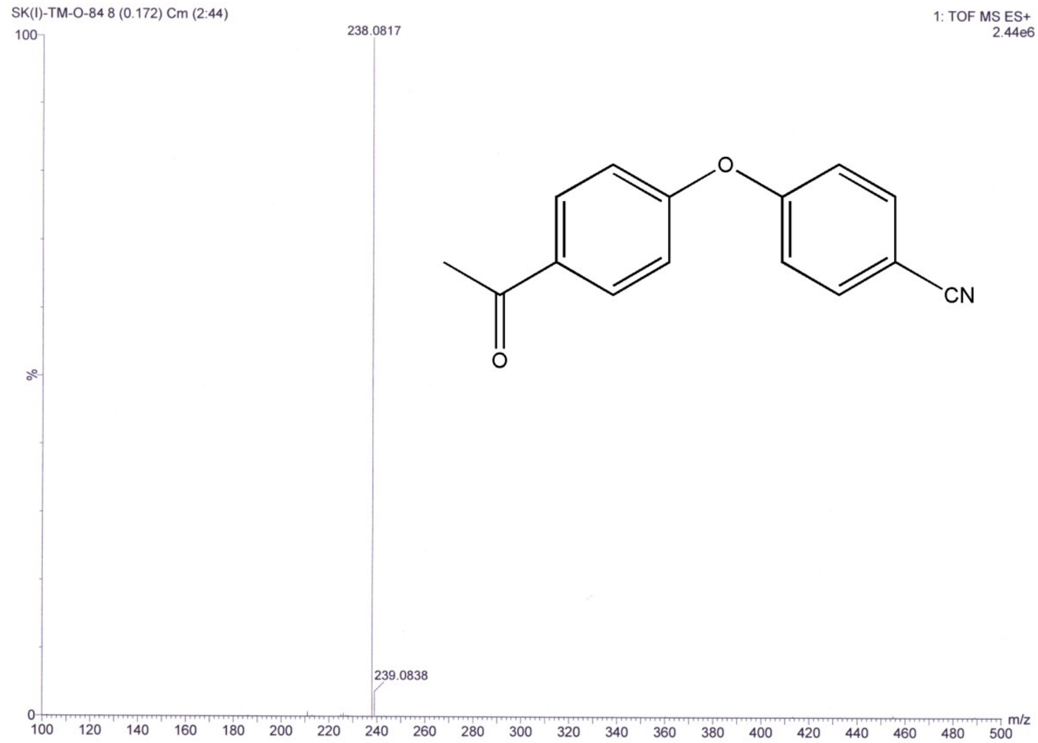
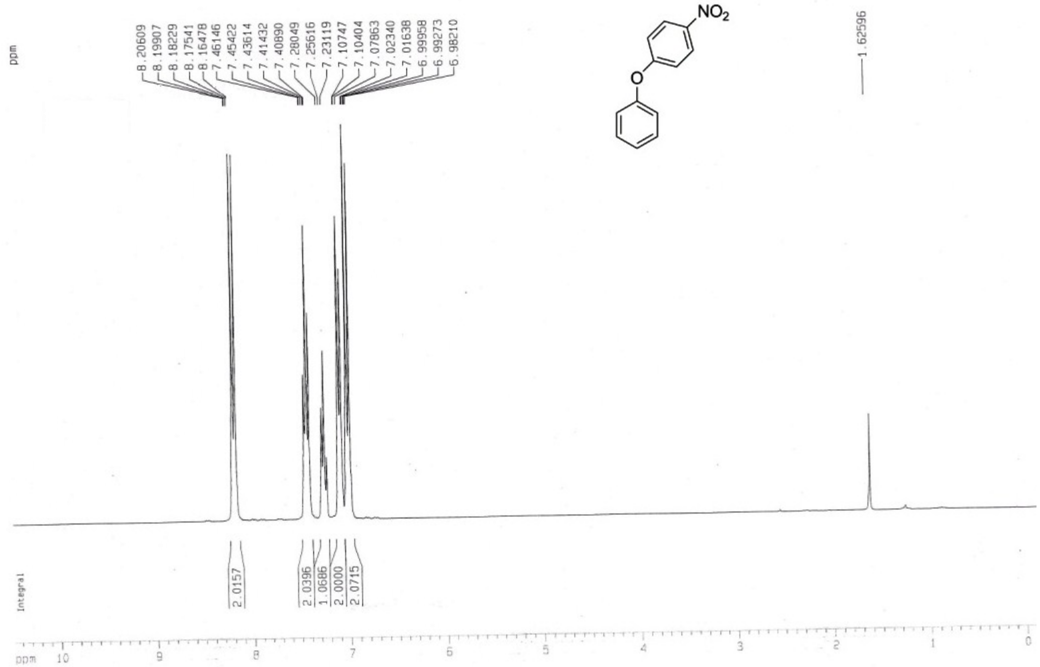
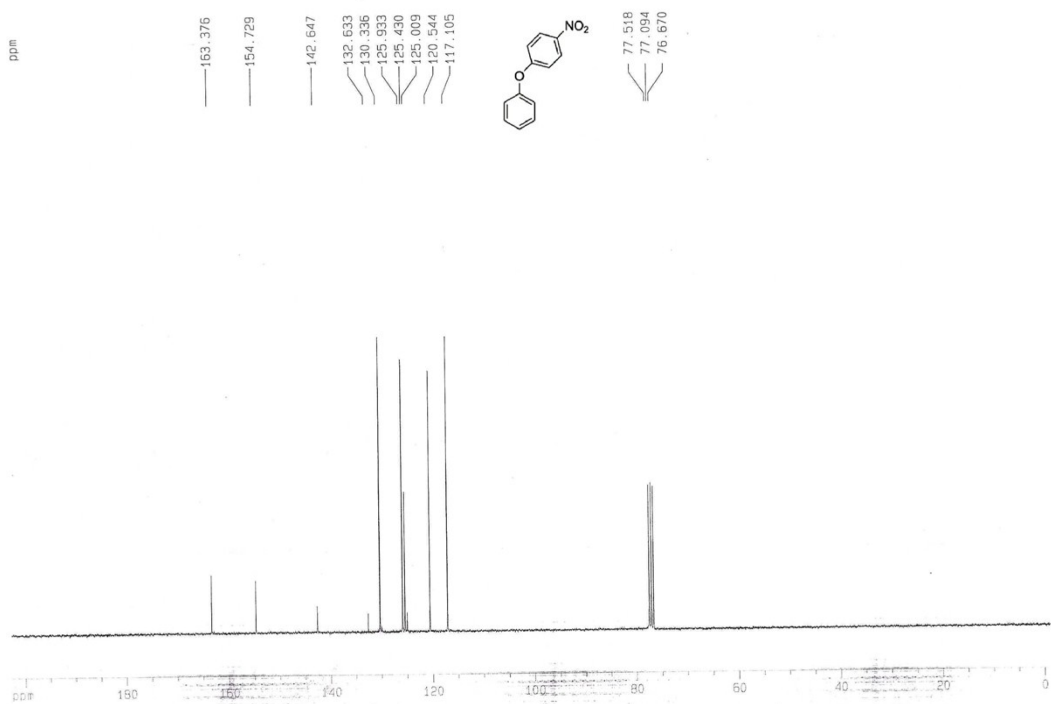
159.361
134.356
133.645
130.842
119.242
118.467
107.323
77.454
77.030
76.607
26.540
-0.006
CN
O
ppm
200
150
100
50
0
Current Data Parameters
NAME SK(I)-TM084-13C
EXPNO 1
PROCNO 2
F2 - Acquisition Parameters
Date_ 20141212
Time 15.50
INSTRUM av300
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 259
DS 4
SWH 18115.941 Hz
FIDRES 0.276427 Hz
AQ 1.8088436 sec
RG 32768
DW 27.600 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 75.4764278 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 235.024 ppm
F1 17736.76 Hz
F2P -5.024 ppm
F2 -379.18 Hz
PPMCM 10.00203 ppm/cm
HZCM 754.83099 Hz/cm

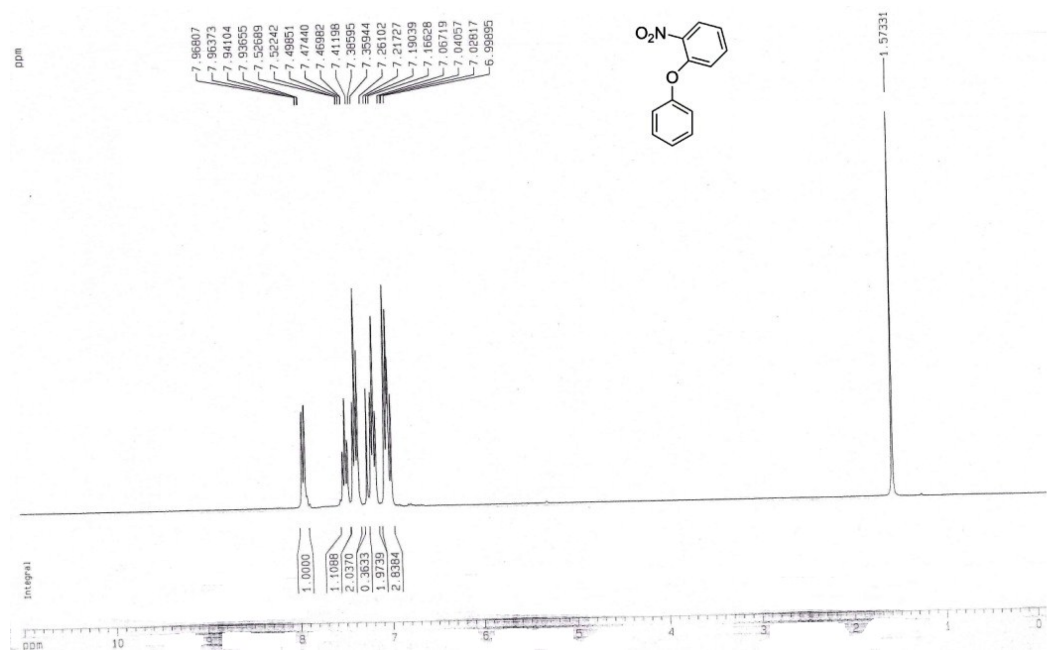


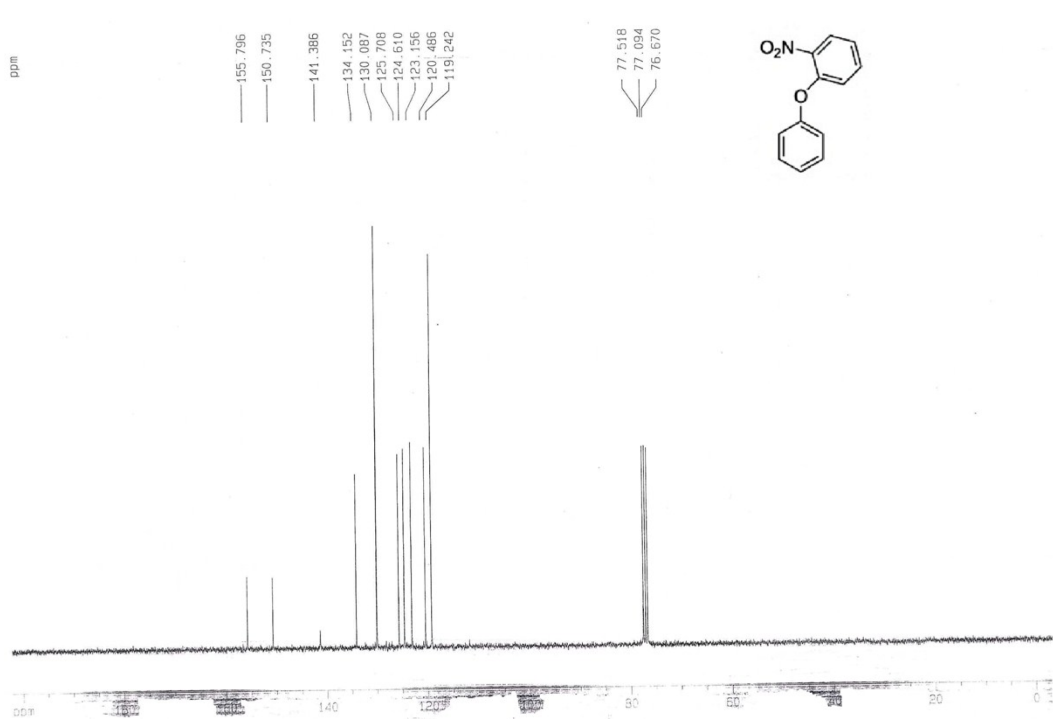
13; δ_H (300 MHz, $CDCl_3$, ppm) 6.98–7.11 (m, 4H), 7.23–7.28 (m, 1H), 7.41–7.46 (m, 2H), 8.16–8.20 (m, 2H); δ_C (75 MHz; $CDCl_3$) 117.11, 120.54, 125.43, 125.93, 130.34, 132.63, 142.65, 154.73, 163.38; Elemental analysis (found: C, 66.93; H, 4.24; N, 6.49. for $C_{12}H_9NO_3$ requires: C, 66.97; H, 4.22; N, 6.51%).



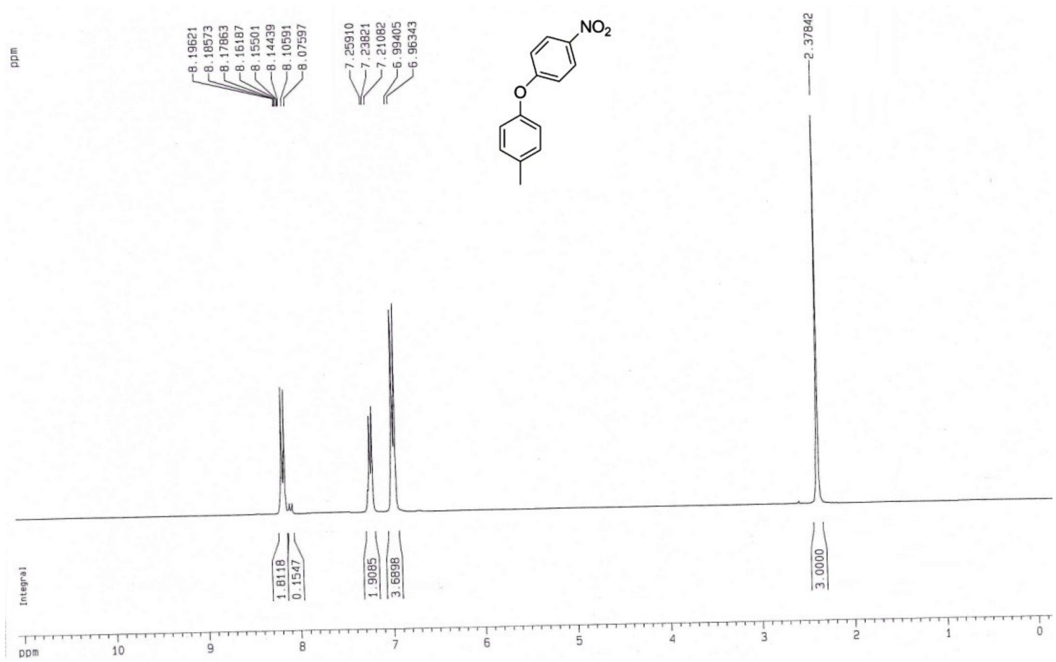


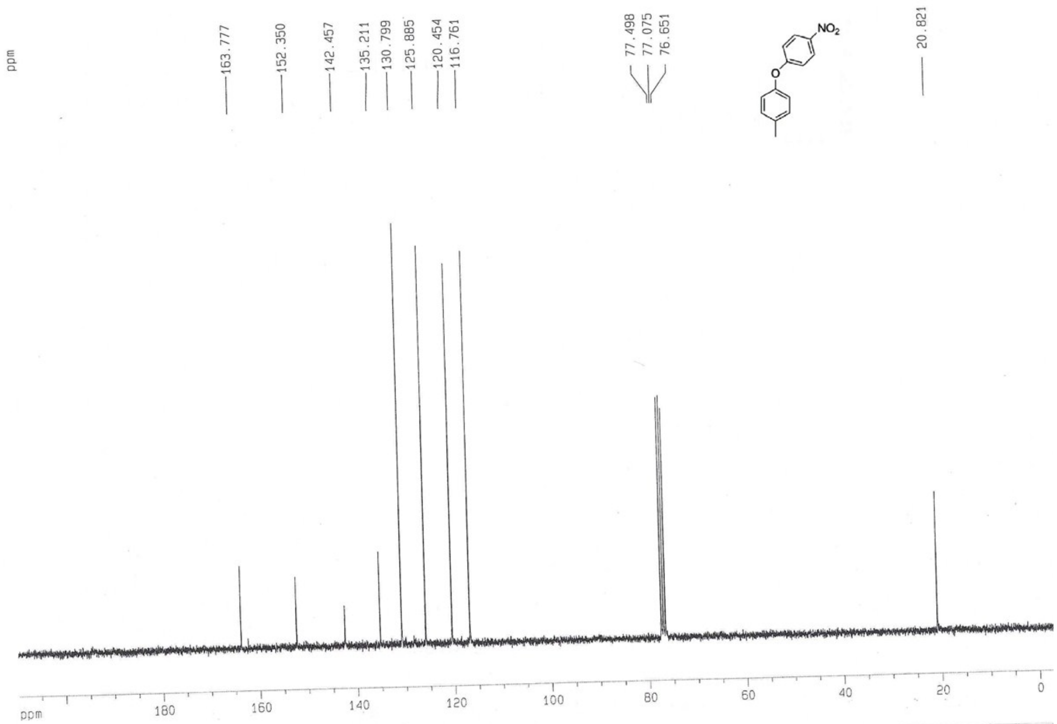
14; δ_H (300 MHz, $CDCl_3$, ppm) 6.99–7.07 (m, 3H), 7.17–7.22 (m, 2H), 7.39 (t, J = 7.9 Hz, 2H), 7.47–7.53 (m, 1H), 7.94–7.97 (m, 1H); δ_C (75 MHz; $CDCl_3$) 117.11, 120.54, 125.43, 125.93, 130.34, 132.63, 142.65, 154.73, 163.38; Elemental analysis (found: C, 66.98; H, 4.24; N, 6.52. for $C_{12}H_9NO_3$ requires: C, 66.97; H, 4.22; N, 6.51%).



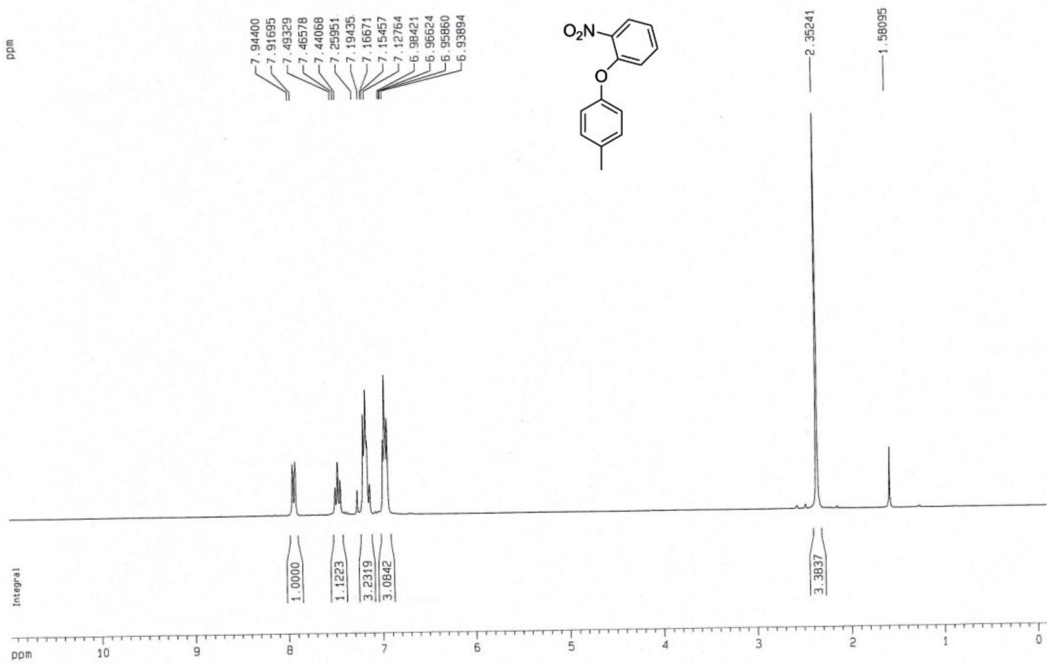


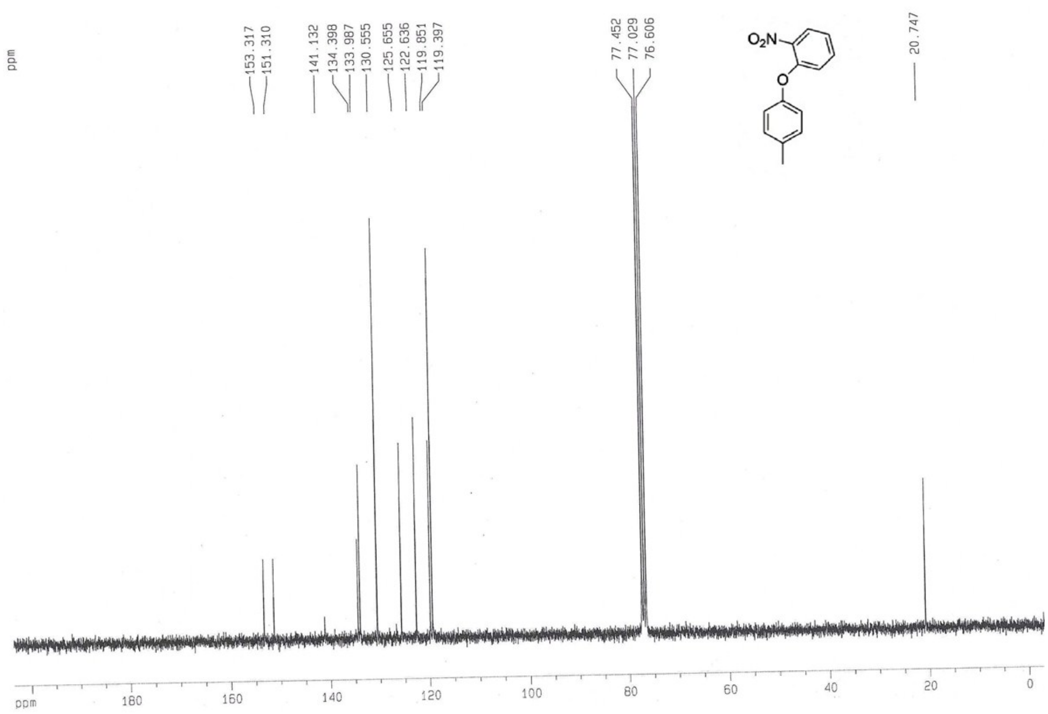
15; δ_H (300 MHz, $CDCl_3$, ppm) 2.38 (s, 3H), 6.98 (d, *J* = 9.2 Hz, 4H), 7.22 (d, *J* = 8.2 Hz, 2H), 8.16–8.19 (m, 2H); δ_C (75 MHz; $CDCl_3$) 20.82, 116.76, 120.45, 125.89, 125.93, 130.79, 135.21, 142.46, 152.35, 163.78; Elemental analysis (found: C, 68.13; H, 4.86; N, 6.12. for $C_{13}H_{11}NO_3$ requires: C, 68.11; H, 4.84; N, 6.11%).





16; δ_H (300 MHz, $CDCl_3$, ppm) 2.35 (s, 3H), 6.94–6.98 (m, 3H), 7.13–7.19 (m, 3H), 7.47 (m, 1H), 7.93 (d, *J* = 8.3 Hz, 1H); δ_C (75 MHz; $CDCl_3$) 20.75, 119.39, 119.85, 122.64, 125.66, 130.56, 133.99, 134.39, 141.13, 151.31, 153.32; Elemental analysis (found: C, 68.15; H, 4.85; N, 6.14. for $C_{13}H_{11}NO_3$ requires: C, 68.11; H, 4.84; N, 6.11%).





17; δ_H (300 MHz, $CDCl_3$, ppm) 3.83 (s, 3H), 6.93–7.03 (m, 6H), 8.17 (d, J = 9.2 Hz, 2H); δ_C (75 MHz; $CDCl_3$) 55.67, 115.31, 116.38, 121.83, 125.89, 142.34, 147.86, 157.19, 164.17; Elemental analysis (found: C, 63.69; H, 4.54; N, 5.73. for $C_{13}H_{11}NO_4$ requires: C, 63.67; H, 4.52; N, 5.71%).