

## Supporting Information

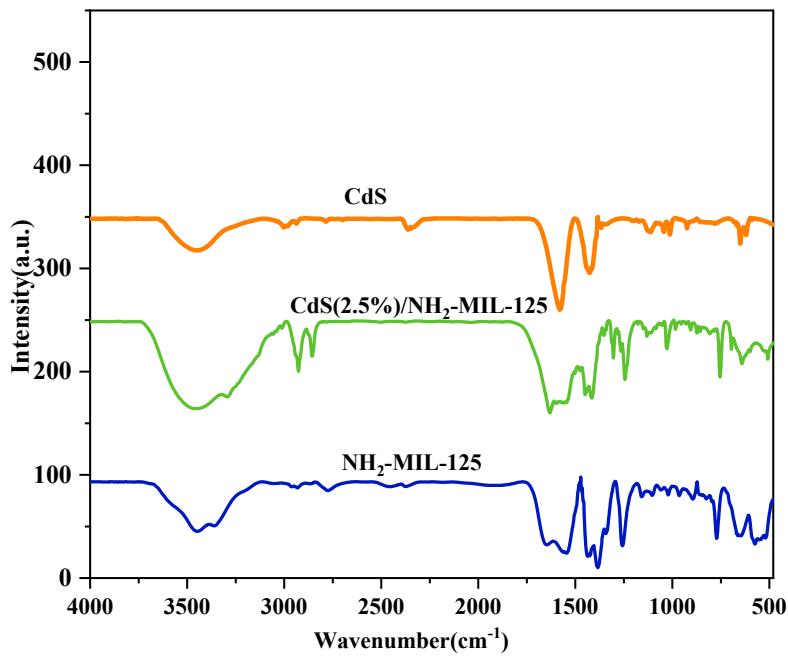
### Selective Photocatalytic Reduction of Nitrobenzene to Anilines, Azoxybenzene, and Azobenzene: A Solvent-Dependent and Light-Induced Process Mediated by CdS/NH<sub>2</sub>-MIL-125 Nanocomposite

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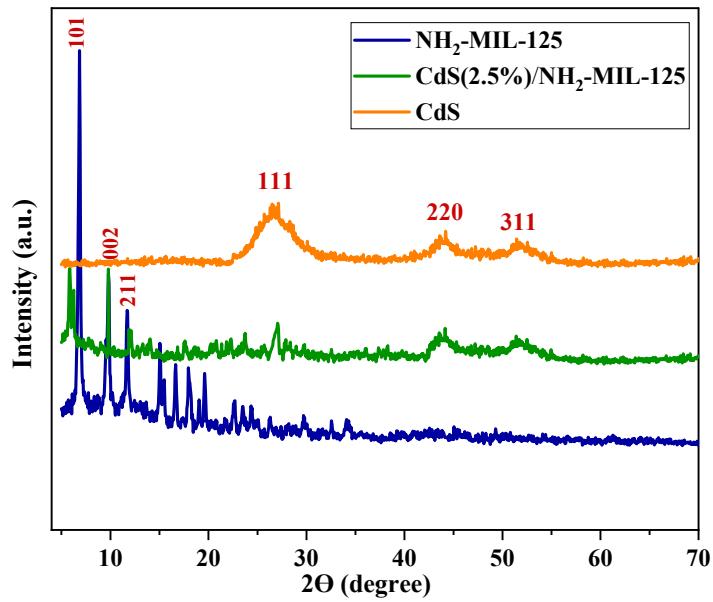
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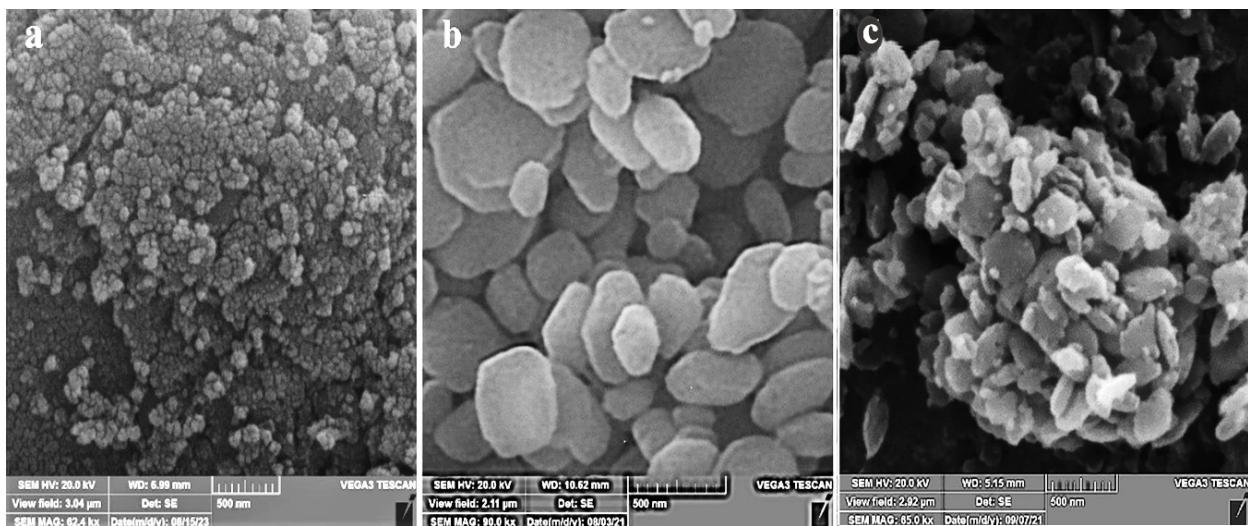
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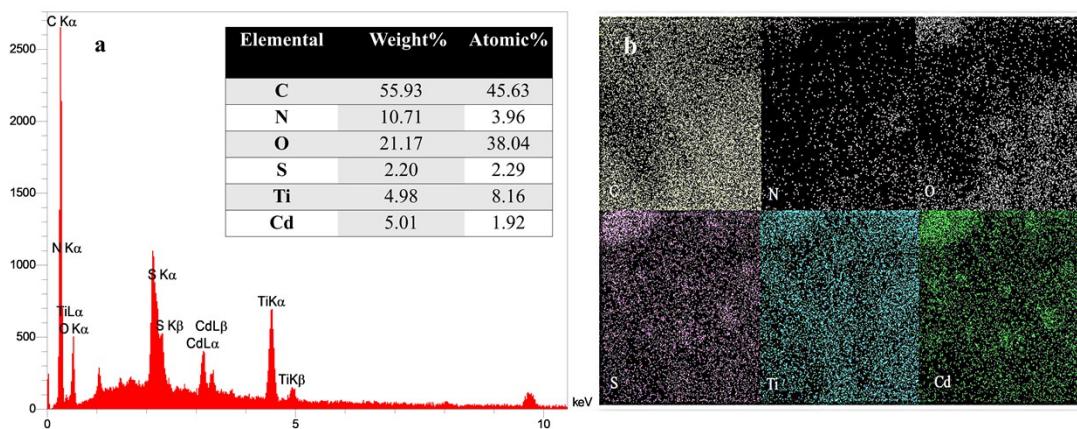
**Fig. S1.** FTIR Spectra of CdS, NH<sub>2</sub>-MIL-125, and CdS/NH<sub>2</sub>-MIL-125 nanocomposite



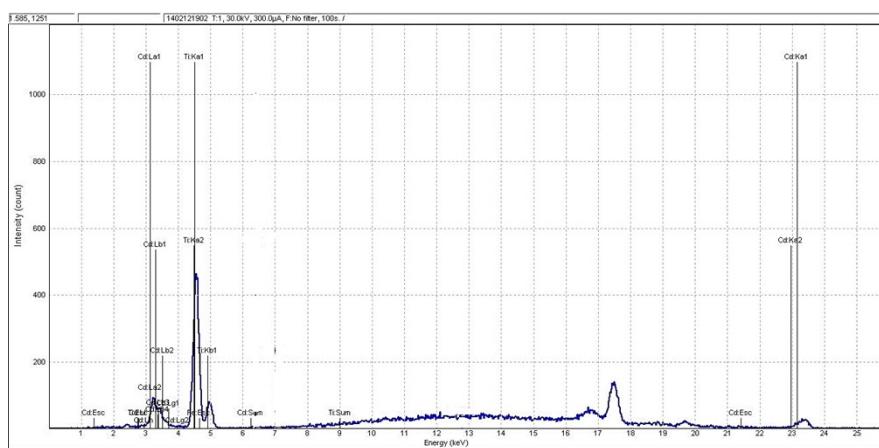
**Fig. S2.** XRD patterns of CdS, NH<sub>2</sub>-MIL-125, and CdS/NH<sub>2</sub>-MIL-125 nanocomposite



**Fig. S3.** SEM images of a) CdS, b) NH<sub>2</sub>-MIL-125, and c) CdS/NH<sub>2</sub>-MIL-125 nanocomposite

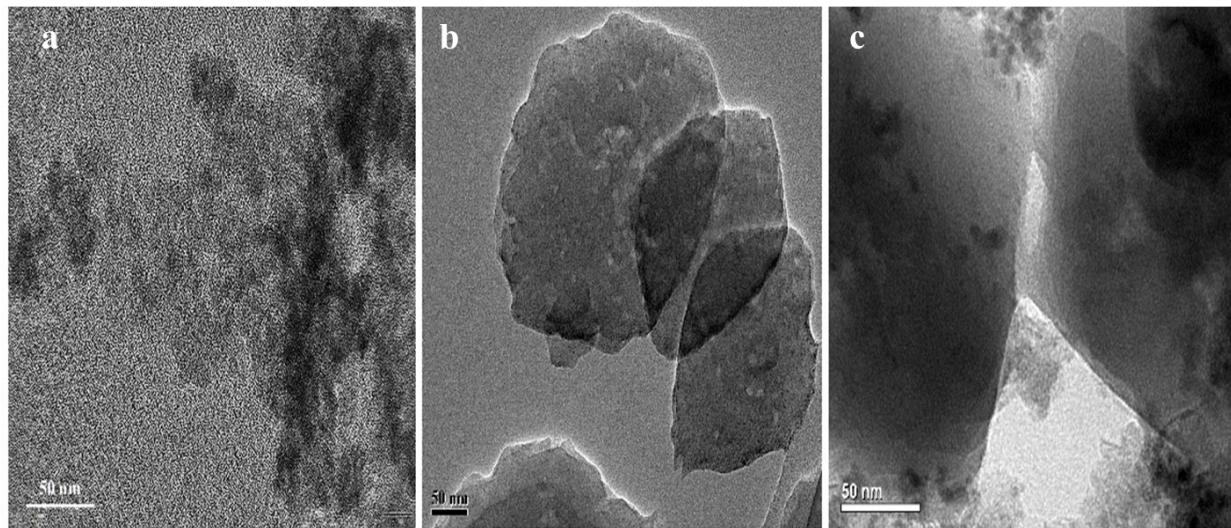


**Fig. S4.** (a) EDX point scan of the surface of CdS(2.5%)/NH<sub>2</sub>-MIL-125 nanocomposite; (b) EDX mapping of CdS(2.5%)/NH<sub>2</sub>-MIL-125

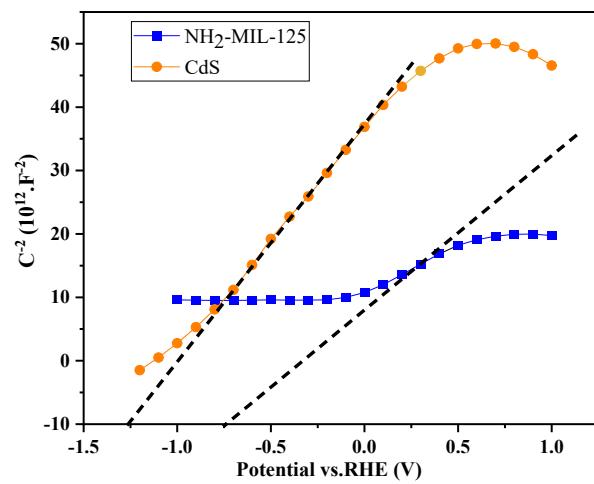


**Fig. S5.** XRF spectrum of the CdS(2.5%)/NH<sub>2</sub>-MIL-125 nanocomposite.

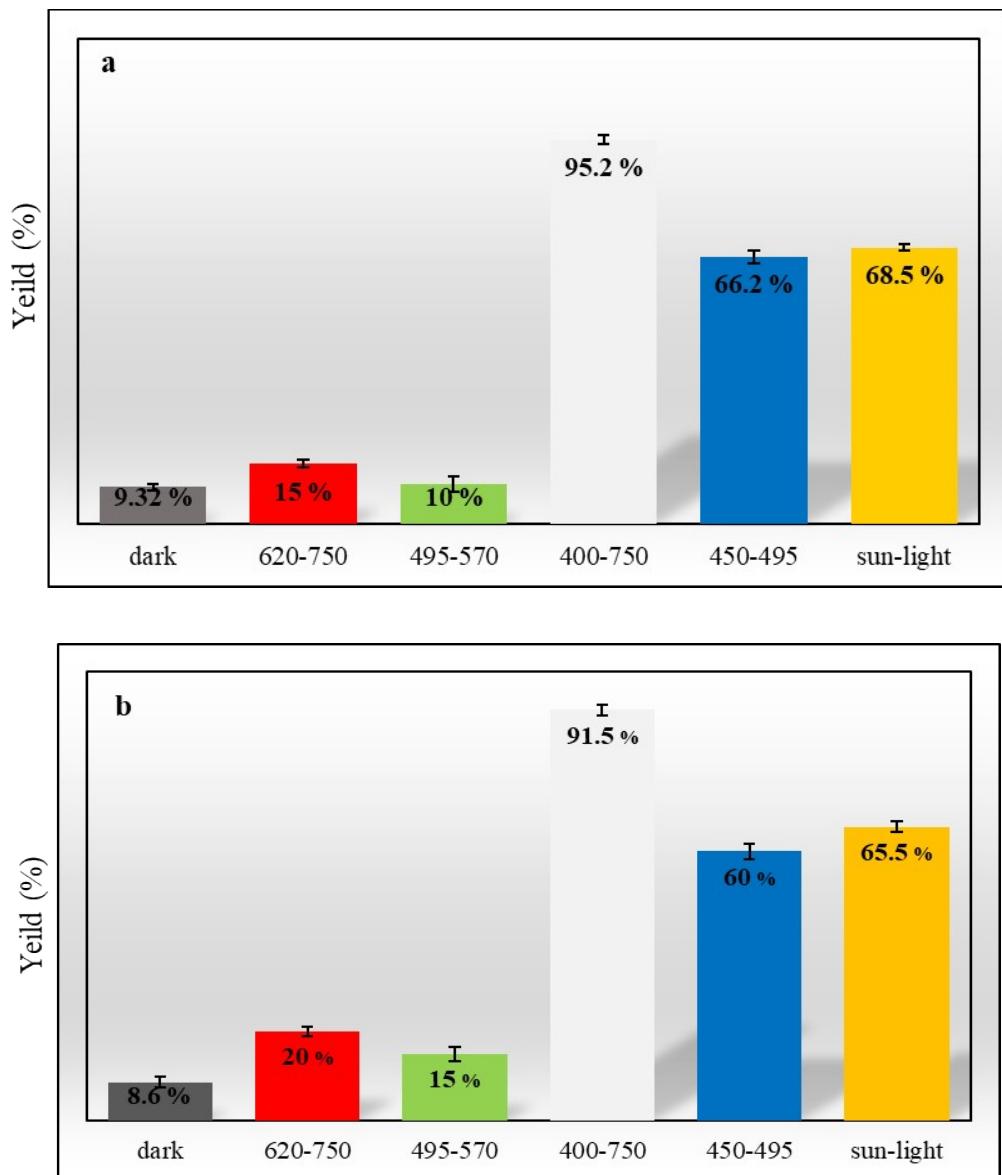
The concentration and intensity of cadmium (Cd) in the XRF spectrum of the CdS(2.5%)/NH<sub>2</sub>-MIL-125 nanocomposite.



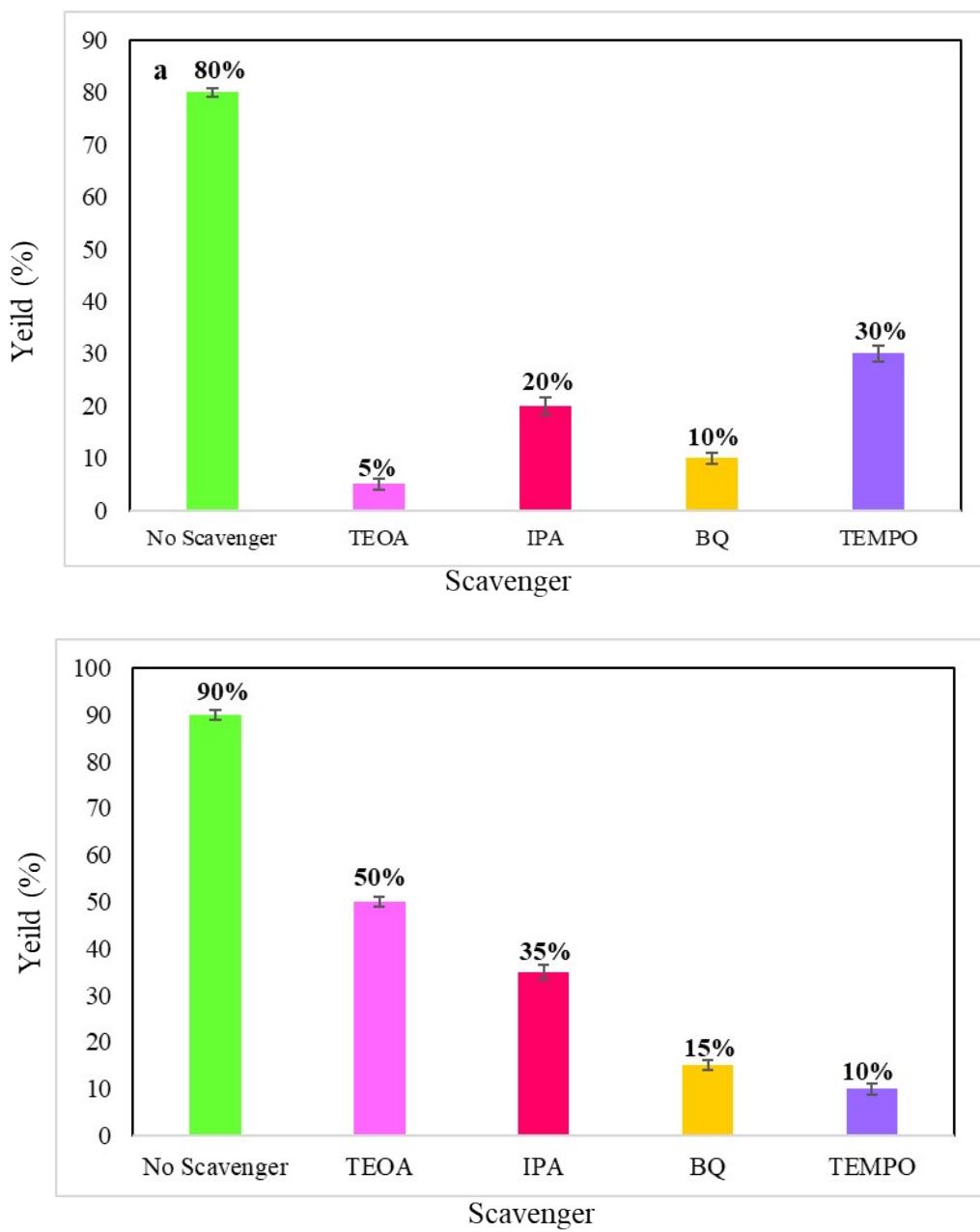
**Fig. S6.** HRTEM images of a) CdS, b) NH<sub>2</sub>-MIL-125, and c) CdS/NH<sub>2</sub>-MIL-125 nanocomposite



**Fig. S7.** Mott-Schottky plots of the (a) pure CdS and (b) NH<sub>2</sub>-MIL-125.



**Fig S8.** Efficiency of reduction reaction of nitroarenes to a) aniline, b) azobenzene product under different LEDs.



**Fig S9.** Control experiments for a) reduction of nitroarenes to azo-compounds b) reduction of nitroarenes to aniline.

**Table S1.** The concentration and intensity of cadmium (Cd) in the XRF spectrum of the CdS(2.5%)/NH<sub>2</sub>-MIL-125 nanocomposite.

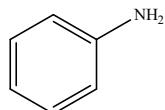
Composition	Elements	Concentration	Intensity
CdS (2.5%)/NH <sub>2</sub> -MIL-125	Ti Cd	38.7 16.6	97.9 62.1

**Table S2.** Results BET for NH<sub>2</sub>-MIL-125 and CdS/NH<sub>2</sub>-MIL-125 nanocomposite

Photocatalyst	BET surface area (m <sup>2</sup> .g <sup>-1</sup> )	Total pore volume (cm <sup>3</sup> .g <sup>-1</sup> )	Average pore diameter (nm)
NH <sub>2</sub> -MIL-125	396.9	0.39	3.9
CdS/NH <sub>2</sub> -MIL-125	199.5	0.33	6.55

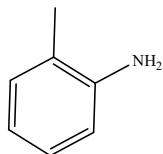
### **<sup>1</sup>H NMR spectra of the synthesized compounds:**

#### Aniline (**2a**)



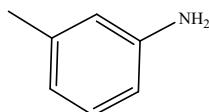
Yellow liquid, 85% yield; <sup>1</sup>H NMR (400 MHz, DMSO-*d*6); δ (ppm) = 7.0 (t, *J* = 8 Hz, 2H, ArH), 6.5 (d, *J* = 8 Hz, 2H, ArH), 6.64 (t, *J* = 8 Hz, 1H, ArH), 4.98 (s, 2H, NH<sub>2</sub>). <sup>13</sup>C NMR (DMSO, 101 MHz) δ (ppm) = 148.9, 129.3, 122.9, 115.4.

#### *o*-Toluidine (**2b**)

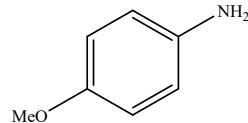


Black liquid, 80% yield; <sup>1</sup>H NMR (400 MHz, DMSO); δ (ppm) = 6.92 (t, *J* = 8 Hz, 1H, ArH), 6.87 (t, *J* = 8 Hz, 1H, ArH), 6.62 (d, *J* = 8 Hz, 1H, ArH), 6.47 (d, *J* = 8 Hz, 1H, ArH), 4.78 (s, 2H, NH<sub>2</sub>), 2.05 (s, 3H, CH<sub>3</sub>). <sup>13</sup>C NMR (101 MHz, DMSO) δ (ppm) = 146.9, 130.3, 126.9, 121.4, 116.4, 114.3, 17.9.

#### *m*-Toluidine (**2c**)



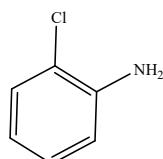
Yellow liquid, 78.3.3% yield;  $^1\text{H}$  NMR (400 MHz, DMSO);  $\delta$  (ppm) = 6.89 (t,  $J$  = 8 Hz, 1H, ArH), 6.41 (d,  $J$  = 8 Hz, 1H, ArH), 6.35 (s, 1H, ArH), 6.33 (d,  $J$  = 8 Hz, 1H, ArH), 5.93 (s, 2H, NH<sub>2</sub>), 2.16 (s, 3H, Me).  $^{13}\text{C}$  NMR (101 MHz, DMSO)  $\delta$  (ppm) = 148.9, 138.2, 129.1, 117, 114.7, 111.6, 21.7.



4-Methoxyaniline (**2d**)

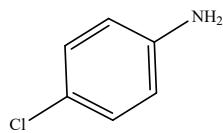
Black solid, 88% yield;  $^1\text{H}$  NMR (400 MHz, DMSO);  $\delta$  (ppm) = 7.50 (d,  $J$  = 8 Hz, 2H, ArH), 7.09 (d,  $J$  = 8 Hz, 2H, ArH), 5.42 (s, 2H, NH<sub>2</sub>), 4.04 (s, 3H, OCH<sub>3</sub>).  $^{13}\text{C}$  NMR (101 MHz, DMSO)  $\delta$  (ppm) = 142.6, 141.8, 129.3, 121.5, 118.8, 118.

2-Chloroaniline (**2e**)



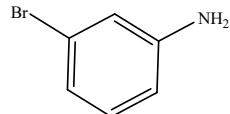
Yellow solid; 75% yield;  $^1\text{H}$  NMR (400 MHz, DMSO)  $\delta$  (ppm) = 8.34 (d,  $J$  = 4 Hz, 1H, ArH), 8.08 (t,  $J$  = 4 Hz, 1H, ArH), 7.38 (t,  $J$  = 8 Hz, 1H, ArH), 7.18 (d,  $J$  = 8 Hz, 1H, ArH), 6.68 (s, 2H, NH<sub>2</sub>).  $^{13}\text{C}$  NMR (101 MHz, DMSO)  $\delta$  (ppm) = 142.6, 141.8, 129.3, 121.5, 118.8, 118.

4-Chloroaniline (**2f**)



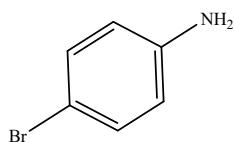
Yellow solid, 80% yield;  $^1\text{H}$  NMR (400 MHz, DMSO)  $\delta$  (ppm) = 7.13 (d,  $J$  = 8 Hz, 2H, ArH), 6.54 (d,  $J$  = 8 Hz, 2H, ArH), 5.27 (s, 2H, NH<sub>2</sub>).  $^{13}\text{C}$  NMR (76 MHz, DMSO)  $\delta$  (ppm) = 148.5, 131.8, 116.2, 106.5.

3-Bromoaniline (**2g**)



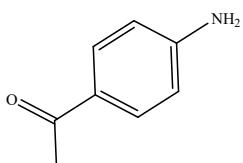
Brown solid, 83% yield;  $^1\text{H}$  NMR (301 MHz, DMSO)  $\delta$  (ppm) = 6.96 (d,  $J$  = 8 Hz, 1H, ArH), 7.26 (s, 1H, ArH), 7.30 (d,  $J$  = 8 Hz, 1H, ArH), 7.37 (t,  $J$  = 8 Hz, 1H, ArH), 5.83 (s, 2H, NH<sub>2</sub>).  $^{13}\text{C}$  NMR (75 MHz, DMSO)  $\delta$  (ppm) = 131.8, 129.1, 128.8, 127, 124.6, 120.2.

#### 4-Bromoaniline (**2h**)

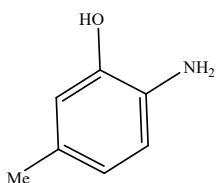


Brown solid; 73% yield;  $^1\text{H}$  NMR (400 MHz, DMSO)  $\delta$  (ppm) = 7.59 (d,  $J$  = 8 Hz, 2H, ArH), 6.56 (d,  $J$  = 8 Hz, 2H, ArH), 5.87 (s, 2H, NH<sub>2</sub>).  $^{13}\text{C}$  NMR (101 MHz, DMSO)  $\delta$  (ppm) = 147.6, 128.4, 118.6, 115.1.

#### 1-(4-Aminophenyl) ethenone (**2i**)



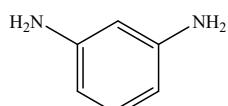
Yellow solid, 70% yield;  $^1\text{H}$  NMR (400 MHz, DMSO)  $\delta$  (ppm) = 7.72 (d,  $J$  = 12 Hz, 2H, ArH), 6.62 (d,  $J$  = 12 Hz, 2H, ArH), 6.05 (s, 2H, NH<sub>2</sub>), 2.40 (s, 3H, Me).  $^{13}\text{C}$  NMR (101 MHz, DMSO)  $\delta$  (ppm) = 195.4, 154, 131, 125.3, 112.9, 26.3.



#### 2-Amino-5-methylphenol (**2j**)

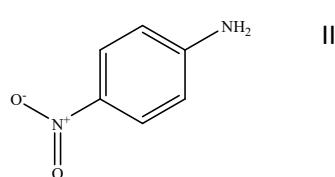
Yellow solid; 75% yield.  $^1\text{H}$  NMR (400 MHz, DMSO)  $\delta$  (ppm) = 8.70 (s, 1H, OH), 6.51 (d,  $J$  = 8 Hz, 2H, ArH), 6.42 (s, 2H, ArH), 6.22 (d,  $J$  = 8 Hz, 2H, ArH), 4.41 (s, 2H, NH<sub>2</sub>), 2.10 (s, 3H, Me).  $^{13}\text{C}$  NMR (101 MHz, DMSO)  $\delta$  (ppm) = 142.2, 136.7, 128.2, 117.1, 115.6, 114.7, 21.

#### Benzene-1,3-diamine (**2k**)



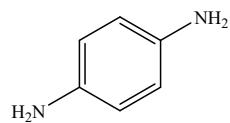
Yellow solid; 60% yield;  $^1\text{H}$  NMR (400 MHz, DMSO)  $\delta$  (ppm) = 7.38 (t, 1H, ArH), 7.26 (s, 1H, ArH), 6.96 (dd, 1H, ArH), 6.94 (dd,  $J$  = 8 Hz, 1H, ArH), 5.82 (s, 4H, NH<sub>2</sub>).  $^{13}\text{C}$  NMR (101 MHz, DMSO)  $\delta$  (ppm) = 131.2, 130.2, 122.6, 121.9.

#### 4-Nitroaniline (**2l**)



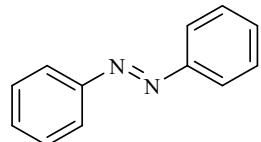
Yellow solid; 20% yield;  $^1\text{H}$  NMR (400 MHz, DMSO);  $\delta$  (ppm) = 7.96 (d,  $J$  = 12 Hz, 2H, ArH), 6.61 (d,  $J$  = 12 Hz, 2H, ArH), 6.75 (s, 2H, NH<sub>2</sub>).  $^{13}\text{C}$  NMR (101 MHz, DMSO)  $\delta$  (ppm) = 167.4, 152.2, 131.1, 113.1.

### Benzene-1,4-diamine (**2m**)



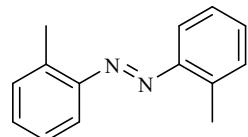
White solid; 50% yield;  $^1\text{H}$  NMR (400 MHz, DMSO)  $\delta$  (ppm) = 6.39 (s, 4H, ArH), 4.20 (s, 4H, NH<sub>2</sub>).  $^{13}\text{C}$  NMR (76 MHz, DMSO)  $\delta$  (ppm) = 139.4, 115.9.

### 1,2-Diphenyldiazene (**3a**)



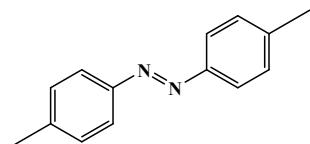
Yellow solid, 18 mg, 80% yield; m.p.: 67-68 °C [Lit, (Wang et, al. 2023, Yan et, al. 2020) 66-67 °C].  $^1\text{H}$  NMR (400 MHz, CDCl<sub>3</sub>);  $\delta$  (ppm) = 8.27 (d,  $J$  = 12 Hz, 4H, ArH), 7.75 (t,  $J$  = 12 Hz, 2H, ArH), 7.57 (d,  $J$  = 12 Hz, 4H, ArH).  $^{13}\text{C}$  NMR (CDCl<sub>3</sub>, 125 MHz)  $\delta$  (ppm) = 163.7, 147, 129.1, 120.2.

### 1,2-Di-o-tolyldiazene (**3b**)



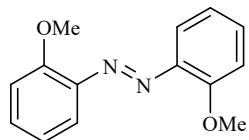
Dark Yellow solid, 16.2 mg, 75% yield; m.p.: 140-141 °C [Lit, (Wang et, al. 2023, Yan et, al. 2020) 142-143 °C].  $^1\text{H}$  NMR (400 MHz, CDCl<sub>3</sub>);  $\delta$  (ppm) = 7.75 (m, 2H, ArH), 7.49 (d,  $J$  = 12 Hz, 2H, ArH), 7.31 (m, 2H, ArH), 7.09 (d,  $J$  = 14.6 Hz, 2H, ArH), 2.45 (s, 6H, Me).  $^{13}\text{C}$  NMR (CDCl<sub>3</sub>, 125 MHz)  $\delta$  (ppm) = 130, 129.5, 124.9, 122.8, 119.9, 115.7, 29.7.

### 1,2-Di-p-tolyldiazene (**3c**)



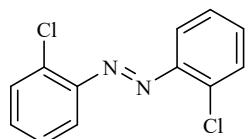
Yellow solid, 20 mg, 73% yield; m.p.: 139-140 °C [Lit, (Wang et, al. 2023, Yan et, al. 2020) 140-142 °C].  
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>); δ (ppm) = 8.12 (d, *J* = 8.1 Hz, 4H, ArH), 7.30 (d, *J* = 8.1 Hz, 4H, ArH), 2.49 (s, 6H, CH<sub>3</sub>). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 125 MHz) δ (ppm) = 146.2, 129.7, 123.7, 21.6.

#### 1,2-Bis(2-methoxyphenyl) diazene (**3d**)



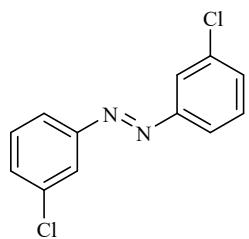
Orang solid, 14.2 mg, 78% yield; m.p.: 154-156 °C [Lit, (Wang et, al. 2023, Yan et, al. 2020) 156-157 °C].  
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>); δ (ppm) = 8.26 (d, *J* = 8 Hz, 2H), 7.92 (d, *J* = 8 Hz, 2H, ArH), 6.93-6.96 (dd, 4H, ArH), 3.82 (s, 6H, OCH<sub>3</sub>). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 125 MHz) δ (ppm) = 161.6, 147.1, 124.4, 114.2, 55.6.

#### 1,2-Bis(2-chlorophenyl) diazene (**3e**)



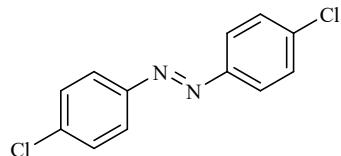
Orang solid, 12 mg, 65% yield; m.p.: 189-191 °C [Lit, (Wang et, al. 2023, Yan et, al. 2020) 186-187 °C].  
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>); δ (ppm) = 9.11 (t, *J* = 8 Hz, 2H, ArH), 8.62 (dd, 4H, ArH), 7.84 (t, *J* = 8 Hz, 2H, ArH). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 125 MHz) δ (ppm) = 159.6, 135.26, 131.3, 130.2, 122.4, 121.9.

#### 1,2-Bis(3-chlorophenyl) diazene (**3f**)



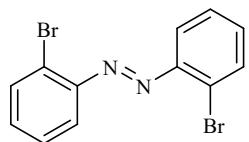
Yellow solid, 16.2 mg, 70% yield; m.p.: 182-183 °C [Lit, (Wang et, al. 2023, Yan et, al. 2020) 180-181 °C]. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>); δ (ppm) = 7.92 (s, 2H, ArH), 7.87 (t, 2H, ArH), 7.50 (m, 4H, ArH). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 125 MHz) δ (ppm) = 159.1, 136.2, 131.3, 130.2, 129.4, 124.2.

#### 1,2-Bis(4-chlorophenyl) diazene (**3g**)



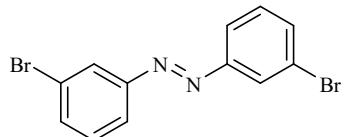
Yellow solid, 17mg, 73% yield; m.p: 180-182°C [Lit, (Wang et, al. 2023, Yan et, al. 2020) 178-179 °C].  
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>); δ (ppm) = 7.87 (d, *J* = 8 Hz, 4H, ArH), 7.49 (d, *J* = 8 Hz, 4H, ArH). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 125 MHz) δ (ppm) = 163.9, 147.9, 128.6, 120.8.

**1,2-Bis(2-bromophenyl) diazene (**3h**)**



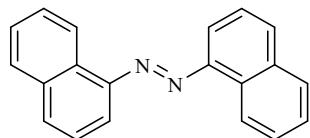
Yellow solid, 11 mg, 63% yield; m.p.: 202-204 °C [Lit, (Wang et, al. 2023, Yan et, al. 2020) 200-201 °C].  
 $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ );  $\delta$  (ppm) = 7.75 (dd, 2H, ArH), 7.64 (t,  $J=2.4$ , 2H, ArH), 7.35 (t,  $J=8$ , 2H, ArH), 7.10 (dd, 2H, ArH).  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125 MHz)  $\delta$  (ppm) = 129.6, 129.3, 128.5, 128.4, 127.1, 123.6.

**1,2-Bis(3-bromophenyl) diazene (**3i**)**

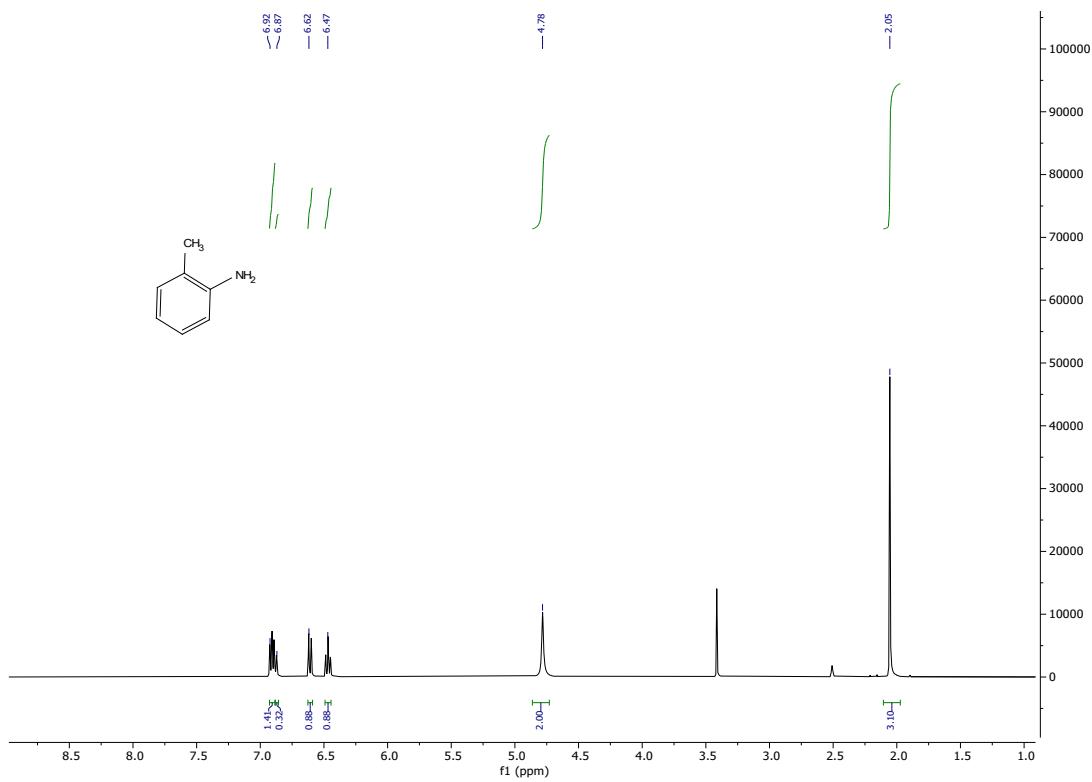


Yellow solid, 13 mg, 65% yield; m.p: 201-203 °C [Lit, (Wang et, al. 2023, Yan et, al. 2020) 198-199 °C].  
 $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ );  $\delta$  (ppm) = 7.64 (d,  $J=8$ , 2H, ArH), 7.42 (d,  $J=8$ , 2H, ArH), 7.32 (s, 2H, ArH), 7.24 (t,  $J=8$ , 2H, ArH).  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125 MHz)  $\delta$  (ppm) = 129.3, 123.2, 122.6, 118.2, 117.6, 108.4.

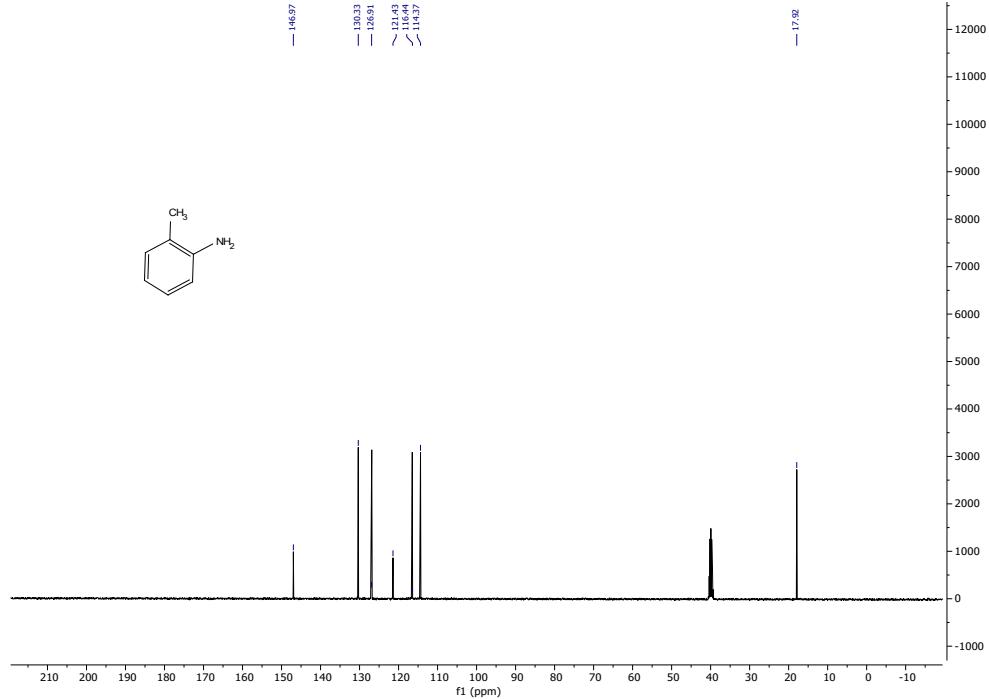
**1,2-Di(naphthalen-1-yl) diazene (**3j**)**



Yellow solid, 20 mg, 74% yield; m.p.: 250-252 °C. [Lit, (Wang et, al. 2023, Yan et, al. 2020) 249-250 °C].  
 $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ );  $\delta$  (ppm) = 7.42 (m, 2H, ArH), 7.26 (d,  $J=8$  Hz, 4H, ArH), 6.98 (m, 2H, ArH), 6.84 (d,  $J=8.4$  Hz, 4H, ArH), 6.66 (t,  $J=6$  Hz, 2H, ArH).  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125 MHz)  $\delta$  (ppm) = 129.6, 129.3, 128.5, 128.4, 127.1, 123.6.

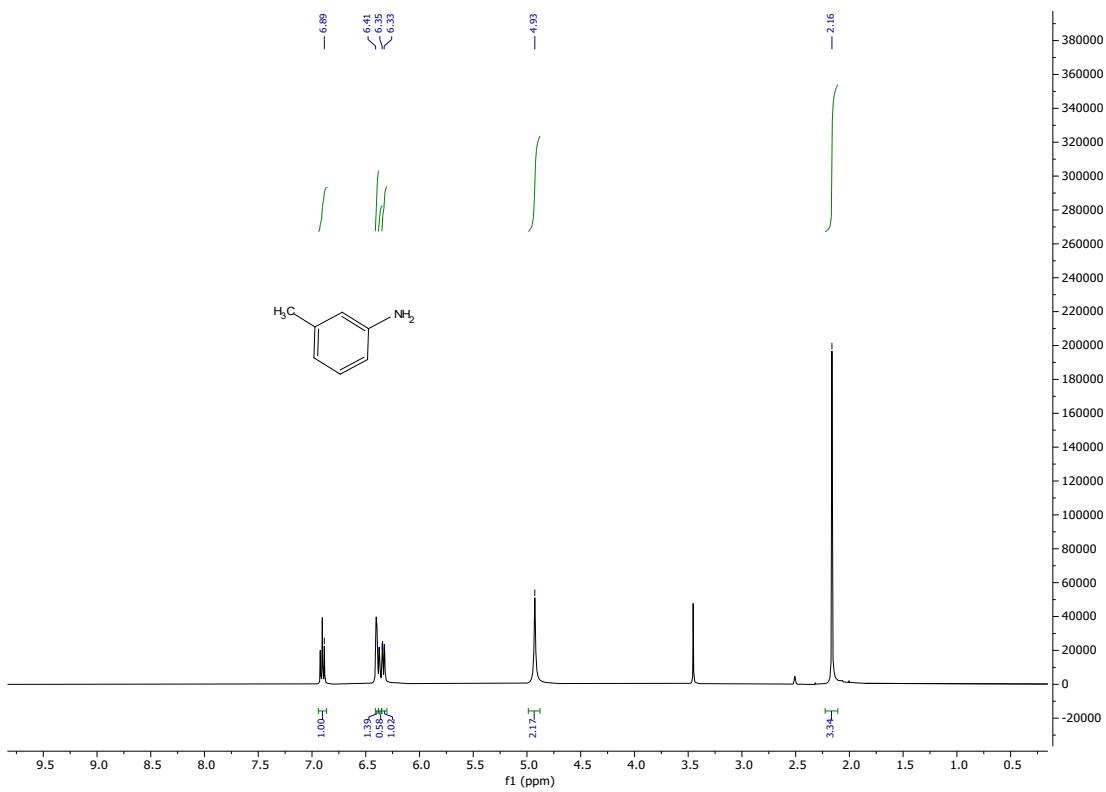


**Fig. S7.**  $^1\text{H}$ NMR (400 MHz, DMSO) spectrum of 2b.

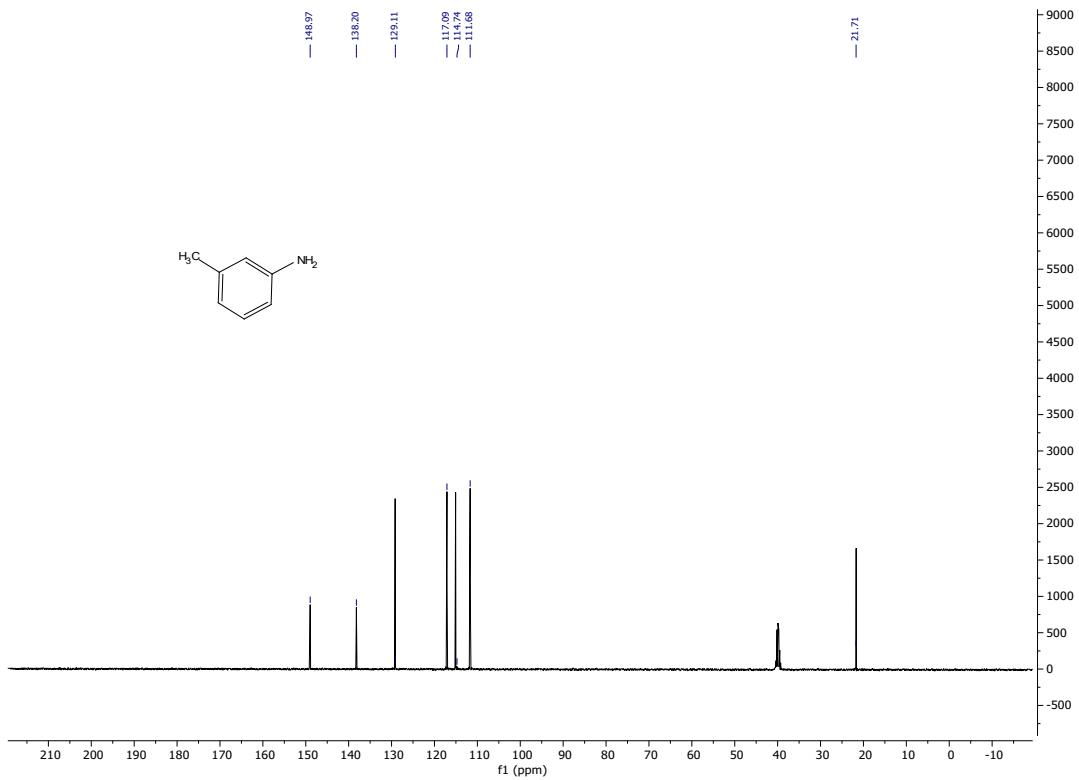


**Fig. S8.**  $^{13}\text{C}$ NMR (400 MHz, DMSO) spectrum of 2b.

MM

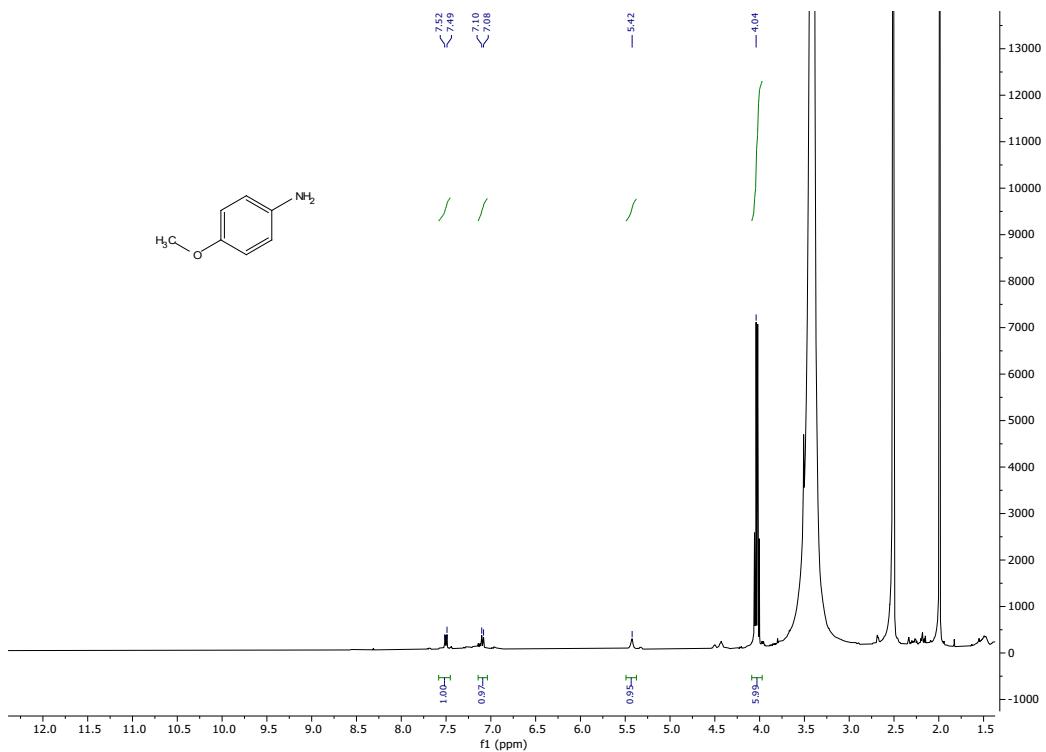


**Fig. S9.**  $^1\text{H}$ NMR (400 MHz, DMSO) spectrum of 2c.

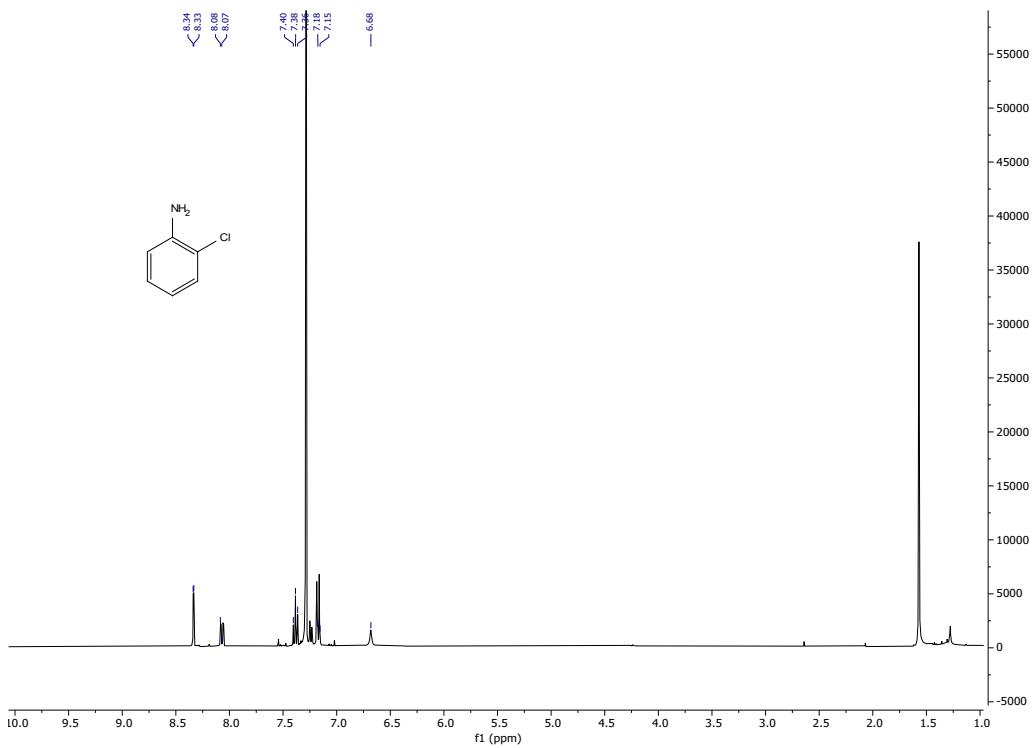


**Fig. S10.**  $^{13}\text{C}$ NMR (400 MHz, DMSO) spectrum of 2c.

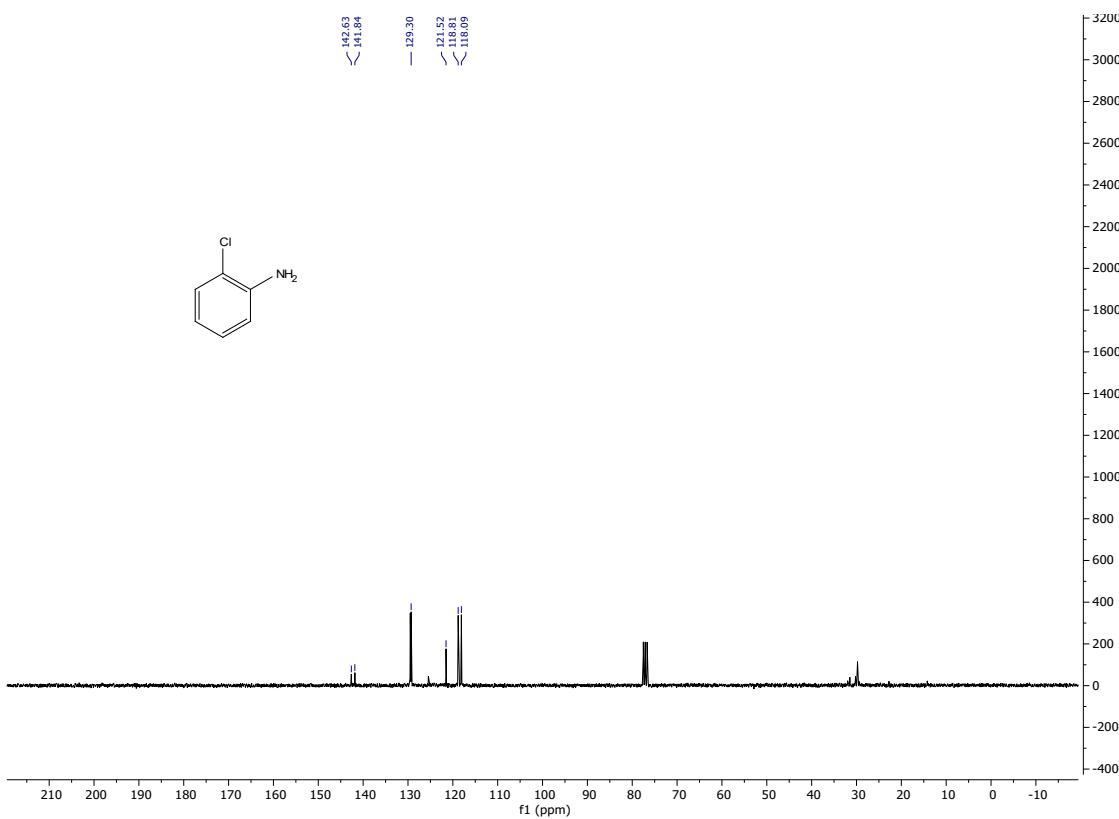
NN



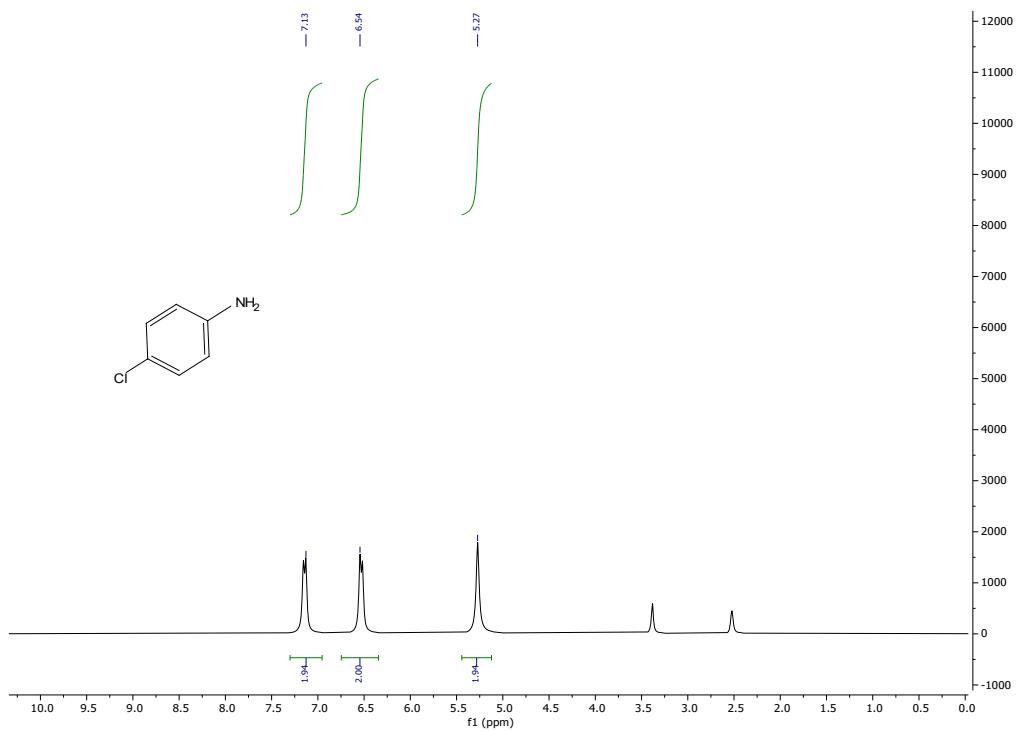
**Fig. S11.**  $^1\text{H}$ NMR (400 MHz, DMSO) spectrum of 2d.



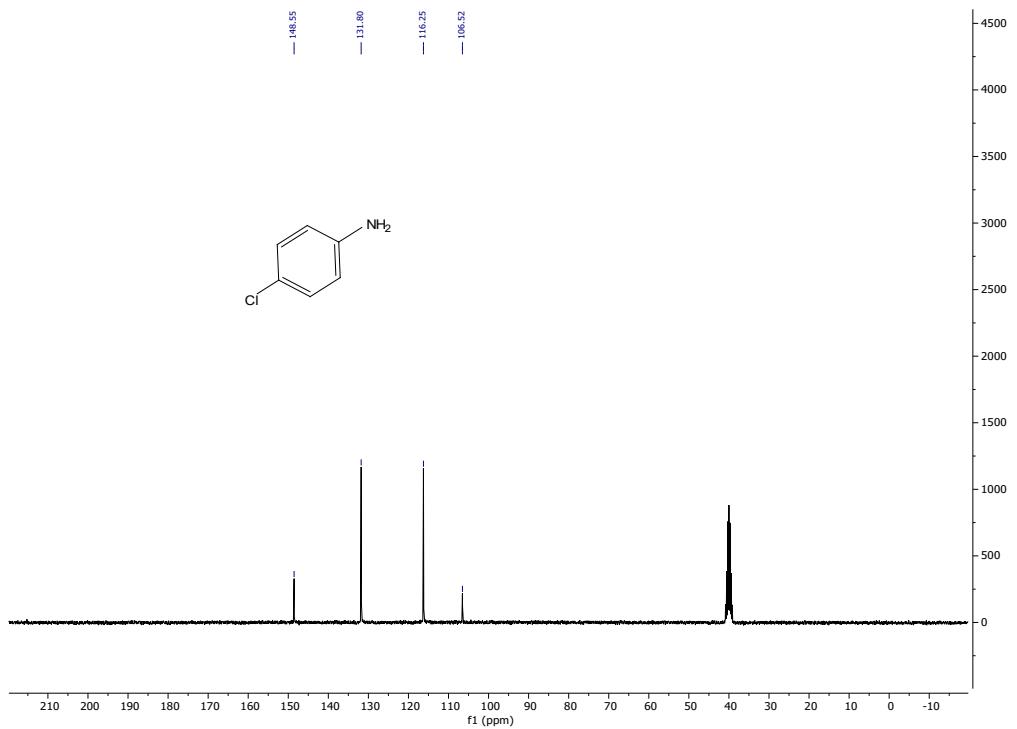
**Fig. S12.**  $^1\text{H}$ NMR (400 MHz, DMSO) spectrum of 2e.



**Fig. S13.**  $^{13}\text{C}$ NMR (400 MHz, DMSO) spectrum of 2e.

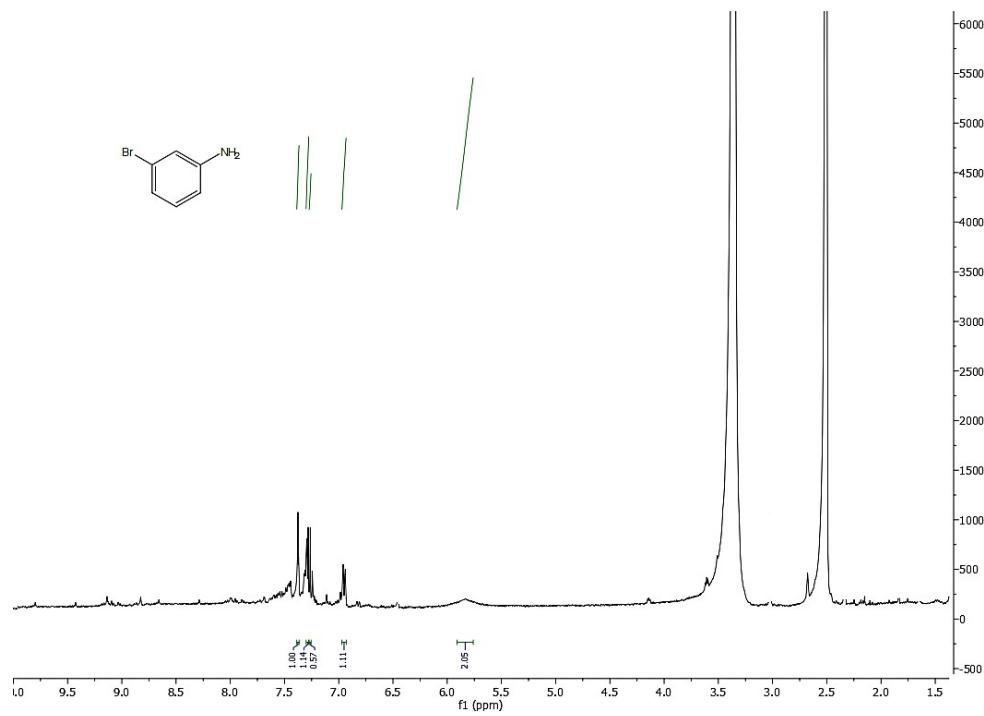


**Fig. S14.**  $^1\text{H}$ NMR (400 MHz, DMSO) spectrum of 2f.

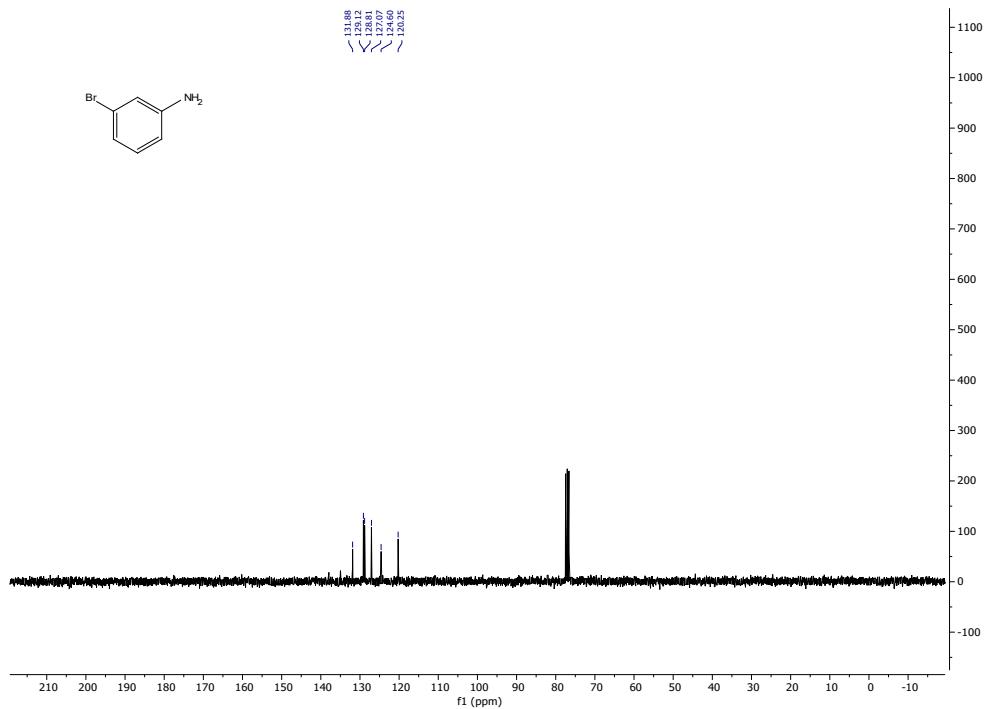


**Fig. S15.**  $^{13}\text{C}$ NMR (400 MHz, DMSO) spectrum of 2f.

QQ

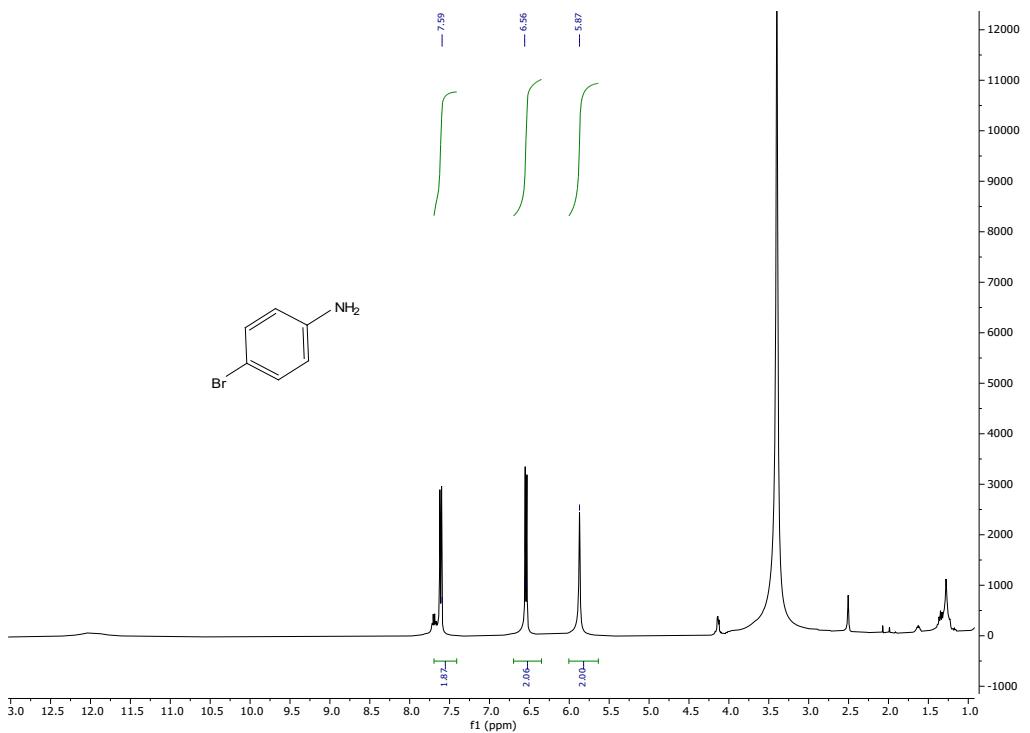


**Fig. 16.**  $^1\text{H}$ NMR (400 MHz, DMSO) spectrum of 2g.

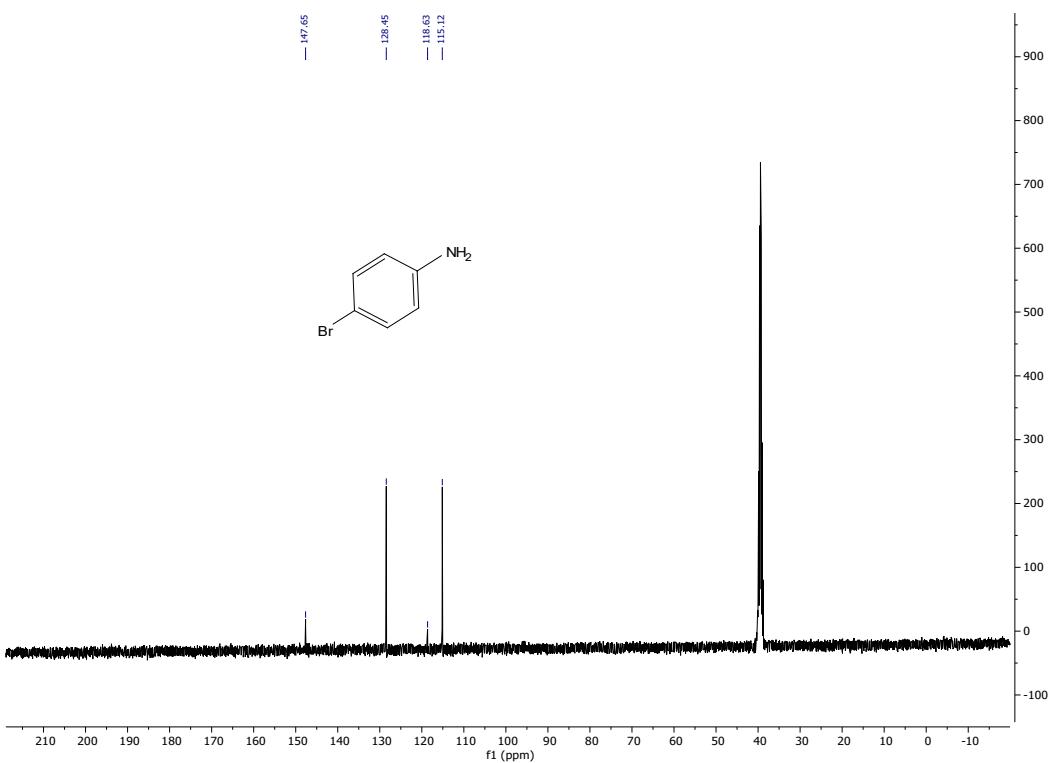


**Fig. S17.**  $^1\text{H}$ NMR (400 MHz, DMSO) spectrum of 2g.

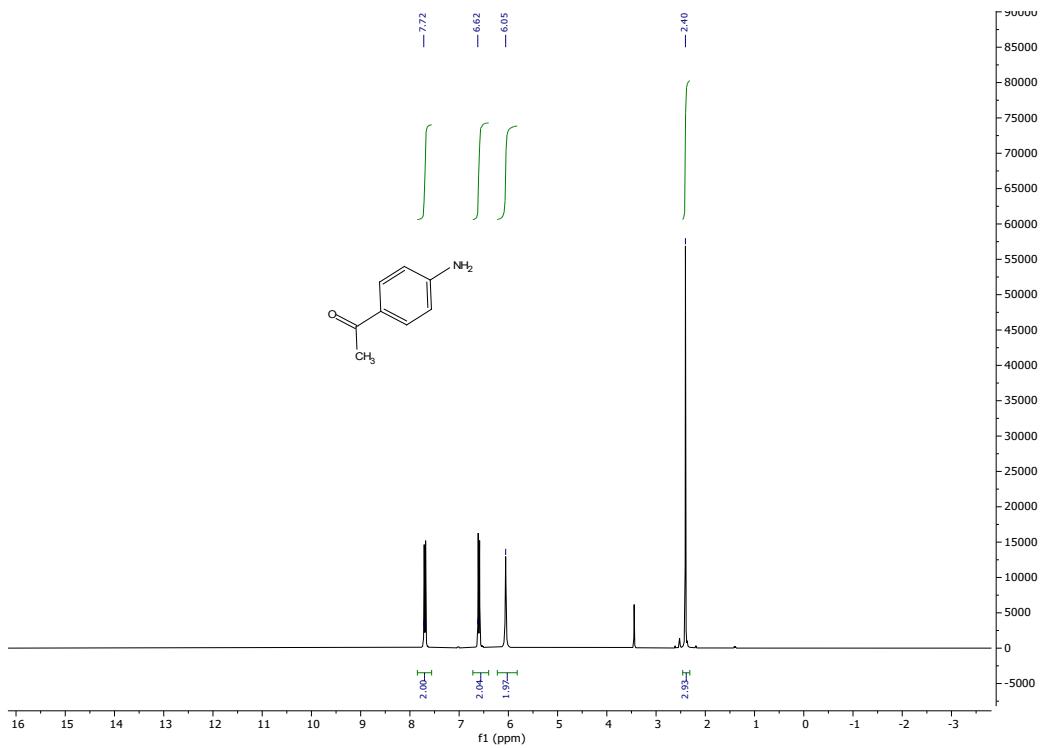
RR



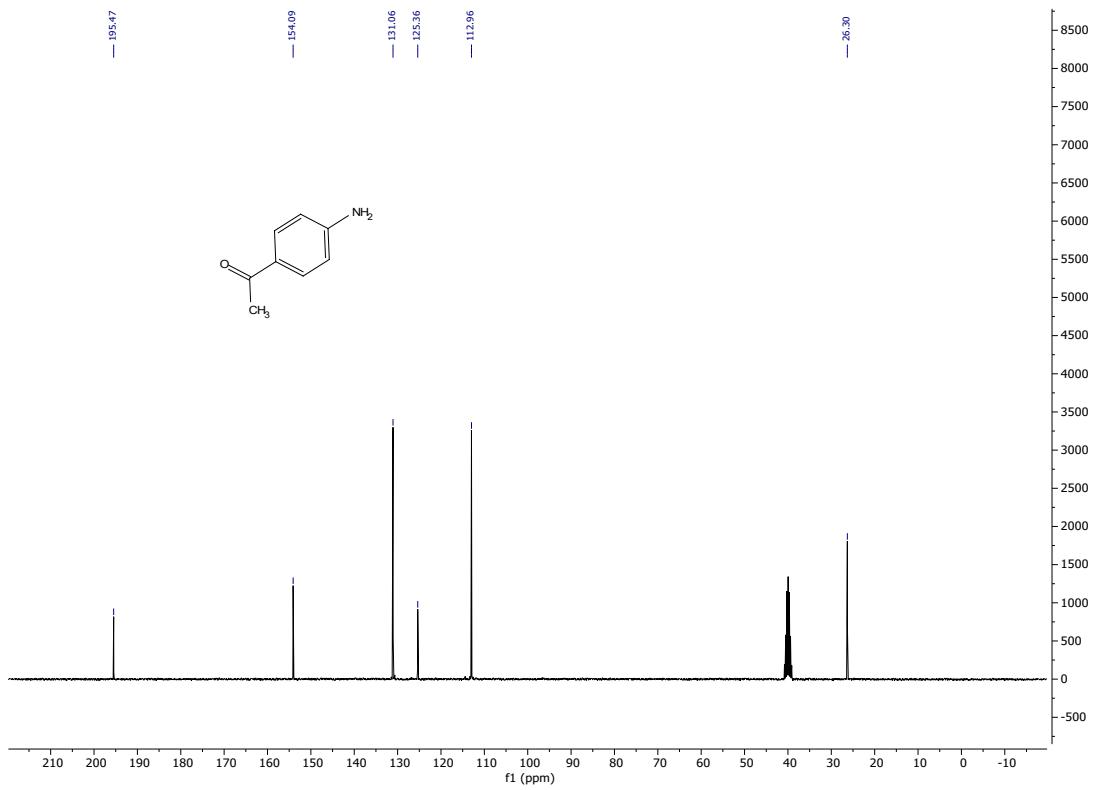
**Fig. S18.**  $^1\text{H}$ NMR (400 MHz, DMSO) spectrum of 2h.



**Fig. S19.**  $^{13}\text{C}$ NMR (400 MHz, DMSO) spectrum of 2h.

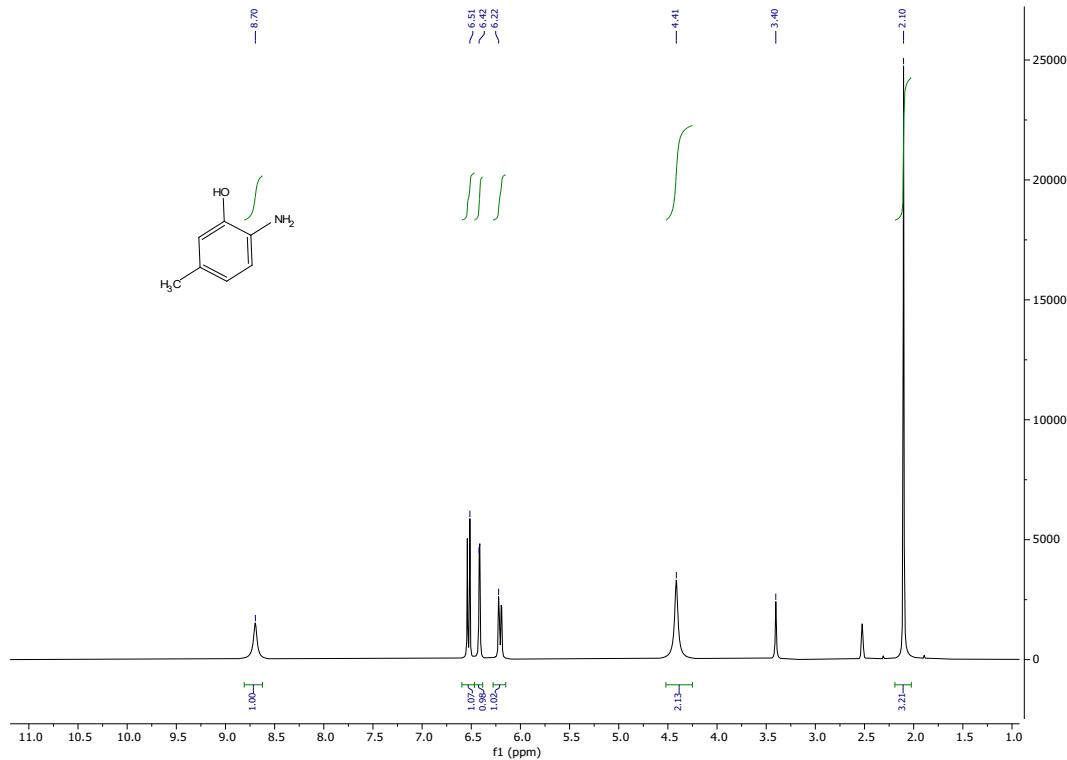


**Fig. S20.**  $^1\text{H}$ NMR (400 MHz, DMSO) spectrum of 2i.



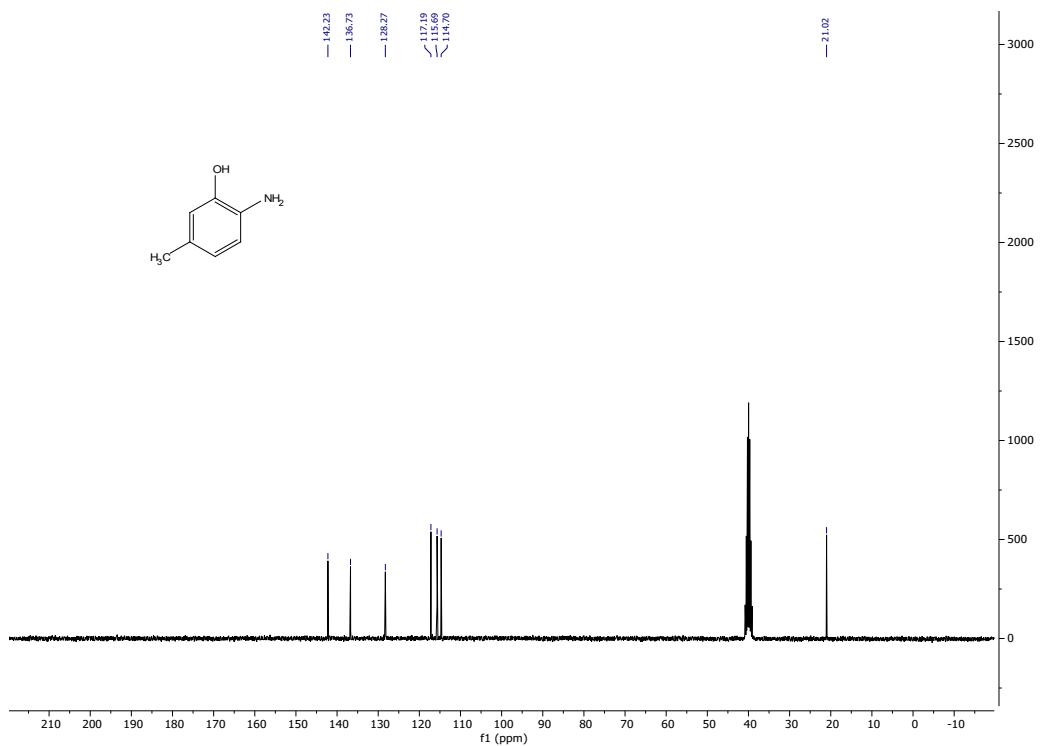
TT

**Fig. S21.**  $^{13}\text{C}$ NMR (400 MHz, DMSO) spectrum of 2i.

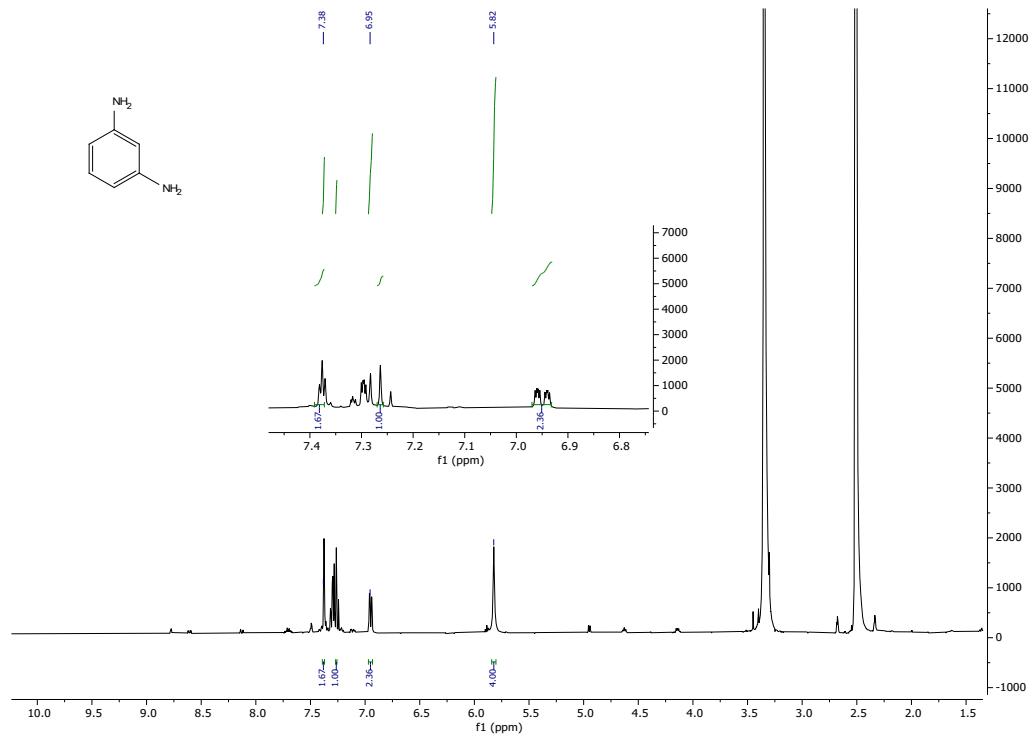


**Fig. S22.**  $^1\text{H}$ NMR (400 MHz, DMSO) spectrum of 2j.

UU

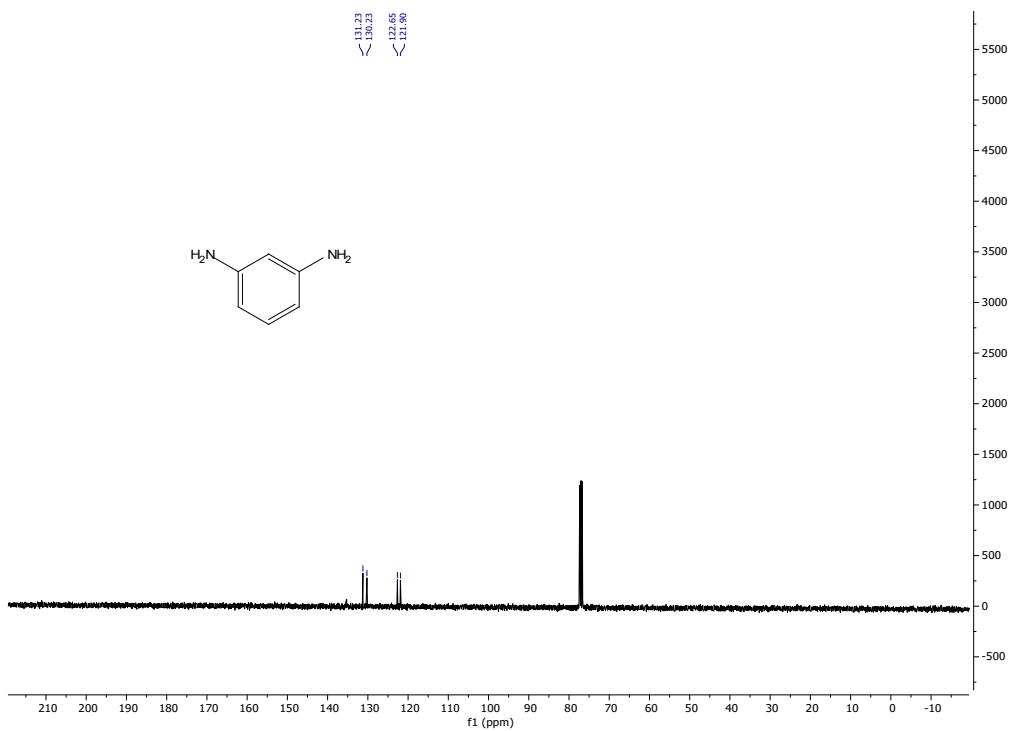


**Fig. S23.**  $^{13}\text{C}$ NMR (400 MHz, DMSO) spectrum of 2j.



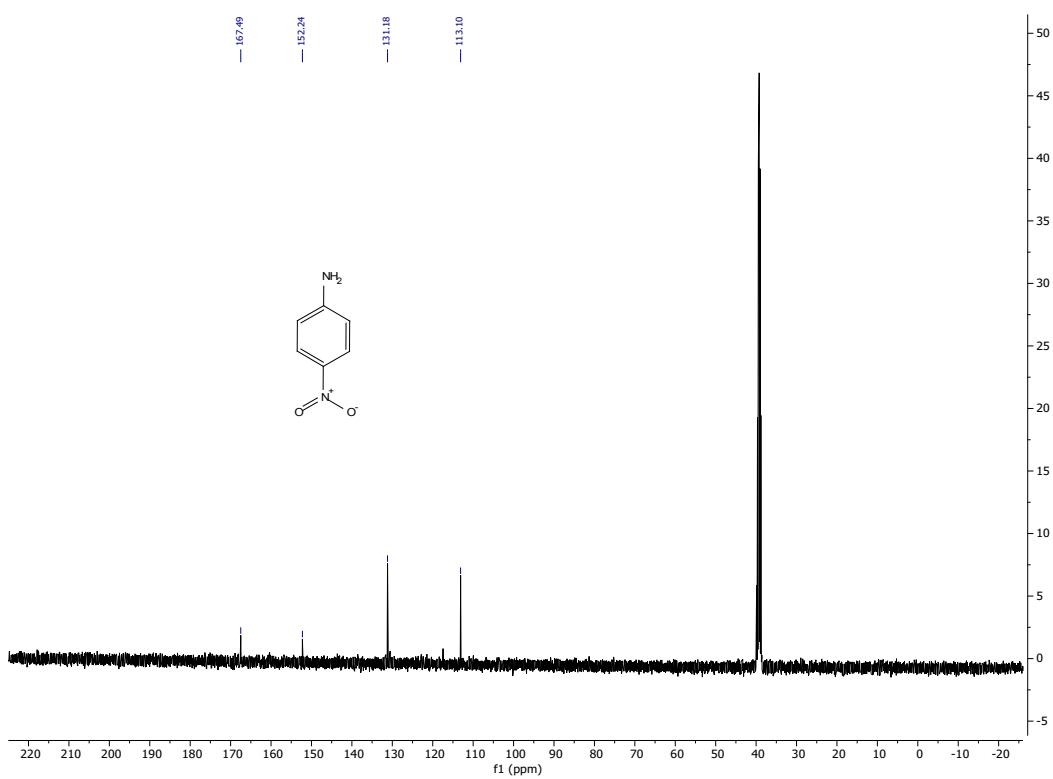
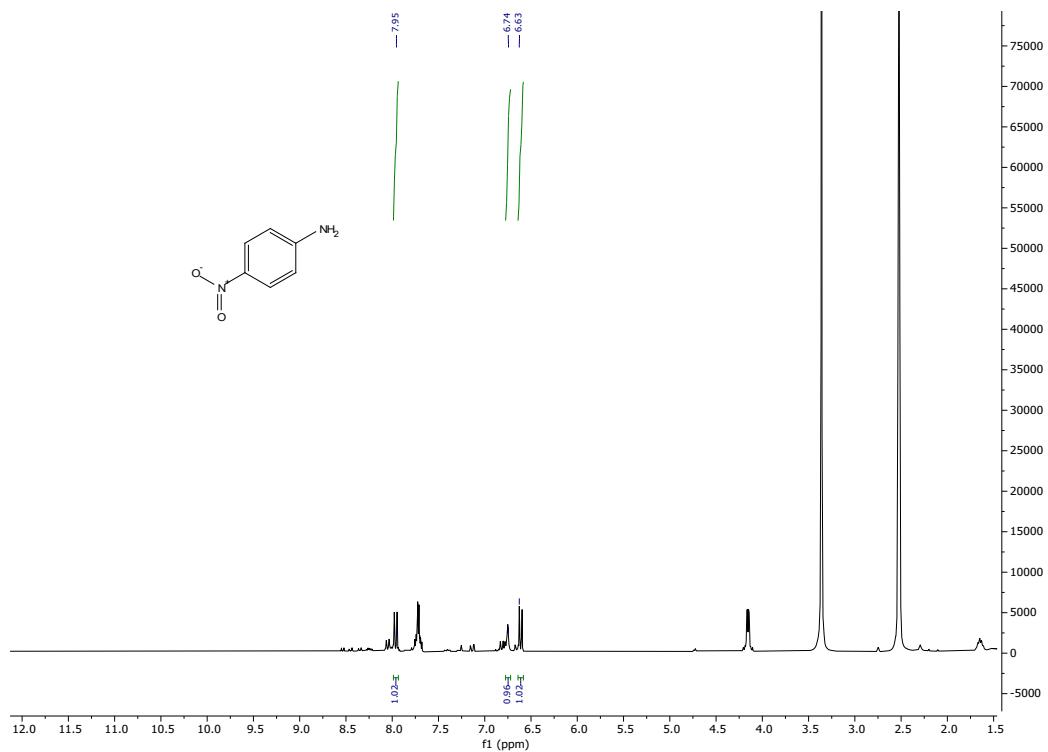
VV

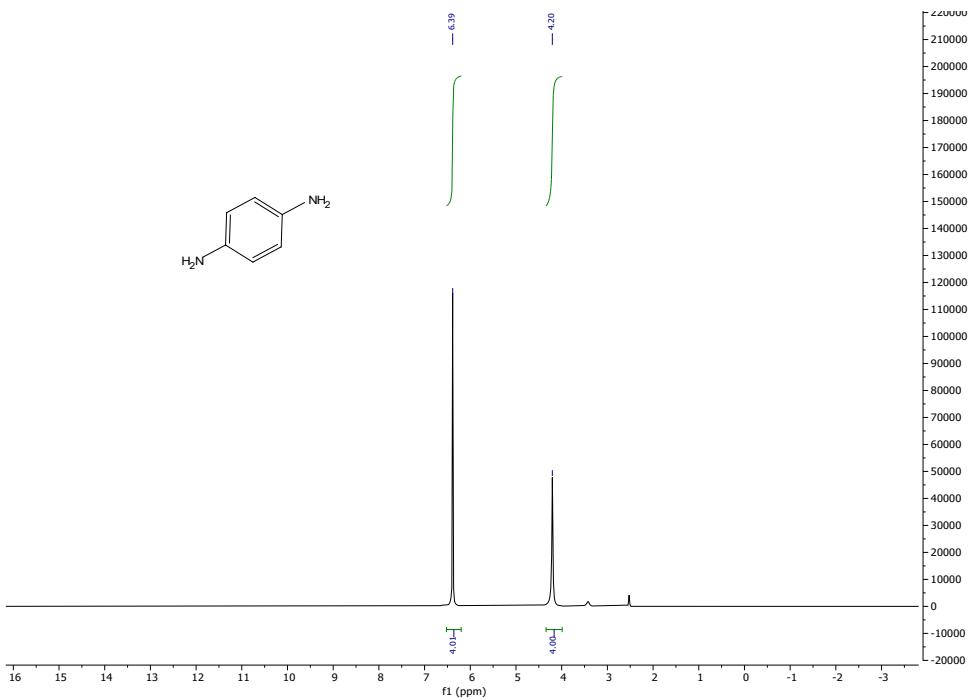
**Fig. S24.**  $^1\text{H}$ NMR (400 MHz, DMSO) spectrum of 2k.



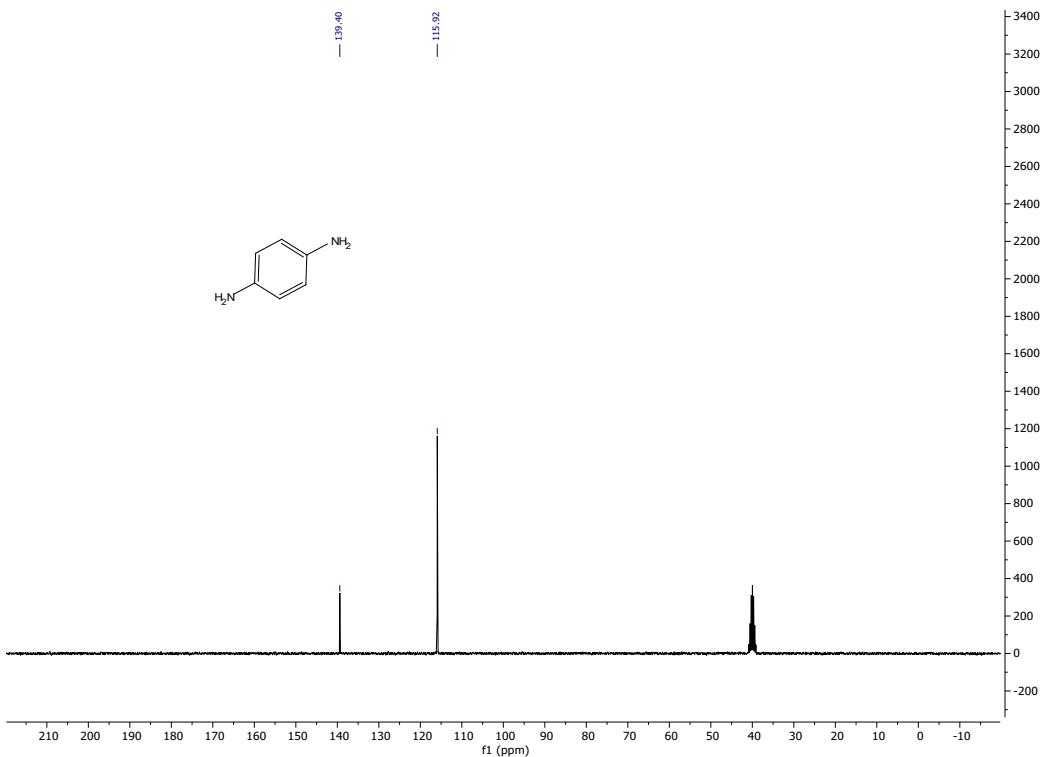
**Fig. S25.**  $^{13}\text{C}$ NMR (400 MHz, DMSO) spectrum of 2k.

WW

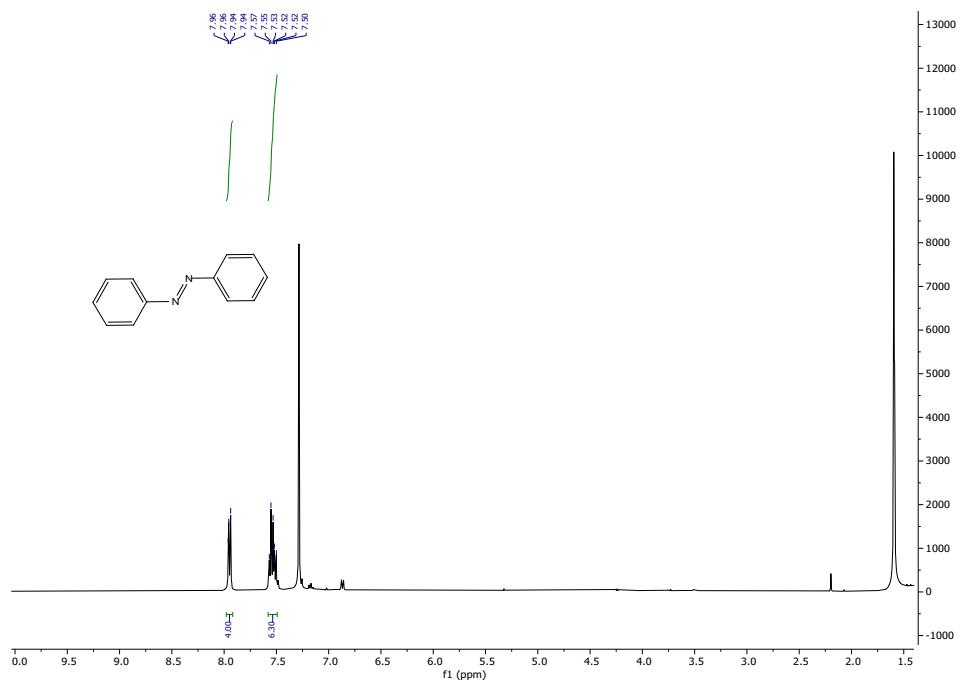




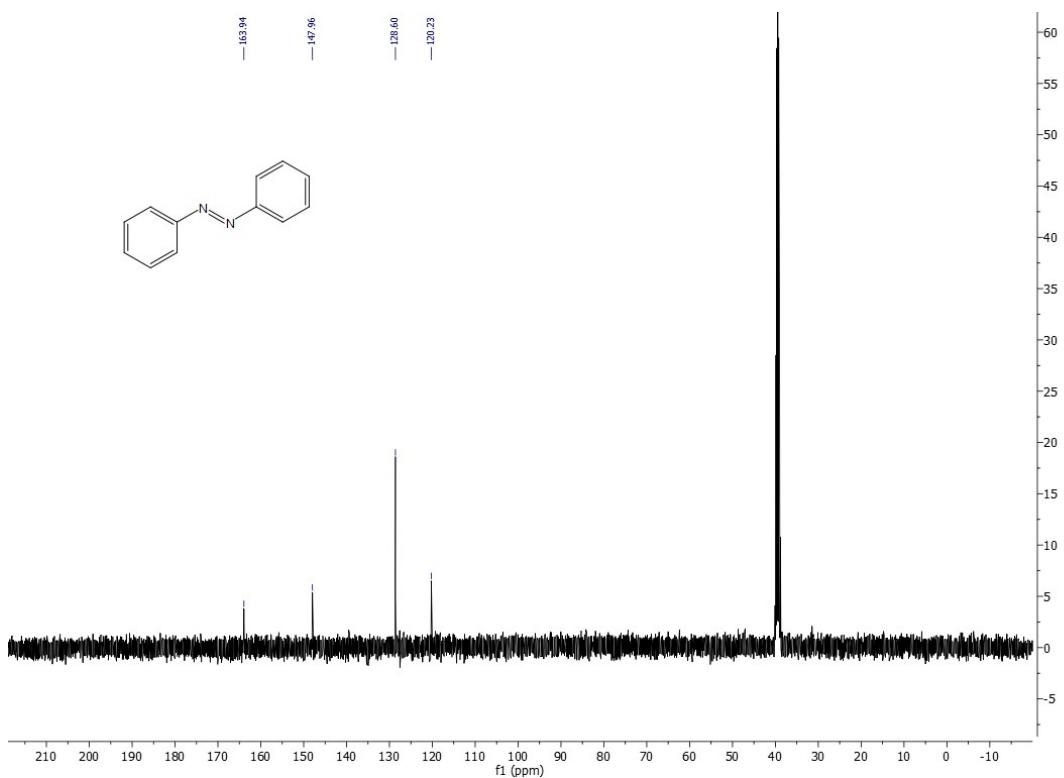
**Fig. S28.**  $^1\text{H}$ NMR (400 MHz, DMSO) spectrum of 2m.



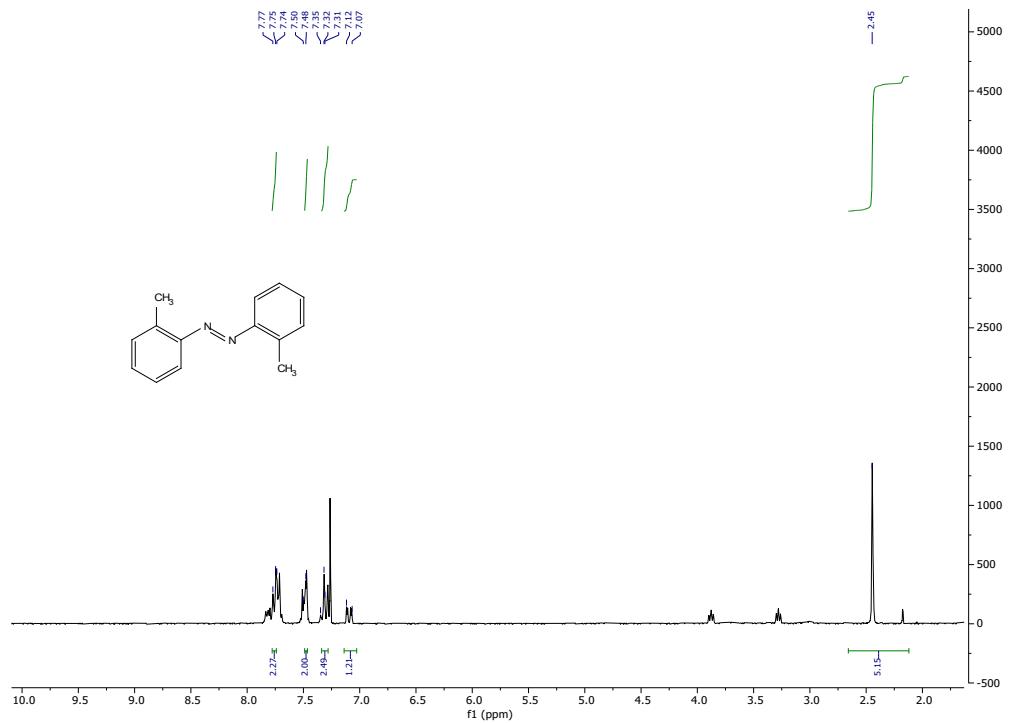
**Fig. S29.**  $^{13}\text{C}$ NMR (400 MHz, DMSO) spectrum of 2m.



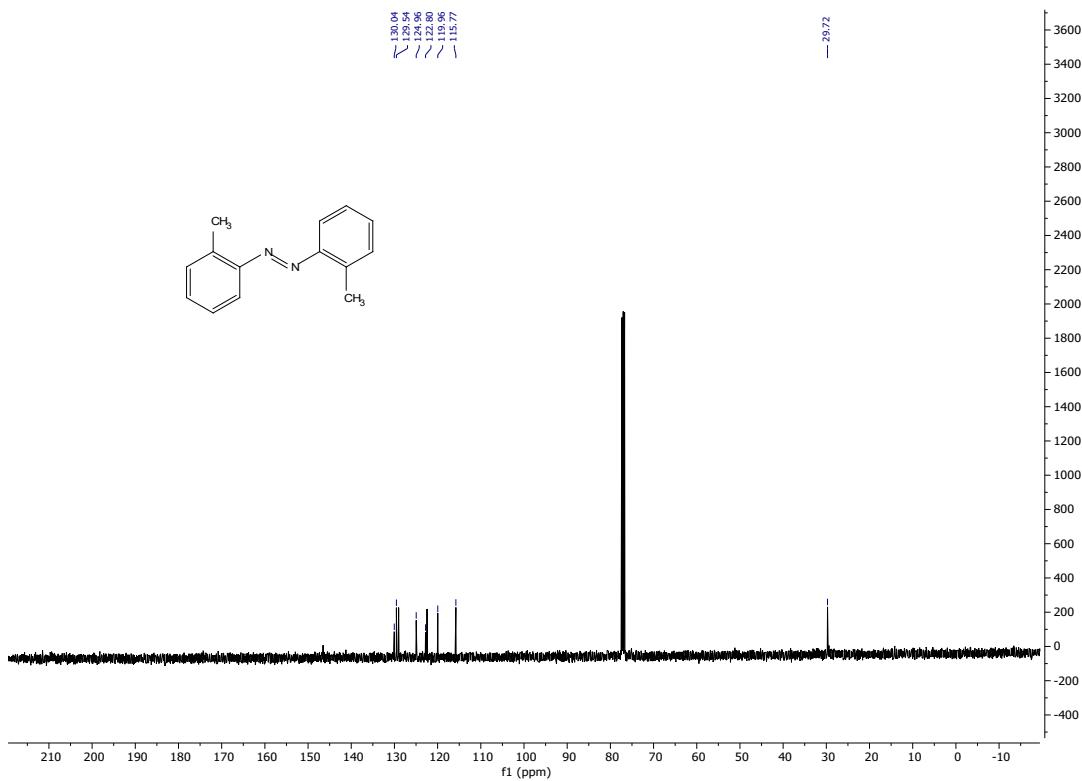
**Fig. S30.**  $^1\text{H}$ NMR (400 MHz,  $\text{CDCl}_3$ ) spectrum of 3a.



**Fig. S31.**  $^{13}\text{C}$ NMR (125 MHz,  $\text{CDCl}_3$ ) spectrum of 3a.

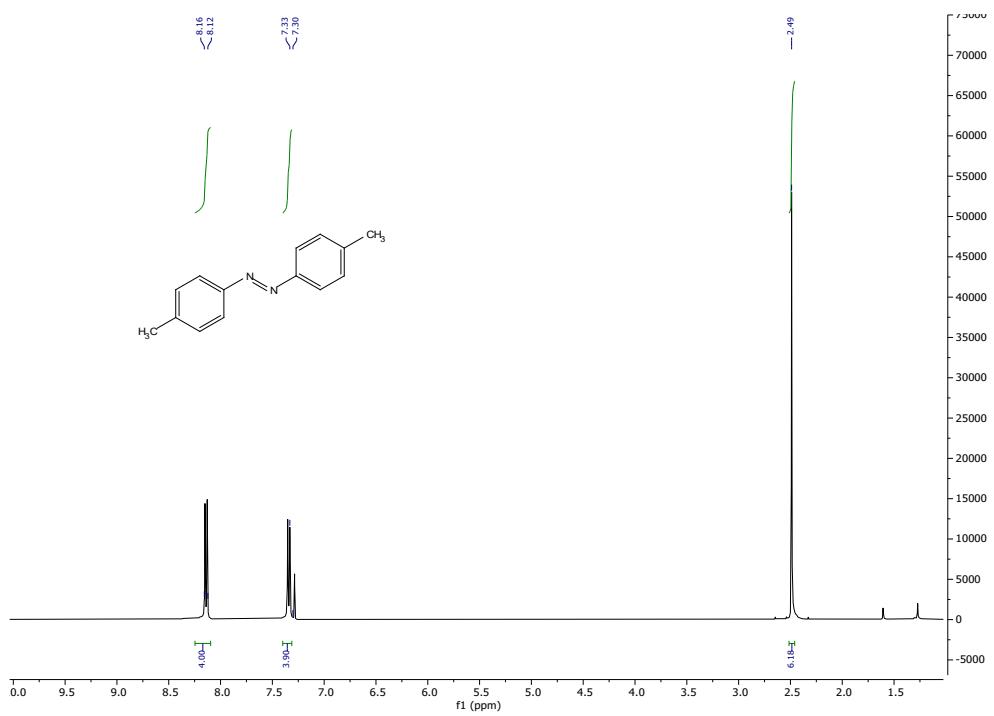


**Fig. S32.** <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) spectrum of 3b.

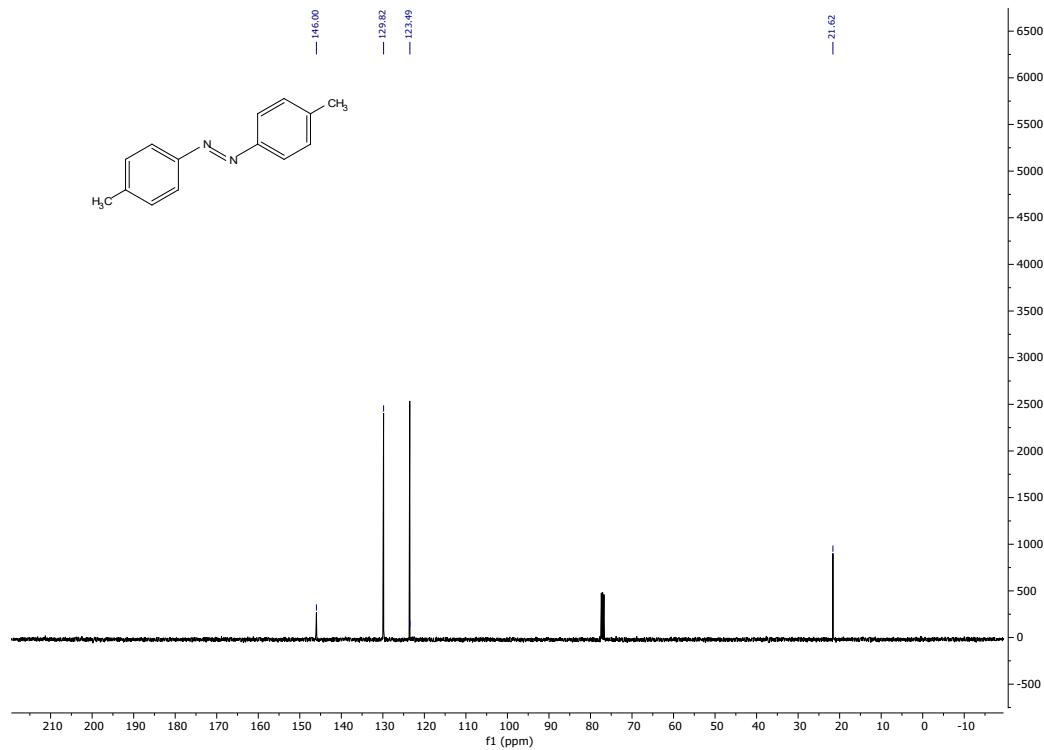


**Fig. S33.** <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) spectrum of 3b.

AAA

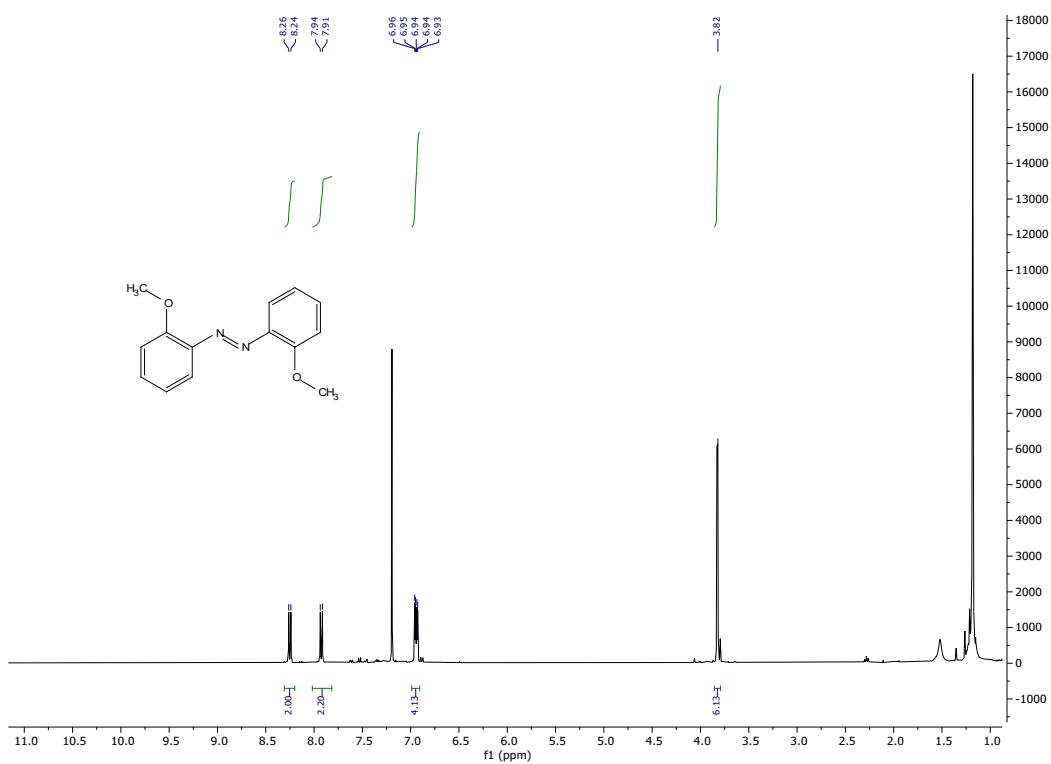


**Fig. S34.**  $^1\text{H}$ NMR (400 MHz,  $\text{CDCl}_3$ ) spectrum of 3c.

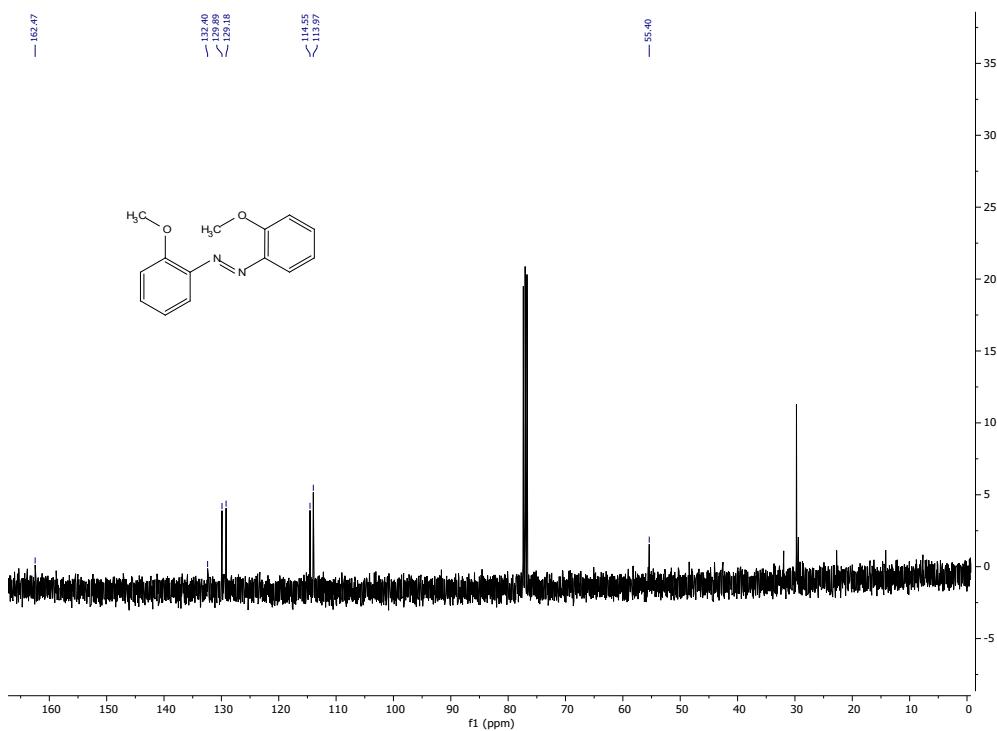


BBB

**Fig. S35.**  $^{13}\text{C}$ NMR (125 MHz,  $\text{CDCl}_3$ ) spectrum of 3c.

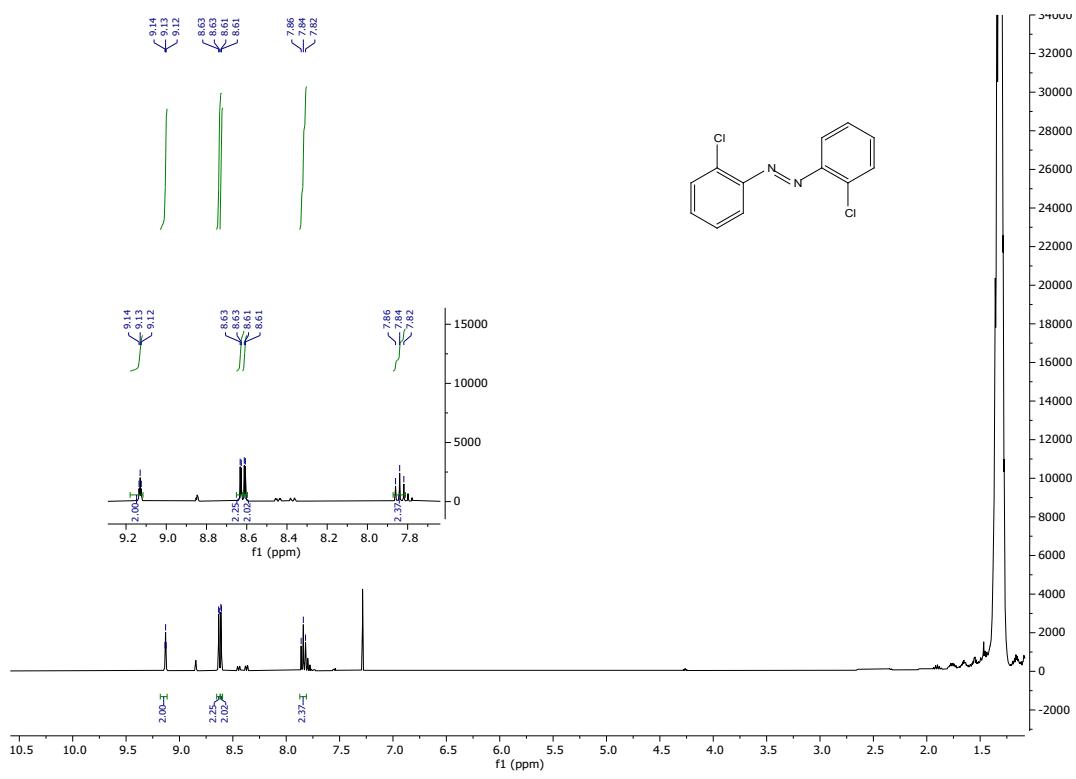


**Fig. S36.**  $^1\text{H}$ NMR (400 MHz,  $\text{CDCl}_3$ ) spectrum of 3d.

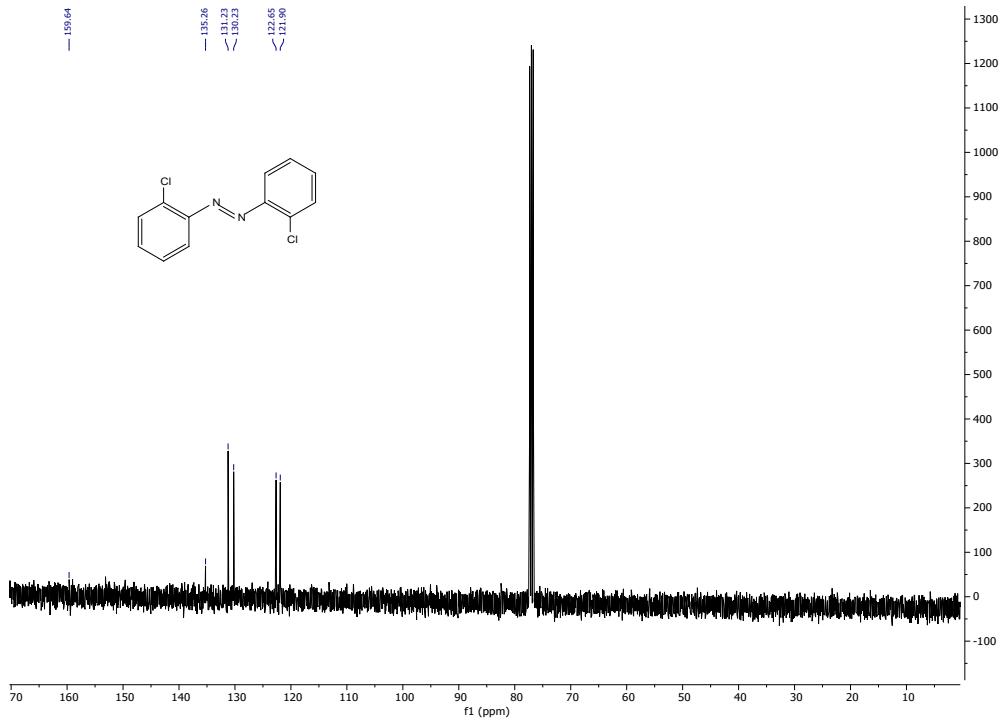


**Fig. S37.**  $^{13}\text{C}$ NMR (400 MHz,  $\text{CDCl}_3$ ) spectrum of 3d.

CCC

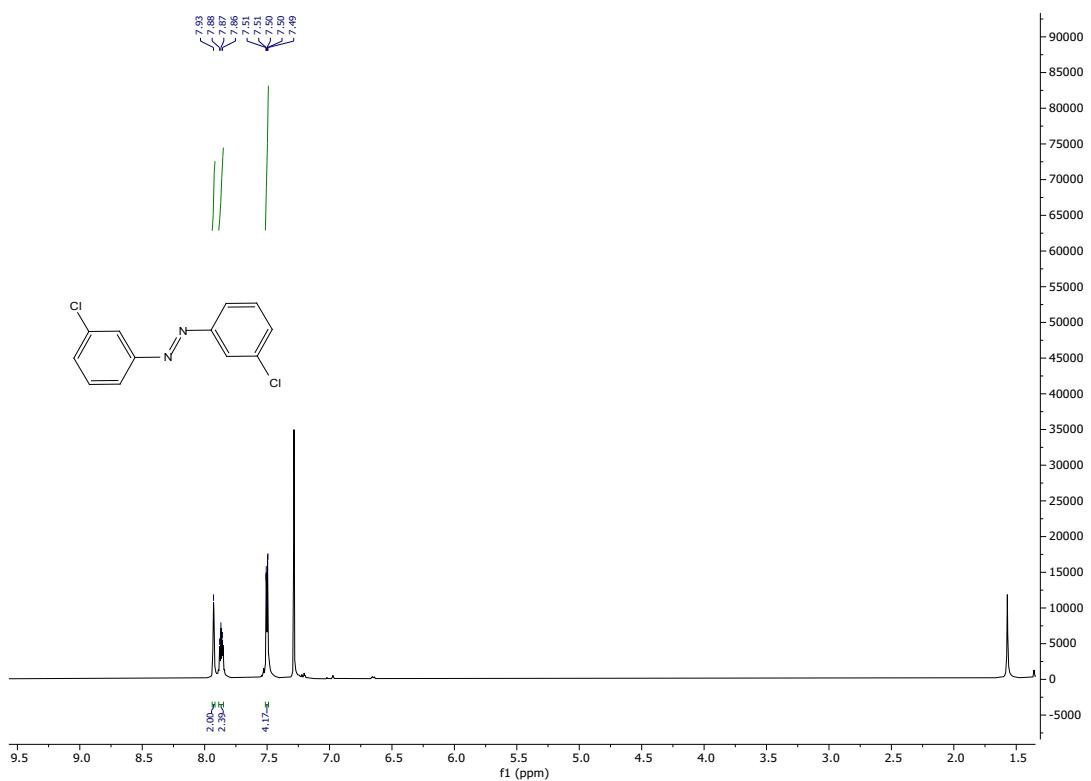


**Fig. S38.**  $^1\text{H}$ NMR (400 MHz,  $\text{CDCl}_3$ ) spectrum of 3e.

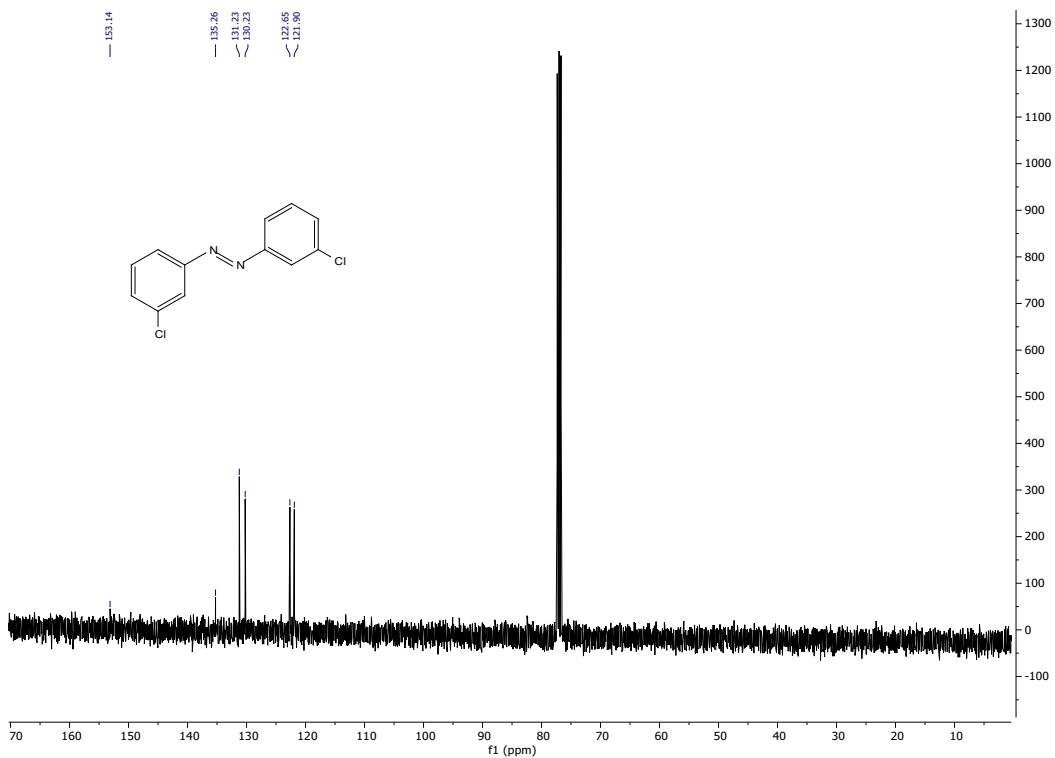


**Fig. S39.**  $^{13}\text{C}$ NMR (400 MHz,  $\text{CDCl}_3$ ) spectrum of 3e.

DDD

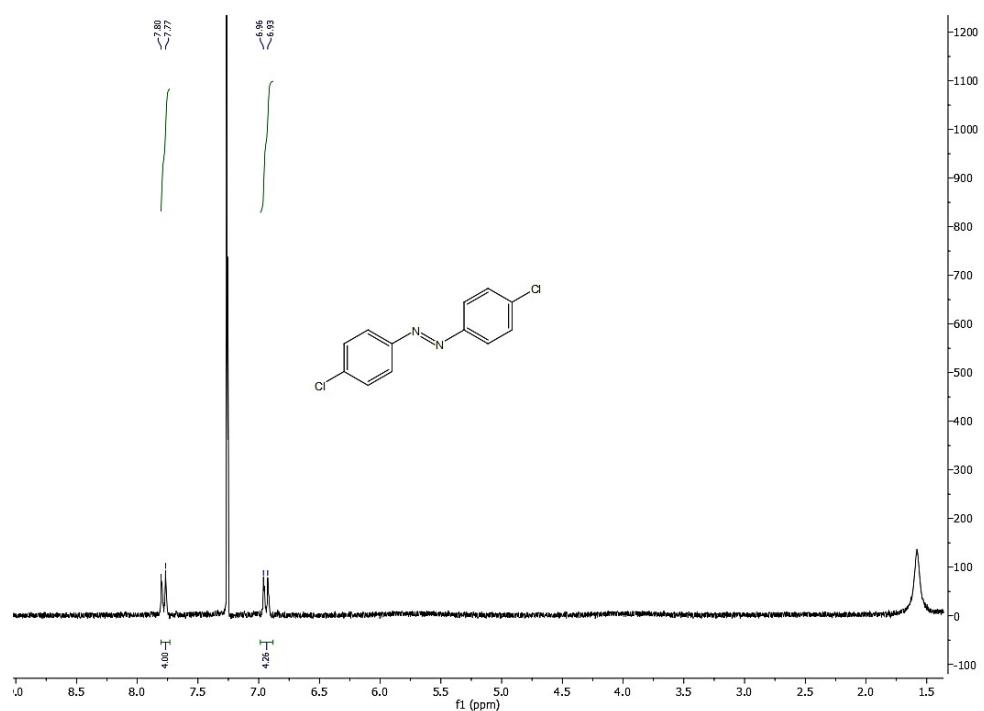


**Fig. S40.** <sup>1</sup>HNMR (400 MHz, CDCl<sub>3</sub>) spectrum of 3f.

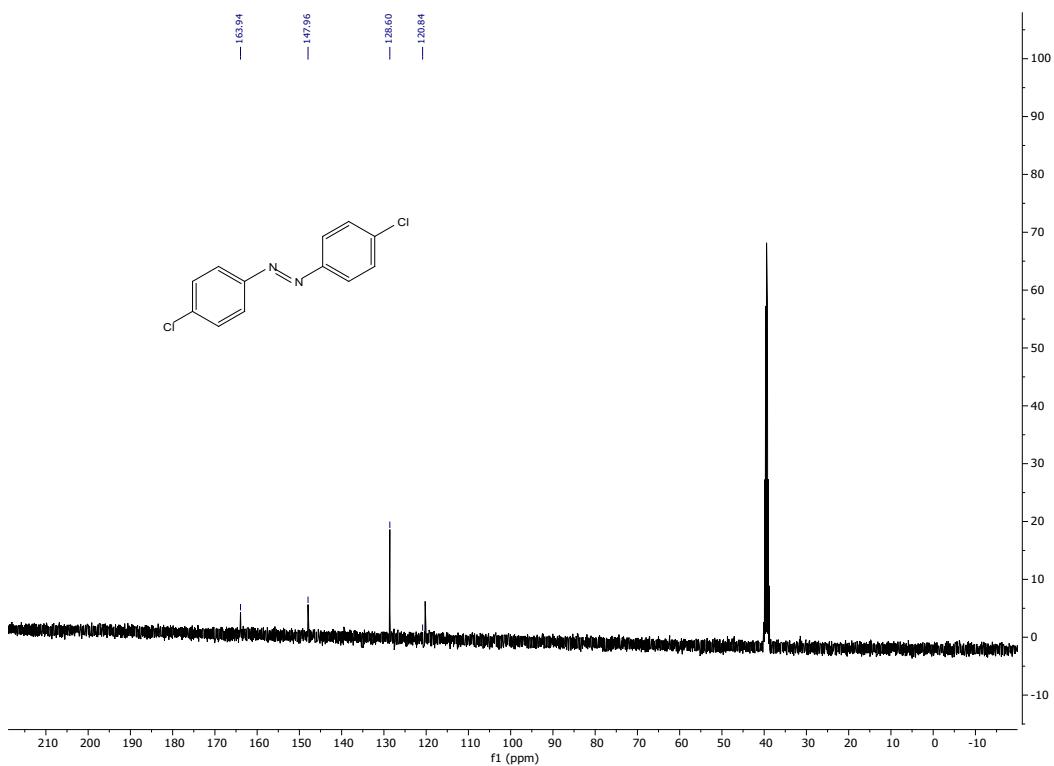


EEE

**Fig. S41.**  $^{13}\text{C}$ NMR (400 MHz,  $\text{CDCl}_3$ ) spectrum of 3f.

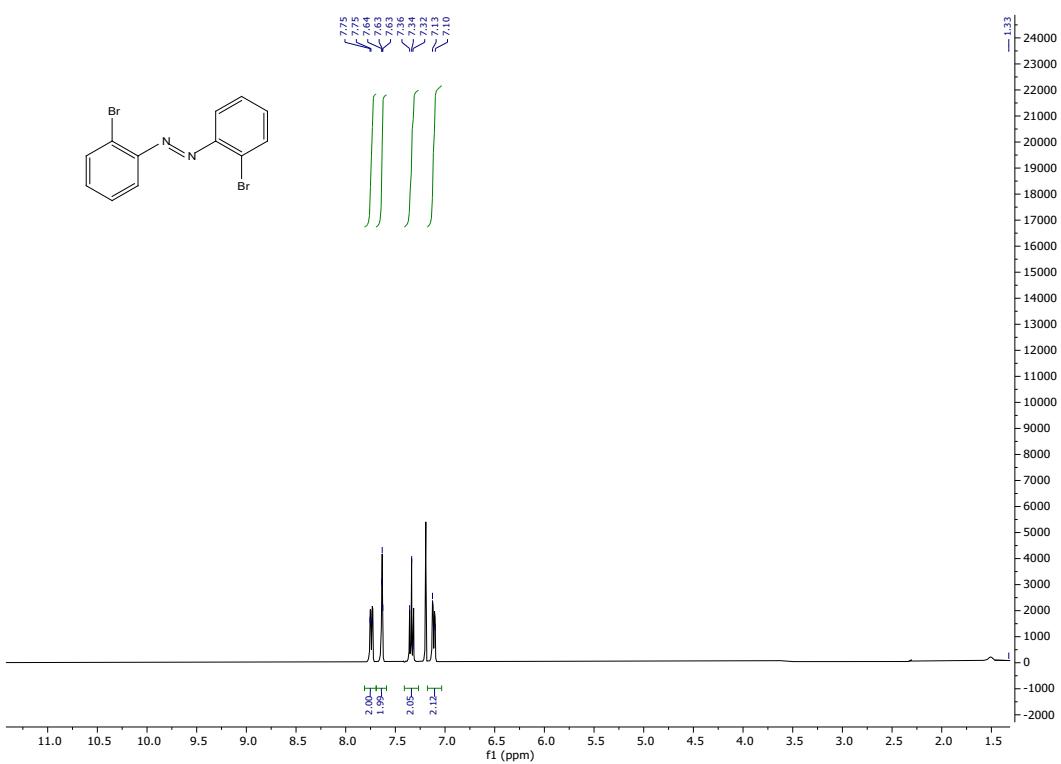


**Fig. S42.**  $^1\text{H}$ NMR (400 MHz,  $\text{CDCl}_3$ ) spectrum of 3g.

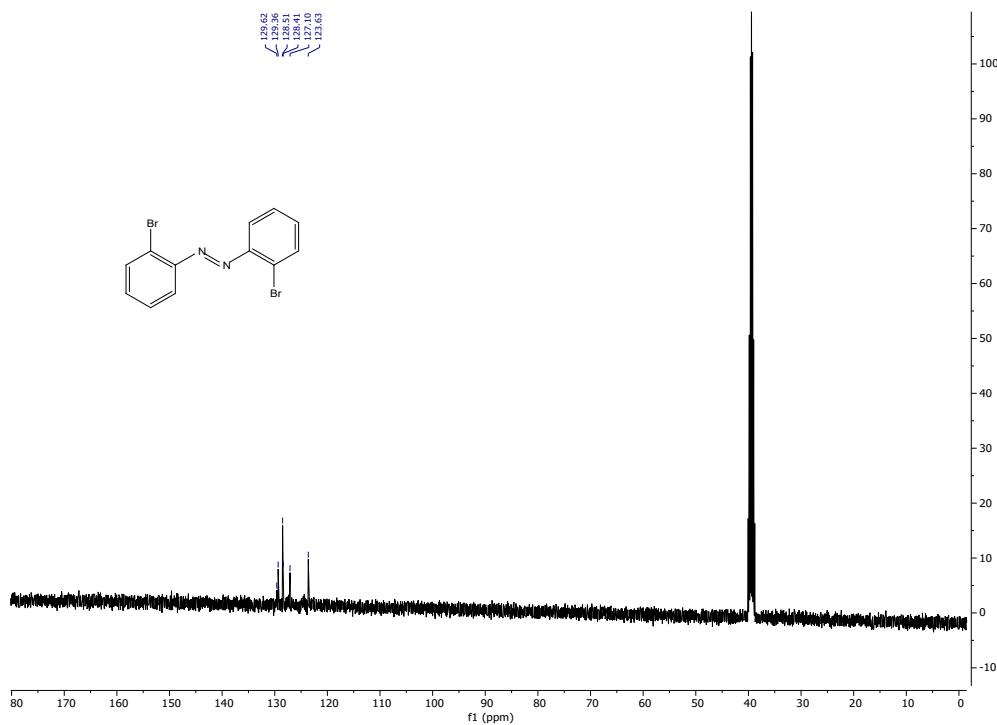


FFF

**Fig. S43.**  $^1\text{H}$ NMR (400 MHz,  $\text{CDCl}_3$ ) spectrum of 3g.

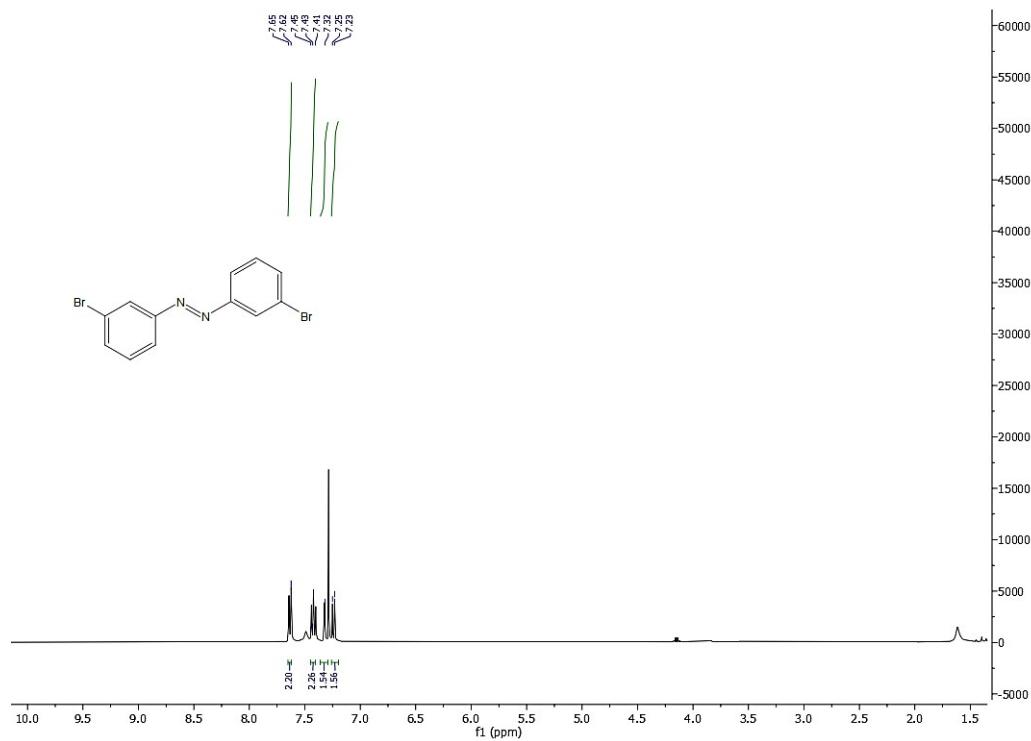


**Fig. S44.**  $^1\text{H}$ NMR (400 MHz,  $\text{CDCl}_3$ ) spectrum of 3h.

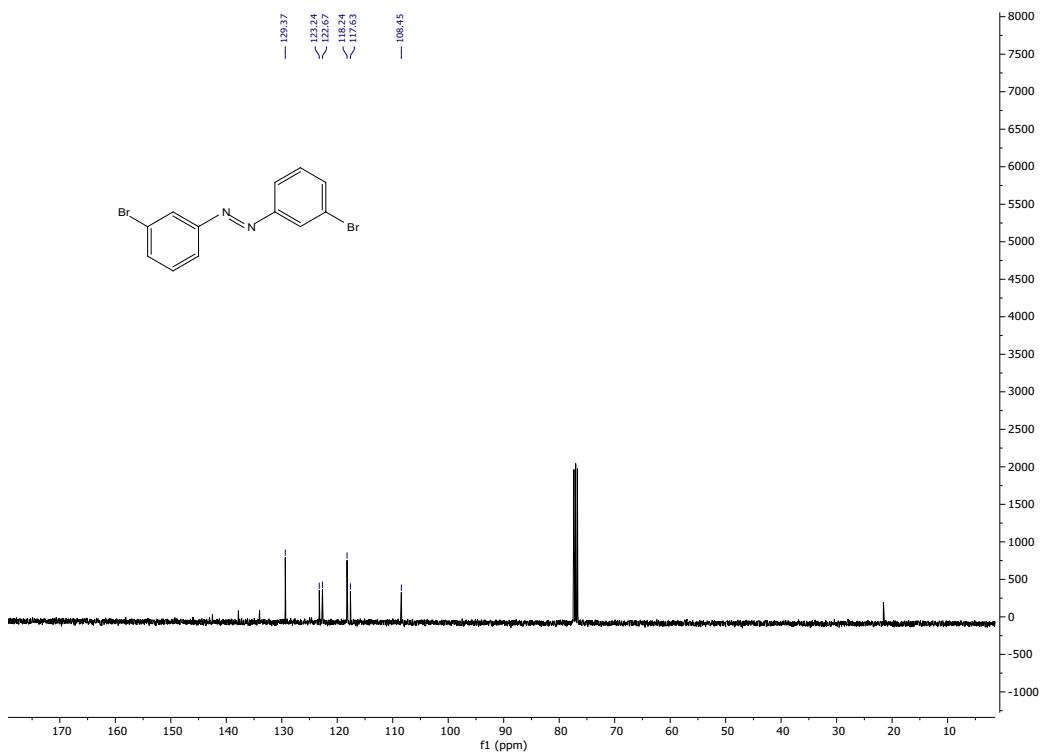


GGG

**Fig. S45.**  $^{13}\text{C}$ NMR (400 MHz,  $\text{CDCl}_3$ ) spectrum of 3h.

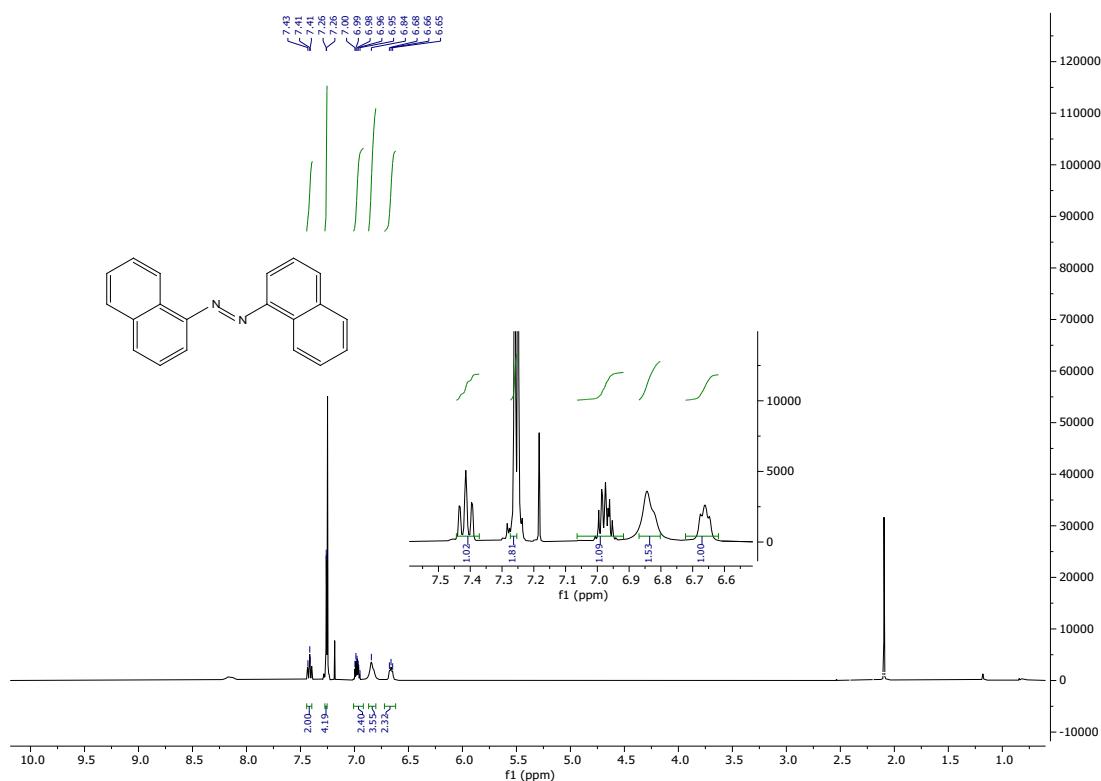


**Fig. S46.**  $^1\text{H}$ NMR (400 MHz,  $\text{CDCl}_3$ ) spectrum of 3i.

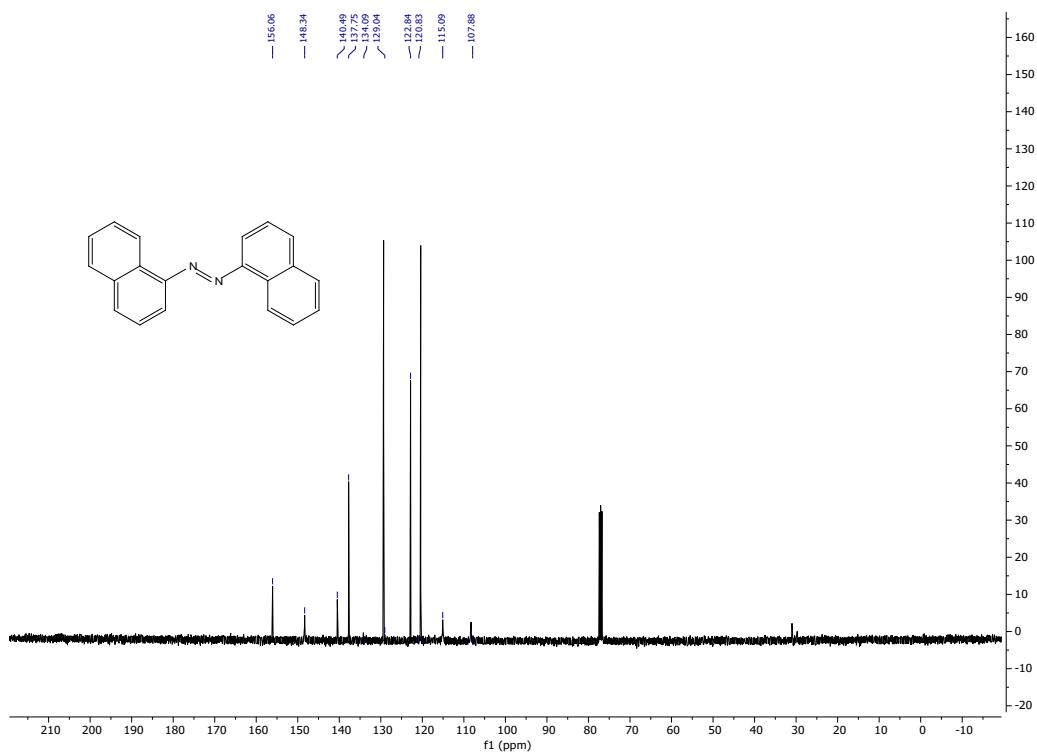


HHH

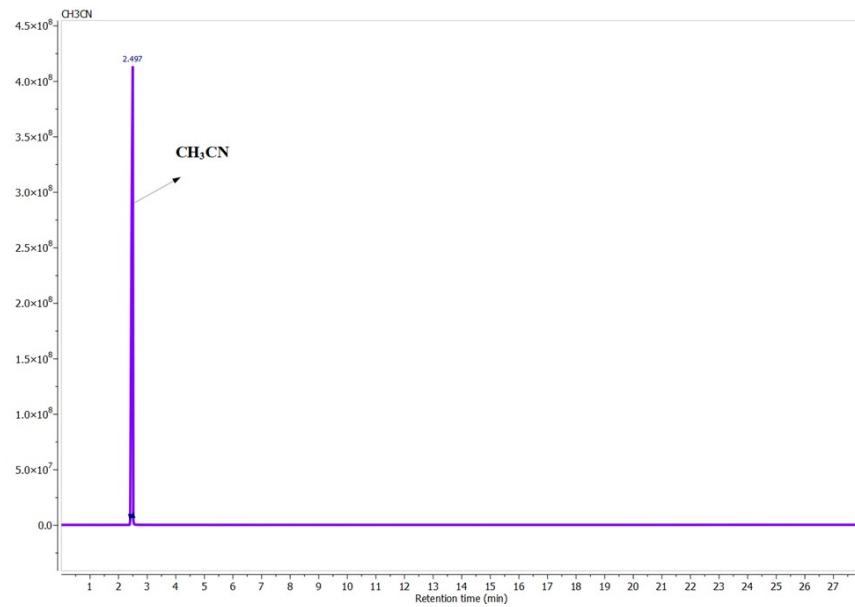
Fig. S47.  $^{13}\text{C}$ NMR (400 MHz,  $\text{CDCl}_3$ ) spectrum of 3i.



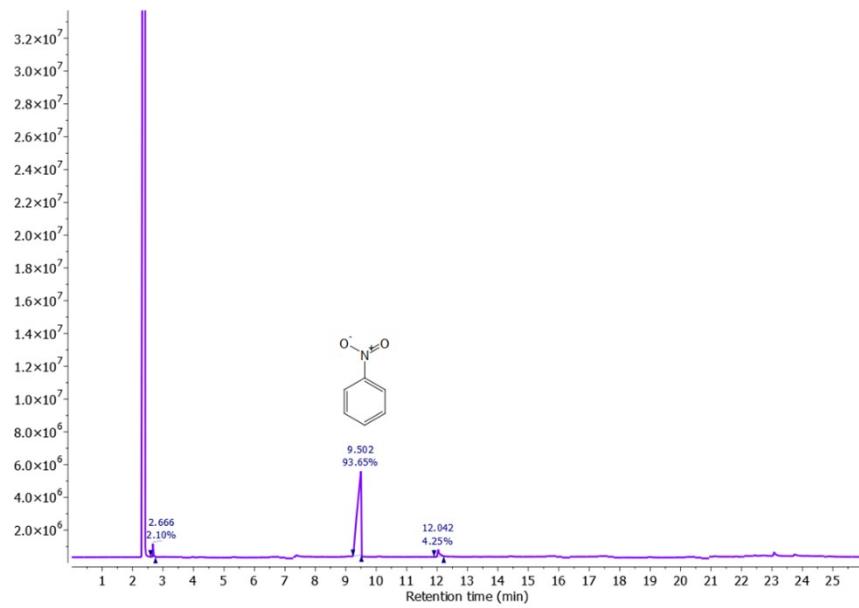
**Fig. S48.**  $^1\text{H}$ NMR (400 MHz,  $\text{CDCl}_3$ ) spectrum of 3j.



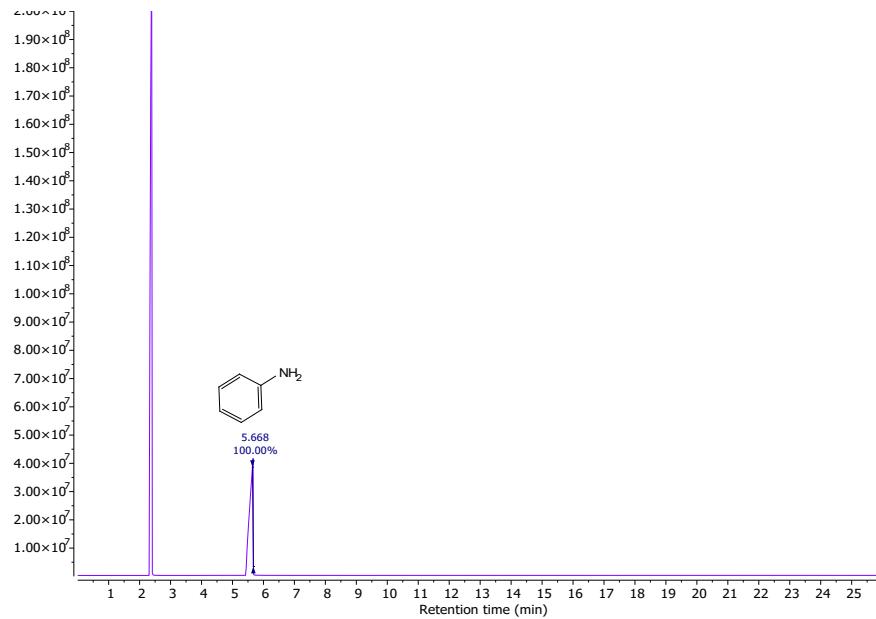
**Fig. S49.**  $^{13}\text{C}$ NMR (400 MHz,  $\text{CDCl}_3$ ) spectrum of 3j.



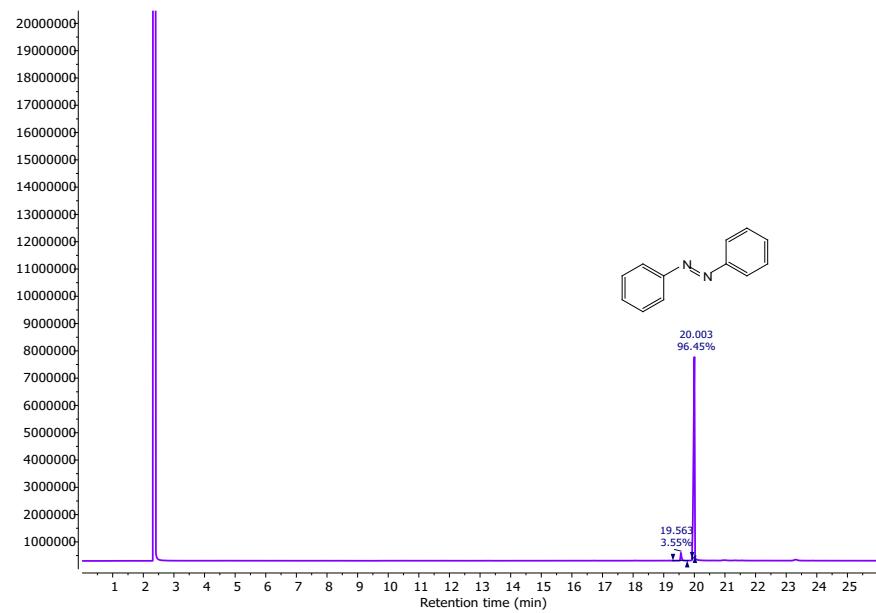
**Fig. S50.** Gas Chromatography (GC): CH<sub>3</sub>CN



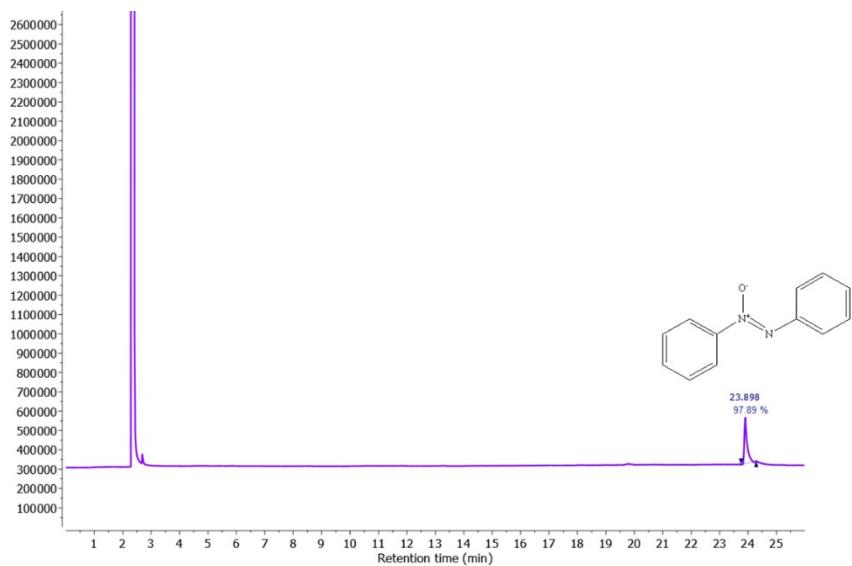
**Fig. S51.** Gas Chromatography (GC): CH<sub>3</sub>CN, Nitrobenzene



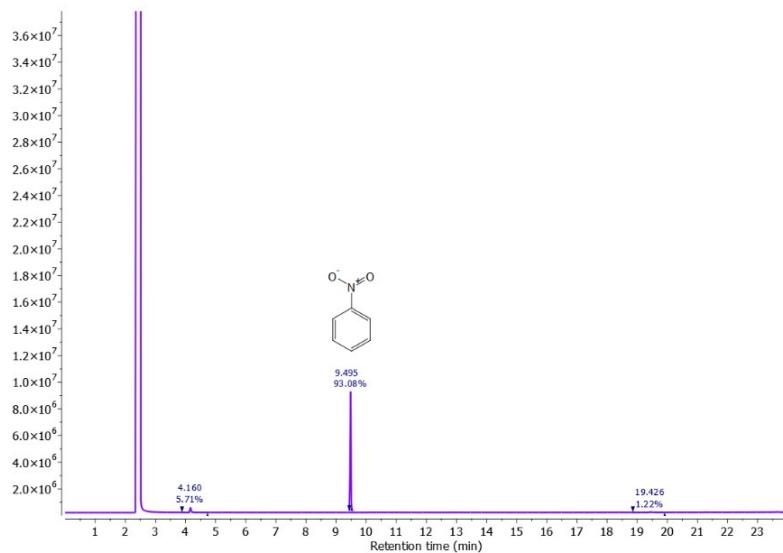
**Fig. S52.** Gas Chromatography (GC): CH<sub>3</sub>CN, Aniline



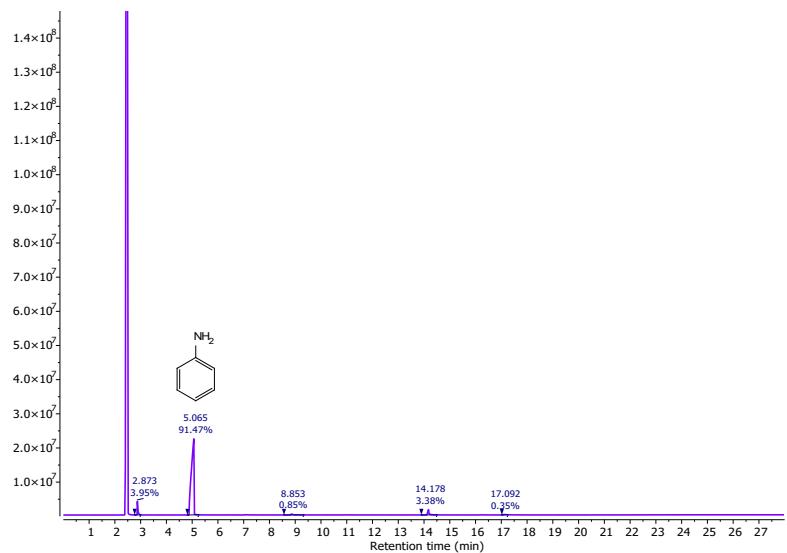
**Fig. S53.** Gas Chromatography (GC): CH<sub>3</sub>CN, Azobenzene



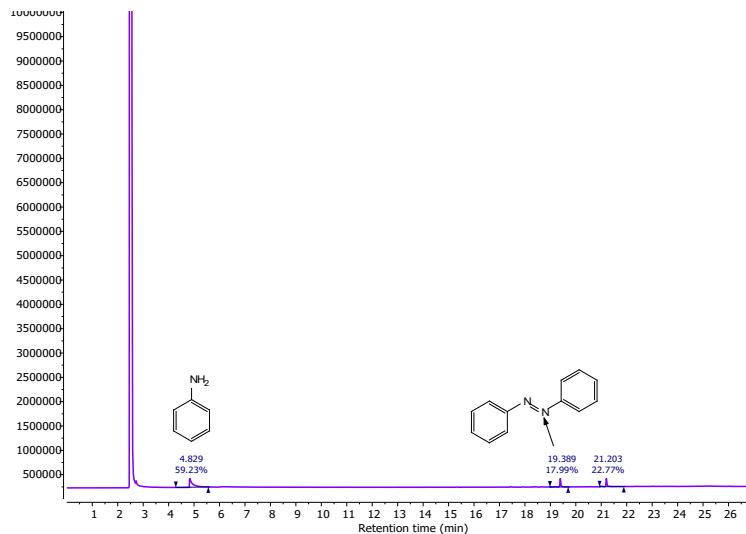
**Fig. S54.** Gas Chromatography (GC): CH<sub>3</sub>CN, Azoxybenzene



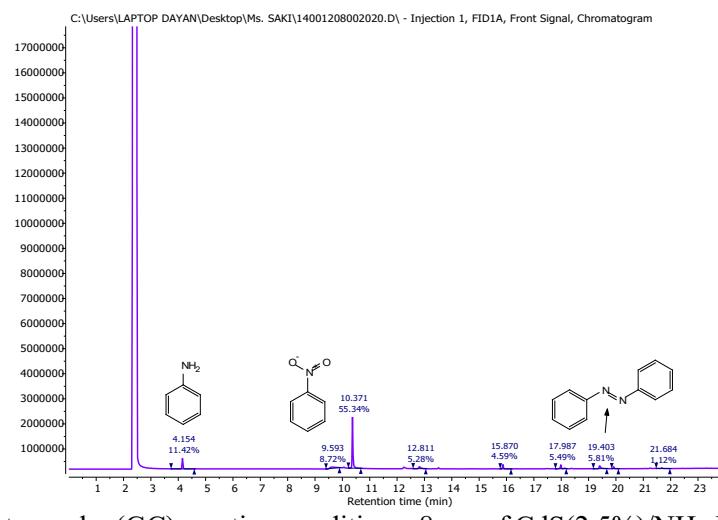
**Fig. S55.** Gas Chromatography (GC) Reaction Conditions: without catalyst, 0.2 mmol nitrobenzene, 2 mL methanol, 6 eq hydrazine hydrate, 12 W white lamp, argon atmosphere, time: 24 hours, room temperature



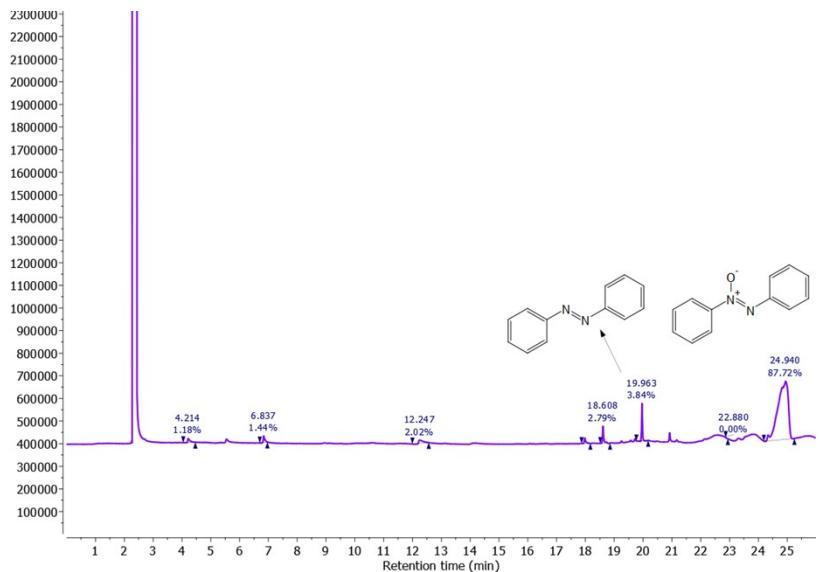
**Fig. S56.** Gas Chromatography (GC) reaction conditions: 8 mg of CdS(2.5%)/NH<sub>2</sub>-MIL-125 photocatalyst, 0.2 mmol of nitrobenzene, 2 mL of methanol, 6 eq of hydrazine hydrate, 12 W white lamp, argon atmosphere, time: 24 hours, room temperature.



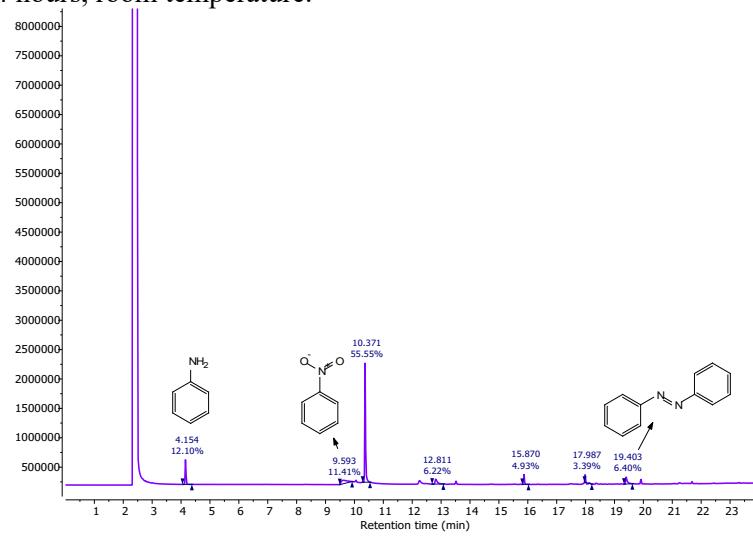
**Fig. S57.** Gas Chromatography (GC) reaction conditions: 8 mg of CdS(2.5%)/NH<sub>2</sub>-MIL-125 photocatalyst, 0.2 mmol of nitrobenzene, 2 mL of ethanol, 6 eq of hydrazine hydrate, 12 W white lamp, argon atmosphere, time: 24 hours, room temperature.



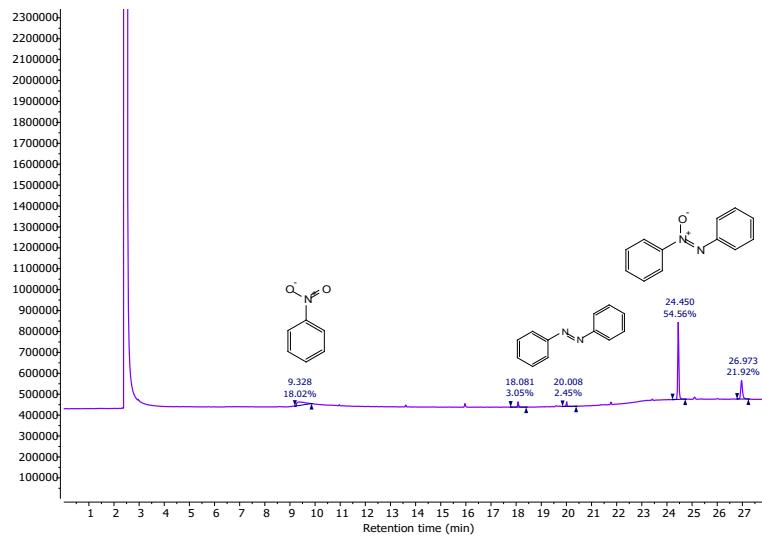
**Fig. S58.** Gas Chromatography (GC) reaction conditions: 8 mg of CdS(2.5%)/NH<sub>2</sub>-MIL-125 photocatalyst, 0.2 mmol of nitrobenzene, 2 mL of water solvent, 6 eq of hydrazine hydrate, 12 W white lamp, argon atmosphere, time: 24 hours, room temperature.



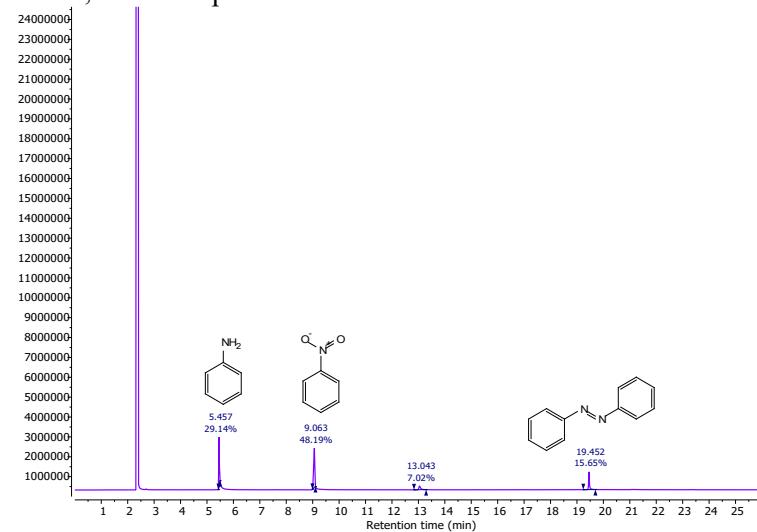
**Fig. S59.** Gas Chromatography (GC) reaction conditions: 8 mg of CdS(2.5%)/NH<sub>2</sub>-MIL-125 photocatalyst, 0.2 mmol of nitrobenzene, 2 mL of tetrahydrofuran, 6 eq of hydrazine hydrate, 12 W white lamp, argon atmosphere, time: 24 hours, room temperature.



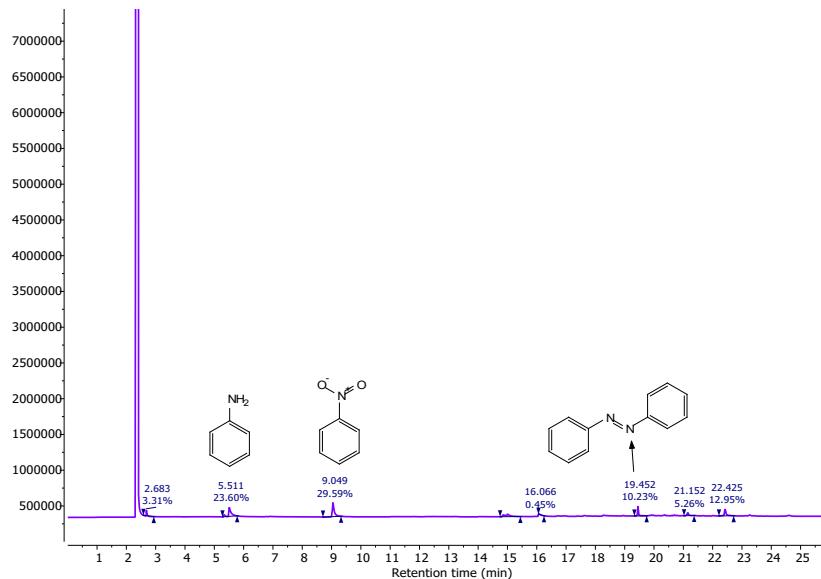
**Fig. S60.** Gas Chromatography (GC) reaction conditions: 8 mg of CdS(2.5%)/NH<sub>2</sub>-MIL-125 photocatalyst, 0.2 mmol of nitrobenzene, 2 mL of methanol, 6 eq of hydrazine hydrate, 12 W white lamp, air atmosphere, time: 24 hours, room temperature.



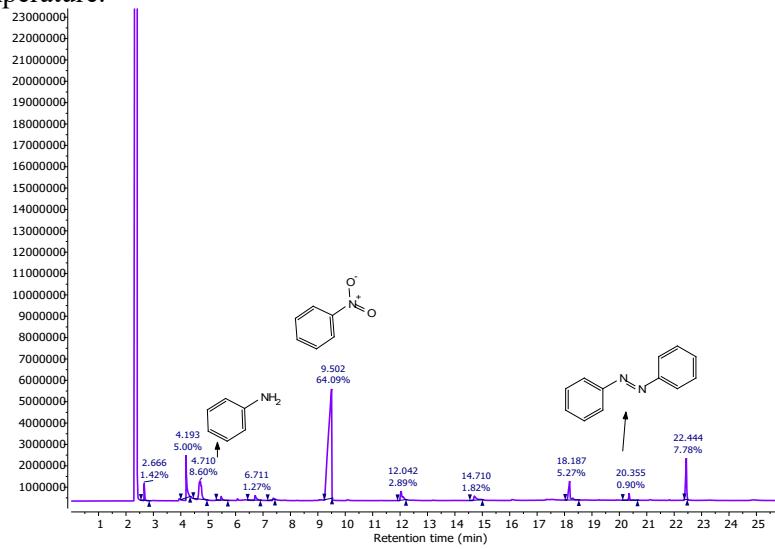
**Fig. S61.** Gas Chromatography (GC) reaction conditions: 8 mg of CdS(2.5%)/NH<sub>2</sub>-MIL-125 photocatalyst, 0.2 mmol of nitrobenzene, 2 mL of methanol, 6 eq of hydrazine hydrate, 12 W white lamp, oxygen atmosphere, time: 24 hours, room temperature.



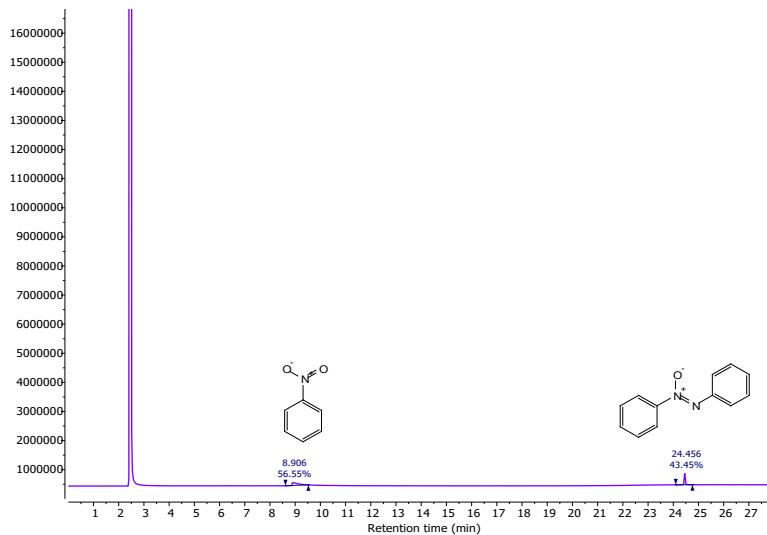
**Fig. S62.** Gas Chromatography (GC) Reaction conditions: 8 mg CdS photocatalyst, 0.2 mmol nitrobenzene, 2 mL methanol, 6 eq hydrazine hydrate, 12 W white lamp, argon atmosphere, time: 24 hours, room temperature .



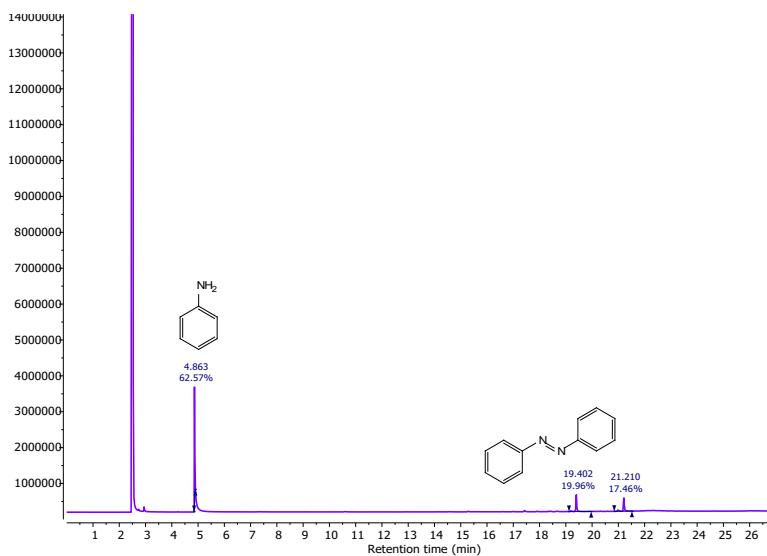
**Fig. S63.** Gas chromatography (GC) reaction conditions: 8 mg of  $\text{NH}_2\text{-MIL-125}$  photocatalyst, 0.2 mmol of nitrobenzene, 2 ml of methanol, 6 eq of hydrazine hydrate, 12 W white lamp, argon atmosphere, time: 24 hours, room temperature.



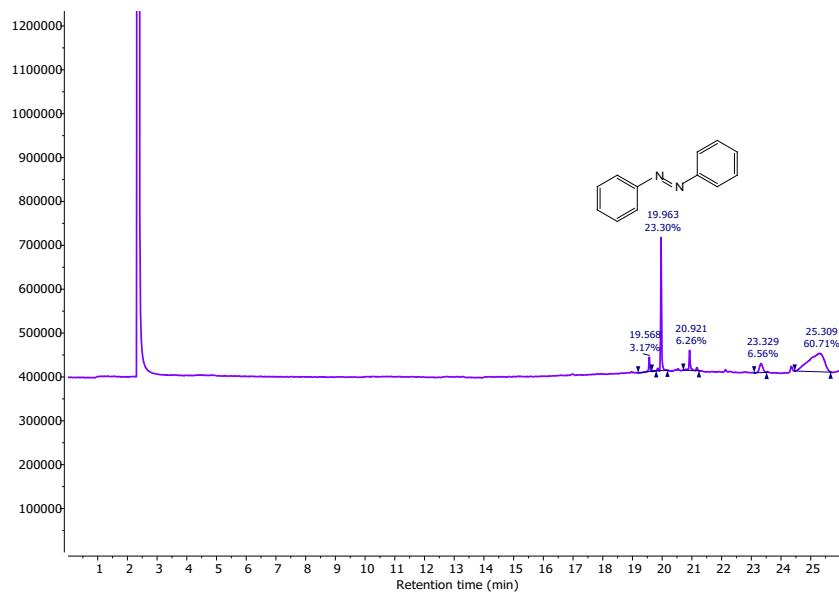
**Fig. S64.** Gas Chromatography (GC) reaction conditions: 8 mg of  $\text{CdS}(2.5\%)\text{/NH}_2\text{-MIL-125}$  photocatalyst, 0.2 mmol of nitrobenzene, 2 mL of methanol, 6 eq of hydrazine hydrate, darkness, argon atmosphere, time: 24 hours, room temperature



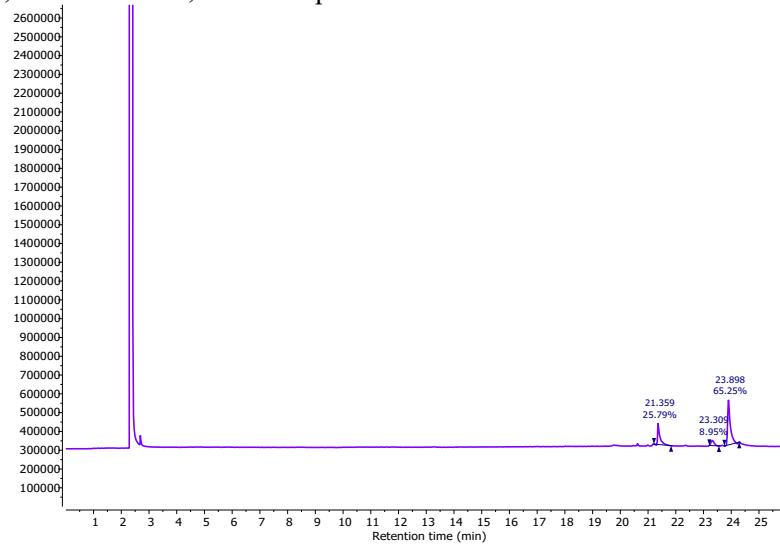
**Fig. S65.** Gas Chromatography (GC) Reaction conditions: 8 mg of CdS(2.5%)/NH<sub>2</sub>-MIL-125 photocatalyst, 0.2 mmol of nitrobenzene, 2 mL of methanol, 6 eq of hydrazine hydrate, 12 W white lamp, argon atmosphere, time: 6 hours, room temperature.



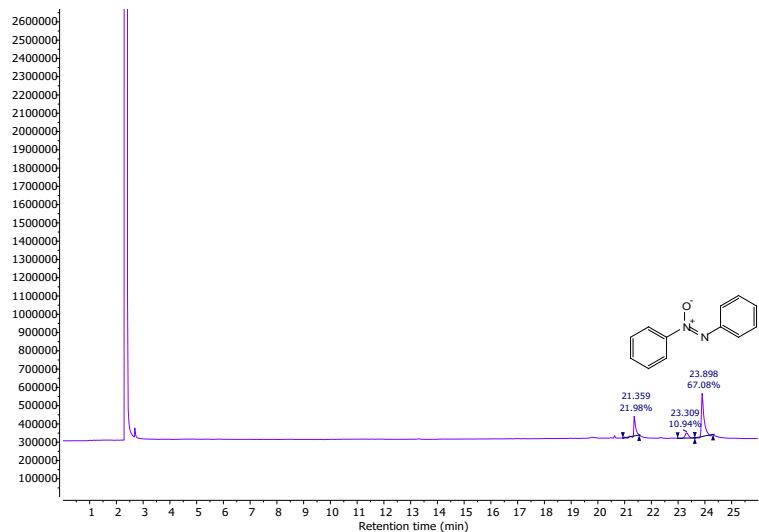
**Fig. 66** Gas Chromatography (GC) reaction conditions: 8 mg of CdS(2.5%)/NH<sub>2</sub>-MIL-125 photocatalyst, 0.2 mmol of nitrobenzene, 2 mL of methanol, 6 eq of hydrazine hydrate, 12 W white lamp, argon atmosphere, time: 18 hours, room temperature.



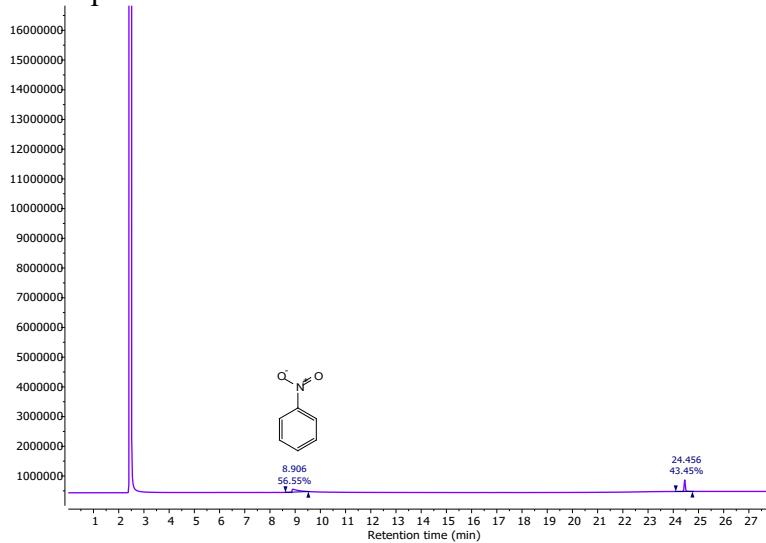
**Fig. S67.** Gas Chromatography (GC) Reaction conditions: 8 mg of CdS(2.5%)/NH<sub>2</sub>-MIL-125 photocatalyst, 0.2 mmol of nitrobenzene, 2 mL of methanol, 6 eq of hydrazine hydrate, 12 W white lamp, oxygen atmosphere, time: 24 hours, room temperature.



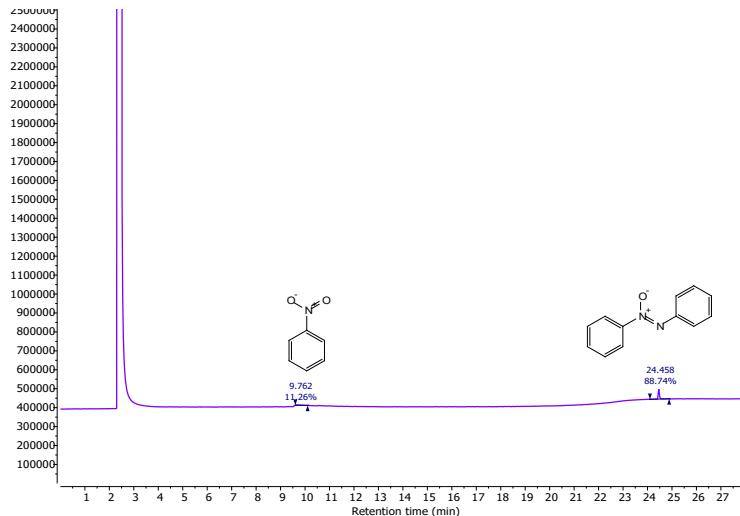
**Fig. S68.** Gas Chromatography (GC) Reaction Conditions: 8 mg of CdS(2.5%)/NH<sub>2</sub>-MIL-125 photocatalyst, 0.5 mmol of nitrobenzene, 2 mL of tetrahydrofuran, 4 eq of hydrazine hydrate, 12 W white lamp, argon atmosphere, time: 24 hours, room temperature



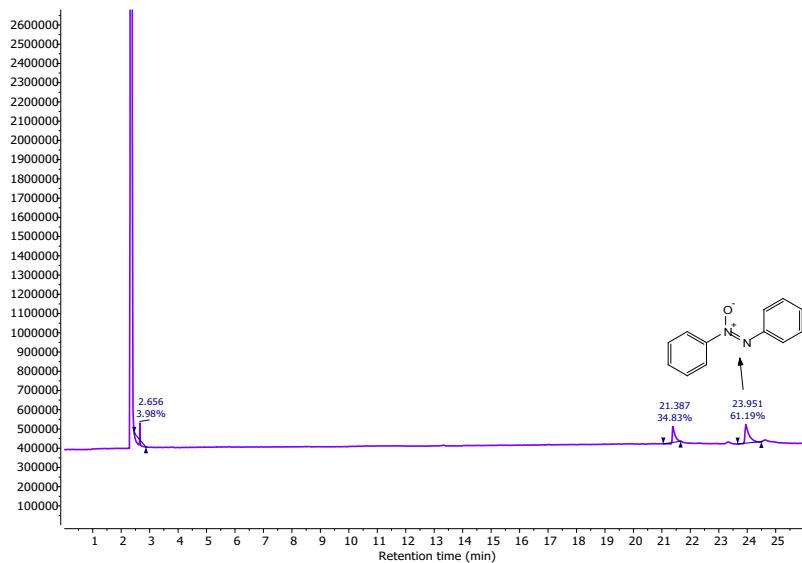
**Fig. S69.** Gas Chromatography (GC) reaction conditions: 8 mg CdS photocatalyst, 0.2 mmol nitrobenzene, 2 mL tetrahydrofuran, 6 eq hydrazine hydrate, 12 W white lamp, argon atmosphere, time: 24 hours, room temperature.



**Fig. S70.** Gas Chromatography (GC) Reaction conditions: 8 mg NH<sub>2</sub>-MIL-125 photocatalyst, 0.2 mmol nitrobenzene, 2 mL tetrahydrofuran, 6 eq hydrazine hydrate, 12 W white lamp, argon atmosphere, time: 24 hours, room temperature

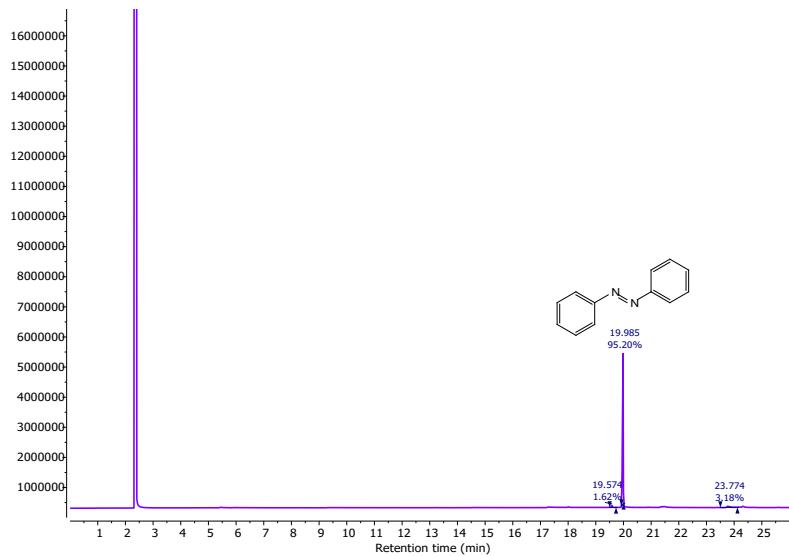


**Fig. S71.** Gas Chromatography (GC) reaction conditions: 10 mg of CdS(2.5%)/NH<sub>2</sub>-MIL-125 photocatalyst, 0.2 mmol of nitrobenzene, 2 mL of tetrahydrofuran, 6 eq of hydrazine hydrate, 12 W white lamp, argon atmosphere, time: 24 hours, room temperature.

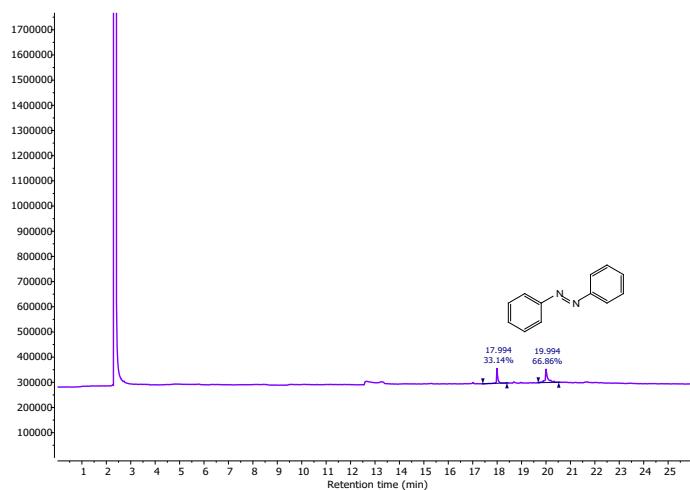


**Fig. S72.** Gas Chromatography (GC) reaction conditions: 8 mg of CdS(2.5%)/NH<sub>2</sub>-MIL-125 photocatalyst, 0.2 mmol of nitrobenzene, 2 mL of tetrahydrofuran solvent, 6 eq of hydrazine hydrate, 9Wblue lamp, argon atmosphere, time: 24 hours, room temperature.

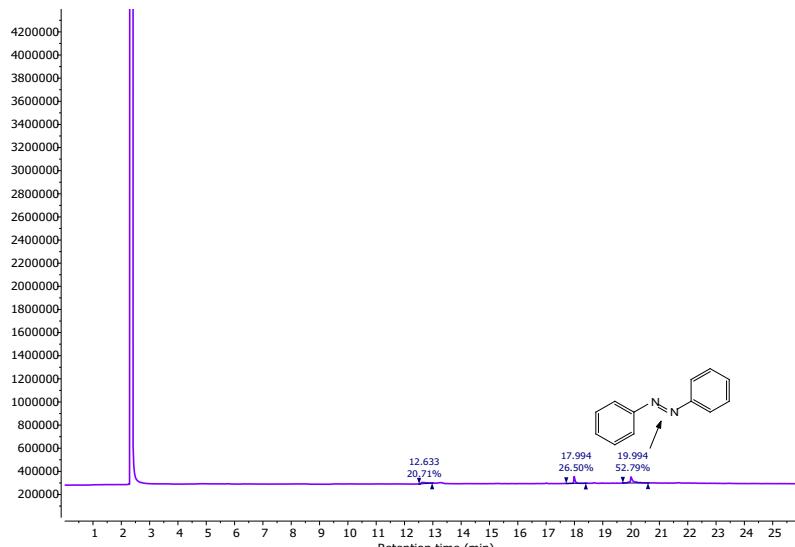
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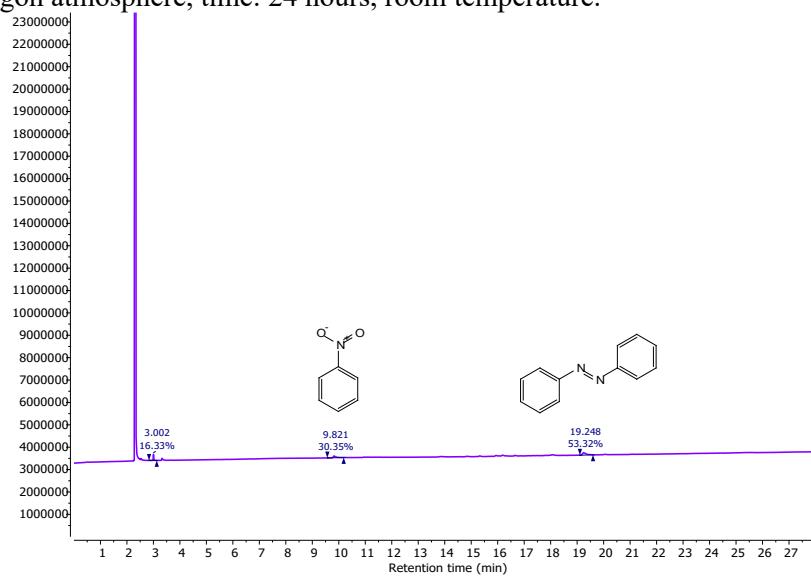
**Fig. S73.** Gas Chromatography (GC) Reaction Conditions: 8 mg of CdS(2.5%)/NH<sub>2</sub>-MIL-125 photocatalyst, 0.2 mmol of nitrobenzene, 2 mL of ethanol, 6 eq of hydrazine hydrate, 0.5 mmol of potassium carbonate, 12 W white lamp, argon atmosphere, time: 24 hours, room temperature.



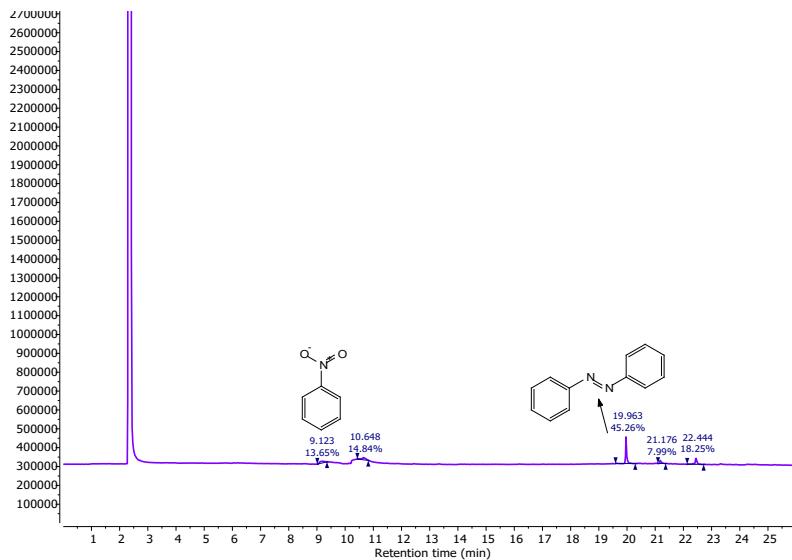
**Fig. S74.** Gas Chromatography (GC) reaction conditions: 8 mg of CdS(2.5%)/NH<sub>2</sub>-MIL-125 photocatalyst, 0.2 mmol of nitrobenzene, 2 mL of methanol, 6 eq of hydrazine hydrate, 0.5 mmol of potassium carbonate, 12 W white lamp, argon atmosphere, time: 24 hours, room temperature.



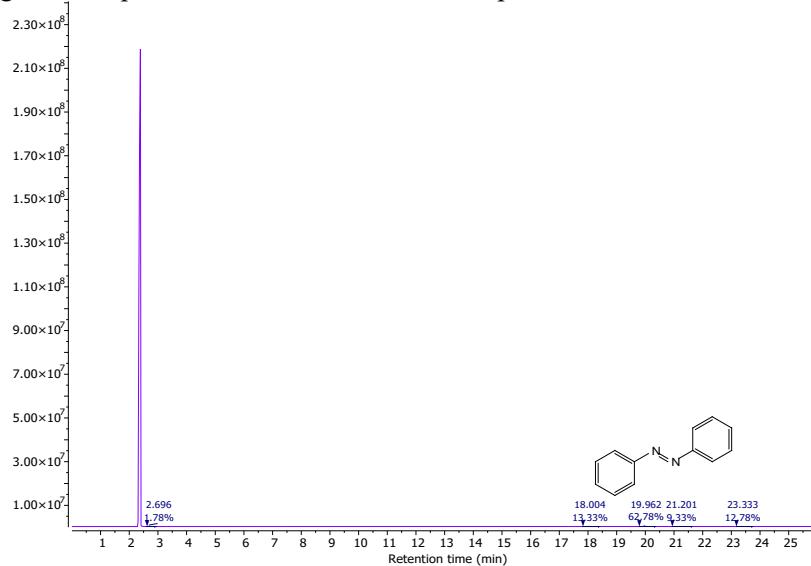
**Fig. S75.** Gas Chromatography (GC) reaction conditions: 8 mg of CdS(2.5%)/NH<sub>2</sub>-MIL-125 photocatalyst, 0.2 mmol of nitrobenzene, 2 mL of acetonitrile solvent, 0.5 mmol of K<sub>2</sub>CO<sub>3</sub>, 6 eq of hydrazine hydrate, 12 W white lamp, argon atmosphere, time: 24 hours, room temperature.



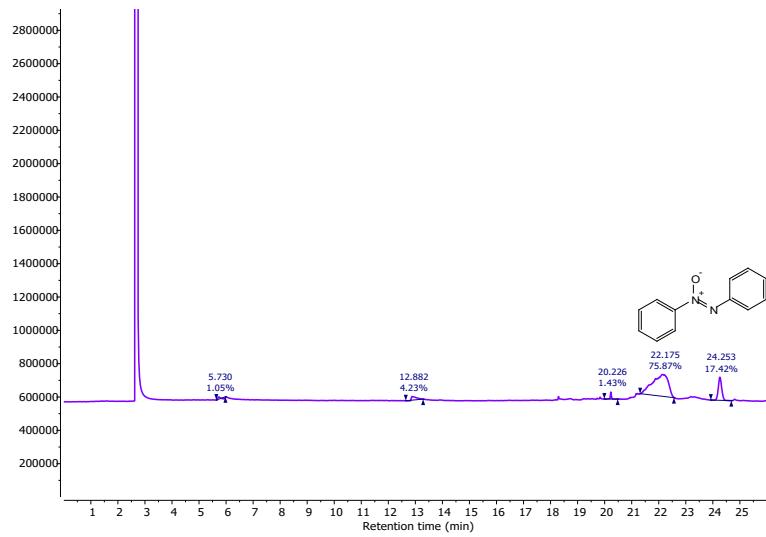
**Fig. S76.** Gas Chromatography (GC) reaction conditions: 8 mg of CdS(2.5%)/NH<sub>2</sub>-MIL-125 photocatalyst, 0.2 mmol of nitrobenzene, 2 ml of ethanol, 6 eq of hydrazine hydrate, 0.4 mmol of potassium carbonate, 12 W white lamp, argon atmosphere, time: 24 hours, room temperature.



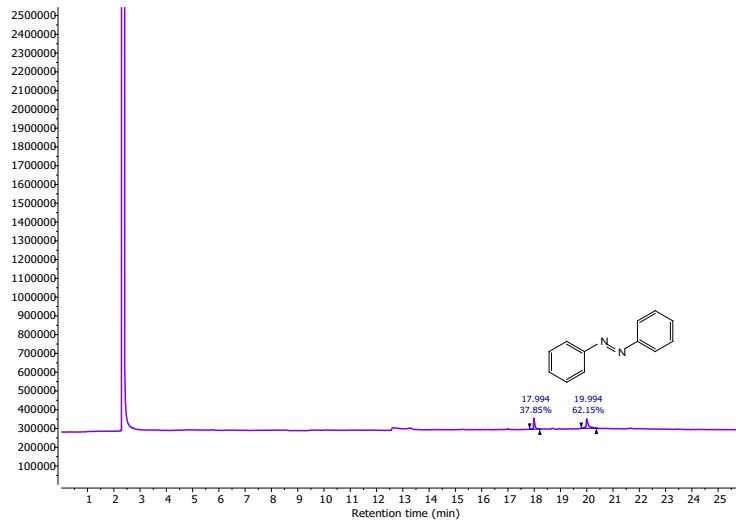
**Fig. S77.** Gas Chromatography (GC) Reaction conditions: 8 mg of CdS(2.5%)/NH<sub>2</sub>-MIL-125 catalyst, 0.2 mmol of nitrobenzene, 2 mL of ethanol, 6 eq of hydrazine hydrate, 0.5 mmol of potassium carbonate, 12 W white lamp, argon atmosphere, time: 12 hours, room temperature.



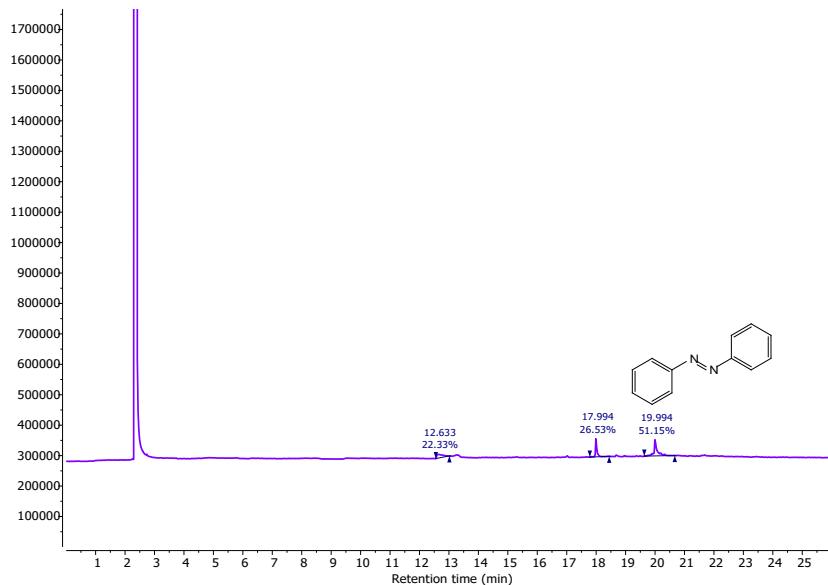
**Fig. S77.** Gas Chromatography (GC) reaction conditions: 8 mg of CdS(2.5%)/NH<sub>2</sub>-MIL-125 photocatalyst, 0.2 mmol of nitrobenzene, 2 mL of methanol, 4 eq of hydrazine hydrate, 0.5 mmol of potassium carbonate, 12 W white lamp, argon atmosphere, time: 24 hours, room temperature.



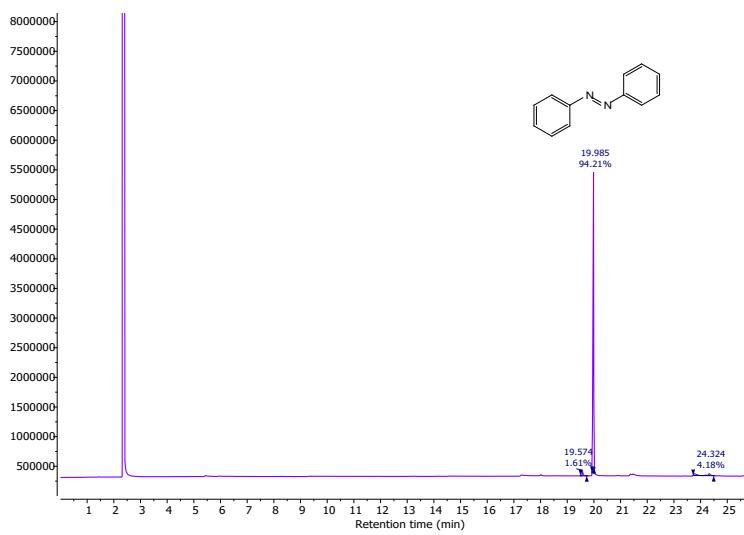
**Fig. S78.** Gas Chromatography (GC) reaction conditions: 8 mg of CdS(2.5%)/NH<sub>2</sub>-MIL-125 photocatalyst, 0.2 mmol of nitrobenzene, 2 mL of ethanol, 6 eq of hydrazine hydrate, 0.5 mmol of potassium carbonate, 12 W white lamp, oxygen atmosphere, time: 24 hours, room temperature.



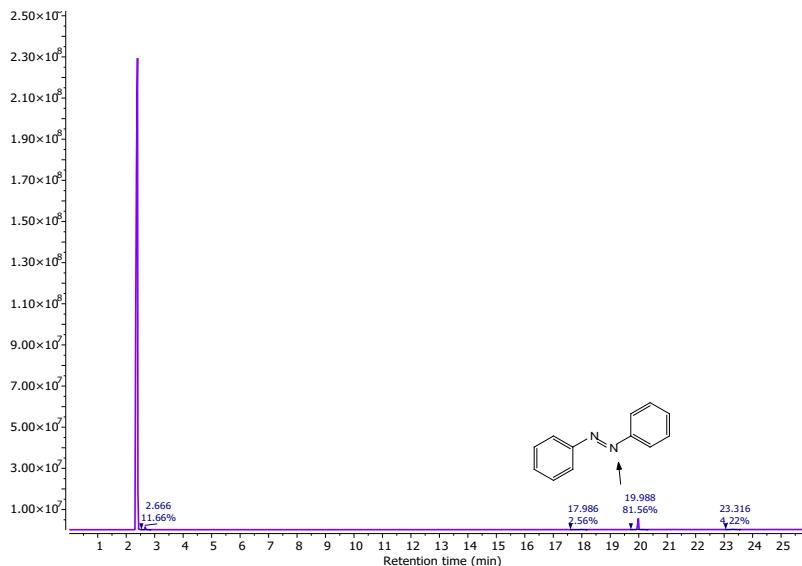
**Fig. S79.** Gas Chromatography (GC) reaction conditions: 8 mg CdS photocatalyst, 0.2 mmol nitrobenzene, 2 mL ethanol, 6 eq hydrazine hydrate, 0.5 mmol potassium carbonate, 12 W white lamp, argon atmosphere, time: 24 hours, room temperature.



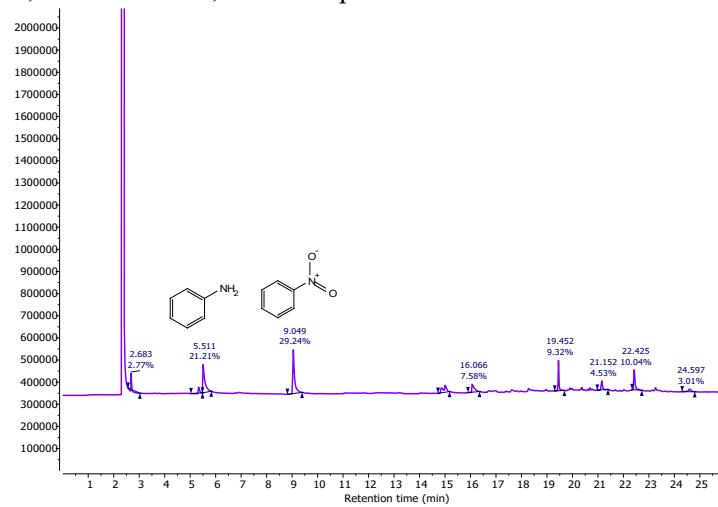
**Fig. S80.** Gas Chromatography (GC) reaction conditions: 8 mg of NH<sub>2</sub>-MIL-125 photocatalyst, 0.2 mmol of nitrobenzene, 2 mL of ethanol, 6 eq of hydrazine hydrate, 0.5 mmol of potassium carbonate, 12-W white lamp, argon atmosphere, time: 24 hours, room temperature.



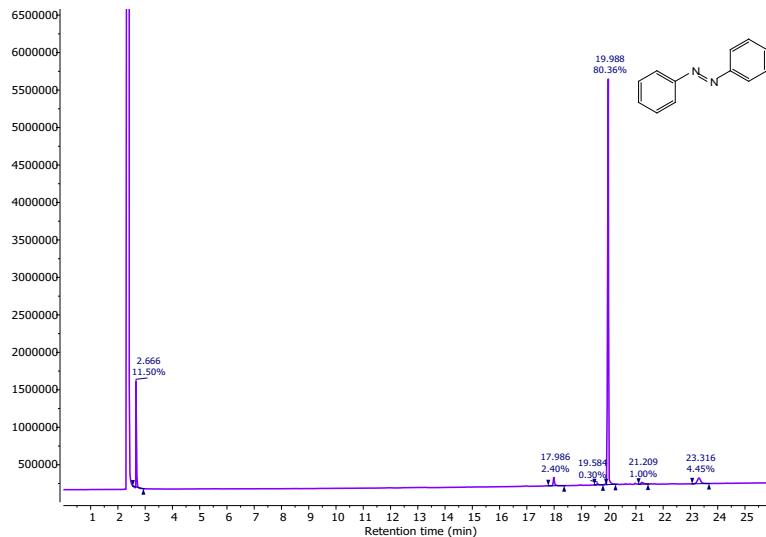
**Fig. S81.** Gas Chromatography (GC) Reaction conditions: 8 mg of CdS(2.5%)/NH<sub>2</sub>-MIL-125 photocatalyst, 0.2 mmol of nitrobenzene, 2 ml of ethanol, 8 eq of hydrazine hydrate, 0.5 mmol of potassium carbonate, 12 W white lamp, argon atmosphere, time: 24 hours, room temperature .



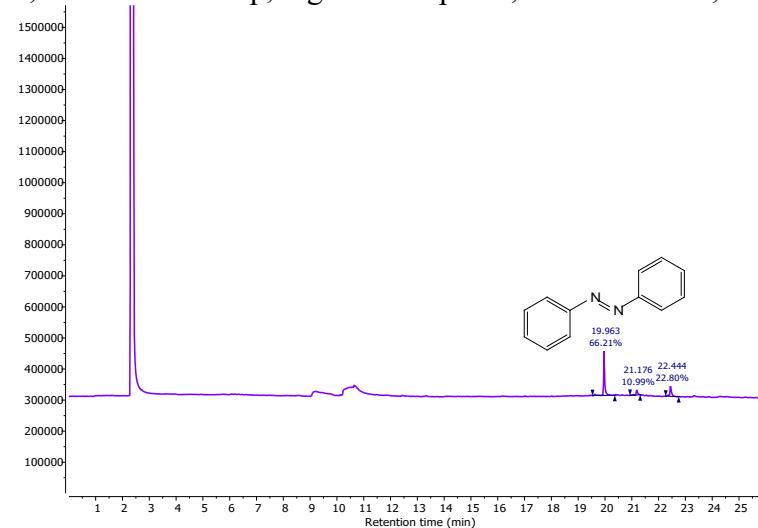
**Fig. S82.** Gas Chromatography (GC) reaction conditions: 8 mg of CdS(3.5%)/NH<sub>2</sub>-MIL-125 photocatalyst, 0.2 mmol of nitrobenzene, 2 ml of ethanol, 0.5 mmol of K<sub>2</sub>CO<sub>3</sub>, 6 eq of hydrazine hydrate, 12 W white lamp, argon atmosphere, time: 24 hours, room temperature .



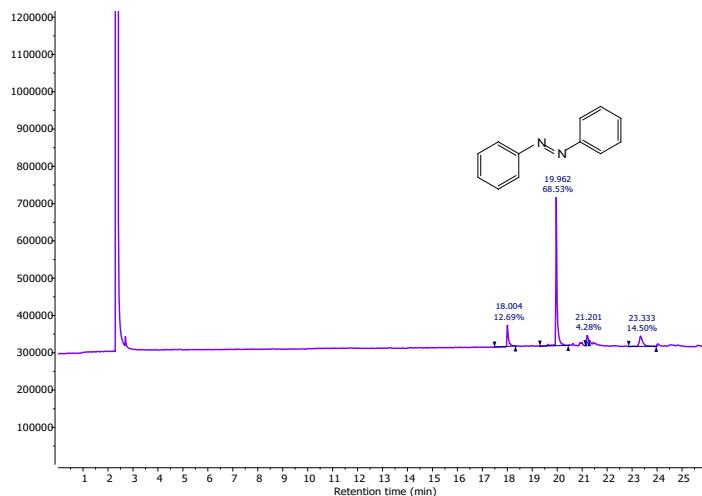
**Fig. S83.** Gas Chromatography (GC) reaction conditions: 8 mg of CdS(2.5%)/NH<sub>2</sub>-MIL-125 photocatalyst, 0.2 mmol of nitrobenzene, 2 mL of ethanol, 8 eq of hydrazine hydrate, 0.5 mmol of potassium carbonate, darkness, argon atmosphere, time: 24 hours, room temperature.



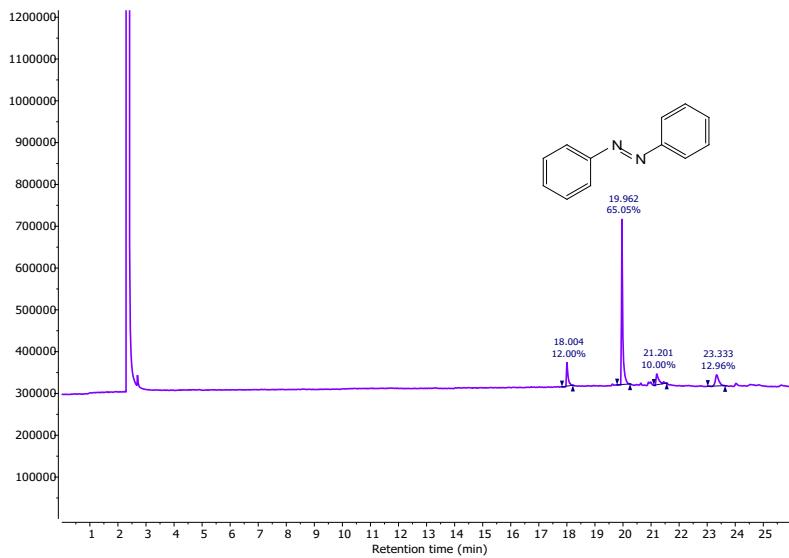
**Fig. S84.** Gas Chromatography (GC) Reaction conditions: 10 mg of CdS(2.5%)/NH<sub>2</sub>-MIL-125 photocatalyst, 0.5 mmol of nitrobenzene, 2 mL of ethanol, 6 eq of hydrazine hydrate, 0.5 mmol of potassium carbonate, 12 W white lamp, argon atmosphere, time: 24 hours, room temperature.



**Fig. S85.** Gas Chromatography (GC) reaction conditions: 8 mg of CdS(2.5%)/NH<sub>2</sub>-MIL-125 photocatalyst, 0.2 mmol of nitrobenzene, 2 mL of ethanol, 0.5 mmol of K<sub>2</sub>CO<sub>3</sub>, 6 eq of hydrazine hydrate, 12 W blue lamp, argon atmosphere, time: 24 hours, room temperature.



**Fig. S86.** Gas Chromatography (GC) reaction conditions: 8 mg of CdS(2.5%)/NH<sub>2</sub>-MIL-125 photocatalyst, 0.2 mmol of nitrobenzene, 2 mL of ethanol, 0.5 mmol of K<sub>2</sub>CO<sub>3</sub>, 6 eq of hydrazine hydrate, natural sunlight, argon atmosphere, time: 24 hours, room temperature.



**Fig. S87.** Gas Chromatography (GC) reaction conditions: 8 mg of recycled CdS(2.5%)/NH<sub>2</sub>-MIL-125 photocatalyst, 0.2 mmol of nitrobenzene, 2 mL of ethanol, 6 eq of hydrazine hydrate, 0.5 mmol of potassium carbonate, 12 W white lamp, argon atmosphere, time: 24 hours, room temperature.