

## Supporting Information

### ***In-situ generated chiral iron complex as efficient catalyst for enantioselective sulfoxidation using aqueous H<sub>2</sub>O<sub>2</sub> as an oxidant***

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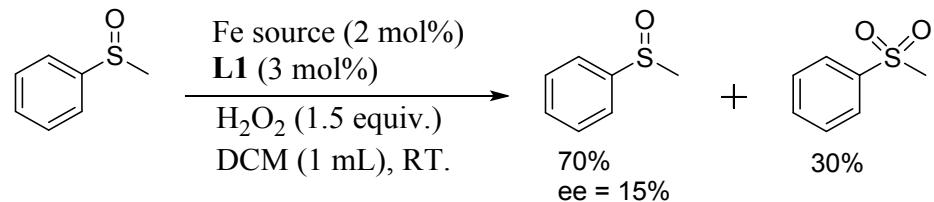
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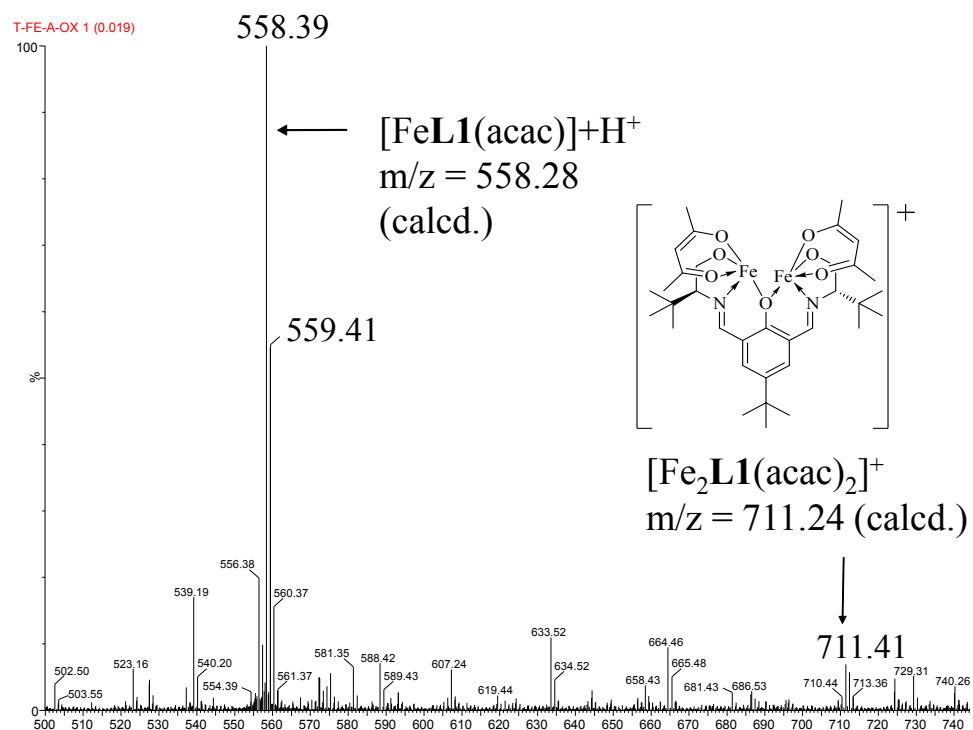
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## 1. Oxidative kinetic resolution of sulfoxide

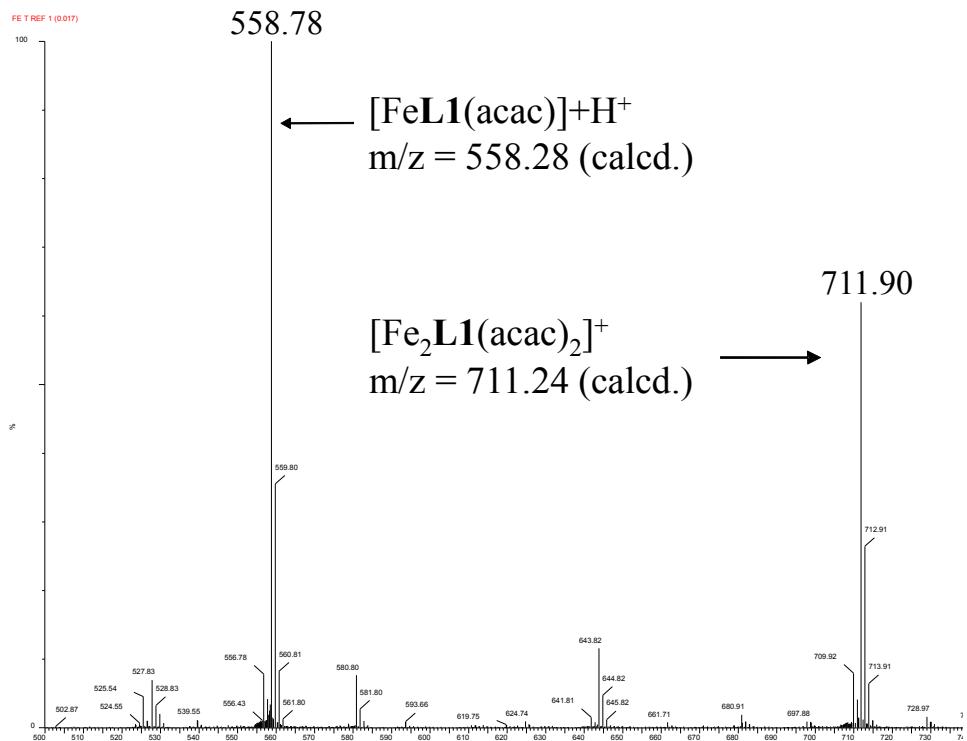
The oxidative kinetic resolution was carried out under the same reaction as mentioned for the asymmetric sulfoxidation taking phenyl methyl sulfoxide as model substrate. The catalyst resolve the racemic sulfoxide with 15% ee after 30% conversion of the starting sulfoxide.



## 2. ESI-MS spectra of the in situ generated complex



**Fig. S1** ESI-MS spectra of the in situ generated complex with 1:1 metal to ligand ratio.



**Fig. S2** ESI-MS spectra of the *in situ* generated complex with 2:1 metal to ligand ratio.

### 3. HRMS spectra of the in situ generated complexes

Elemental Composition Report

Page 1

#### Single Mass Analysis

Tolerance = 50.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

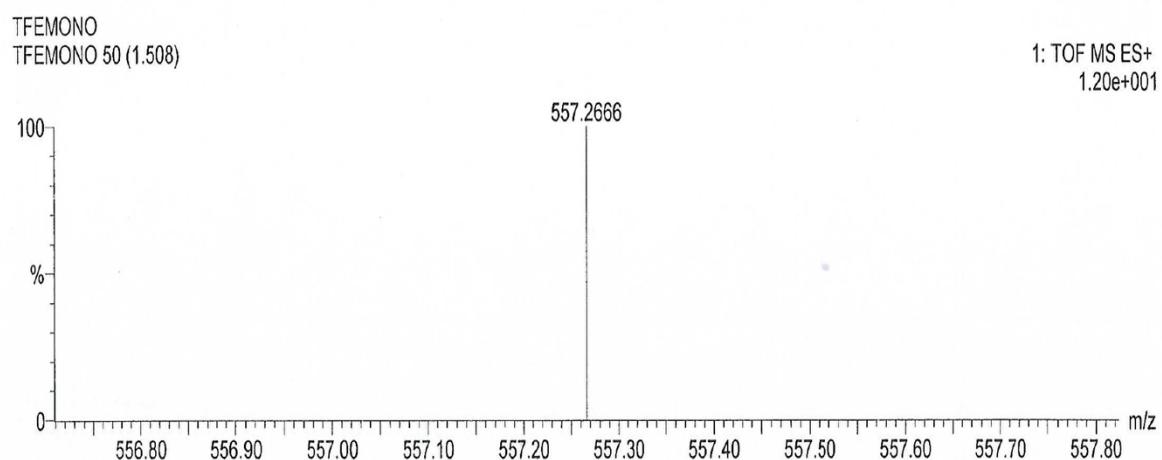
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

687 formula(e) evaluated with 10 results within limits (up to 50 closest results for each mass)

Elements Used:

C: 0-30 H: 0-46 N: 0-2 O: 0-5 Al: 0-1 P: 0-1 Fe: 0-1 I: 0-1



Minimum: -1.5  
Maximum: 5.0 50.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
557.2666	557.2678	-1.2	-2.2	8.5	n/a	C29 H45 N2 O5 Fe
	557.2622	4.4	7.9	7.5	n/a	C28 H46 N2 O4 Al Fe
	557.2725	-5.9	-10.6	10.5	n/a	C29 H43 N2 O5 Al P
	557.2604	6.2	11.1	5.5	n/a	C27 H46 N2 O2 I
	557.2595	7.1	12.7	8.5	n/a	C29 H46 N2 O3 P Fe
	557.2510	15.6	28.0	7.5	n/a	C29 H46 O5 Al Fe
	557.2492	17.4	31.2	5.5	n/a	C28 H46 O3 I
	557.2483	18.3	32.8	8.5	n/a	C30 H46 O4 P Fe
	557.2452	21.4	38.4	1.5	n/a	C23 H46 N2 O5 I
	557.2393	27.3	49.0	10.5	n/a	C30 H42 N2 I

**Fig. S3** HRMS spectra of [FeL1(acac)]

**Single Mass Analysis**

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Element prediction: Off

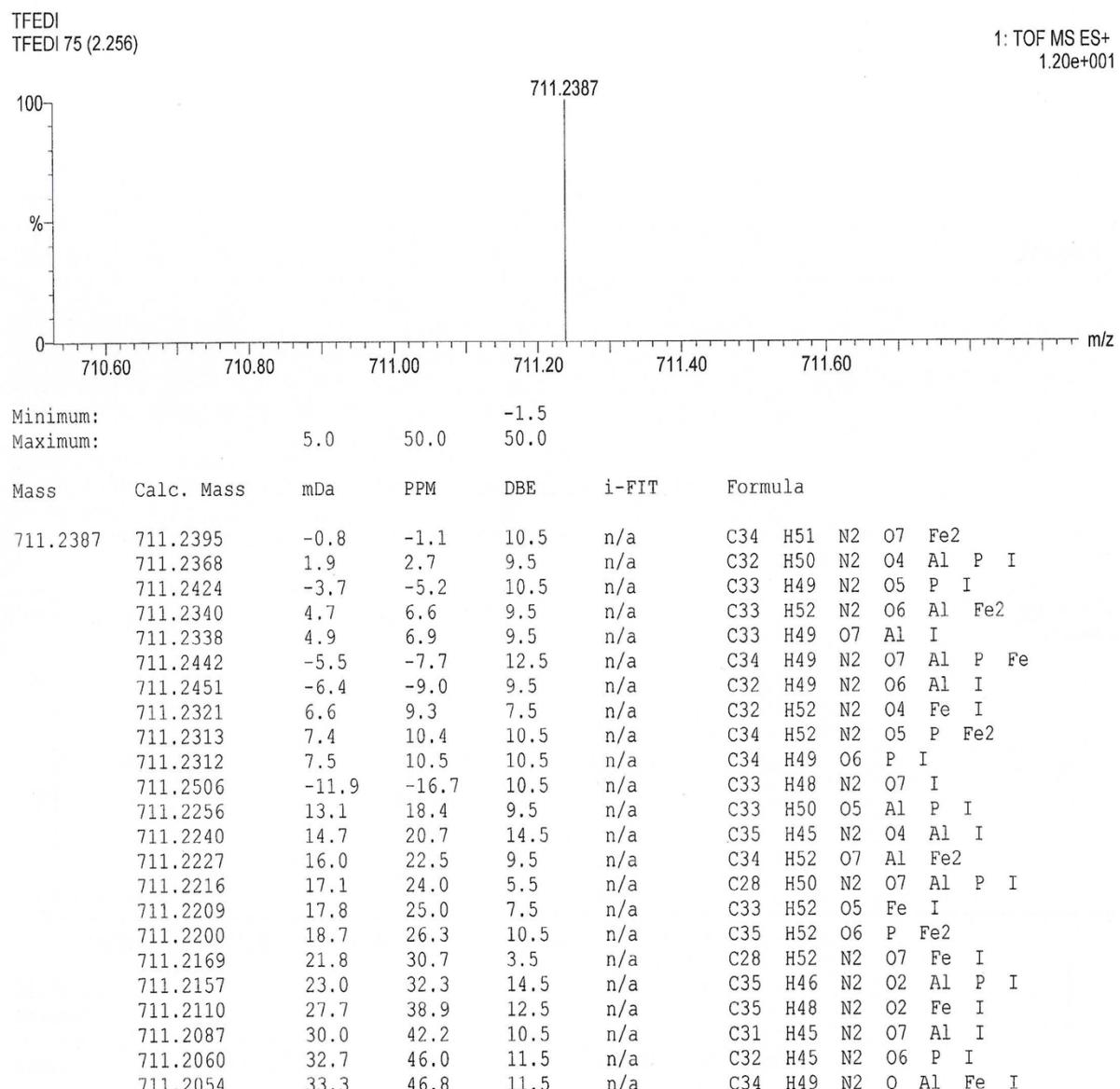
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

1146 formula(e) evaluated with 23 results within limits (up to 50 closest results for each mass)

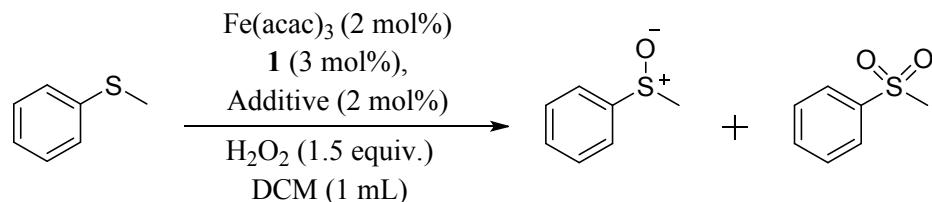
Elements Used:

C: 0-35 H: 0-52 N: 0-2 O: 0-7 Al: 0-1 P: 0-1 Fe: 0-2 I: 0-1

**Fig. S4** HRMS spectra of  $[\text{Fe}_2\text{L1}(\text{acac})_2]^+$

#### 4. Screening of benzoic acid derivatives as additive

**Table S1.** Screening of benzoic acid derivatives as additive for the enantioselective oxidation of methyl phenyl sulfide with **1**/Fe(acac)<sub>3</sub> system<sup>a</sup>



Entry	Additive	Conversion <sup>b</sup> (%)	Selectivity <sup>b</sup> (%)	ee <sup>c</sup> (%)
1	<i>p</i> -OHC <sub>6</sub> H <sub>4</sub> COOH	89	95	83
2	<i>p</i> -OMeC <sub>6</sub> H <sub>4</sub> COOH	91	95	88
3	<i>p</i> -MeC <sub>6</sub> H <sub>4</sub> COOH	87	96	74
4	<i>o</i> -OmeC <sub>6</sub> H <sub>4</sub> COOH	87	95	81
5	<i>p</i> -NH <sub>2</sub> C <sub>6</sub> H <sub>4</sub> COOH	90	94	87
6	<i>p</i> -OmeC <sub>6</sub> H <sub>4</sub> COONa	90	94	88

<sup>a</sup> Reaction condition: methyl phenyl sulfide (0.25 mmol), Fe(acac)<sub>3</sub> (2 mol%), **L1** (3 mol%), additive (2 mol%), aqueous H<sub>2</sub>O<sub>2</sub> (30%, 1.2 equiv.), in organic solvent (1 ml) at 15 °C for 12 h. <sup>b</sup> Conversion and selectivity were calculated by <sup>1</sup>H NMR analysis. <sup>c</sup> Enantiomeric excess were determined by HPLC analysis on a chiral phase Daicel Chiralcel OD column.

## 5. Characterization data of the sulfoxides

**Phenyl methyl sulfoxide<sup>1</sup>:** Colourless oil; Yield: 86%; ee: 88%; <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ = 7.66-7.64 (m, 2H), 7.53-7.48 (m, 3H), 2.71 (s, 3H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>): δ = 145.21, 130.71, 129.02, 123.14, 43.50; The enantiomeric excess was determined by HPLC analysis. HPLC condition: Daicel Chiralcel OD column, 80:20 Hex/IPA, 0.5 mL/min, 30 °C, 254 nm; t<sub>r</sub> (**R**) = 13.7 min, t<sub>r</sub> (**S**) = 15.4 min.

**4-Fluorophenyl methyl sulfoxide<sup>2</sup>:** Colourless oil; Yield: 76%; ee: 95%; <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ = 7.69-7.66 (m, 2H), 7.25-7.22 (m, 2H), 2.73 (s, 3H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>): δ = 165.19, 163.19, 140.99, 125.83, 125.76, 116.67, 116.50, 43.99; The enantiomeric excess was determined by HPLC analysis. HPLC condition: Daicel Chiralcel OD column, 92:08 Hex/IPA, 0.4 mL/min, 30 °C, 254 nm; t<sub>r</sub> (**R**) = 31.9 min, t<sub>r</sub> (**S**) = 33.7 min.

**4-Chlorophenyl methyl sulfoxide<sup>1</sup>:** Colourless oil; Yield: 80%; ee: 95%; <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ = 7.61 (d, J = 8.5 Hz, 2H), 7.51 (d, J = 8.5 Hz, 2H), 2.73 (s, 3H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>): δ = 144.00, 136.90, 129.39, 124.80, 43.74; The enantiomeric excess was determined by HPLC analysis. HPLC condition: Daicel Chiralcel OB column, 80:20 Hex/IPA, 0.7 mL/min, 30 °C, 254 nm; t<sub>r</sub> (**R**) = 11.4 min, t<sub>r</sub> (**S**) = 16.9 min.

**4-Bromophenyl methyl sulfoxide<sup>1</sup>:** White solid; Yield: 79%; ee: 92%; <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ = 7.67 (d, J = 8.5 Hz, 2H), 7.53 (d, J = 8.5 Hz, 2H), 2.73 (s, 3H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>): δ = 144.81, 132.63, 125.21, 43.99; The enantiomeric excess was determined by HPLC analysis. HPLC condition: Daicel Chiralcel OB column, 80:20 Hex/IPA, 0.8 mL/min, 30 °C, 254 nm; t<sub>r</sub> (**R**) = 10.8 min, t<sub>r</sub> (**S**) = 15.3 min.

**4-Nitrophenyl methyl sulfoxide<sup>1</sup>:** White solid; Yield: 69%; ee: 96%; <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ = 8.40 (d, J = 8.5 Hz, 2H), 7.86 (d, J = 8.5 Hz, 2H), 2.82 (s, 3H); <sup>13</sup>C NMR (125

MHz, CDCl<sub>3</sub>): δ = 153.29, 149.54, 124.76, 124.55, 43.92; The enantiomeric excess was determined by HPLC analysis. HPLC condition: Daicel Chiralcel OJ column, 65:35 Hex/IPA, 0.5 mL/min, 30 °C, 254 nm; t<sub>r</sub> (**R**) = 23.9 min, t<sub>r</sub> (**S**) = 27.4 min.

**4-Methylphenyl methyl sulfoxide<sup>1</sup>:** White solid; Yield: 86%; ee: 87%; <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ = 7.54 (d, J = 8.0 Hz, 2H), 7.32 (d, J = 8 Hz, 2H), 2.70 (s, 3H), 2.41 (s, 3H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>): δ = 142.27, 141.37, 129.90, 123.41, 43.79, 21.25; The enantiomeric excess was determined by HPLC analysis. HPLC condition: Daicel Chiralcel OD column, 94:06 Hex/IPA, 0.5 mL/min, 30 °C, 254 nm; t<sub>r</sub> (**R**) = 33.5 min, t<sub>r</sub> (**S**) = 36.4 min.

**4-Methoxyphenyl methyl sulfoxide<sup>1</sup>:** Yellow oil; Yield: 86%; ee: 85%; <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ 7.45 (d, J = 7.8 Hz, 1H), 6.88 (d, J = 7.6 Hz, 1H), 3.70 (s, 3H), 2.56 (s, 3H); <sup>13</sup>C NMR (50 MHz, CDCl<sub>3</sub>): δ = 161.52, 136.02, 125.04, 114.43, 55.09, 34.44; The enantiomeric excess was determined by HPLC analysis. HPLC condition: Daicel Chiralcel OD column, 90:10 Hex/IPA, 0.7 mL/min, 30 °C, 254 nm; t<sub>r</sub> (**R**) = 21.7 min, t<sub>r</sub> (**S**) = 23.3 min.

**3-Chlorophenyl methyl sulfoxide<sup>3</sup>:** Colourless oil; Yield: 77%; ee: 98%; <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ = 7.67 (s, 1H), 7.52-7.49 (m, 1H), 7.48-7.46(m, 2H), 2.75 (s, 3H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>): δ = 147.65, 135.54, 131.08, 130.53, 123.50, 121.56, 43.85; The enantiomeric excess was determined by HPLC analysis. HPLC condition: Daicel Chiralcel OB column, 90:10 Hex/IPA, 1.0 mL/min, 30 °C, 254 nm; t<sub>r</sub> (**R**) = 12.5 min, t<sub>r</sub> (**S**) = 18.8 min.

**3-Bromophenyl methyl sulfoxide<sup>4</sup>:** Colorless oil; Yield: 78%; ee: 92%; <sup>1</sup>H NMR (200 MHz, CDCl<sub>3</sub>): δ = 7.82 (s, 1H), 7.65-7.53 (m, 2H), 7.47-7.32 (m, 1H), 2.75 (s, 3H); <sup>13</sup>C NMR (50 MHz, CDCl<sub>3</sub>): δ = 147.91, 134.06, 130.83, 126.41, 123.53, 122.08, 43.97; The enantiomeric excess was determined by HPLC analysis. HPLC condition: Daicel Chiralcel OB column, 80:20 Hex/IPA, 1.0 mL/min, 30 °C, 254 nm; t<sub>r</sub> (**R**) = 8.67 min, t<sub>r</sub> (**S**) = 13.9 min.

**2-Chlorophenyl methyl sulfoxide<sup>3</sup>:** Colourless oil; Yield: 76%; ee: 91%; <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ = 7.96 (d, *J* = 8 Hz, 1H), 7.54 (t, *J* = 7.5 Hz, 1H), 7.45 (t, *J* = 7.5 Hz, 1H), 7.40 (d, *J* = 8 Hz, 1H), 2.83 (s, 3H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>): δ = 143.63, 132.06, 129.84, 128.23, 125.36, 41.72; The enantiomeric excess was determined by HPLC analysis. HPLC condition: Daicel Chiralcel AD-H column, 90:10 Hex/IPA, 0.5 mL/min, 30 °C, 254 nm; t<sub>r</sub> (**R**) = 17.9 min, t<sub>r</sub> (**S**) = 21.7 min.

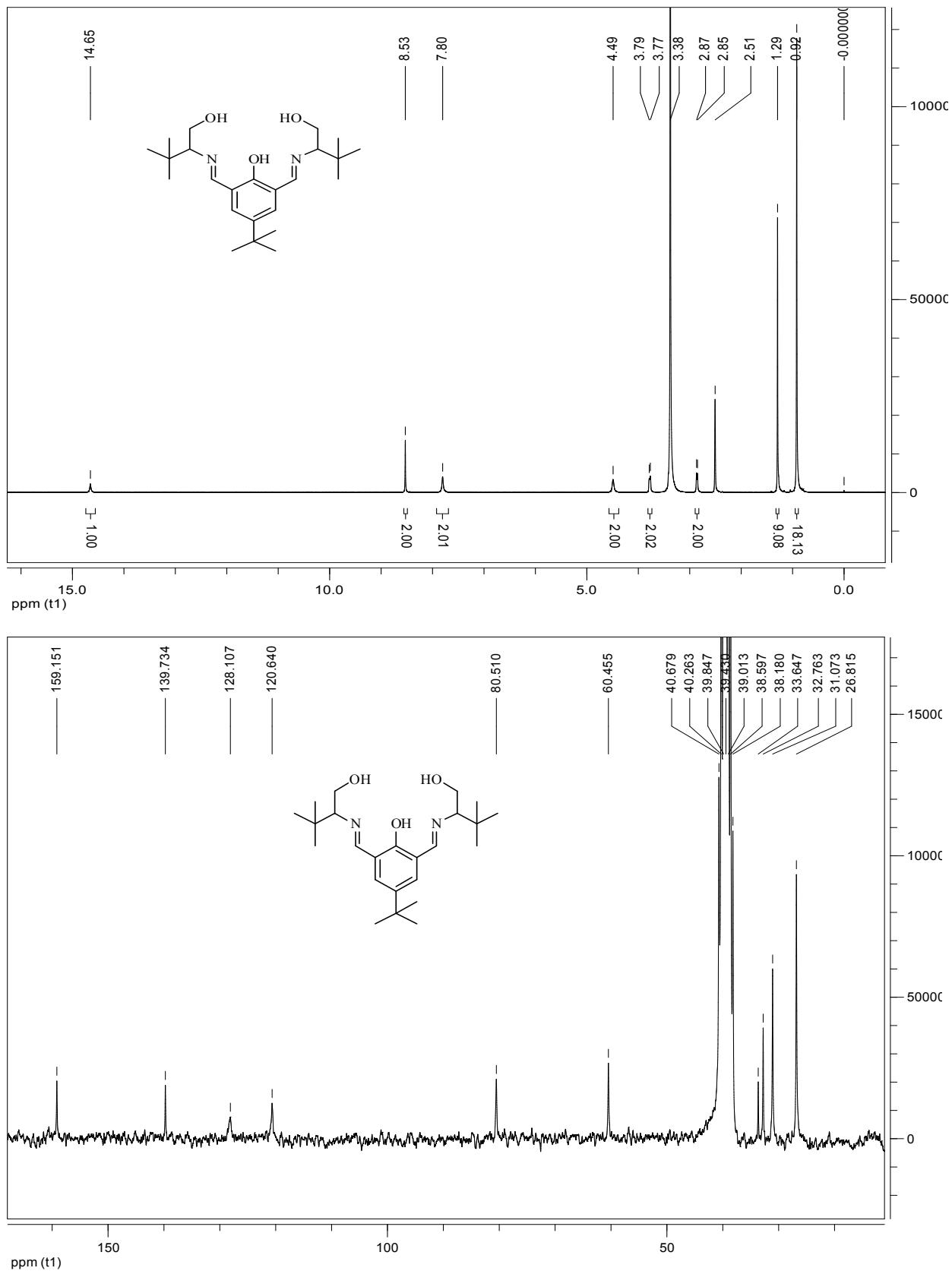
**2-Bromophenyl methyl sulfoxide<sup>1</sup>:** Yellow oil; Yield: 73%; ee: 91%; <sup>1</sup>H NMR (200 MHz, CDCl<sub>3</sub>): δ = 7.98-7.93 (m, 1H), 7.62-7.49 (m, 2H), 7.42-7.34 (m, 1H), 2.83 (s, 3H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>): δ = 145.29, 132.91, 132.26, 128.73, 125.69, 118.40, 41.84; The enantiomeric excess was determined by HPLC analysis. HPLC condition: Daicel Chiralcel AD-H column, 90:10 Hex/IPA, 0.5 mL/min, 30 °C, 254 nm; t<sub>r</sub> (**R**) = 15.2 min, t<sub>r</sub> (**S**) = 17.8 min.

**Ethyl phenyl sulfoxide<sup>1</sup>:** Colourless oil; Yield: 87%; ee: 85%; <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ = 7.62-7.60 (m, 2H), 7.54-7.49 (m, 3H), 2.95-2.87 (m, 1H), 2.81-2.74 (m, 1H), 1.20 (t, *J* = 7.5, 3H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>): δ = 143.20, 130.94, 129.14, 124.17, 50.24, 5.95; The enantiomeric excess was determined by HPLC analysis. HPLC condition: Daicel Chiralcel OD column, 90:10 Hex/IPA, 0.5 mL/min, 30 °C, 254 nm; t<sub>r</sub> (**R**) = 17.6 min, t<sub>r</sub> (**S**) = 20.9 min.

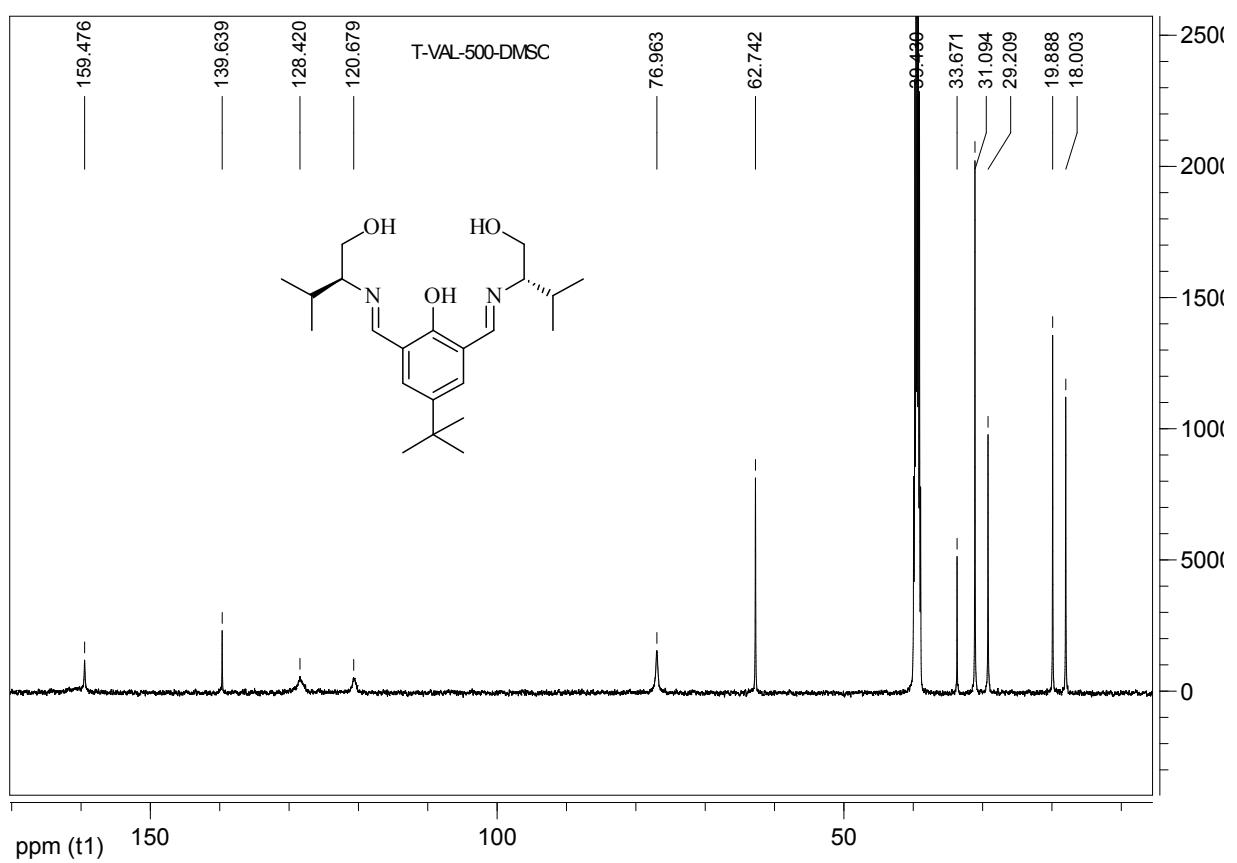
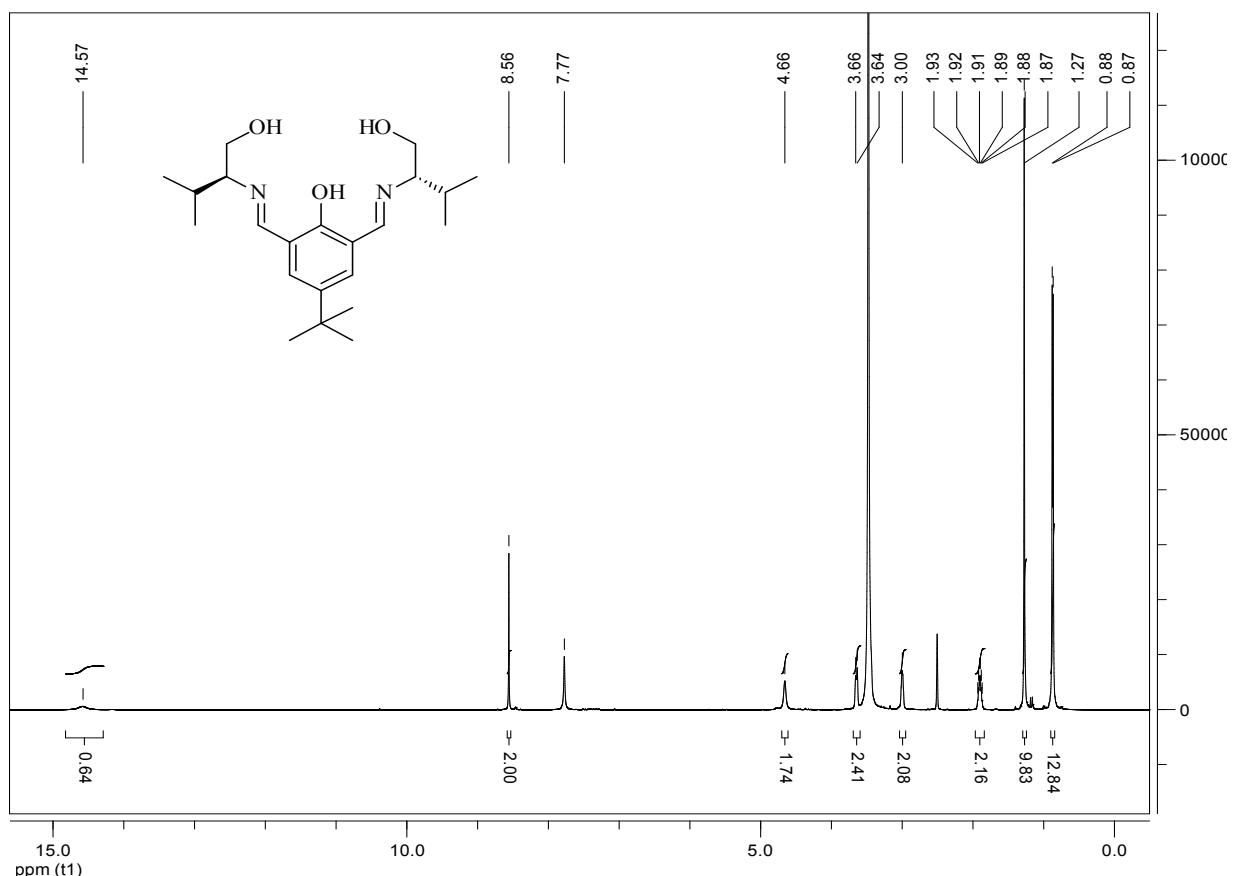
**Benzyl phenyl sulfoxide<sup>1</sup>:** White solid; Yield: 85%; ee: 85%; <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ = 7.46-7.37 (m, 5H), 7.29-7.23 (m, 3H), 6.98 (m, 2H), 4.12 (d, *J* = 12.5 Hz, 1H), 4.00 (d, *J* = 12.5 Hz, 1H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>): δ = 142.63, 131.16, 130.33, 129.06, 128.83, 128.42, 128.23, 124.43, 63.52; The enantiomeric excess was determined by HPLC analysis. HPLC condition: Daicel Chiralcel OD column, 90:10 Hex/IPA, 0.5 mL/min, 30 °C, 254 nm; t<sub>r</sub> (**R**) = 17.6 min, t<sub>r</sub> (**S**) = 20.9 min.

## 6. $^1\text{H}$ and $^{13}\text{C}$ -NMR spectra of ligands (L1-L4)

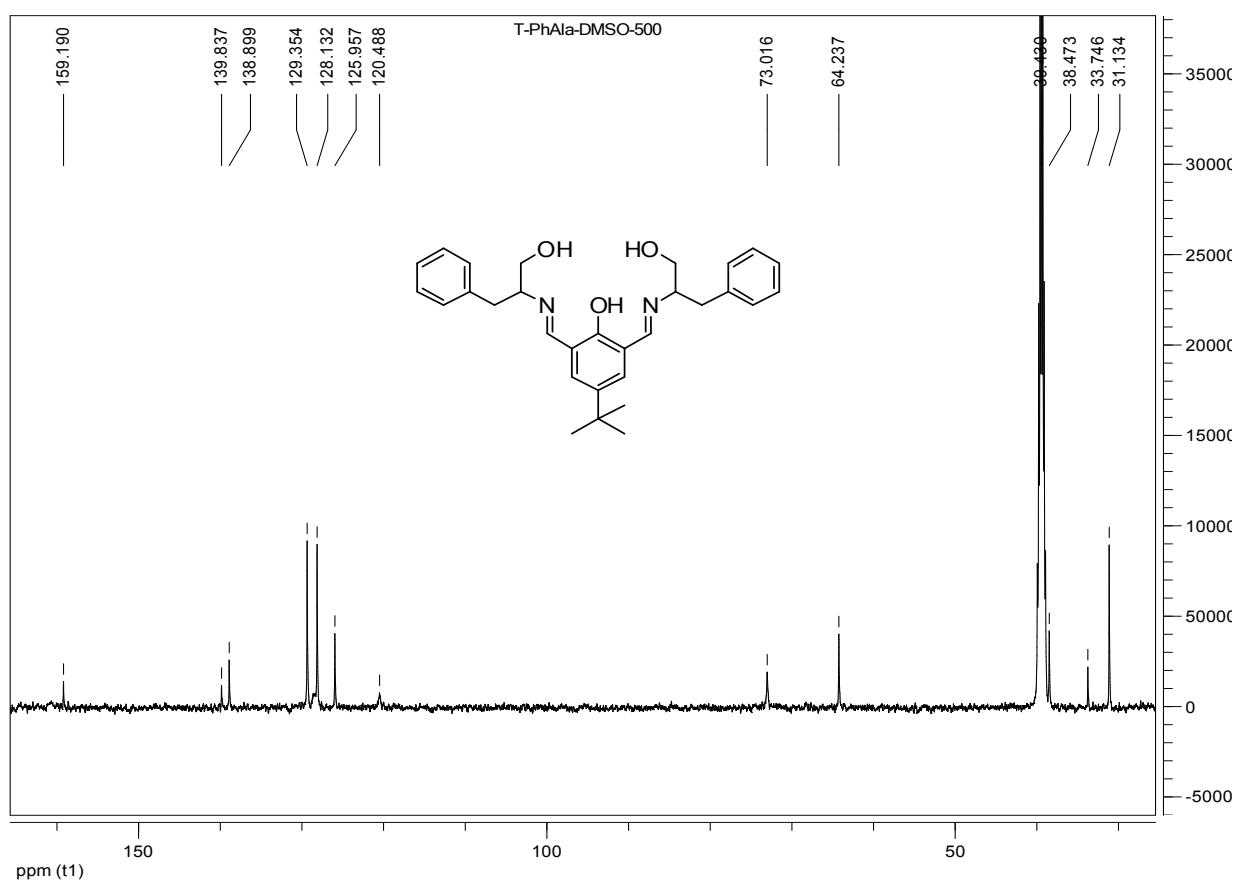
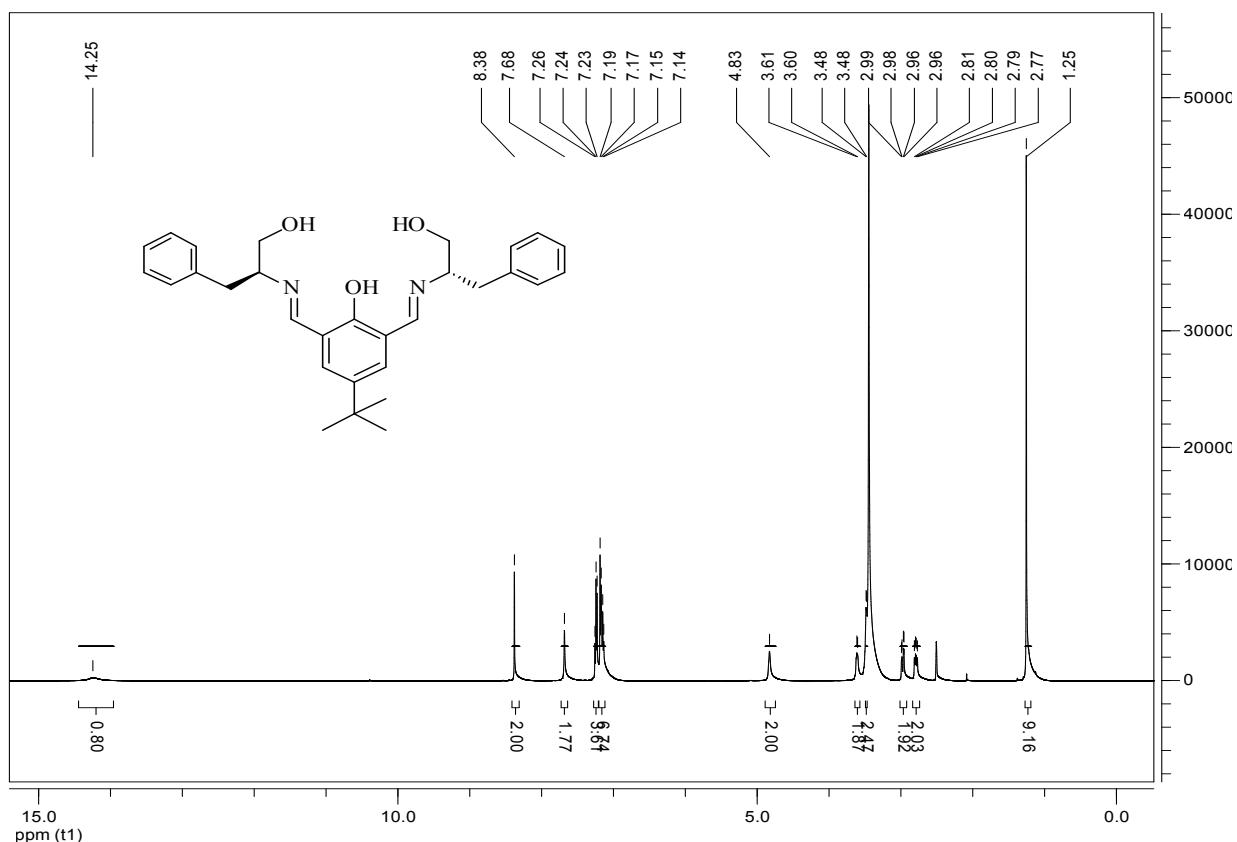
### Ligand L1



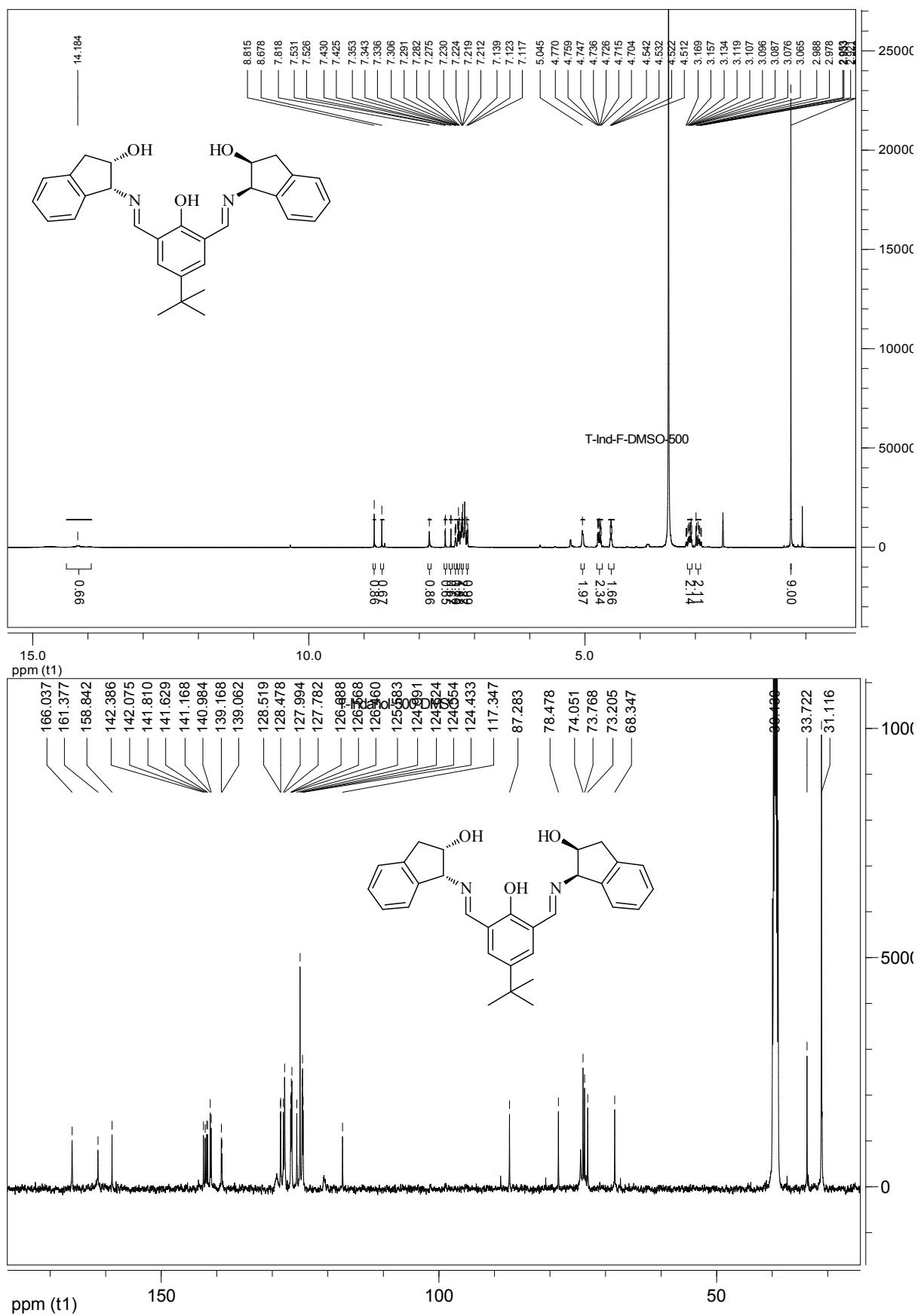
**Ligand L2:**



**Ligand L3:**

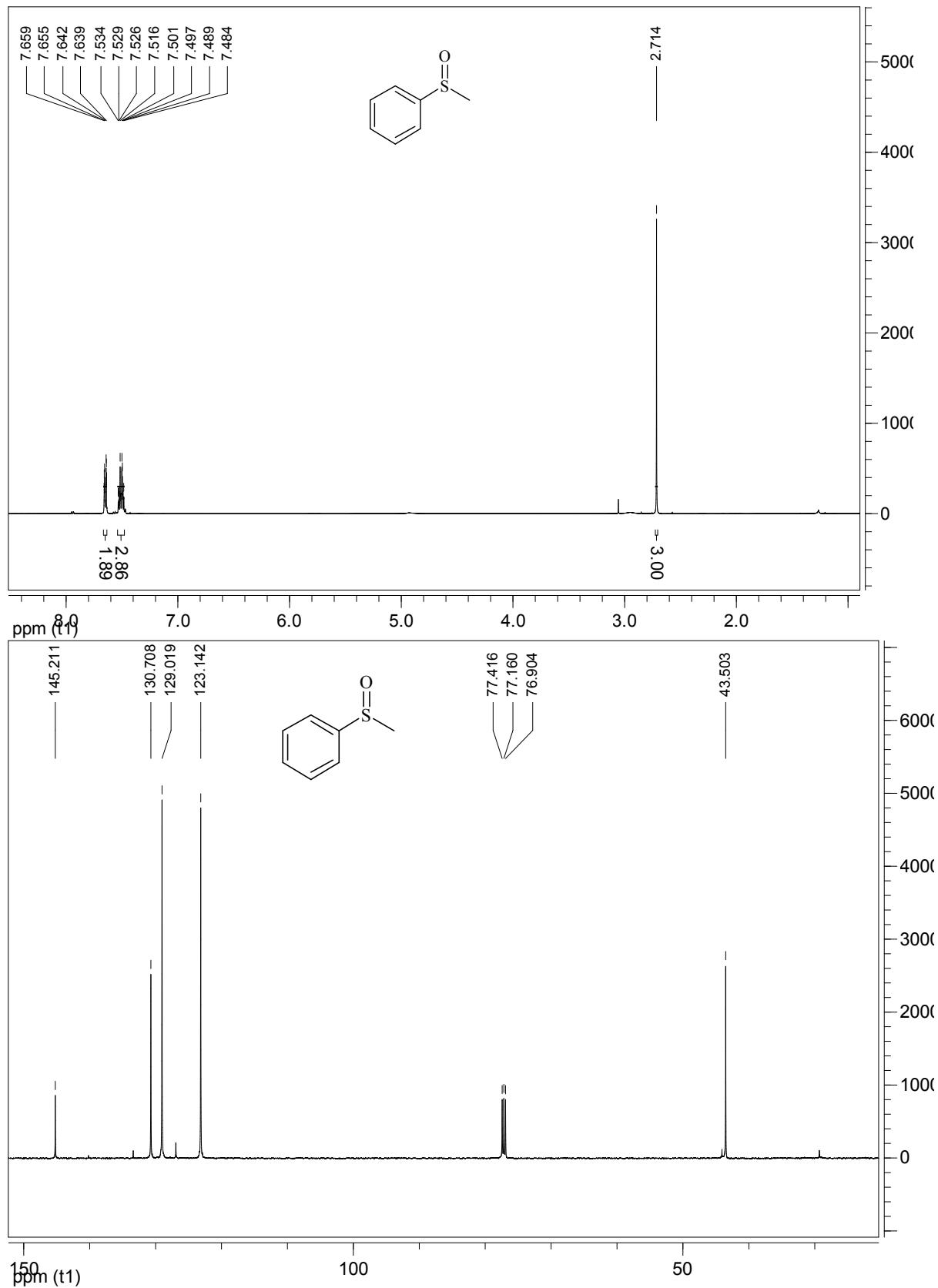


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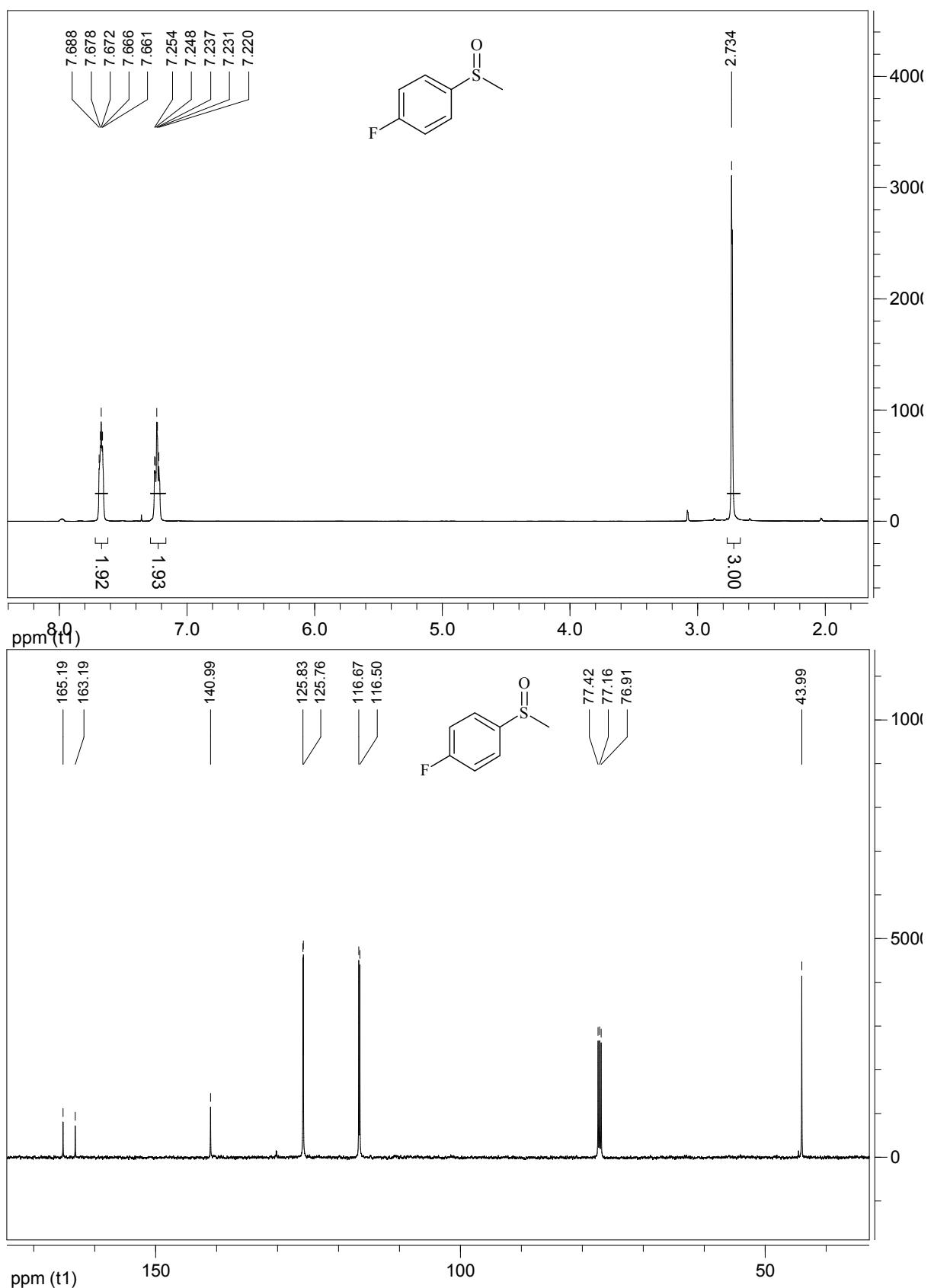


## 7. $^1\text{H}$ and $^{13}\text{C}$ -NMR spectra of sulfoxides

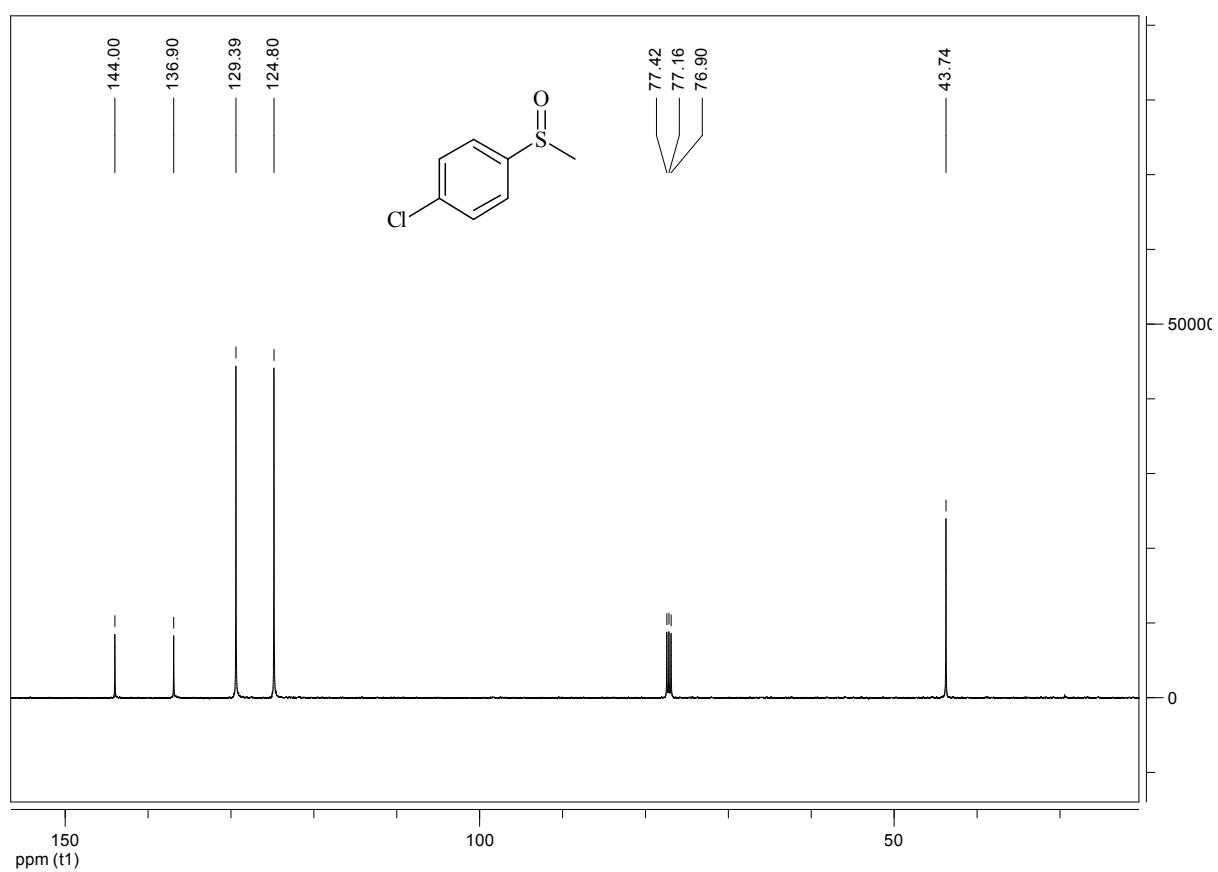
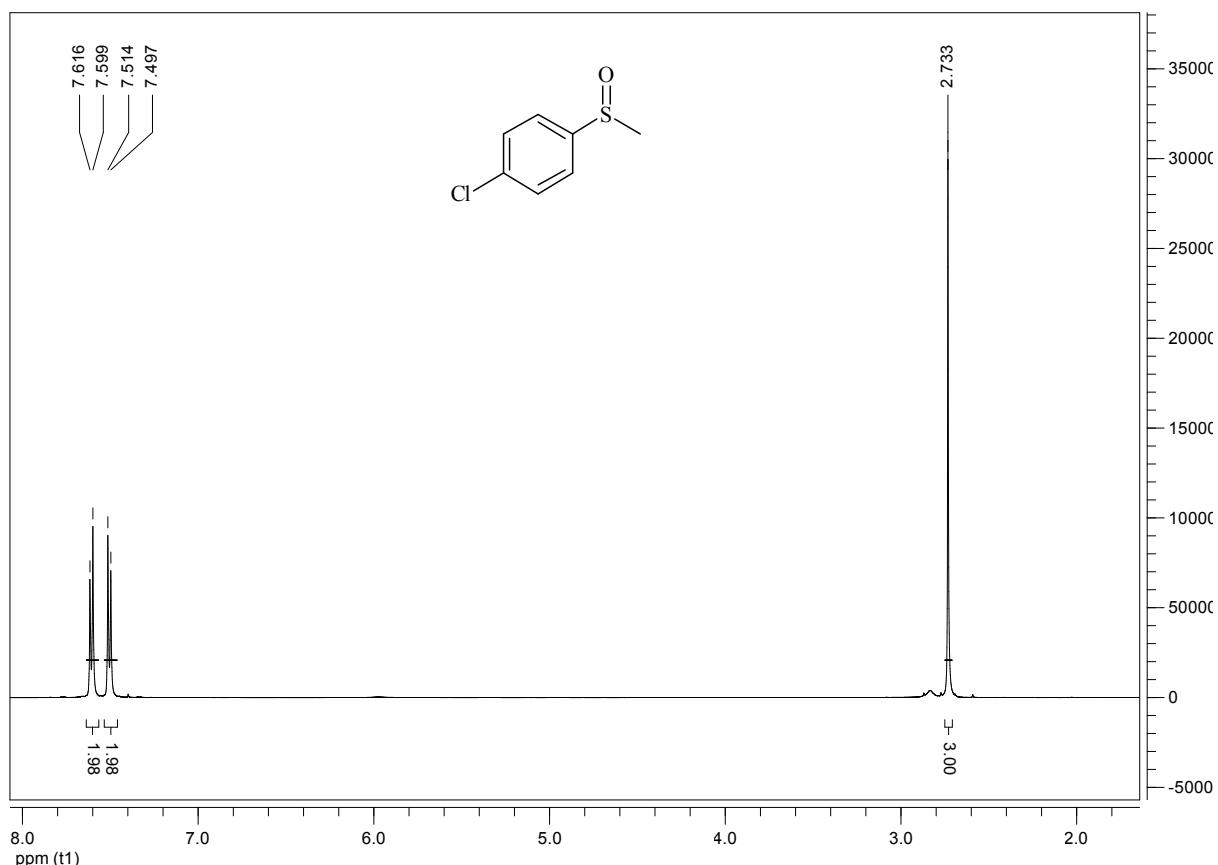
### Phenyl methyl sulfoxide



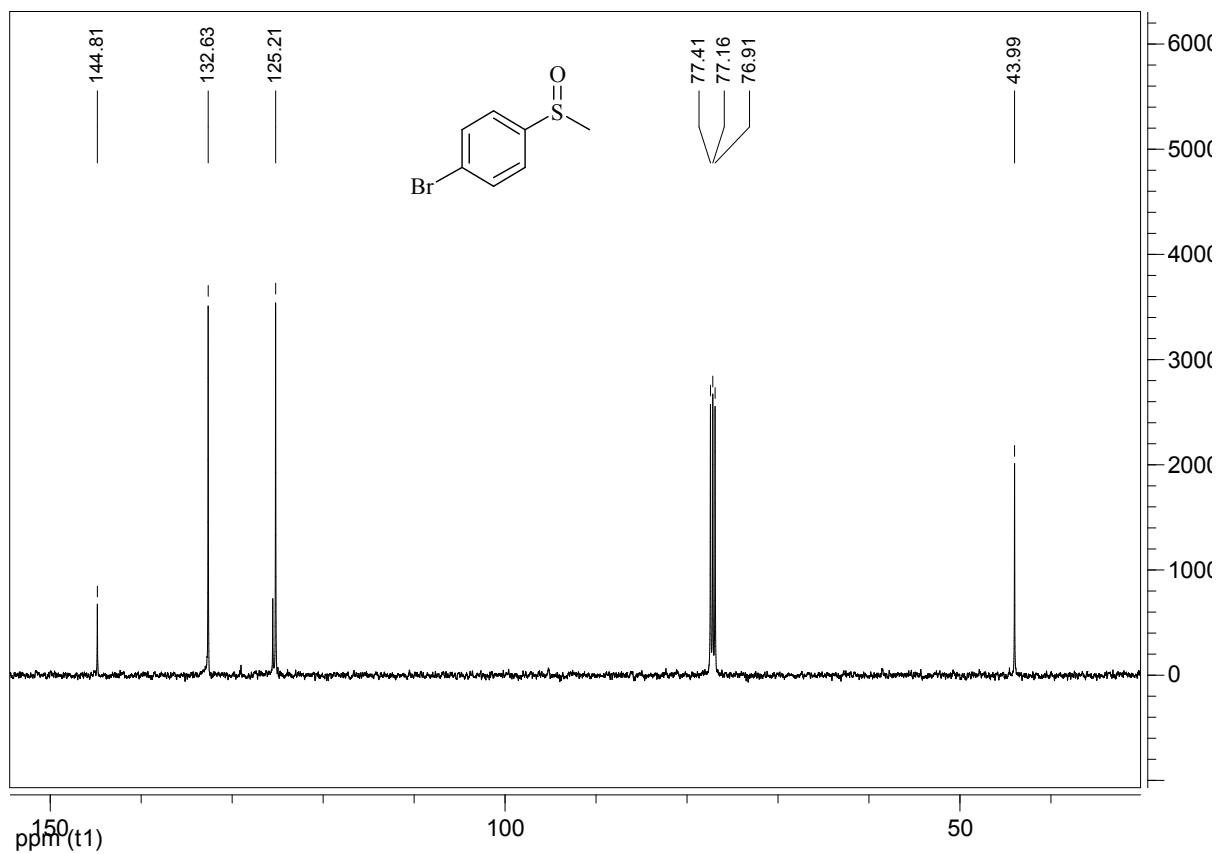
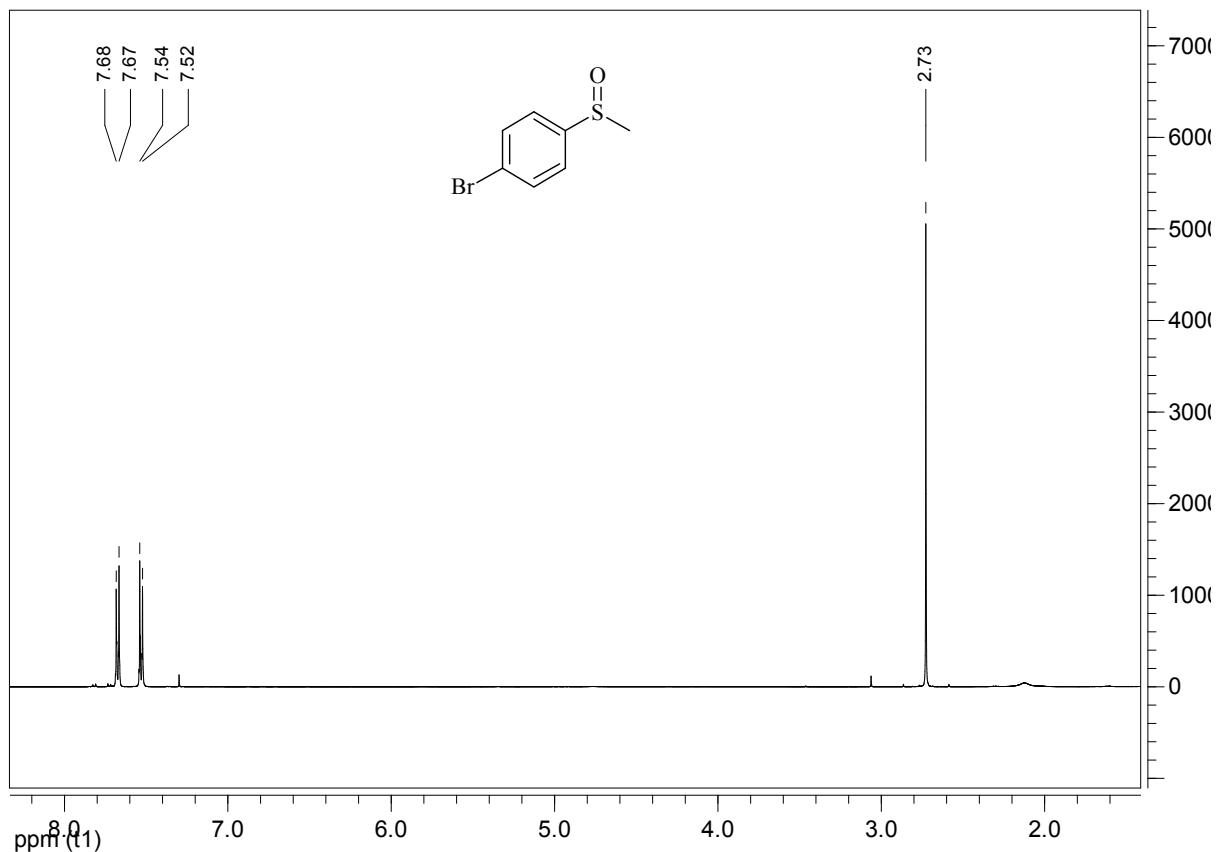
**4-Fluorophenyl methyl sulfoxide**



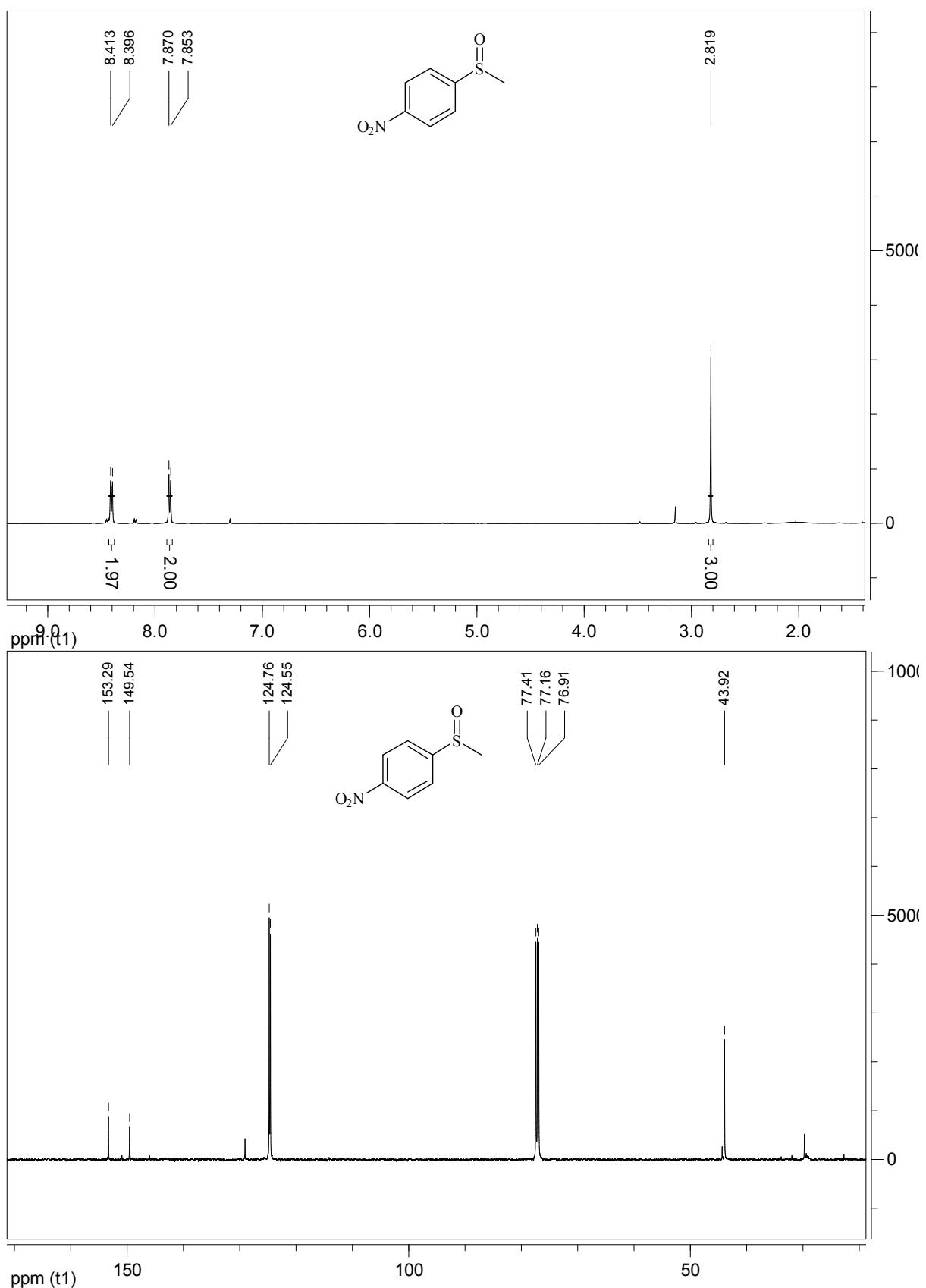
**4-Chlorophenyl methyl sulfoxide**



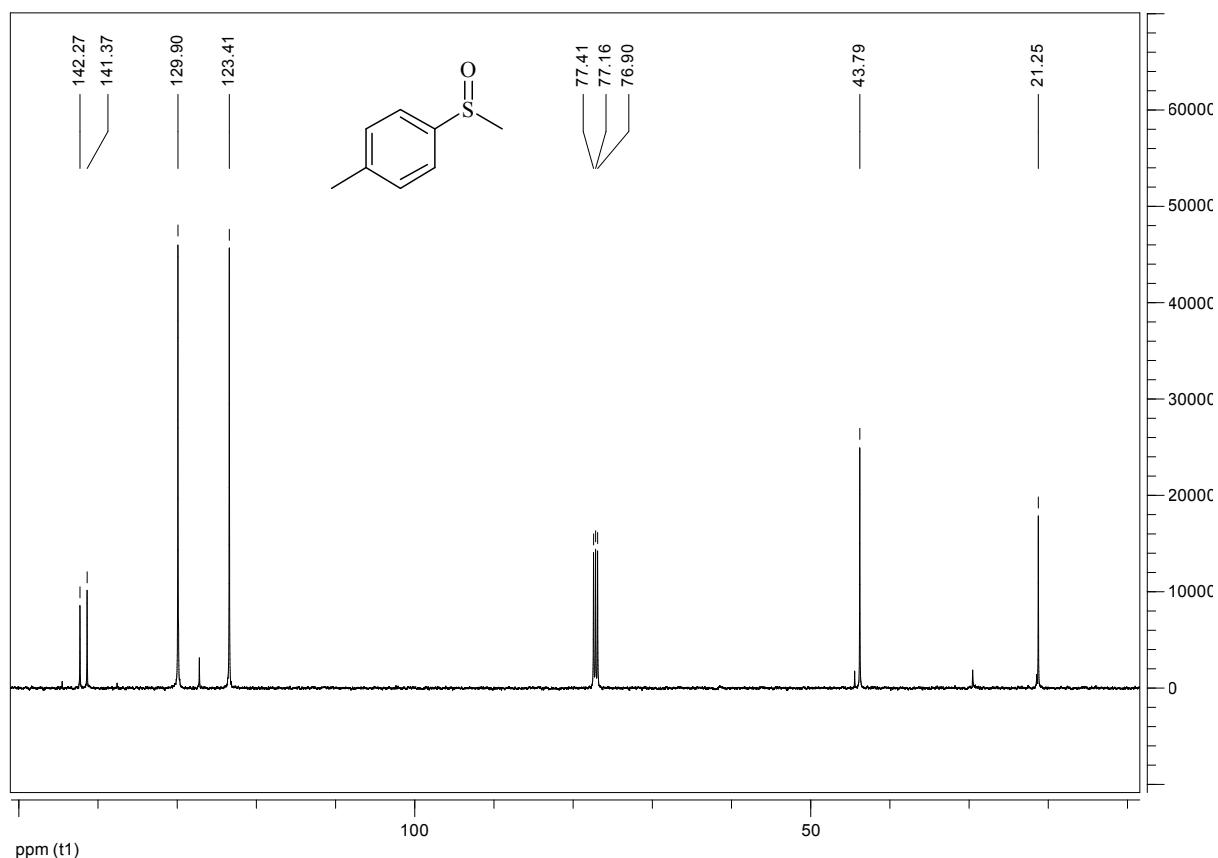
**4-Bromophenyl methyl sulfoxide**



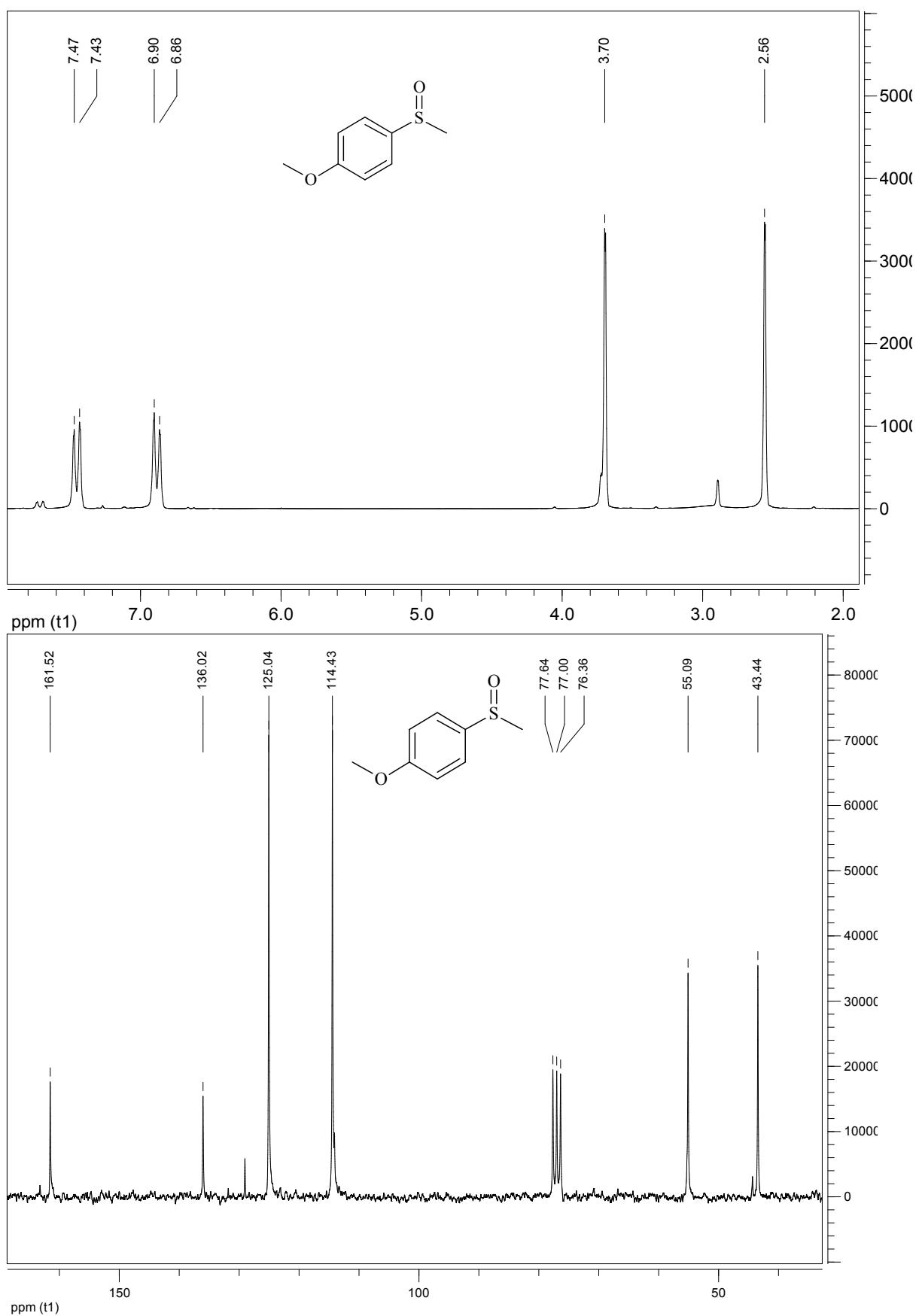
**4-Nitrophenyl methyl sulfoxide**



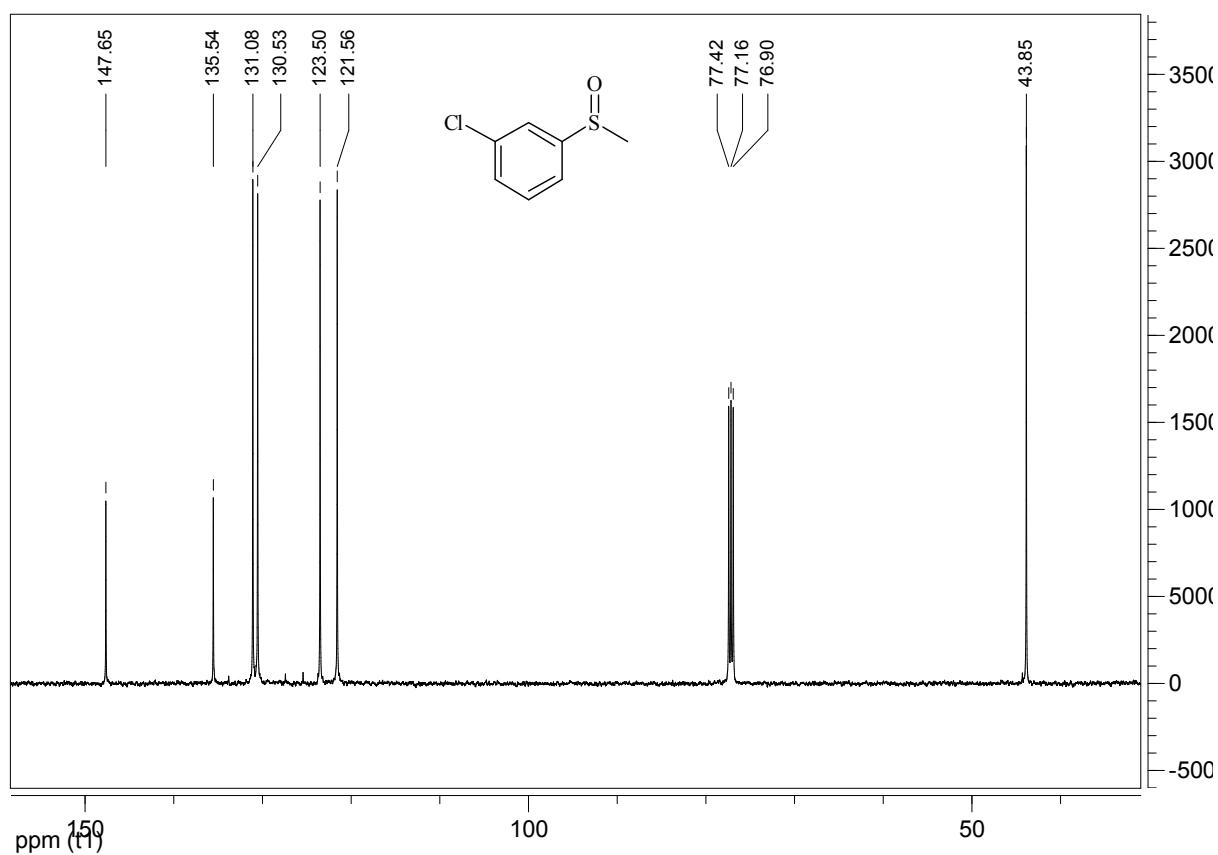
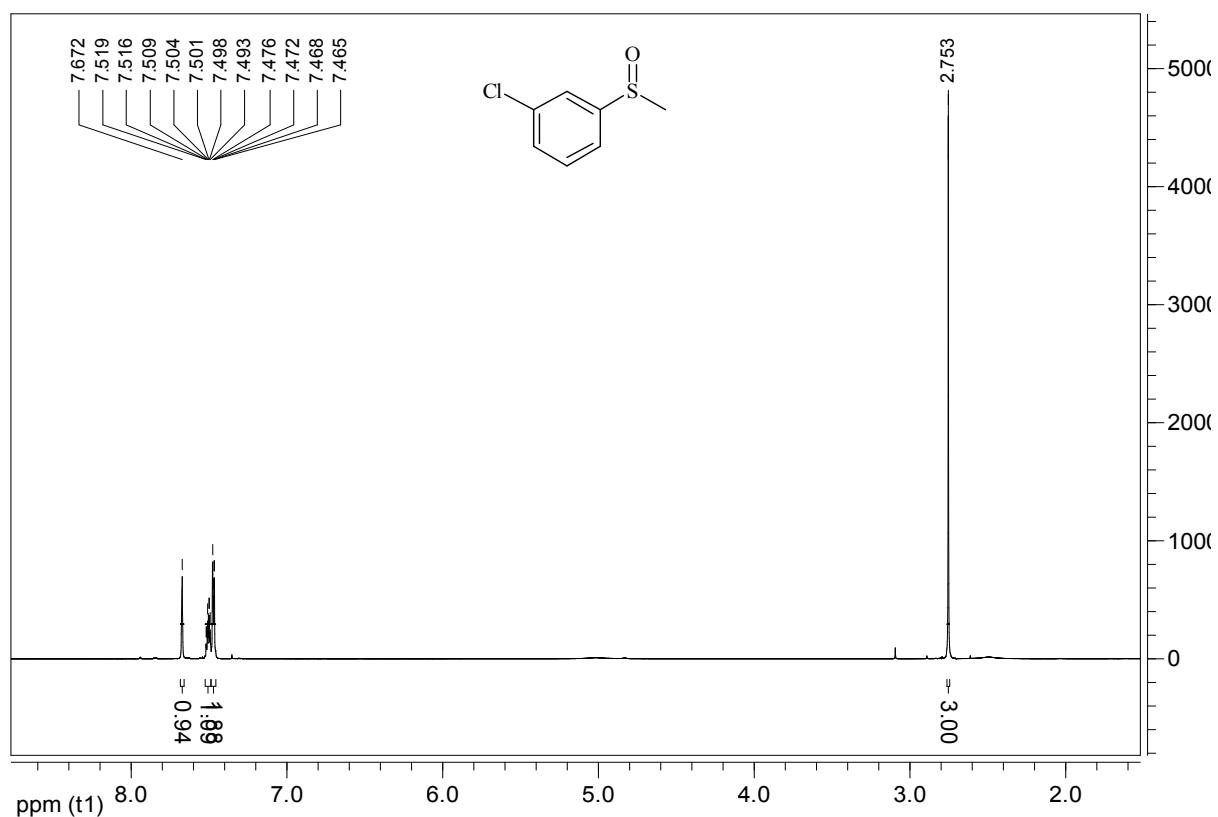
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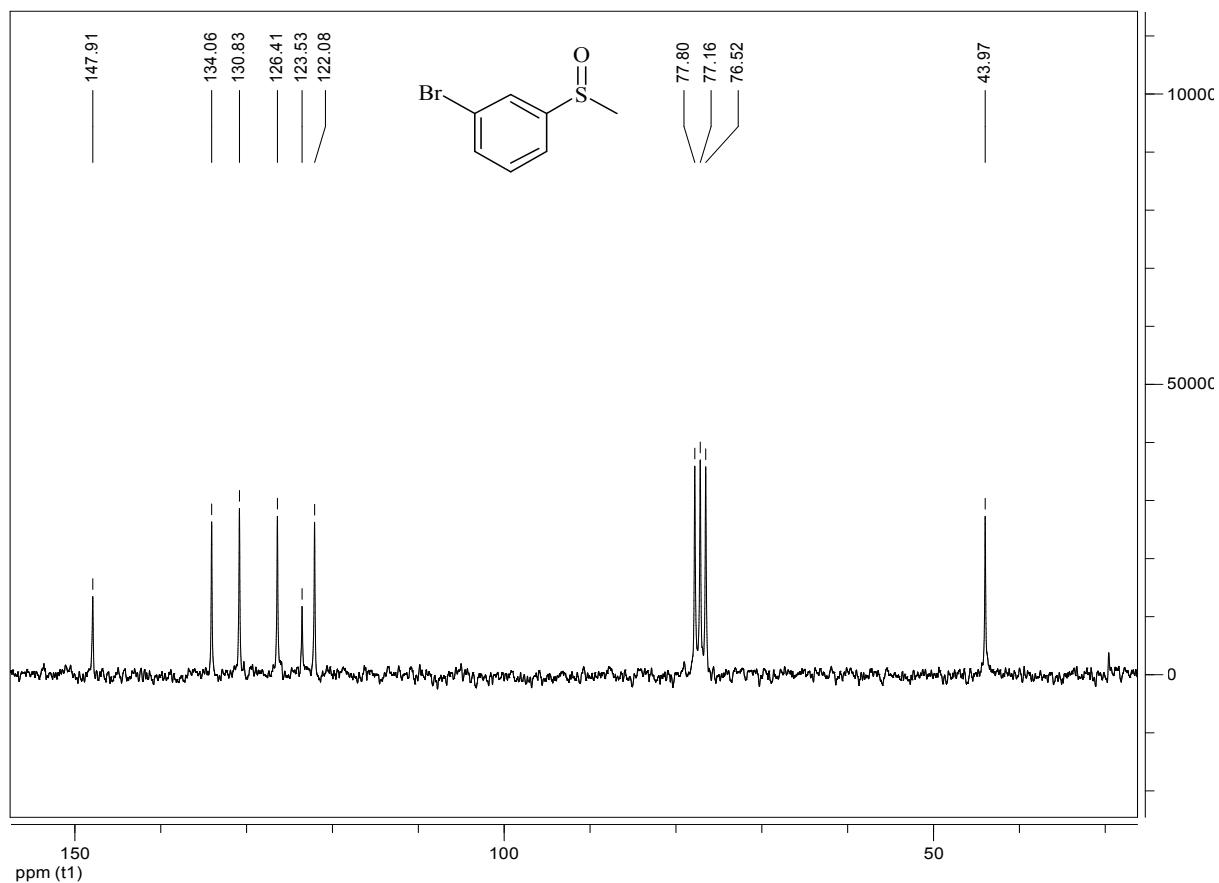
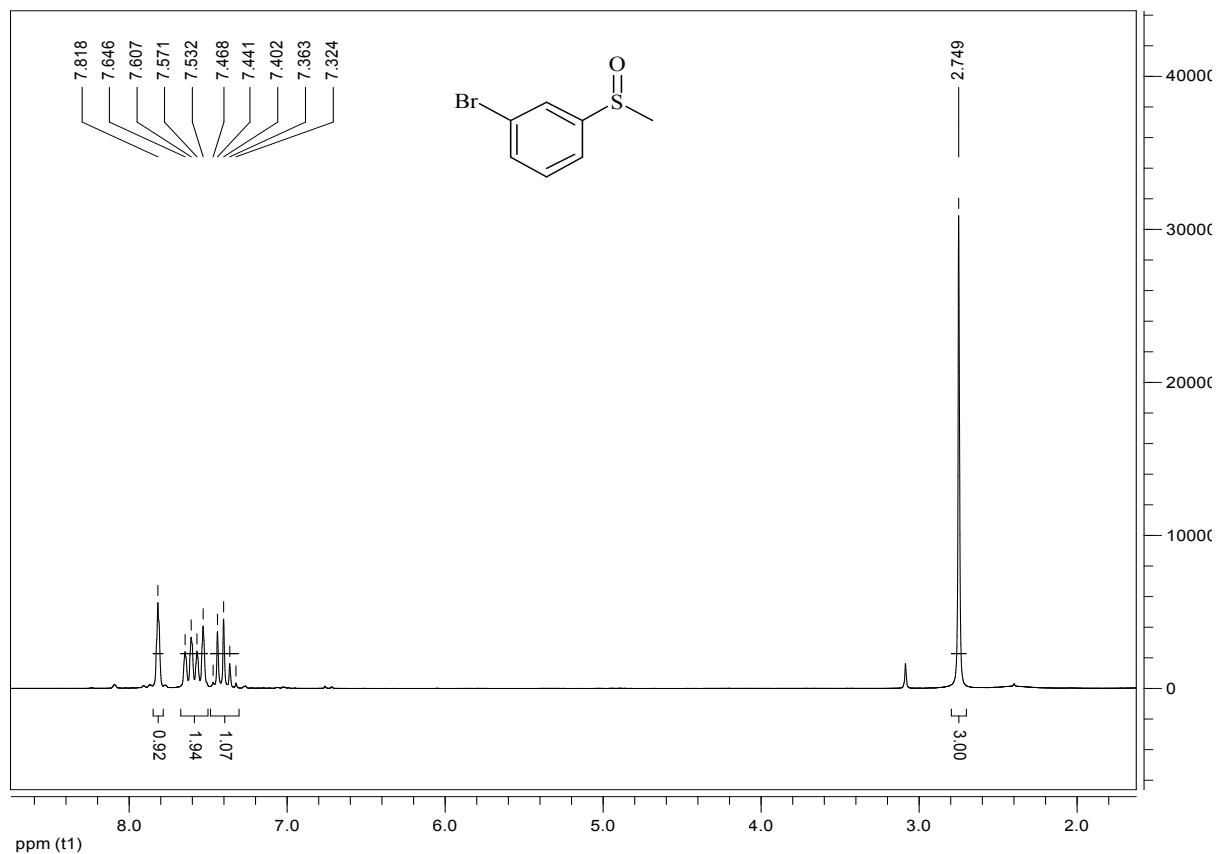
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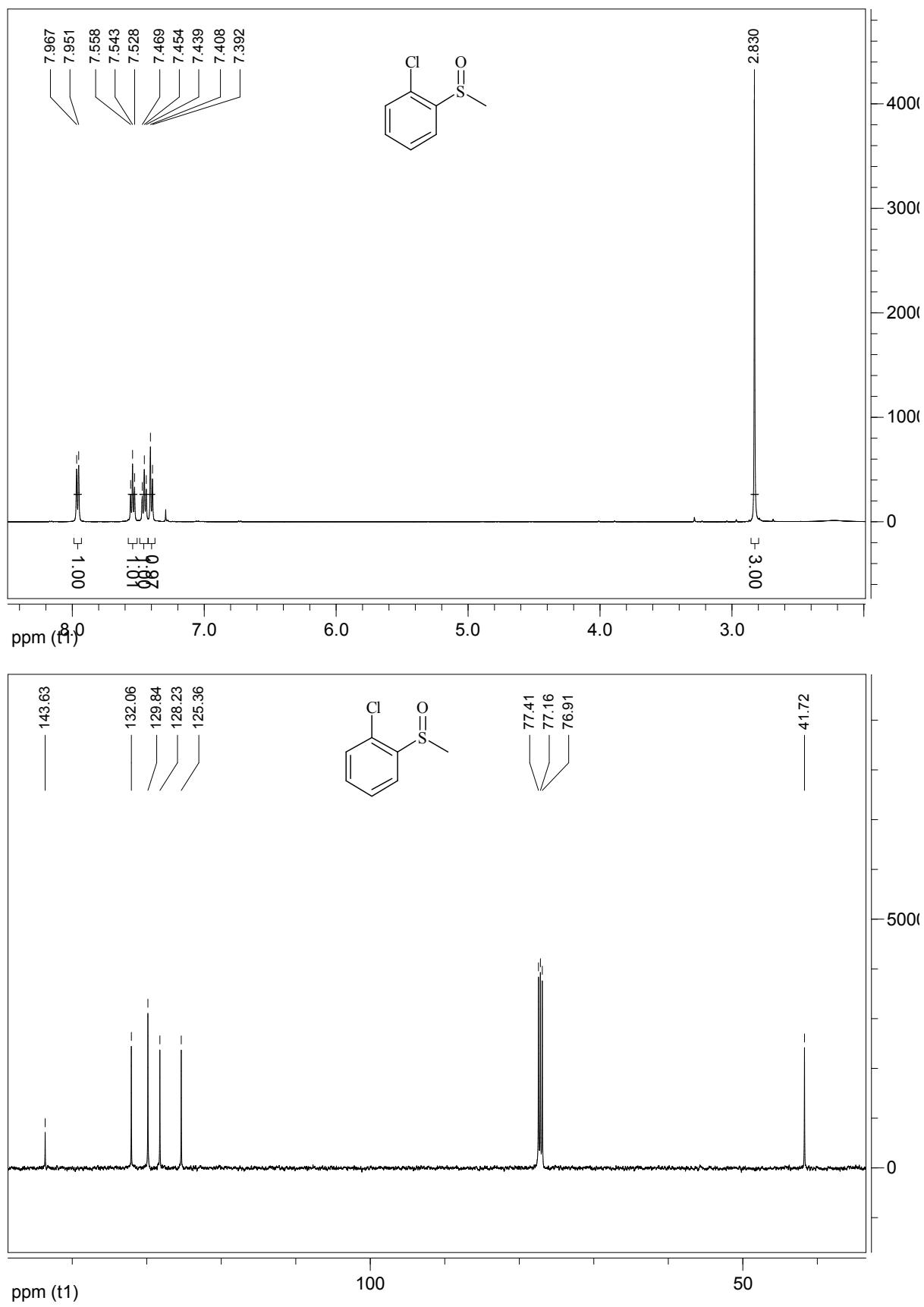
**3-Chlorophenyl methyl sulfoxide**



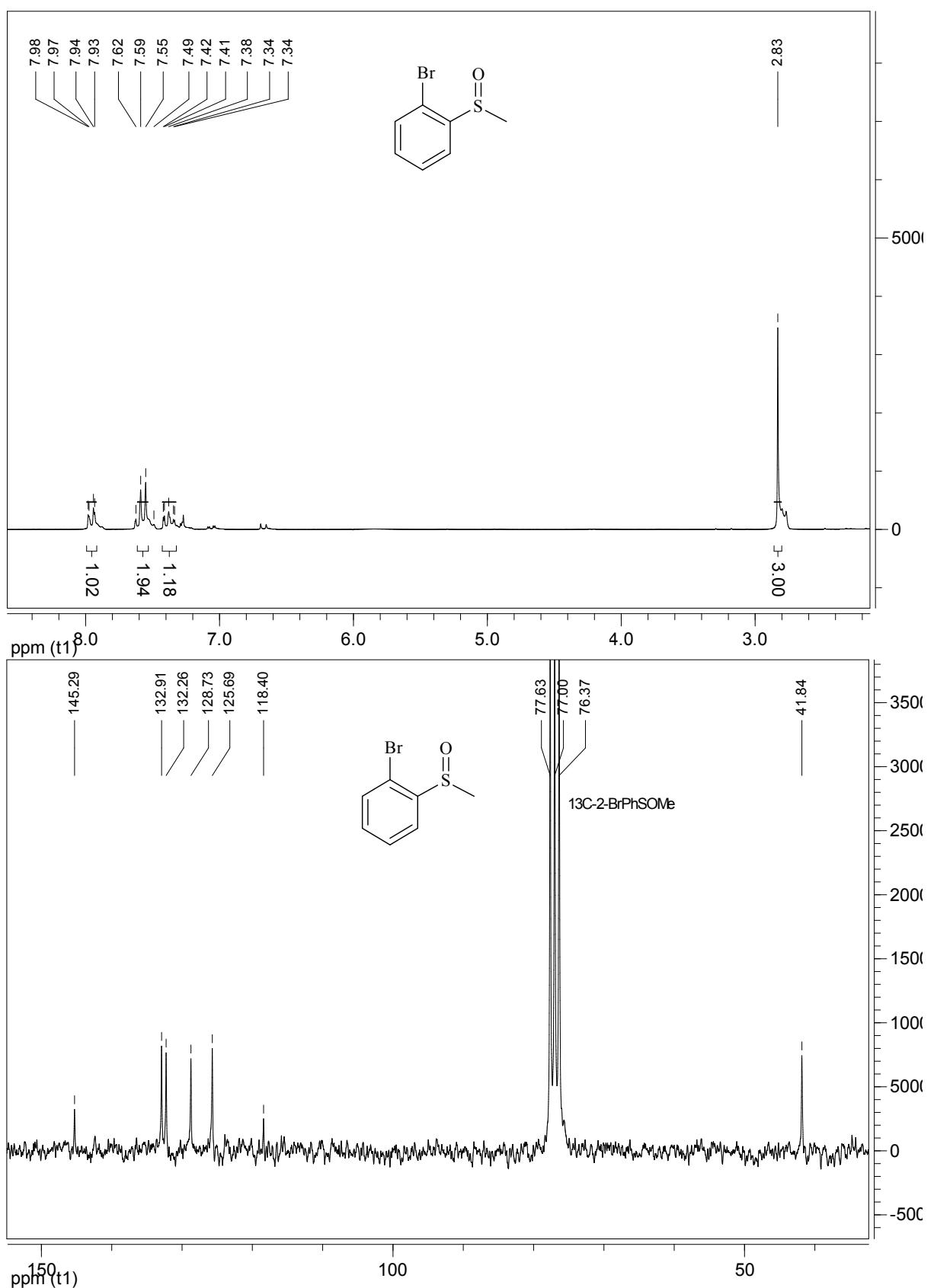
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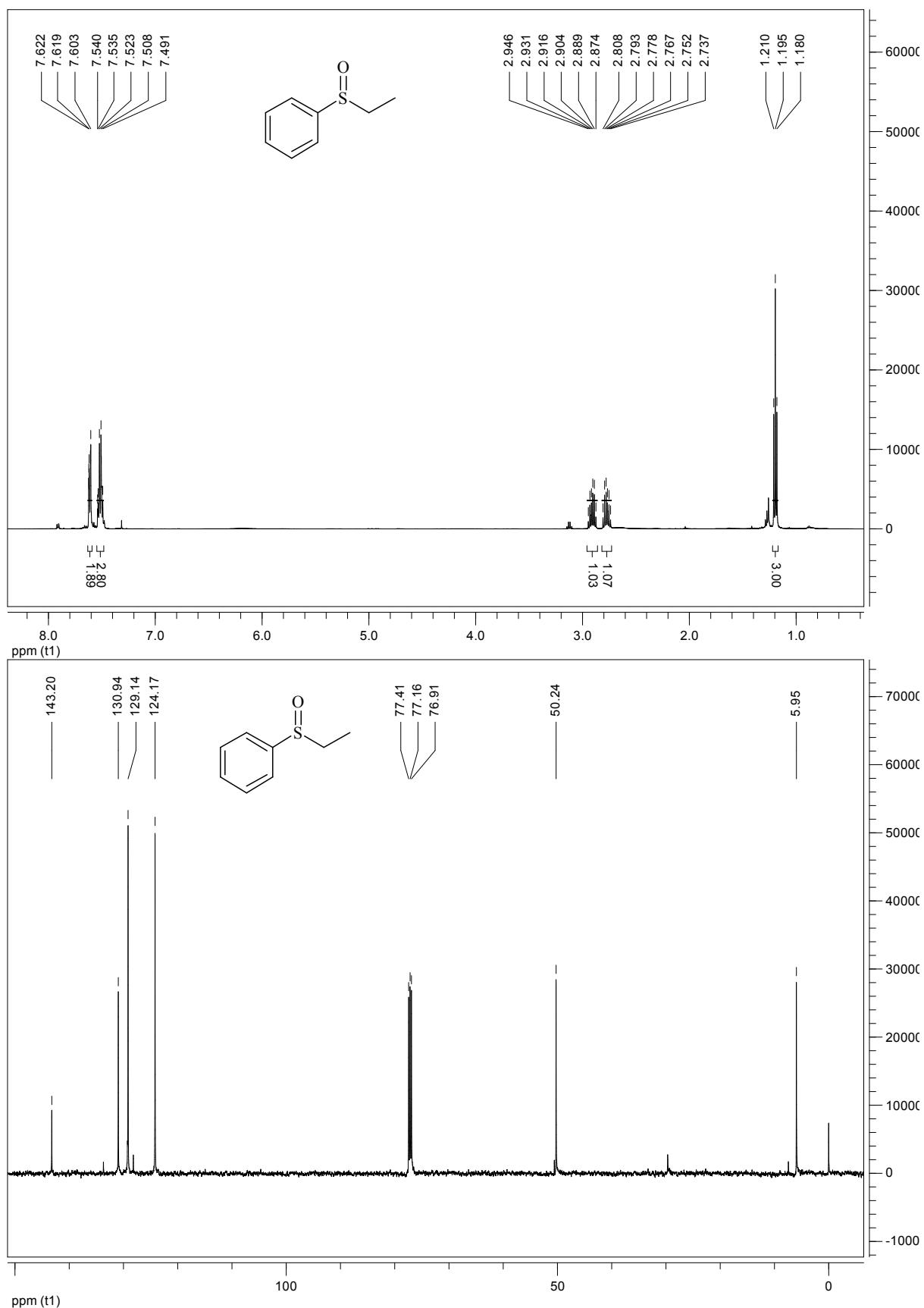
**2-Chlorophenyl methyl sulfoxide**



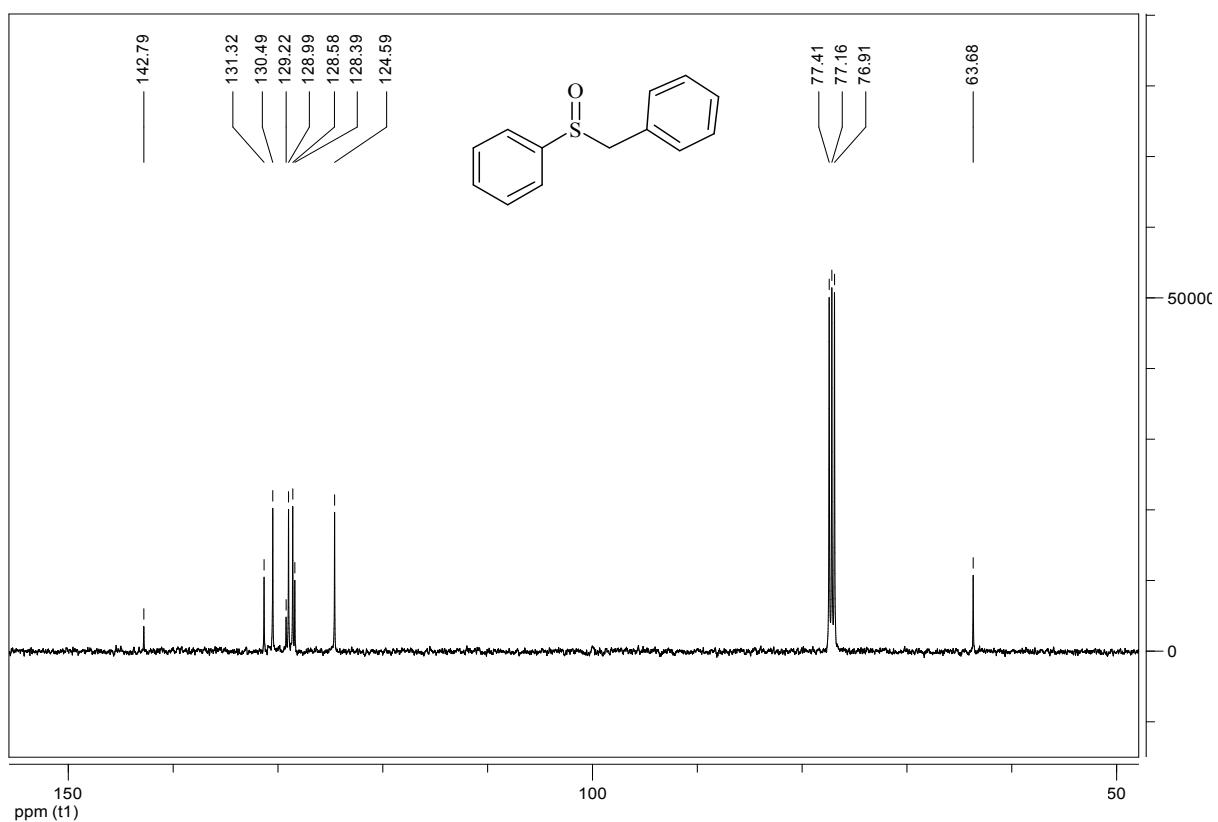
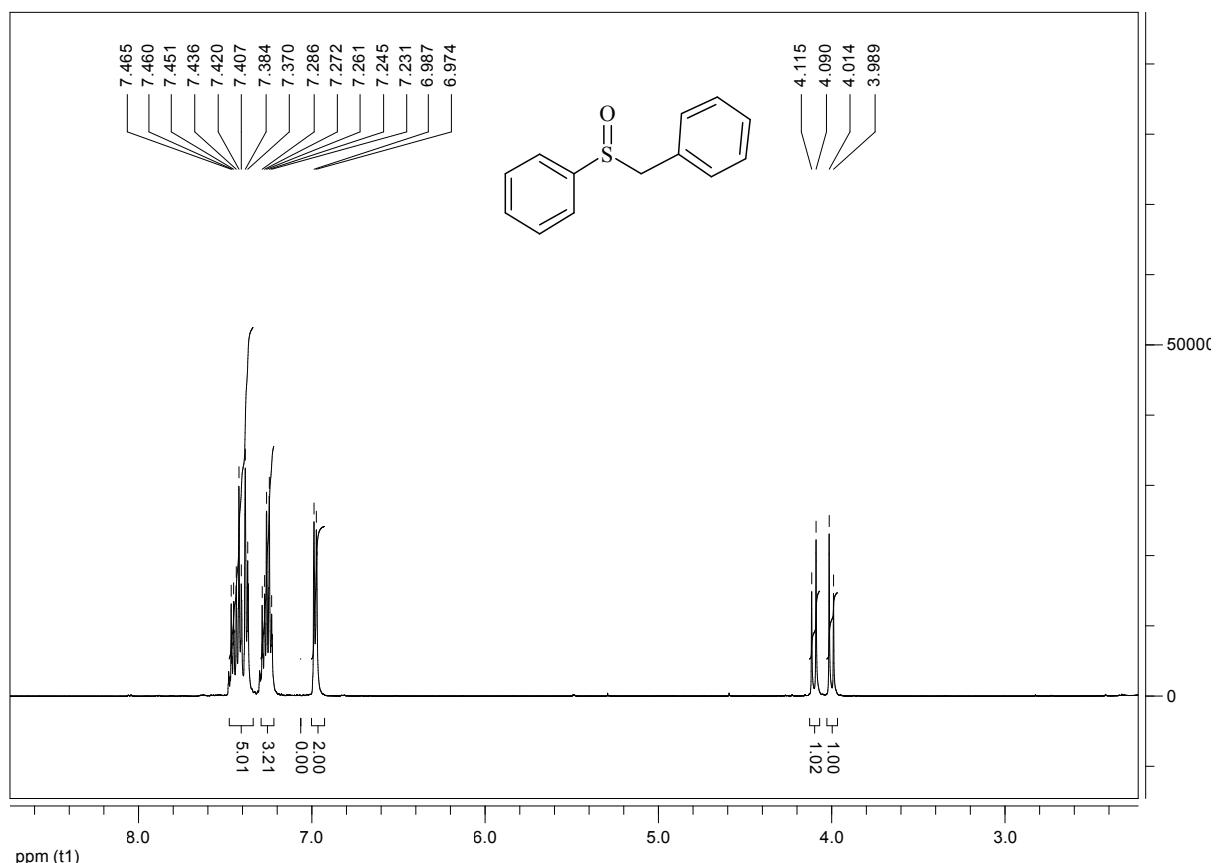
**2-Bromophenyl methyl sulfoxide**



### Ethyl phenyl sulfoxide

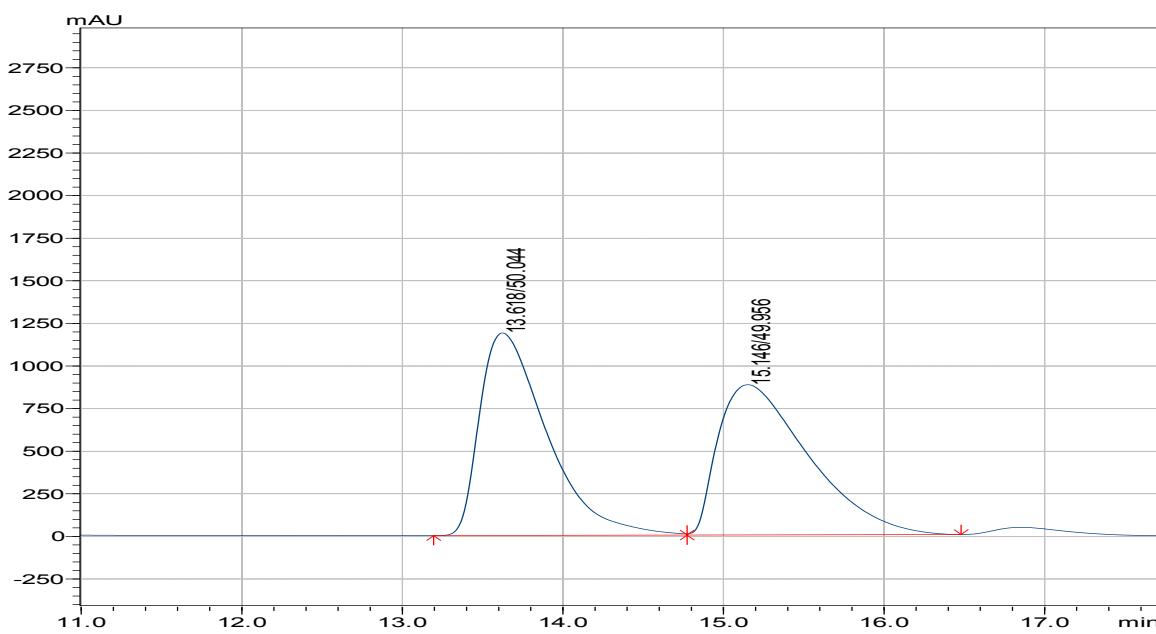


### **Benzyl phenyl sulfoxide**

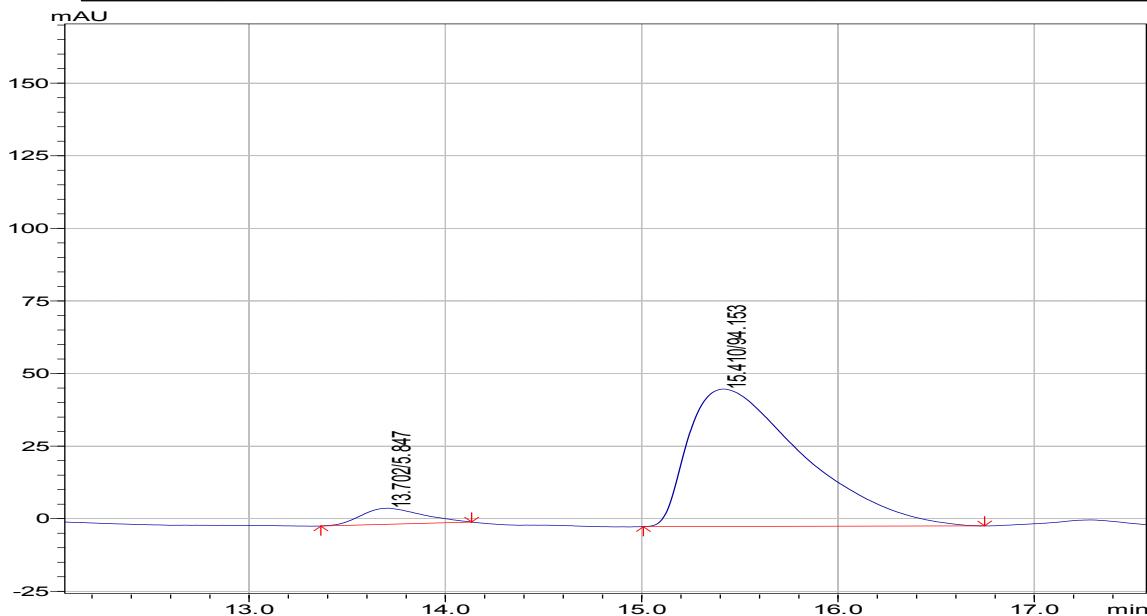


## 8. HPLC chromatogram of sulfoxides

### Phenyl methyl sulfoxide

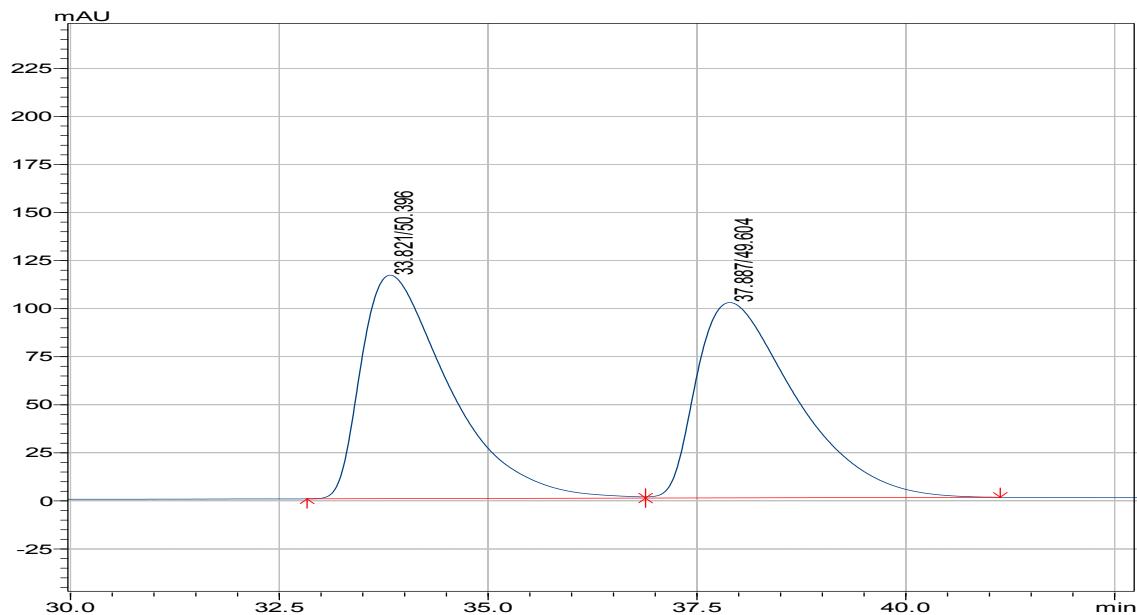


Peak#	Ret. Time	Area	Peak Start	Peak End	Area%
1	13.618	34533456	13.195	14.773	50.0444
2	15.146	34472223	14.773	16.480	49.9556

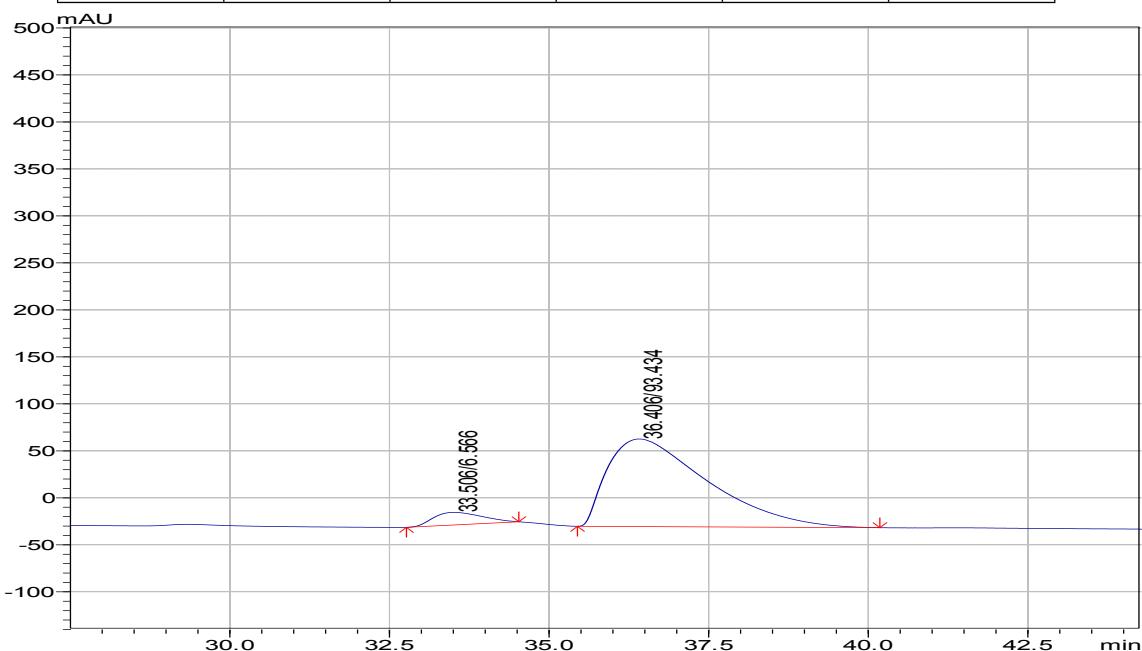


Peak#	Ret. Time	Area	Peak Start	Peak End	Area%
1	13.702	119036	13.365	14.133	5.8472
2	15.410	1916761	15.008	16.747	94.1528

### 4-Methylphenyl methyl sulfoxide

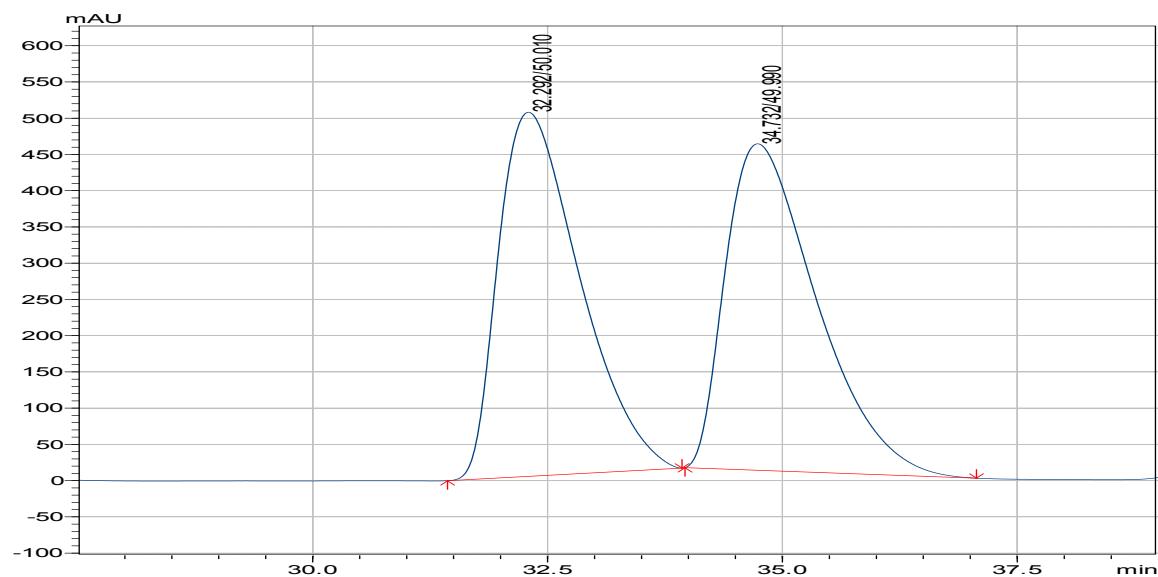


Peak#	Ret. Time	Area	Peak Start	Peak End	Area%
1	33.821	8527787	32.832	36.885	50.3958
2	37.887	8393825	36.885	41.131	49.6042

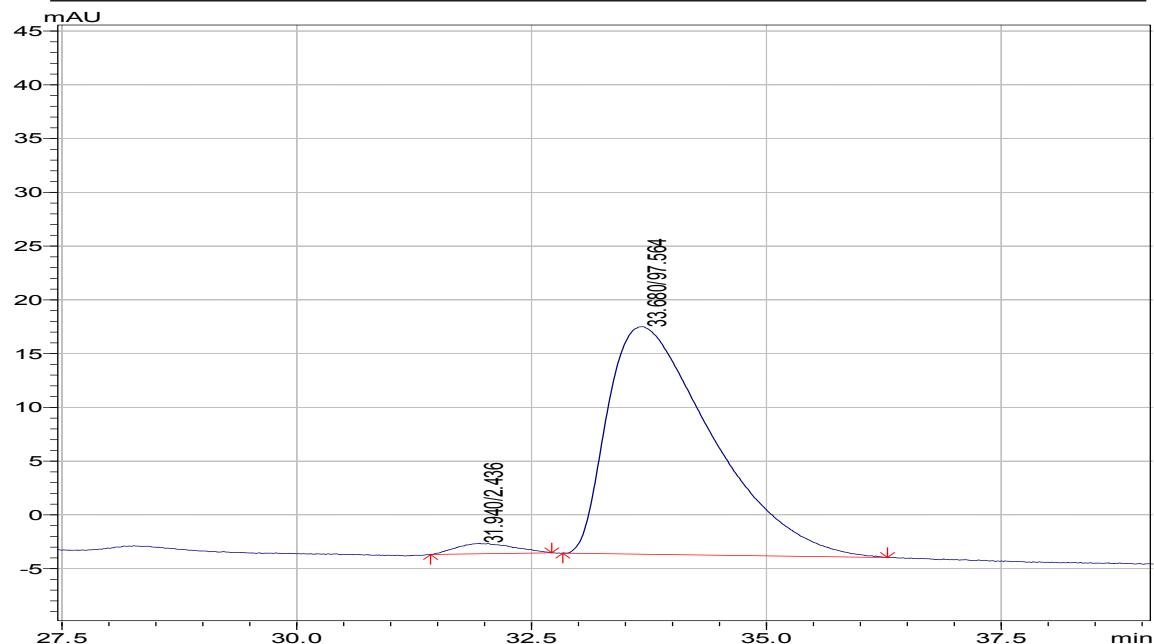


Peak#	Ret. Time	Area	Peak Start	Peak End	Area%
1	33.506	713478	32.768	34.528	6.5657
2	36.406	10153346	35.445	40.181	93.4343

### 4-Fluorophenyl methyl sulfoxide

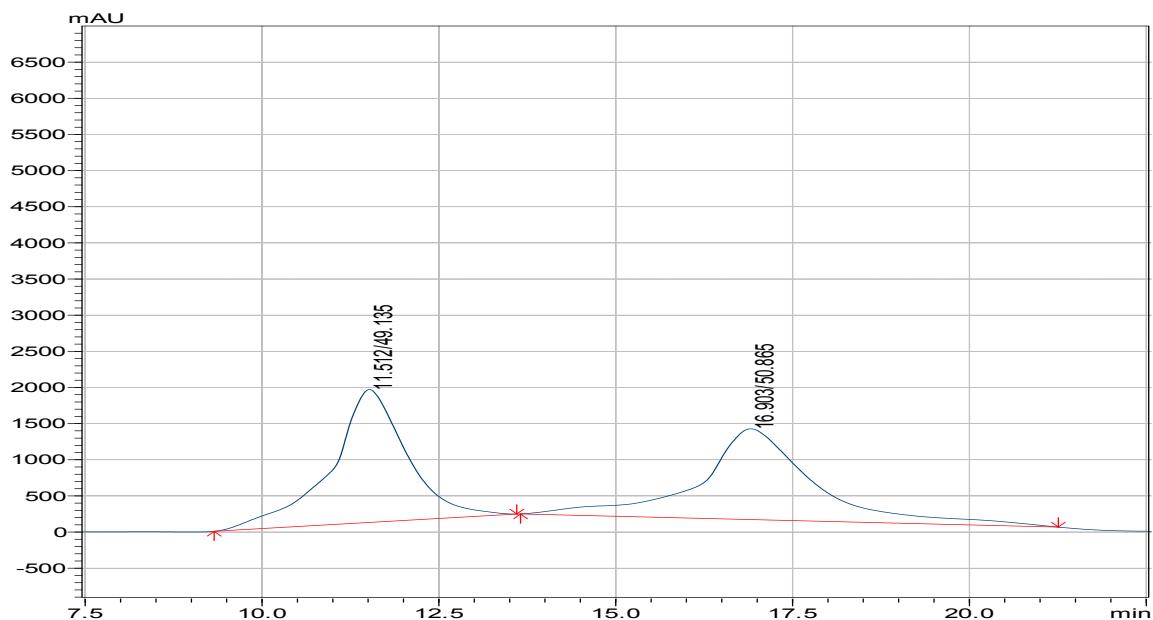


Peak#	Ret. Time	Area	Peak Start	Peak End	Area%
1	32.292	30331958	31.435	33.931	50.0102
2	34.732	30319639	33.963	37.067	49.9898

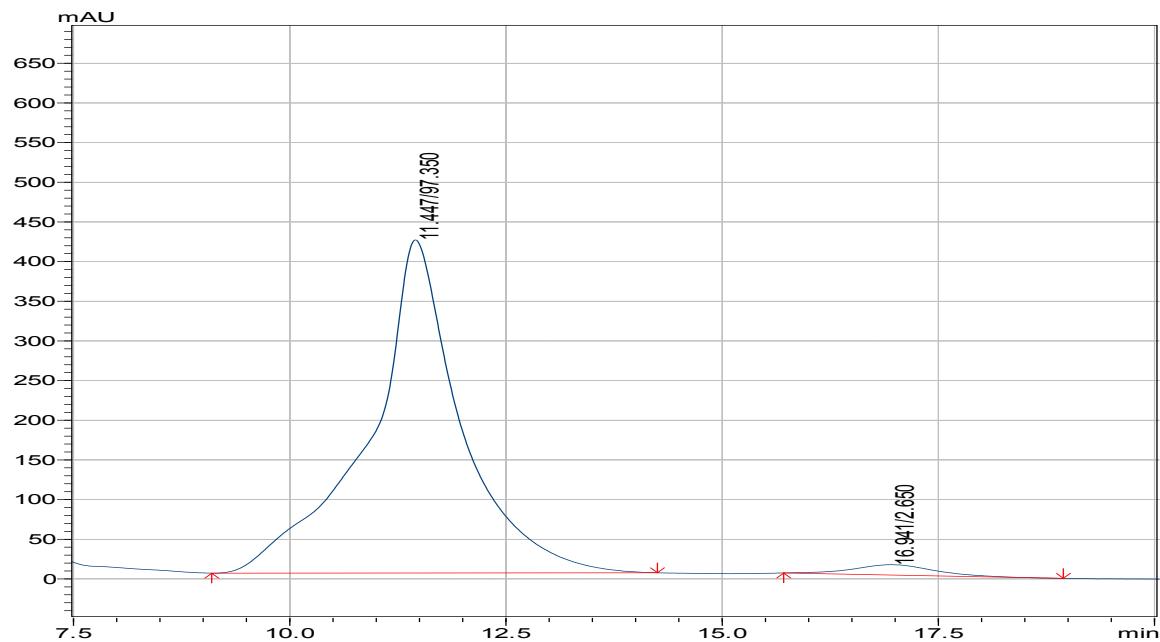


Peak#	Ret. Time	Area	Peak Start	Peak End	Area%
1	31.940	40969	31.424	32.715	2.4355
2	33.680	1641175	32.832	36.288	97.5645

### 4-Chlorophenyl methyl sulfoxide

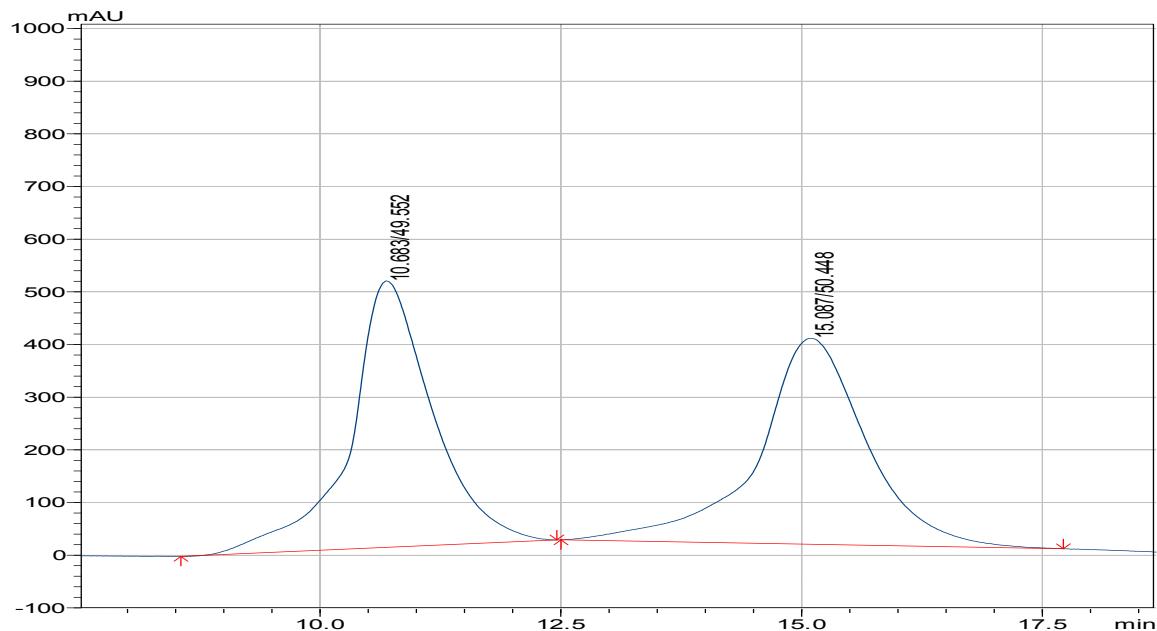


Peak#	Ret. Time	Area	Peak Start	Peak End	Area%
1	11.512	137217214	9.323	13.600	49.1353
2	16.903	142046813	13.653	21.259	50.8647

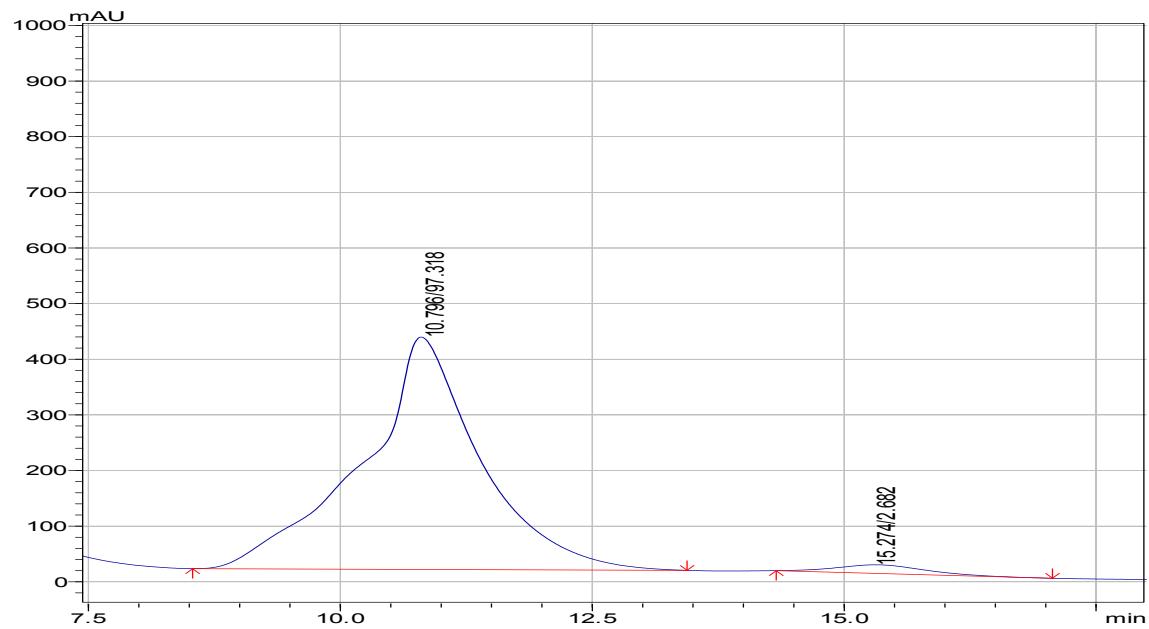


Peak#	Ret. Time	Area	Peak Start	Peak End	Area%
1	11.447	31089574	9.099	14.251	97.3501
2	16.941	846271	15.712	18.944	2.6499

### 4-Bromophenyl methyl sulfoxide

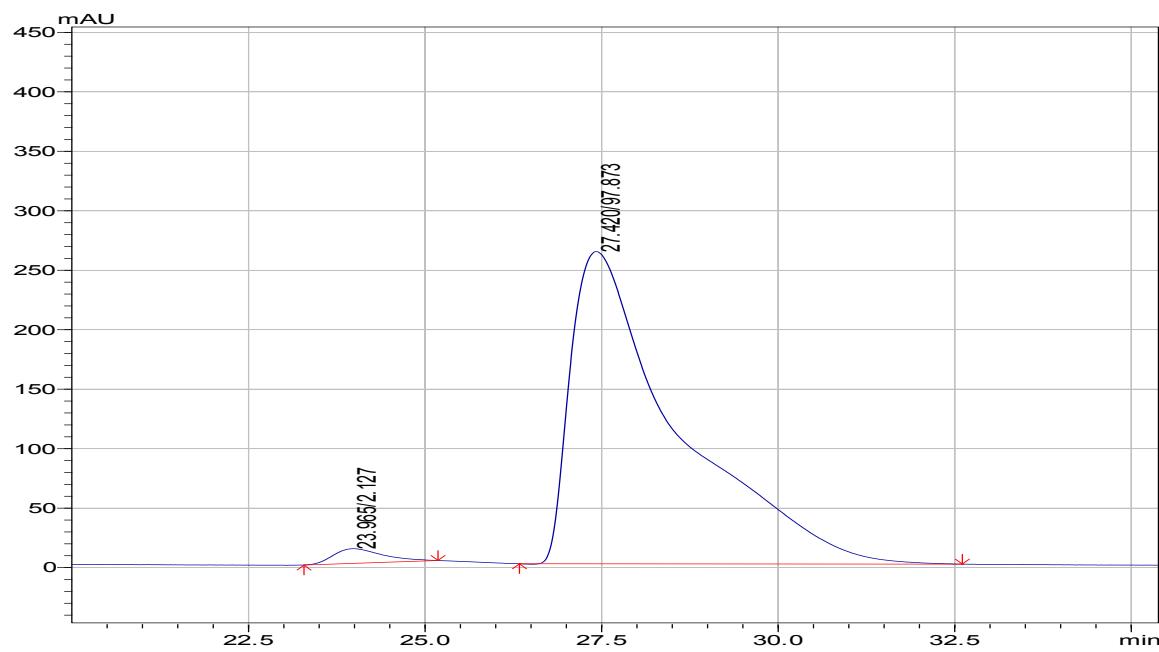
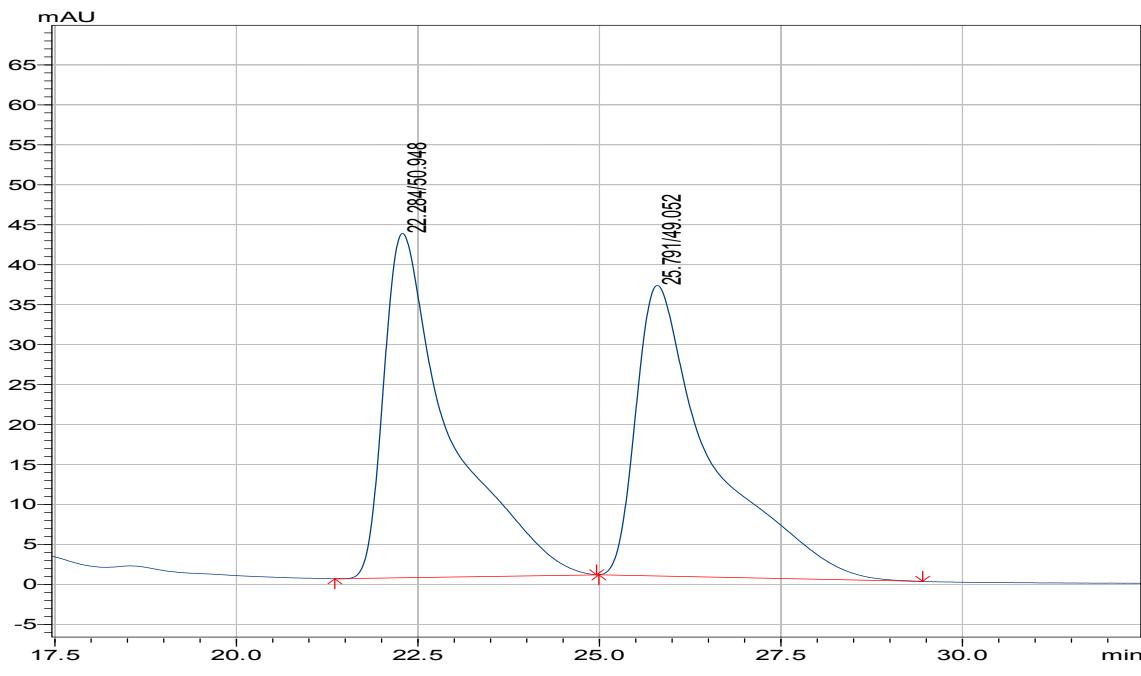


Peak#	Ret. Time	Area	Peak Start	Peak End	Area%
1	10.683	30273114	8.555	12.459	49.5516
2	15.087	30821033	12.501	17.717	50.4484

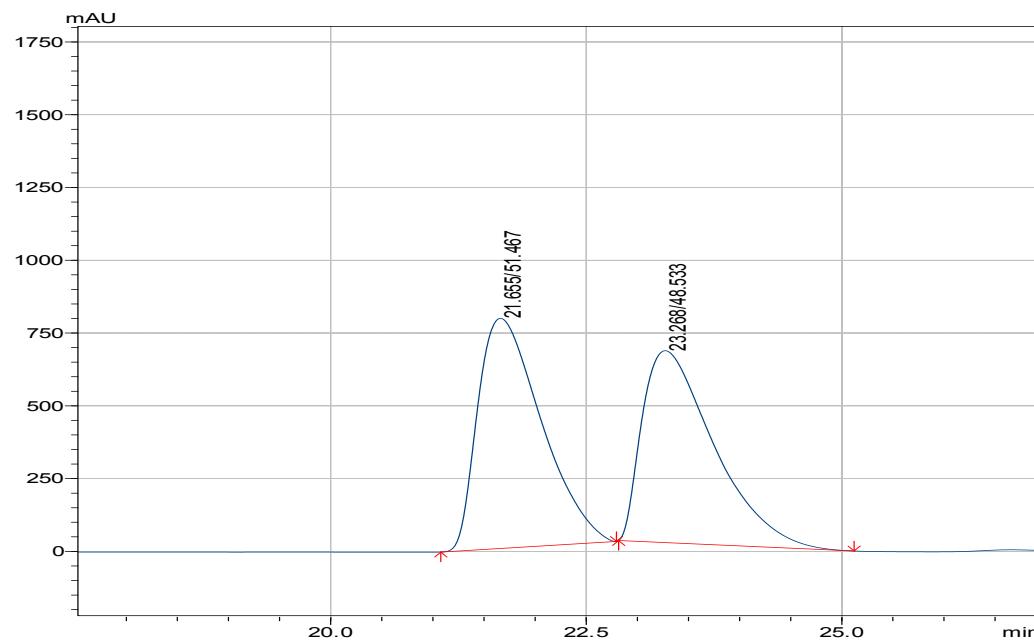


Peak#	Ret. Time	Area	Peak Start	Peak End	Area%
1	10.796	34015374	8.533	13.440	97.1864
2	15.274	984774	14.101	17.579	2.8136

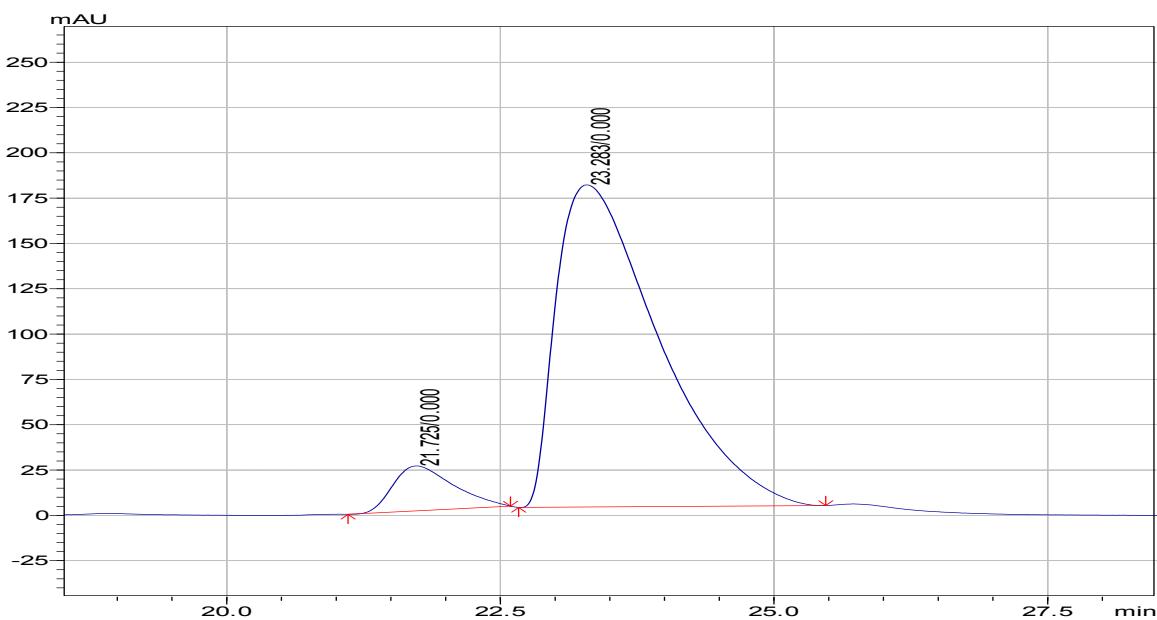
### 4-Nitrophenyl methyl sulfoxide



### 4-Methoxyphenyl methyl sulfoxide

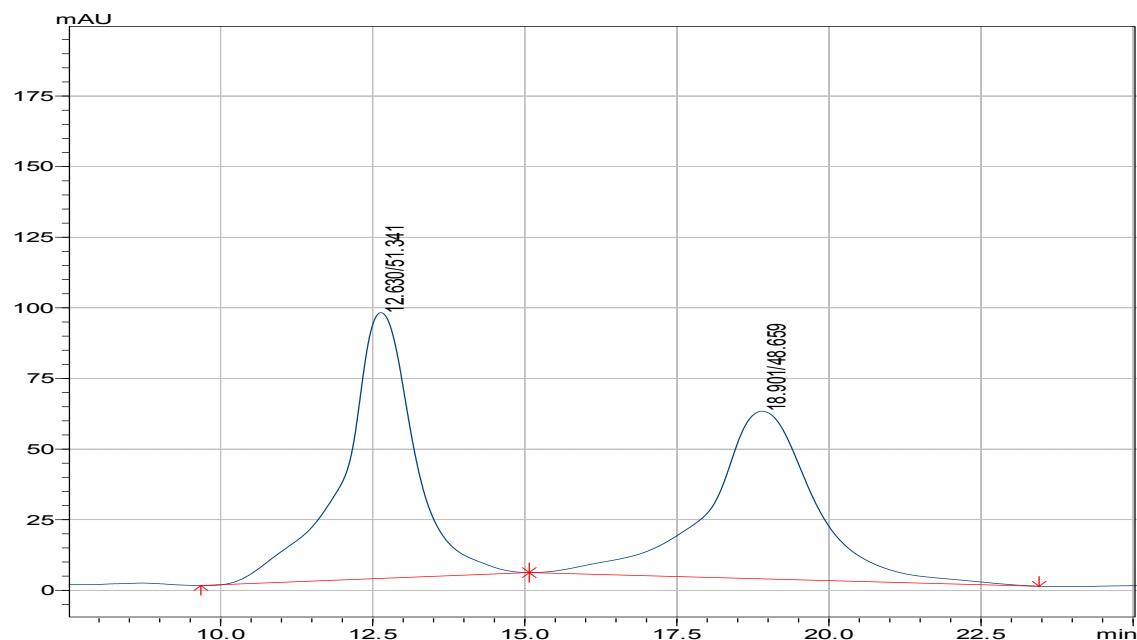


Peak#	Ret. Time	Area	Peak Start	Peak End	Area%
1	21.655	34179408	21.077	22.795	51.4667
2	23.268	32231251	22.816	25.120	48.5333

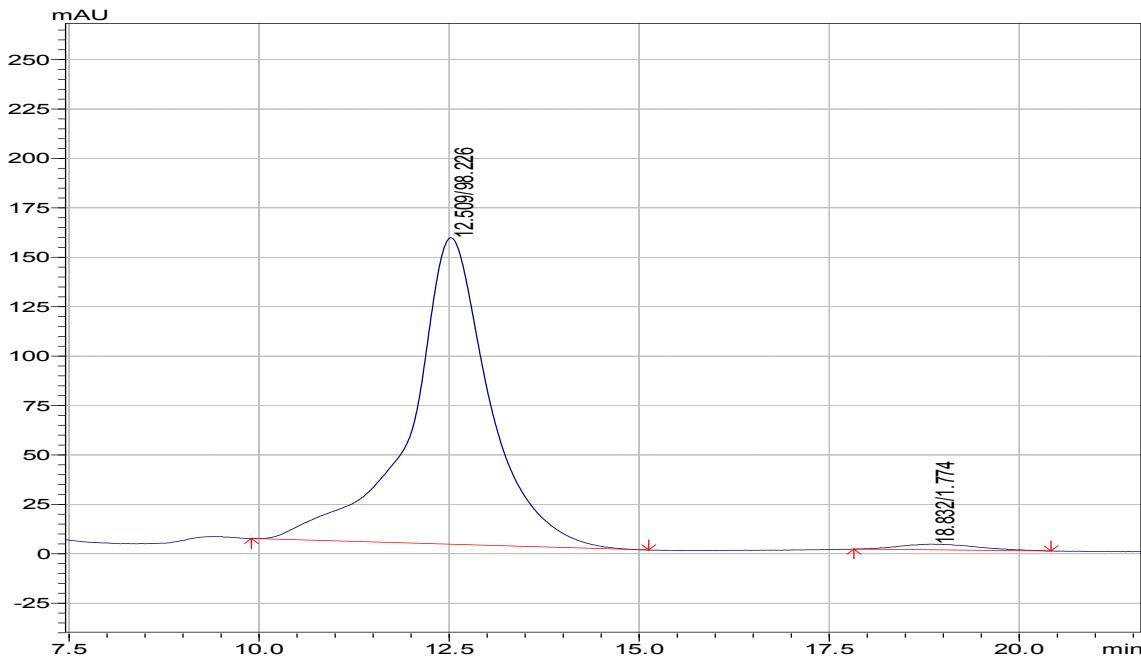


Peak#	Ret. Time	Area	Peak Start	Peak End	Area%
1	21.725	947437	21.109	22.592	7.5712
2	23.283	11566286	22.667	25.472	92.4288

### 3-Chlorophenyl methyl sulfoxide

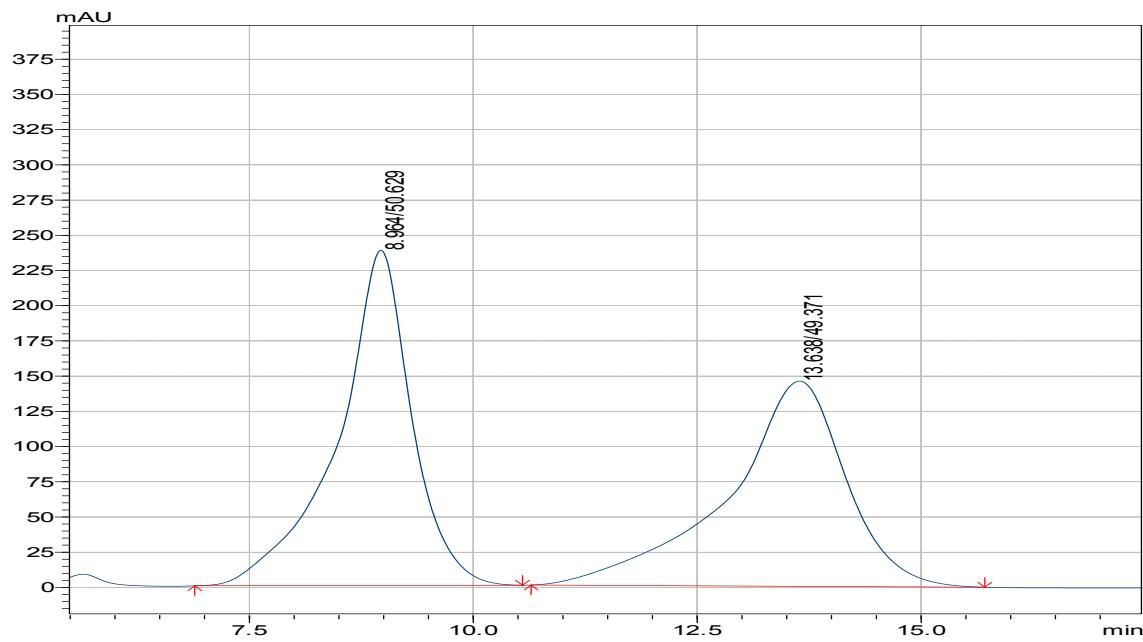


Peak#	Ret. Time	Area	Peak Start	Peak End	Area%
1	12.630	7616071	9.675	15.072	51.3408
2	18.901	7218260	15.072	23.456	48.6592

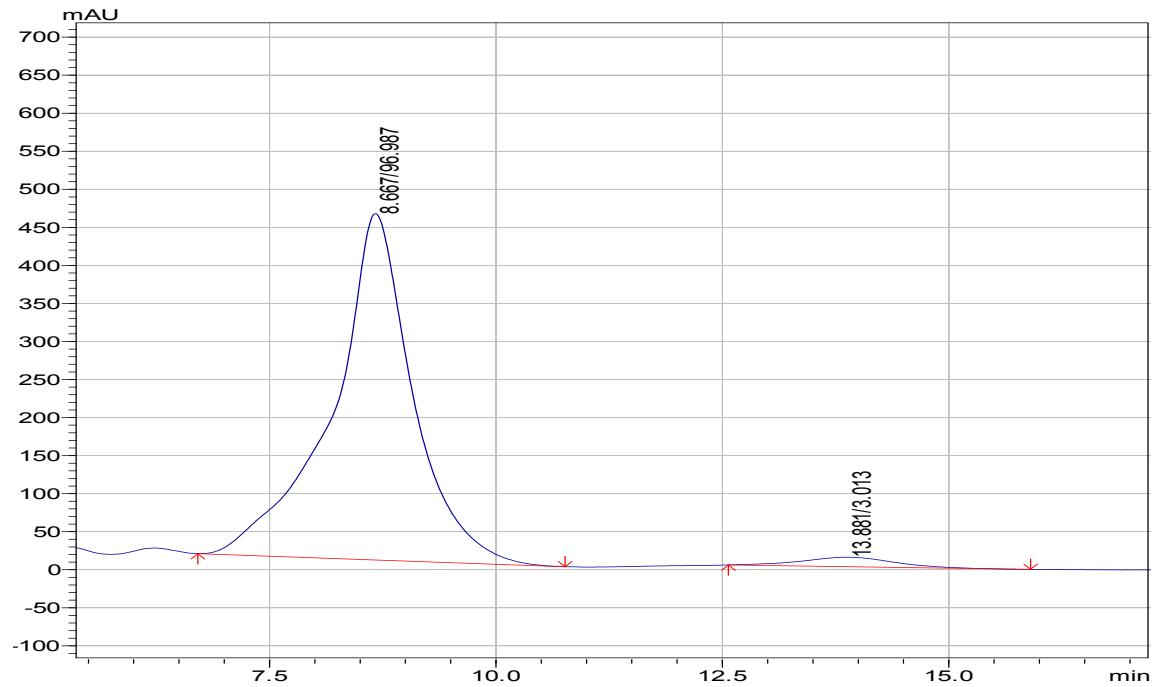


Peak#	Ret. Time	Area	Peak Start	Peak End	Area%
1	12.509	11177935	9.899	15.125	98.2256
2	18.832	201929	17.824	20.416	1.7744

### 3-Bromophenyl methyl sulfoxide

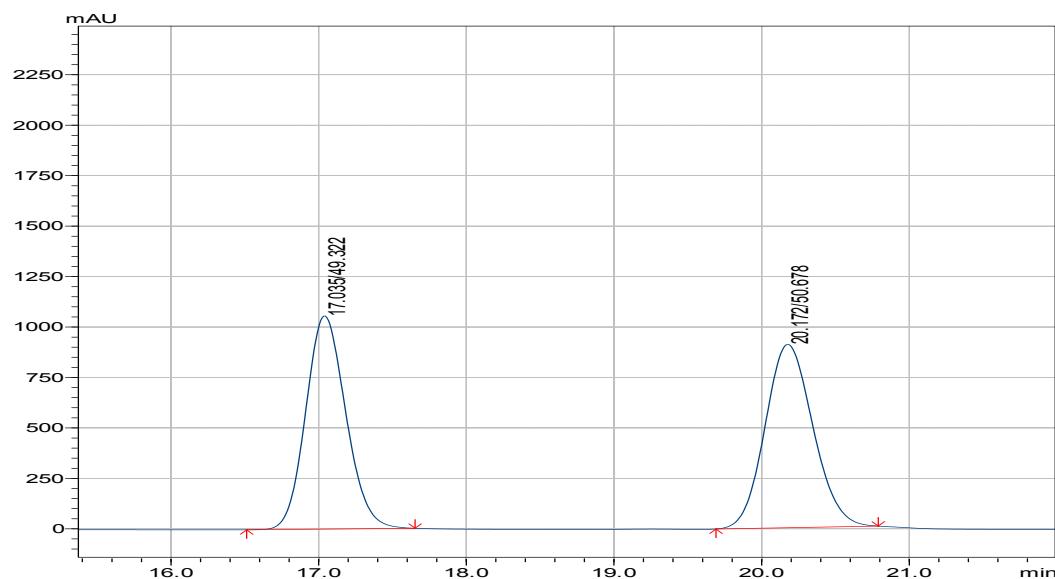


Peak#	Ret. Time	Area	Peak Start	Peak End	Area%
1	8.964	13612213	6.891	10.549	50.6293
2	13.638	13273847	10.645	15.712	49.3707

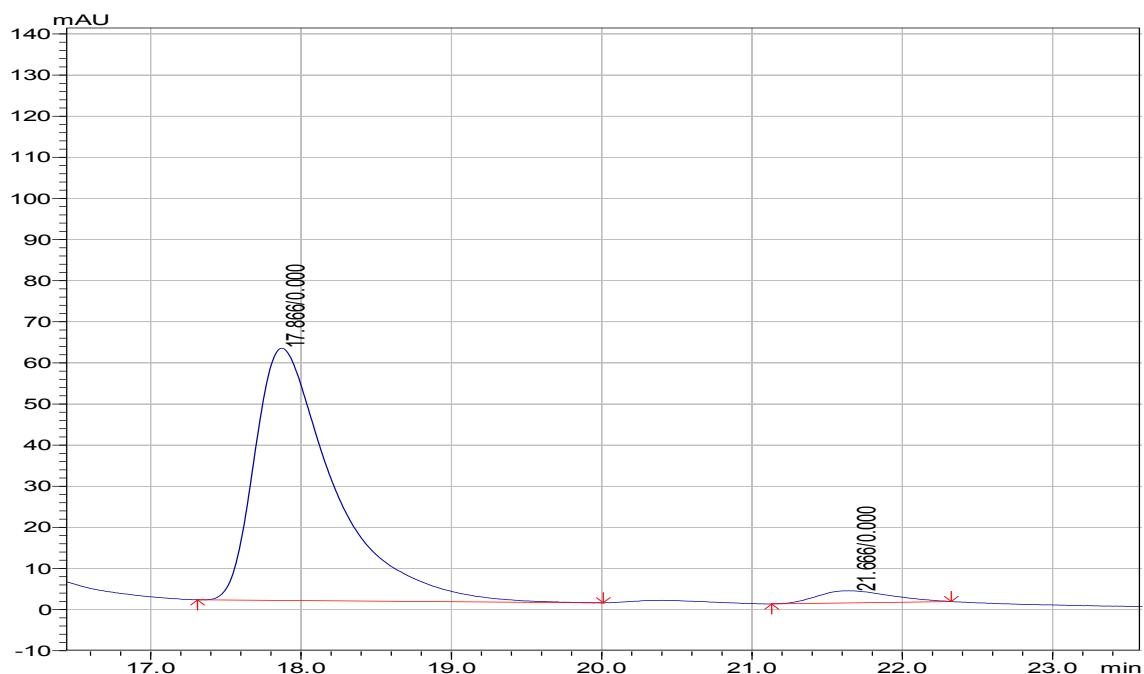


Peak#	Ret. Time	Area	Peak Start	Peak End	Area%
1	8.667	28183869	6.709	10.763	96.9872
2	13.881	875488	12.565	15.904	3.0128

## 2-Chlorophenyl methyl sulfoxide

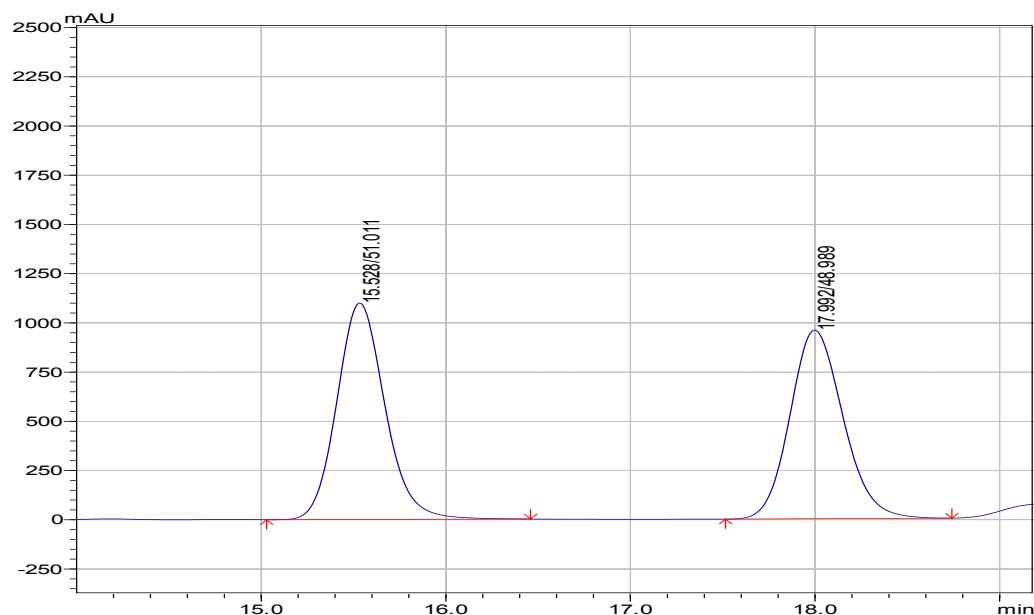


Peak#	Ret. Time	Area	Peak Start	Peak End	Area%
1	17.035	19936115	16.512	17.653	49.3216
2	20.172	20484572	19.691	20.789	50.6784

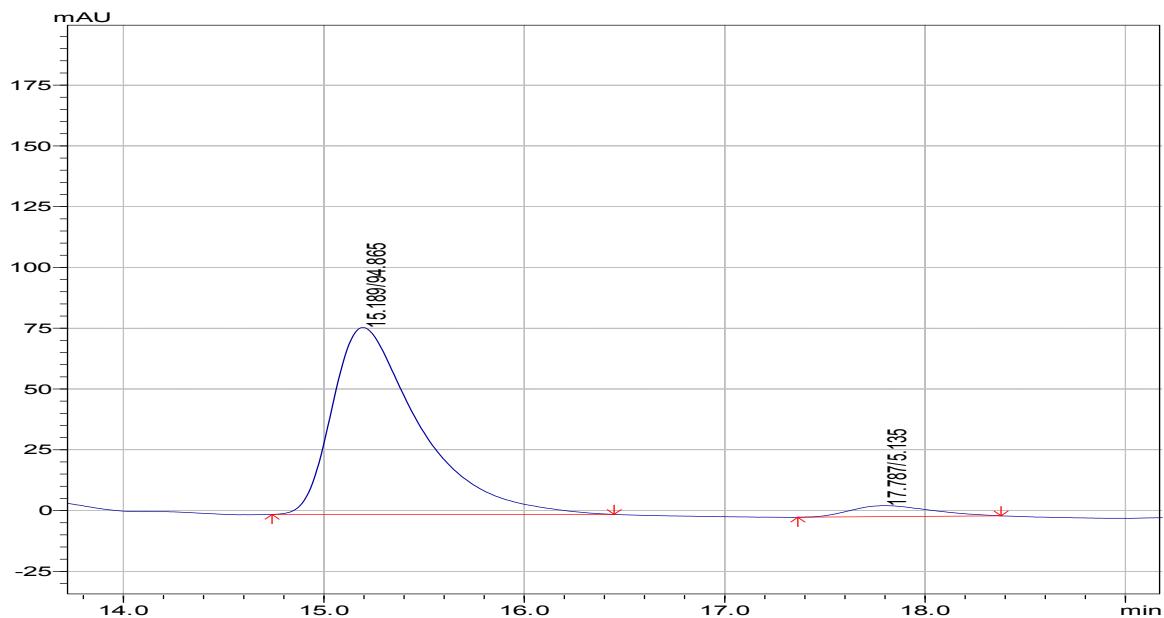


Peak#	Ret. Time	Area	Peak Start	Peak End	Area%
1	17.866	2240487	17.312	20.011	95.7105
2	21.666	100414	21.131	22.325	4.2895

## 2-Bromophenyl methyl sulfoxide

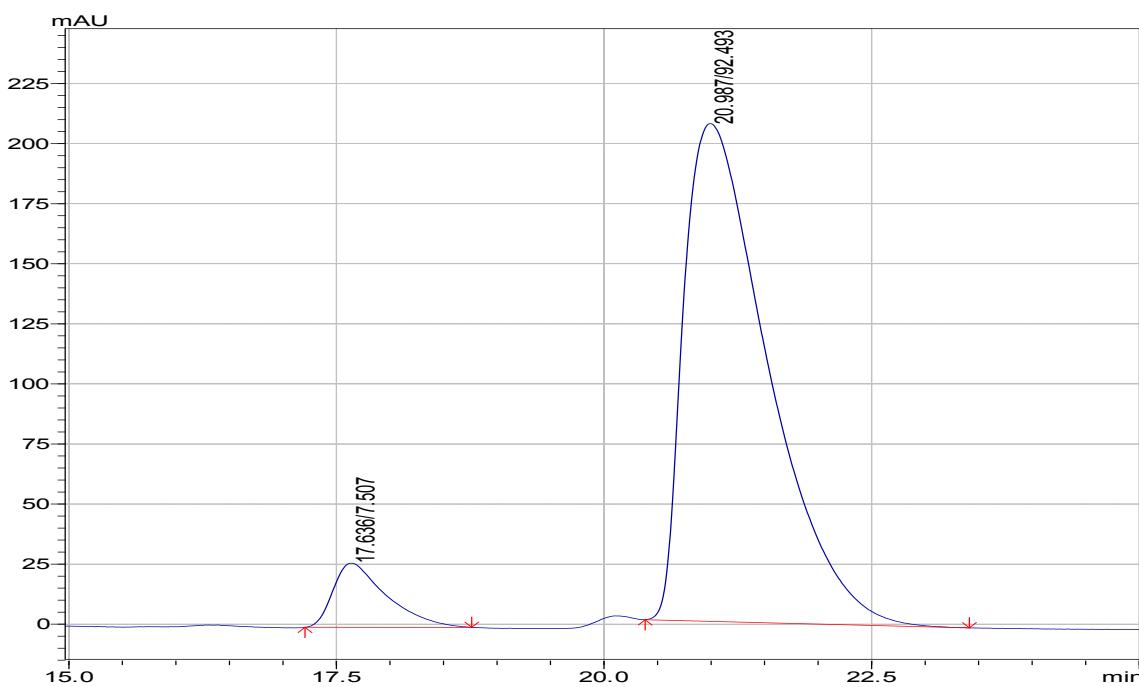
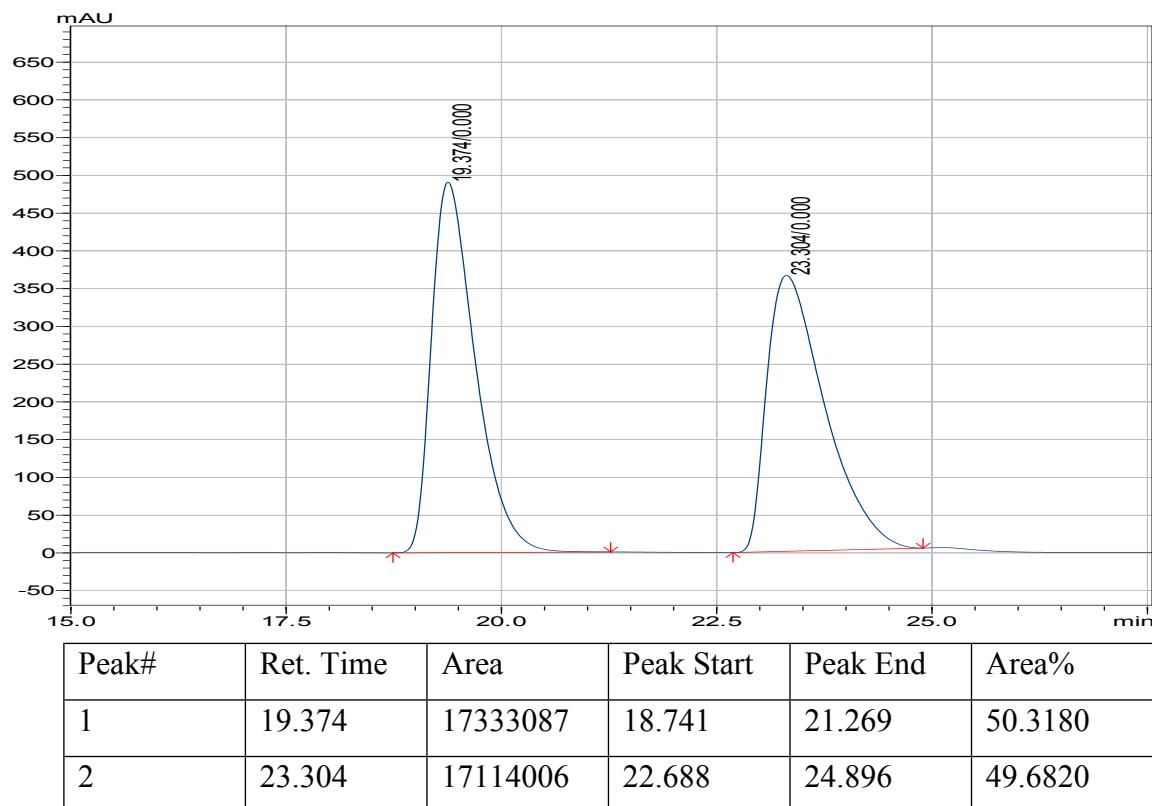


Peak#	Ret. Time	Area	Peak Start	Peak End	Area%
1	15.528	19914903	15.029	16.459	51.0112
2	17.992	19125317	17.515	18.741	48.9888

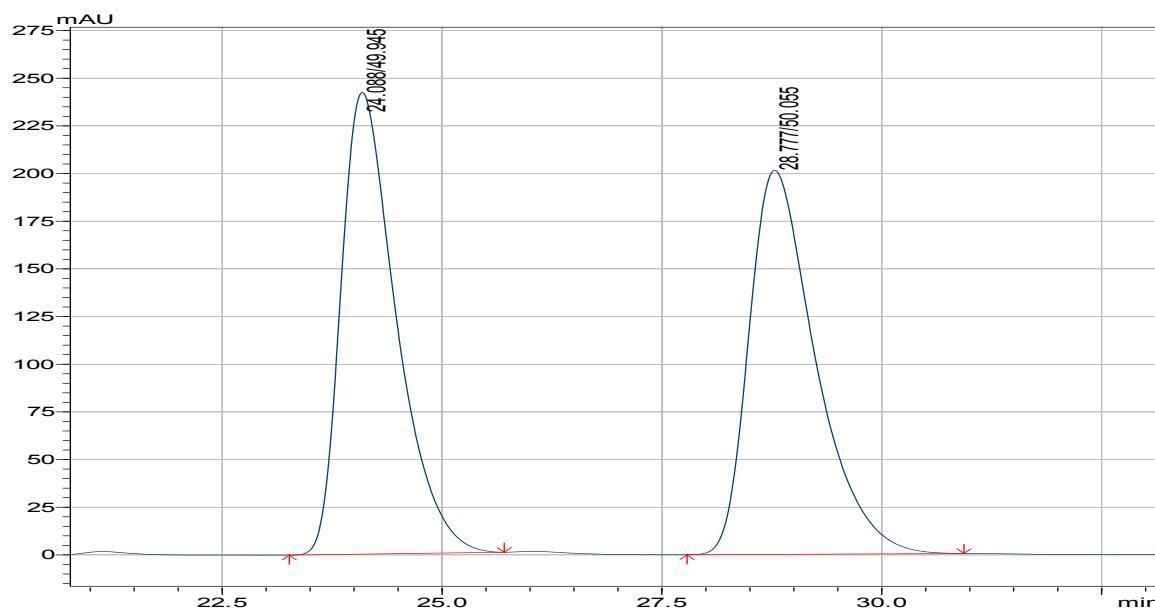


Peak#	Ret. Time	Area	Peak Start	Peak End	Area%
1	15.189	2322010	14.741	16.448	94.8646
2	17.787	125699	17.365	18.379	5.1354

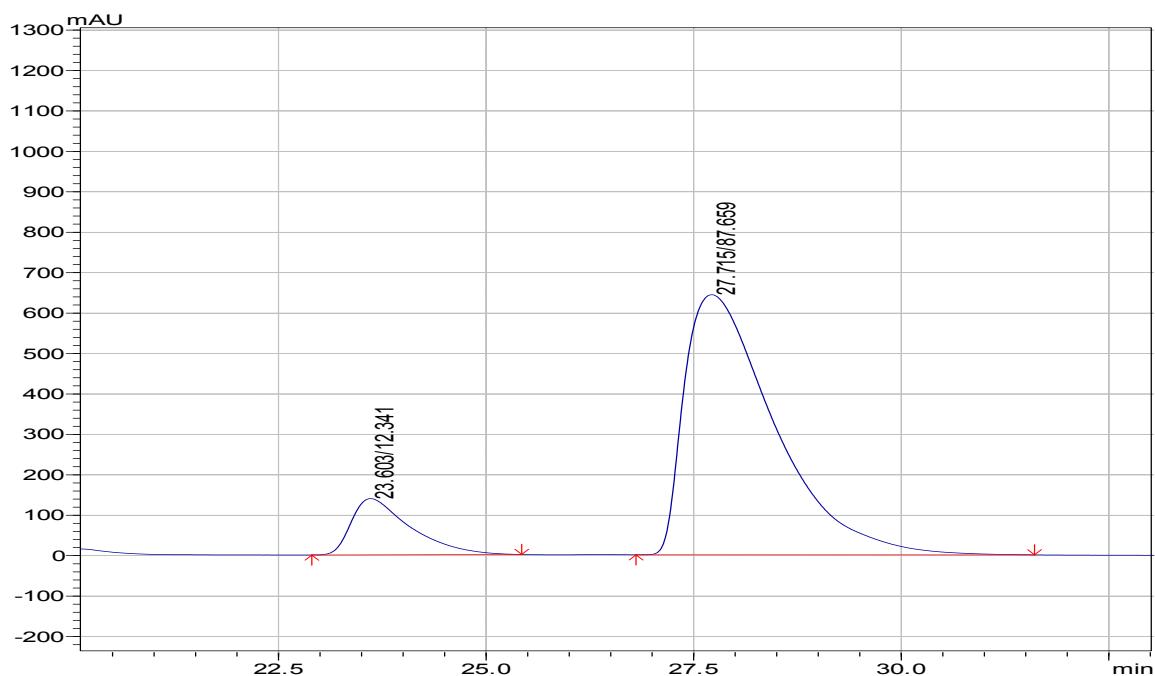
### Ethyl phenyl sulfoxide



### Benzyl phenyl sulfide



Peak#	Ret. Time	Area	Peak Start	Peak End	Area%
1	24.088	10564866	23.264	25.707	49.9445
2	28.777	10588329	27.787	30.933	50.0555



Peak#	Ret. Time	Area	Peak Start	Peak End	Area%
1	23.603	6841224	22.901	25.429	12.3411
2	27.715	48593255	26.805	31.605	87.658

## 9. Notes and references

- 1 J. Legros, C. Bolm, *Chem. Eur. J.*, 2005, **11**, 1086–1092.
- 2 J. Sun, C. Zhu, Z. Dai, M. Yang, Y. Pan, H. Hu, *J. Org. Chem.* 2004, **69**, 8500–8503.
- 3 S. Liao, B. List, *Adv. Synth. Catal.*, 2012, **354**, 2363–2367.
- 4 T. Yamaguchi, K. Matsumoto, B. Saito, T. Katsuki, *Angew. Chem. Int. Ed.*, 2007, **46**, 4729–4731.