

Supporting Information

Bismuth nanoparticles: an efficient catalyst for reductive coupling of nitroarenes to azo-compounds

Kishore Pothula, Lin Tang, Zhenggen Zha, Zhiyong Wang*

Hefei National Laboratory for Physical Sciences at Microscale, CAS Key Laboratory of Soft Matter Chemistry & Collaborative Innovation Center of Suzhou Nano Science and Technology, University of Science and Technology of China, Hefei, 230026, P. R. China. Fax: (+86) 551-360-3185; E-mail: zwang3@ustc.edu.cn.

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General Remarks

All substrates were purchased commercially and used without further purification. BiCl₃ and Zinc dust were purchased from Sinopharm Chemical Reagent Co., Ltd. The morphology and size of the nanoparticles were characterized by Transmission electron microscope (TEM) (JEOL-2010 and Hitachi H7650). The as-synthesized BiNPs were dispersed in ethanol and dried on the carbon-coated Cu grids. The accurate metal loading was directly determined by ICP-OES (Inductively Coupled Plasma Optical Emission Spectrometer) using Perkin Elmer Optima 7300 DV. X-ray powder diffraction patterns (XRD) of the product were obtained on a Panalytical X'pert PRO X-ray diffractometer with Cu K α radiation. GC-MS samples were recorded on a Shimadzu QP-5050 GC-MS system. ¹H and ¹³C NMR spectra were recorded on a Bruker AC-300 FT spectrometer at 400 MHz and 100 MHz, respectively, with tetramethylsilane as an internal reference. Chemical shifts (δ) and coupling constants (J) were expressed in ppm and Hz, respectively. Infrared Spectra of samples were recorded on a Bruker EQUINOX 55 spectrometer. HRMass were recorded on a Micro Mass UK LTD GCT TOF-MS. GC-MS samples were recorded on a Shimadzu QP-5050 GC-MS system.

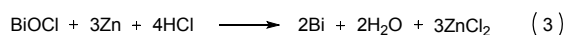
General procedure for the preparation of Bismuth Nano particles

0.5 mmol BiCl₃ was suspended in demineralized water 30 ml. After stirring for about 15 min at room temperature (RT, ~25-30 °C), the white precipitation was observed. Then 0.75 mmol of zinc dust was added in portions and the reaction mixture was further stirred for 1 hours. Black particles were observed in the reaction. PH was adjusted to 3 with aqueous hydrochloric acid. The reaction mixture was filtered and washed with water followed by ethanol then dried under vacuum for 12 hours at 50 °C to obtain BiNPs yield 92 %. The reported procedure for BiNPs were Bamboo raft nano tubes, here we got Nano bismuth Particles at PH 3.

The overall reaction can be simply formulated as shown in below equation (1).



The above reaction includes two following steps as equations (2) and (3).



First, BiCl₃ hydrolyzed in water to form BiOCl, a white precipitate equation (2), which is then reduced by Zinc dust to form bismuth nanoparticles.

General procedure for the synthesis of aromatic azo compounds

1 mmol Nitrobenzene was taken in a schlenk tube then added 20.8 mg (10 mol %) of Bismuth nanoparticles. Reaction mixture was degassed and slowly added NaBH_4 in Ethanol (37.83 mg NaBH_4 dissolved in 4 ml Ethanol) followed by stirring the reaction mass for 24 hours. Progress of the reaction mass was monitored by TLC (Mobile phase petether/ethyl acetate : 9/1). Catalyst was filtered and filtrate was evaporated under reduced pressure to dryness. The product dissolved in water and extracted with ethylacetate. The organic phase was dried on MgSO_4 and distilled to get crude product which was subjected to flash column chromatography (Petether/Ethyl acetate : 98/2) to afford pure azobenzene.

X-Ray Diffraction Analysis (XRD) : The phase purity of the Bismuth was examined by Panalytical X'pert PRO X-ray diffractometer with $\text{Cu K}\alpha$ radiation. The detection range was 20° to 70° . Figure 1 shows the typical XRD pattern of the As-synthesized bismuth nanoparticles, all the diffraction peaks were consistent with the literature values⁹. Undetection of any impurity in this pattern, indicating the formation of pure bismuth under current synthetic condition. Sharp and strong diffraction peaks in Figure 1 also confirm the well crystallization of the products.

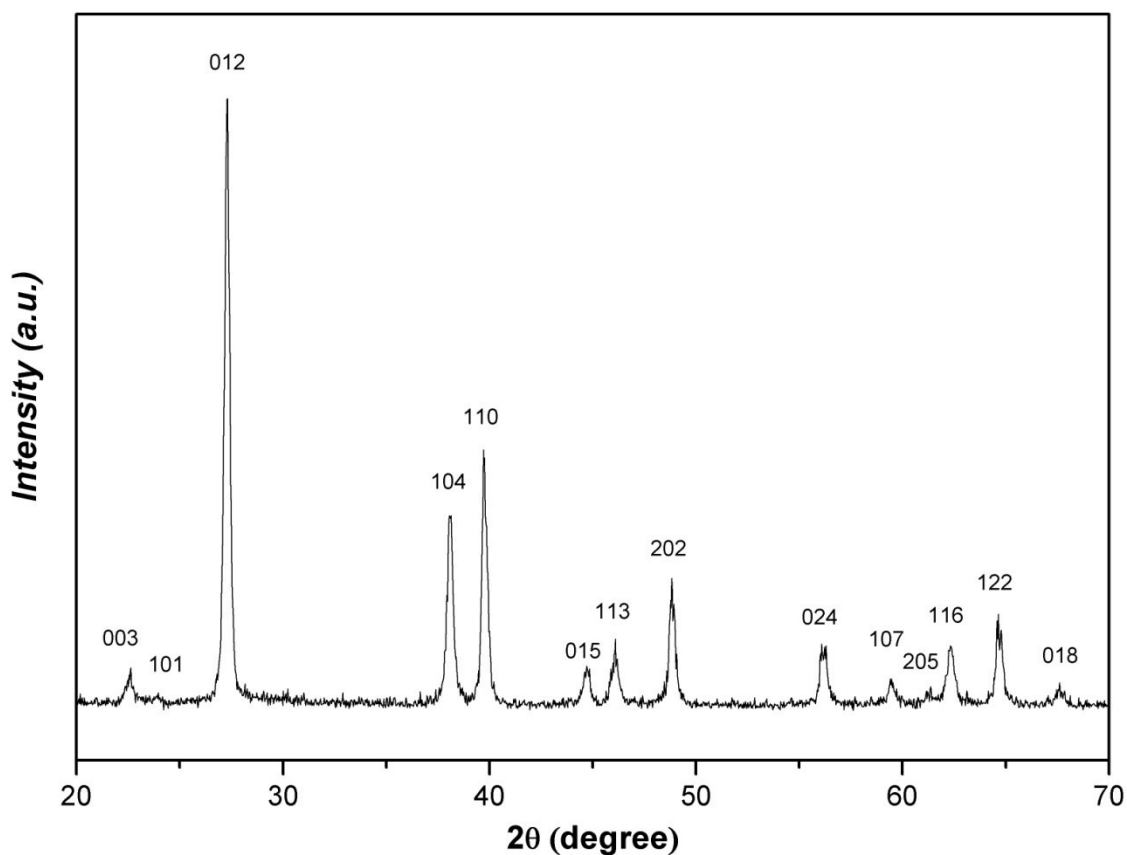


Figure 1 Powder XRD pattern of the bismuth nano particles.

Transmission electron microscope (TEM) : The morphologies and structures of the as-prepared bismuth nanoparticles were investigated by TEM images. Figure 2 shows the representative TEM images of the prepared bismuth nanoparticles with 30 and 10 nm magnifications. Diameters of these nanoparticles varied from 3 to 8 nm and most of them were about 5 nm. Figure 3 shows the representative TEM images of the recovered bismuth nanoparticles with 30 nm magnifications. Diameters of these nanoparticles varied from 15 to 30 nm.

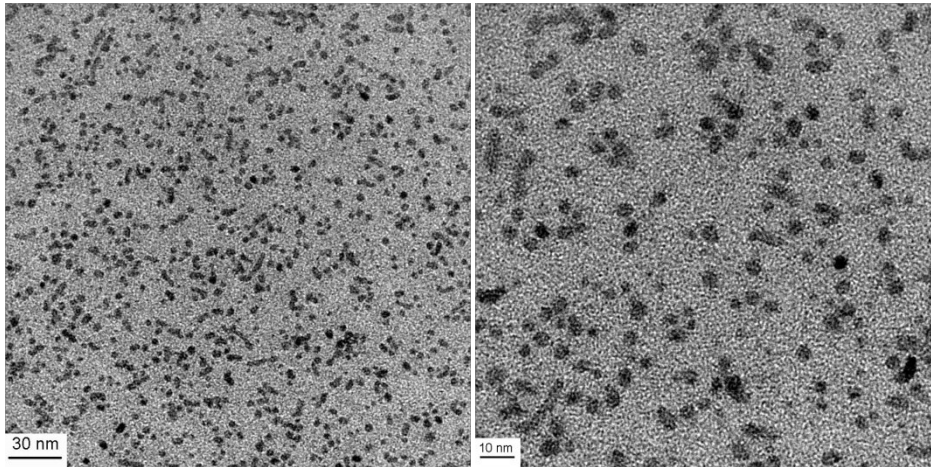


Figure 2 TEM images of Bismuth nanoparticles.

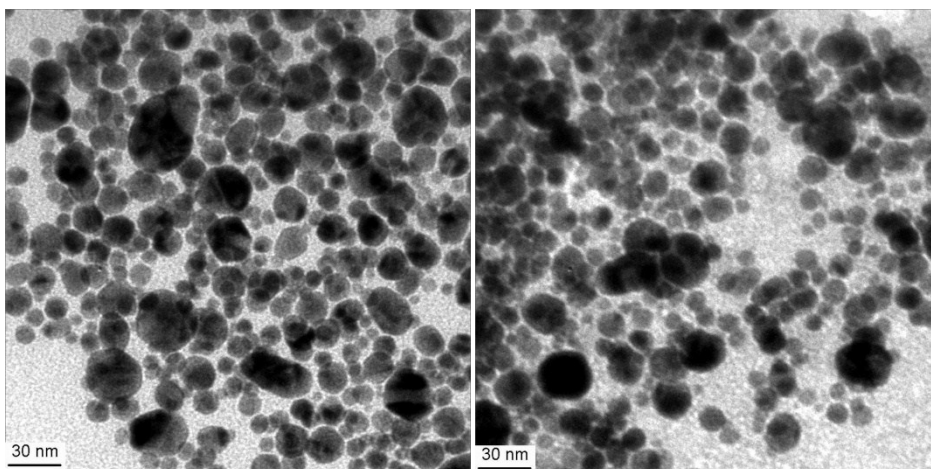


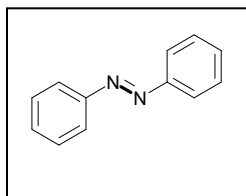
Figure 3 TEM images of Bismuth nanoparticles after 5th run.

Table S1: Comparative studies of the reaction (aryl nitro to aryl azo) at different temperatures .^a

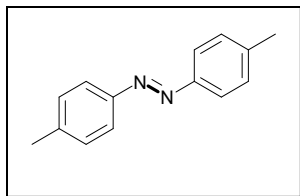
Entry	Catalyst	Temperature (°C)	Yield (%) ^b
1	0.1	30	Traces
2	0.1	50	9
3	0.1	80	12
4	1	80	16

(a) Reaction conditions: Nitro benzene (1 mmol), Catalyst bismuth nanoparticles, NaBH₄ (1.0 mmol), solvent 4 ml of water, 24 h. (b) Determined by GC-MS.

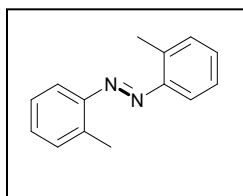
Characterization data of products



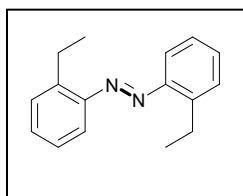
Azobenzene (2a). The title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a orange crystal: 92% yield; mp = 68 °C; ¹H NMR (400 MHz, CDCl₃) δ 7.52 (m, 6H), 7.96 (d, J = 7 Hz, 4H); ¹³C NMR (100 MHz, CDCl₃) δ 125 MHz, CDCl₃) δ 122.8, 129.0, 130.9, 152.6. This compound was known¹.



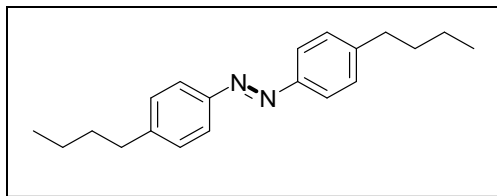
1,2-di-p-tolyldiazene (2b). The title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a yellow crystal: 87% yield; mp = 143 °C; ^1H NMR (400 MHz, CDCl_3) δ 2.43 (s, 6H), 7.30 (d, $J=8.0$ Hz, 4H), 7.81 (d, $J=8.4$ Hz, 4H); ^{13}C NMR (100 MHz, CDCl_3) δ 21.4, 122.7, 129.7, 141.2, 150.8; This compound was known¹.



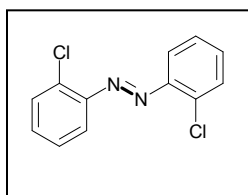
1,2-Di-o-tolyldiazene (2c). The title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a red crystal: 85% yield; mp = 53-54 °C; ^1H NMR (400 MHz, CDCl_3) δ 2.73 (s, 6H), 7.28 (m, 2H), 7.35 (m, 4H), 7.61 (d, $J = 7.9$ Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 17.6, 115.8, 126.3, 130.6, 131.0, 138.0, 151.0; This compound was known².



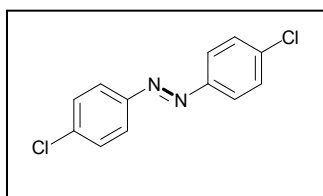
1,2-Bis(2-ethylphenyl)diazene (2d). The title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a red crystal: 82% yield; mp = 47 °C; ^1H NMR (400 MHz, CDCl_3) δ 1.45 (m, 6H), 3.33 (q, $J = 7.6$ Hz, 4H), 7.36 - 7.4 (m, 2H), 7.46 - 7.51 (m, 2H), 7-78 (d, $J = 8.0$ Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 16.6, 24.9, 115.7, 126.5, 129.9, 131, 144.2, 150.6; This compound was known³.



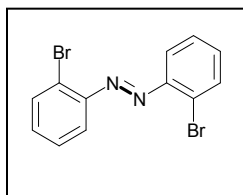
Bis-(4-butyl-phenyl)-diazene (2e). The title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a red crystal: 80% yield; mp = 33 – 34 °C; ^1H NMR (400 MHz, CDCl_3) δ 1.0 (t, 6H), 1.44 (m, 4H), 1.71 (m, 4H), 2.74 (t, J = 7.6 Hz, 4H), 7.37 (d, J = 8.0 Hz, 4H), 7.92 (dd, J = 1.9, 8.4 Hz, 4H); ^{13}C NMR (100 MHz, CDCl_3) δ 13.9, 22.1, 33.2, 35.5, 122.7, 128.7, 146.0, 151.3; This compound was known⁴.



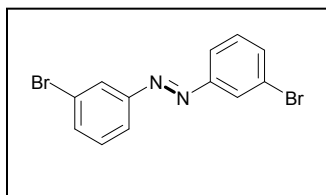
Bis-(2-chloro-phenyl)-diazene (2f). The title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a orange crystal: 95% yield; mp = 137-138 °C; ^1H NMR (400 MHz, CDCl_3) δ 7.39 (m, 4H), 7.58 (dd, J = 1.1, 7.9 Hz, 2H), 7.78 (dd, J = 1.5, 7.9 Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 118.1, 127.4, 130.7, 132.2, 135.8, 148.7 ; This compound was known².



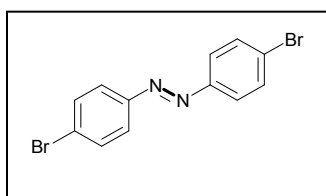
Bis-(4-chloro-phenyl)-diazene (2g). The title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a orange crystal: 94% yield; mp = 184 °C; ^1H NMR (400 MHz, CDCl_3) δ 7.47 – 7.5 (m, 4H), 7.84 – 7.88 (m, 4H); ^{13}C NMR (100 MHz, CDCl_3) δ 124.2, 129.4, 137.2, 150.8; This compound was known¹.



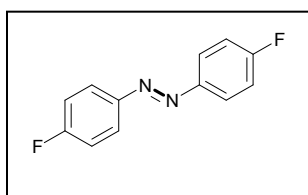
Bis-(2-bromo-phenyl)-diazene (2h). The title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a yellow crystal: 94% yield; mp = 144 °C; ¹H NMR (400 MHz, CDCl₃) δ 7.32 – 7.36 (m,2H) 7.39 – 7.43 (m, 2H) 7.75 – 7.78 (m,4H); ¹³C NMR (100 MHz, CDCl₃) δ 118.4,126.3,128.1,132.4,133.8,149.5 ; This compound was known⁵.



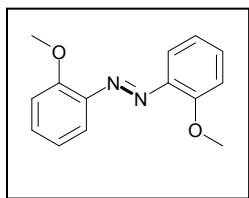
Bis-(3-bromo-phenyl)-diazene (2i). The title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a yellow crystal: 96% yield; mp = 140 °C; ¹H NMR (400 MHz, CDCl₃) δ 7.4 (t, J = 7.9 Hz, 2H) 7.62(d, J = 2.3 Hz, 2H) 7.89 (dd, J = 7.9 Hz, 2H) 8.0 (s, 2H); ¹³C NMR (100 MHz, CDCl₃) δ 123.2, 124.7, 130.5, 134.1, 153.1; This compound was known¹.



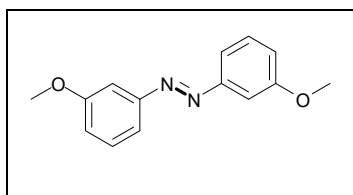
Bis-(4-bromo-phenyl)-diazene (2j). The title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a yellow crystal: 93% yield; mp = 204 °C; ¹H NMR (400 MHz, CDCl₃) δ 7.65 (m, 4H), 7.78 (m, 4H) ; ¹³C NMR (100 MHz, CDCl₃) δ 124.4, 125.7, 132.4, 151.1; This compound was known².



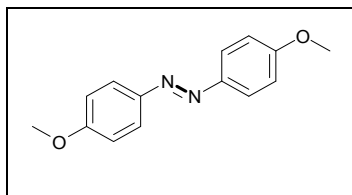
Bis-(4-fluoro-phenyl)-diazene (2k). The title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a orange crystal: 89% yield; mp = 101-102 °C; ^1H NMR (400 MHz, CDCl_3) δ 7.2 (m, 4H), 7.9 (m, 4H); ^{13}C NMR (100 MHz, CDCl_3) δ 116.1 (d, $J_{\text{C-F}} = 22.8$ Hz), 124.8.8 (d, $J_{\text{C-F}} = 8.9$ Hz), 148.9 (d, $J_{\text{C-F}} = 2.3$ Hz), 165.6 (d, $J_{\text{C-F}} = 250.7$ Hz); This compound was known¹.



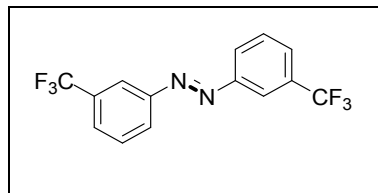
Bis-(2-methoxy-phenyl)-diazene (2l). The title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a orange crystal: 62% yield; mp = 154-156 °C; ^1H NMR (400 MHz, CDCl_3) δ 4.01(s, 6H), 7.0 (M, 2H), 7.08 (dd, $J = 1.1, 8.4$ Hz, 2H), 7.41 (m, 2H), 7.63 (dd, $J = 1.8, 8.0$ Hz, 2H), ^{13}C NMR (CDCl_3) δ 56.5, 112.7, 117.1, 117.7, 120.9, 132.3, 143.1, 156.9; This compound was known⁶.



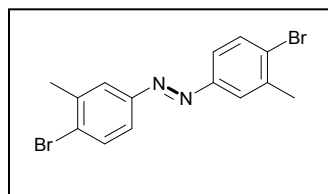
Bis-(3-methoxy-phenyl)-diazene (2m). The title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a orange crystal: 64% yield; mp = 75 °C; ^1H NMR (400 MHz, CDCl_3) 3.9 (s, 6H), 7.05 (dd, $J = 1.9, 8.1$ Hz, 2H), 7.41-7.47 (m, 4H), 7.57 (d, $J = 7.8$ Hz, 2H), ^{13}C NMR (CDCl_3) δ 55.4, 105.6, 117.1, 117.8, 129.7, 153.7, 160.2; This compound was known⁶.



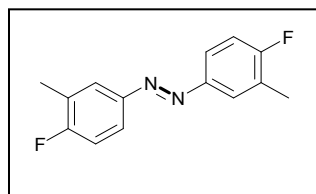
Bis-(3-methoxy-phenyl)-diazene (2n). The title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a orange crystal: 68% yield; mp = 172 °C; ^1H NMR (400 MHz, CDCl_3) δ 3.88 (s, 6H), 7.0 (dd, $J = 2.2, 6.9$ Hz, 4H), 7.88 (dd, $J = 2.1, 6.9$ Hz, 4H); ^{13}C NMR (100 MHz, CDCl_3) δ 55.5, 114.1, 124.3, 147, 161.5; This compound was known⁷.



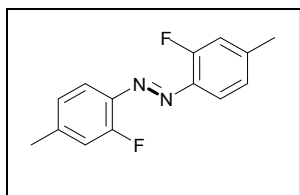
Bis-(3-trifluoromethyl-phenyl)-diazene (2o). The title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a orange crystal: 88% yield; mp = 85 °C; ^1H NMR (400 MHz, CDCl_3) δ 7.69 (t, $J = 7.9$ Hz, 2H), 7.77 (d, $J = 7.8$ Hz, 2H), 8.1 (d, $J = 8.0$ Hz, 2H), 8.22 (s, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 119.9, 126.6, 128, 129.9, 152.3; IR (KBr): $\nu = 2962, 1581, 1492, 1309, 1251, 1197, 1099, 892, 825$ cm^{-1} ; IR spectral data not reported previously. This compound was known¹.



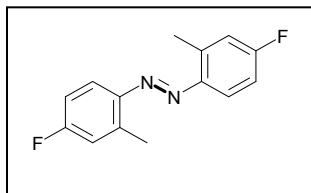
Bis-(4-Bromo-3-methyl-phenyl)-diazene (2p). The title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a orange crystal: 86% yield; mp = 146 °C; ^1H NMR (400 MHz, CDCl_3) δ 2.49 (s, 6H), 7.59 (m, 2H), 7.67 (d, $J = 8.4$ Hz, 2H), 7.78 (d, $J = 2.0$ Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 23.0, 121.6, 124.9, 128.0, 133.0, 139, 151.5; IR (KBr): $\nu = 2919, 1743, 1652, 1558, 1409, 1295, 1195, 1029, 946, 821$ cm^{-1} ; IR spectral data not reported previously. This compound was known⁸.



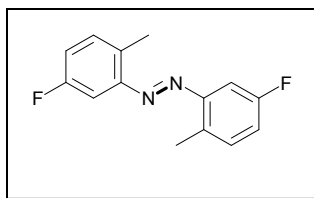
Bis-(4-fluoro-3-methyl-phenyl)-diazene (2q). The title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a shiny yellow crystal: 82% yield; mp = 112 °C; ^1H NMR (400 MHz, CDCl_3) δ 2.37 (s, 6H), 7.13 (t, $J=8.8$ Hz, 2H), 7.76 (m, 4H,); ^{13}C NMR (100 MHz, CDCl_3) δ 14.8 (d, $J_{\text{C-F}}=3.1$ Hz), 115.7 (d, $J_{\text{C-F}}=23.9$ Hz), 122.6 (d, $J_{\text{C-F}}=8.9$ Hz), 125.5 (d, $J_{\text{C-F}}=6.2$ Hz), 125.9 (d, $J_{\text{C-F}}=18.8$ Hz), 148.8 (d, $J_{\text{C-F}}=2.7$ Hz), 163.0 (d, $J_{\text{C-F}}=249.4.7$ Hz), IR (KBr): $\nu = 2962, 1581, 1492, 1450, 1251, 1197, 1099, 892, 825$ cm^{-1} ; HRMS-ESI: m/z (calculated for $[\text{M}+\text{H}]^+$ $\text{C}_{14}\text{H}_{13}\text{N}_2\text{F}_2$ 247.1047, found 247.1048 . This compound was known².



Bis-(2-fluoro-4-methyl-phenyl)-diazene (2r). The title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a yellow crystal: 85% yield; mp = 139 – 140 °C; ^1H NMR (400 MHz, CDCl_3) δ 2.4 (s, 6H), 7.0 (d, $J = 8.8$ Hz, 4H), 7.05 – 7.08 (m, 2H), 7.7 (t, $J = 8.1$ Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 21.7 , 117.6 (d, $J_{\text{C-F}}=9.0$ Hz), 125.3 (d, $J_{\text{C-F}}=3.0$ Hz), 139.0 (d, $J_{\text{C-F}}=7.1$ Hz), 144.2 (d, $J_{\text{C-F}}=8.1$ Hz), 159, 161.6; IR (KBr): $\nu=2923, 1583, 1492, 1421, 1267, 1124, 939, 850, 823$ cm^{-1} ; HRMS-ESI: m/z (calculated for $[\text{M} + \text{H}]^+$ $\text{C}_{14}\text{H}_{13}\text{N}_2\text{F}_2$ 247.1047, found 247.1049. This compound was not reported.



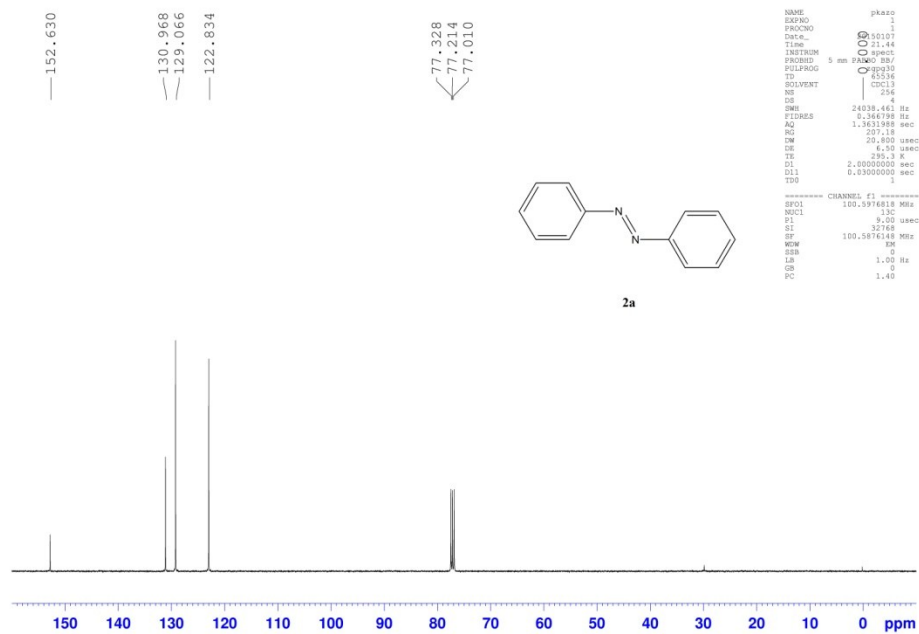
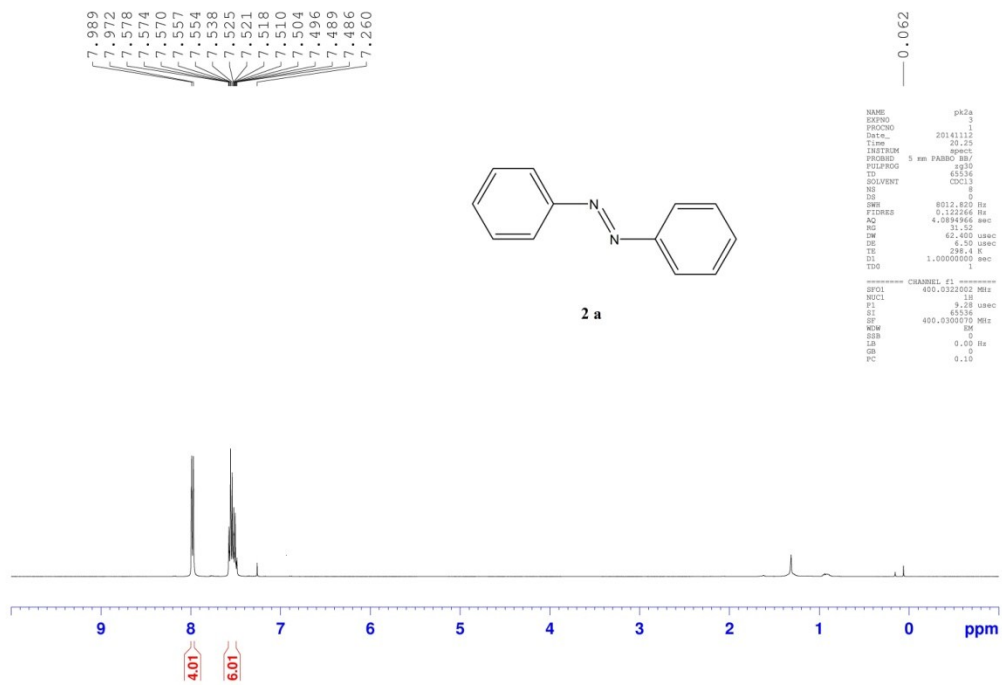
Bis-(4-fluoro-2-methyl-phenyl)-diazene (2s). The title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a yellow crystal: 82% yield; mp = 110 °C; ^1H NMR (400 MHz, CDCl_3) δ 2.37 (s, 6H), 7.13 (t, $J = 8.1$ Hz, 2H), 7.74 (m, Hz, 4H); ^{13}C NMR (100 MHz, CDCl_3) δ 14.8 (d, $J_{\text{C-F}}=3.1$ Hz) , 115.6 (d, $J_{\text{C-F}}=23.8$ Hz), 122.6 (d, $J_{\text{C-F}}=9.0$ Hz), 125.6 (d, $J_{\text{C-F}}=6.2$ Hz), 126 (d, $J_{\text{C-F}}=18.8$ Hz), 148.9 (d, $J_{\text{C-F}}=2.6$ Hz), 164 (d, $J_{\text{C-F}}=249.3$ Hz), IR (KBr): $\nu=2921, 1581, 1490, 1436, 1251, 1197, 1101, 892, 825$ cm^{-1} ; HRMS-ESI: m/z (calculated for $[\text{M} + \text{H}]^+$ $\text{C}_{14}\text{H}_{13}\text{N}_2\text{F}_2$ 247.1047, found 247.1053 . This compound identification data was not reported.

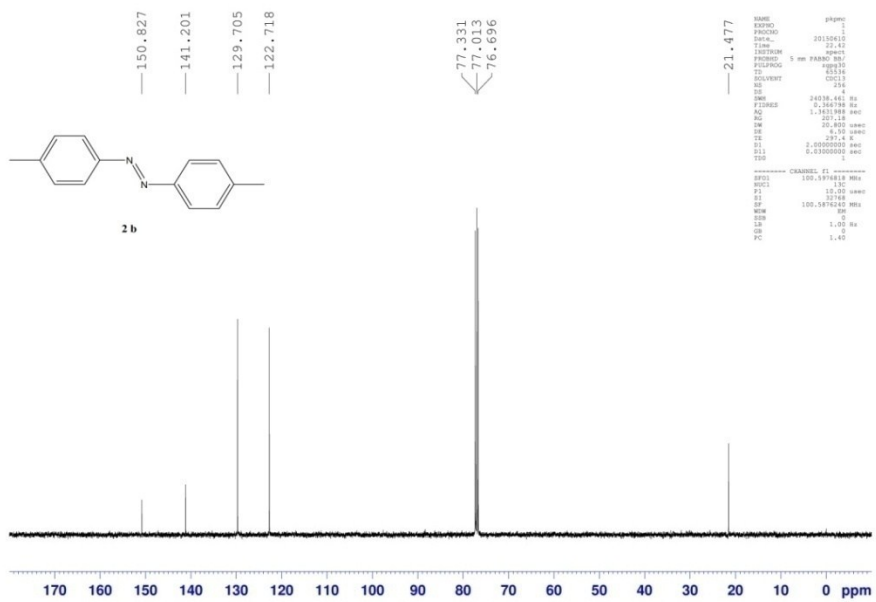
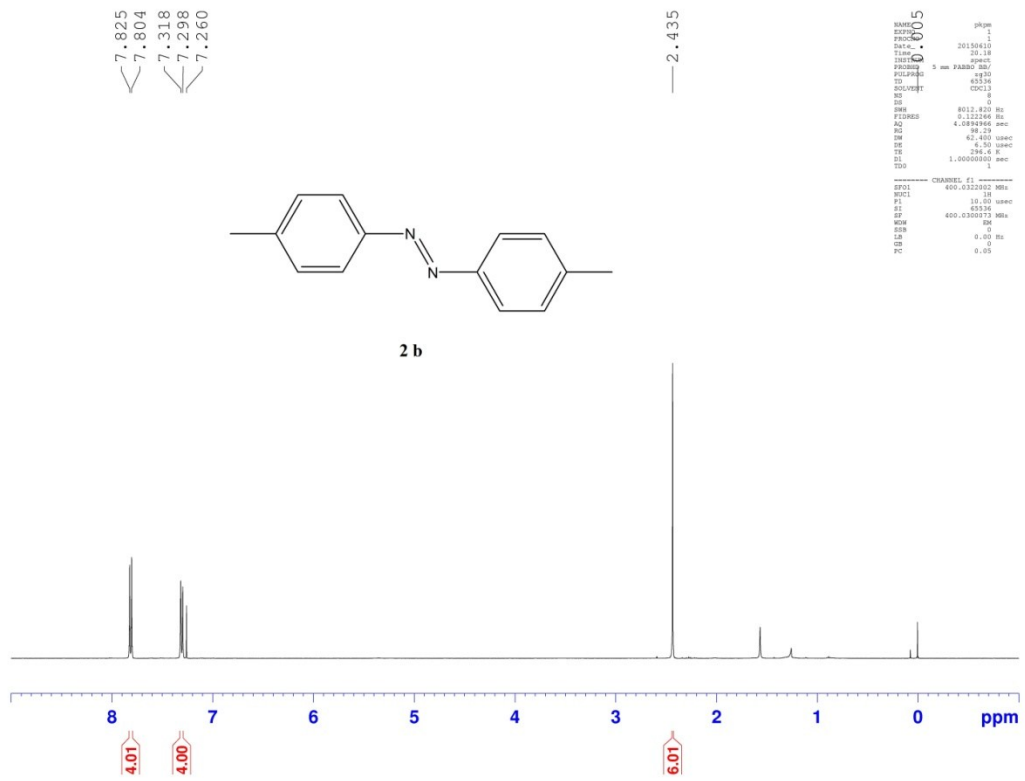


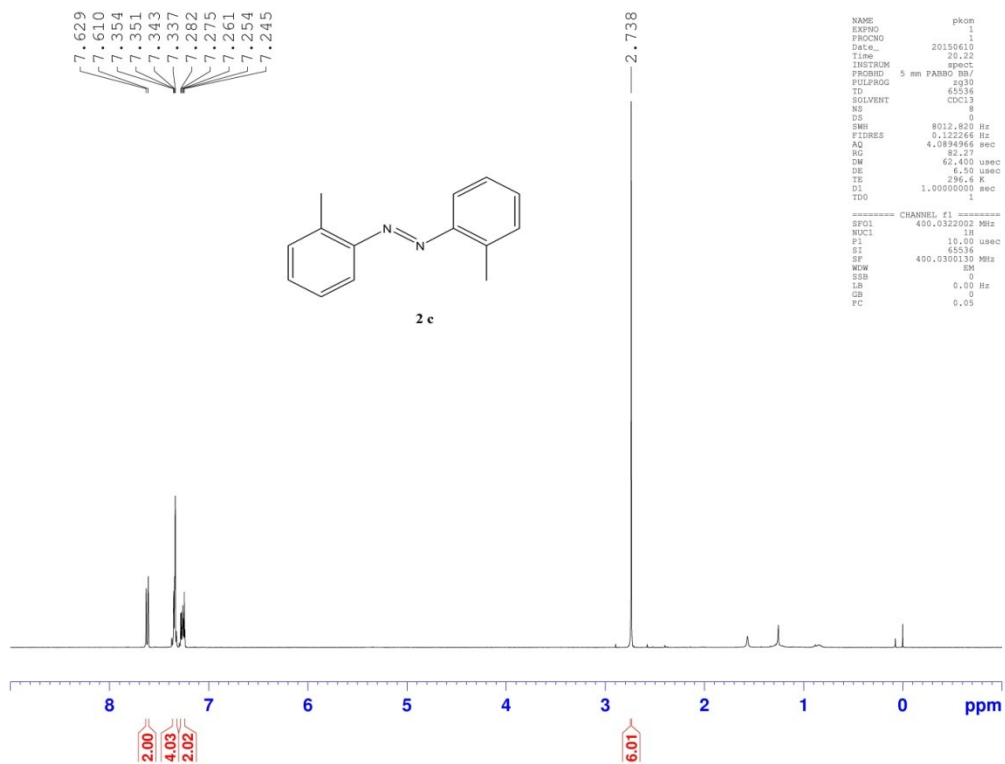
Bis-(5-fluoro-2-methyl-phenyl)-diazene (2t). The title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a orange crystal: 86% yield; mp = 121 °C; ¹H NMR (400 MHz, CDCl₃) δ 2.7 (s, 6H), 7.1 (m, 2H), 7.3 (m, 2H), 7.38 (dd, J = 2.8 , 9.9 Hz, 2H) ; ¹³C NMR (100 MHz, CDCl₃) δ 16.8, 102.3 (d, J_{C-F}= 22.6 Hz), 117.9 (d, J_{C-F}= 22.1 Hz), 132.3 (d, J_{C-F}= 7.9 Hz), 134.5 (d, J_{C-F}= 2.9 Hz), 151.2 (d, J_{C-F}= 5.2 Hz), 161 (d, J_{C-F}= 244.1 Hz), IR (KBr): ν=2931, 1591, 1496, 1378, 1245, 1132, 1037, 962, 806 cm⁻¹ ; HRMS-ESI: m/z (calculated for [M + H]⁺ C₁₄H₁₃N₂F₂ 247.1047, found 247.1053 . This compound identification data was not reported.

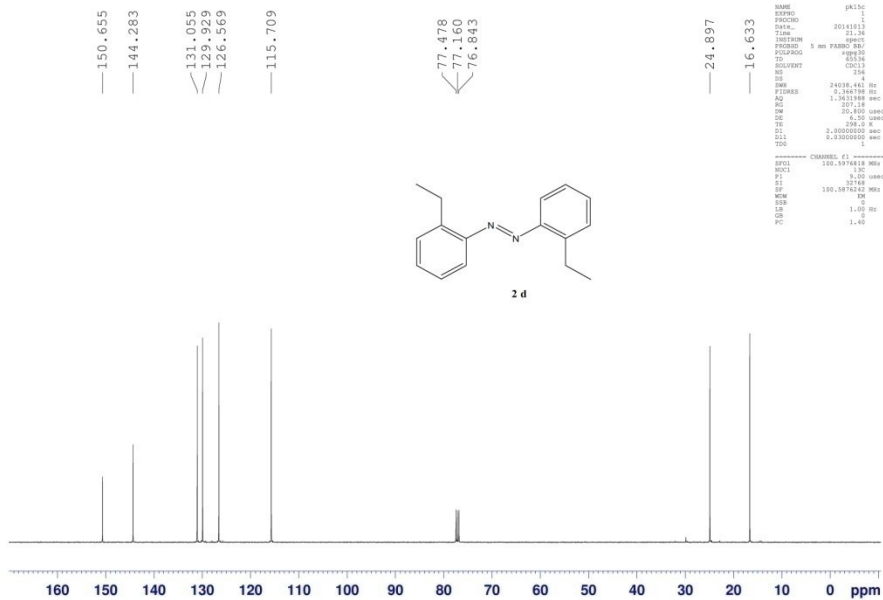
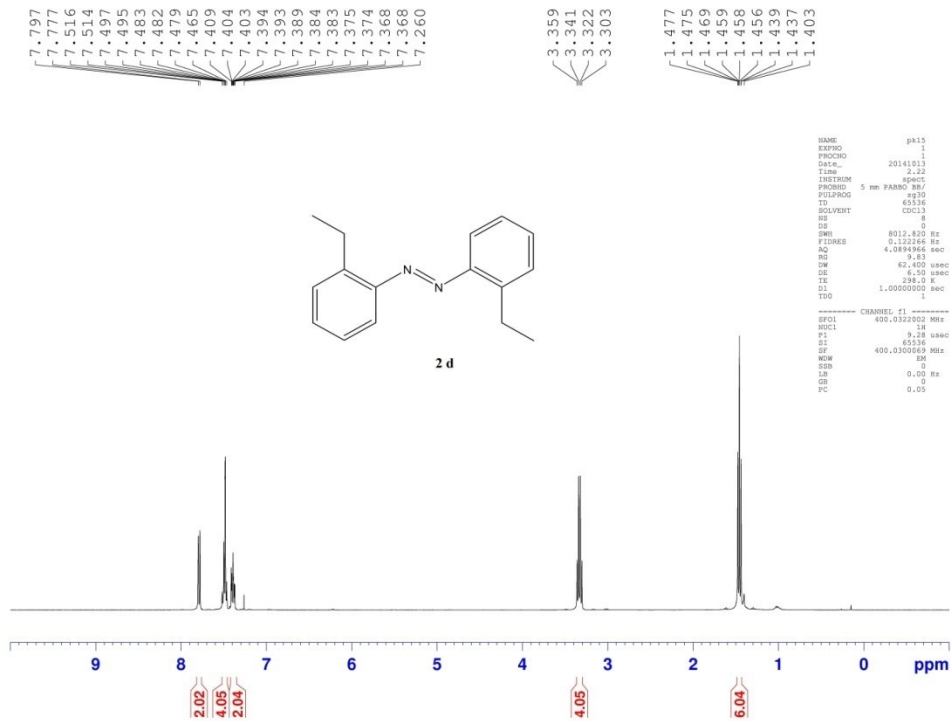
References

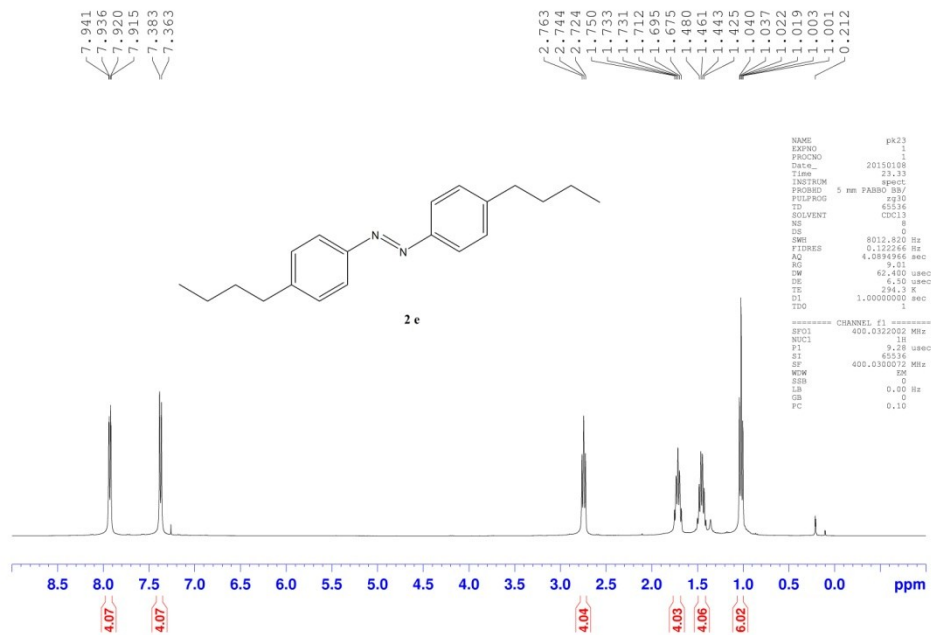
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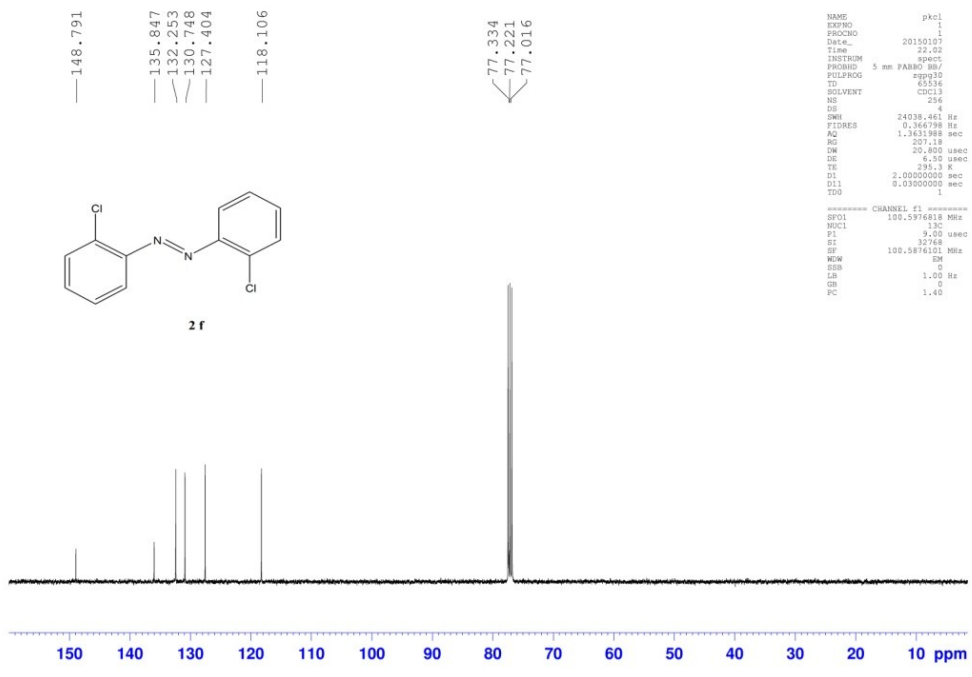
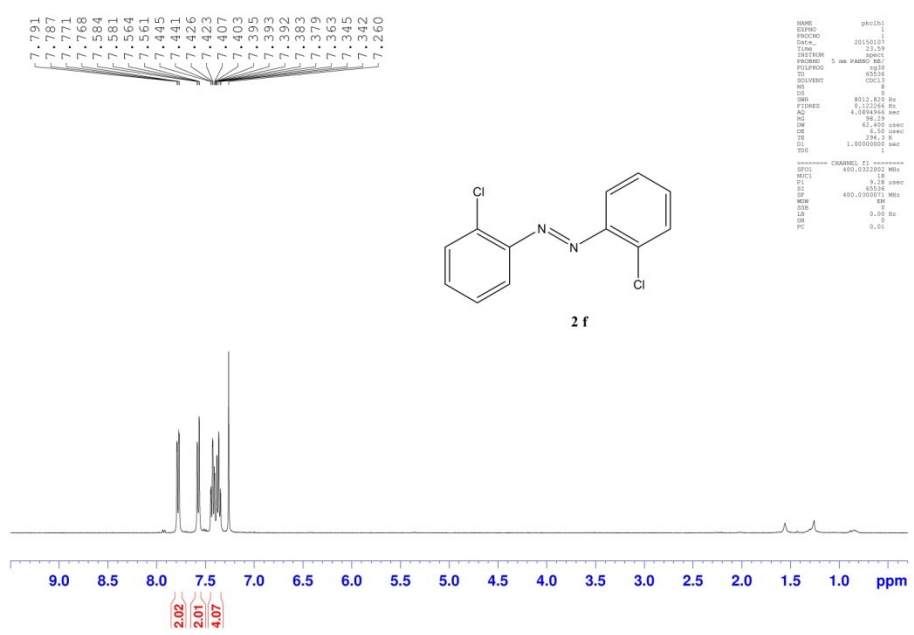


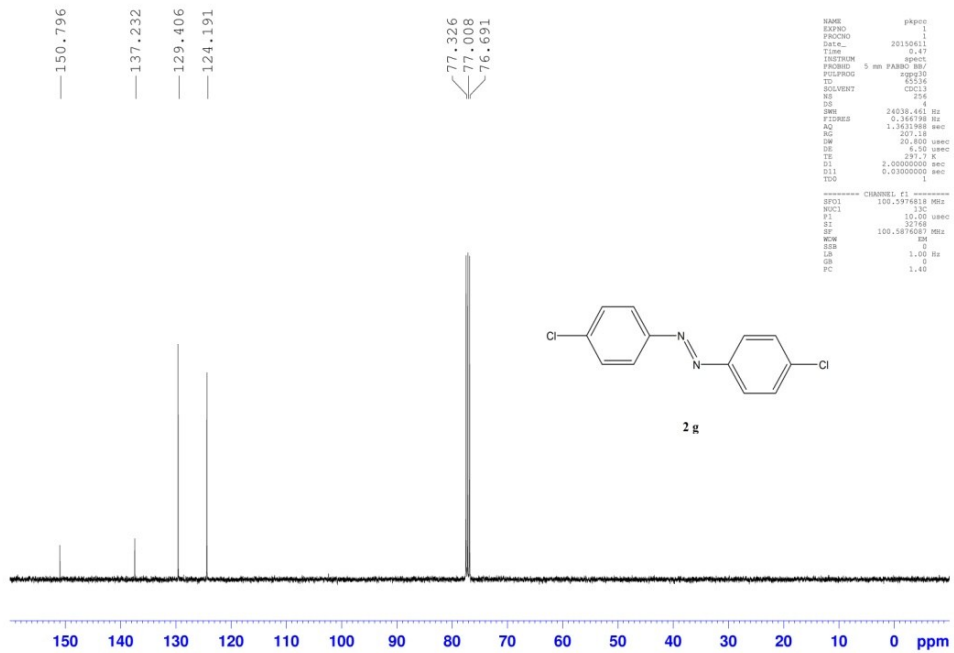
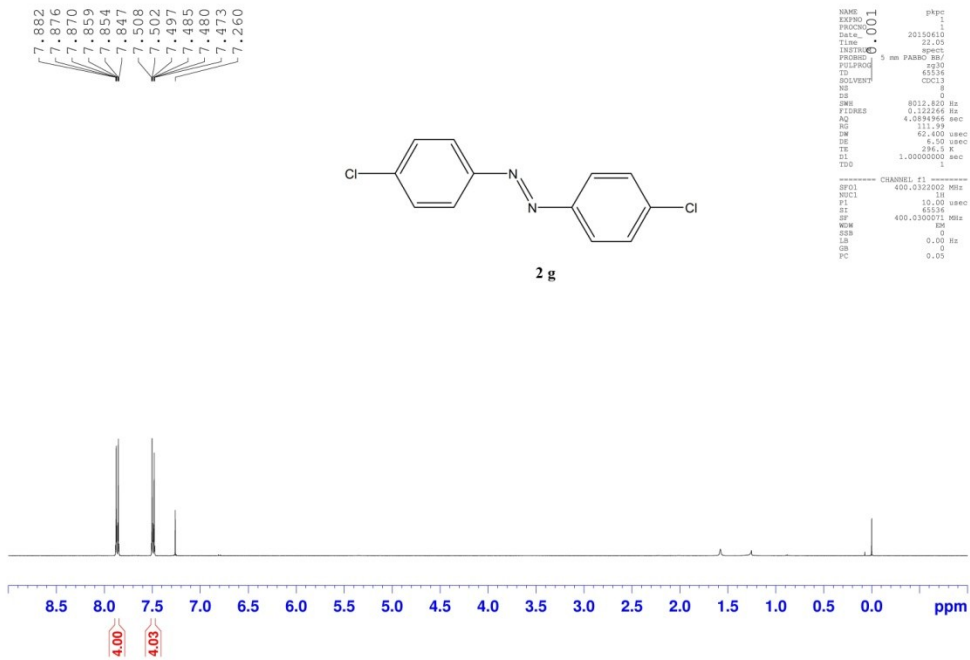








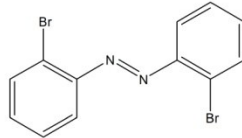




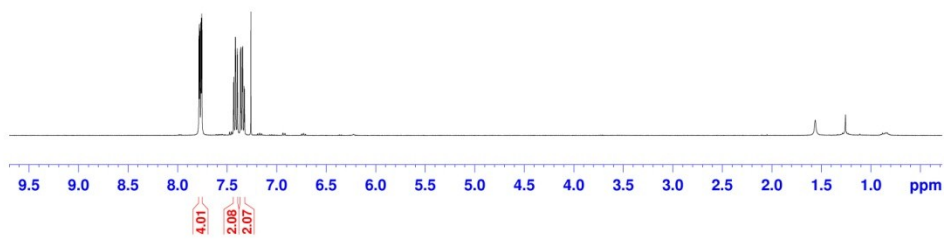
7.781
7.777
7.772
7.765
7.761
7.757
7.753
7.735
7.731
7.725
7.717
7.715
7.712
7.704
7.597
7.593
7.566
7.561
7.548
7.546
7.543
7.542
7.536
7.528
7.524
7.260

```

NAME      pkabrc
EXPNO     1
PROCNO    1
Date_     20150616
Time      4.31
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         4
DS         4
SWH        8012.400 Hz
FIDRES     0.122566 Hz
AQ         4.084966 sec
RG         111.99
WDW         62.400 usec
DE         6.50 usec
TE         296.6 K
D1         1.0000000 sec
TDO        1
  
```



2 h

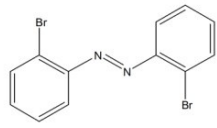


```

----- CHANNEL f1 -----
SFO1     400.032002 MHz
NUC1     1H
P1        9.28 usec
SI        65536
SF        400.030093 MHz
WDW       EM
SSB       0
LB        0.00 Hz
GB        0
PC        0.05
  
```

149.56
133.82
132.48
128.12
126.31
118.45

77.33
77.00
76.70



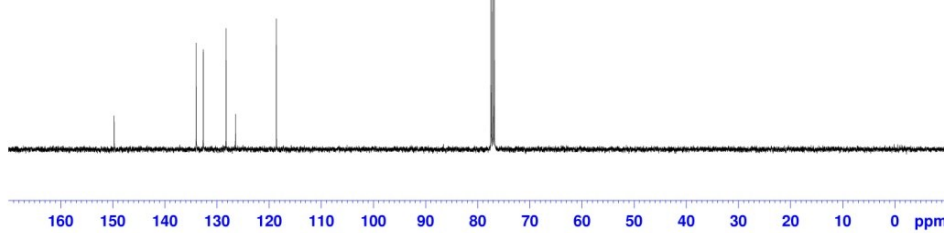
2 h

```

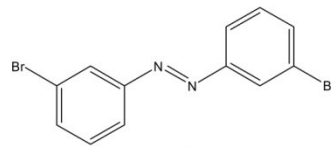
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EXPNO     2
PROCNO    1
Date_     20150616
Time      4.31
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         4
DS         4
SWH        24038.461 Hz
FIDRES     0.366798 Hz
AQ         1.3631988 sec
RG         207.18
WDW         20.800 usec
DE         6.50 usec
TE         297.2 K
D1         2.0000000 sec
D11        0.0300000 sec
TDO        1
  
```

```

----- CHANNEL f1 -----
SFO1     100.5976818 MHz
NUC1     13C
P1        13C
SI        32768
SF        100.5876093 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40
  
```



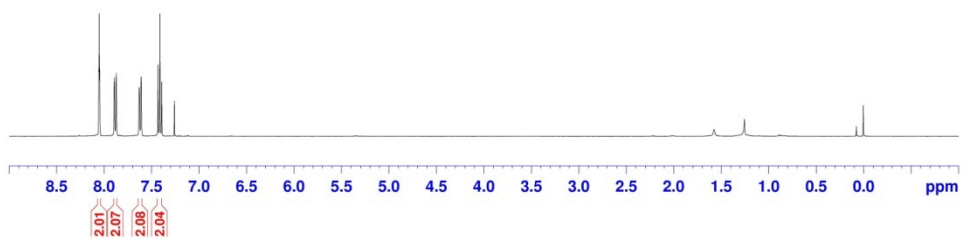
8.052
7.894
7.893
7.875
7.873
7.633
7.628
7.431
7.414
7.394
7.260



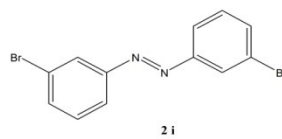
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NAME      pxhbc
EXPNO     1
PROCNO    1
Date_     20150610
Time      23.01
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         8
DS         8
SMB       8012.820 Hz
FIDRES    0.122268 Hz
AQ         4.0894964 sec
RG         85.28
DW         62.400 usec
DE         35.00 usec
TE         300.2 K
D1         1.0000000 sec
TD0        1

===== CHANNEL f1 =====
SF01      400.032002 MHz
NUC1      13C
P1         10.00 usec
PT         65536
SP         400.030073 MHz
Waltz16   on
SSB        0
LB         0.00 Hz
GB         0
PC         0.50
  
```



153.170
134.123
130.516
124.741
123.215

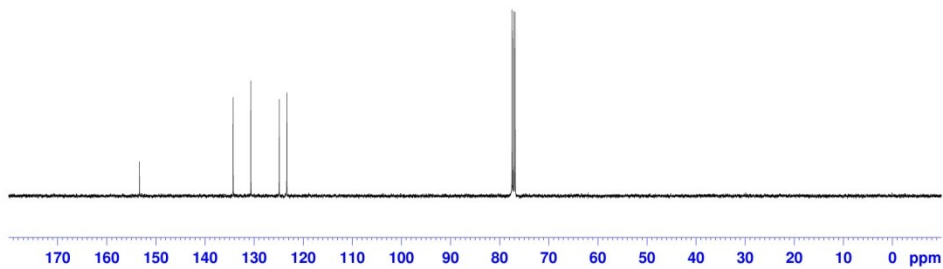


```

NAME      pxhbc
EXPNO     1
PROCNO    1
Date_     20150611
Time      0.28
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         8
DS         8
SMB       24038.461 Hz
FIDRES    0.366798 Hz
AQ         1.3621988 sec
RG         207.18
DW         20.800 usec
DE         65.00 usec
TE         297.5 K
D1         2.0000000 sec
D11        0.0300000 sec
TD0        1

===== CHANNEL f1 =====
SF01      100.5976818 MHz
NUC1      13C
P1         10.00 usec
PT         32768
SP         100.5876094 MHz
Waltz16   on
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
  
```

77.334
77.016
76.698

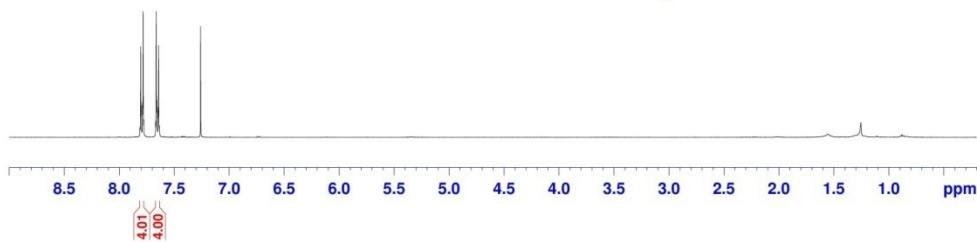
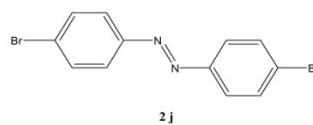


7.805
7.800
7.788
7.783
7.777
7.669
7.663
7.658
7.646
7.641
7.260

```

NAME      pkbrc
EXPNO     1
PROCNO    1
Date_     20150424
Time      22:07
INSTRUM   spect
PROBHD     5 mm PABBO BB/
PULPROG   zg30
TD         65536
SOLVENT    CDCl3
NS         4
DS         2
SWH        8012.820 Hz
FIDRES     0.122246 Hz
AQ         4.0894866 sec
RG         111.99
DW         62.400 usec
DE         6.50 usec
TE         292.7 K
D1         1.0000000 sec
TD0        1

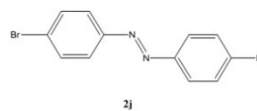
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NUC1       13
P1         9.18 usec
SI         65536
SF         400.432000 MHz
WDW        EM
SSB        0
LB         0.00 Hz
GB         0
PC         0.05
  
```



151.15
132.41
126.77
124.43

77.33
77.22
77.01

0.00



```

NAME      pkbrc
EXPNO     1
PROCNO    1
Date_     20150424
Time      22:56
INSTRUM   spect
PROBHD     5 mm PABBO BB/
PULPROG   zg30
TD         65536
SOLVENT    CDCl3
NS         4
DS         2
SWH        24038.461 Hz
FIDRES     0.366798 Hz
AQ         13.1631988 sec
RG         207.18
DW         20.800 usec
DE         6.50 usec
TE         296.5 K
D1         2.0000000 sec
D11        0.0300000 sec
TD0        1

===== CHANNEL f1 =====
SFO1      100.5976818 MHz
NUC1       13C
P1         9.00 usec
SI         32768
SF         100.5976818 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
  
```

