Supplementary Material

A highly efficient synthesis of 1-trimethylsilyl-2-arylcyclopentenes using two consecutive stages of aqueous and anhydrous reactions

Jeevan Chakravarthy A. S., Krishnamurthy M. S., Noor Shahina Begum, and HariPrasad Suresh*
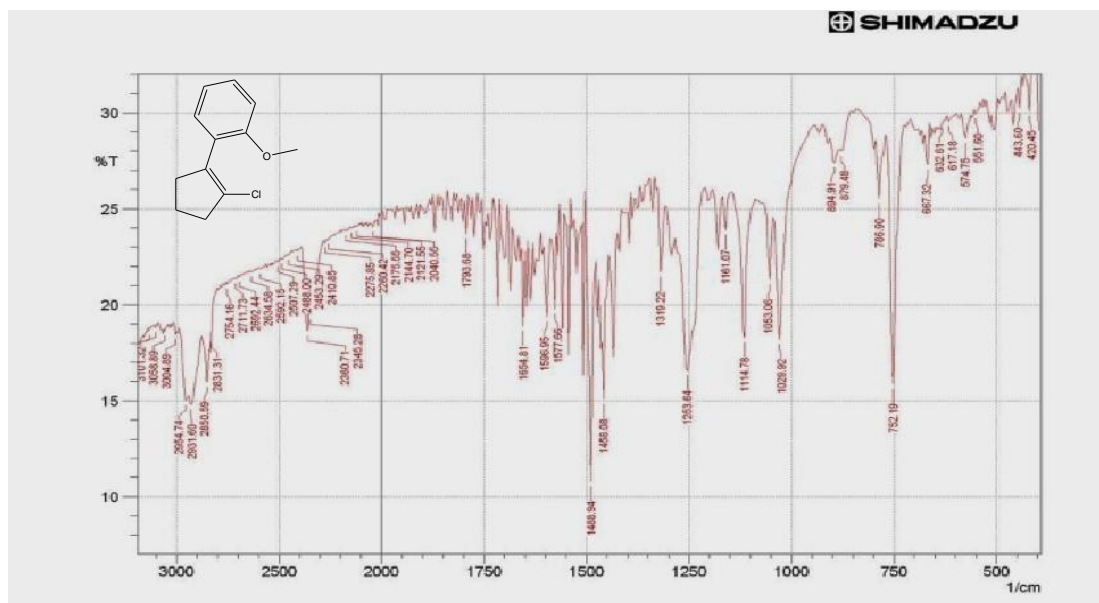
Department of Post Graduate Studies in Chemistry, Central College Campus, Bangalore University,
Palace Road, Bangalore – 560 001, India
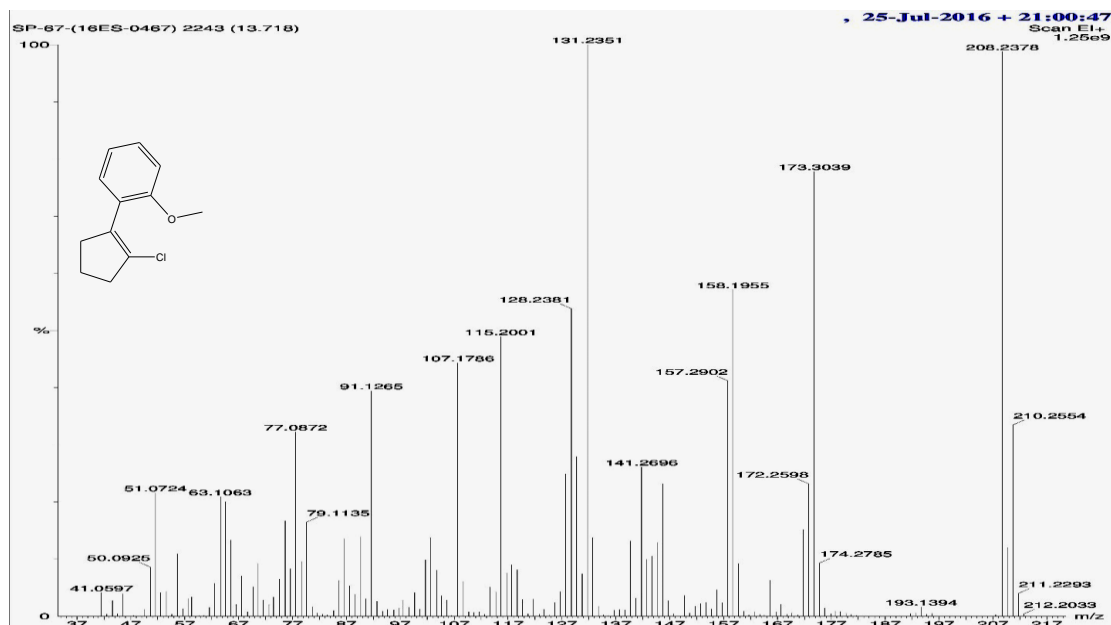*Email: hariprasad@bub.ernet.in*
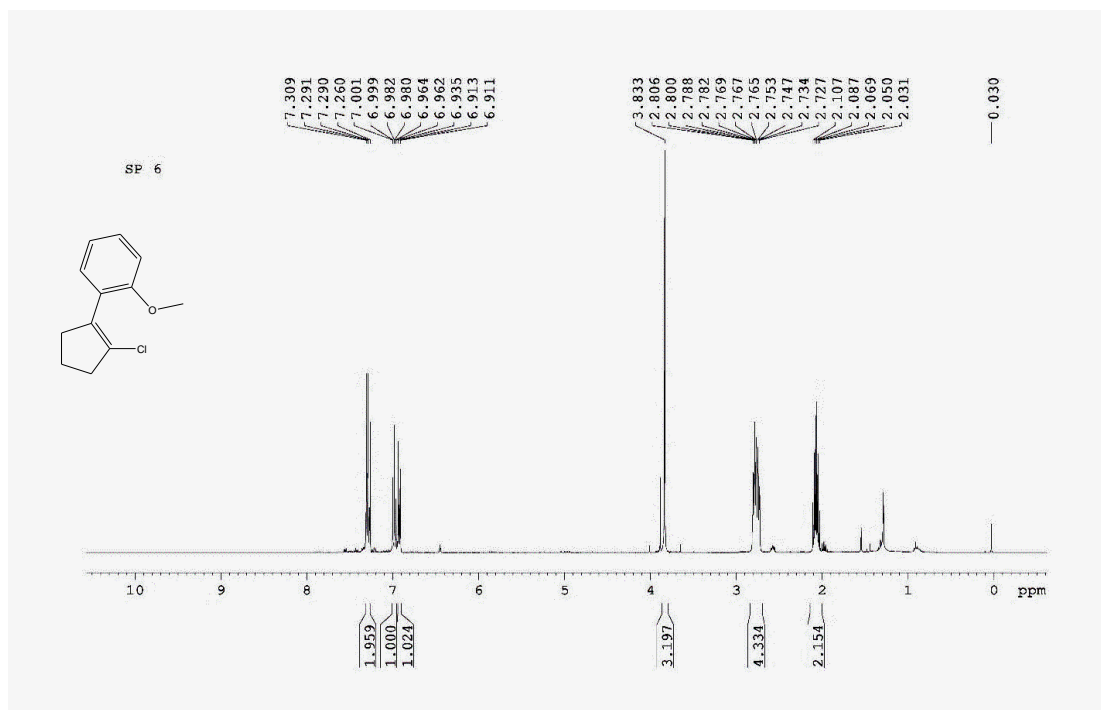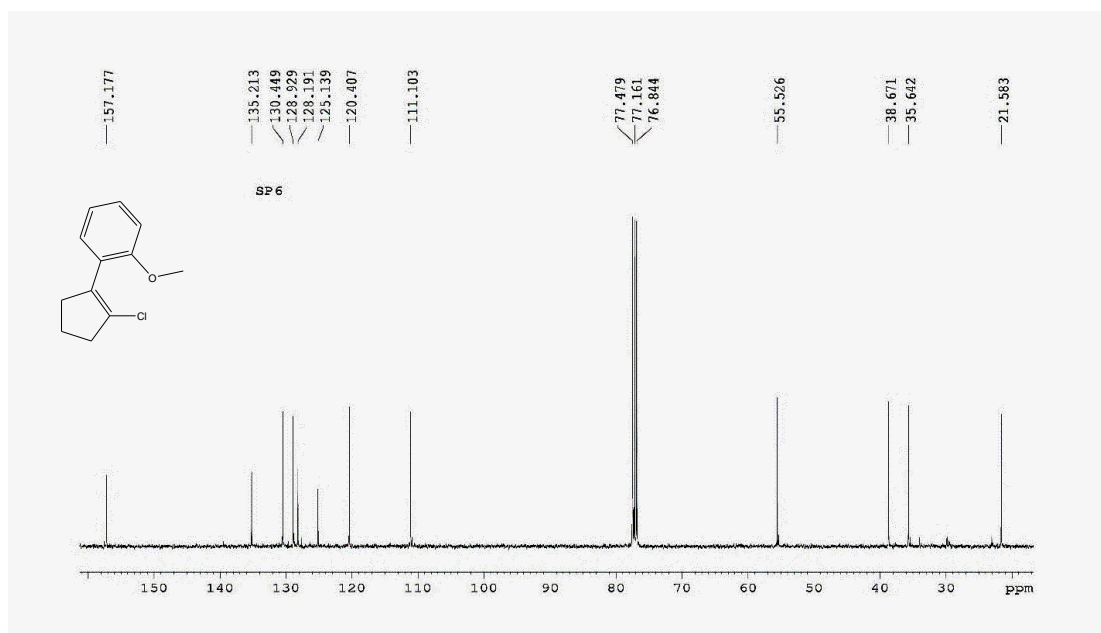
Table of Contents

IR of 1-chloro-2-(2′-methoxyphenyl)cyclopenetene 3a:

MS of 1-chloro-2-(2′-methoxyphenyl)cyclopentene 3a:

¹H NMR of 1-chloro-2-(2′-methoxyphenyl)cyclopentene 3a:

¹³C NMR of 1-chloro-2-(2′-methoxyphenyl)cyclopentene 3a:

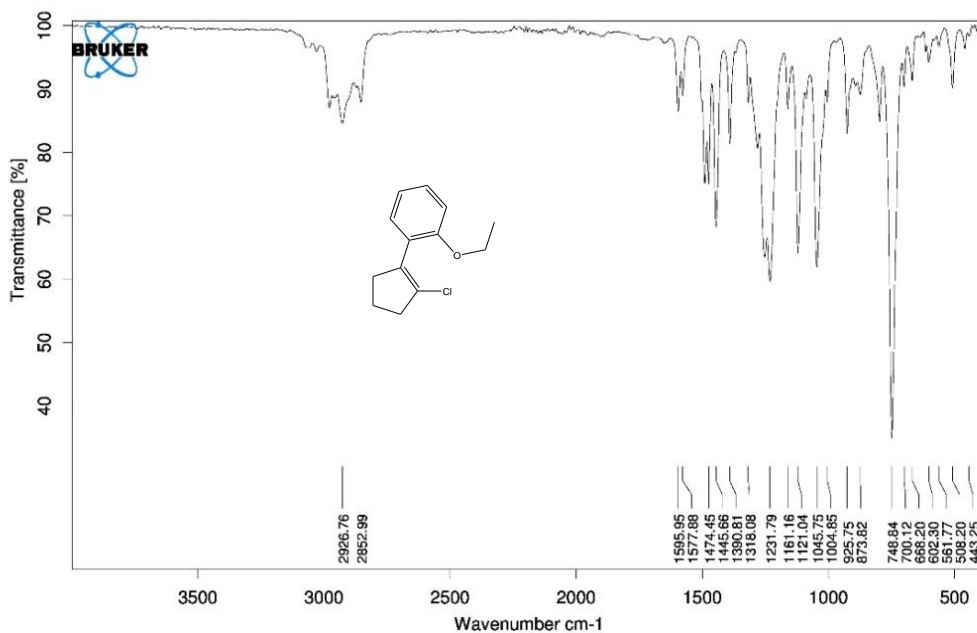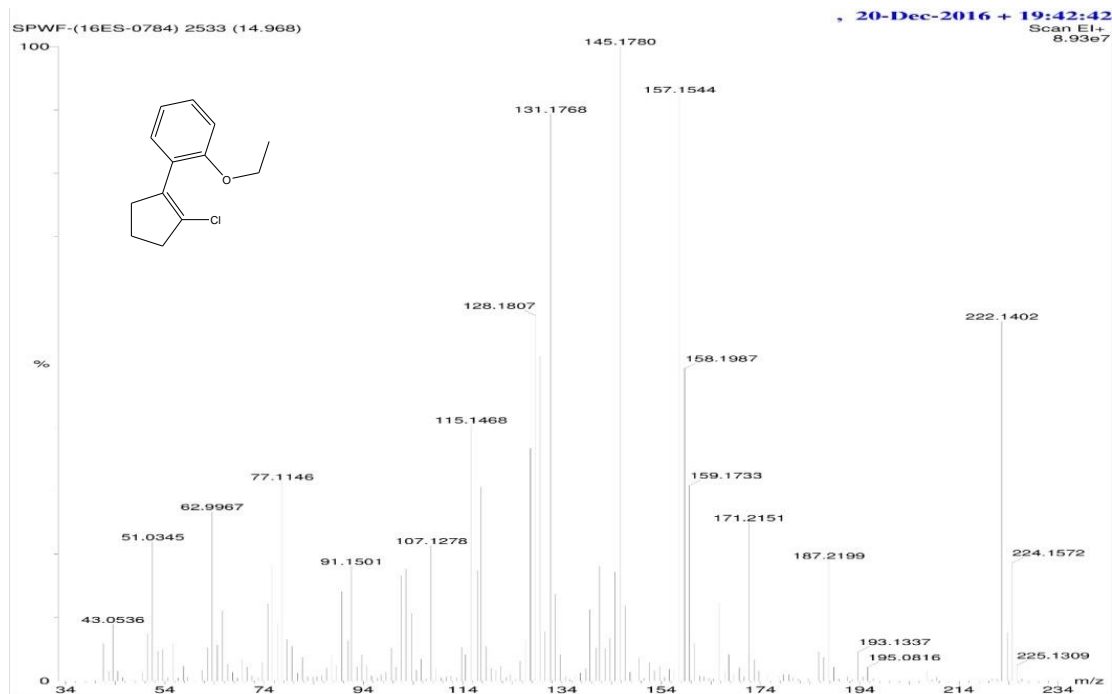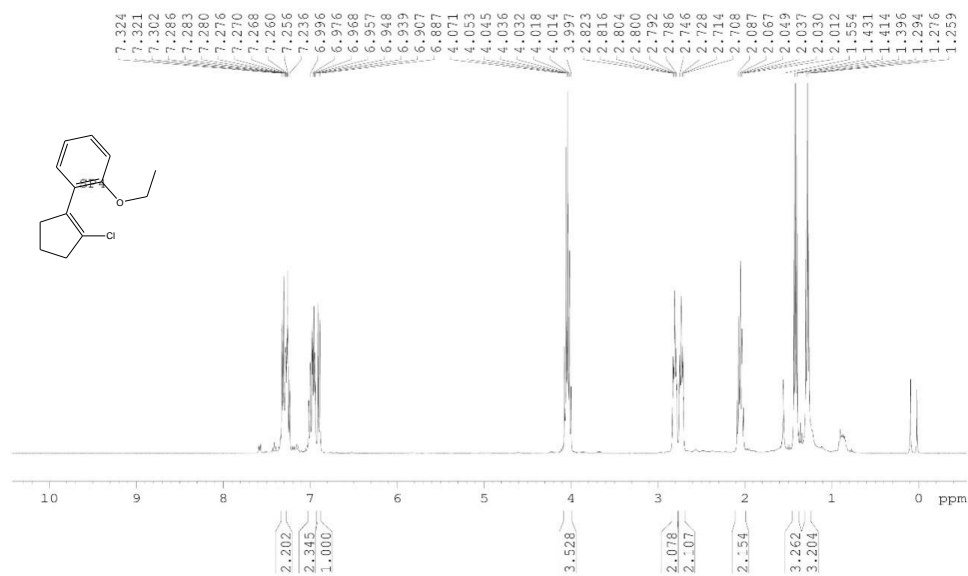**IR of 1-chloro-2-(2′-ethoxyphenyl)cyclopentene 3b:**

MS of 1-chloro-2-(2′-ethoxyphenyl)cyclopentene 3b:

¹H NMR of 1-chloro-2-(2′-ethoxyphenyl)cyclopentene 3b:

¹³C NMR of 1-chloro-2-(2′-ethoxyphenyl)cyclopentene 3b:

IR of 1-chloro-2-(4′-methylphenyl)cyclopentene 3c:

MS of 1-chloro-2-(4′-methylphenyl)cyclopentene 3c:
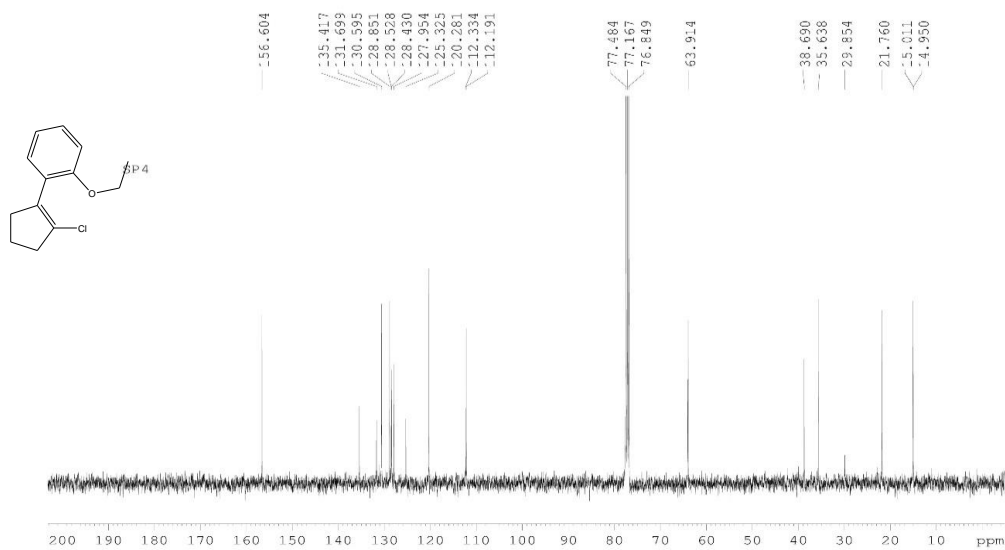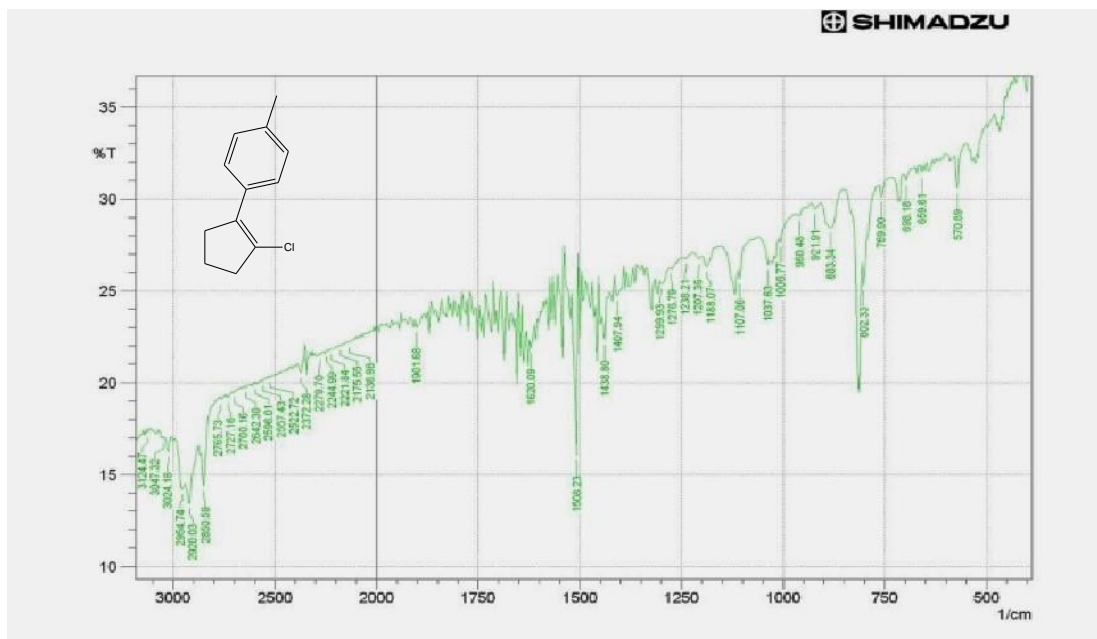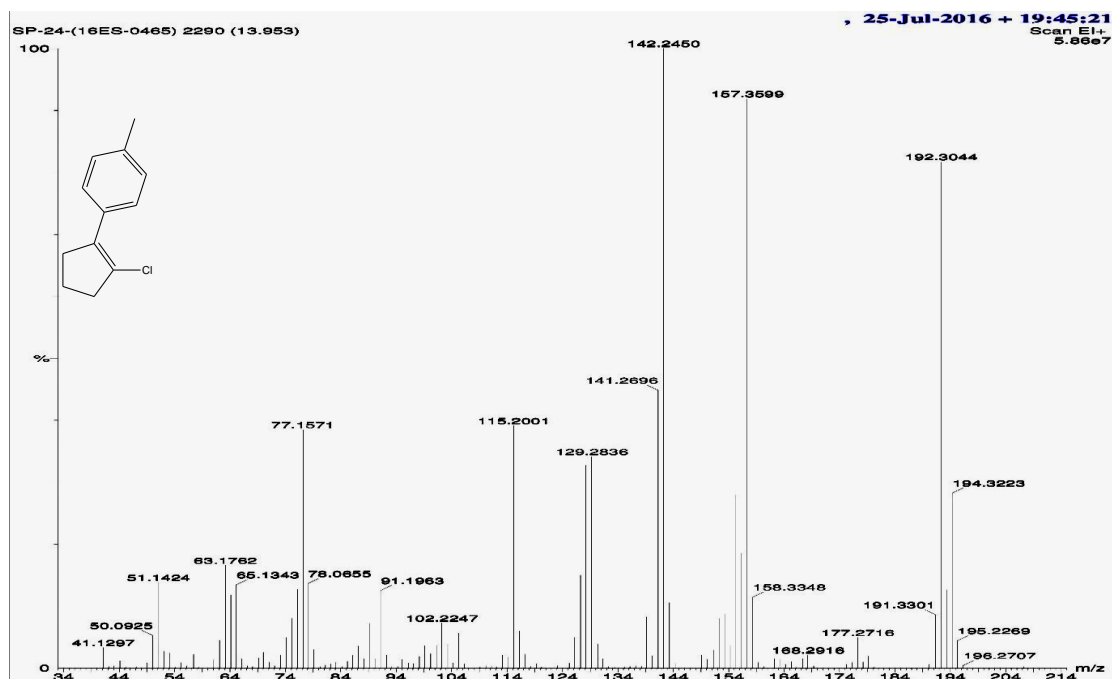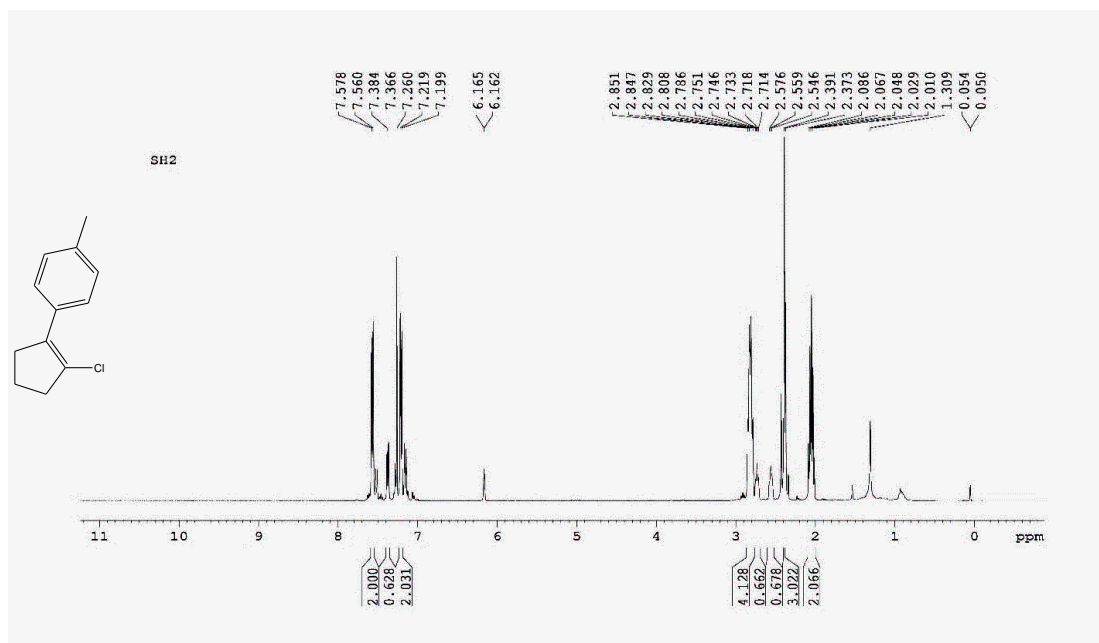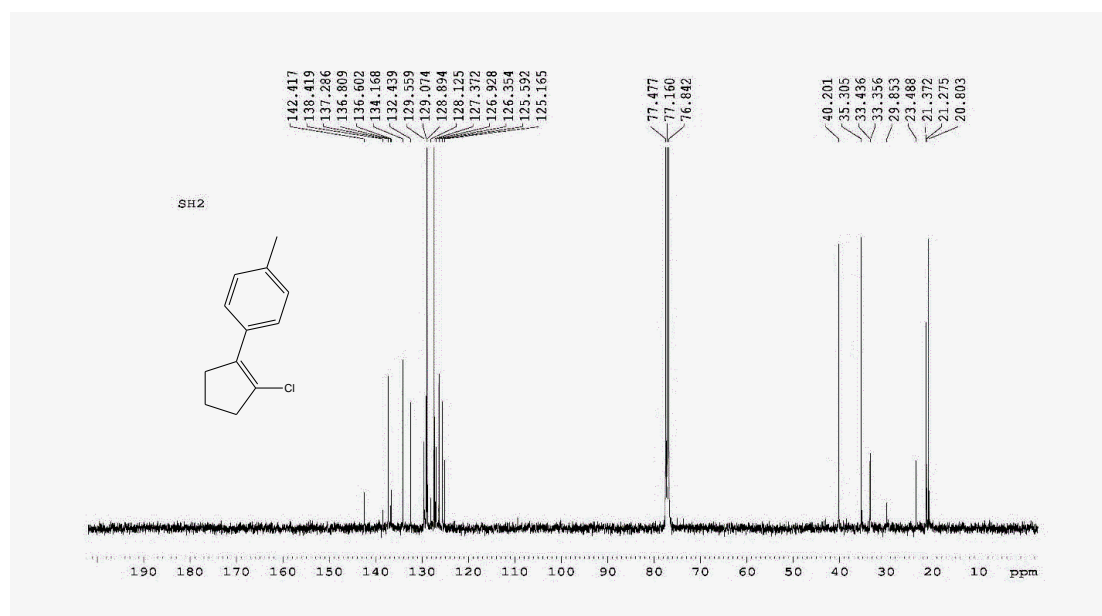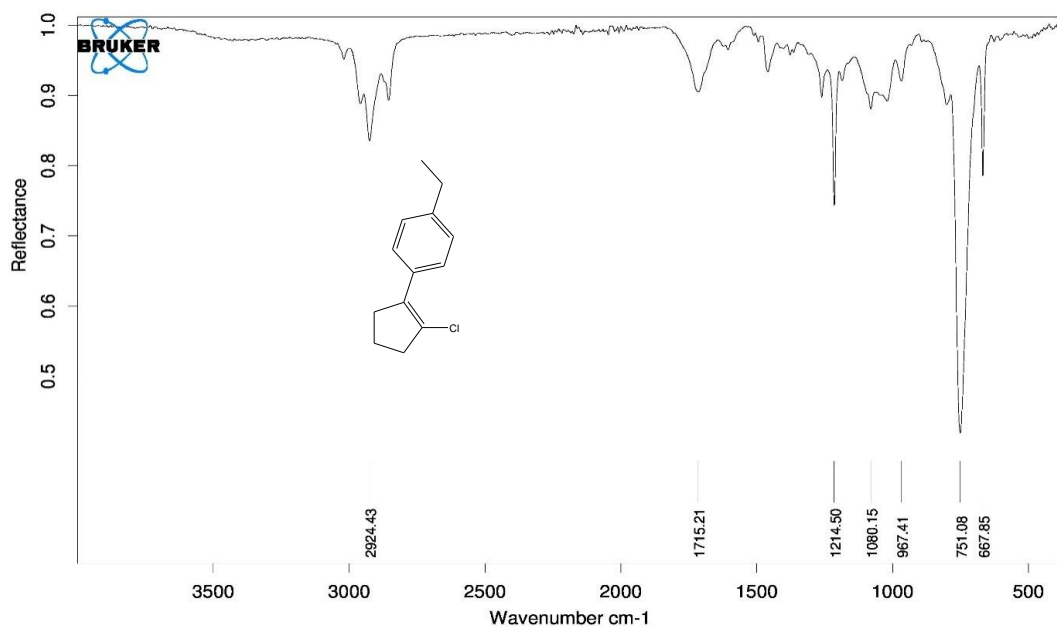
^{1}H NMR of 1-chloro-2-(4′-methylphenyl)cyclopentene 3c:

SH2

7.578
7.560
7.384
7.366
7.260
7.219
7.199
6.165
6.162
2.851
2.847
2.829
2.808
2.786
2.751
2.746
2.733
2.718
2.714
2.576
2.559
2.546
2.391
2.373
2.086
2.067
2.048
2.029
2.010
1.309
0.054
0.050

2.000
0.628
2.031

4.128
0.662
0.678
3.022
2.066

11 10 9 8 7 6 5 4 3 2 1 0 ppm

^{13}C NMR of 1-chloro-2-(4′-ethoxyphenyl)cyclopentene 3c:

SH2

142.417
138.419
137.286
136.809
136.602
134.168
132.439
129.559
129.074
128.894
128.125
127.372
126.928
126.354
125.592
125.165

77.477
77.160
76.842

40.201
35.305
33.436
33.356
29.853
23.488
21.372
21.275
20.803

190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

IR of 1-chloro-2-(4′-ethylphenyl)cyclopentene 3d:

MS of 1-chloro-2-(4′-ethylphenyl)cyclopentene 3d:
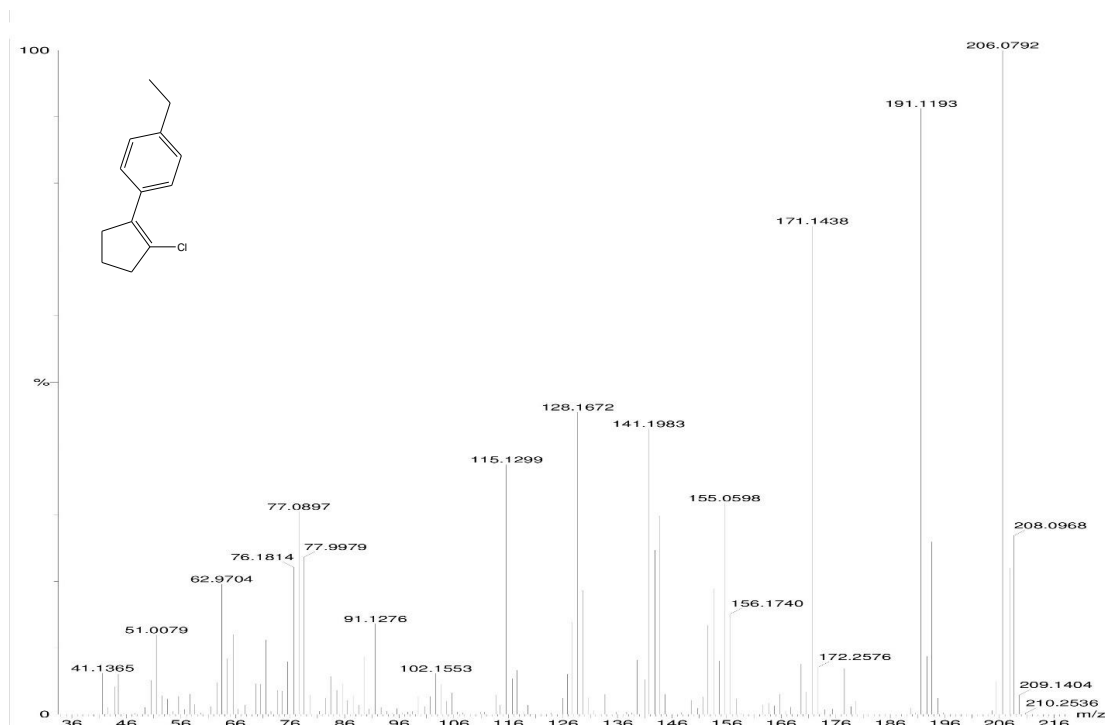
¹H NMR of 1-chloro-2-(4′-ethylphenyl)cyclopentene 3d:
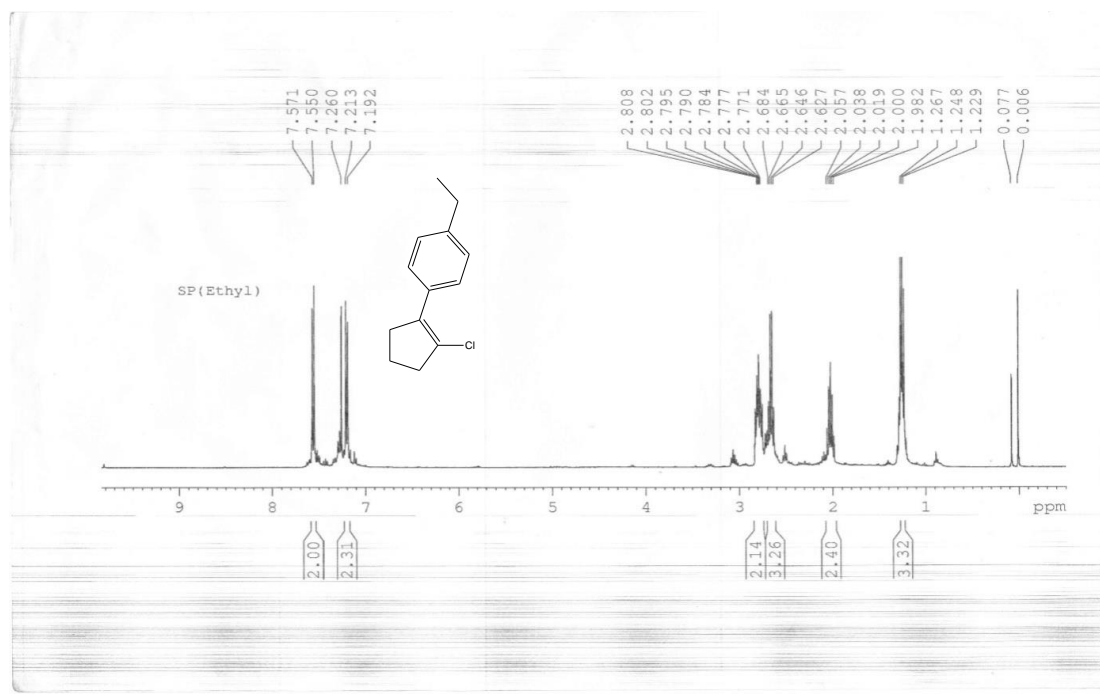
¹³C NMR of 1-chloro-2-(4′-ethylphenyl)cyclopentene 3d:
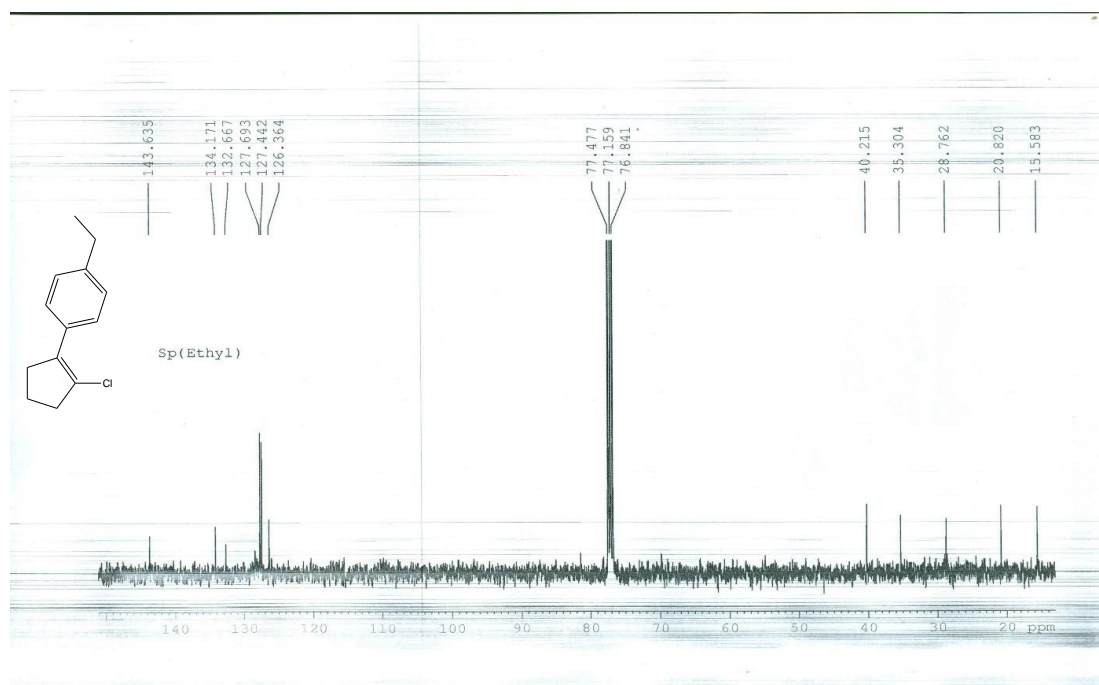
IR of 1-chloro-2-(4′-tertiatybutylphenyl)cyclopentene 3e:

MS of 1-chloro-2-(4′-tertiatybutylphenyl)cyclopentene 3e:

^{1}H NMR of 1-chloro-2-(4′-tertiatybutylphenyl)cyclopentene 3e:

^{13}C NMR of 1-chloro-2-(4′-tertiatybutylphenyl)cyclopentene 3e:

IR of 1-chloro-2-(4′-ethylsulfanephenyl)cyclopentene 3f:
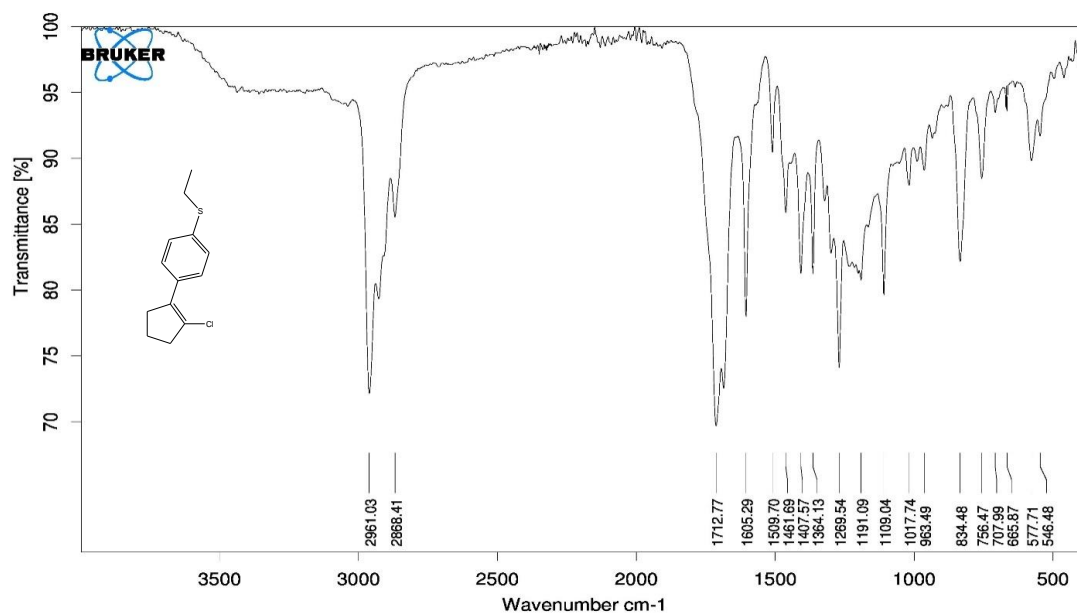
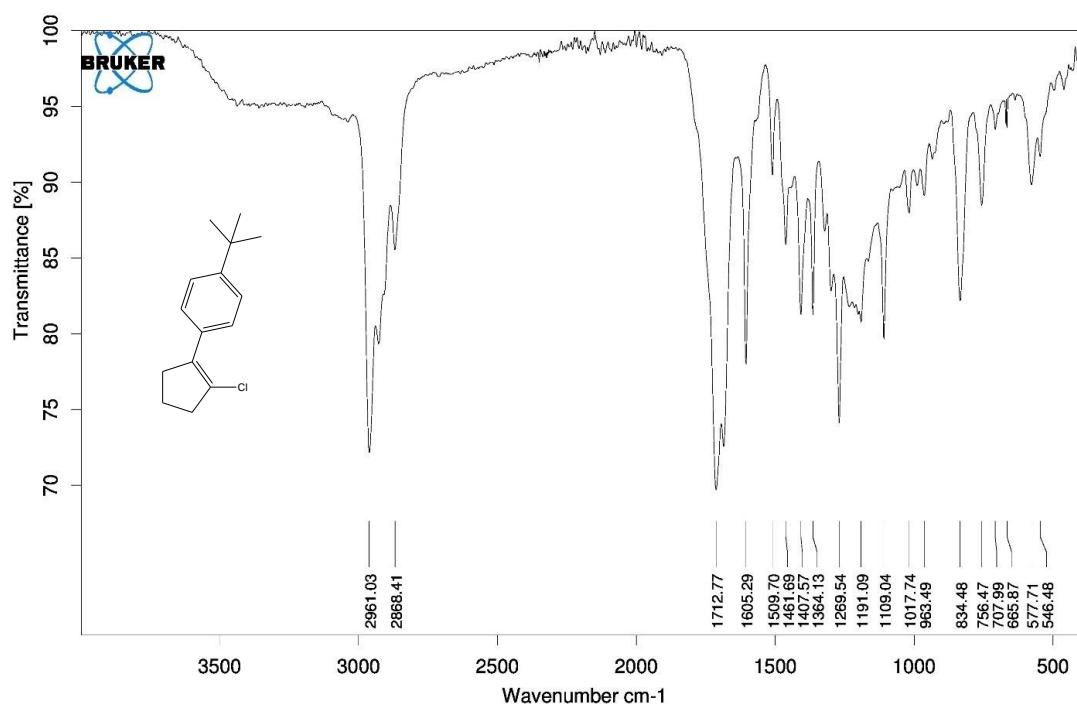**MS of 1-chloro-2-(4′-ethylsulfanephenyl)cyclopentene 3f:**
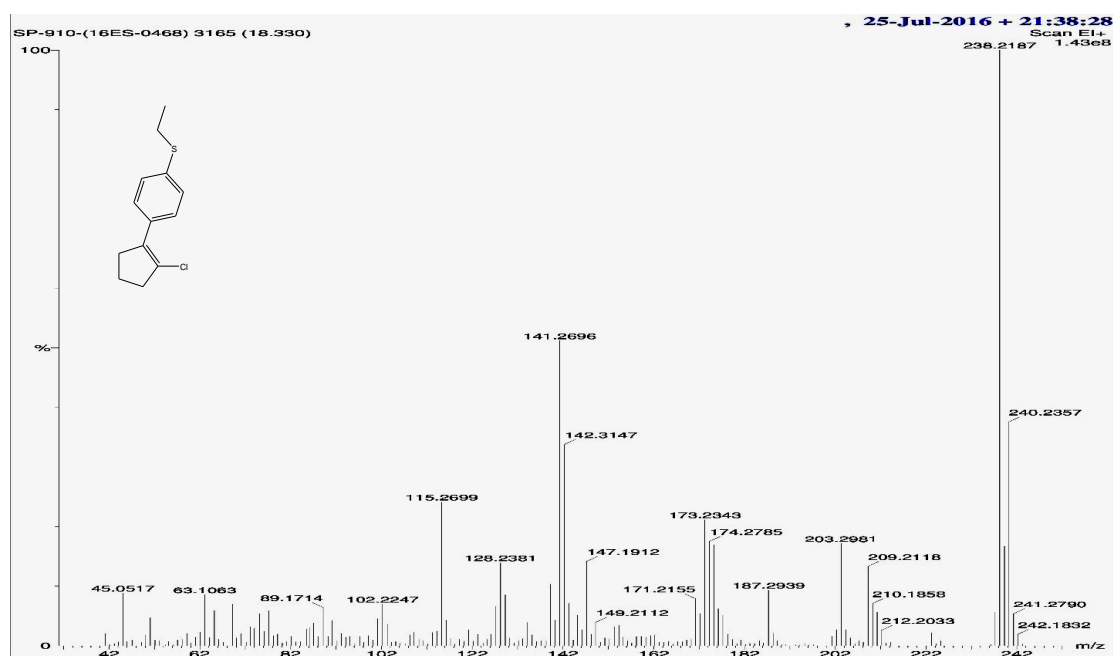
¹H NMR of 1-chloro-2-(4′-ethylsulfanephenyl)cyclopentene 3f:
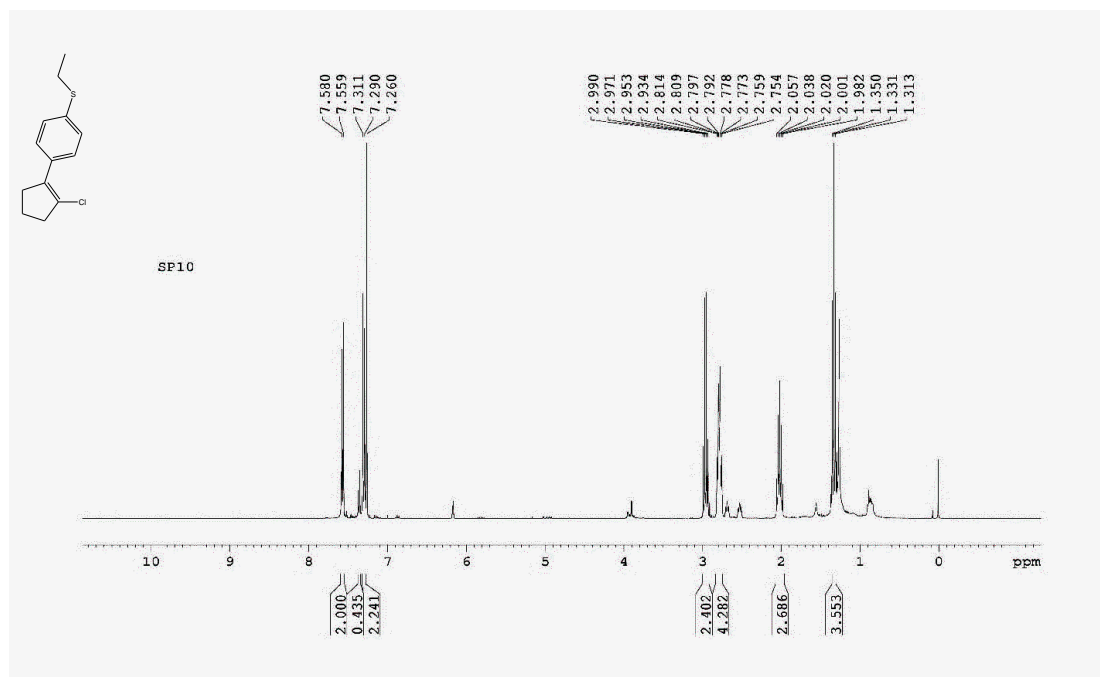
IR of 1-chloro-2-(4′-ethylsulfanephenyl)cyclopentene 3f:
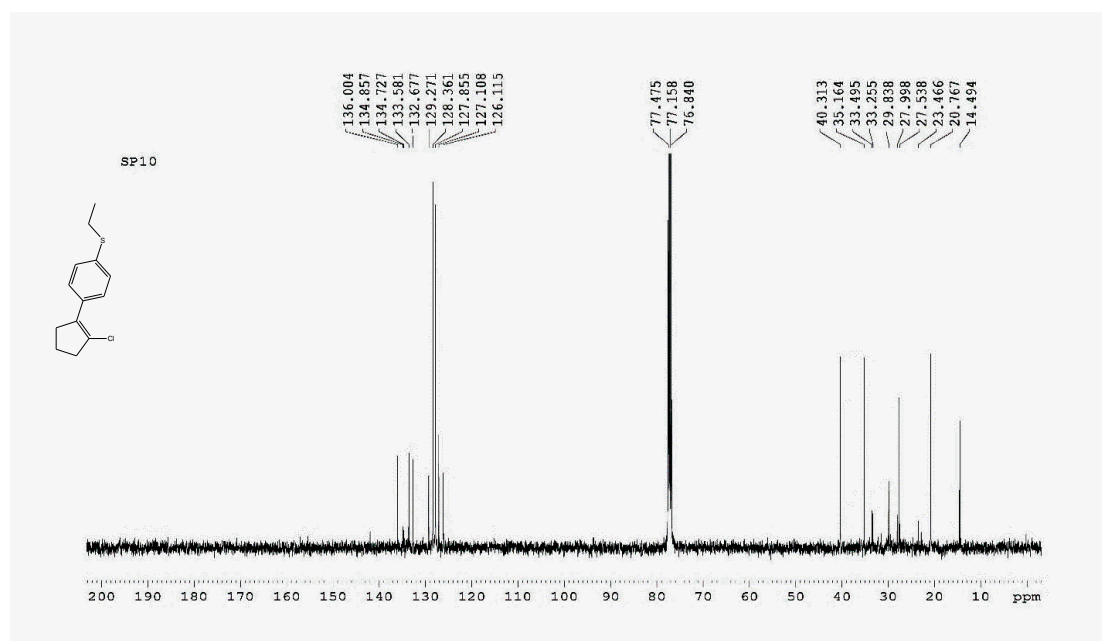
IR of 1-chloro-2-(4′-ethoxyphenyl)cyclopentene 3g:
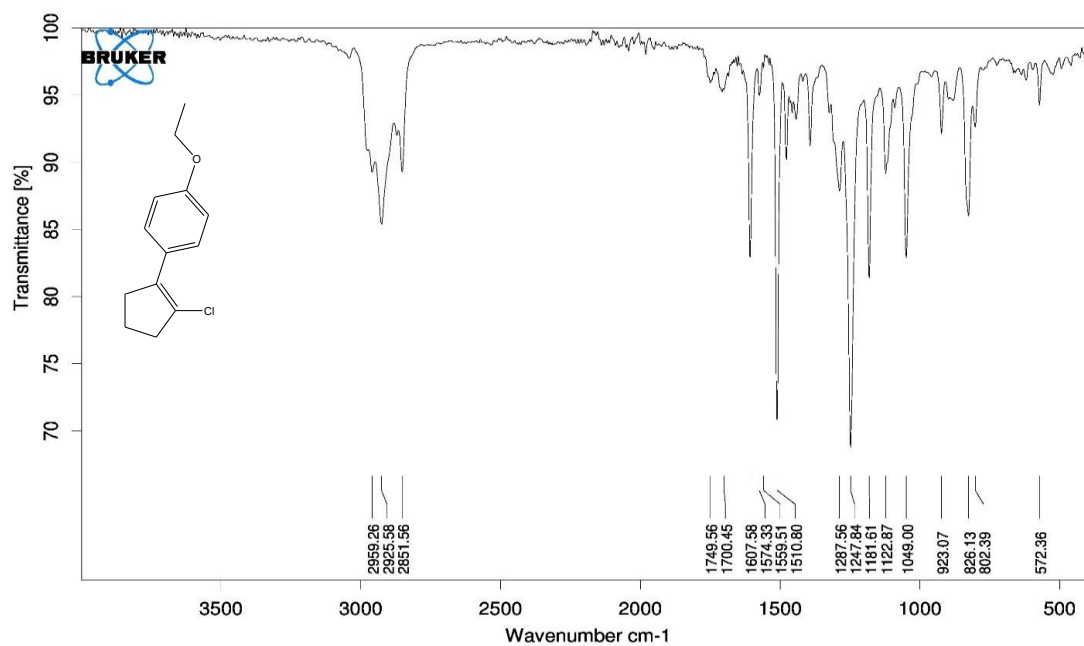
MS of 1-chloro-2-(4′-ethoxyphenyl)cyclopentene 3g:
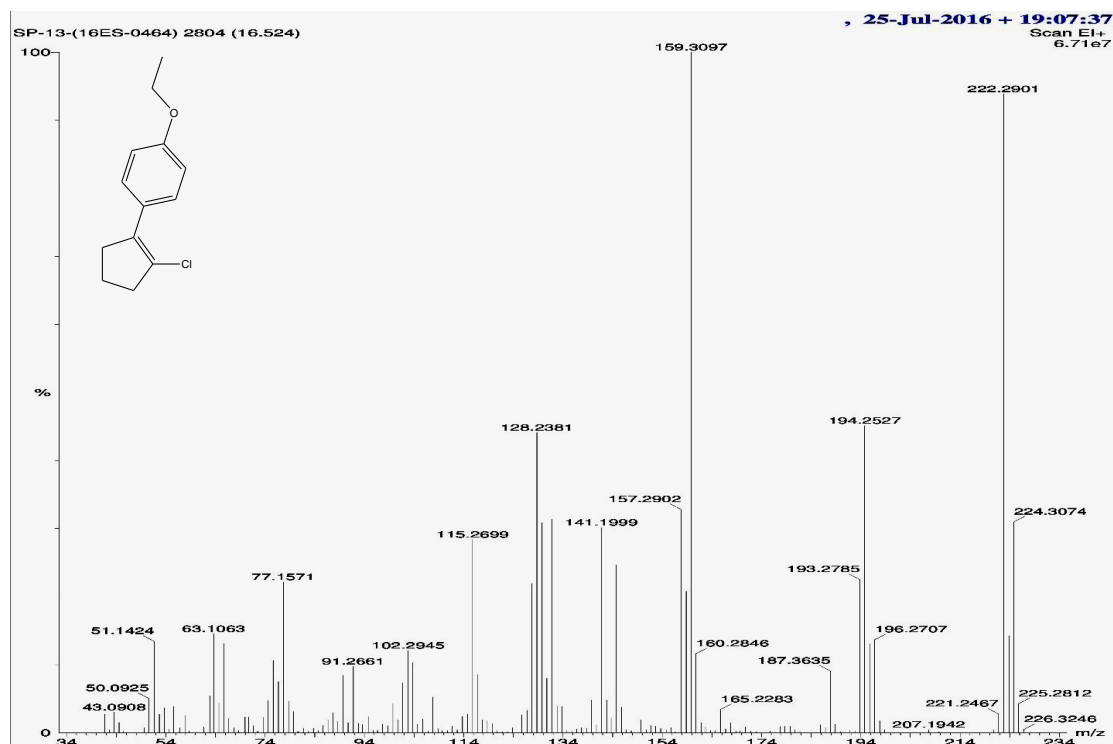
^{1}H NMR of 1-chloro-2-(4′-ethoxyphenyl)cyclopentene 3g:
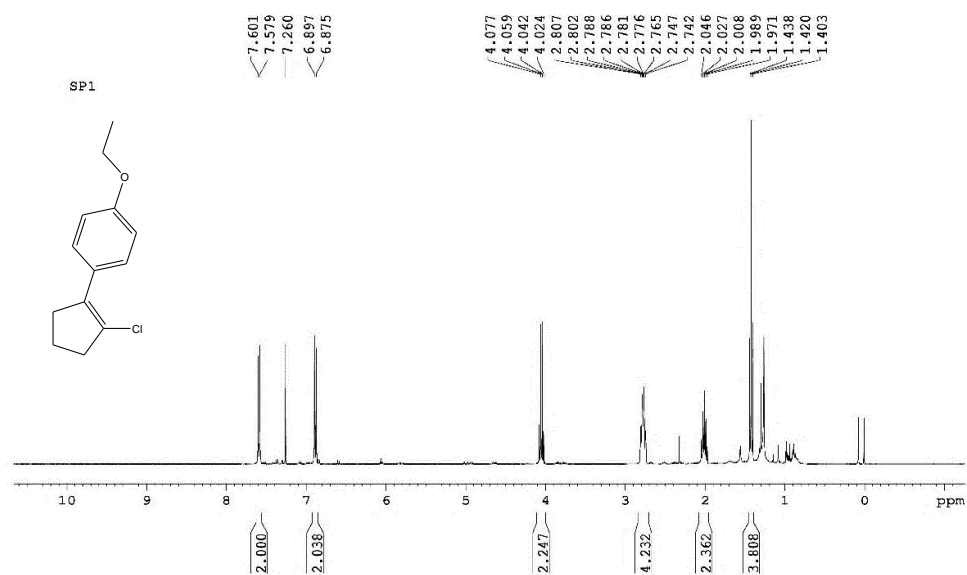
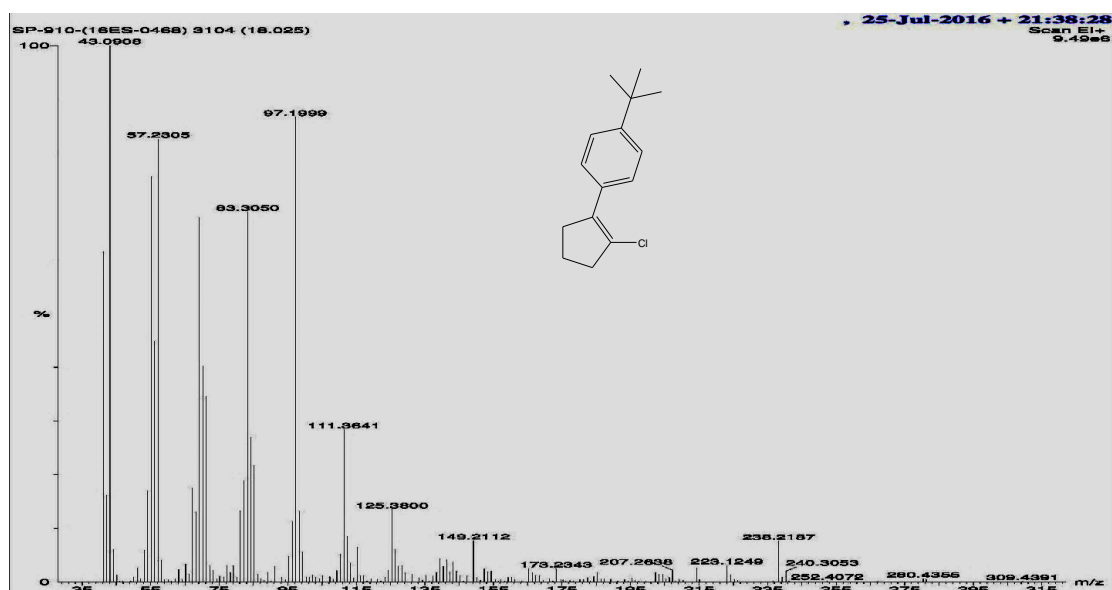# ^{13}C NMR of 1-chloro-2-(4′-ethoxyphenyl)cyclopentene 3g:
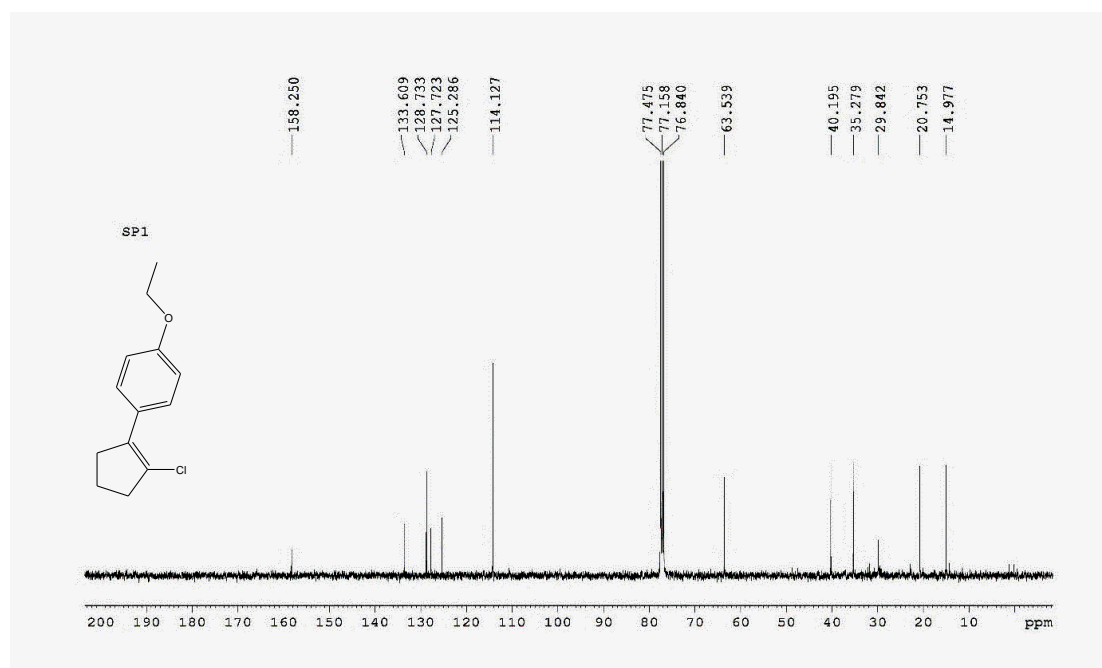
IR of 1-chloro-2-(3′-methylphenyl)cyclopentene 3h:

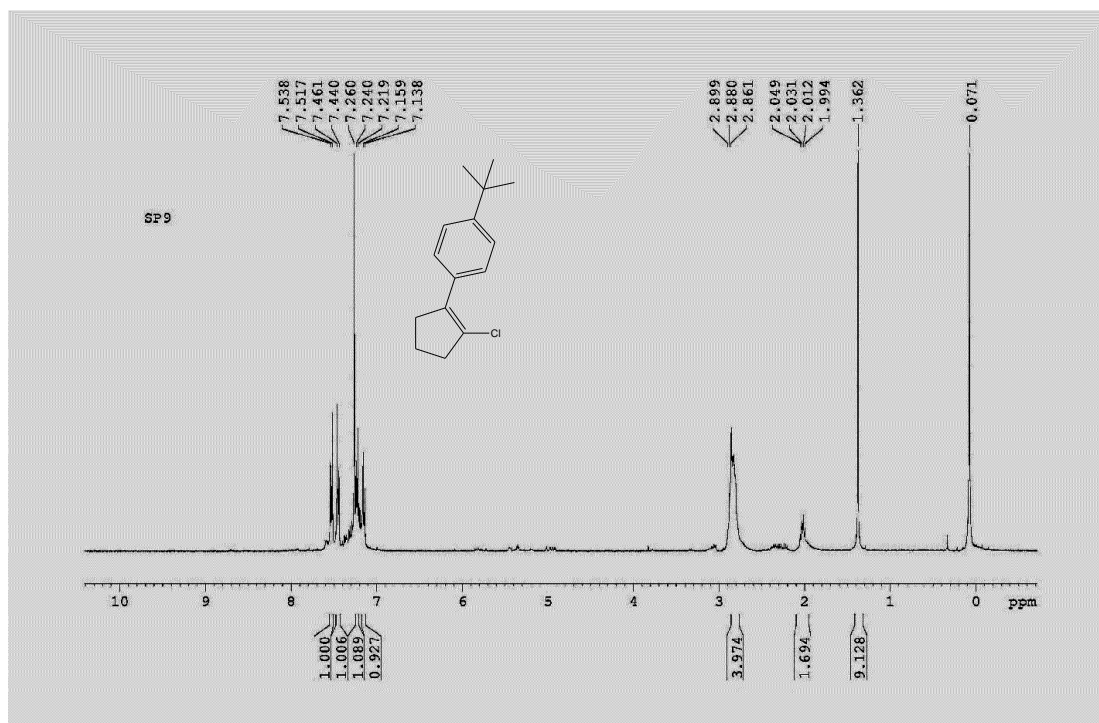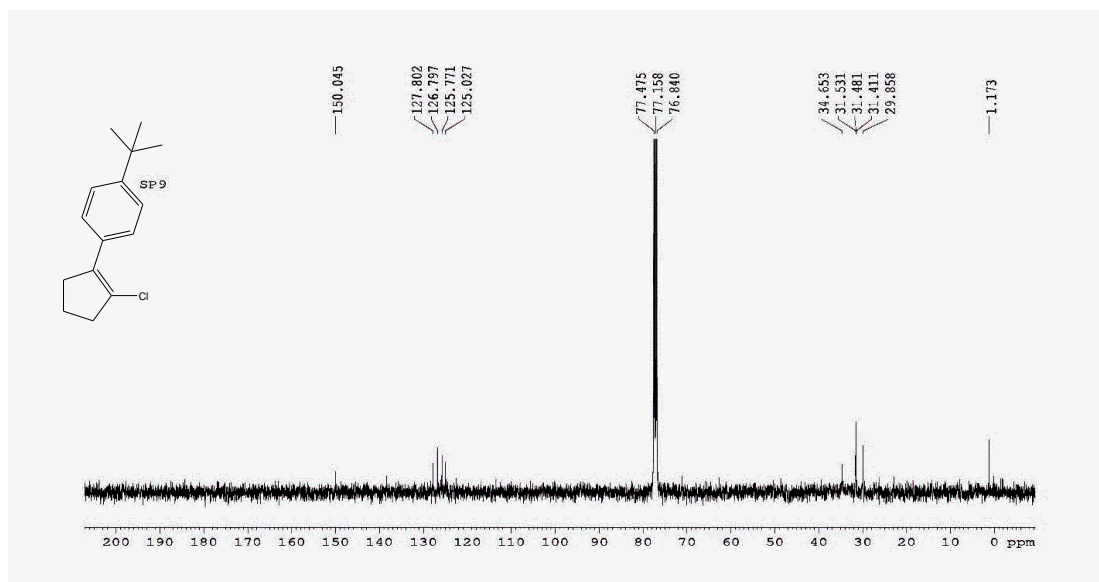**MS of 1-chloro-2-(3′-methylphenyl)cyclopentene 3h:**
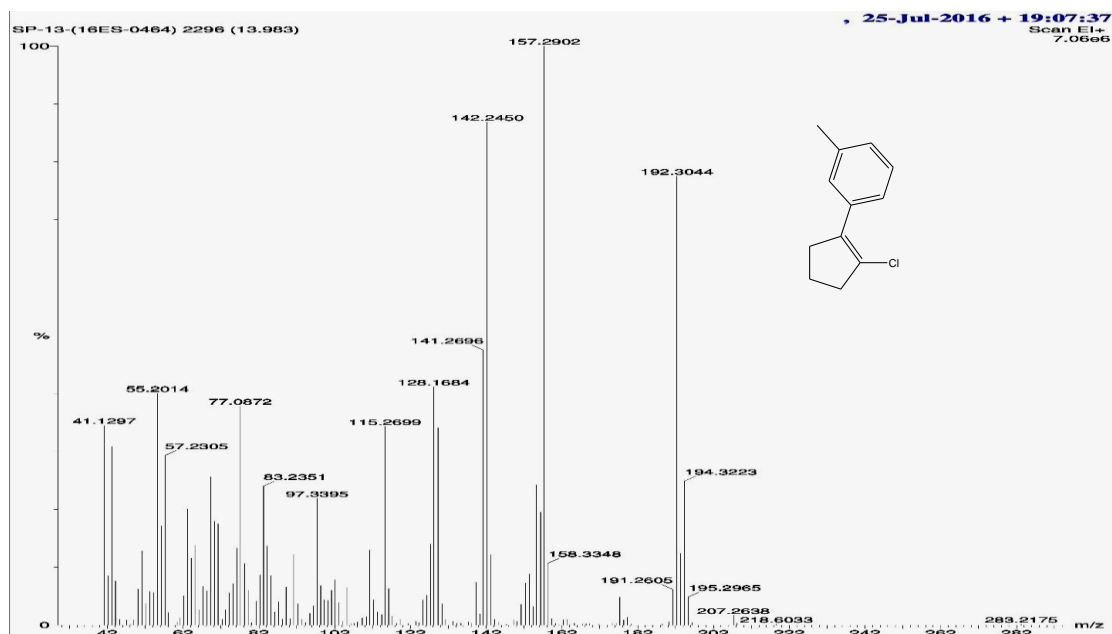
¹H NMR of 1-chloro-2-(3′-methylphenyl)cyclopentene 3h:
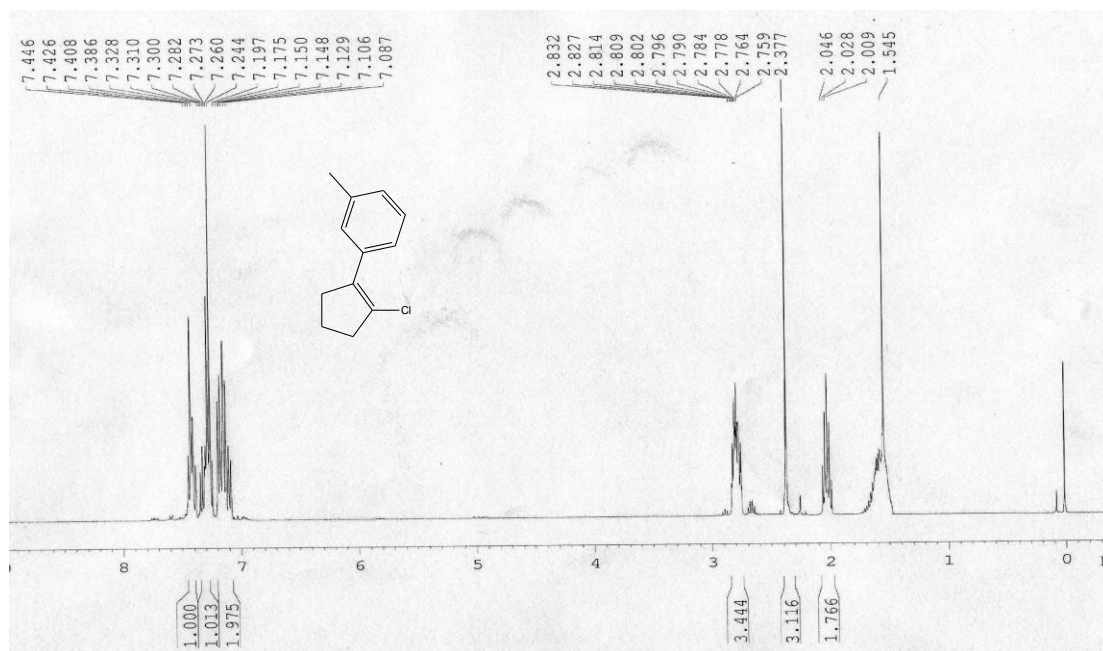
¹³C NMR of 1-chloro-2-(3′-methylphenyl)cyclopentene 3h:
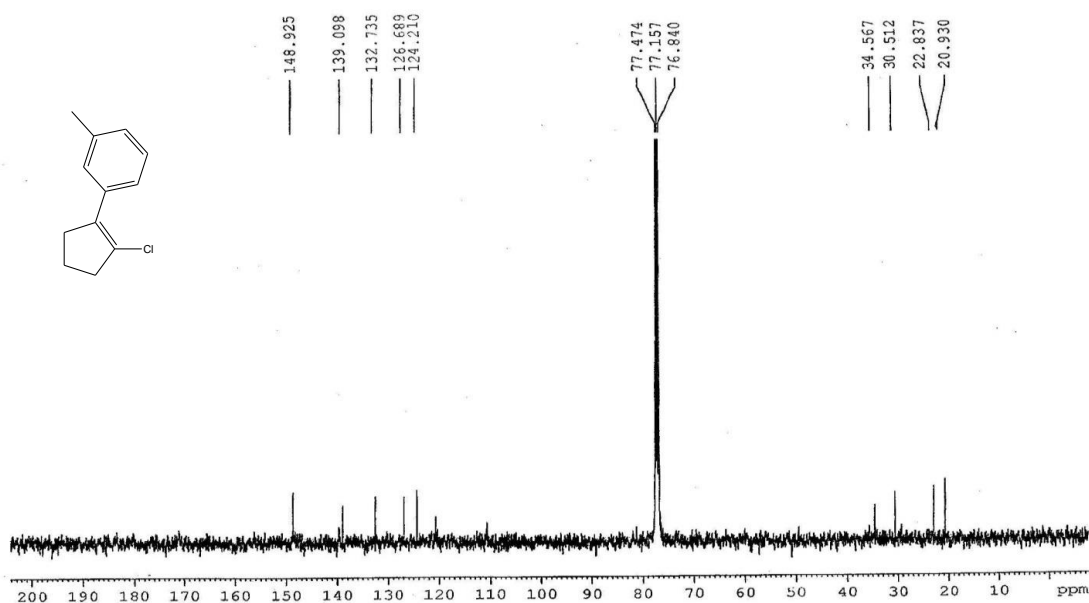
IR of 1-chloro-2-(3′,4′-dimethoxyphenyl)cyclopentene 3i:
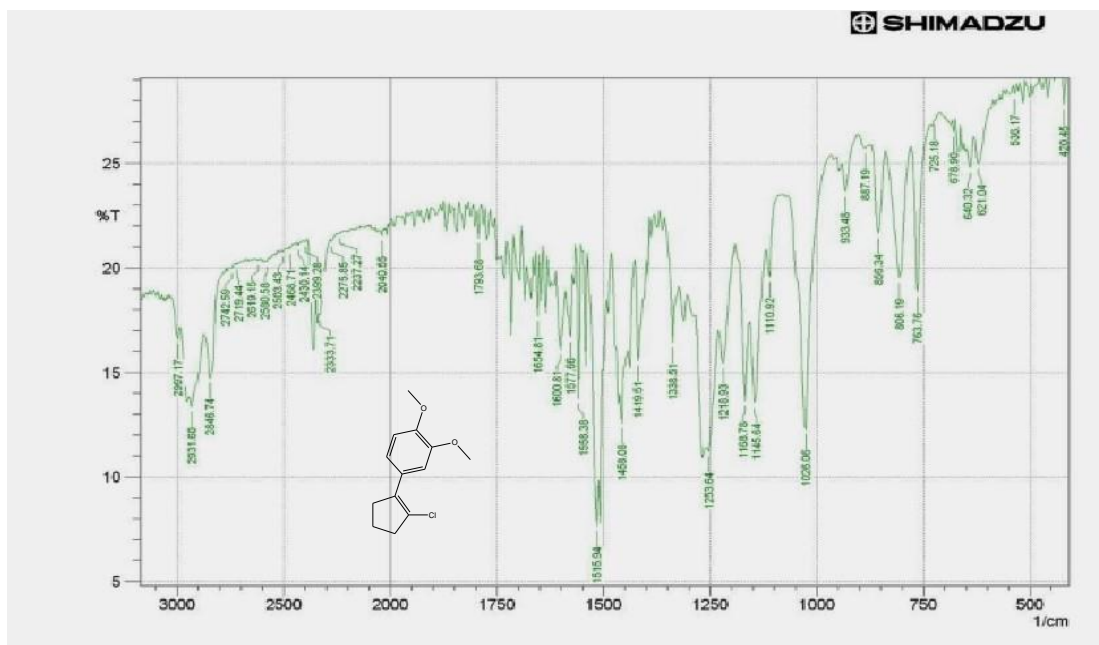
MS of 1-chloro-2-(3′,4′-dimethoxyphenyl)cyclopentene 3i:
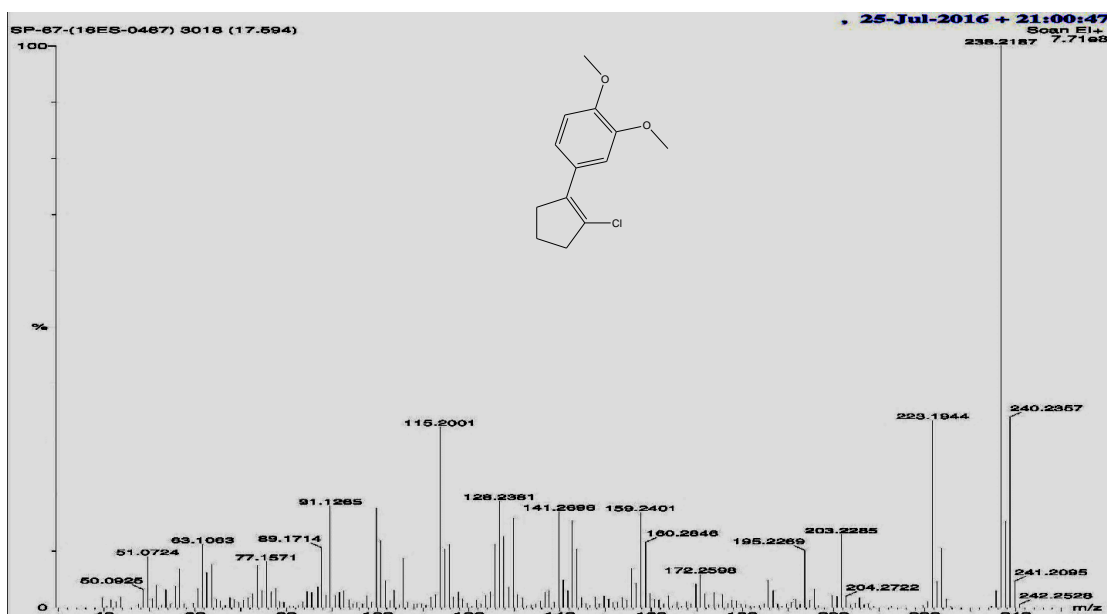
^{1}H NMR of 1-chloro-2-(3′,4′-dimethoxyphenyl)cyclopentene 3i:

SP7

7.343 7.338 7.260 7.137 7.132 7.116 7.111 6.860 6.839 6.817 6.796

3.893 3.881 3.868 2.810 2.807 2.805 2.789 2.768 2.749 2.745 2.705 2.700 2.692 2.685 2.682 2.667 2.662 2.538 2.532 2.519 2.515 2.501 2.495 2.044 2.025 2.006 1.988 1.968

0.700 0.714 1.100   0.337   3.083 3.205   2.865 0.741 0.725   2.198

^{13}C NMR of 1-chloro-2-(3′,4′-dimethoxyphenyl)cyclopentene 3i:

SP7

148.763 148.380 148.302 142.098 133.582 130.117 128.044 125.571 124.391 120.209 118.210 110.974 110.755 110.720 108.841

77.477 77.359 77.159 76.841

55.869

40.171 35.210 33.413 33.319 29.749 23.443 20.629
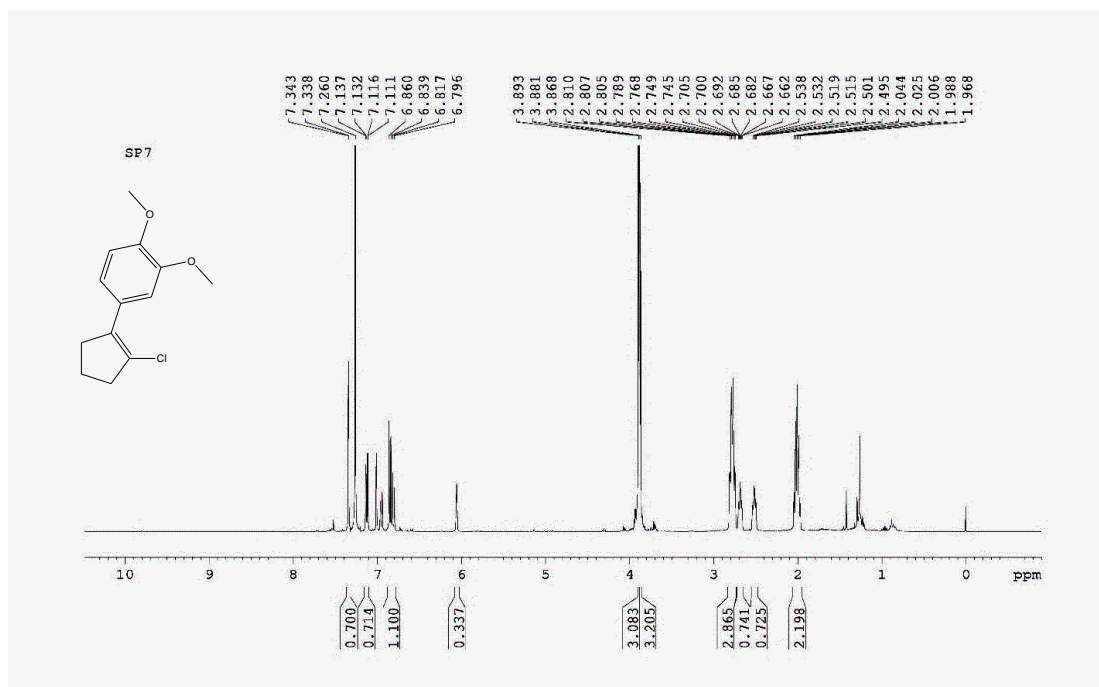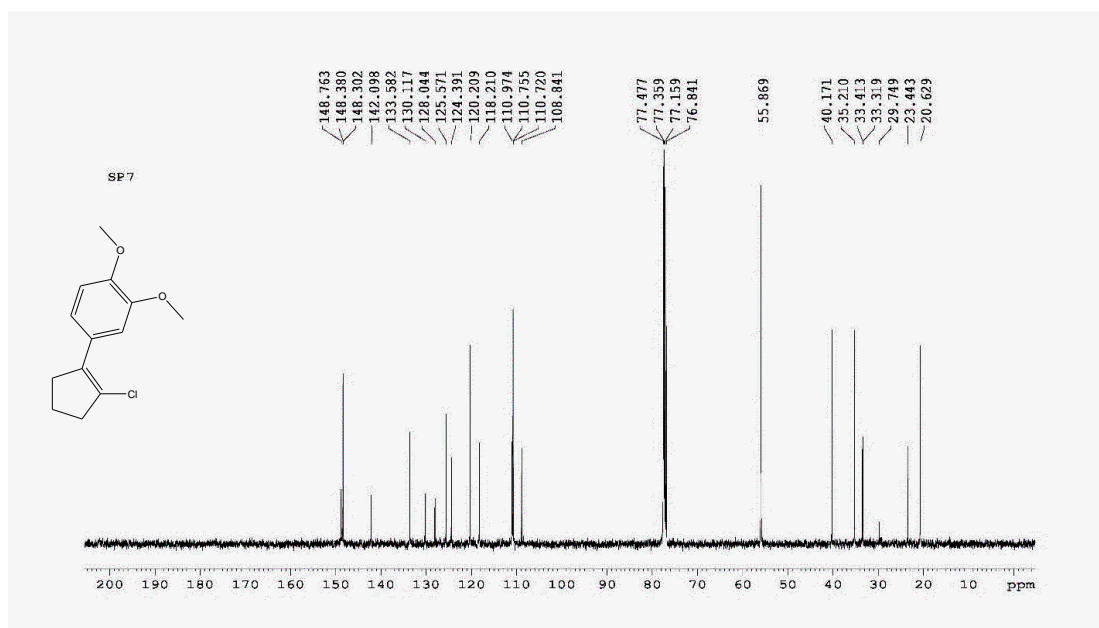
IR of 1-chloro-2-(4′-biphenyl)cyclopentene 3j:
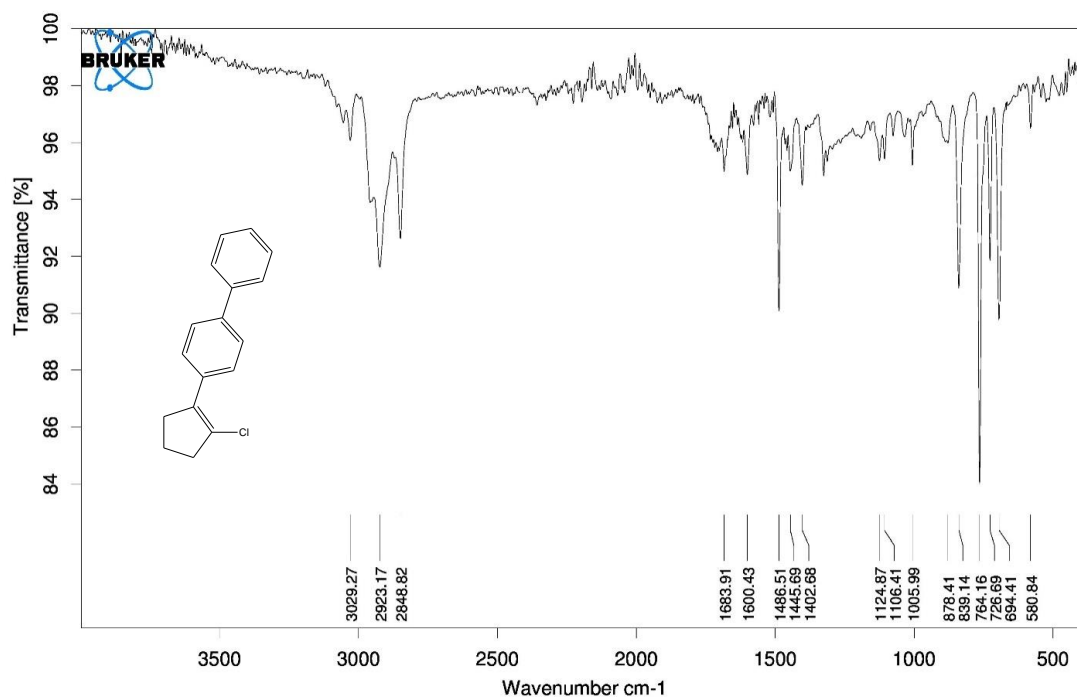
MS of 1-chloro-2-(4′-biphenyl)cyclopentene 3j:

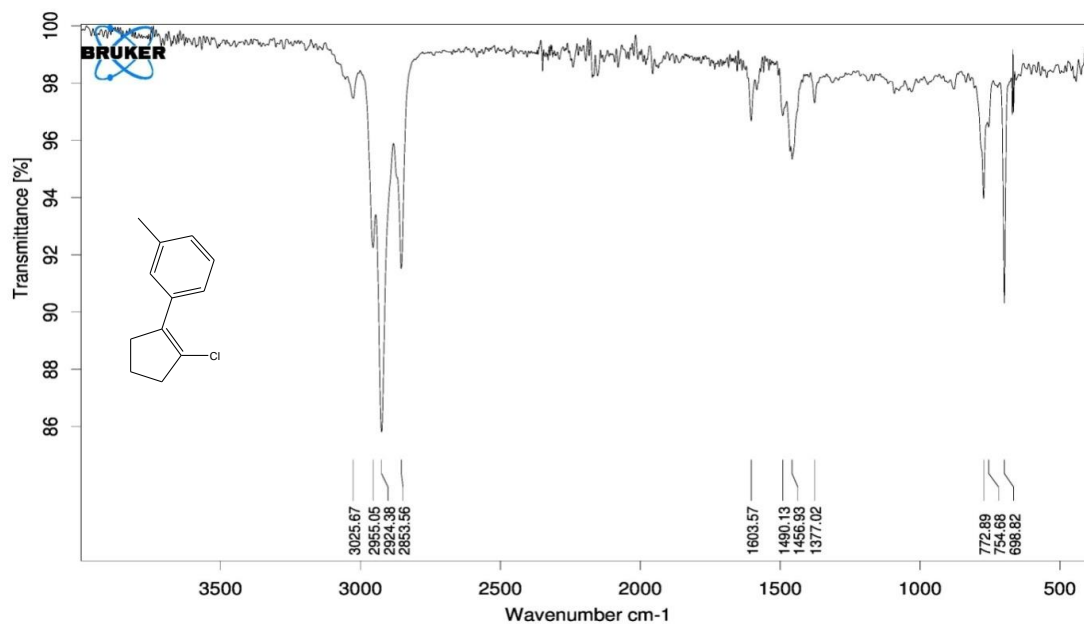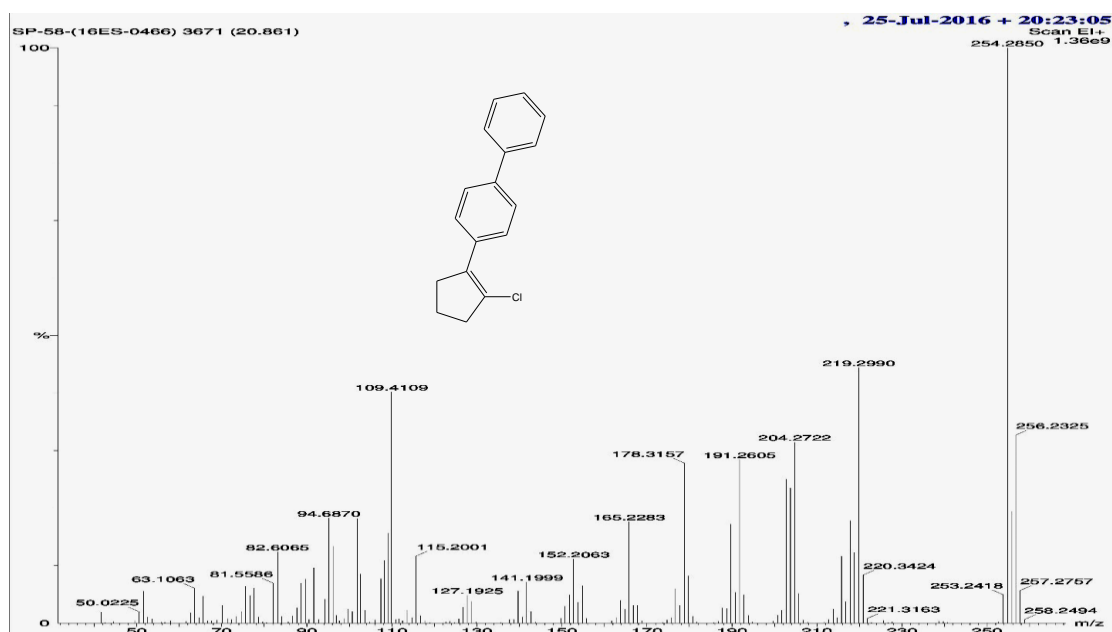## ¹H NMR of 1-chloro-2-(4′-biphenyl)cyclopentene 3j:
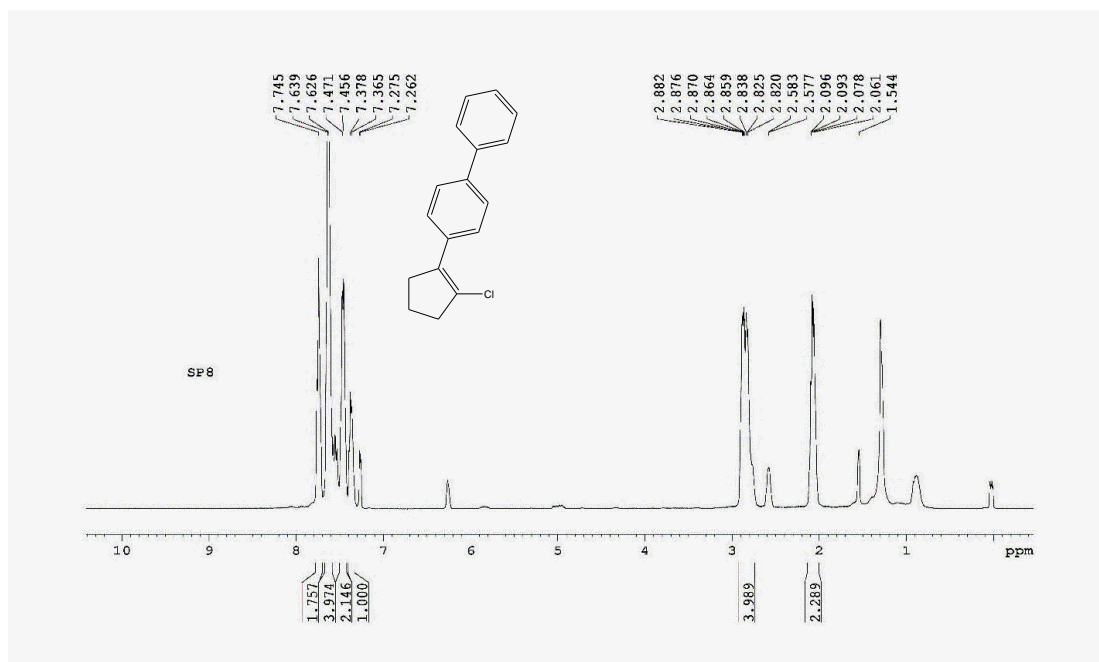
¹³C NMR of 1-chloro-2-(4′-biphenyl)cyclopentene 3j:
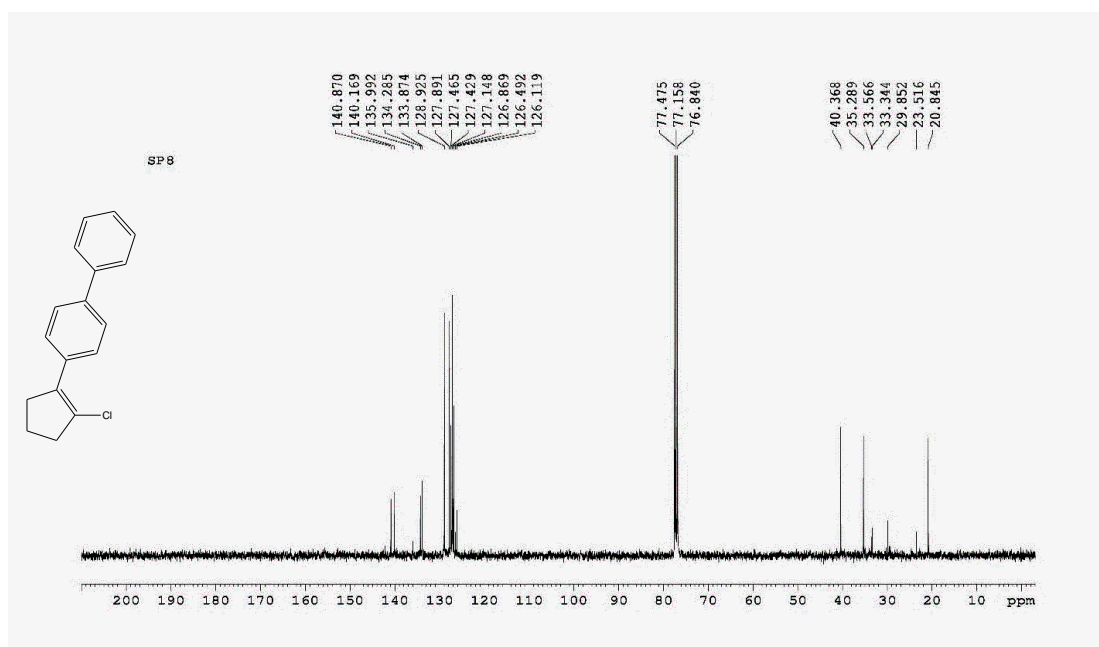
IR of 1-chloro-2-(1-napthyl)cyclopentene 3k:

MS of 1-chloro-2-(1-napthyl)cyclopentene 3k:

¹H NMR of 1-chloro-2-(1-napthyl)cyclopentene 3k:
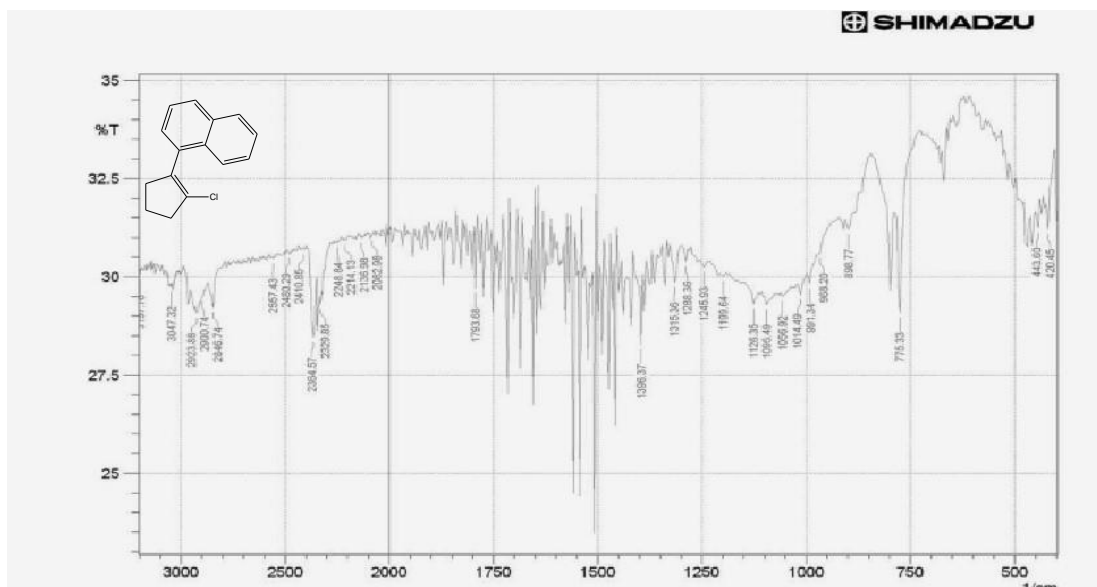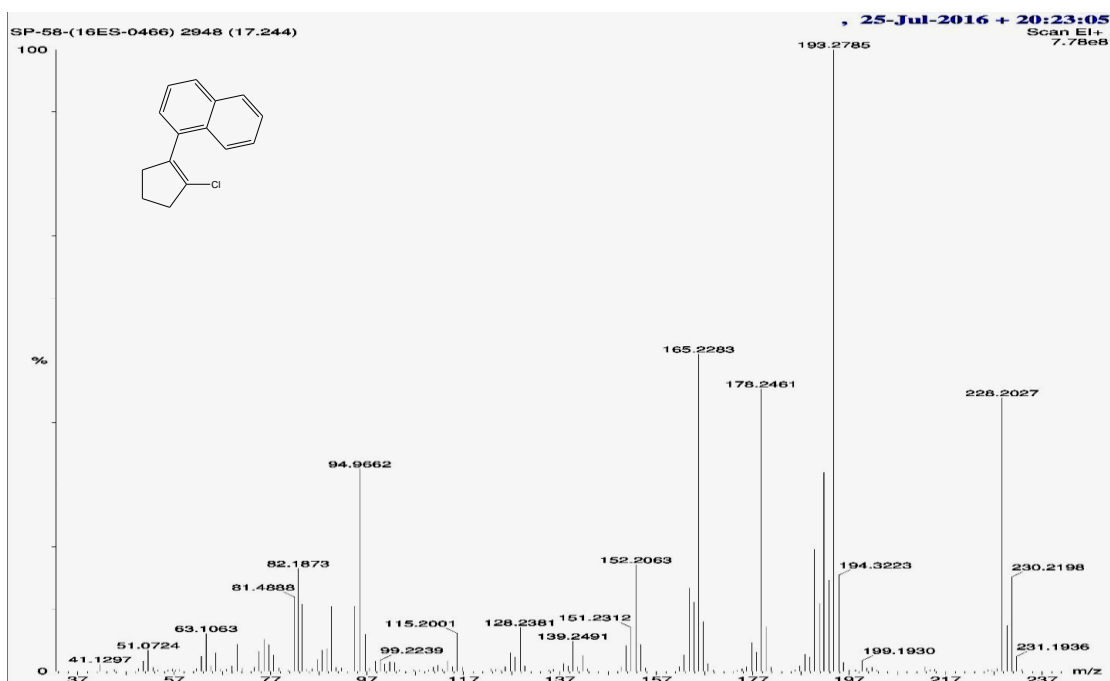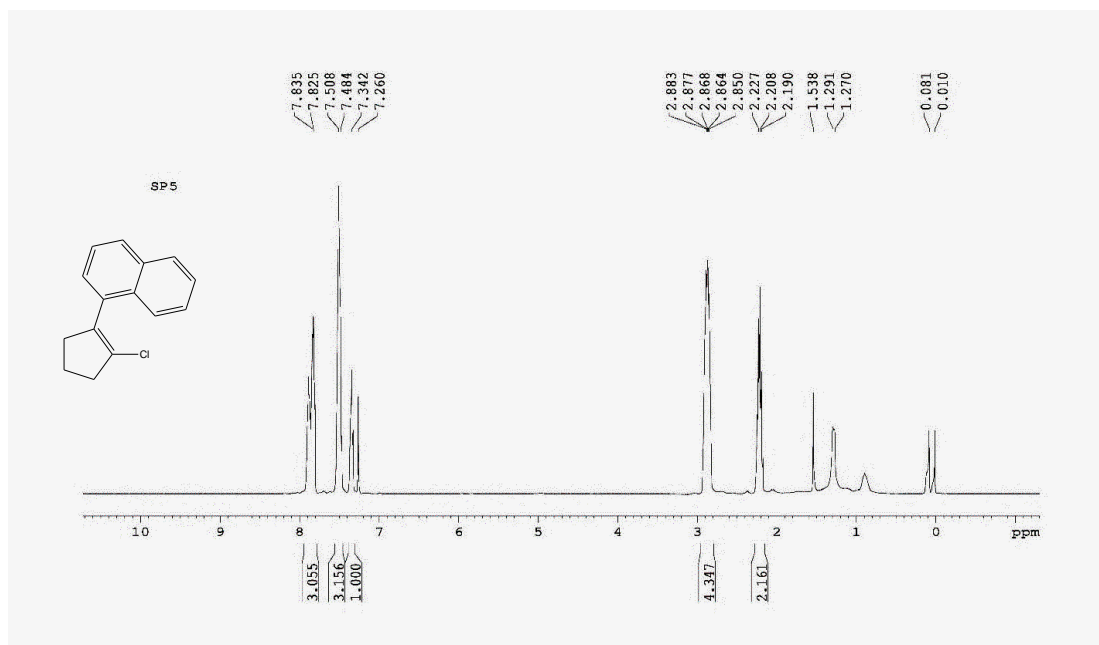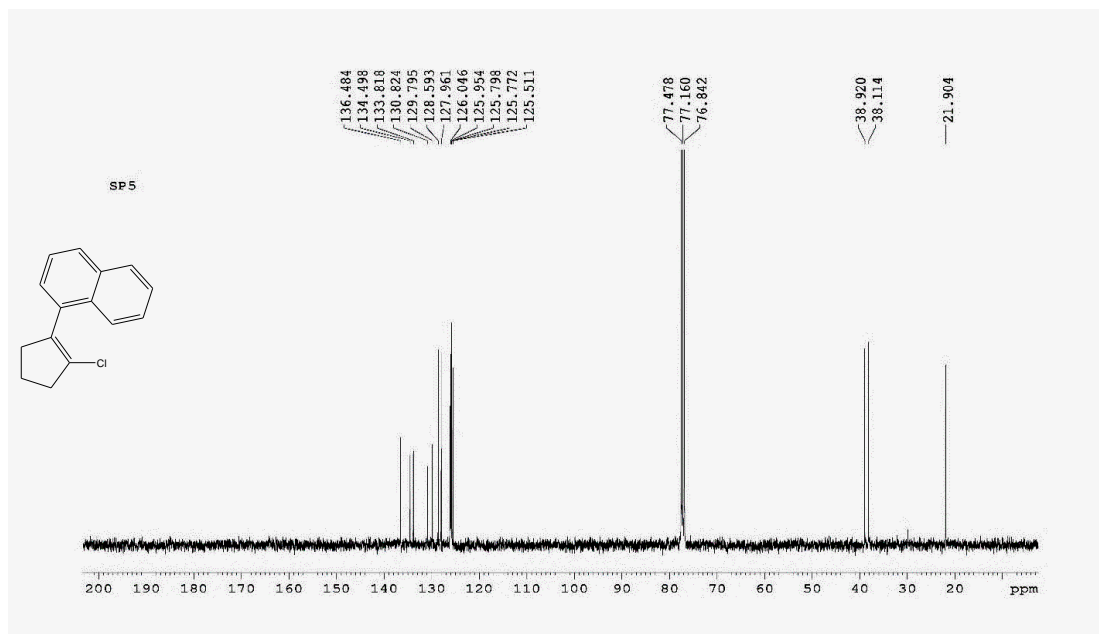

¹³C NMR of 1-chloro-2-(1-napthyl)cyclopentene 3k:

X-ray structure analysis

To our knowledge there exist no literature reports for the single crystal XRD analysis of 1-chloro-2-aryl-substituted cyclopentenes (**3**). We now report the XRD crystal structures of some representative 1-chloro-2-arylcyclopentens. Good quality crystals of the 1-chloro-2-aryl compounds **3j**, and **3k** were obtained by slow evaporation technique. Suitable crystals of the chlorine bearing compounds **3j** and **3k** of appropriate quality and size for single crystal X-ray diffraction were obtained from slow evaporation method using hexane at room temperature. A good quality single crystal in each case was mounted along its largest dimension and used for data collection. The intensity data were collected on a Bruker Smart CCD Area Detector System using MoKα (0.7103Å) radiation in ω – φ scan mode. The data were reduced using SAINT-Plus.[1] The structures in each case was solved by Direct Methods and refined on F^2 using SHELX-97[2] software package. All the non-hydrogen atoms were refined anisotropically. As the hydrogen atoms were not readily revealed from difference Fourier maps, they were included in the ideal positions with fixed isotropic U values, and they were riding with their respective non-hydrogen atoms. The difference Fourier map, after the refinement, was essentially featureless in all the cases. The mean plane calculations were done using the program PARST.[3] Diagrams were generated using ORTEP-3,[4] PLATON,[5] CAMERON[6] and DIAMOND.[7]

The ORTEP view of the representative molecule **3k** with atomic labeling (thermal ellipsoids drawn at 50% probability) is given in **Figure 2**. Packing of molecules for compounds are shown in **Figures 3** and orientation of the planes containing the ring structures are depicted in **Figure 4. Table 2** gives the interatomic interaction parameters in compounds **3j** and **3k**. Summary of crystallographic data and other structure refinement parameters of the compounds **3j** and **3k** are given in **Table 3**.

Intermolecular features

The compounds **3j** and **3k** are not prospective candidates for any robust weak interactions, as all the molecules are devoid of O, N, F, S atoms that would result in hydrogen bonds and other weak interactions . The only weak interactions observed are C-H···Cl and C-H···Cg (**Table 2**).

Compound **3j** is stabilized by, C10'-H10'…Cg and C7'-H7'…Cg (Cg is the centroid of aryl ring C6-C11) which forms a chain along 'a' axis with a distance of 2.754 Å and 2.753 Å respectively. The C-H…π interactions is seen in compounds **3k.** This interaction, in compound **3k**, C15-H15…Cg (Cg is the centroid of naphthalene ring C7-C16) has a value of 2.697 Å (**Figures 2 and 3**).

Figure 2: ORTEP view of compound **3k** with two molecule in the asymmetric unity, showing 50% probability ellipsoids and the atom-numbering scheme

3k

Figure 3: Packing of the compound **3j** showing intermolecular C-H···π interactions and **3k** showing intermolecular C-H···π
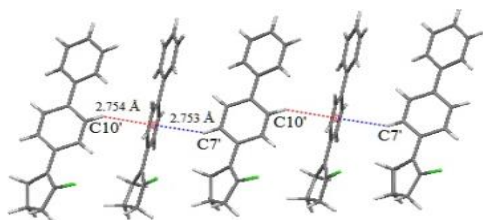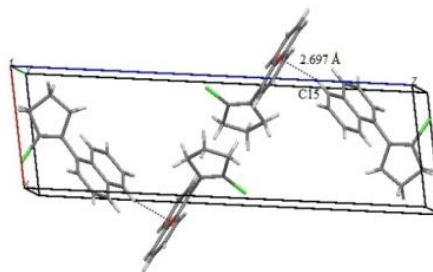
3j   **3k**

Figure 4: The orientation of the ring structures present in the compounds **3j** and **3k**
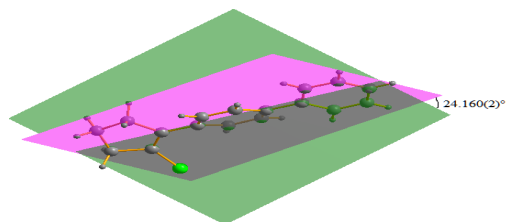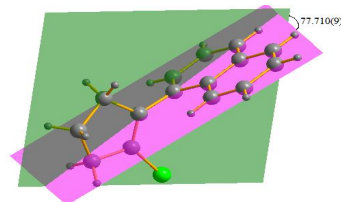
3j   **3k**

The dihedral angles between the planes containing cycloalkenyl and biphenyl **3j** is found to be coplanar, whereas molecule with naphthalene substituted cycloalkenyl ring **3k** is oriented orthogonally.

Table 2: Non-bonded interactions and possible hydrogen bonds in **3j** and **3k** (Å, °)

Compound	D–H···A[a]	D–H	H···A	D···A	D–H···A
3j	C7'-H7'...Cg [i]	0.950 (2)	2.753 (4)	3.595 (2)	148
	C10'-H10'...Cg [ii]	0.950 (2)	2.754 (2)	3.604 (4)	149
3k	C15-H15...Cg [iii]	0.950 (2)	2.697 (3)	3.532 (4)	147

[a] D - donor; A - acceptor; H–hydrogen.

Symmetry codes: (i) -x+1,-y,-z-1, (ii) –x,-y+1,-z, (iii) x+1,+y,+z.

**Table 3:** Crystal data and refinement parameters for compounds **3j** and **3k.**

|	**3j**	**3k**
CCDC No	1553574	1553573
Empirical formula	$C_{17}H_{15}Cl$	$C_{15}H_{13}Cl$
Formula weight	254.74	228.70
T [K]	100(2) K	100(2) K
Λ[Å]	0.71073	0.71073
Crystal system	triclinic	triclinic
Space group	$P\bar{1}$	$P\bar{1}$
a [Å]	9.780(2)	7.4359(5)
b [Å]	10.385(2)	7.6628(5)
c [Å]	13.578(3)	23.3162(16)
α [°]	93.131(6)	81.474(3)
β [°]	107.106(6)	80.862(2)
γ [°]	97.036(7)	61.022(2)
V [Å^3]	1280.4(5)	1143.56(13)
Z	4	4
ρ_{calc} [Mg/m^3]	1.321	1.328
μ [mm^{-1}]	0.276	0.300
F(000)	536	480
Crystal size [mm]	0.18 x 0.16 x 0.16	0.18 x 0.16 x 0.16
θ [°]	2.02 to 25	2.66 to 25.00
Index ranges	-11 ≤ h ≤ 11, -12 ≤ k ≤ 12, -16 ≤ l ≤ 16	-8 ≤ h ≤ 8, -9 ≤ k ≤ 9, -27≤ l ≤ 27
Reflections collected	15246	13789
Independent reflections	4503 [R(int) = 0.0869]	4008 [R(int) = 0.0496]
Completeness to theta	99.8%	99.8 %
	θ = 25.00°	θ = 25.00°
Data/restraints/parameters	4503/0/325	4008 / 0 / 289
Goodness-of-fit on F^2	1.007	0.955
Final R indices [I>2sigma(I)]	R1 = 0.0678, wR2 = 0.1517	R1 = 0.0462, wR2 = 0.0982
R indices (all data)	R1 = 0.1255, wR2 = 0.1766	R1 = 0.0793, wR2 = 0.1067
Largest diff. peak and hole([e Å^{-3}])	0.680 and -0.530	0.270 and -0.270
Refinement method	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2

XRD References

1. Bruker, SMART, SAINT-Plus, SADABS; Bruker Axs inc.: Madison, Wisconcin, USA, **1998**.
2. Sheldrick, G. M. *Acta Cryst. A*. **2008,** *64*, 112-122.doi:10.1107/S0108767307043930
3. Nardelli, M. *Acta Cryst*. *C*. **1983,** *39,* 1141-1142.doi.org/10.1107/S0108270183007696
4. Farrugia, L. J. *J. Appl. Cryst*. **2012,** *45*, 849-854.   doi:10.1107/S0021889812029111
5. Spek, A. L. *Acta Cryst. D*. **2009,** *65*, 148-155.*Acta Cryst.* (2009).doi:10.1107/S090744490804362X
6. Watkin, D. J.; Prout, C. J.; Pearce, L. J. **1996,** CAMERON. Chemical Crystallography Laboratory, University Of Oxford, England.
7. Brandenburg, K.; Putz, H. *Crystal Impact*. **2005,** GbR, Bonn, Germany.

IR of 1-Trimethylsilyl-2-(2′-methoxyphenyl)cyclopenetene 4a:
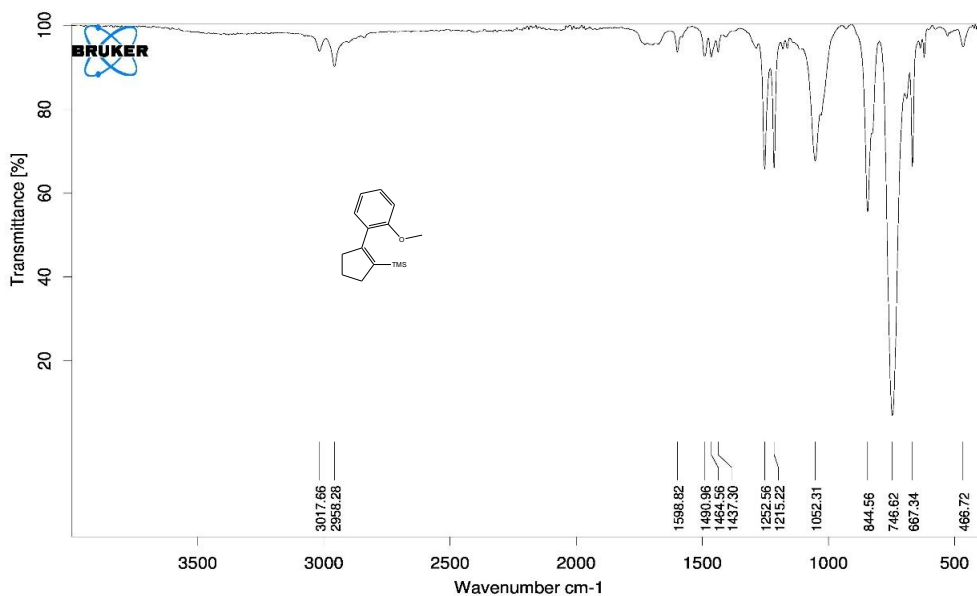
MS of 1-Trimethylsilyl-2-(2′-methoxyphenyl)cyclopentene 4a:
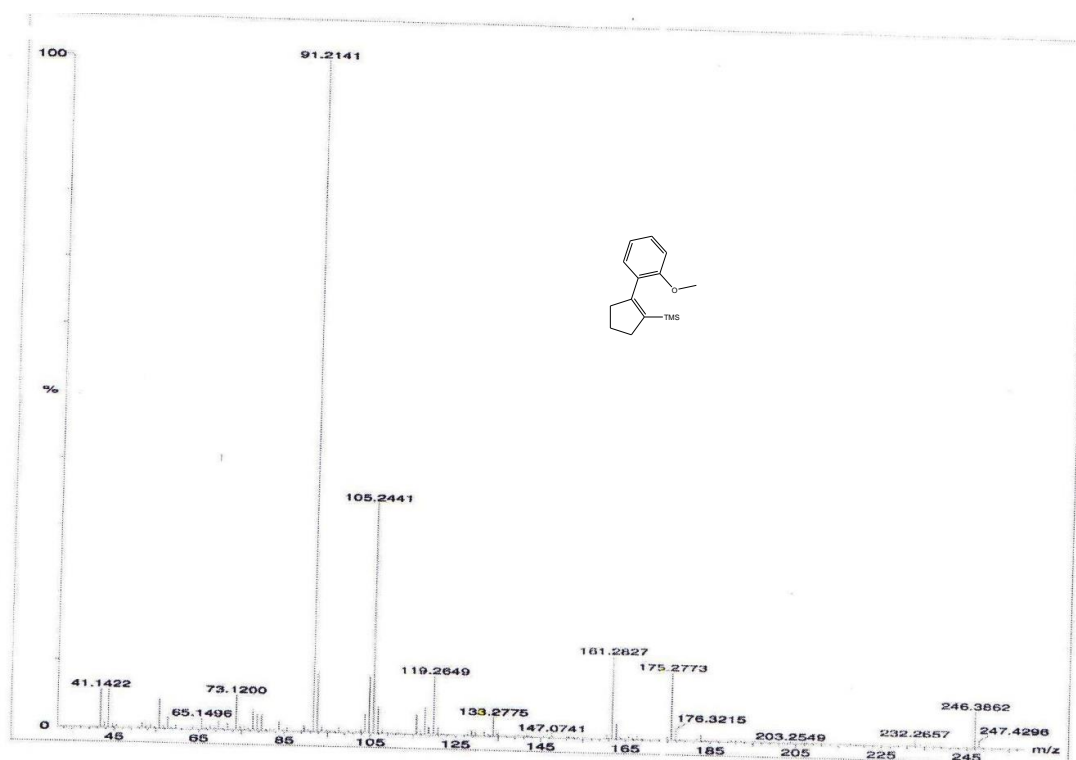
¹H NMR of 1-Trimethylsilyl-2-(2′-methoxyphenyl)cyclopentene 4a:
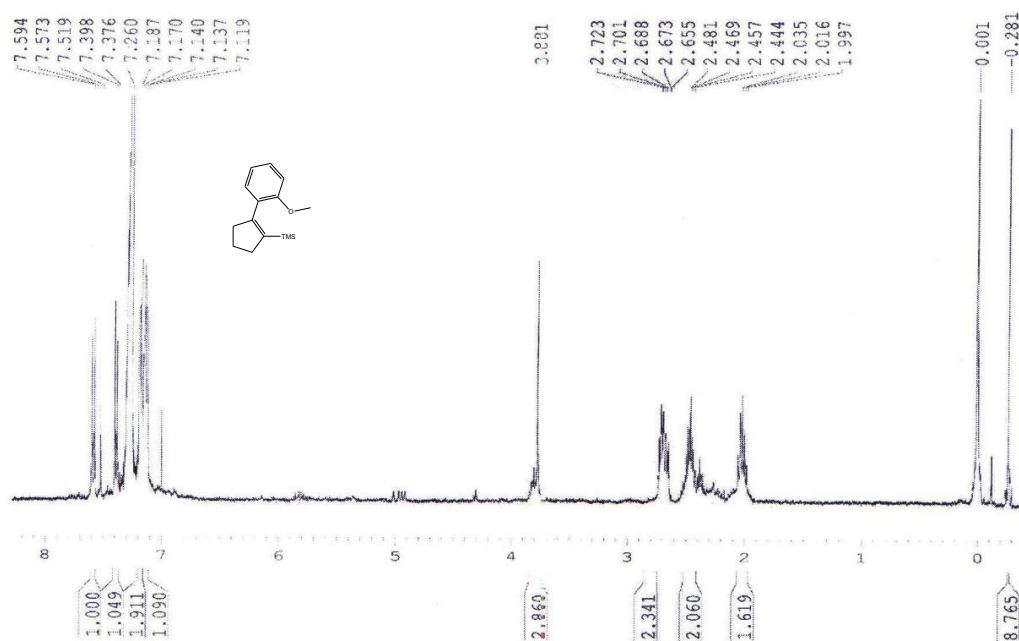
¹³C NMR of 1-Trimethylsilyl-2-(2′-methoxyphenyl)cyclopentene 4a:
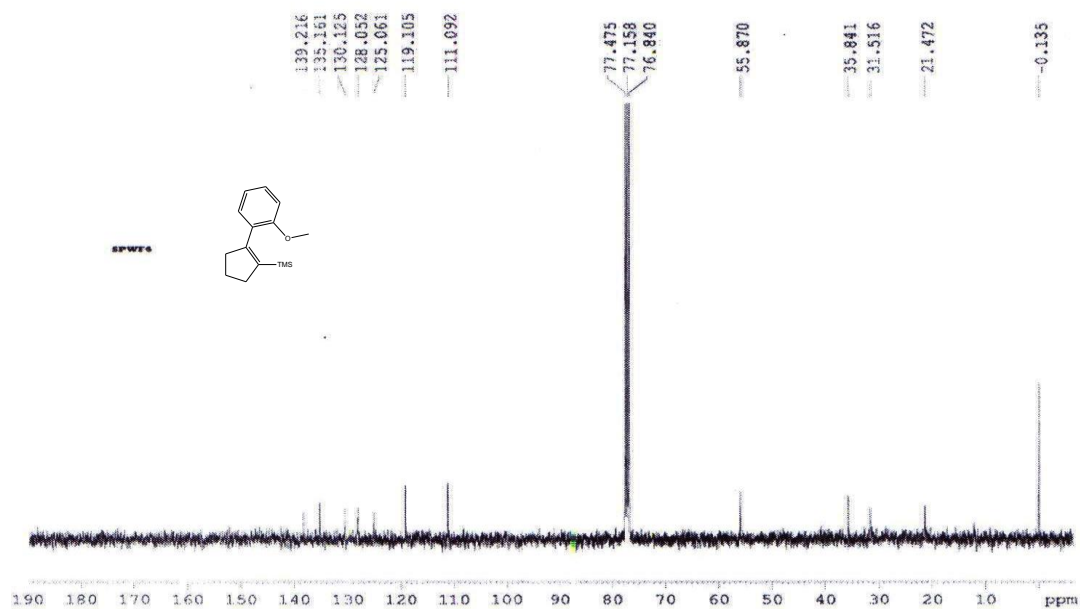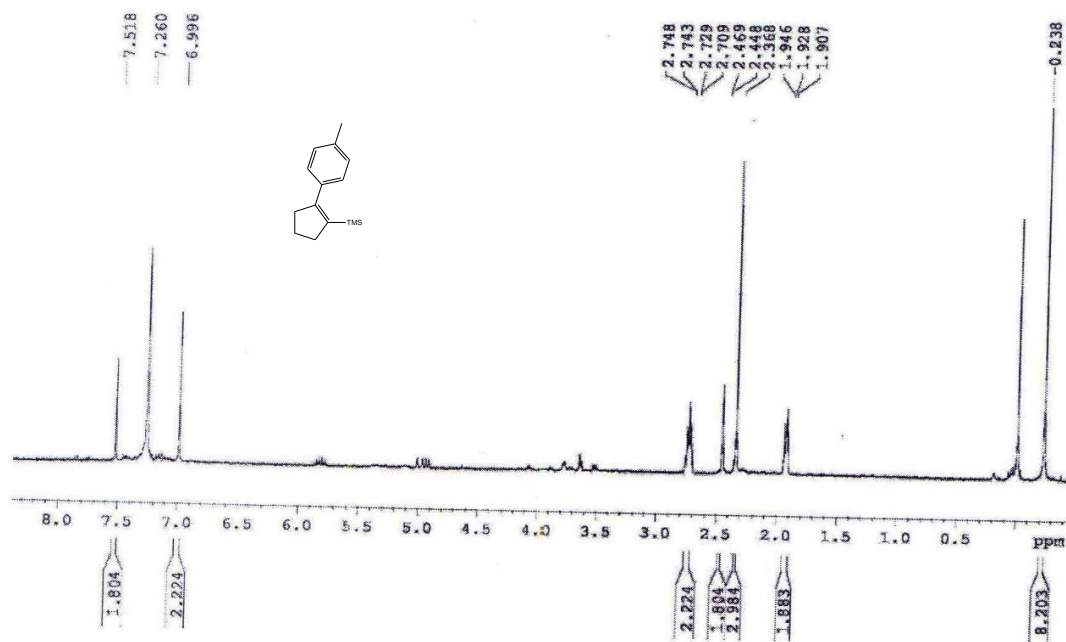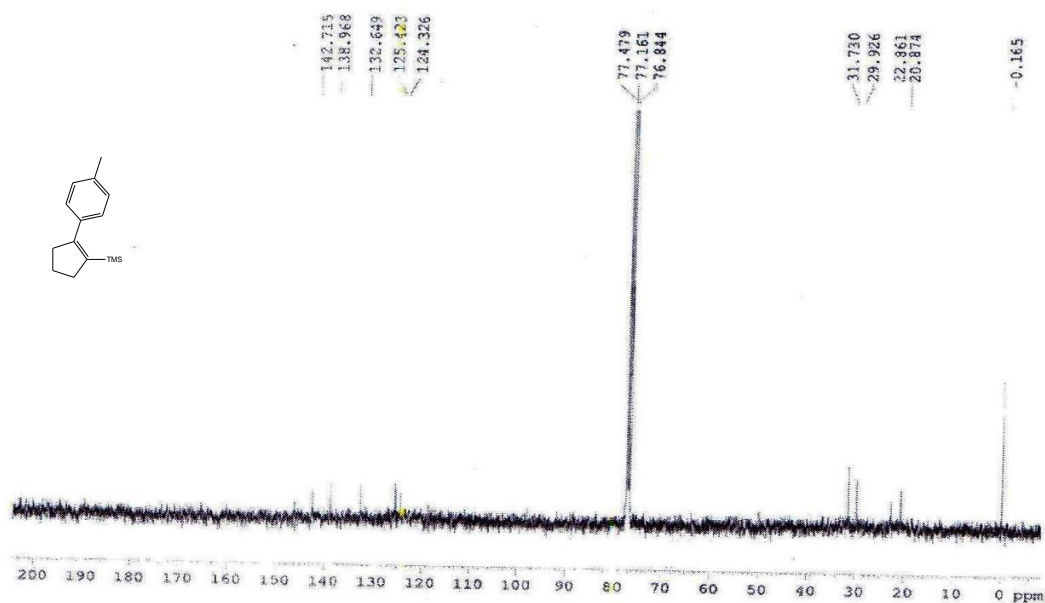
IR of 1-Trimethylsilyl-2-(2′-ethoxyphenyl)cyclopentene 4b:
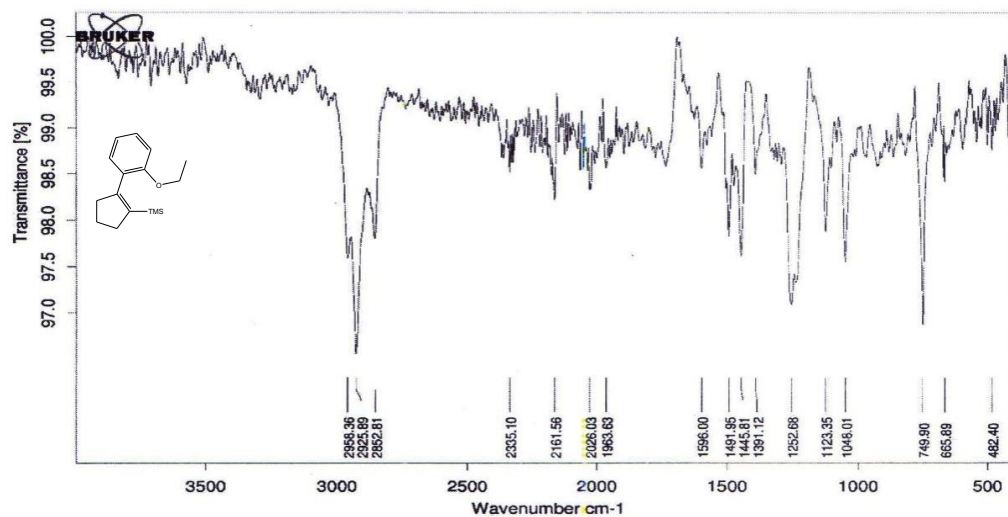
MS of 1-Trimethylsilyl-2-(2′-ethoxyphenyl)cyclopentene 4b:
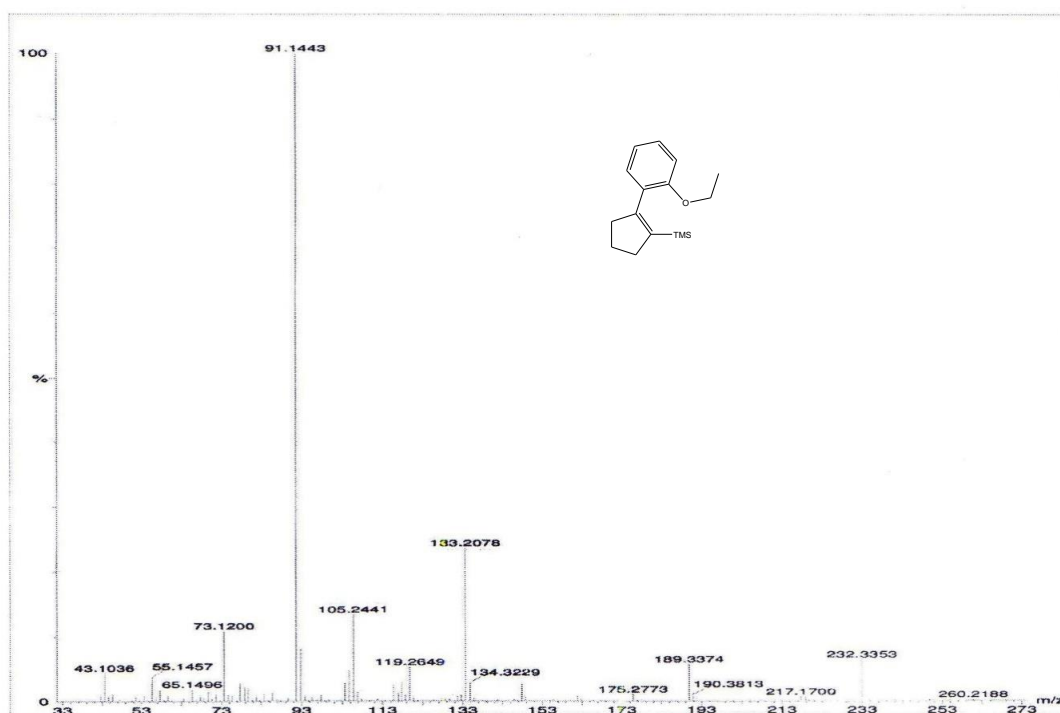
¹H NMR of 1-Trimethylsilyl-2-(2′-ethoxyphenyl)cyclopentene 4b:
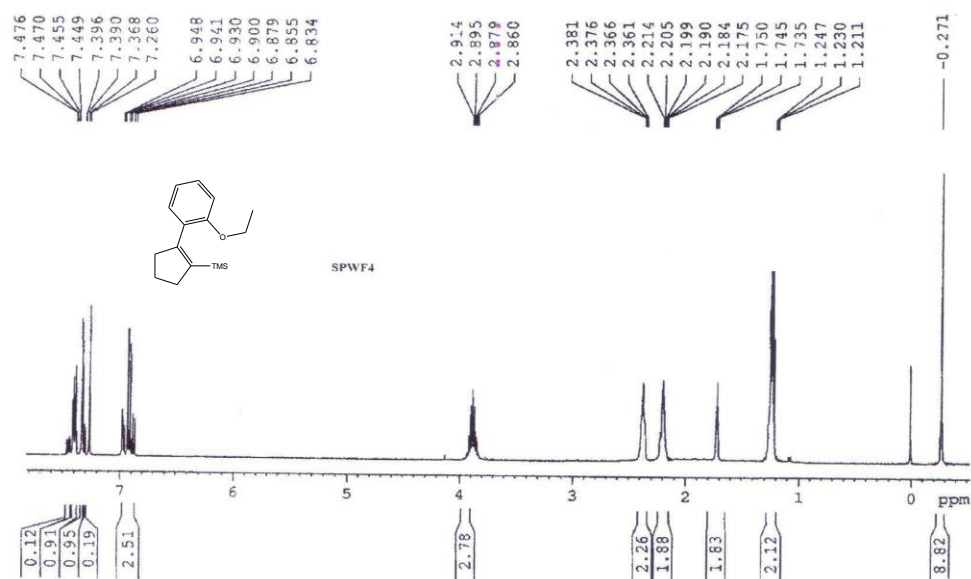
¹³C NMR of 1-Trimethylsilyl-2-(2′-ethoxyphenyl)cyclopentene 4b:
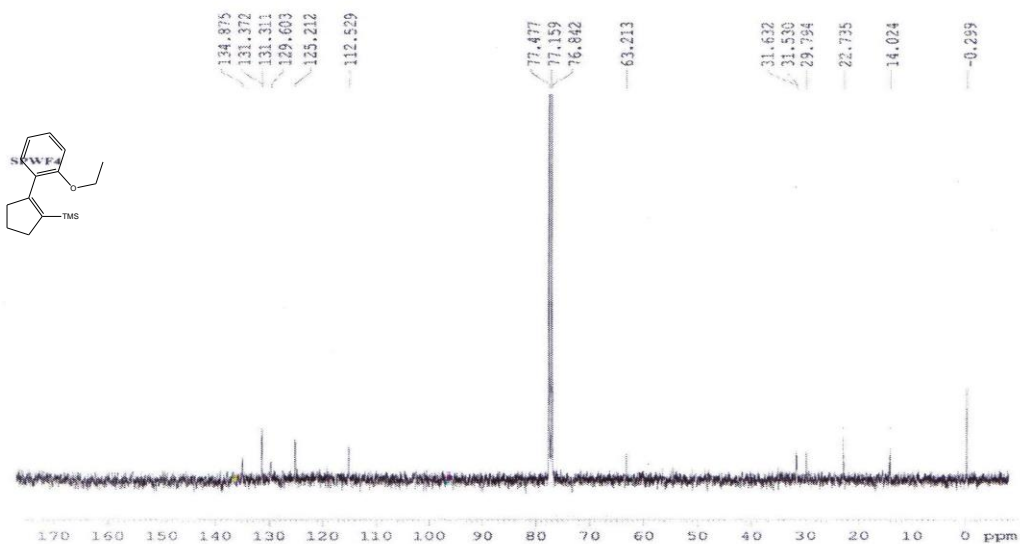
IR of 1-Trimethylsilyl-2-(4′-methylphenyl)cyclopentene 4c:
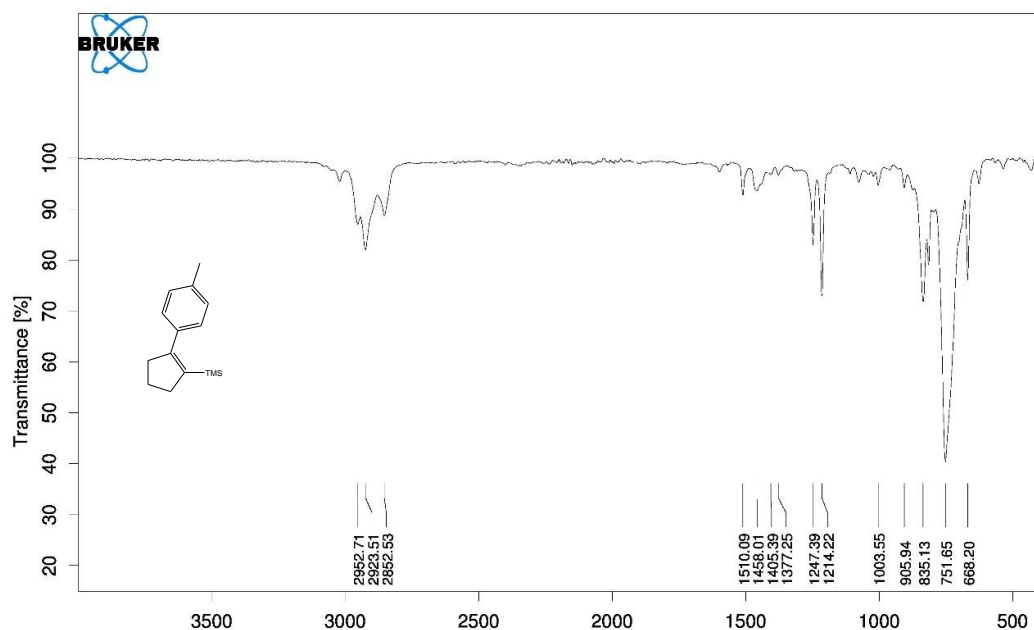
MS of 1-Trimethylsilyl-2-(4′-methylphenyl)cyclopentene 4c:
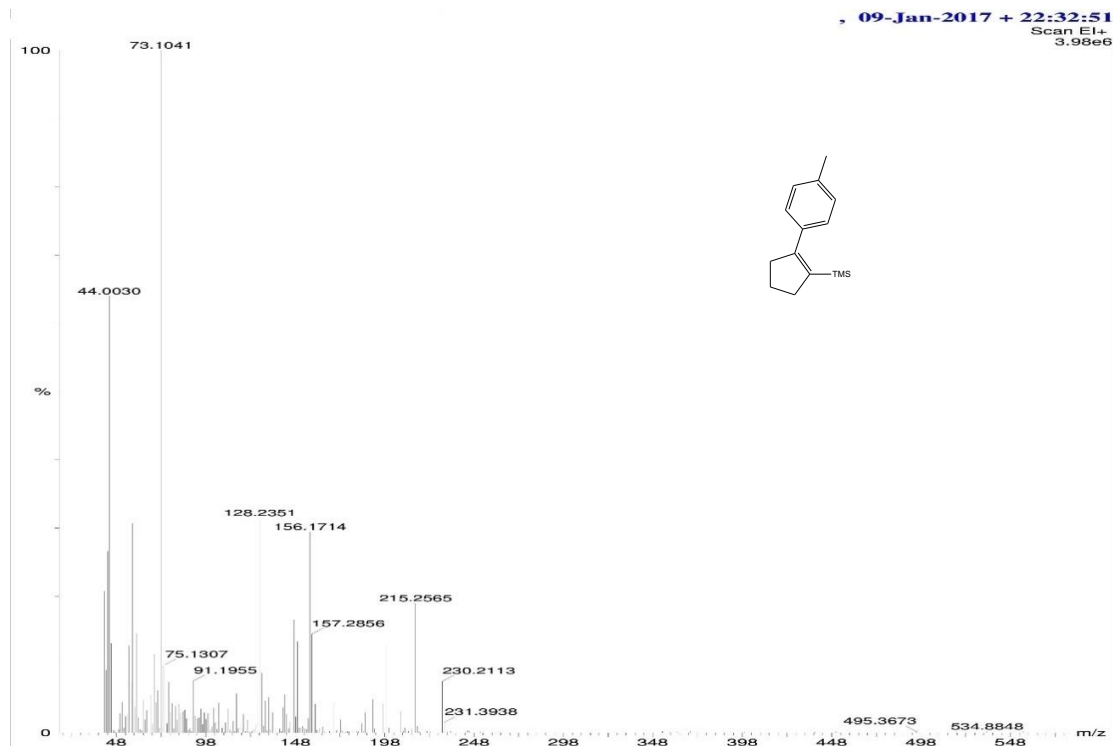
^{1}H NMR of 1-Trimethylsilyl-2-(4′-methylphenyl)cyclopentene 4c:

^{13}C NMR of 1-Trimethylsilyl-2-(4′-methylphenyl)cyclopentene 4c:

                                    ©ARKAT USA, Inc

IR of 1-Trimethylsilyl-2-(4′-ethylphenyl)cyclopentene 4d:

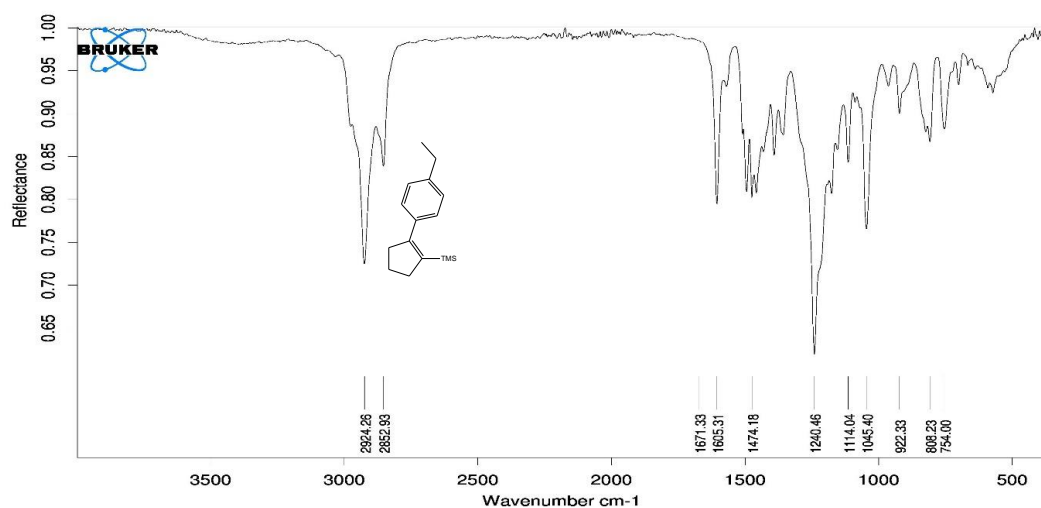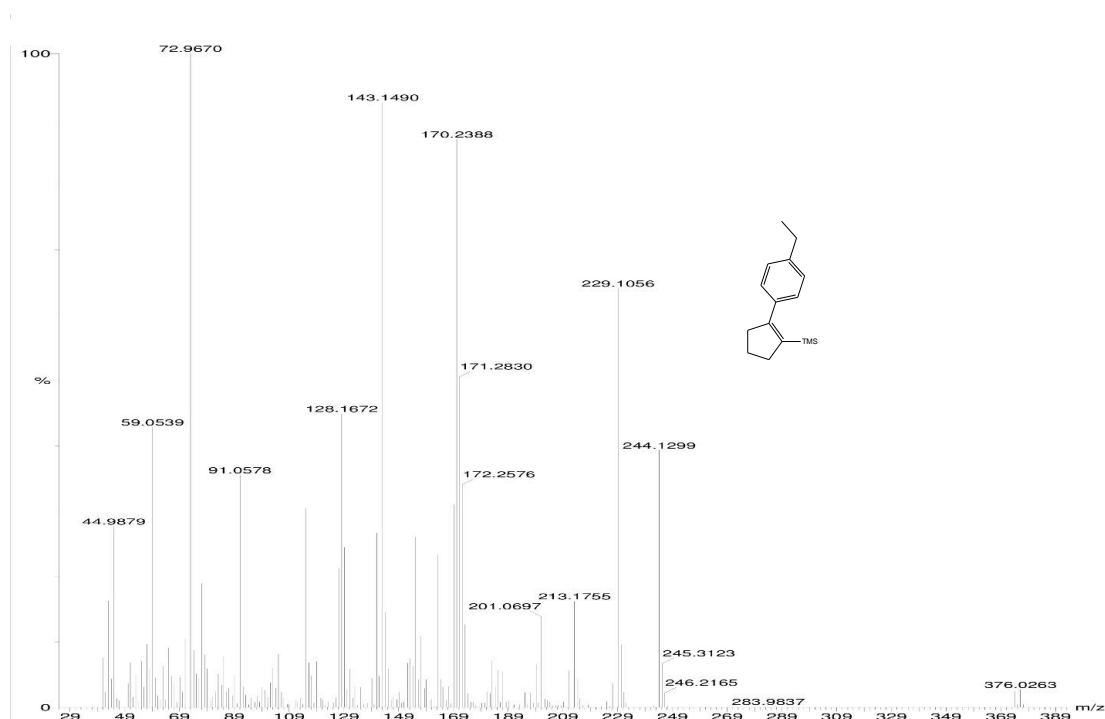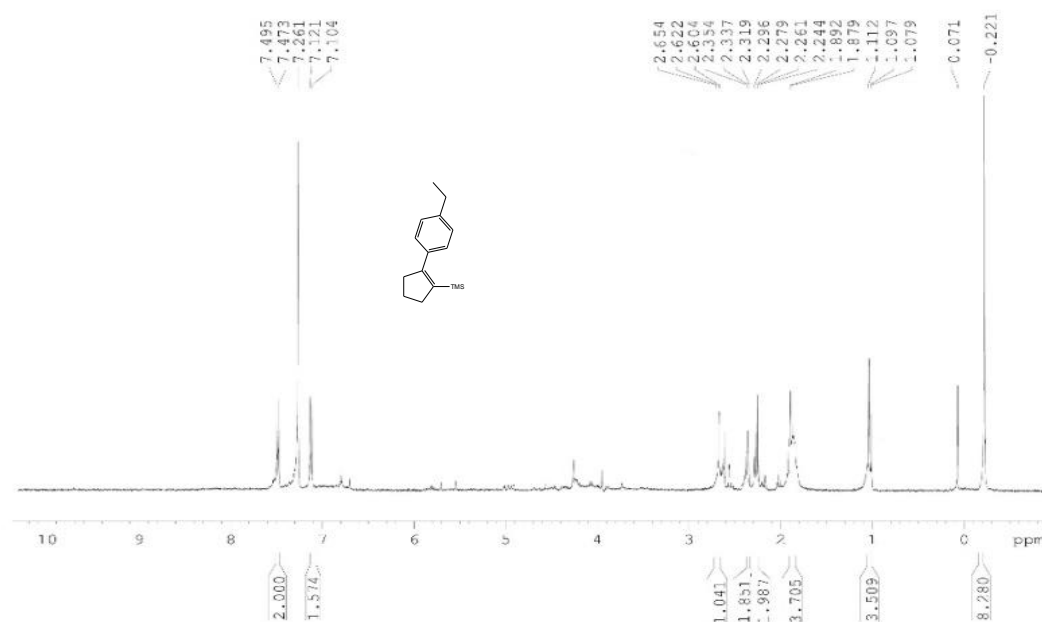**MS of 1-Trimethylsilyl-2-(4′-ethylphenyl)cyclopentene 4d:**

^{1}H NMR of 1-Trimethylsilyl-2-(4′-ethylphenyl)cyclopentene 4d:

^{13}C NMR of 1-Trimethylsilyl-2-(4′-ethylphenyl)cyclopentene 4d:

IR of 1-Trimethylsilyl-2-(4′-tertiatybutylphenyl)cyclopentene 4e:

MS of 1-Trimethylsilyl-2-(4′-tertiatybutylphenyl)cyclopentene 4e:

^{1}H NMR of 1-Trimethylsilyl-2-(4′-tertiatybutylphenyl)cyclopentene 4e:

SPWF9

^{13}C NMR of 1-Trimethylsilyl-2-(4′-tertiatybutylphenyl)cyclopentene 4e:

IR of 1-Trimethylsilyl-2-(4′-ethylsulfanephenyl)cyclopentene 4f:

MS of 1-Trimethylsilyl-2-(4′-ethylsulfanephenyl)cyclopentene 4f:

¹H NMR of 1-Trimethylsilyl-2-(4′-ethylsulfanephenyl)cyclopentene 4f:

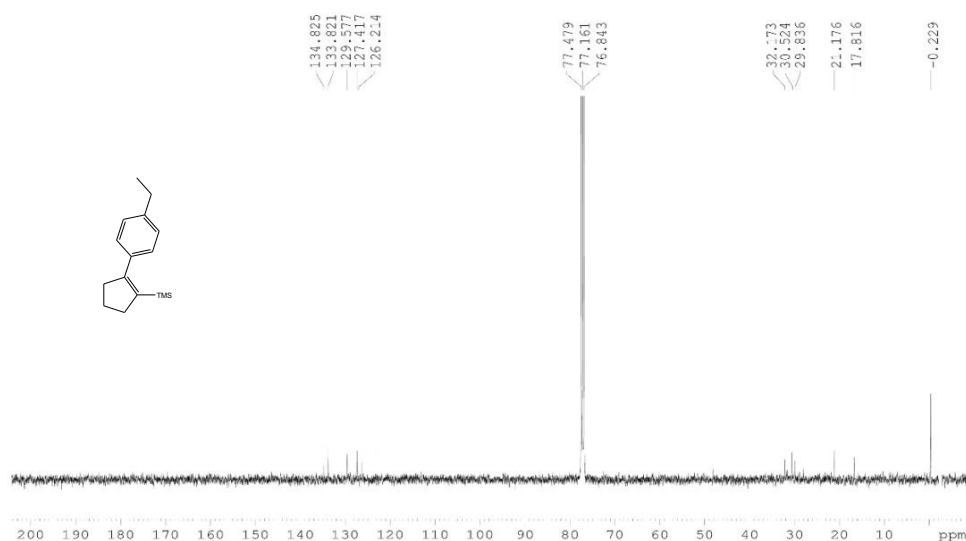# ¹³C of 1-Trimethylsilyl-2-(4′-ethylsulfanephenyl)cyclopentene 4f:
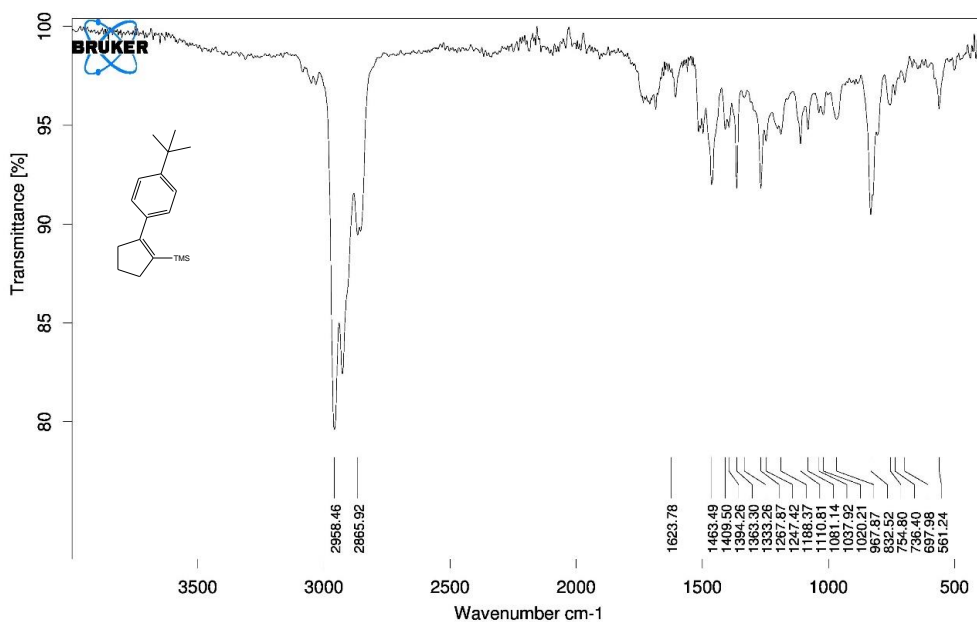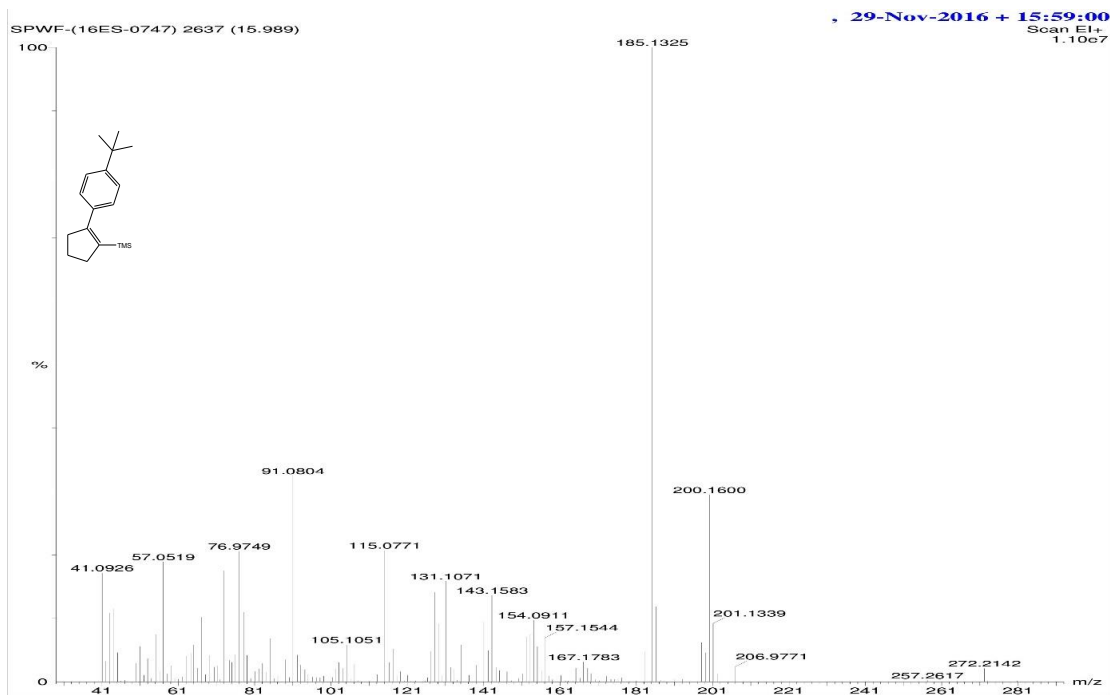
IR of 1-Trimethylsilyl-2-(4′-ethoxyphenyl)cyclopentene 4g:

MS of 1-Trimethylsilyl-2-(4′-ethoxyphenyl)cyclopentene 4g:
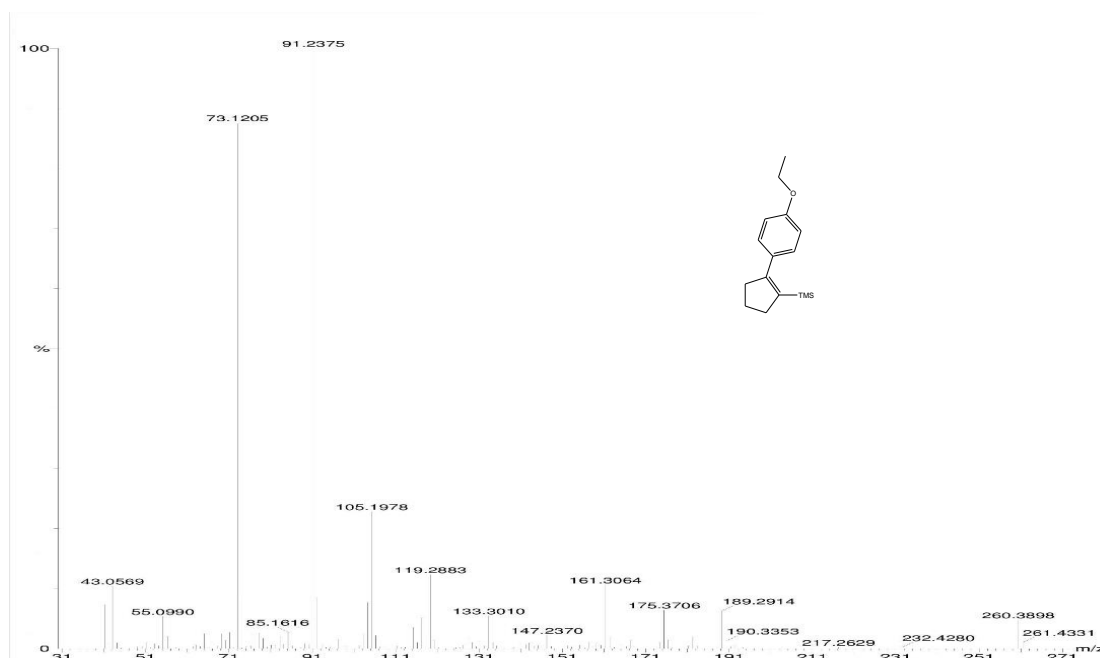
¹H NMR of 1-Trimethylsilyl-2-(4′-ethoxyphenyl)cyclopentene 4g:
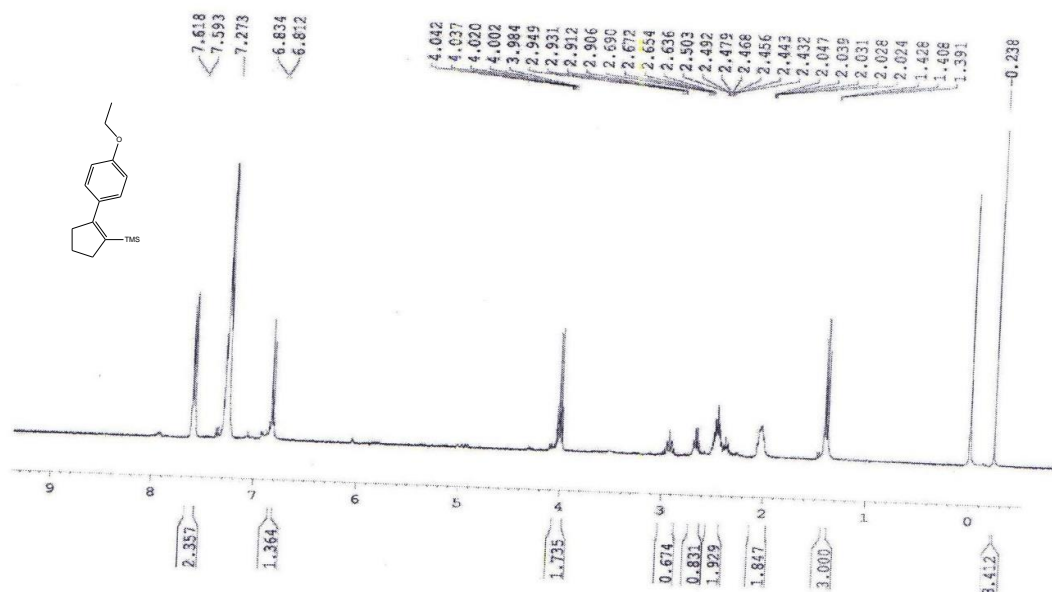
¹³C NMR of 1-Trimethylsilyl-2-(4′-ethoxyphenyl)cyclopentene 4g:

IR of 1-Trimethylsilyl-2-(3′-methylphenyl)cyclopentene 4h:

E:\JEEVAN\SPWF2-3.3   SPWF2-3   Instrument type and / or accessory   11/02/2017
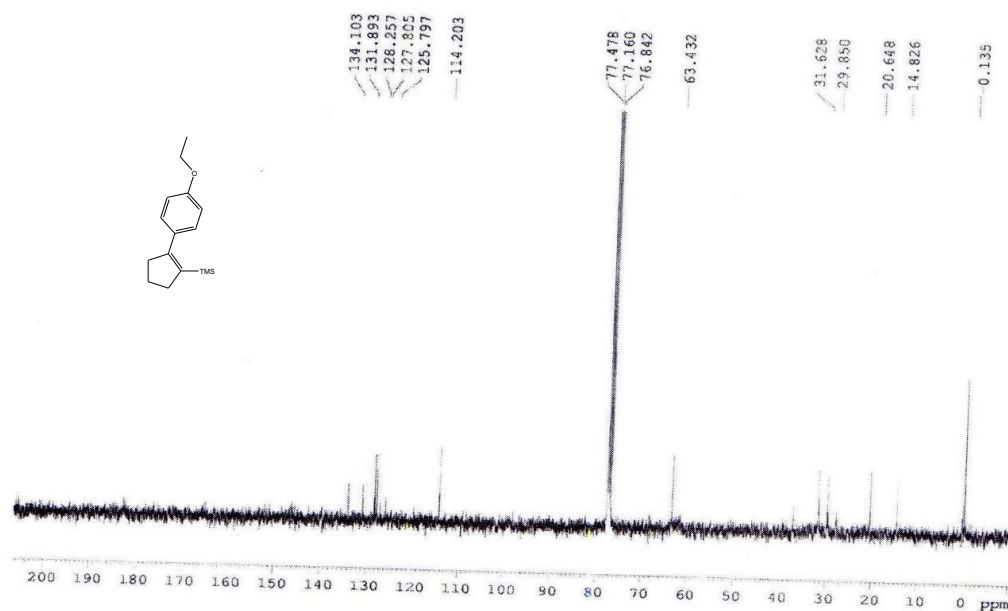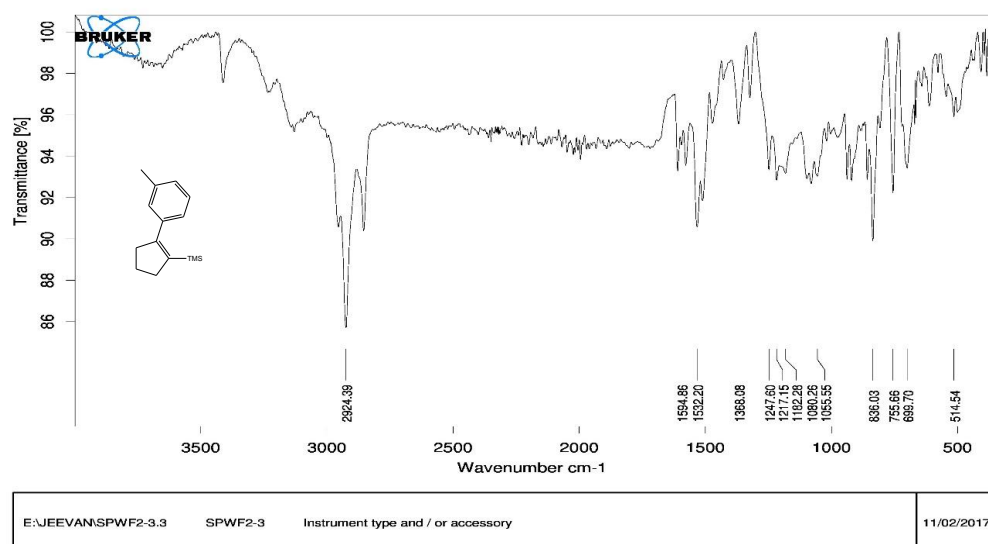
Page 1/1

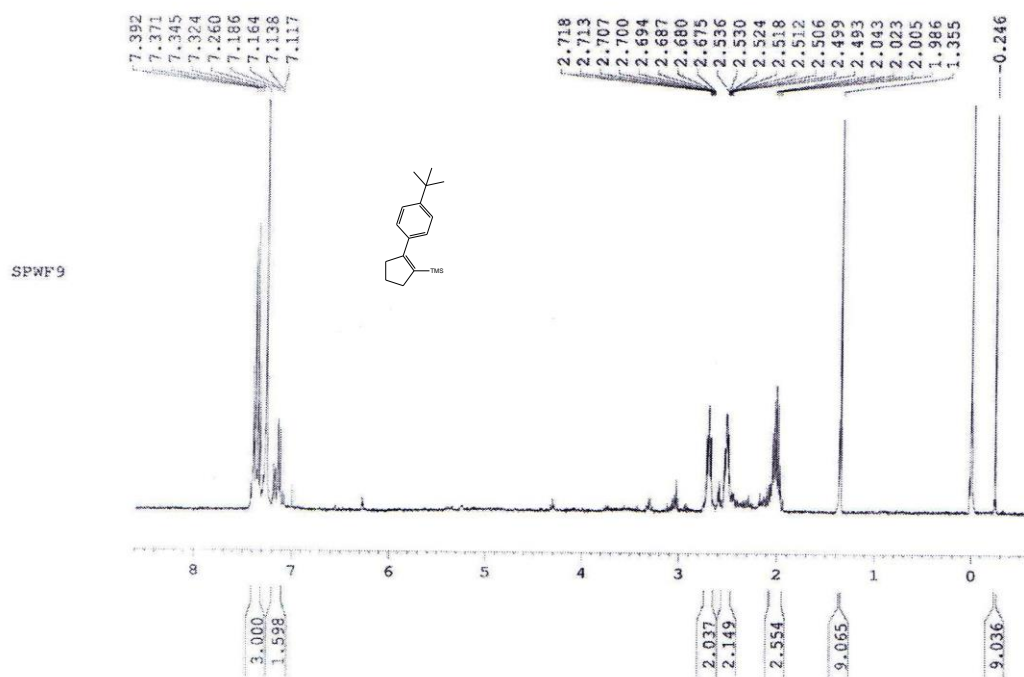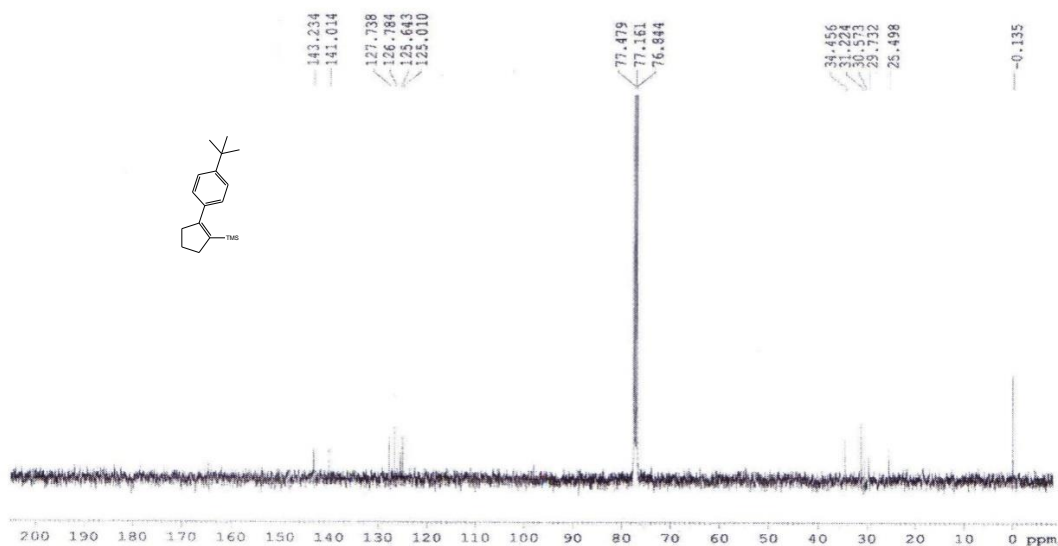**MS of 1-Trimethylsilyl-2-(3′-methylphenyl)cyclopentene 4h:**
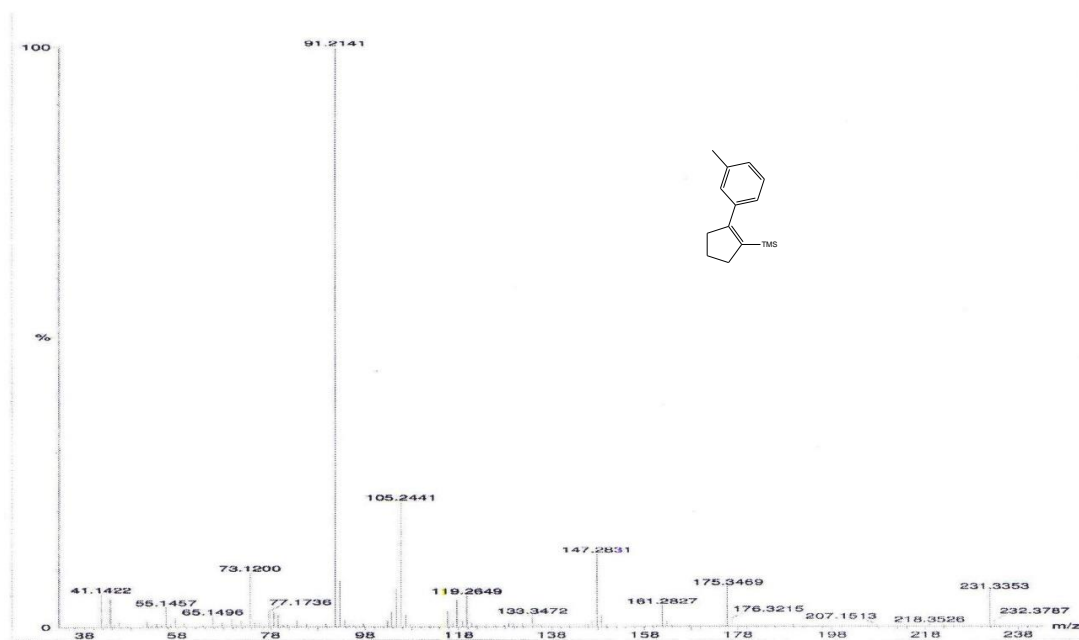
¹H NMR of 1-Trimethylsilyl-2-(3′-methylphenyl)cyclopentene 4h:

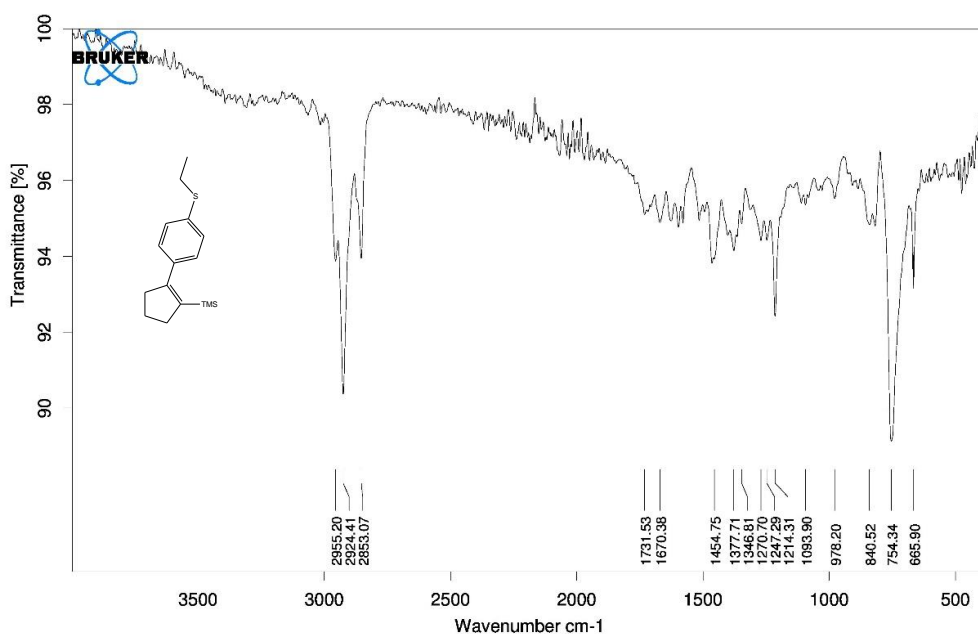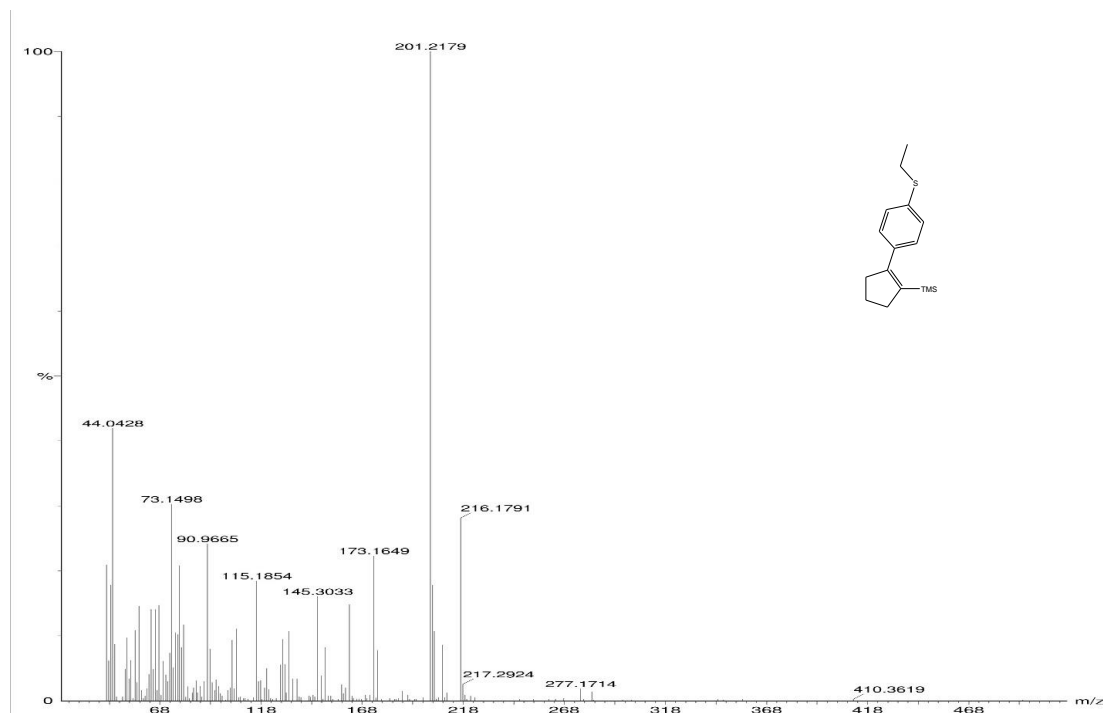**¹³C NMR of 1-Trimethylsilyl-2-(3′-methylphenyl)cyclopentene 4h:**

IR of 1-Trimethylsilyl-2-(3′,4′-dimethoxyphenyl)cyclopentene 4i:
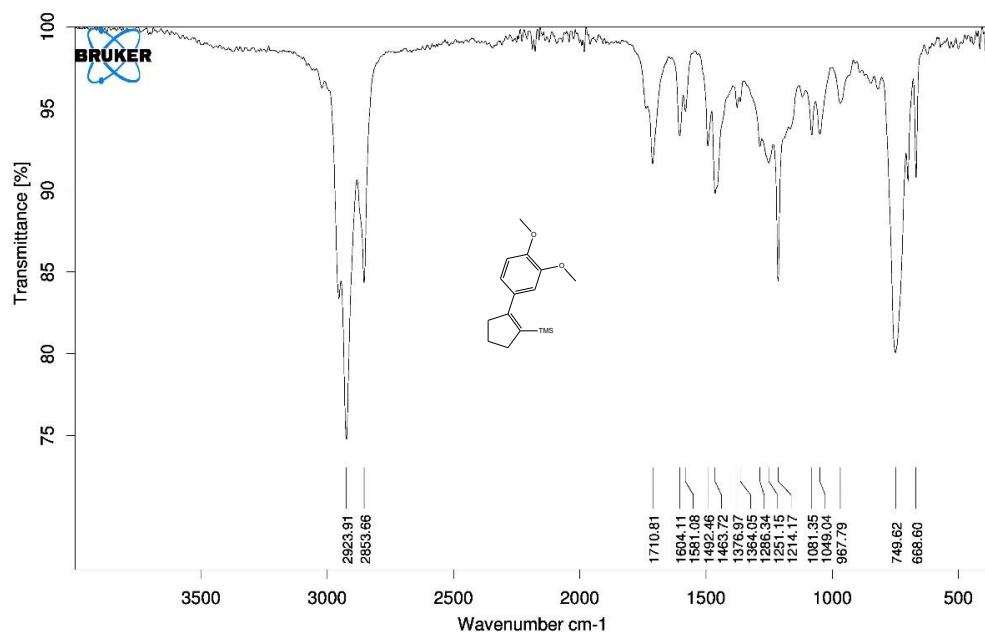
MS of 1-Trimethylsilyl-2-(3′,4′-dimethoxyphenyl)cyclopentene 4i:
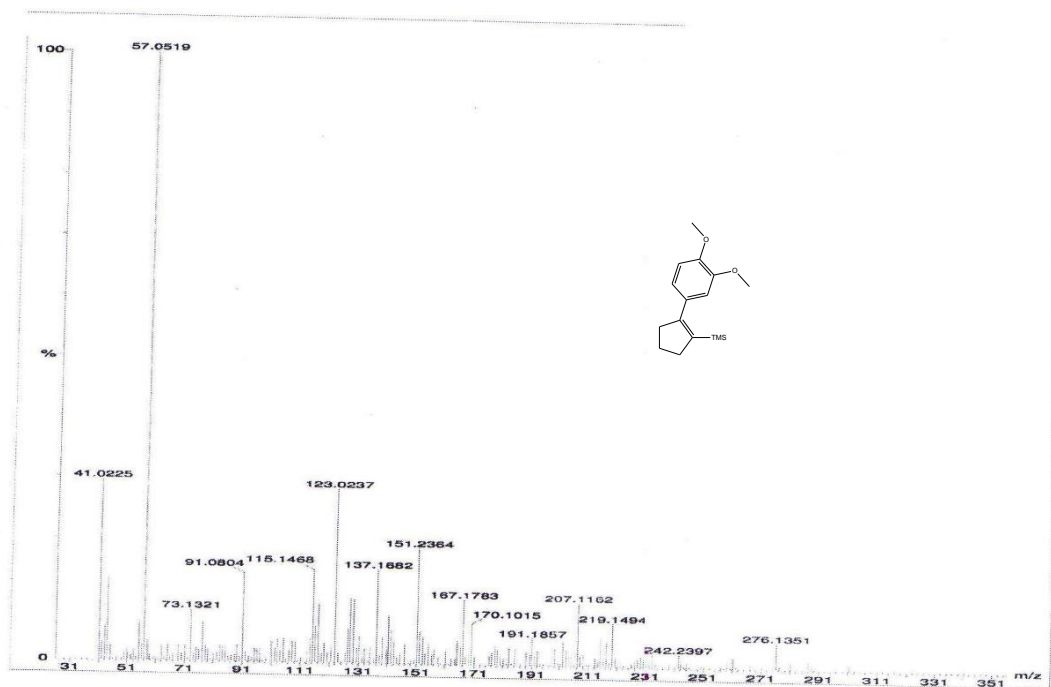
1H NMR of 1-Trimethylsilyl-2-(3′,4′-dimethoxyphenyl)cyclopentene 4i:

13C NMR of 1-Trimethylsilyl-2-(3′,4′-dimethoxyphenyl)cyclopentene 4i:
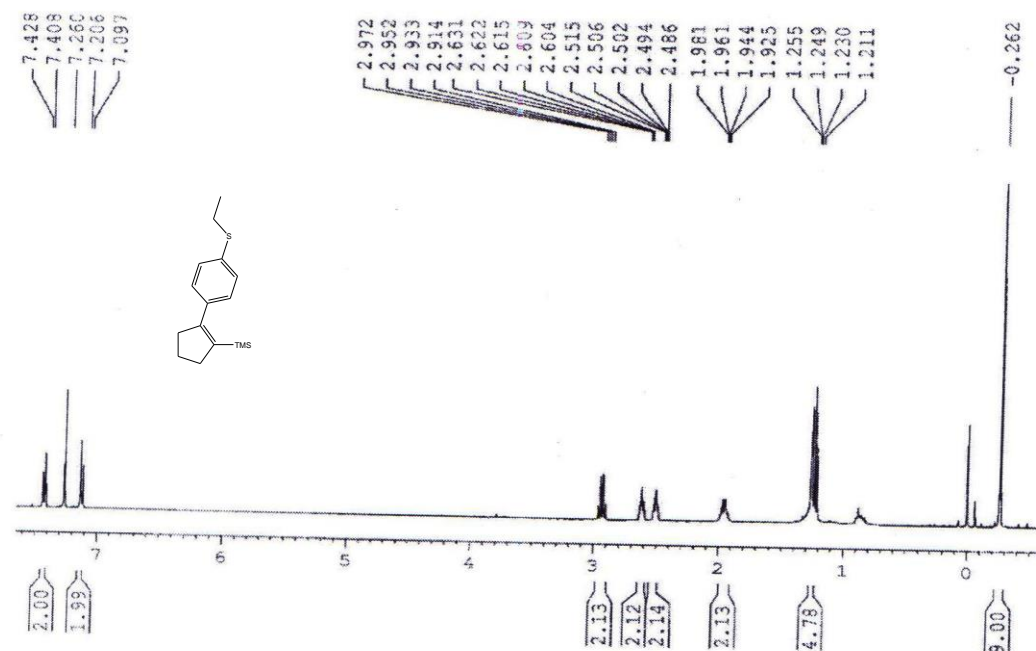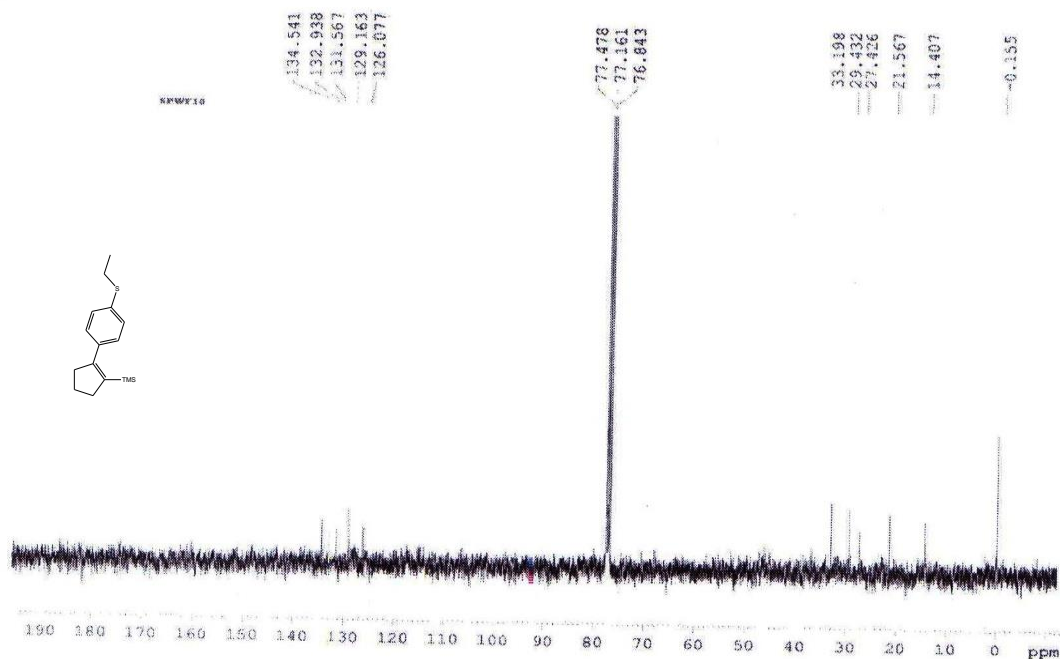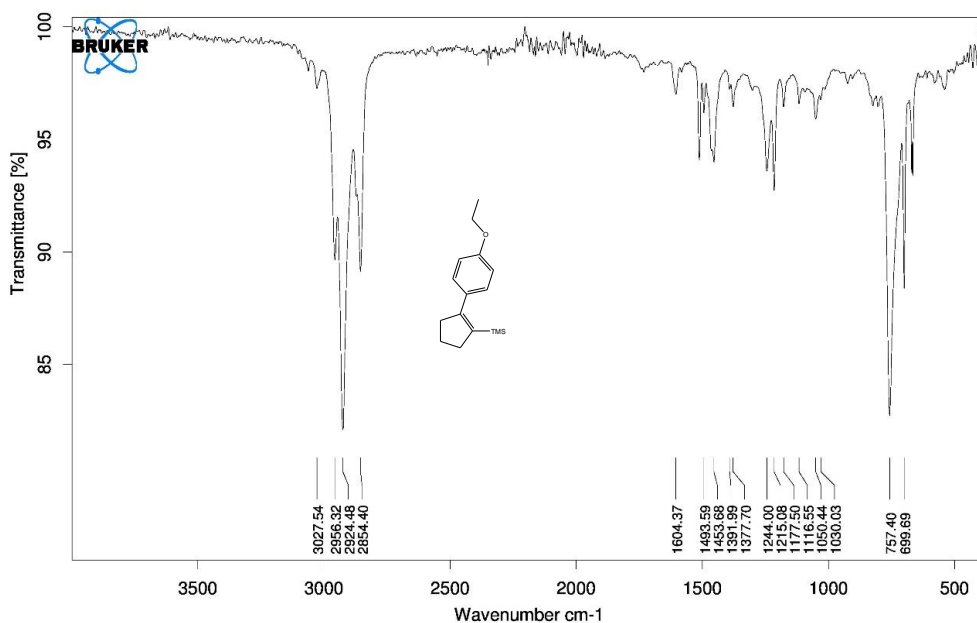
IR of 1-Trimethylsilyl-2-(4′-biphenyl)cyclopentene 4j:
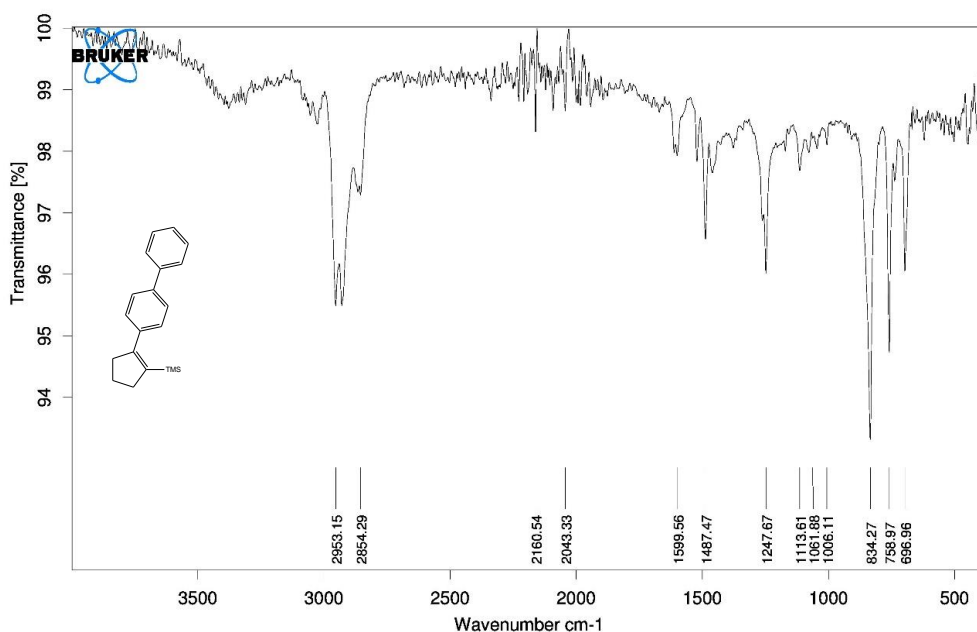
MS of 1-Trimethylsilyl-2-(4′-biphenyl)cyclopentene 4j:
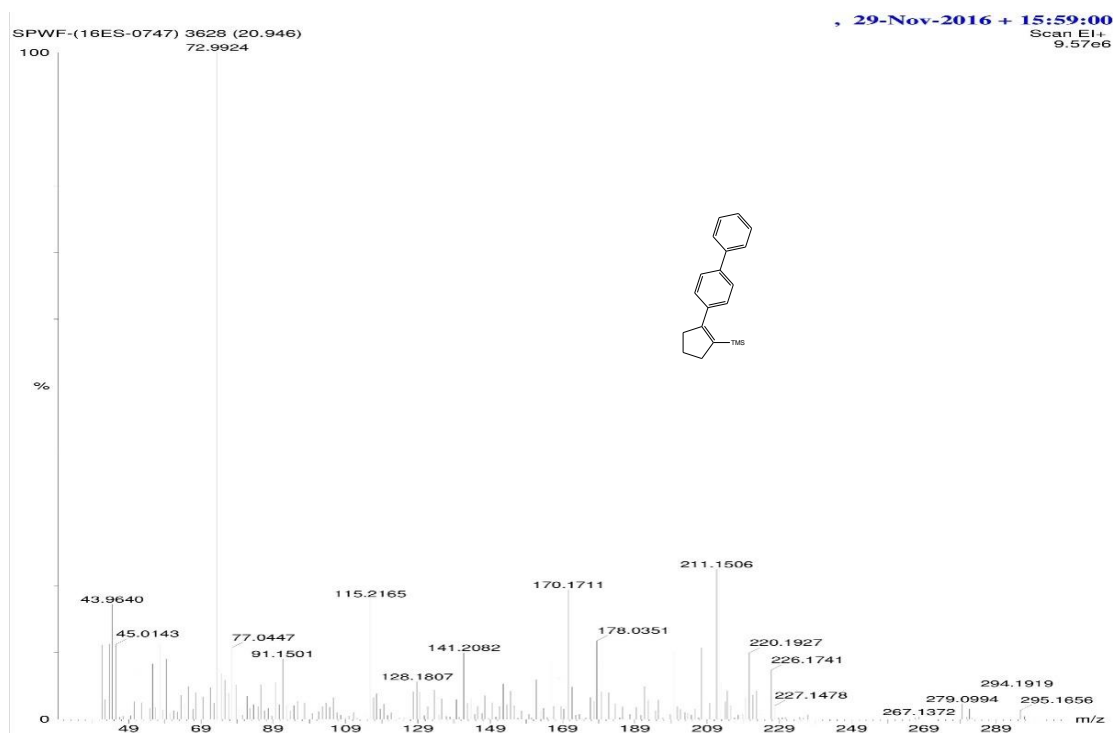
^{1}H NMR of 1-Trimethylsilyl-2-(4′-biphenyl)cyclopentene 4j:
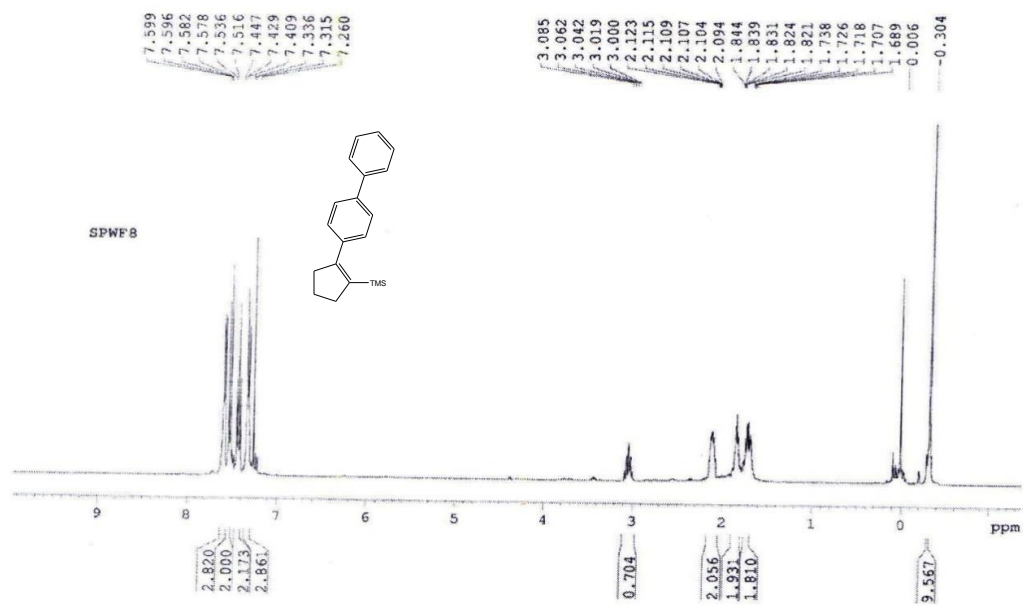
^{13}C NMR of 1-Trimethylsilyl-2-(4′-biphenyl)cyclopentene 4j:
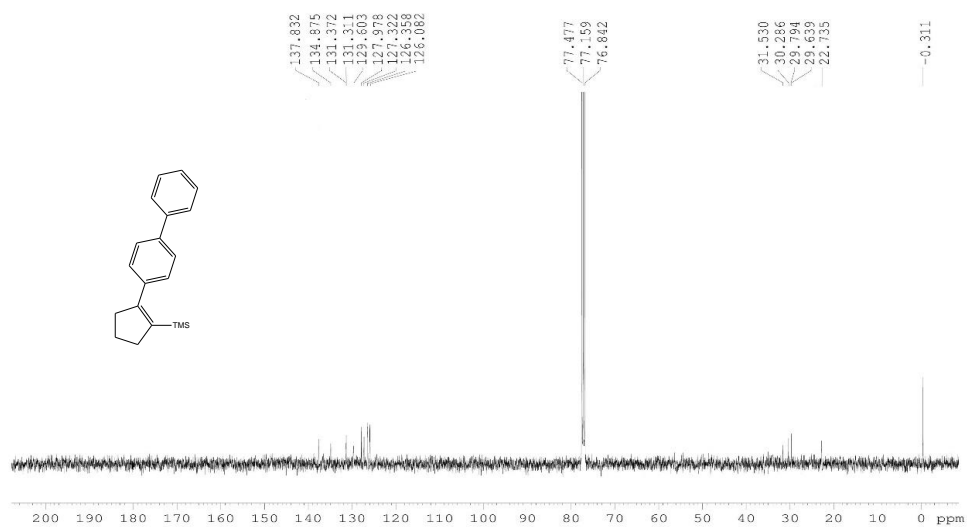
IR of 1-Trimethylsilyl-2-(1-napthyl)cyclopentene 4k:
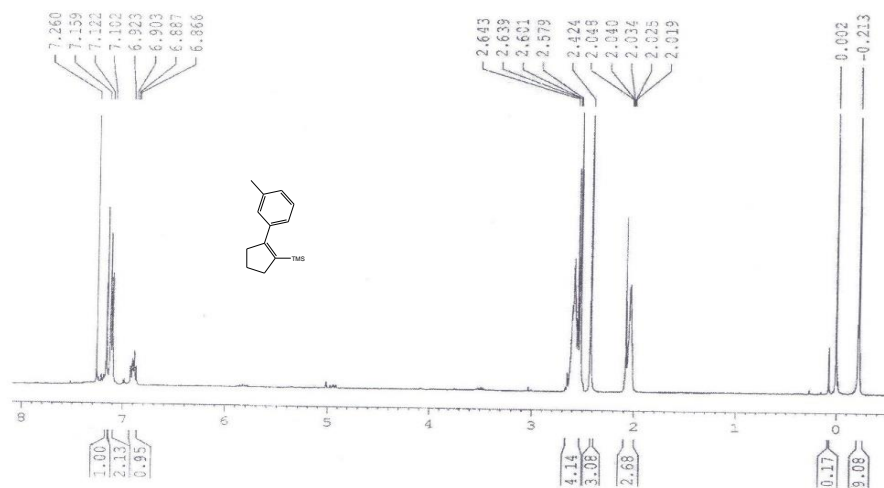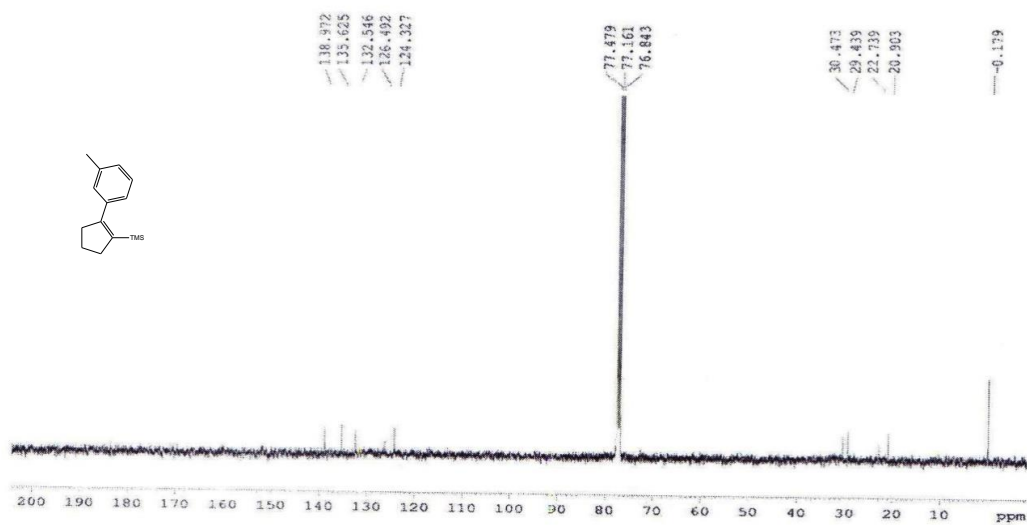

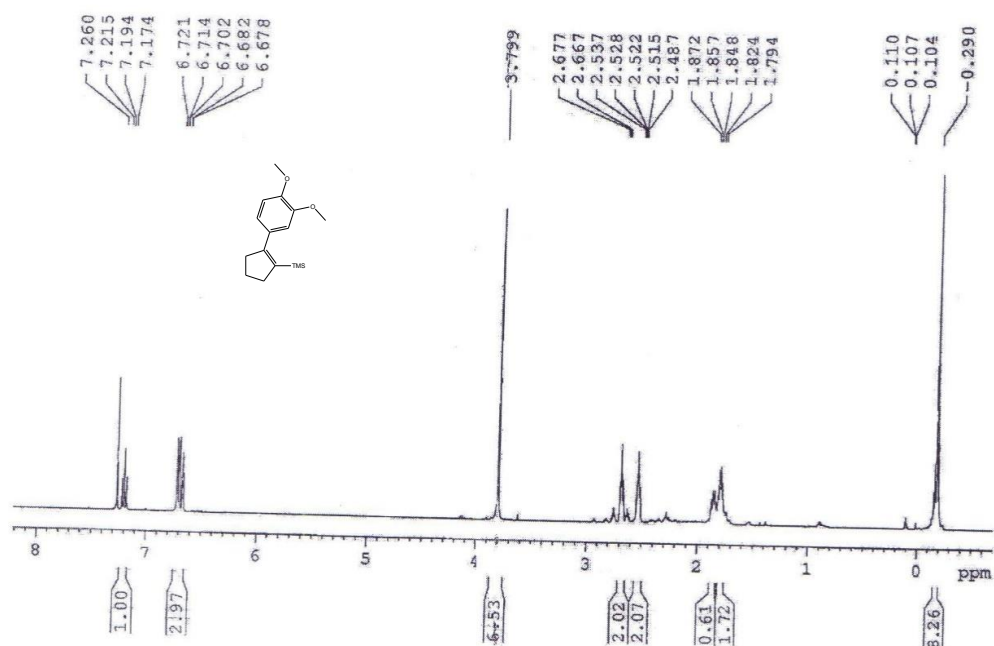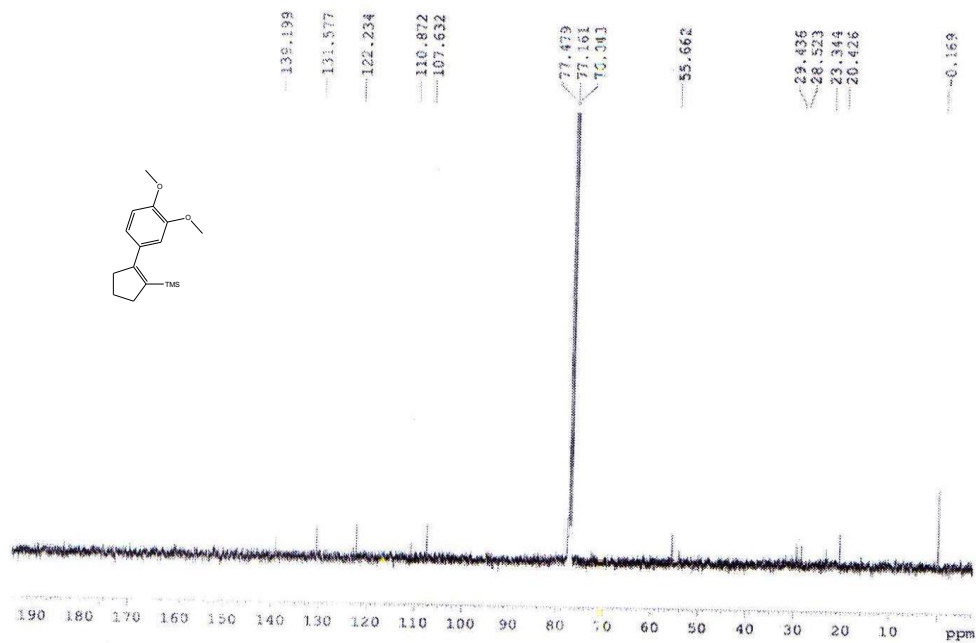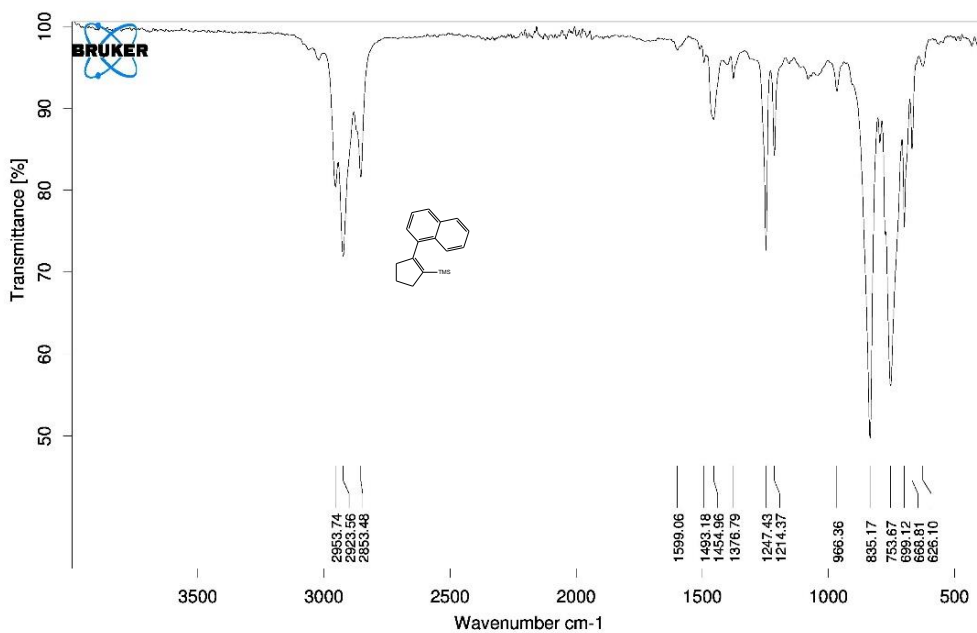**MS of 1-Trimethylsilyl-2-(1-napthyl)cyclopentene 4k:**
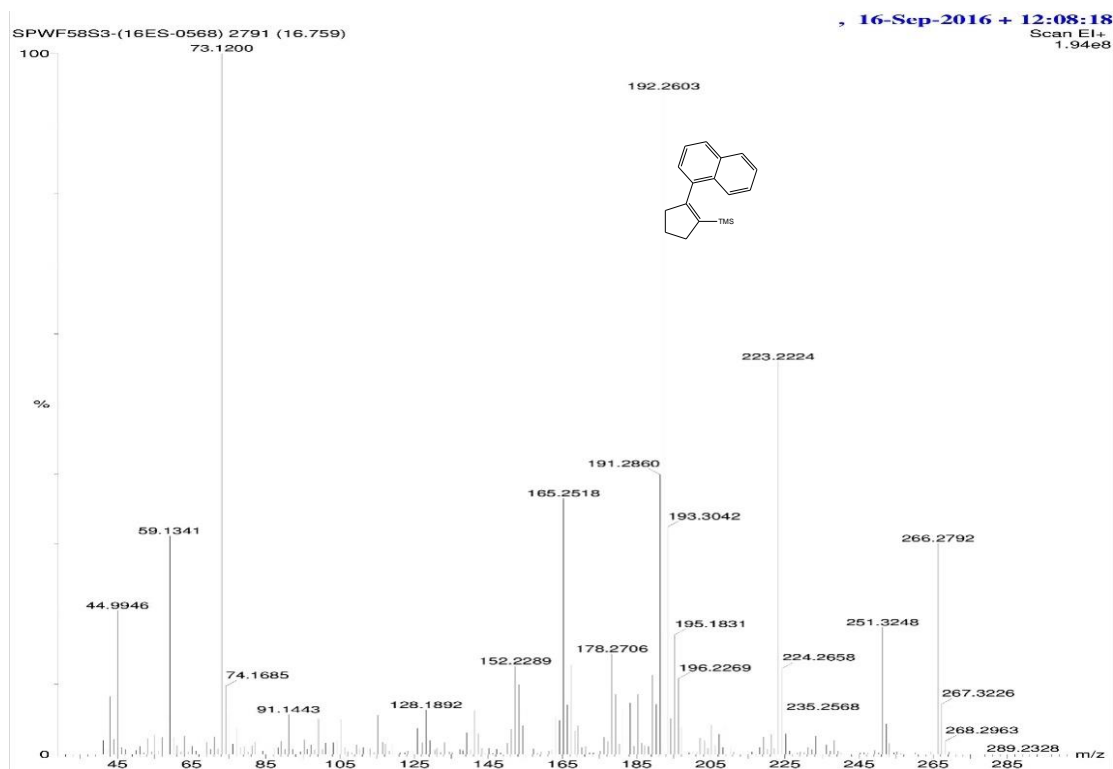
^{1}H NMR of 1-Trimethylsilyl-2-(1-napthyl)cyclopentene 4k:
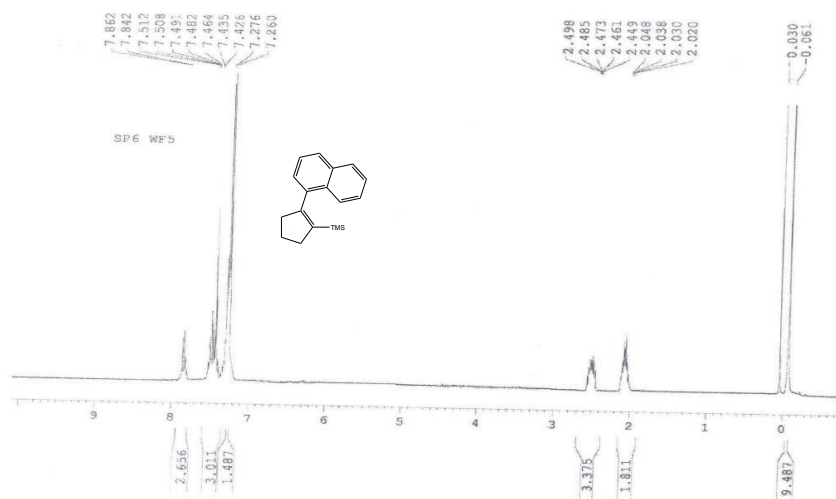
^{13}C NMR of 1-Trimethylsilyl-2-(1-napthyl)cyclopentene 4k:
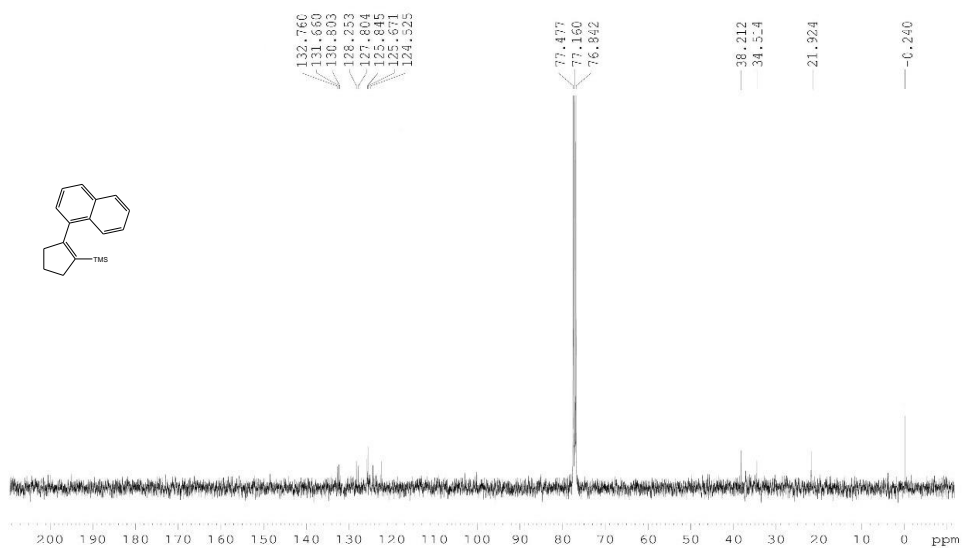
X-ray structure analysis

To our knowledge there exist no literature reports for the single crystal XRD analysis of 1-chloro-2-aryl-substituted cyclopentenes (**3**).

We now report the XRD crystal structures of some representative 1-chloro-2-arylcyclopentens. Very good quality crystals of the 1-chloro-2-aryl compounds **3j**, and **3k** were obtained by slow evaporation technique.

Attempts were made to isolate single crystals of the silyl compounds **4j** and **4k** by recrystallization techniques using hexane (60-80 ºC fraction). However, due to the improper crystal growth, the single crystal XRD details of **4j** and **4k** could not be obtained, probably due to the presence of the trimethylsilyl- group.

Suitable crystals of the chlorine bearing compounds **3j** and 3**k** of appropriate quality and size for single crystal X-ray diffraction were obtained from slow evaporation method from hexane at room temperature. A good quality single crystal in each case was mounted along its largest dimension and used for data collection. The intensity data were collected on a Bruker Smart CCD Area Detector System using MoKα (0.7103Å) radiation in $\omega - \phi$ scan mode. The data were reduced using SAINT-Plus.[38] The structures in each case was solved by Direct Methods and refined on F^2 using SHELX-97[39] software package. All the non-hydrogen atoms were refined anisotropically. As the hydrogen atoms were not readily revealed from difference Fourier maps, they were included in the ideal positions with fixed isotropic U values, and they were riding with their respective non-hydrogen atoms. The difference Fourier map, after the refinement, was essentially featureless in all the cases. The mean plane calculations were done using the program PARST.[40] Diagrams were generated using ORTEP-3,[41] PLATON,[42] CAMERON[43] and DIAMOND.[44]

The ORTEP view of the molecules **3j** and **3k** with atomic labeling (thermal ellipsoids drawn at 50% probability) is given in **Figure 2**. Packing of molecules for compounds are shown in **Figures 3** and **4** and orientation of the planes containing the ring structures are depicted in **Figure 5. Table 2** gives the interatomic interaction parameters in compounds **3j** and **3k**. Summary of crystallographic data and other structure refinement parameters of the compounds **3i** and **3j** are given in **Table 3**.

Figure 2: ORTEP view of compounds **3j** and **3k** with two and two molecule in the asymmetric unit respectively, showing 50% probability ellipsoids and the atom-numbering scheme
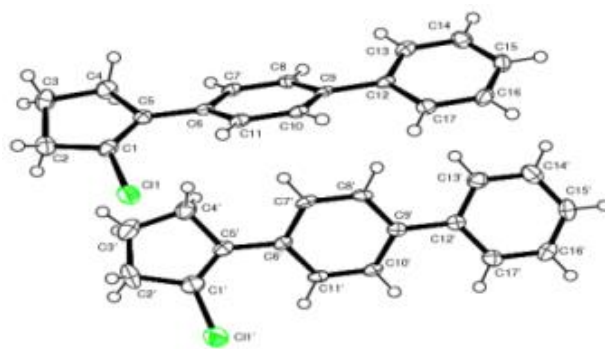
3j

3k

Intermolecular features

The compounds **3j** and **3k** are not prospective candidates for any robust weak interactions, as all the molecules are devoid of O, N, F, S atoms that would result in hydrogen bonds and other weak interactions . The only weak interactions observed are C-H···Cl and C-H···Cg (**Table 2**).

Compound **3j** is stabilized by, C10'-H10'…Cg and C7'-H7'…Cg (Cg is the centroid of aryl ring C6-C11) which forms a chain along 'a' axis with a distance of 2.754 Å and 2.753 Å respectively. The C-H…π interactions is seen in compound **3k.** This interaction, in compound **3k**, C15-H15…Cg (Cg is the centroid of naphthalene ring C7-C16) has a value of 2.697 Å (**Figures 3 and 4**).

Figure 3: Packing of the compound **3j** showing intermolecularC-H···π interactions
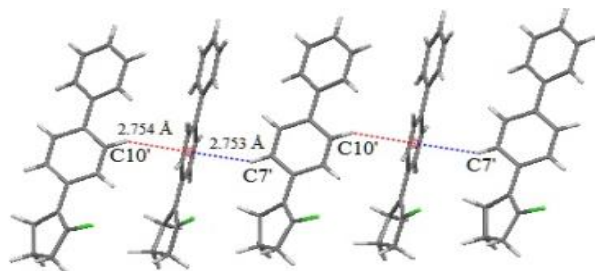
Figure 4: Packing of th
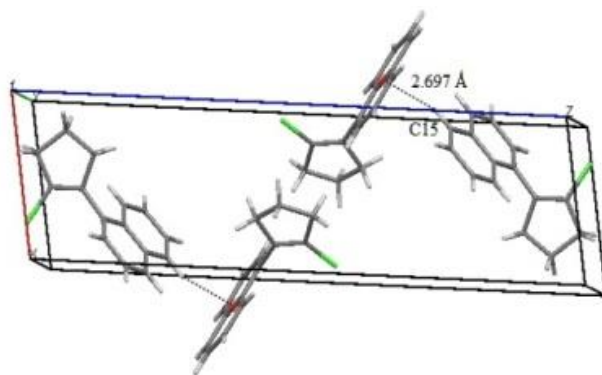
Figure 5: The orientation of the ring structures present in the compounds **3j** and **3k.**

3j   **3k**

The dihedral angles between the planes containing cycloalkenyl and biphenyl **3j** is found to be coplanar,

whereas molecule with naphthalene substituted cycloalkenyl ring **3k** is oriented orthogonally.

Table 2: Non-bonded interactions and possible hydrogen bonds in **3j** and **3k** (Å, °)

Compound	D–H⋯A[a]	D–H	H⋯A	D⋯A	D–H⋯A	
3j	C7'-H7'...Cg [i]	0.950 (2)	2.753 (4)	3.595 (2)	148	[a] D - donor;
	C10'-H10'...Cg [ii]	0.950 (2)	2.754 (2)	3.604 (4)	149	A - acceptor;
3k	C15-H15...Cg [iii]	0.950 (2)	2.697 (3)	3.532 (4)	147	H– hydrogen.

Symmetry codes:(i) -x+1,-y,-z-1, (ii) −x,-y+1,-z, (iii) x+1,+y,+z.

Table 3: Crystal data and refinement parameters for compounds **3j** and **3k.**

	3j	3k
CCDC No	1553574	1553573
Empirical formula	$C_{17}H_{15}Cl$	$C_{15}H_{13}Cl$
Formula weight	254.74	228.70
T [K]	100(2) K	100(2) K
Λ[Å]	0.71073	0.71073
Crystal system	triclinic	triclinic
Space group	$P\bar{1}$	$P\bar{1}$
a [Å]	9.780(2)	7.4359(5)
b [Å]	10.385(2)	7.6628(5)
c [Å]	13.578(3)	23.3162(16)
α [°]	93.131(6)	81.474(3)
β [°]	107.106(6)	80.862(2)
γ [°]	97.036(7)	61.022(2)
V [Å^3]	1280.4(5)	1143.56(13)
Z	4	4
ρ_{calc} [Mg/m^3]	1.321	1.328
μ [mm^{-1}]	0.276	0.300
F(000)	536	480
Crystal size [mm]	0.18 x 0.16 x 0.16	0.18 x 0.16 x 0.16
θ [°]	2.02 to 25	2.66 to 25.00
Index ranges	-11 ≤ h ≤ 11, -12 ≤ k ≤ 12, -16 ≤ l ≤ 16	-8 ≤ h ≤ 8, -9 ≤ k ≤ 9, -27≤ l ≤ 27
Reflections collected	15246	13789
Independent reflections	4503 [R(int) = 0.0869]	4008 [R(int) = 0.0496]
Completeness to theta	99.8%	99.8 %
	θ = 25.00°	θ = 25.00°
Data/restraints/parameters	4503/0/325	4008 / 0 / 289
Goodness-of-fit on F^2	1.007	0.955
Final R indices [I>2sigma(I)]	R1 = 0.0678, wR2 = 0.1517	R1 = 0.0462, wR2 = 0.0982
R indices (all data)	R1 = 0.1255, wR2 = 0.1766	R1 = 0.0793, wR2 = 0.1067
Largest diff. peak and hole([e Å^{-3}])	0.680 and -0.530	0.270 and -0.270
Refinement method	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2

REFERENCES

38. Bruker, SMART, SAINT-Plus, SADABS; Bruker Axs inc.: Madison, Wisconcin, USA, **1998**.

39. Sheldrick, G. M. *Acta Cryst. A*. **2008,** *64*, 112-122.
    doi:10.1107/S0108767307043930

40. Nardelli, M. *Acta Cryst*. *C*. **1983,** *39,* 1141-1142.
    doi.org/10.1107/S0108270183007696

41. Farrugia, L. J. *J. Appl. Cryst*. **2012,** *45*, 849-854.
    doi:10.1107/S0021889812029111

42. Spek, A. L. *Acta Cryst. D*. **2009,** *65*, 148-155.*Acta Cryst.* (2009).
    doi:10.1107/S090744490804362X

43. Watkin, D. J.; Prout, C. J.; Pearce, L. J. **1996,** CAMERON. Chemical Crystallography Laboratory, University Of Oxford, England.

44. Brandenburg, K.; Putz, H. *Crystal Impact*. **2005,** GbR, Bonn, Germany.