

Supporting Information

Visible Light Active CdS Nanorods: One-Pot Synthesis of Aldonitrones

Moosa Ramdar^a, Foad Kazemi*^{a,b}, Babak Kaboudin^a, Zahra Taran^a and Adel Partovi^a

^aDepartment of Chemistry, Institute for Advanced Studies in Basic Sciences (IASBS), 49195-1159, Zanjan, Iran

^bCenter for Climate and Global Warming(CCGW), Institute for Advanced Studies in Basic Sciences (IASBS), GavaZang, Zanjan, Iran *e-mail: kazemi_f@iasbs.ac.ir

Table of contents:

1. General Information
2. Fig. S1.TGA analysis of cadmium sulfide nanorod.
3. Fig. S2. Dependences of photovoltage (V vs Ag/AgCl) on pH value of electrolyte for nanorodCdS.
4. Fig. S3. The UV–Vis diffuses reflectance spectrum of the CdS nanorod catalyst.
5. NMR spectra of nitrone compounds.
 - 5.1 .Fig. S4.¹H NMR Spectrum of (Z)-N,1-diphenylmethanimine oxide in CDCl₃
 - 5.2. Fig.S5.¹³C NMR Spectrum of (Z)-N,1-diphenylmethanimine oxide in CDCl₃
 - 5.3. Fig.S6.¹H NMR Spectrum of (Z)-1-phenyl-N-(*p*-tolyl)methanimine oxide in CDCl₃
 - 5.4. Fig.S7.¹³C NMR Spectrum of (Z)-1-phenyl-N-(*p*-tolyl)methanimine oxide in CDCl₃
 - 5.5.Fig. S8.¹H NMR Spectrum of (Z)-1-(*p*-chlorophenyl)-N-phenylmethanimine oxide in CDCl₃
 - 5.6.Fig. S9.¹³C NMR Spectrum of (Z)-1-(*p*-chlorophenyl)-N-phenylmethanimine oxide in CDCl₃
 - 5.7. Fig.S10.¹H NMR Spectrum of (Z)-N-m-tolyl-1-(*p*-tolyl)methanimine oxide in CDCl₃
 - 5.8. Fig. S11.¹³C NMR Spectrum of (Z)-N-m-tolyl-1-(*p*-tolyl)methanimine oxide in CDCl₃
 - 5.9. Fig.S12.¹H NMR Spectrum of (Z)-1-(*p*-chlorophenyl)-N-(m-tolyl)methanimine oxide in CDCl₃
 - 5.10. Fig.S13.¹³C NMR Spectrum of (Z)-1-(*p*-chlorophenyl)-N-(m-tolyl)methanimine oxide in CDCl₃
 - 5.11.Fig. S14.¹H NMR Spectrum of (Z)-1-phenyl-N-(m-tolyl)methanimine oxide in CDCl₃
 - 5.12. Fig. S15.¹³C NMR Spectrum of (Z)-1-phenyl-N-(m-tolyl)methanimine oxide in CDCl₃
 - 5.13. Fig.S16.¹H NMR Spectrum of (Z)-1-(*p*-(methylthio)phenyl)-N-phenylmethanimine oxide in CDCl₃
 - 5.14. Fig. S17.¹³C NMR Spectrum of (Z)-1-(*p*-(methylthio)phenyl)-N-phenylmethanimine oxide in CDCl₃
 - 5.15. Fig. S18. ¹H NMR Spectrum of (Z)-N,1-di-m-tolylmethanimine oxide in CDCl₃
 - 5.16. Fig. S19.¹³C NMR Spectrum of (Z)-N,1-di-m-tolylmethanimine oxide in CDCl₃
6. Spectral data
7. Table S1. CHNS analysis of nitrone compounds.
8. Table S2. CHNS analysis of the CdS catalysts.

1. General Information

Nitro compounds, poly ethylene glycol-400 (PEG-400), cadmium chloride and oxalic acid were purchased from Merck Co. Ammonium formates (HCO_2NH_4) was supplied by Fluka Co. All chemicals were used as received without further purification. Deionized water was used in all experiments. ^1H and ^{13}C nuclear magnetic resonance (NMR) spectra were recorded on Bruker 400 MHz-Avance III. CHNS analysis was recorded with ElementarVario EL III. UV-Visible diffusive reflectance spectra (DRS) were measured on Varian Cary 100 UV-Vis. TEM images was obtained with Philips CM120, VEGA TESCAN-XMU. Thermogravimetric analysis was conducted from room temperature to 700 °C in a nitrogen flow using a NETZSCH STA 409 PC/PG instrument. Nitrogen sorption analysis (Belsorp, BELMAX, Japan)was used for further analysis of the catalyst.

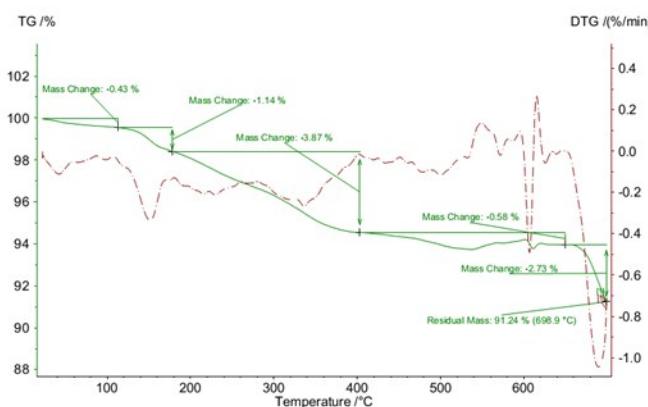


Fig. S1 TGA analysis of the CdS nanorod.

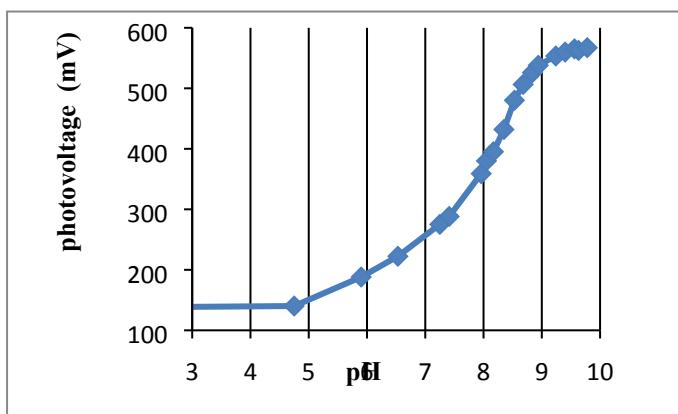


Fig. S2Dependences of photovoltage (V vs Ag/AgCl) on pH value of electrolyte for nanorod CdS

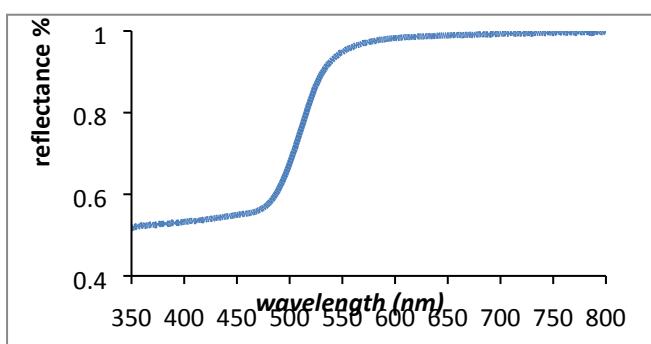


Fig. S3The UV-Vis diffuses reflectance spectrum of the CdS nanorod catalyst.

5. NMR spectra of nitrone compounds

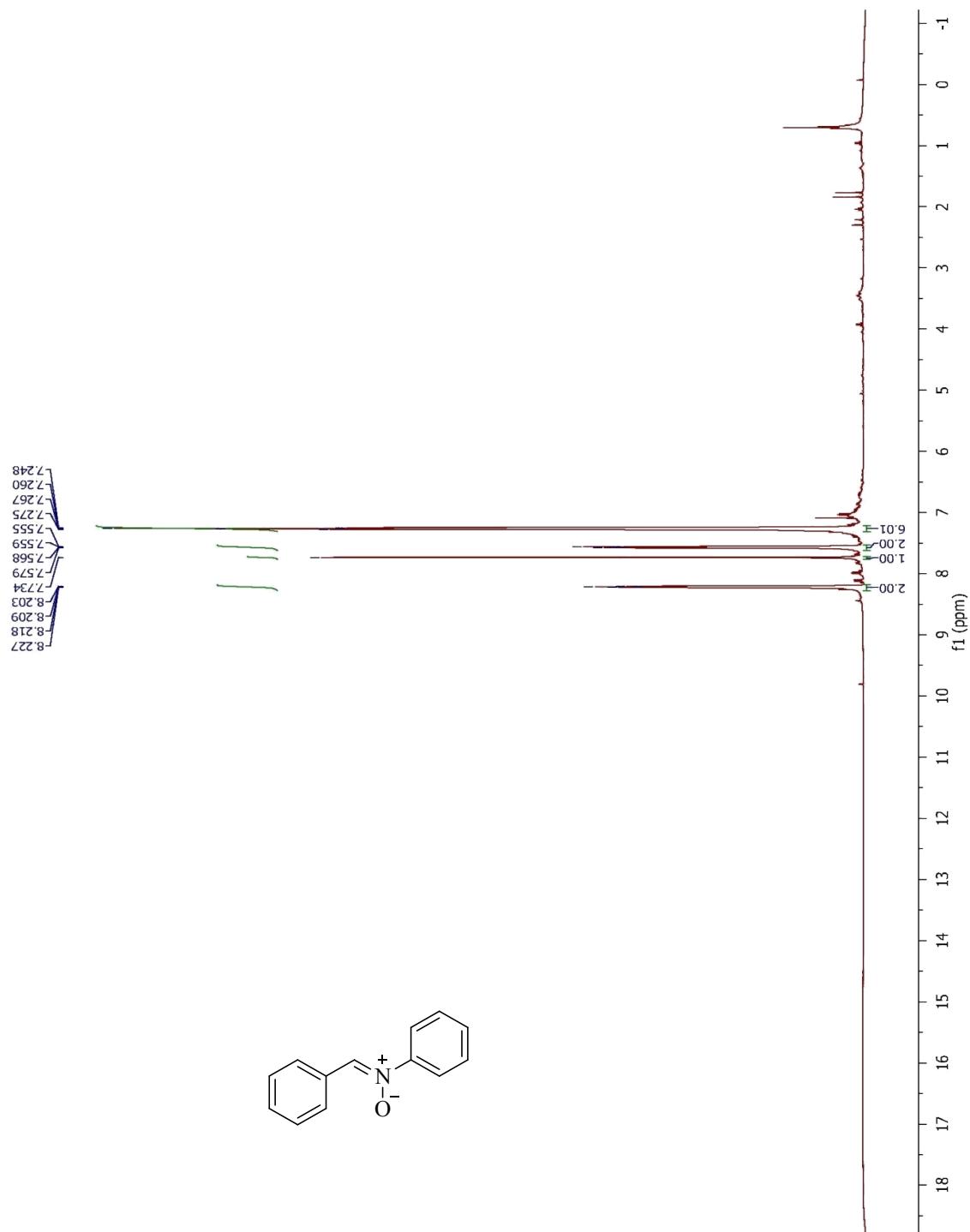


Fig.S 4 ^1H NMR Spectrum of (Z)-N,1-diphenylmethanimine oxide in CDCl_3 .

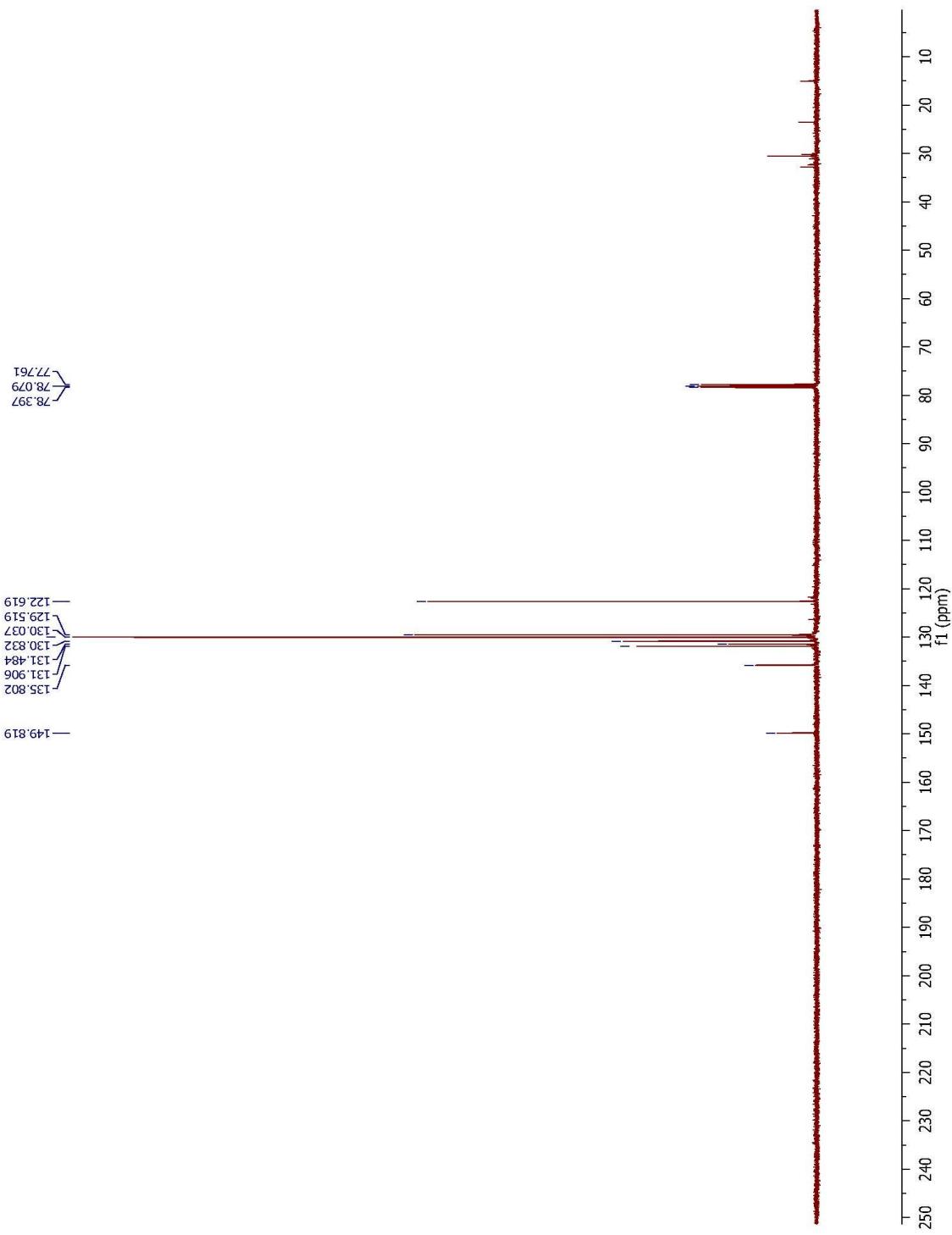


Fig.S5 ^{13}C NMR Spectrum of (Z)-N,1-diphenylmethanimine oxide in CDCl_3 .

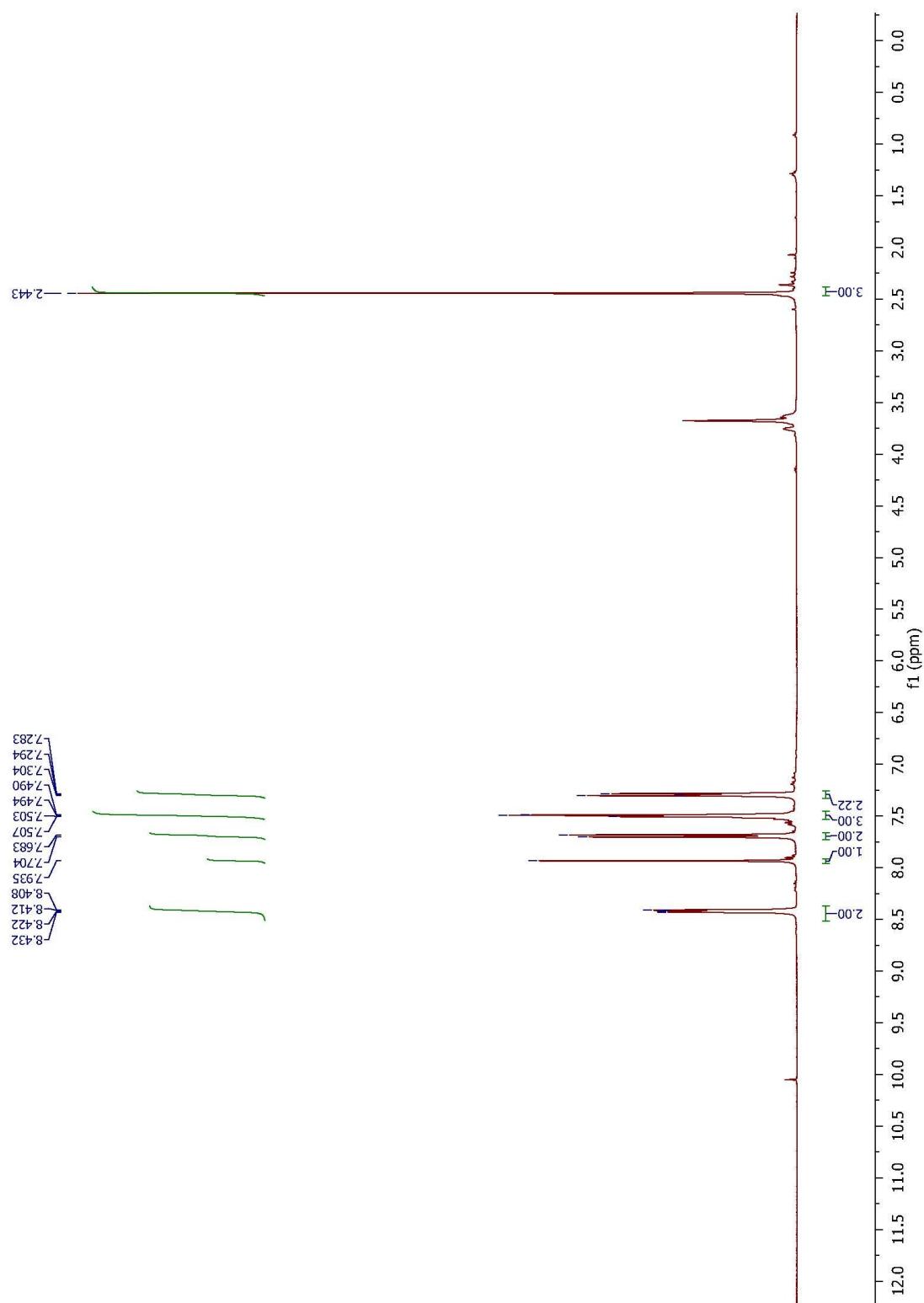


Fig.S6 ${}^1\text{H}$ NMR Spectrum of (Z)-1-phenyl-*N*-(*p*-tolyl)methanimine oxide in CDCl_3 .

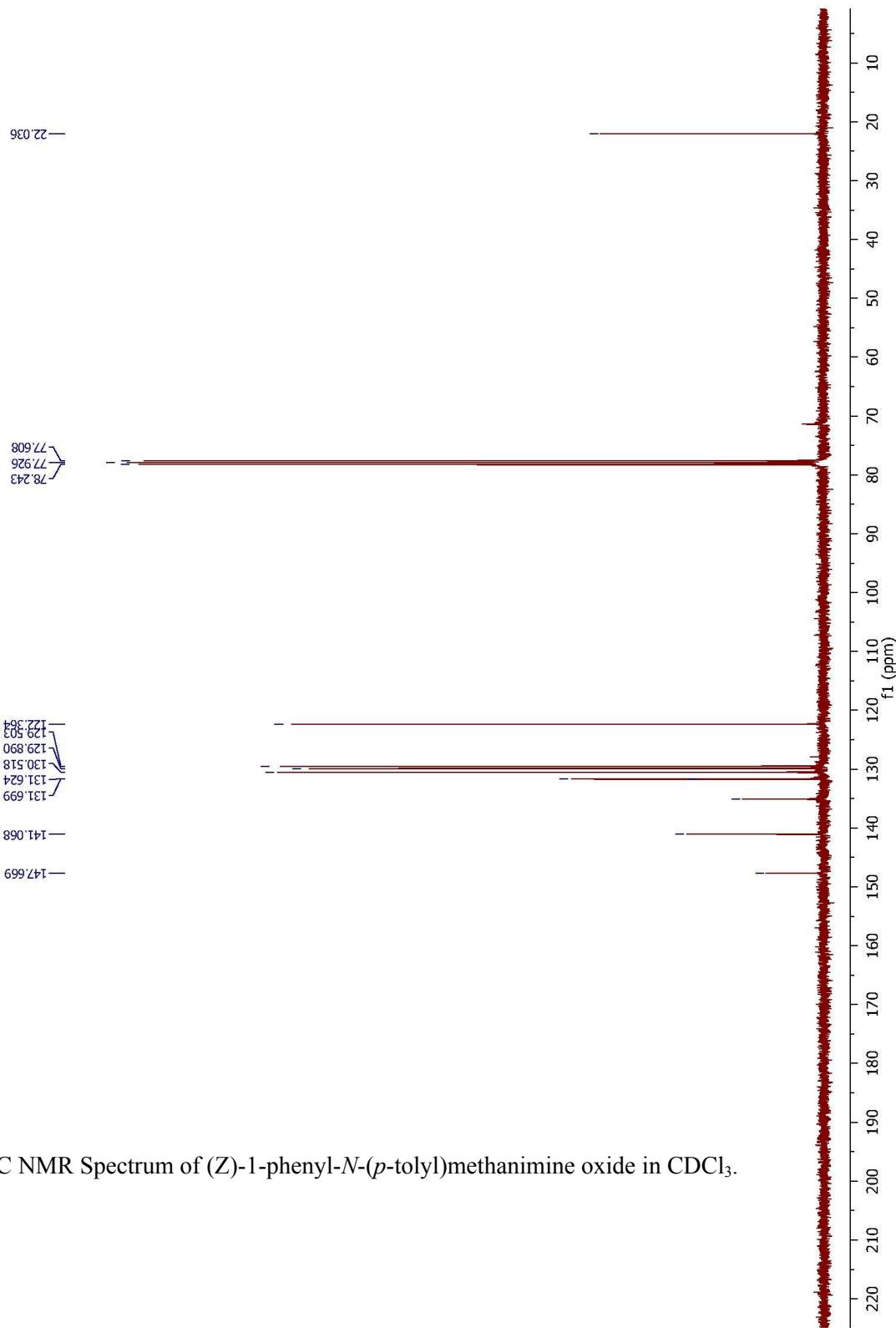
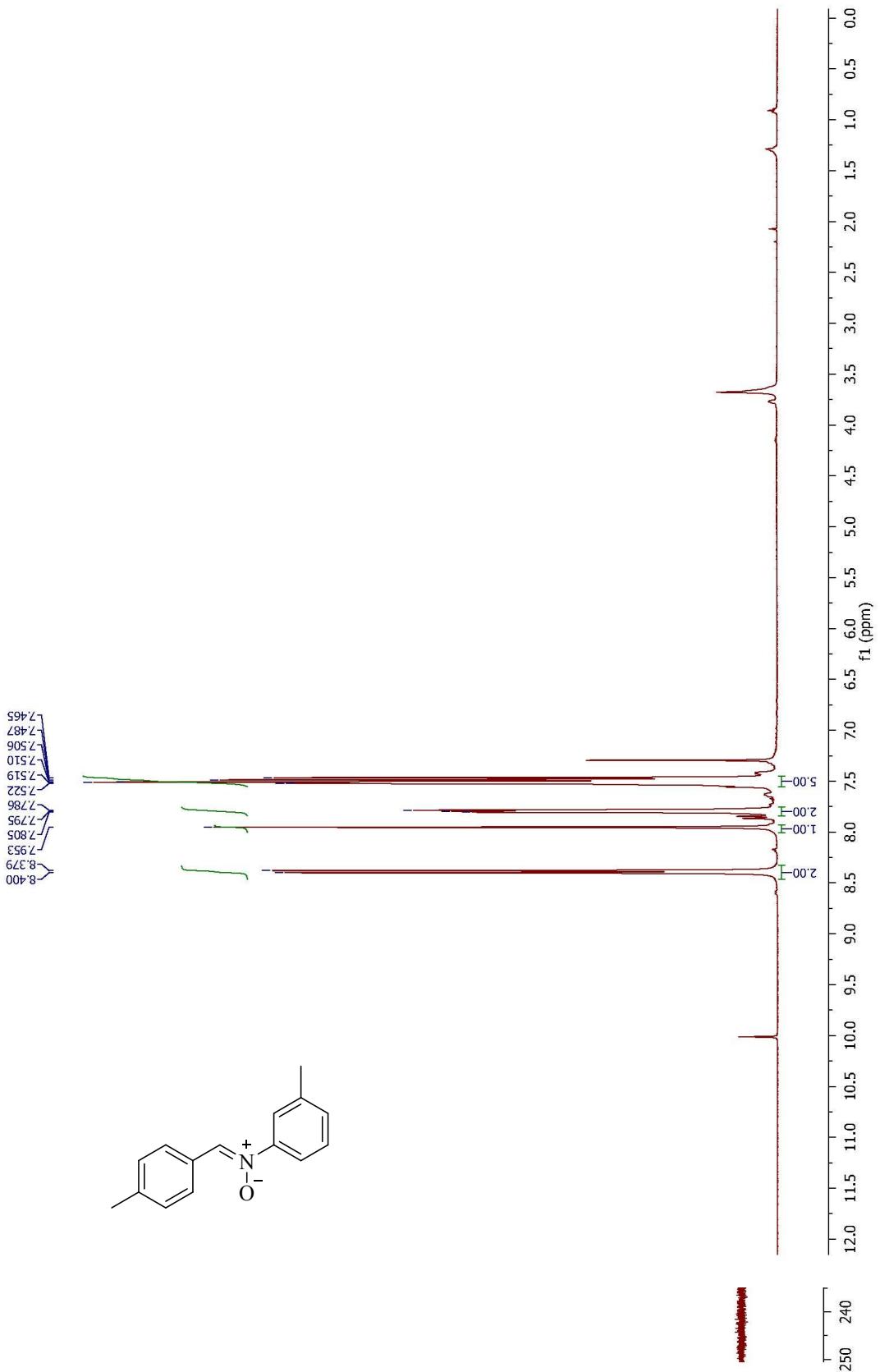


Fig.S7 ^{13}C NMR Spectrum of (Z)-1-phenyl-*N*-(*p*-tolyl)methanimine oxide in CDCl_3 .

Fig**Fig.S9** ^{13}C NMR Spectrum of (Z)-1-(*p*-chlorophenyl)-*N*-phenylmethanimine oxide in CDCl_3 .

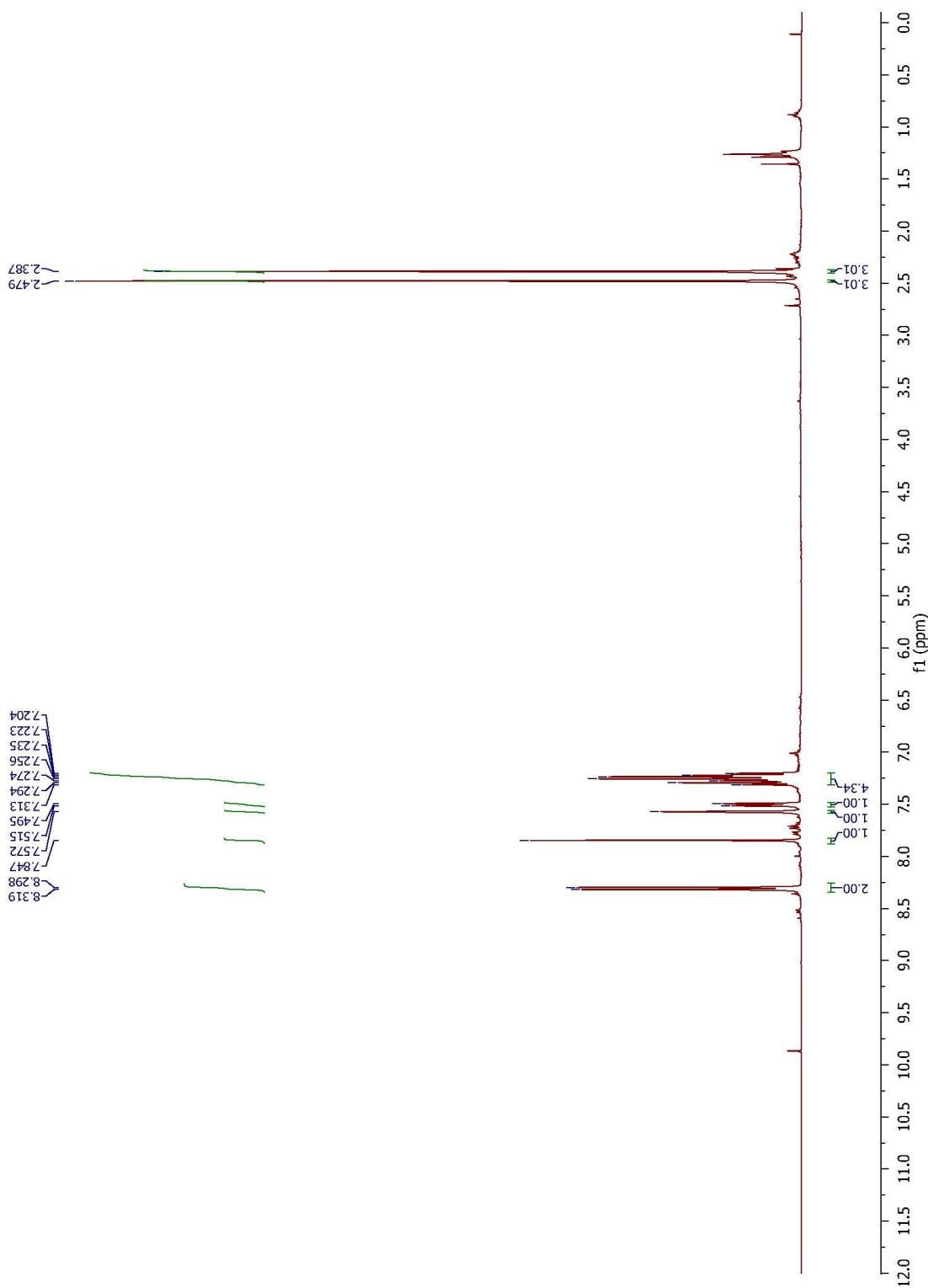


Fig.S10 ^1H NMR Spectrum of (Z)-*N*-*m*-tolyl-1-(*p*-tolyl)methanimine oxide in CDCl_3 .

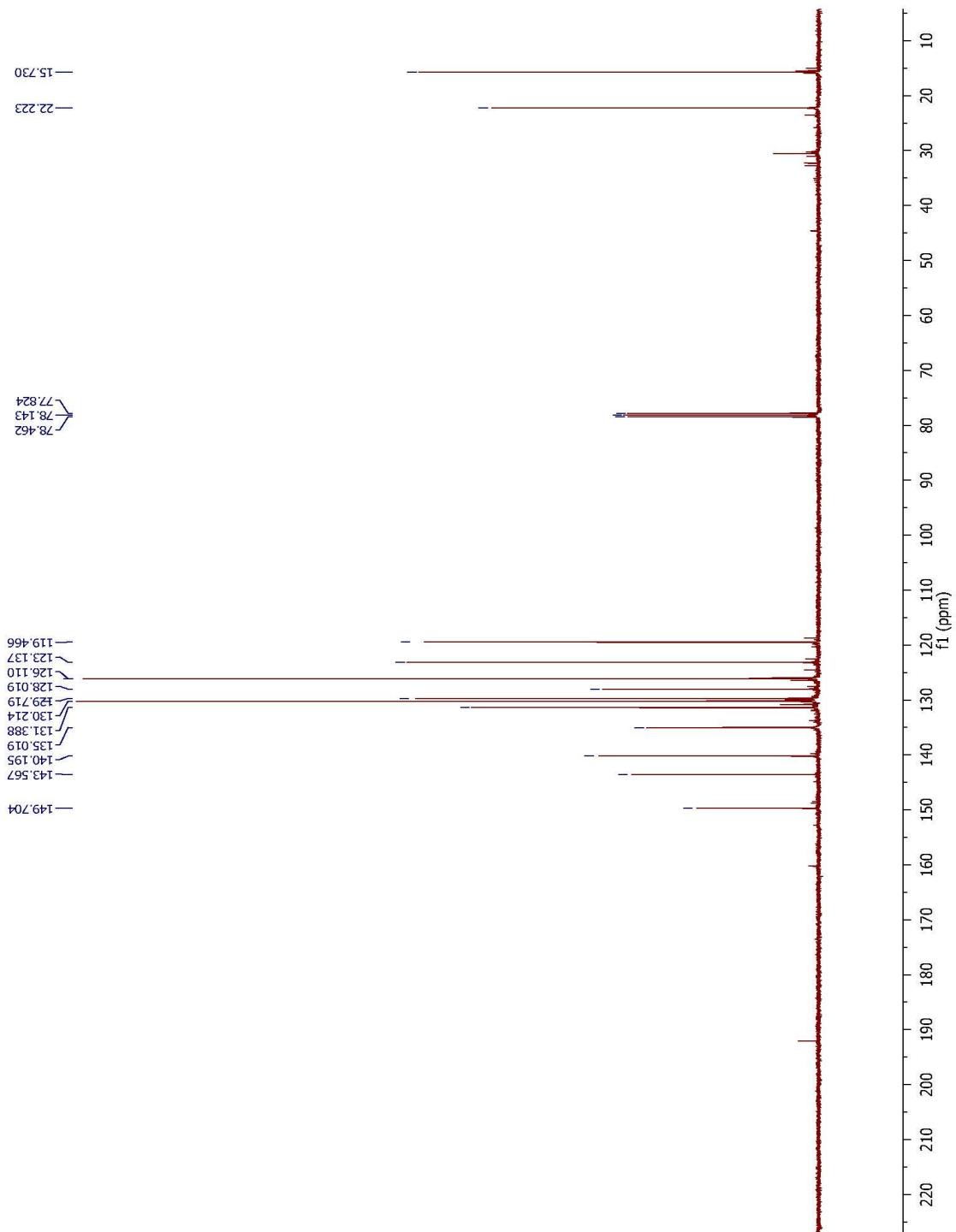


Fig.S11 ^{13}C NMR Spectrum of (Z)-*N*-*m*-tolyl-1-(*p*-tolyl)methanimine oxide in CDCl_3 .

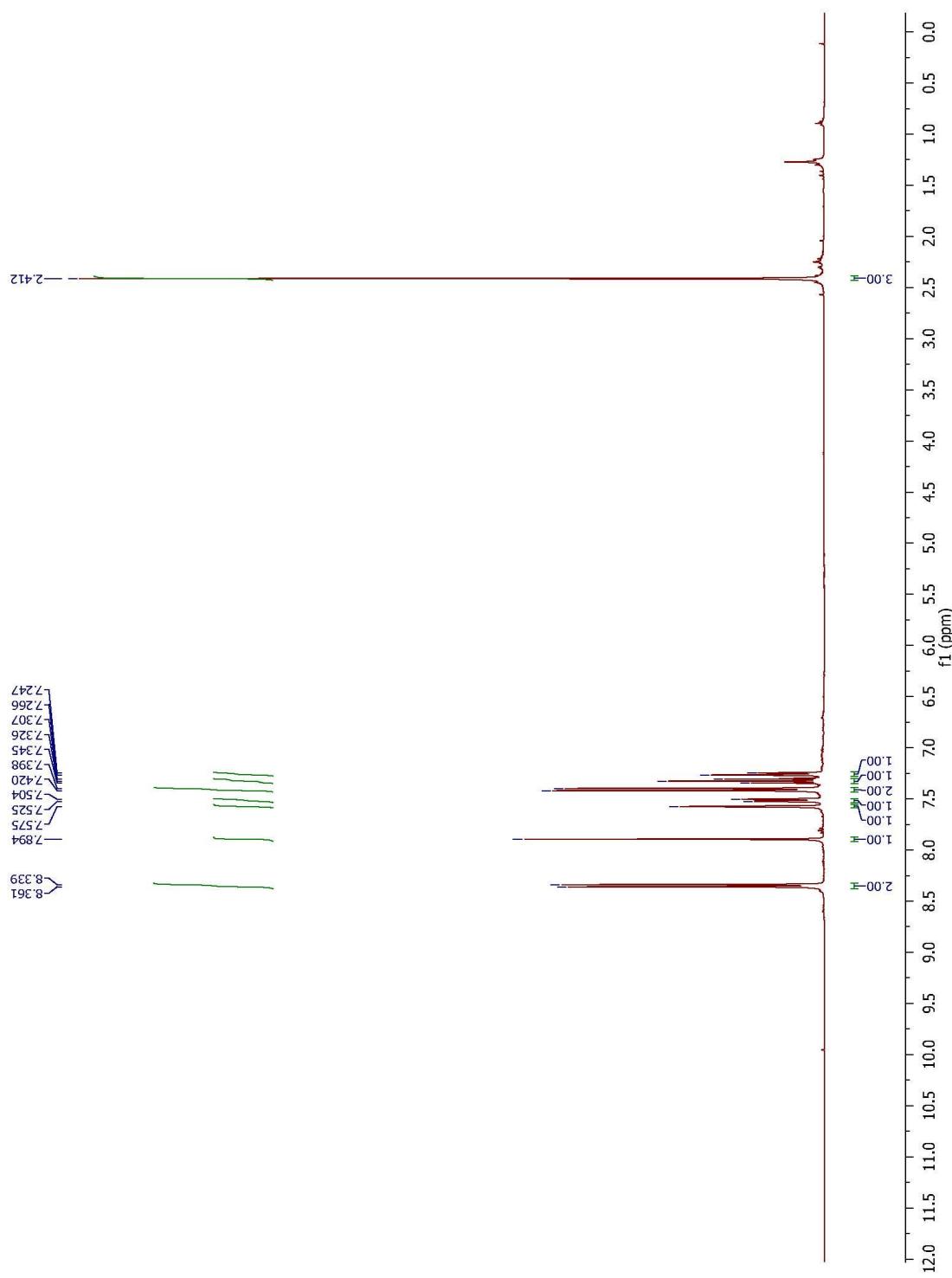


Fig.S12¹H NMR Spectrum of (Z)-1-(*p*-chlorophenyl)-*N*-(*m*-tolyl)methanimine oxide in CDCl₃.

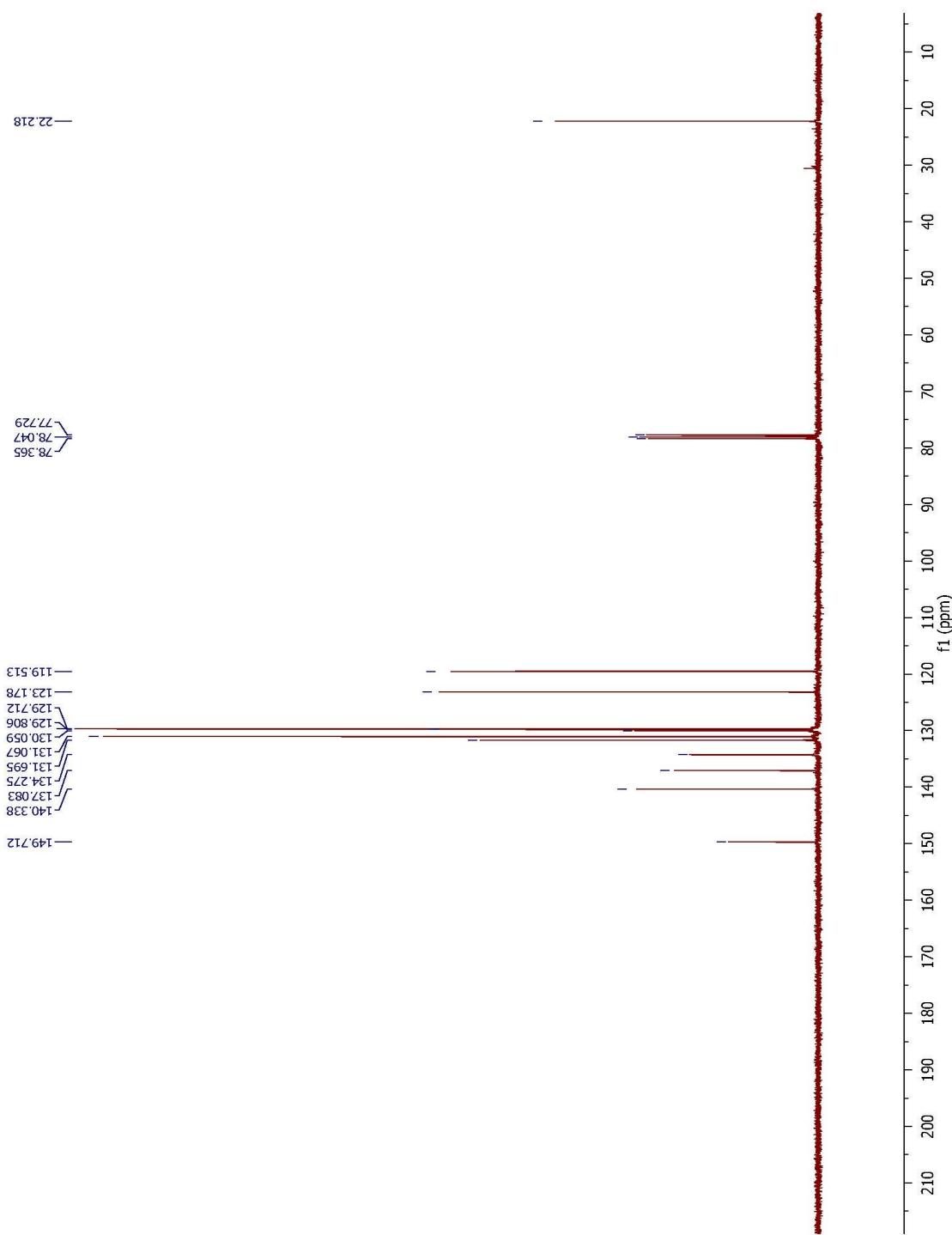


Fig.S13 ^{13}C NMR Spectrum of (Z)-1-(*p*-chlorophenyl)-*N*-(*m*-tolyl)methanimine oxide in CDCl_3 .

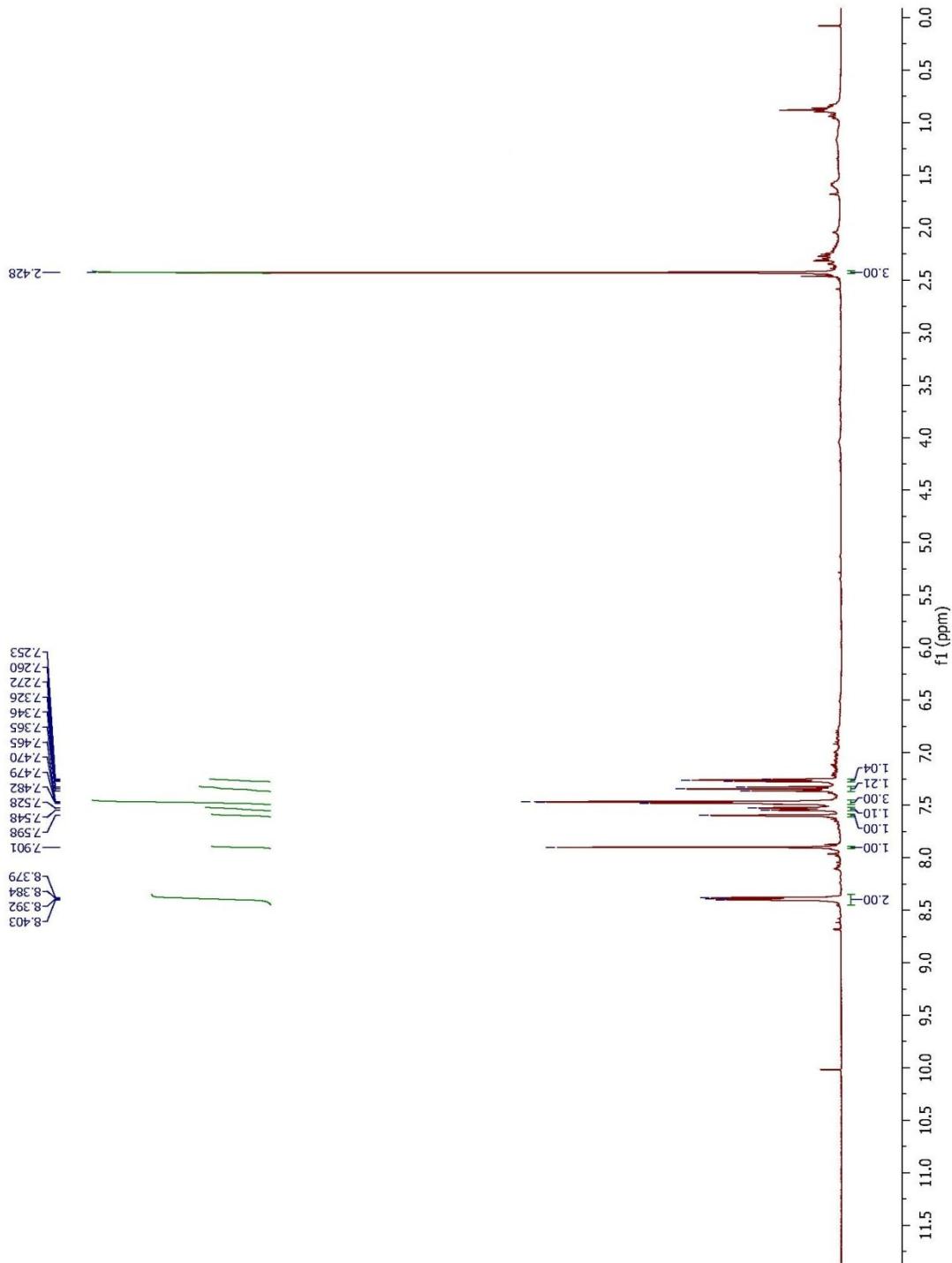


Fig.S14¹H NMR Spectrum of (Z)-1-phenyl-*N*-(*m*-tolyl)methanimine oxide in CDCl₃.

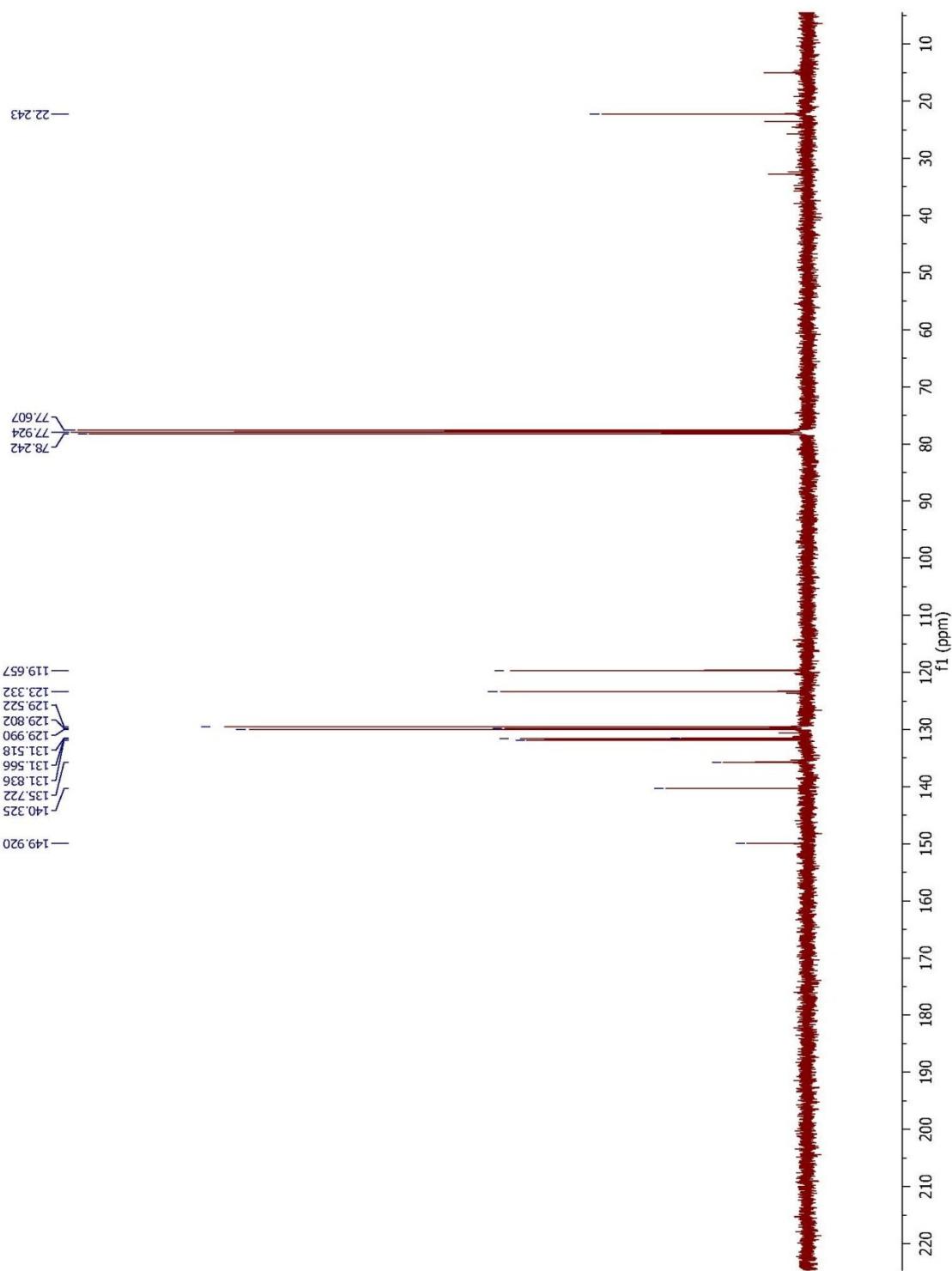


Fig.S15 ^{13}C NMR Spectrum of (Z)-1-phenyl- N -(*m*-tolyl)methanimine oxide in CDCl_3 .

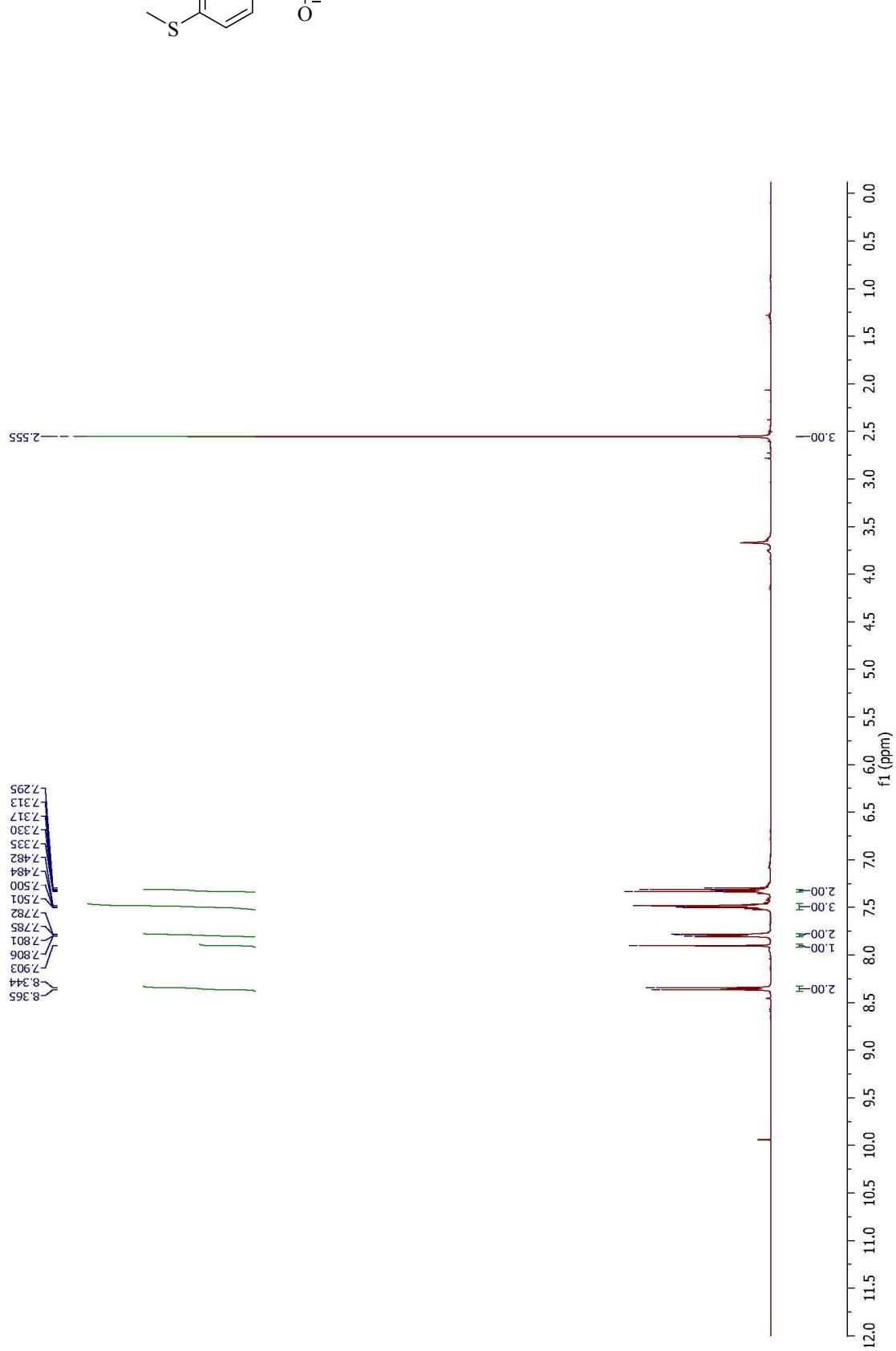


Fig.S16¹H NMR Spectrum of (Z)-1-(*p*-(methylthio)phenyl)-*N*-phenylmethanimine oxide in CDCl₃.

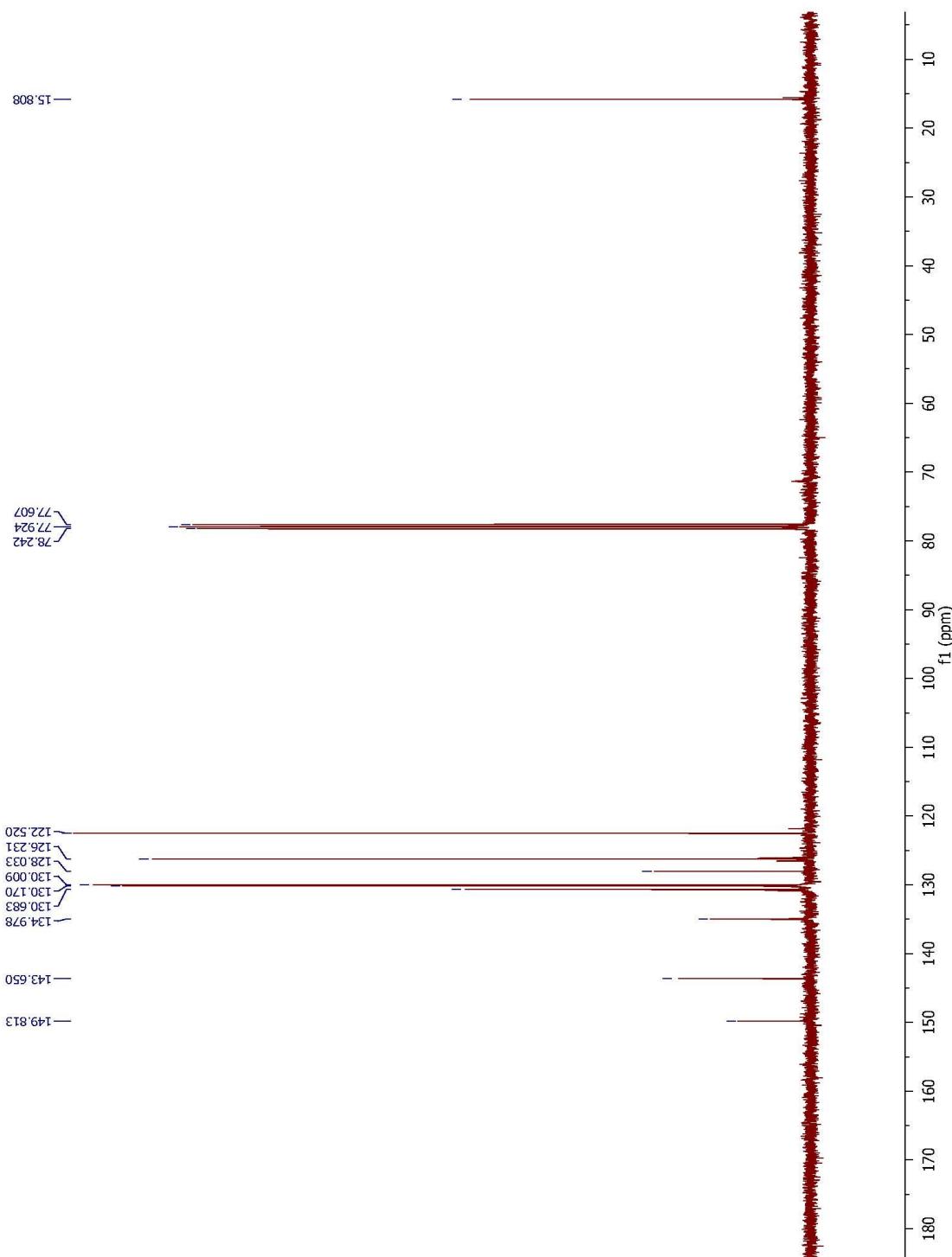


Fig.S17¹³C NMR Spectrum of (Z)-1-(*p*-(methylthio)phenyl)-*N*-phenylmethanimine oxide in CDCl₃.

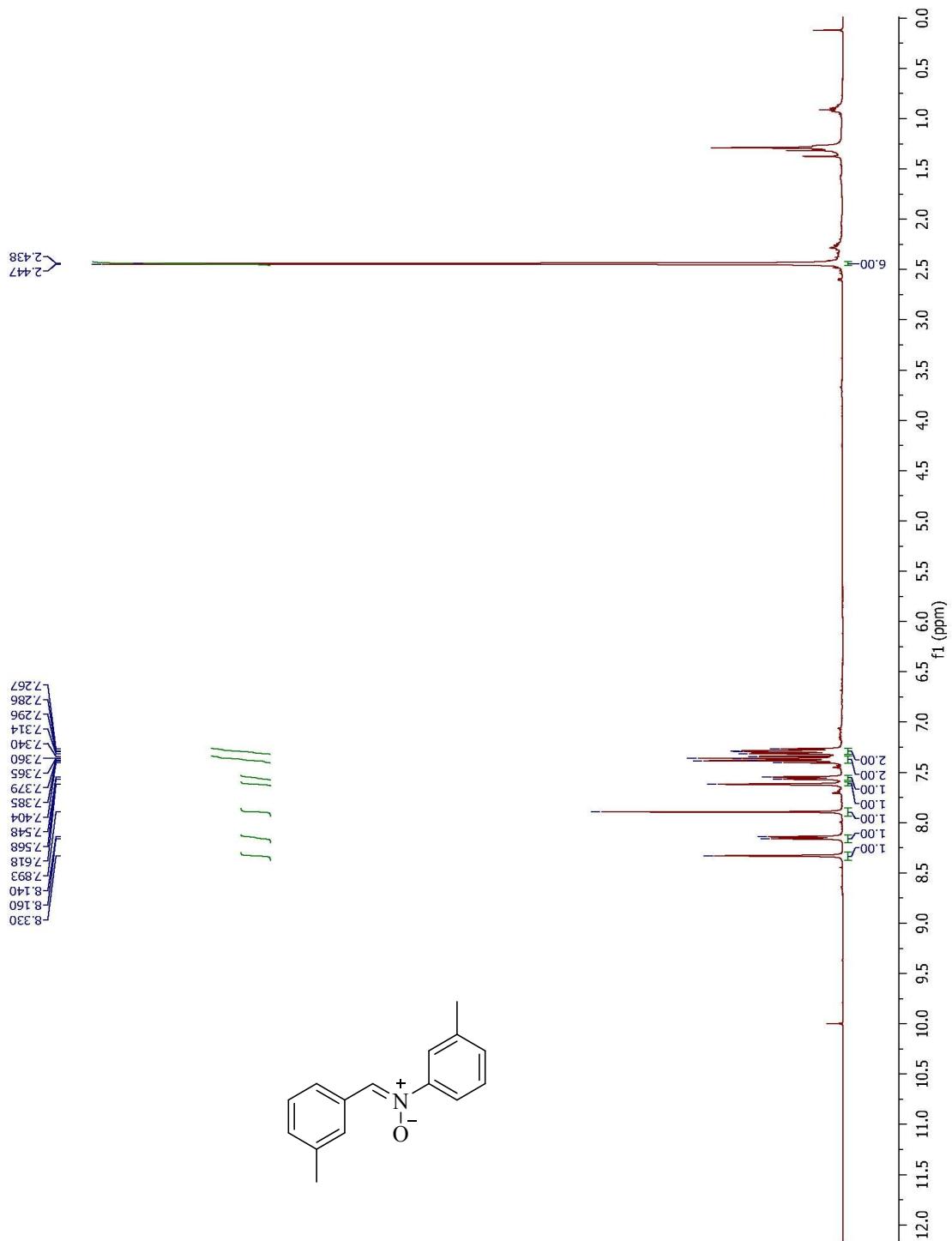
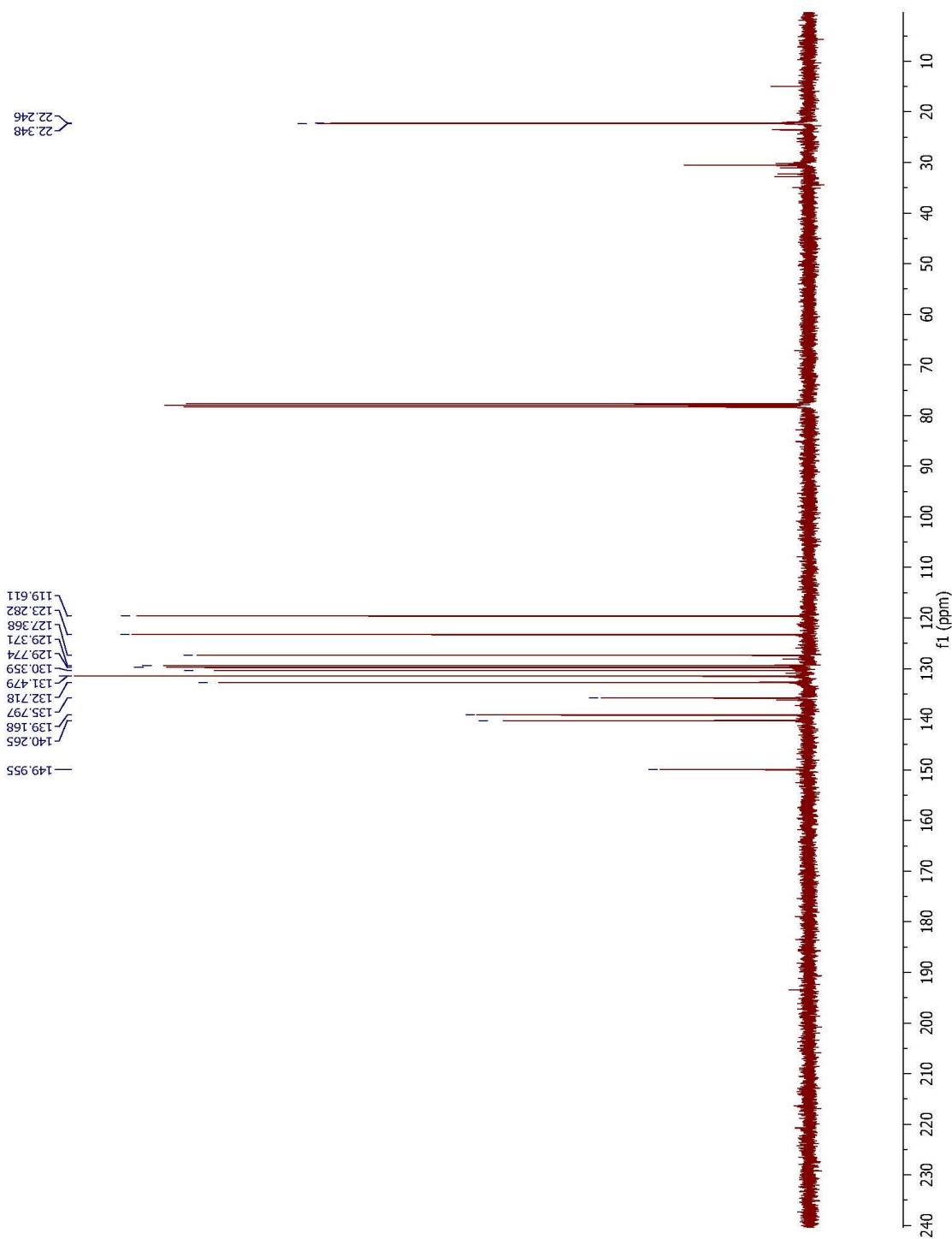


Fig. S18¹H NMR Spectrum of (Z)-N,1-di-*m*-tolylmethanime oxide in CDCl₃.



6

(*Z*)-1-(*p*-tolyl)-1-phenylmethanimine oxide. Brown crystal, mp. 104–110 °C. ^1H NMR (400 MHz, CDCl_3) δ 8.21 (dd, J = 6.6, 3.1 Hz, 1H), 7.73 (s, 1H), 7.57 (dd, J = 6.5, 3.1 Hz, 1H), 7.26 (dd, J = 6.7, 4.0 Hz,

Fig.S19 ^{13}C NMR Spectrum of (*Z*)-*N*,1-di-*m*-tolylmethanimine oxide in CDCl_3 .

1H). ^{13}C NMR (101 MHz, CDCl_3) 149.82, 135.80, 131.91, 131.48, 130.83, 130.04, 129.52, 122.62, 78.40, 78.08, 77.76.

(*Z*)-1-phenyl-*N*-(*p*-tolyl)methanimine oxide. Brown solid, mp:104-110 °C. ^1H NMR (400 MHz, CDCl_3) δ 8.42 (dd, J = 6.7, 2.6 Hz, 1H), 7.93 (s, 1H), 7.69 (d, J = 8.3 Hz, 1H), 7.50 (dd, J = 5.2, 1.6

Hz, 2H), 7.39 – 7.25 (m, 1H), 2.44 (s, 1H). ^{13}C NMR (101 MHz, CDCl_3) 147.67, 141.07, 135.08, 131.70, 131.62, 130.52, 129.89, 129.50, 122.36, 78.24, 77.93, 77.61, 22.04.

(Z)-1-(*p*-chlorophenyl)-*N*-phenylmethanimine oxide. yellow solid, mp: 154–157 °C. ^1H NMR (400 MHz, CDCl_3) δ 8.39 (d, $J = 8.6$ Hz, 1H), 7.95 (s, 1H), 7.85 – 7.72 (m, 1H), 7.58 – 7.43 (m, 3H). ^{13}C NMR (101 MHz, CDCl_3) 148.88, 136.44, 133.61, 130.26, 130.18, 129.27, 129.14, 128.97, 121.72, 77.42, 77.10, 76.78.

(Z)-*N*-*m*-tolyl-1-(*p*-tolyl)methanimine oxide. Yellow oil. ^1H NMR (400 MHz, CDCl_3) δ 8.31 (d, $J = 8.6$ Hz, 1H), 7.85 (s, 1H), 7.57 (s, 1H), 7.51 (d, $J = 8.0$ Hz, 1H), 7.36 – 7.16 (m, 2H), 2.48 (s, 2H), 2.39 (s, 2H). ^{13}C NMR (101 MHz, CDCl_3) 149.70, 143.57, 140.20, 135.02, 131.39, 130.21, 129.72, 128.02, 126.11, 123.14, 119.47, 78.46, 78.14, 77.82, 22.22, 15.73.

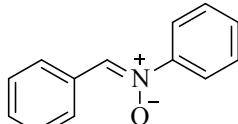
(Z)-1-(*p*-chlorophenyl)-*N*-(*m*-tolyl)methanimine oxide. Brown solid, mp: 77–82 °C. ^1H NMR (400 MHz, CDCl_3) δ 8.35 (d, $J = 8.6$ Hz, 2H), 7.89 (s, 1H), 7.57 (s, 1H), 7.51 (d, $J = 8.4$ Hz, 1H), 7.41 (d, $J = 8.7$ Hz, 2H), 7.33 (t, $J = 7.8$ Hz, 1H), 7.26 (d, $J = 7.6$ Hz, 1H), 2.41 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) 149.71, 140.34, 137.08, 134.28, 131.69, 131.07, 130.06, 129.81, 129.71, 123.18, 119.51, 78.37, 78.05, 77.73, 22.22.

(Z)-1-phenyl-*N*-(*m*-tolyl)methanimine oxide. Brown oil. ^1H NMR (400 MHz, CDCl_3) δ 8.39 (dd, $J = 6.6, 3.2$ Hz, 2H), 7.90 (s, 1H), 7.60 (s, 1H), 7.54 (d, $J = 7.9$ Hz, 1H), 7.47 (dd, $J = 5.1, 1.8$ Hz, 3H), 7.35 (t, $J = 7.8$ Hz, 1H), 7.26 (t, $J = 3.8$ Hz, 1H), 2.43 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) 149.92, 140.33, 135.72, 131.84, 131.57, 131.52, 129.99, 129.80, 129.52, 123.33, 119.66, 78.24, 77.92, 77.61, 22.24.

(Z)-1-(*p*-(methylthio)phenyl)-*N*-phenylmethanimine oxide. Yellow crystal, mp: 147–153 °C. ^1H NMR (400 MHz, CDCl_3) δ 8.35 (d, $J = 8.5$ Hz, 2H), 7.90 (s, 1H), 7.79 (dd, $J = 8.0, 1.8$ Hz, 2H), 7.49 (dd, $J = 7.2, 0.7$ Hz, 3H), 7.34 – 7.31 (m, 2H), 2.55 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) 149.81, 143.65, 134.98, 130.68, 130.17, 130.01, 128.03, 126.23, 122.52, 78.24, 77.92, 77.61, 15.81.

(Z)-*N*,1-di-*m*-tolylmethanimine oxide. Brown oil. ^1H NMR (400 MHz, CDCl_3) δ 8.33 (s, 1H), 8.15 (d, $J = 7.8$ Hz, 1H), 7.89 (s, 1H), 7.62 (s, 1H), 7.56 (d, $J = 7.9$ Hz, 1H), 7.37 (dt, $J = 10.0, 7.8$ Hz, 2H), 7.29 (dd, $J = 11.4, 7.4$ Hz, 2H), 2.44 (d, $J = 3.4$ Hz, 6H). ^{13}C NMR (101 MHz, CDCl_3) 149.96, 140.27, 139.17, 135.80, 132.72, 131.48, 130.36, 129.77, 129.37, 127.37, 123.28, 119.61, 22.35, 22.25.

7.Table S1 CHNS analysis of nitrone compounds

Entry	Nitronate	%C	%N	%H	%S
1		78.23	7.09	5.99	-

2		78.51	6.12	6.82	-
3		67.35	5.59	5.06	-
4		78.86	6.23	6.24	-
5		66.44	5.97	4.82	-
6		78.72	6.545	6.22	-
7		68.14	5.44	5.63	12.34
8		79.2	6.46	6.84	-

8.Table S2 CHNS analysis of the CdS catalysts.

catalyst	C%	N%	H%	S%	Organic compound%

CdS	2.1	1.42	0.51	22.18	4.03
Reused CdS	17.30	2.51	1.42	14.82	21.23
seperated CdS-PEG	3.84	0.63	0.173	20	4.64
Separated CdS-PEG after reaction	2.08	0.68	0.1	20.57	2.86