Electronic Supplementary Material (ESI) for RSC Advances.

A Quinoxaline-based Porous Organic Polymer Containing Copper Nanoparticles CuNPs@Q-POP as a Robust Nanocatalyst toward C-N Coupling Reaction

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Experimental Section

Materials and instruments:

All chemicals were purchased from commercial sources and used without further purification. Solvents used in this study were dried and purified using standard procedures before usage. The powder X-ray diffraction (XRD) patterns of samples were performed on a D8 Advance Bruker X-ray diffractometer using Cu Kα (λ=1.54 °A) radiation. Nitrogen sorption isotherms were carried out using a BEL sorb-mini 2 at 77 K. The specific surface areas and the pore size distributions of materials were determined by the Brunauer-Emmett-Teller (BET) method and the Barrett-Joyner-Halenda (BJH) model, respectively. Before analysis, the samples were outgassed at 120 °C for 4h under vacuum. The Fourier transform infrared (FT-IR) spectra were recorded on Jasco FTIR-6300spectrometer as KBr pellets. Thermal gravimetric analysis (TGA) was obtained using an SDT Q600 instrument by heating samples from 25 to 600 °C in a dynamic argon atmosphere with a heating rate of 10°C min^{-1}. Field-emission scanning electron (FE-SEM) microscopy and energy-dispersive X-ray spectroscopy (EDX) analysis were carried out on a Tescan Mira3 scanning electron microscope. Transmission electron microscopy (TEM) images were performed on Philips-CM120. Inductively coupled plasma (ICP) was measured on an Analytic Jena PQ9000 for the determination of Cu content. Conversion and selectivity were determined by GC-FID on a Shimadzu GC-16A instrument using a 2 m column packed with silicon DC-200 or carbowax 20 M using EtOAc-Hexane (1:5) as eluent. ^{1}H NMR (400 MHz) and ^{13}C NMR (100 MHz) spectra were recorded on a Bruker Avance 400 MHz NMR spectrometer.

Synthesis of bis(salicylidene)-*O*-phenylene diamine (A):

This compound was prepared according to the procedure reported in the literature. *O*-phenylenediamine (2.16 g, 20 mmol) and salicylaldehyde (4.24 mL, 40 mmol) were dissolved in dry methanol (40 mL) at room temperature. After stirring for 16 h, the resulting solid was separated by filtration. The obtained solid was washed with methanol and dried *in vacuo* to afford compound (**A**) as yellow powders (6 g, 95%).

Synthesis of 2,3-di(2-hydroxyphenyl)1,2-dihydro quinoxaline (B):

Under an argon atmosphere, NaCN (0.226 g, 4.6 mmol) was introduced into a solution of bis(salicylidene)-*O*-phenylenediamine (3.7 g, 11.8 mmol) in dry DMF (35 mL), and the mixture was stirred at room temperature for 48 h. Subsequently, the reaction mixture was poured into ice water (50 mL). The resultant solid was filtered *via* Buchner funnel, washed with water for several times and dried. The crude product was purified by recrystallization in acetonitrile. Finally, the pure desired product was obtained as an orange solid (2.96 g, 80%). ^{1}H-NMR (400 MHz, $CDCl_3$): δ 14.92 (s, 1H), 10.18 (s, 1H), 7.44 (d, *J*=8 Hz, 1H), 7.33-7.29 (m, 1H), 7.24 (d, *J*=8 Hz, 1H), 7.08-7.04 (m, 1H), 7.01-6.96 (m, 1H), 6.92-6.86 (m, 4H), 6.81-

6.77 (m, 1H), 6.66 (s, 1H), 6.64 (s, 1H), 6.62-6.59 (m, 1H), 6.225 (d, *J*=4 Hz, 1H); ^{13}C-NMR (100 MHz, $CDCl_3$): δ 161.7, 161.1, 152.6, 136.8, 132.6, 129.3, 129.1, 128.9, 127.4, 127.0, 125.7, 119.4, 118.5, 117.5, 117.2, 117.1, 115.7, 114.1, 45.5.

Synthesis of allyl-functionalized 2,3-di(2-hydroxy phenyl)1,2-dihydroquinoxaline (C):

A solution of 2,3-di(2-hydroxyphenyl)1,2-dihydro quinoxaline (2.96 g, 9.4 mmol) in acetone (60 mL) was shaken with K_2CO_3 (3.9 g, 28.2 mmol). Allyl bromide (2.44 mL, 28.2 mmol) was then added slowly to the solution. After the mixture was stirred at room temperature for 48 h, water was added (20 mL) and the product was extracted with ethyl acetate (3×30 mL) and dried over $MgSO_4$. The solvent was evaporated under vacuum and the crude product was purified by column chromatography on silica gel (EtOAc/hexane 1:4) to yield allyl functionalized 2,3-di(2-hydroxyphenyl)1,2-dihydroquinoxaline monomer as an orange powder (1.86 g, 50%). ^{1}H-NMR (400 MHz, $CDCl_3$): δ 14.98 (s, 1H), 7.31 (d, *J*=8 Hz, 1H), 7.29 (d, *J*=8 Hz, 1H), 7.17-7.02 (m, 4H), 6.88 (d, *J*=8 Hz, 1H), 6.79 (d, *J*=8 Hz, 1H), 6.71-6.64 (m, 3H), 6.55 (d, *J*=8 Hz, 1H), 6.30 (s, 1H), 6.14-6.07 (m, 1H), 5.61-5.54 (m, 1H), 5.46 (dd, *J*=18 Hz, *J*=4 Hz, 1H), 5.32 (dd, *J*=10 Hz, *J*=4 Hz, 1H), 5.03 (dd, *J*=18 Hz, *J*=4 Hz, 1H), 4.97 (dd, *J*=12 Hz, *J*=4 Hz, 1H), 4.63-4.62 (m, 2H), 3.92-3.91 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 161.5, 160.0, 152.3, 135.8, 132.6, 131.9, 131.3, 129.9, 128.9, 128.1, 127.9, 126.4, 126.3, 125.5, 120.8, 117.2, 116.9, 116.6, 116.5, 115.5, 111.3, 110.5, 68.0, 50.6, 50.5.

Synthesis of Q-POP:

Radical copolymerization of ligand **C** with divinylbenzene (DVB) as cross-linker, in the presence of azobisisobutyronitrile (AIBN) as a radical initiator, was used to synthesis the nanoporous polymer under solvothermal conditions. Typically, the monomer **C** (1 g, 2.52 mmol) and divinylbenzene (1.43 mL, 10.1 mmol) were dissolved in dry DMF (10 mL), followed by the addition of AIBN (0.05 g). The reaction mixture was degassed with argon gas for 30 min, poured into a 50 mL autoclave (stainless steel 316), and heated at 100 °C for 24 h. After cooling to room temperature, water (30 mL) was added to the reaction mixture and the resulting polymer was filtered and washed with excess water. Additionally, the as-prepared polymer was further washed with methanol for the elimination of any unreacted monomer **C**. Finally, the desired polymer was obtained as a yellow powder and dried in an oven at 90-100 °C overnight.

Synthesis of CuNPs@Q-POP(7.3% Cu):

To a round-bottom flask were introduced Q-POP (1.5 g), MeOH (50 mL), and Et_3N (1 mL). The resultant suspension was allowed to stir at room temperature for 1 h. Then, $Cu(NO_3)_2.3H_2O$ (0.8 g) in MeOH (20 mL) was added to the above suspension and refluxed for about 12 h. The green Cu^{2+}@Q-POP was isolated by filtration, washed with MeOH for removal of excess $Cu(NO_3)_2.3H_2O$, and then dried *in vacuo* for

overnight. Next, CuNPs@Q-POP were synthesized by the reduction of Cu^{2+}@Q-POP. An aqueous solution of hydrazine hydrate (10 mL, 35%) was added dropwise with vigorous stirring under argon atmosphere to a mixture of Cu^{2+}@Q-POP (1.5 g), EtOH (17.5 mL), H_2O (10 mL), and NH_4OH (17.5 mL, 25%). Afterward, the mixture was transferred into a 100 mL stainless steel autoclave and treated at 100 ˚C for 4 h. Finally, the mixture was filtered and washed with water and methanol successively. The resultant brown solid was dried under a vacuum to obtain CuNPs@Q-POP. Inductively coupled plasma (ICP) analysis displayed a Cu content of 7.3% in the CuNPs@Q-POP.

Synthesis of CuNPs@Q-POP(2.8% Cu):

To a round-bottom flask, Q-POP (1.5 g) was suspended in a mixture of MeOH (50 mL) and Et_3N (1 mL). The resultant suspension was allowed to stir at room temperature for 1 h. Then, $Cu(NO_3)_2.3H_2O$ (0.2 g) in MeOH (6 mL) was added and refluxed for about 12 h and the resultant green Cu^{2+}@Q-POP precipitate was filtered, washed with MeOH, and dried *in vacuo* overnight. Next, an aqueous solution of hydrazine hydrate (5 mL, 35%) was added dropwise with intense stirring under argon atmosphere to the mixture of Cu^{2+}@Q-POP (1.5 g), EtOH (17.5 mL), H_2O (10 mL), and NH_4OH (17.5 mL, 25%). Then, the mixture was heated at 100 ˚C for 4 h in an appropriate stainless steel autoclave. Finally, the mixture was filtered and successively washed with water and methanol. Thereafter, the brown solid was dried under vacuum to obtain CuNPs@Q-POP. Inductively coupled plasma (ICP) analysis revealed that the Cu content of CuNPs@Q-POP was 2.8%.

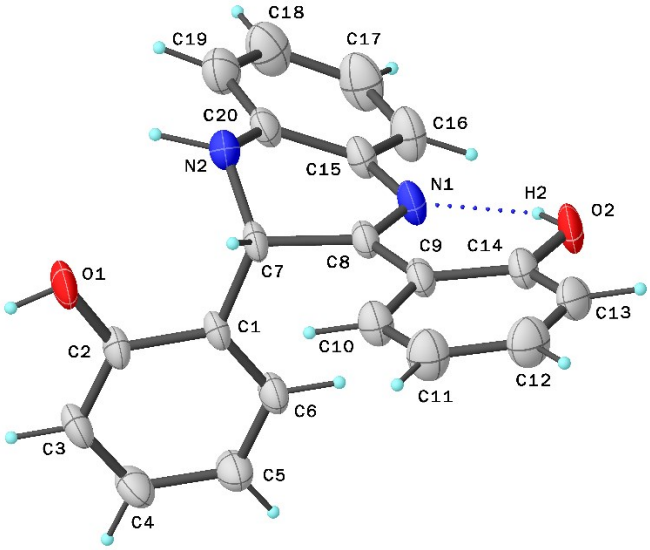
General procedure for N-arylation of anilines with aryl halides catalyzed by CuNPs@Q-POP(2.8% Cu):

In a 25 mL round bottom flask (two necked-flask) a mixture of aryl halide (1 mmol), aniline (1.2 mmol), K_2CO_3 (2 mmol), PEG-200 (2 mL), and CuNPs@Q-POP(2.8% Cu) catalyst (75 mg) was stirred under nitrogen atmosphere at 110 ˚C for 24. After the reaction, ethyl acetate (10 mL) was added to the reaction mixture and the catalyst was separated by filtration and washed with ethyl acetate. Then, water (10 mL) was added to the filtrate and further extracted with ethyl acetate (2 × 10 mL). The extracted organic phases were combined and dried with anhydrous $MgSO_4$. The organic phase was analyzed by GC to determine conversion and selectivity. The conversions were assessed according to the concentration of iodobenzene. Then the solvent was removed under reduced pressure and the crude product was purified by column chromatography over silica gel to obtain the desired product. The product was analyzed by ^{1}H-NMR and ^{13}C-NMR.

General procedure for recyclability of the catalyst.

To examine the recyclability of CuNPs@Q-POP, the *N*-arylation of iodobenzene with aniline was perfomed under the similar condition as mentioned above. After the completion of the reaction, the catalyst was separated from the mixture reaction by filtration and washed three times with water, ethyl acetate, and methanol, then dried under vacuum at 100˚C for 12h. Finally, the reused catalyst was exerted for the next run under optimum condition.

CCDC Number: 1953774



ORTEP representation of compound **B**.

Table 1. Crystal data and structure refinement for compound B.

Identification code	shelx
Empirical formula	C20 H16 N2 O2
Formula weight	316.35
Temperature	298(2) K
Wavelength	0.71073 A

Crystal system, space group	Monoclinic, P 21/c
Unit cell dimensions	a = 9.7420(19) A alpha = 90 deg.
	b = 16.082(3) A beta = 115.05(3) deg.
	c = 11.405(2) A gamma = 90 deg.
Volume	1618.6(7) A^3
Z, Calculated density	4, 1.298 Mg/m^3
Absorption coefficient	0.085 mm^-1
F(000)	664
Theta range for data collection	2.533 to 24.998 deg.
Limiting indices	-11<=h<=11, -18<=k<=19, -13<=l<=13
Reflections collected / unique	10238 / 2860 [R(int) = 0.1897]
Completeness to theta = 24.998	99.9 %
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2860 / 1 / 226
Goodness-of-fit on F^2	0.827
Final R indices [I>2sigma(I)]	R1 = 0.0721, wR2 = 0.1622
R indices (all data)	R1 = 0.1269, wR2 = 0.1852
Largest diff. peak and hole	0.227 and -0.311 e.A^-3

Table 1. Bond lengths [A] and angles [deg] for compound B.

O(1)-C(2)	1.358(4)
O(1)-H(1)	0.8200
O(2)-C(14)	1.345(4)
O(2)-H(2)	0.821(2)
N(1)-C(8)	1.295(4)

N(1)-C(15)	1.397(4)
C(8)-C(9)	1.460(4)
C(8)-C(7)	1.525(4)
C(7)-N(2)	1.467(4)
C(7)-C(1)	1.524(4)
C(1)-C(6)	1.387(4)
C(1)-C(2)	1.416(4)
C(9)-C(10)	1.408(5)
C(9)-C(14)	1.425(4)
N(2)-C(20)	1.388(4)
N(2)-H(2A)	0.99(4)
C(3)-C(4)	1.368(5)
C(3)-C(2)	1.383(5)
C(6)-C(5)	1.372(5)
C(15)-C(16)	1.391(5)
C(15)-C(20)	1.406(5)
C(13)-C(12)	1.368(6)
C(13)-C(14)	1.376(5)
C(10)-C(11)	1.372(5)
C(20)-C(19)	1.375(5)
C(4)-C(5)	1.402(5)
C(16)-C(17)	1.364(6)
C(12)-C(11)	1.402(6)
C(19)-C(18)	1.377(6)

C(18)-C(17)	1.384(6)
C(8)-N(1)-C(15)	120.1(3)
N(1)-C(8)-C(9)	118.3(3)
N(1)-C(8)-C(7)	120.1(3)
C(9)-C(8)-C(7)	121.6(3)
N(2)-C(7)-C(1)	113.8(3)
N(2)-C(7)-C(8)	106.0(2)
C(1)-C(7)-C(8)	111.5(2)
C(6)-C(1)-C(2)	118.4(3)
C(6)-C(1)-C(7)	124.5(3)
C(2)-C(1)-C(7)	117.0(3)
C(10)-C(9)-C(14)	117.8(3)
C(10)-C(9)-C(8)	121.6(3)
C(14)-C(9)-C(8)	120.6(3)
C(20)-N(2)-C(7)	115.9(3)
C(4)-C(3)-C(2)	120.6(3)
O(1)-C(2)-C(3)	123.9(3)
O(1)-C(2)-C(1)	116.5(3)
C(3)-C(2)-C(1)	119.6(3)
C(5)-C(6)-C(1)	122.0(3)
C(16)-C(15)-N(1)	120.4(3)
C(16)-C(15)-C(20)	119.5(3)
N(1)-C(15)-C(20)	119.8(3)
C(12)-C(13)-C(14)	121.5(4)

O(2)-C(14)-C(13)	120.0(3)
O(2)-C(14)-C(9)	120.4(3)
C(13)-C(14)-C(9)	119.6(3)
C(11)-C(10)-C(9)	121.3(3)
C(19)-C(20)-N(2)	123.6(3)
C(19)-C(20)-C(15)	119.5(3)
N(2)-C(20)-C(15)	116.7(3)
C(3)-C(4)-C(5)	120.7(3)
C(6)-C(5)-C(4)	118.7(3)
C(17)-C(16)-C(15)	120.1(4)
C(13)-C(12)-C(11)	119.9(4)
C(20)-C(19)-C(18)	120.1(4)
C(10)-C(11)-C(12)	119.7(4)
C(19)-C(18)-C(17)	120.5(4)
C(16)-C(17)-C(18)	120.1(4)

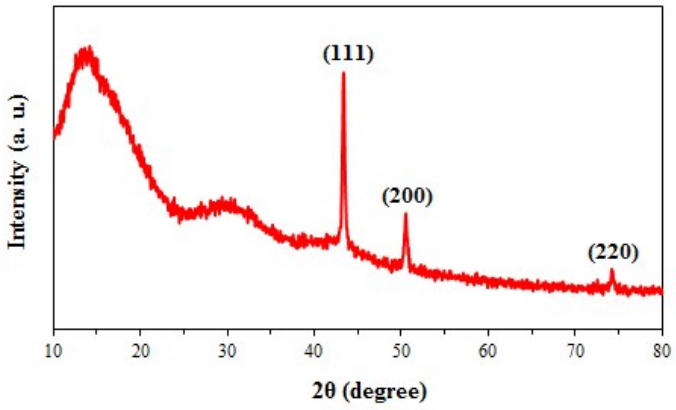


Figure S1. Wide angle powder XRD patterns of reused of CuNPs@Q-POP for *N*-arylation of iodobenzene with aniline.

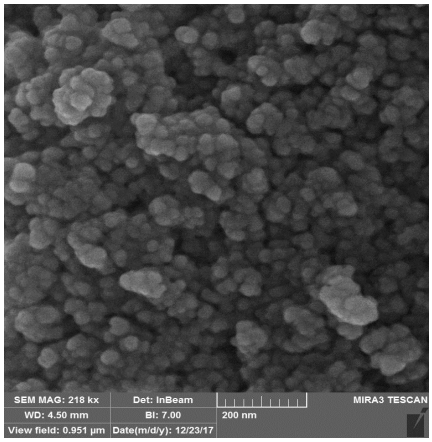
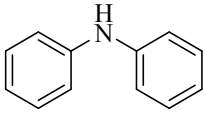


Figure S2. SEM image of reused CuNPs@Q-POP for *N*-arylation of iodobenzene with aniline.

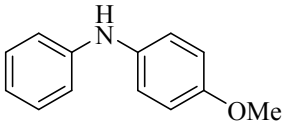
The NMR spectral data of the products

N-phenylaniline



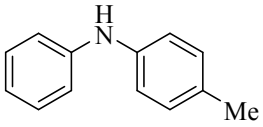
White solid, m.p. 52-53 ˚C; ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm)=7.33-7.28 (m, 4H), 7.12-7.10 (m, 4H), 6.99-6.95 (m, 2H), 5.73 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm)=142.6, 128.9, 120.5, 117.3.

N-(4-methoxyphenyl)aniline



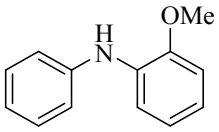
Pale yellow solid, m.p. 103 ˚C; ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm)= 7.17-7.12 (m, 2H), 7.03-6.99 (m, 2H), 6.86-6.74 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm)=155.3, 145.2, 135.7, 129.3, 122.2, 119.6, 115.6, 114.7, 55.6.

N-(4-methylphenyl)aniline



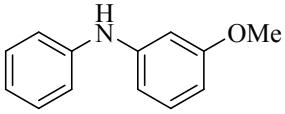
Pale yellow solid, m.p. 81 ˚C; ^{1}H NMR (400 MHz, CDCl3): δ (ppm)=7.17-7.15 (m, 2H), 7.03-7.01 (m, 2H), 6.95-6.92 (m, 4H), 6.83-6.79 (m, 1H), 5.53 (s, 1H), 2.23 (s, 3H); ^{13}C NMR (100 MHz, CDCl3): δ (ppm)=130.9, 129.8, 129.7, 129.3, 122.7, 120.3, 118.9, 116.9, 20.7.

_N_-(2-methoxyphenyl)aniline



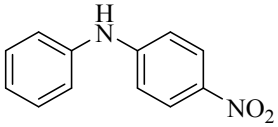
Yellowish oil; ^{1}H NMR (400 MHz, CDCl3): δ (ppm)=7.33-7.30 (m, 3H), 7.17 (d, *J*=8 Hz, 2H), 6.98-9.85 (m, 4H), 6.16 (s, 1H), 3.91 (s, 3H); ^{13}C NMR (100 MHz, CDCl3): δ (ppm)=129.3, 128.8, 121.13, 120.8, 119.9, 118.6, 114.6, 110.5, 55.6.

_N_-(3-methoxyphenyl)aniline



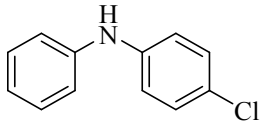
Yellowish oil; ^{1}H NMR (400 MHz, CDCl3): δ (ppm)=7.32-7.28 (m, 2H), 7.22-7.19 (m, 1H), 7.12 (d, *J*=8 Hz, 2H), 6.97 (t, *J*=8 Hz, 1H), 6.69-6.66 (m, 2H), 6.52-6.50 (m, 1H), 5.74 (s, 1H), 3.81 (s, 3H); ^{13}C NMR (100 MHz, CDCl3): δ (ppm)=160.2, 144.1, 142.3, 129.6, 128.8, 120.8, 117.8, 109.7, 105.6, 102.8, 54.7.

_N_-(4-nitrophenyl)aniline



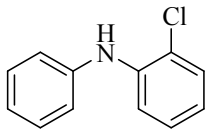
Yellow solid, m.p. 130 ˚C; ^{1}H NMR (400 MHz, CDCl3): δ (ppm)=8.05 (d, *J*=8 Hz, 2H), 7.34-7.30 (m, 2H), 7.15-7.08 (m, 3H), 6.87 (d, *J*=8 Hz, 2H), 6.21 (s, 1H); ^{13}C NMR (100 MHz, CDCl3): δ (ppm)=150.3, 139.7, 139.5, 129.7, 126.2, 124.7, 121.9, 113.7.

_N_-(4-chlorophenyl)aniline



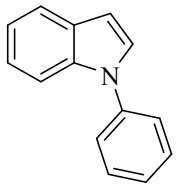
Pale yellow solid, m.p. 71-72 ˚C; ^{1}H NMR (400 MHz, CDCl3): δ (ppm)=7.23-7.20 (m, 2H), 7.14 (d, *J*=8 Hz, 2H), 6.99-6.97 (m, 2H), 6.92 (d, *J*=8 Hz, 2H), 6.88-6.87 (m, 1H); ^{13}C NMR (100 MHz, CDCl3): δ (ppm)=142.6, 141.9, 129.4, 129.3, 121.5, 118.8, 118.1.

_N_-(2-chlorophenyl)aniline



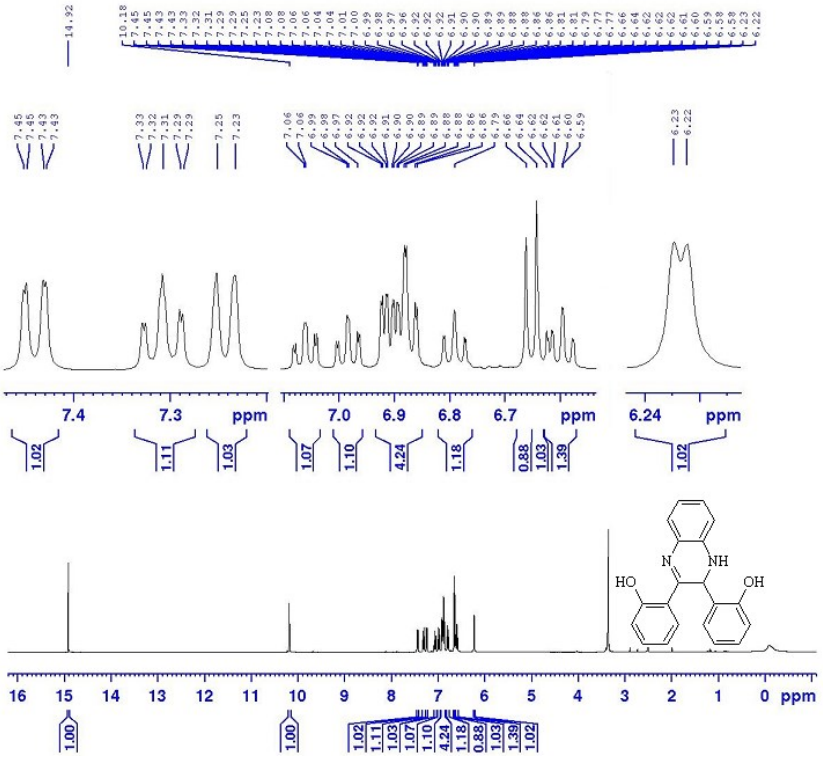
Yellowish oil; ^{1}H NMR (400 MHz, CDCl3): δ (ppm)=7.29-7.21 (m, 4H), 7.10-7.03 (m, 3H), 6.97 (t, *J*=8 Hz, 1H), 6.75-6.71 (m, 1H), 6.03 (s, 1H); ^{13}C NMR (100 MHz, CDCl3): δ (ppm)=141.5, 129.7, 129.4, 127.4, 122.6, 120.4, 120.2, 115.5.

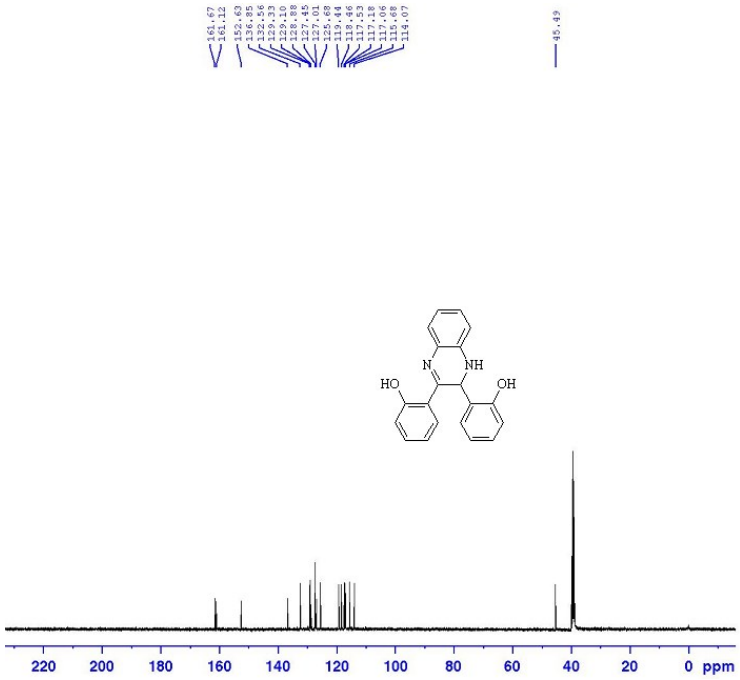
_N_-phenylindole

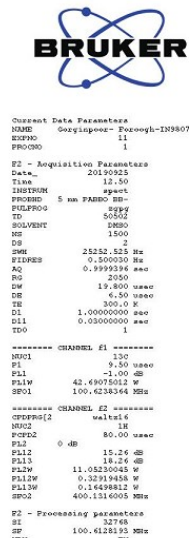


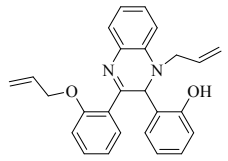
Colorless solid, m.p. 60-62 ˚C; ^{1}H NMR (400 MHz, CDCl3): δ (ppm)=7.79-7.76 (m, 1H), 7.66-7.64 (m, 1H), 7.59-7.56 (m, 4H), 7.44-7.40 (m, 2H), 7.33-7.25 (m, 2H), 6.77-6.76 (m, 1H); ^{13}C NMR (100 MHz, CDCl3): δ (ppm)=139.9, 135.9, 129.7, 129.4, 128.0, 126.5, 124.4, 122.4, 121.2, 120.4, 110.6, 103.6.

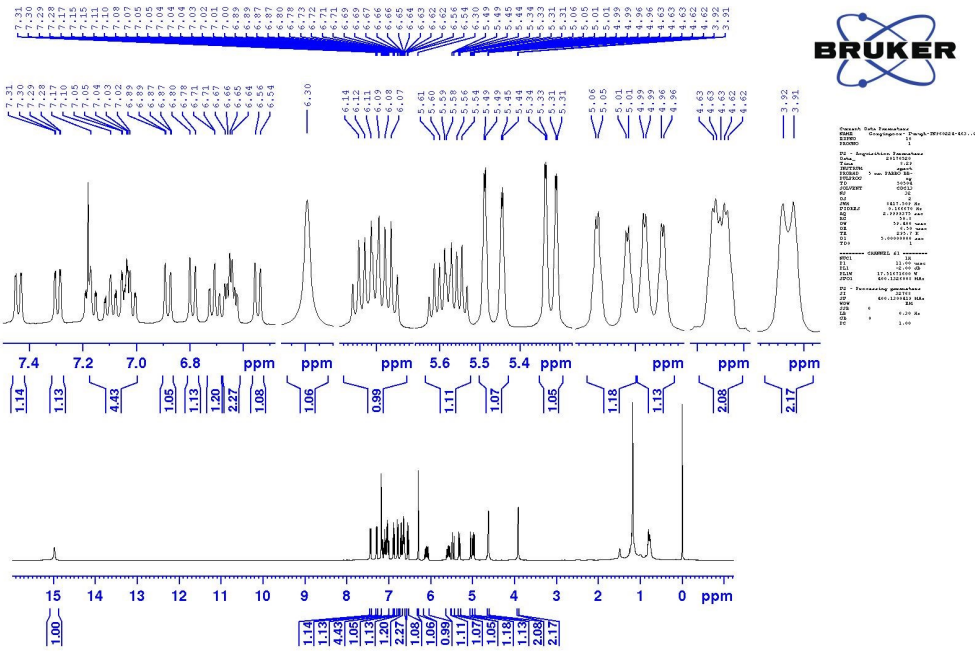
Copies of ^{1}H NMR and ^{13}C NMR spectra

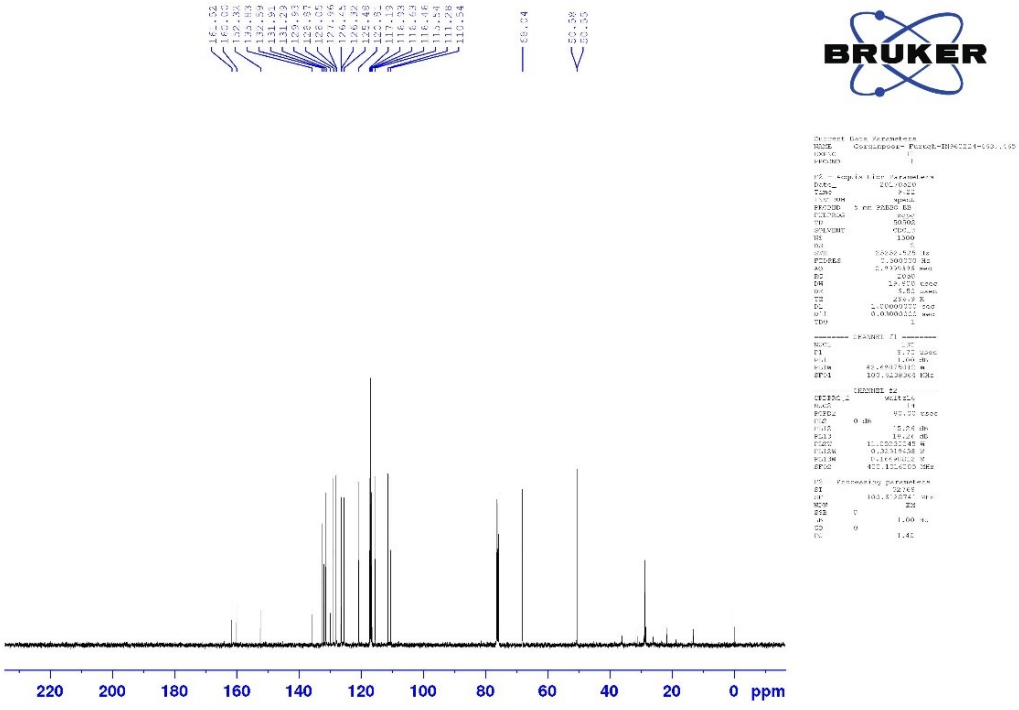


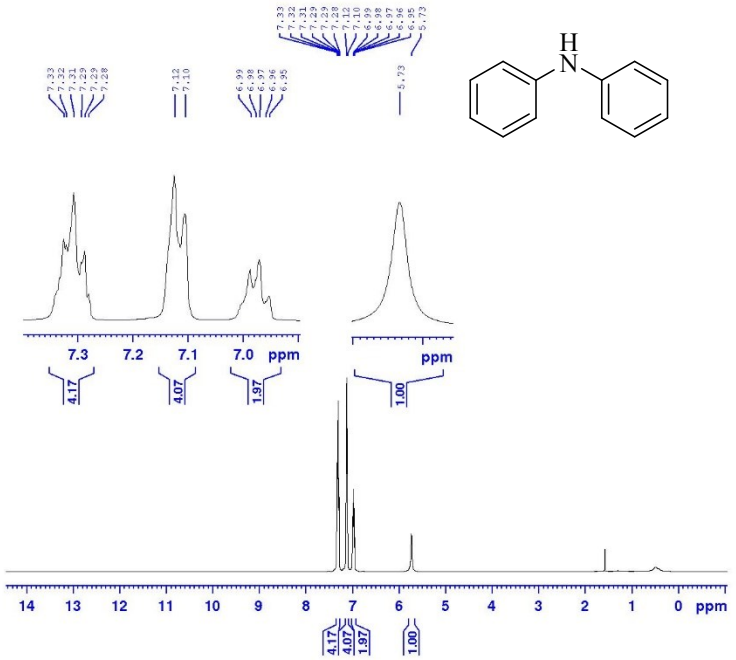




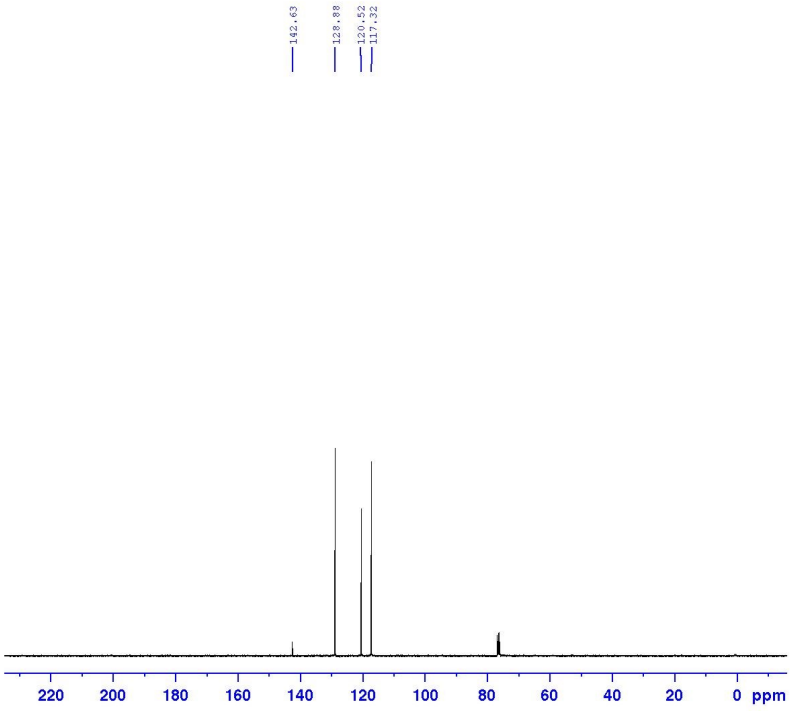

N
N
O
OH



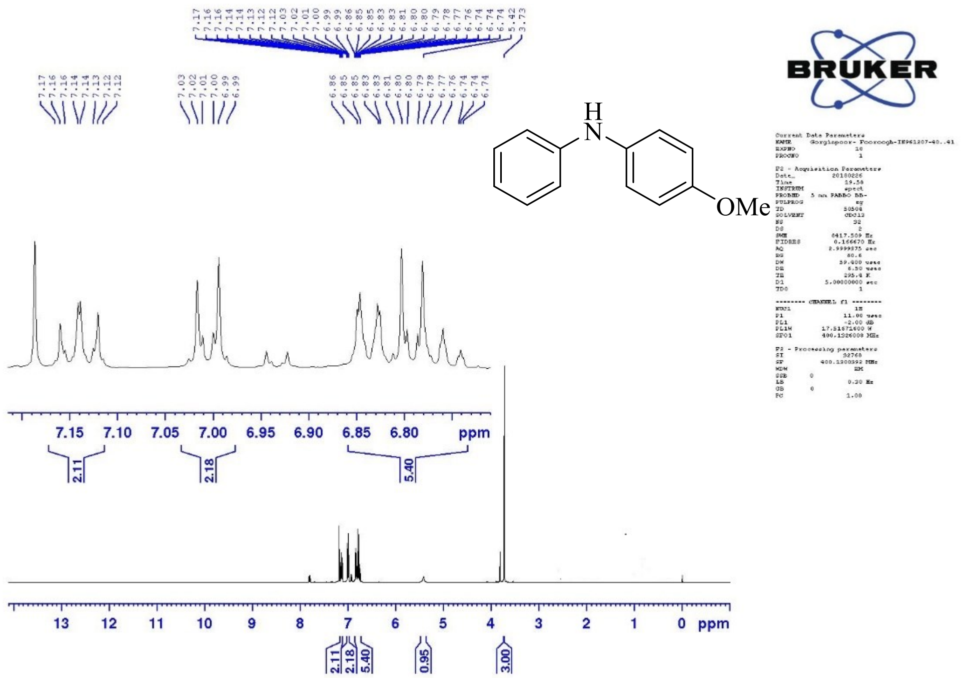


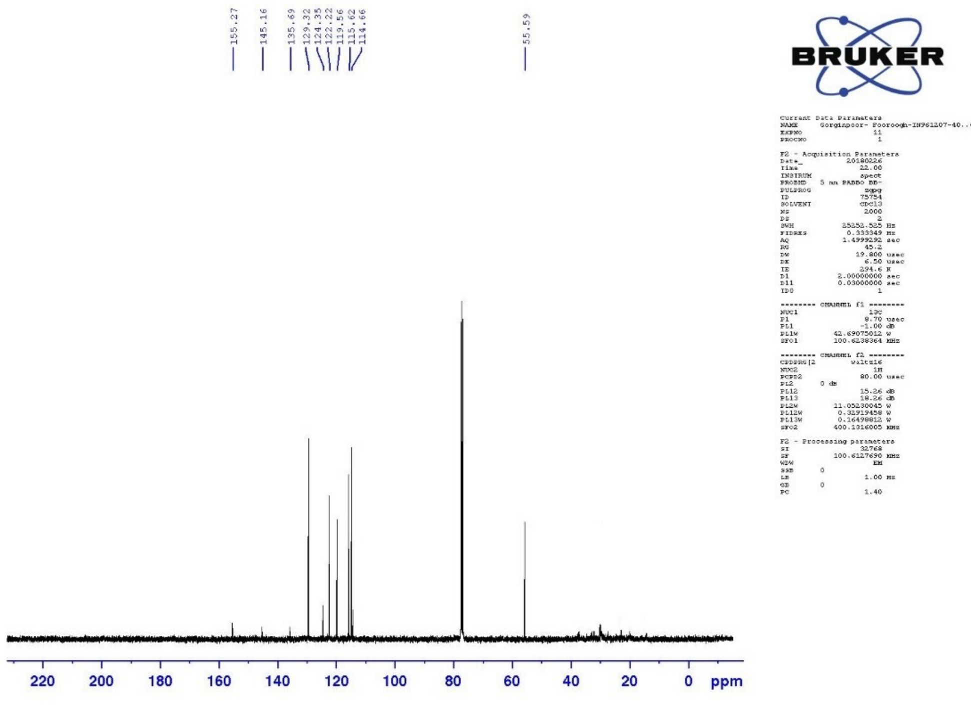


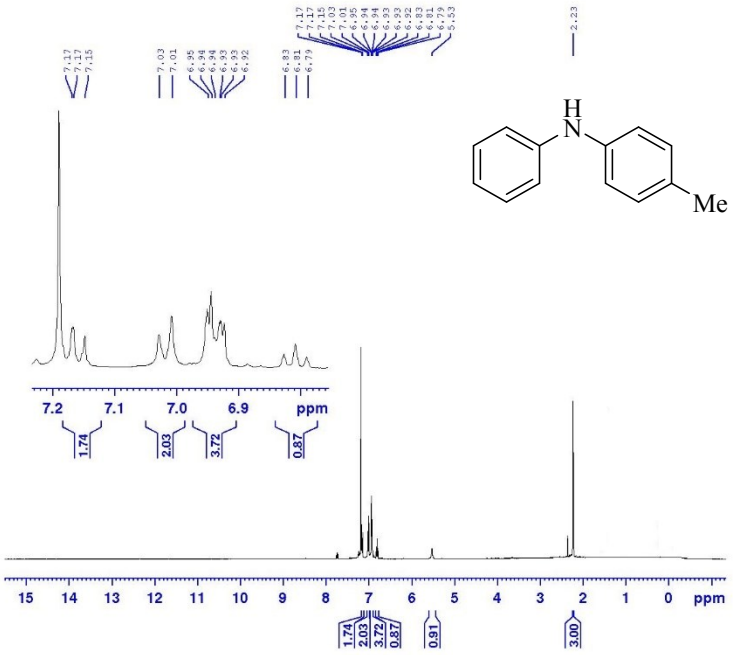

```
Current Data Parameters
NAME      Gorginpour- Furugh-IN980605
EXPNO              10
PROCNO              1

F2 - Acquisition Parameters
Date_          20190827
Time             14.08
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG             zg
TD                50504
SOLVENT           CDCl3
NS                   32
DS                    2
SWH            8802.817 Hz
FIDRES        0.174299 Hz
AQ            2.8686273 sec
RG                 50.8
DW               56.800 usec
DE                6.50 usec
TE               294.5 K
D1            5.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                  1H
P1                11.00 usec
PL1               -2.00 dB
PL1W        17.51671600 W
SFO1        400.1326008 MHz

F2 - Processing parameters
SI                32768
SF          400.1300021 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

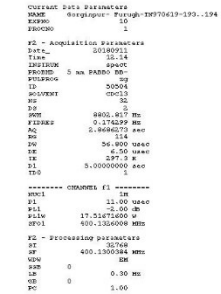


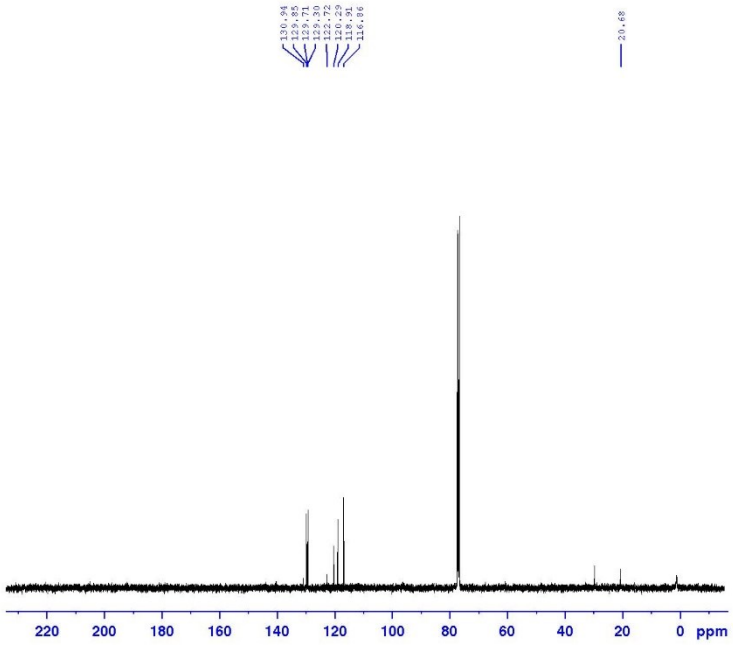

```
Current Data Parameters
NAME      Gorginpour- Furugh-IN980605
EXPNO              11
PROCNO              1

F2 - Acquisition Parameters
Date_          20190827
Time             14.02
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg
TD                50502
SOLVENT           CDCl3
NS                  948
DS                    2
SWH           25252.525 Hz
FIDRES        0.500030 Hz
AQ            0.9999396 sec
RG                 2050
DW               19.800 usec
DE                6.50 usec
TE               295.1 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                9.50 usec
PL1              -1.00 dB
PL1W        42.69075012 W
SFO1        100.6238364 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                  1H
PCPD2             80.00 usec
PL2             0 dB
PL12              15.26 dB
PL13              18.26 dB
PL2W        11.05230045 W
PL12W        0.32919458 W
PL13W        0.16498812 W
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6128193 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

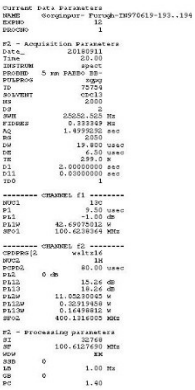

BRUKER
H
N
OMe
7.15 7.10 7.05 7.00 6.95 6.90 6.85 6.80 ppm
2.11
2.18
5.40
13 12 11 10 9 8 7 6 5 4 3 2 1 0 ppm
2.11
2.18
5.40
0.95
3.00


155.27
145.16
135.69
129.32
124.35
122.22
119.56
115.62
114.66
55.59
BRUKER
220 200 180 160 140 120 100 80 60 40 20 0 ppm

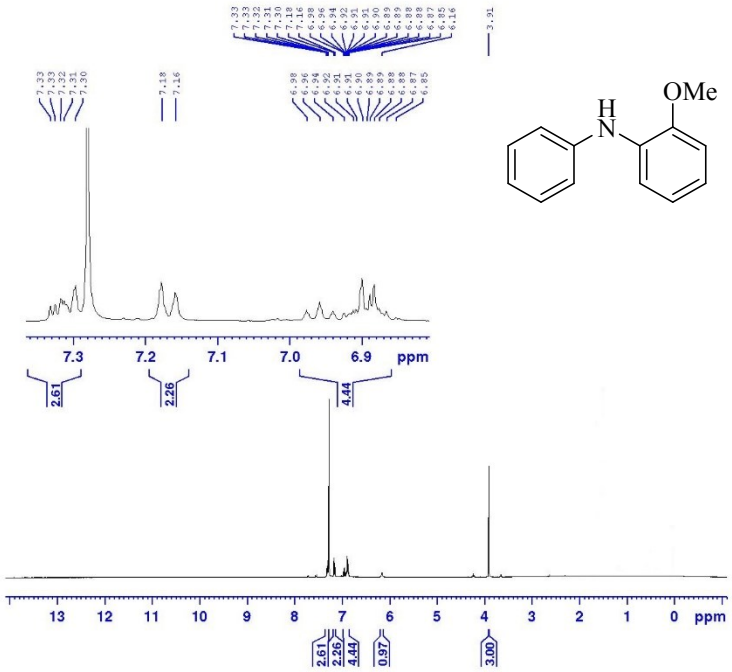

7.17
7.17
7.15
7.03
7.01
6.95
6.94
6.94
6.93
6.93
6.92
6.83
6.81
6.79
6.53
2.23
H
N
Me
7.2
7.1
7.0
6.9
ppm
1.74
2.03
3.72
0.87
15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.74
2.03
3.72
0.87
0.91
3.00

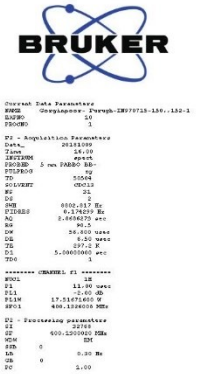


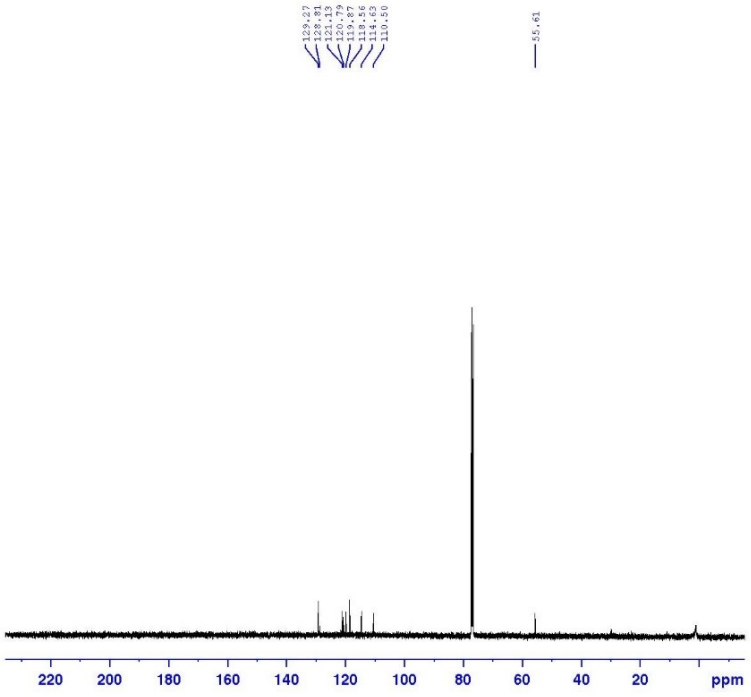

130.94
130.85
129.71
129.30
122.72
120.29
118.91
116.86
20.68
220 200 180 160 140 120 100 80 60 40 20 0 ppm



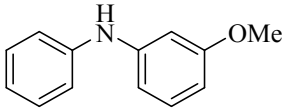
h77

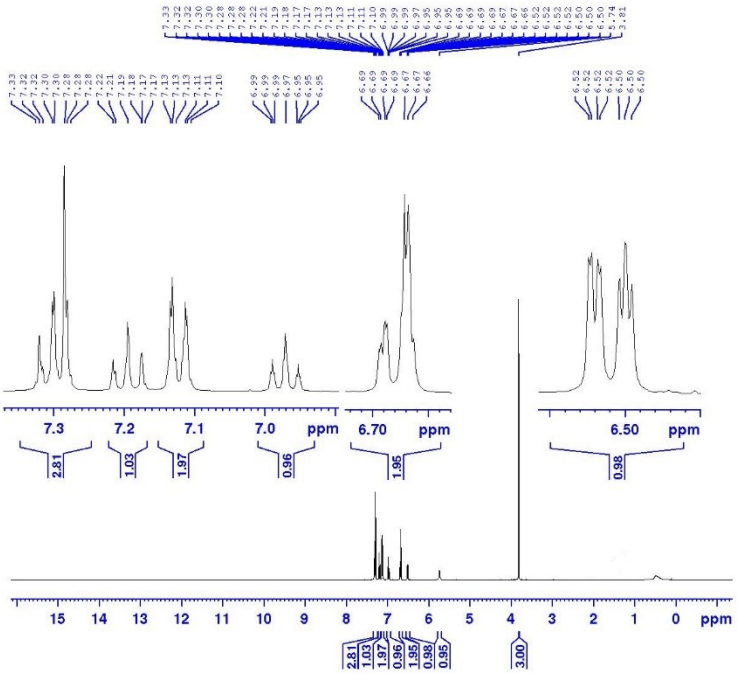

7.33
7.33
7.32
7.31
7.30
7.18
7.16
6.98
6.96
6.94
6.92
6.91
6.91
6.90
6.89
6.89
6.88
6.88
6.87
6.85
6.16
3.91
OMe
H
N
7.3
7.2
7.1
7.0
6.9
ppm
2.61
2.26
4.44
13
12
11
10
9
8
7
6
5
4
3
2
1
0
ppm
2.61
2.26
4.44
0.97
3.00


BRUKER

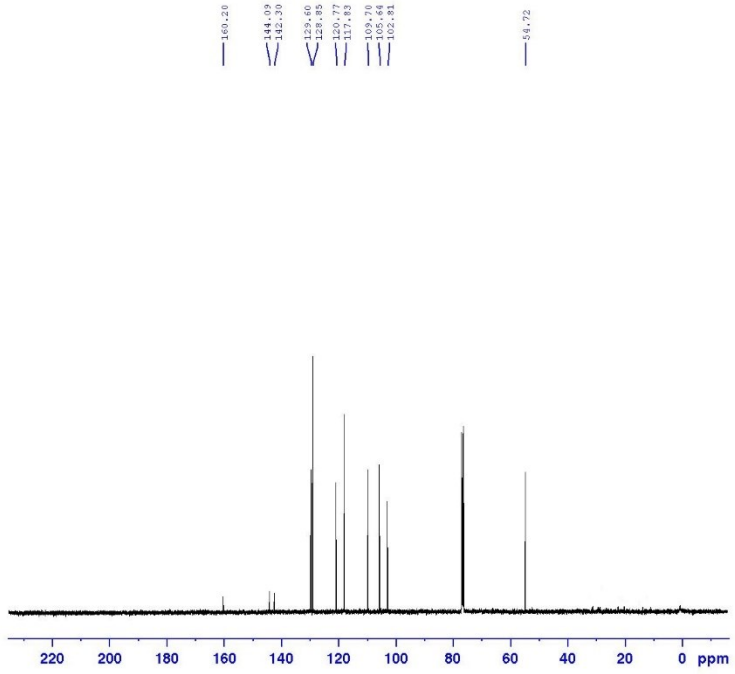

129.27
128.81
121.13
120.79
113.87
113.56
111.63
110.50
55.61
220
200
180
160
140
120
100
80
60
40
20
ppm


BRUKER

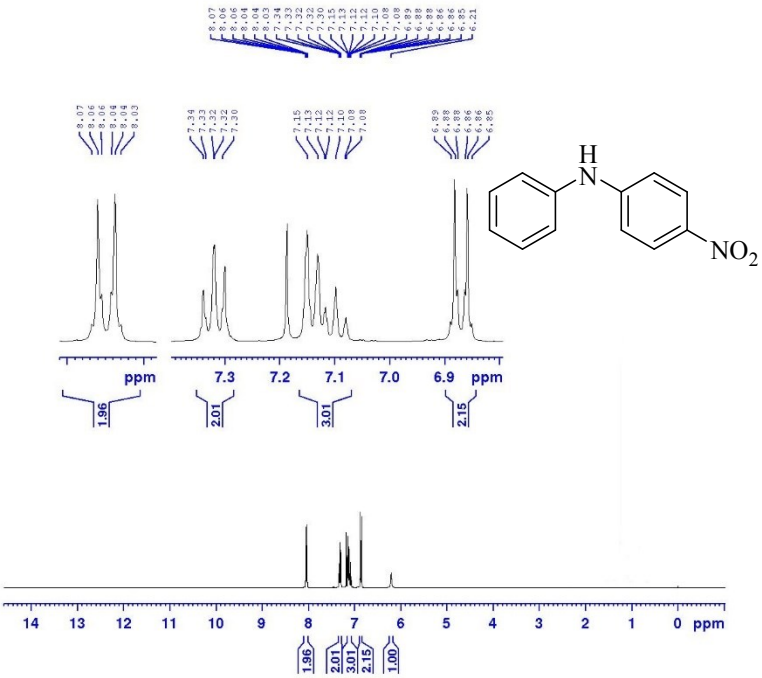


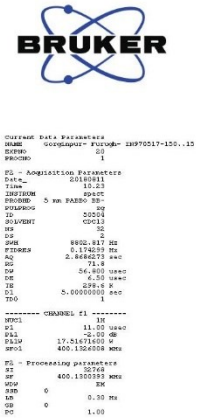



```
Current Data Parameters
NAME      Gorginpur- Furugh-IN970607
EXPNO                 20
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180903
Time              11.15
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG             zg
TD                50504
SOLVENT           CDCl3
NS                   32
DS                    2
SWH            8802.817 Hz
FIDRES         0.174299 Hz
AQ             2.8684273 sec
RG                 71.8
DW               56.800 usec
DE                6.50 usec
TE                296.2 K
D1             5.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                  1H
P1                11.00 usec
PL1               -2.00 dB
PL1W        17.51671600 W
SFO1        400.1326008 MHz

F2 - Processing parameters
SI                   32768
SF          400.1300000 MHz
WDW                     EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

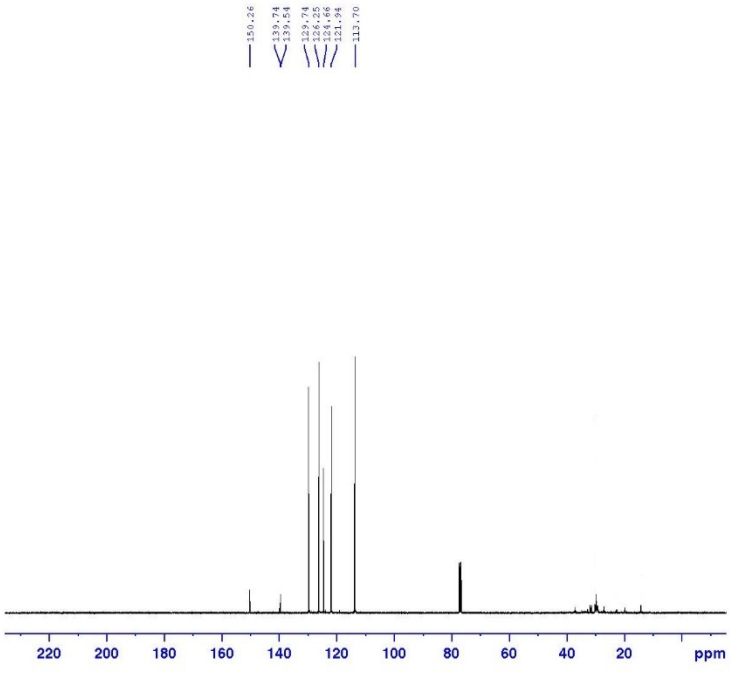



```
Current Data Parameters
NAME      Gorginpur- Furugh-IN970607
EXPNO                 21
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180903
Time              12.39
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg
TD                75754
SOLVENT           CDCl3
NS                 1324
DS                    2
SWH           25252.525 Hz
FIDRES         0.333349 Hz
AQ             1.4999292 sec
RG                 2050
DW               19.800 usec
DE                6.50 usec
TE                297.5 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                9.50 usec
PL1               -1.00 dB
PL1W        42.69075012 W
SFO1        100.6238364 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2       0 dB
PL12              15.26 dB
PL13              18.26 dB
PL2W        11.05230045 W
PL12W        0.32919458 W
PL13W        0.16498812 W
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                   32768
SF          100.6128193 MHz
WDW                     EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

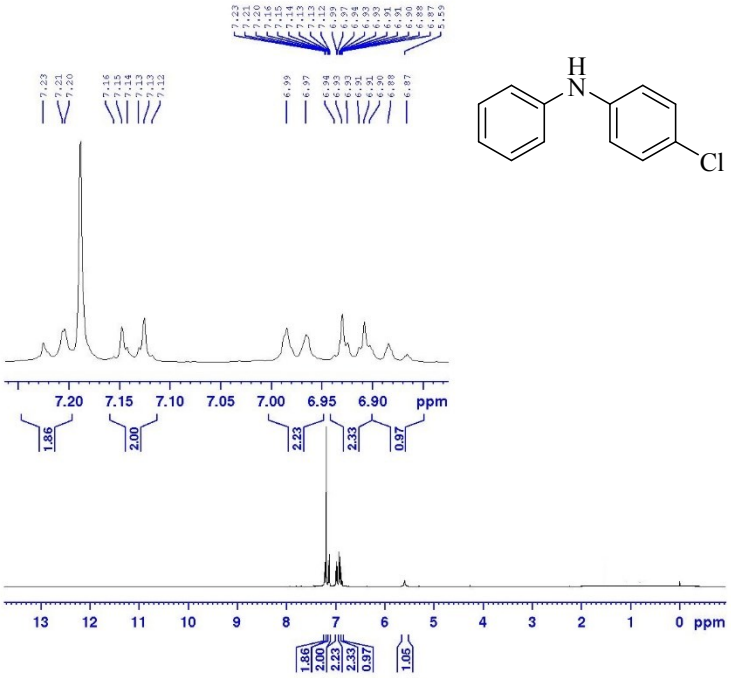


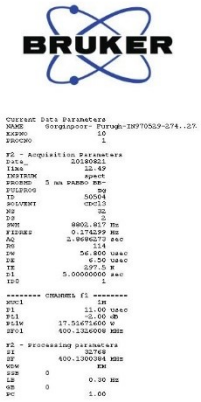


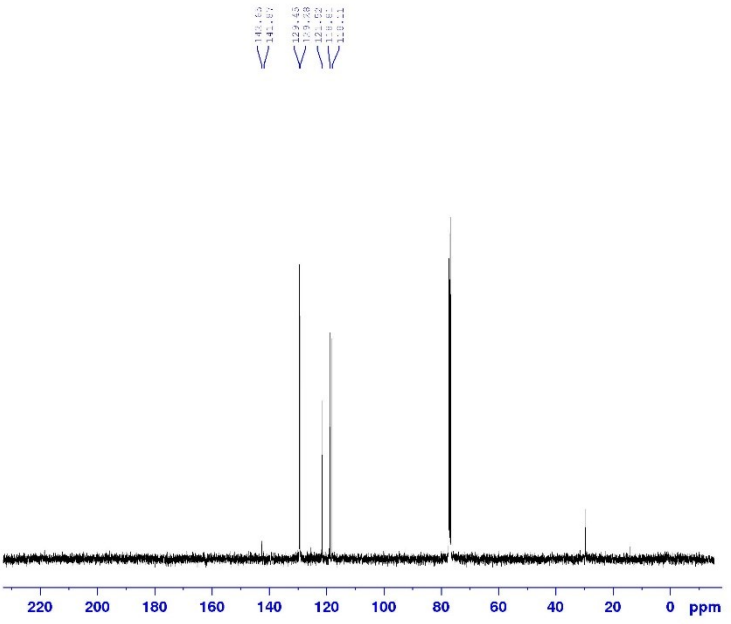
```
Current Data Parameters
NAME      Georgiyevs- Furugh- IN970517-150..15
EXPNO                 20
PROCNO                 1

F1 - Acquisition Parameters
Date_           20180911
Time               10.23
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG                 zg
TD                  32504
SOLVENT             CDCl3
NS                     32
DS                      2
SWH             8802.817 Hz
FIDRES          0.174299 Hz
AQ              2.8486273 sec
RG                   71.8
DW                 56.800 usec
DE                  6.50 usec
TE                 299.6 K
D1              5.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                   1H
P1                  11.00 usec
PL1                 -2.00 dB
PL1W          17.51671600 W
SFO1          400.1324008 MHz

F1 - Processing parameters
SI                  32768
SF            400.1300093 MHz
WDW                    EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



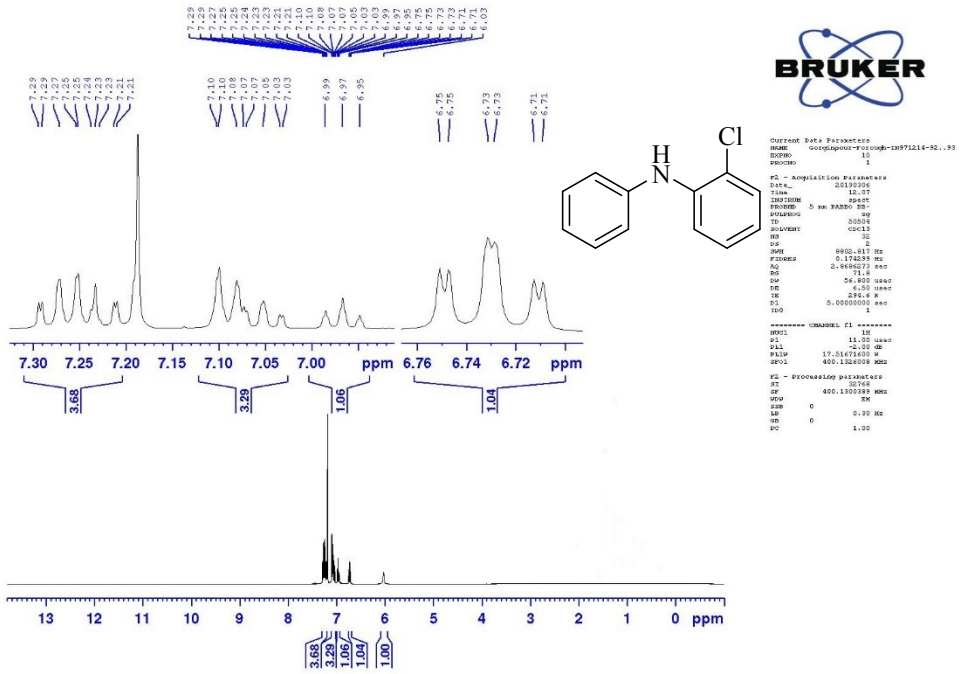
```
Current Data Parameters
NAME      Georgiyevs- Furugh- IN970517-150..15
EXPNO                 21
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180911
Time               8.50
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG             zgpg
TD                  75756
SOLVENT             CDCl3
NS                    990
DS                     16
SWH            25252.525 Hz
FIDRES          0.333369 Hz
AQ              1.4999091 sec
RG                   40.5
DW                 19.800 usec
DE                  6.50 usec
TE                 299.7 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                  9.50 usec
PL1                -1.00 dB
PL1W          92.69075012 W
SFO1          100.6238364 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                 0 dB
PL12               15.26 dB
PL13               19.26 dB
PL2W          11.05230045 W
PL12W          0.32919458 W
PL13W          0.16498812 W
SFO2          400.1316005 MHz

F2 - Processing parameters
SI                  32768
SF            100.6127690 MHz
WDW                    EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

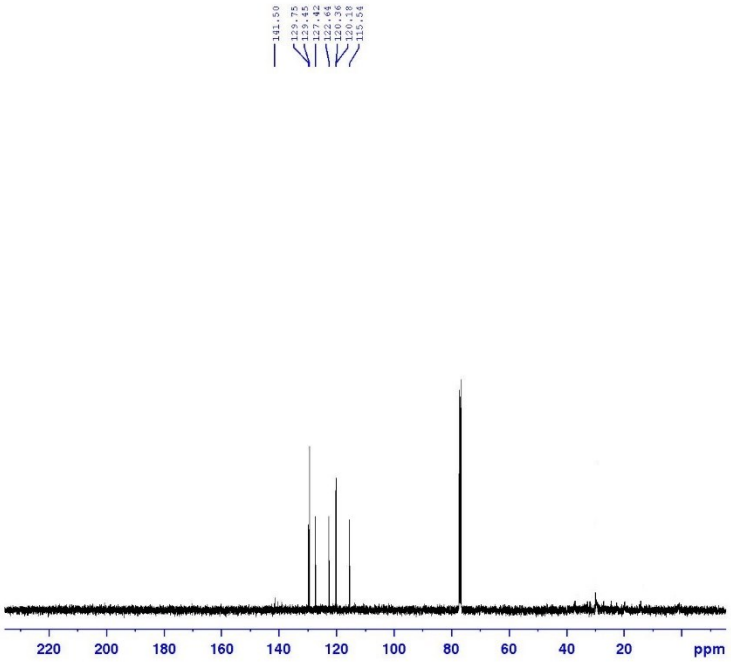

7.23
7.21
7.20
7.16
7.15
7.14
7.13
7.12
6.99
6.97
6.94
6.93
6.91
6.90
6.88
6.87
H
N
Cl
7.20
7.15
7.10
7.05
7.00
6.95
6.90
ppm
1.86
2.00
2.23
2.33
0.97
13
12
11
10
9
8
7
6
5
4
3
2
1
0 ppm
1.86
2.00
2.23
2.33
0.97
1.05

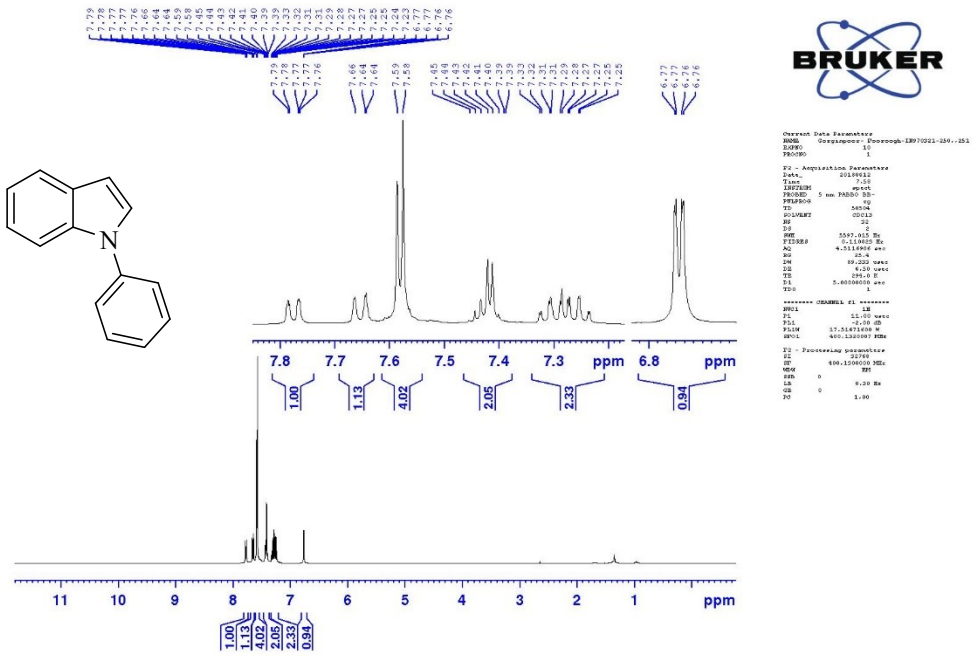

BRUKER


144.69
144.07
129.45
123.36
123.02
116.81
116.14
220
200
180
160
140
120
100
80
60
40
20
0 ppm

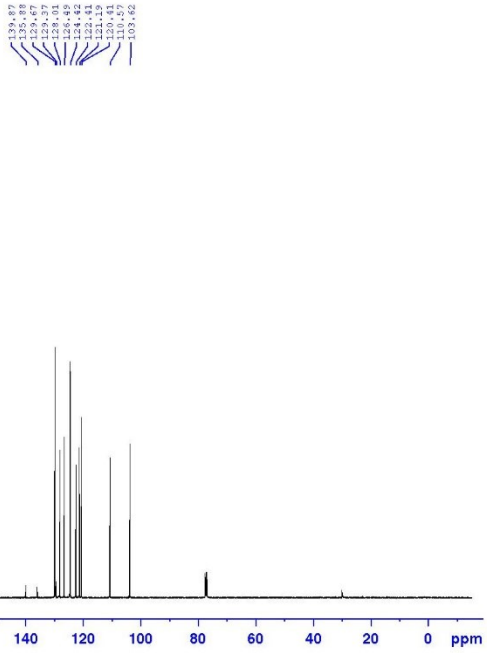

BRUKER


7.30 7.25 7.20 7.15 7.10 7.05 7.00 ppm 6.76 6.74 6.72 ppm
3.68 3.29 1.06 1.04
13 12 11 10 9 8 7 6 5 4 3 2 1 0 ppm
3.68 3.29 1.06 1.04 1.00
H N Cl


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




```
Current Data Parameters
NAME      Gorgiapoor- Poorsogh-IN976221-250..251
EXPNO          10
PROCNO          1

F2 - Acquisition Parameters
Date_        20190612
Time            7.50
INSTRUM         spect
PROBHD   5 mm PABBO BB-
PULPROG         zg
TD             54504
SOLVENT         CDCl3
NS              32
DS               2
SWH          5597.015 Hz
FIDRES       0.102685 Hz
AQ           4.5114906 sec
RG              25.4
DW             89.222 usec
DE              6.50 usec
TE             295.0 K
D1          5.00000000 sec
TD0             1

======== CHANNEL f1 ========
NUC1             1H
P1             11.00 usec
PL1            -2.00 dB
PLW1        17.51671008 W
SFO1       400.1320007 MHz

F2 - Processing parameters
SI             32768
SF          400.1306000 MHz
WDW               EM
SSB             0
LB              0.20 Hz
GB              0
PC              1.00
```




```
Current Data Parameters
NAME      Gorgiapoor- Poorsogh-IN976221-250..251
EXPNO          11
PROCNO          1

F2 - Acquisition Parameters
Date_        20190612
Time            8.55
INSTRUM         spect
PROBHD   5 mm PABBO BB-
PULPROG         zgpg
TD             55562
SOLVENT         CDCl3
NS              1500
DS               2
SWH          25252.525 Hz
FIDRES       0.226629 Hz
AQ           0.9999396 sec
RG              2850
DW             19.800 usec
DE              6.50 usec
TE             295.3 K
D1          1.00000000 sec
D11         0.03000000 sec
TD0             1

======== CHANNEL f1 ========
NUC1             13C
P1              9.50 usec
PL1             -1.06 dB
PLW1        42.69035812 W
SFO1       100.6228296 MHz

======== CHANNEL f2 ========
CPDPRG[2        waltz16
NUC2             1H
PCPD2           80.00 usec
PL2            0 dB
PL12           18.24 dB
PL13           18.24 dB
PLW2        11.05230045 W
PLW12        0.03919454 W
PLW13        0.10493012 W
SFO2       400.1316005 MHz

F2 - Processing parameters
SI             32768
SF          100.6127690 MHz
WDW               EM
SSB             0
LB              1.00 Hz
GB              0
PC              1.40
```