Supplementary Material

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

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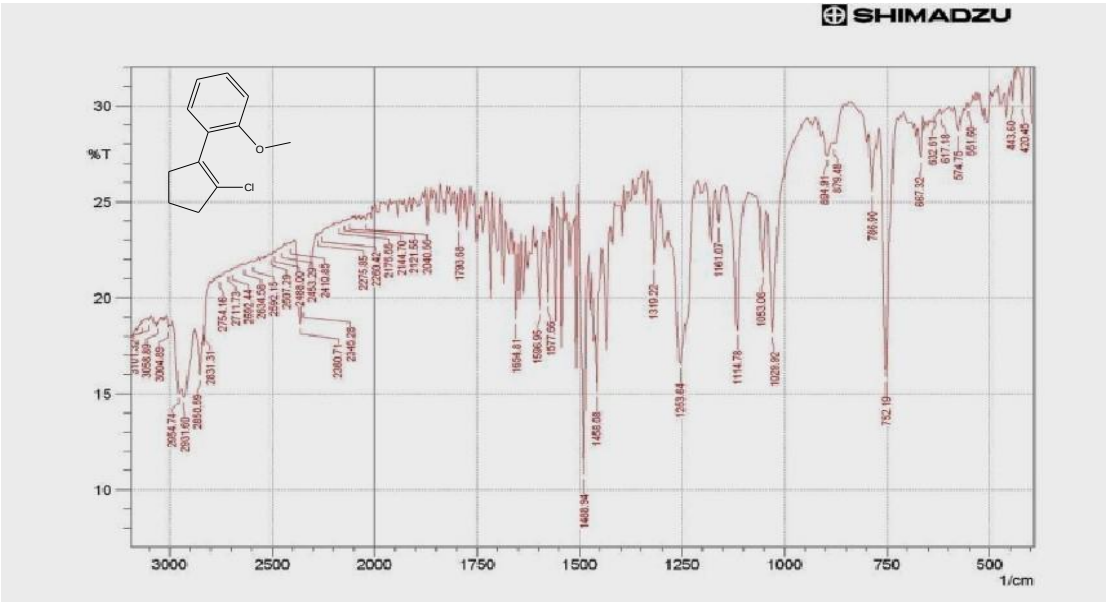
IR of 1-chloro-2-(2′-methoxyphenyl)cyclopenetene 3a:



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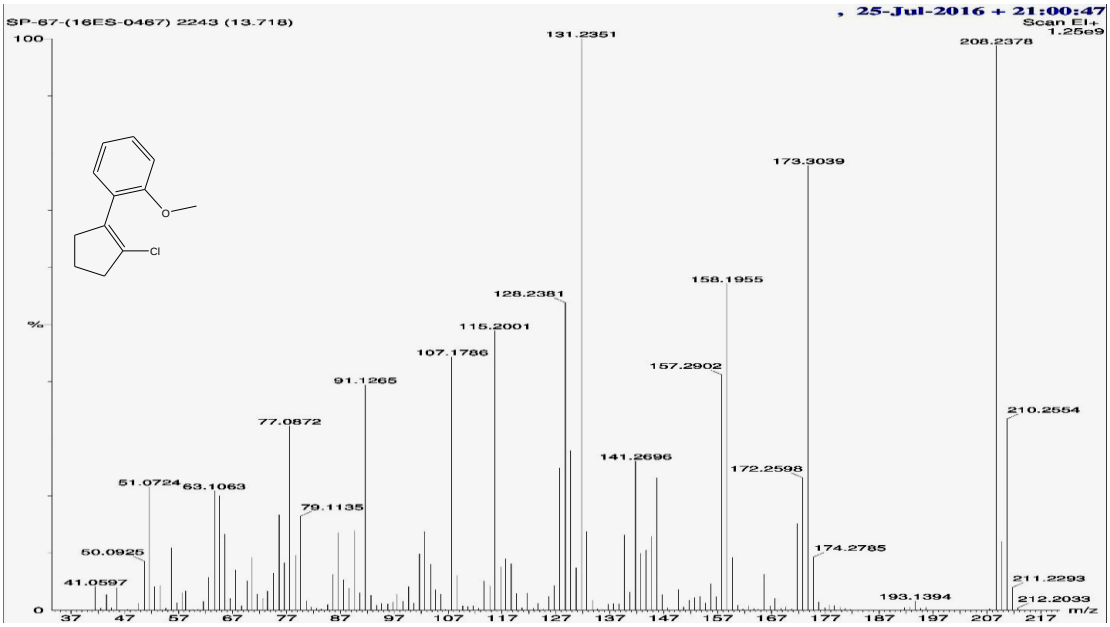

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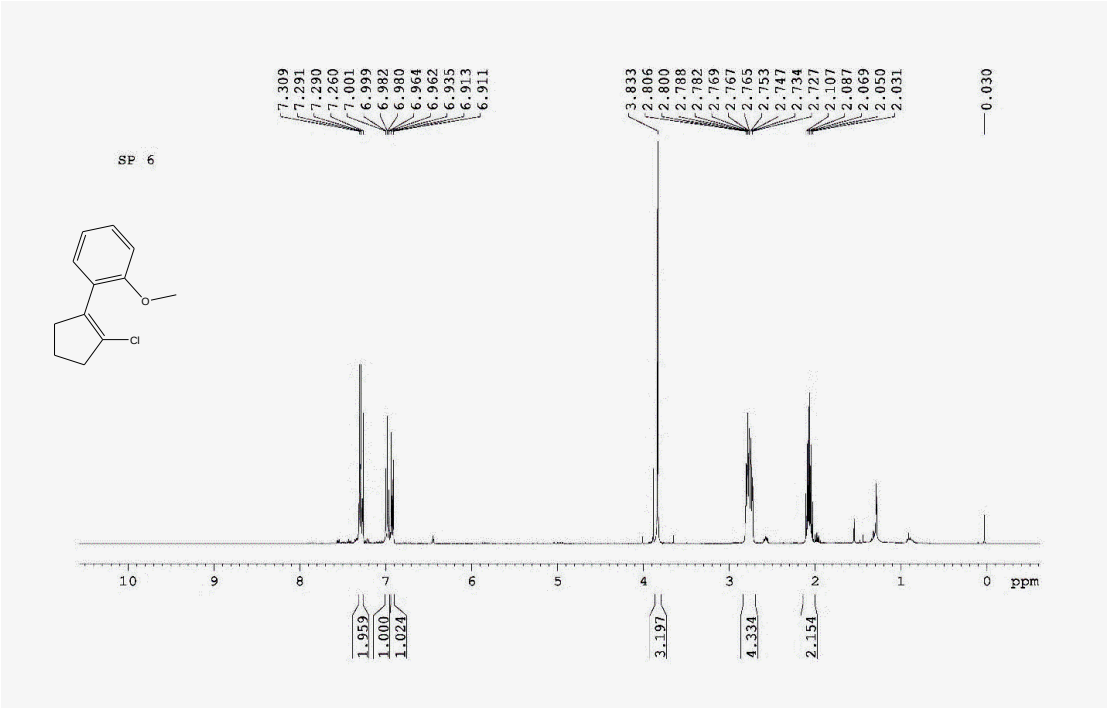

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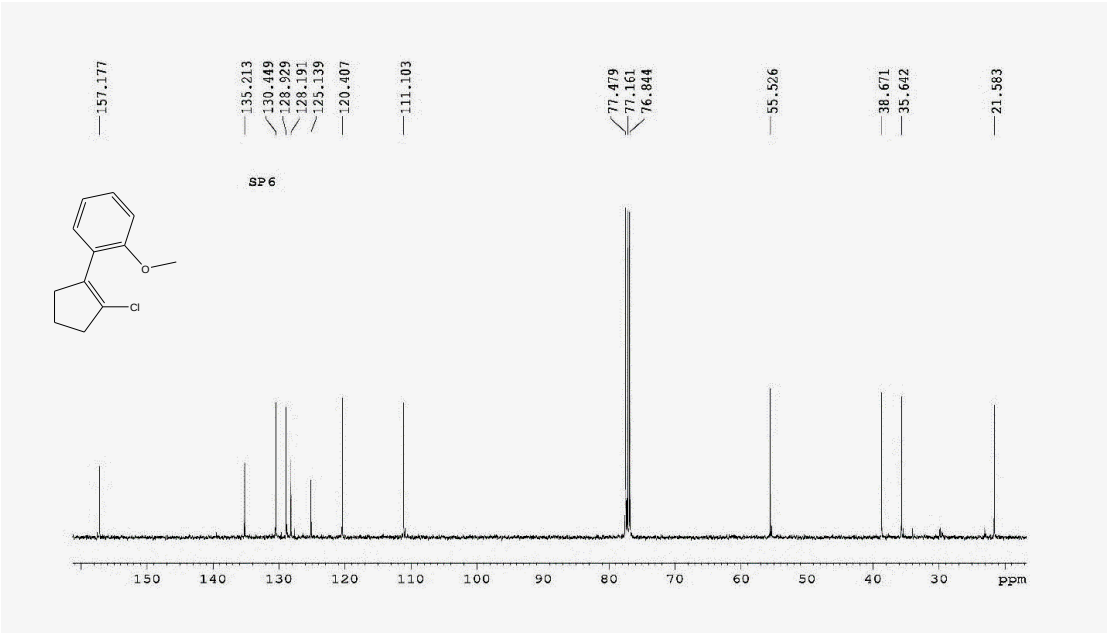

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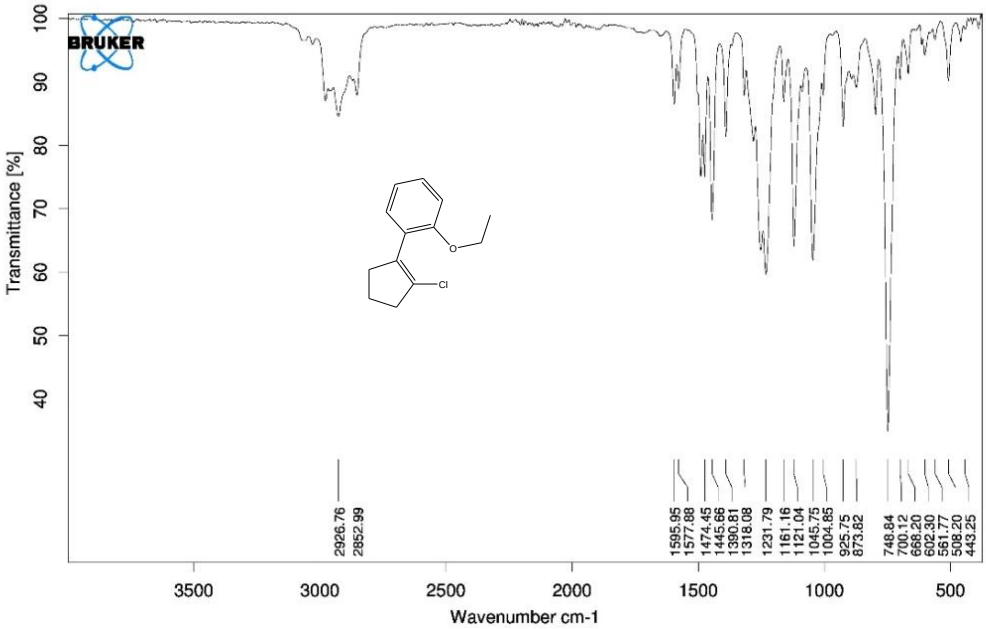

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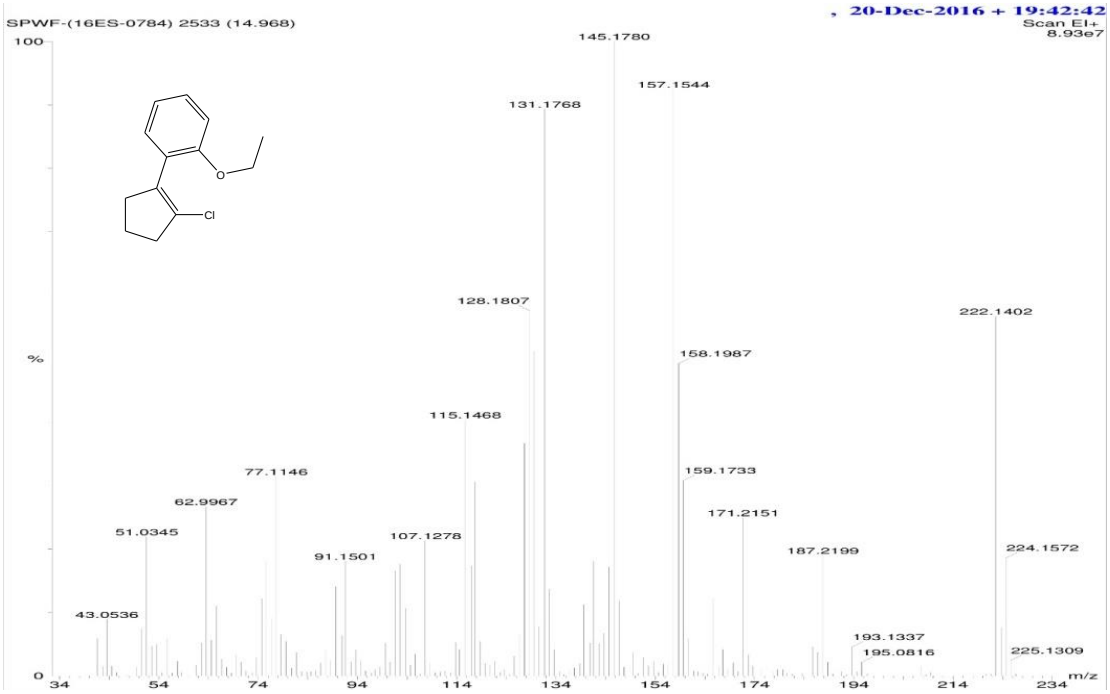

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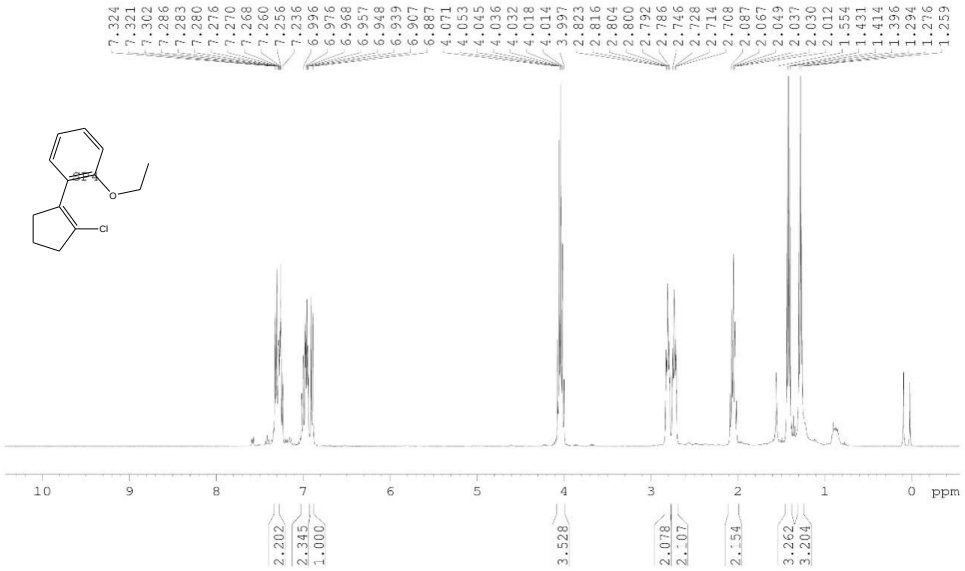

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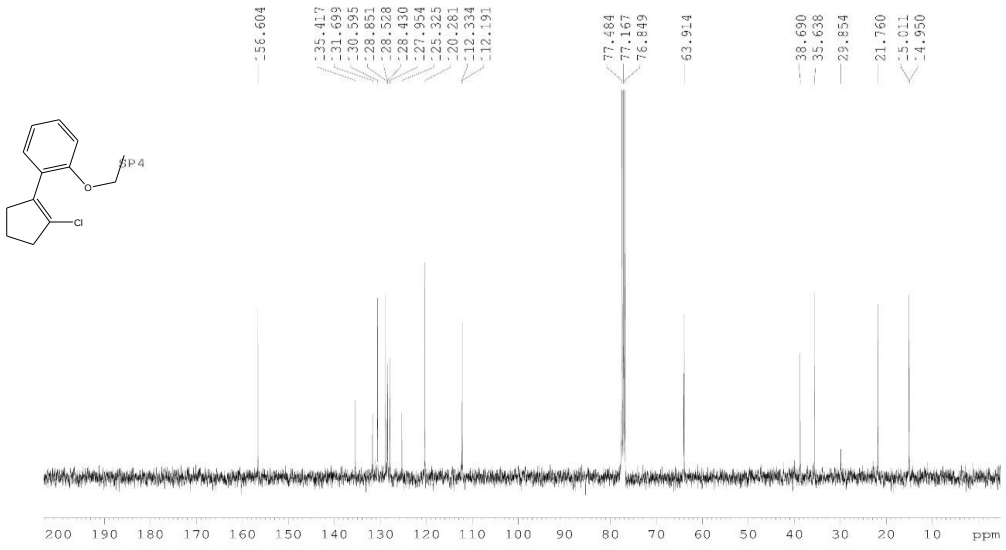

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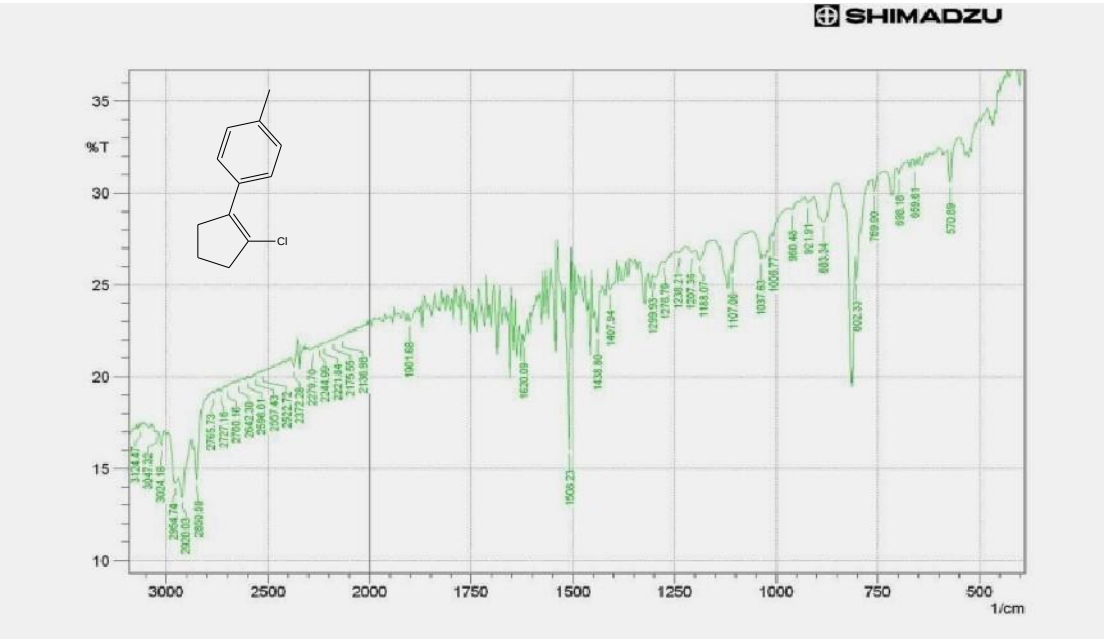

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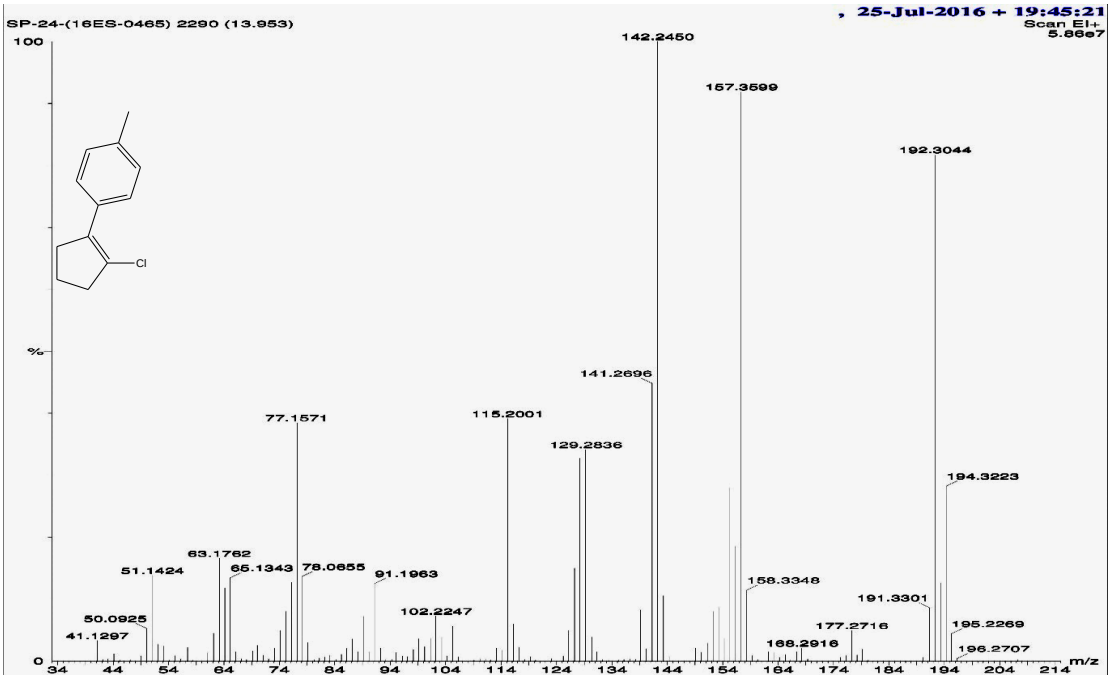

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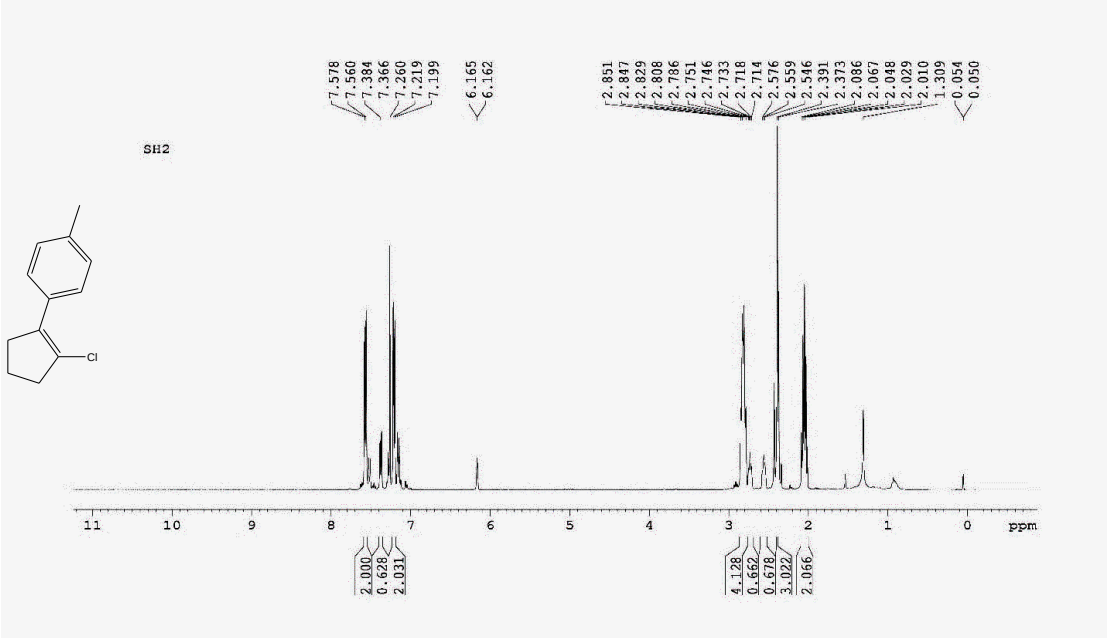

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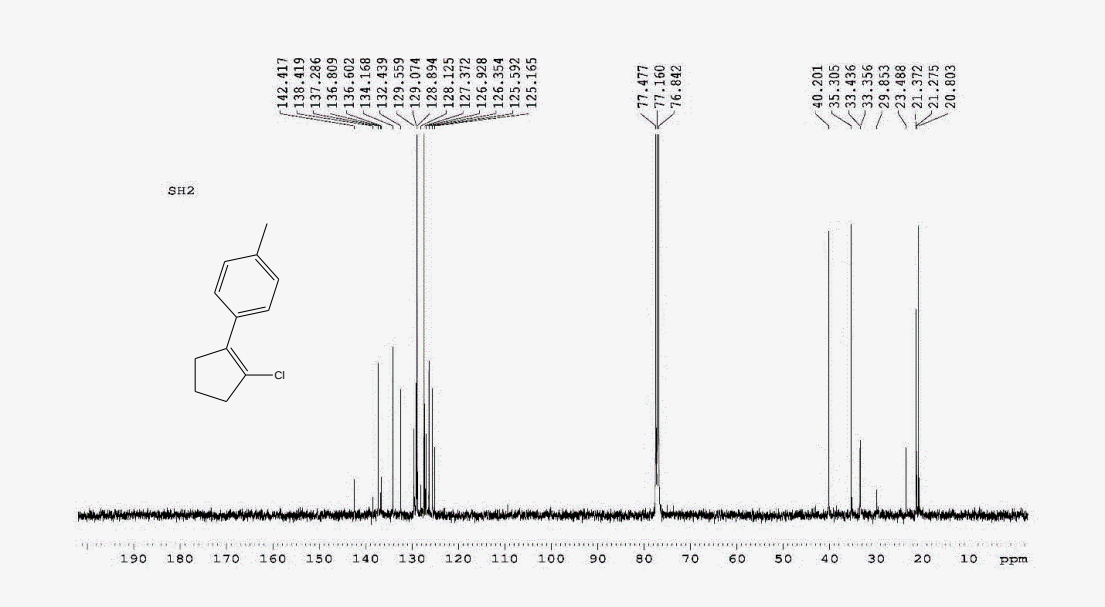

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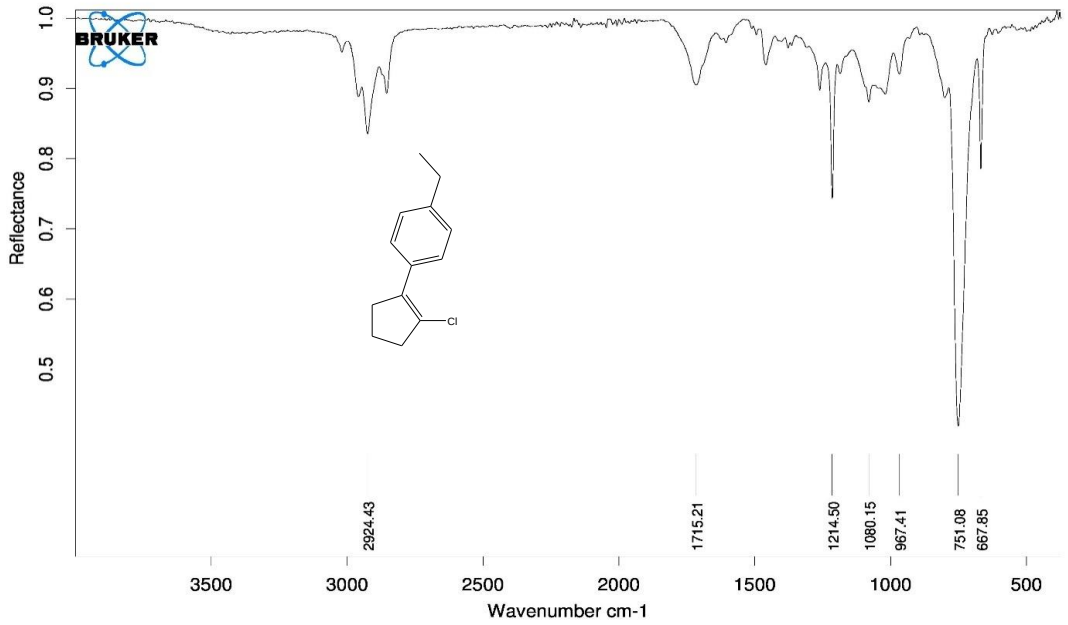

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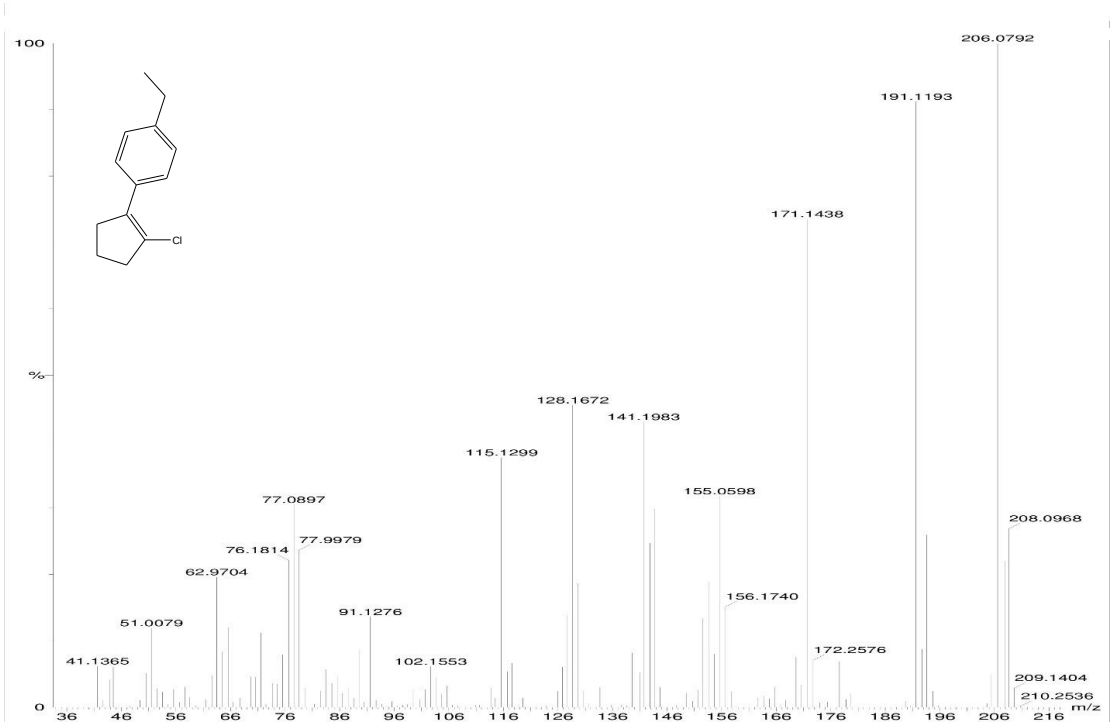

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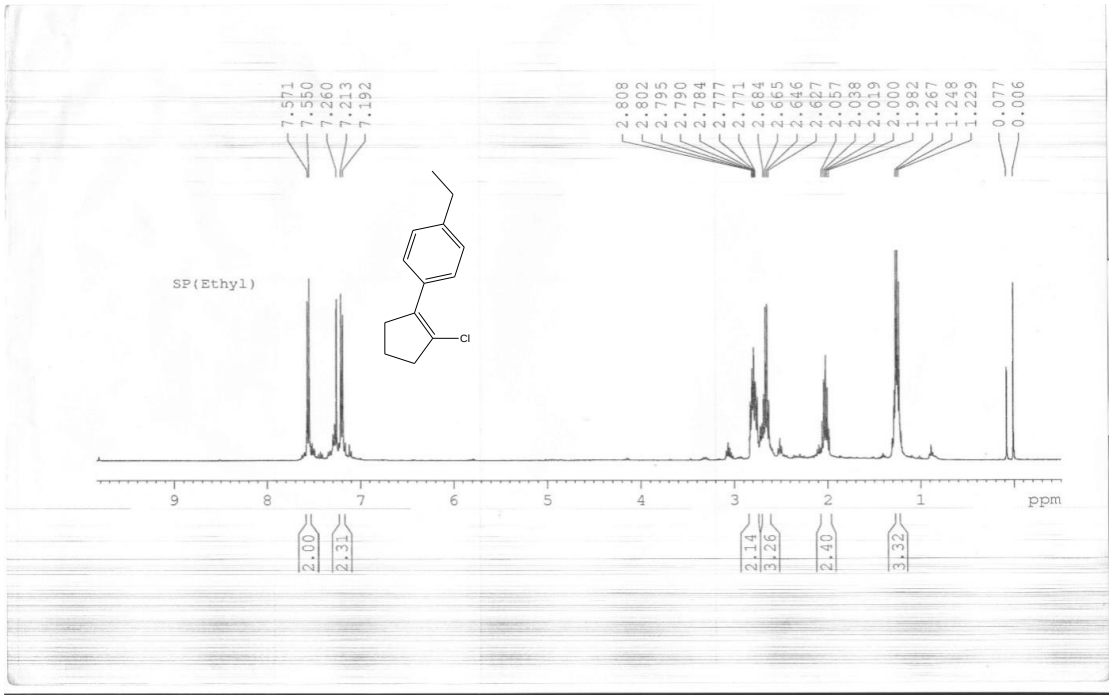

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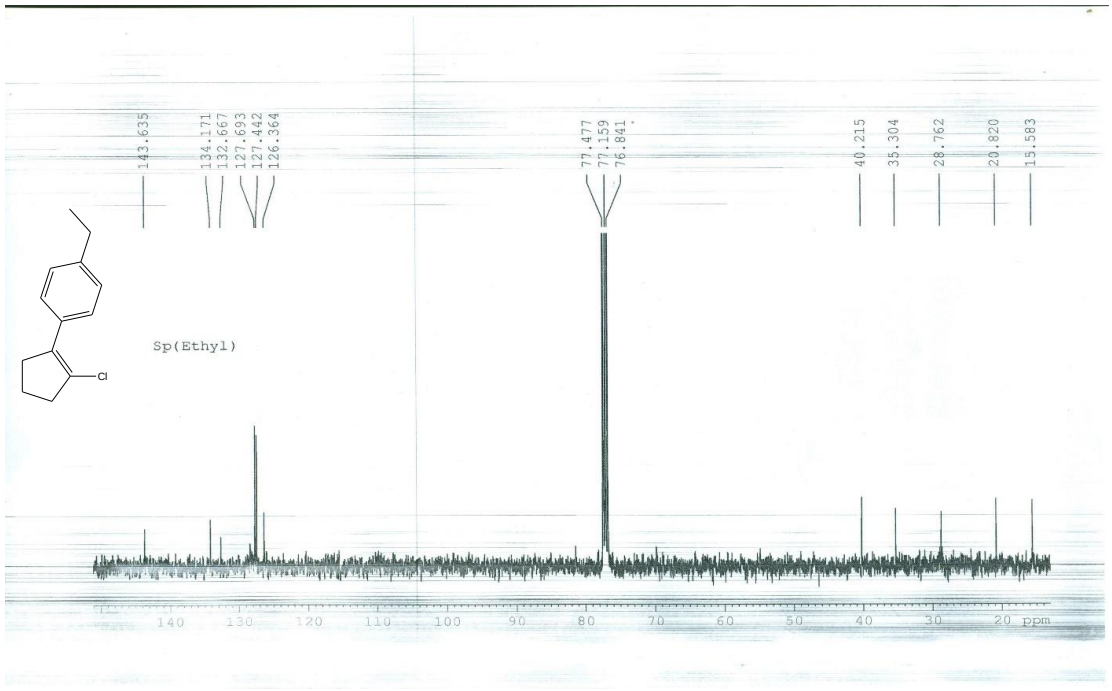

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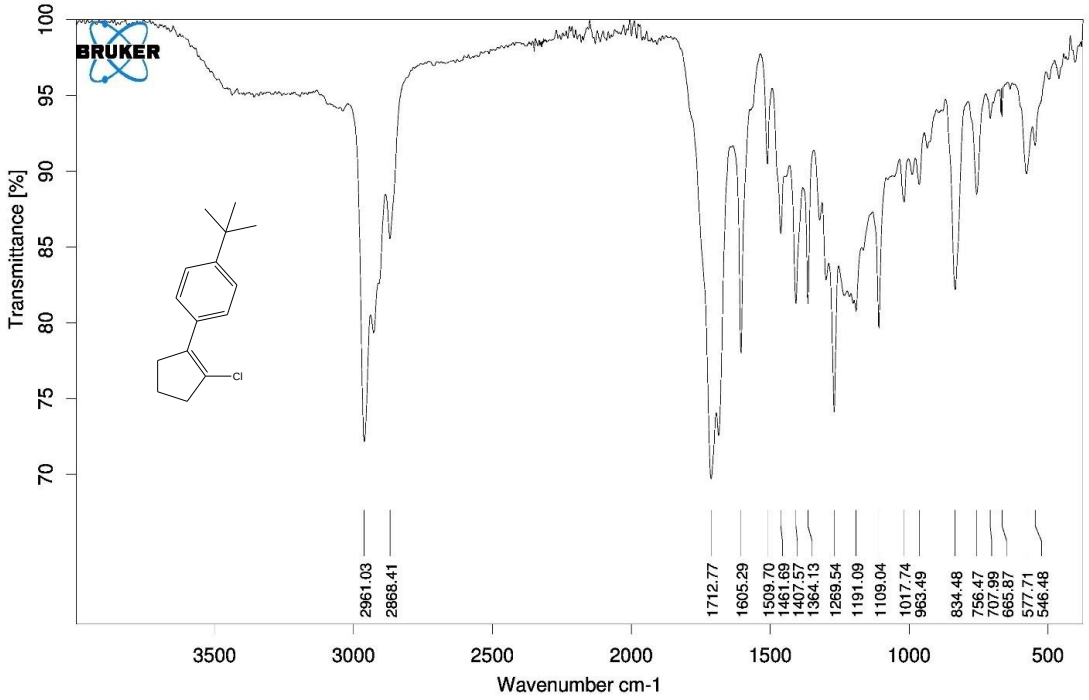

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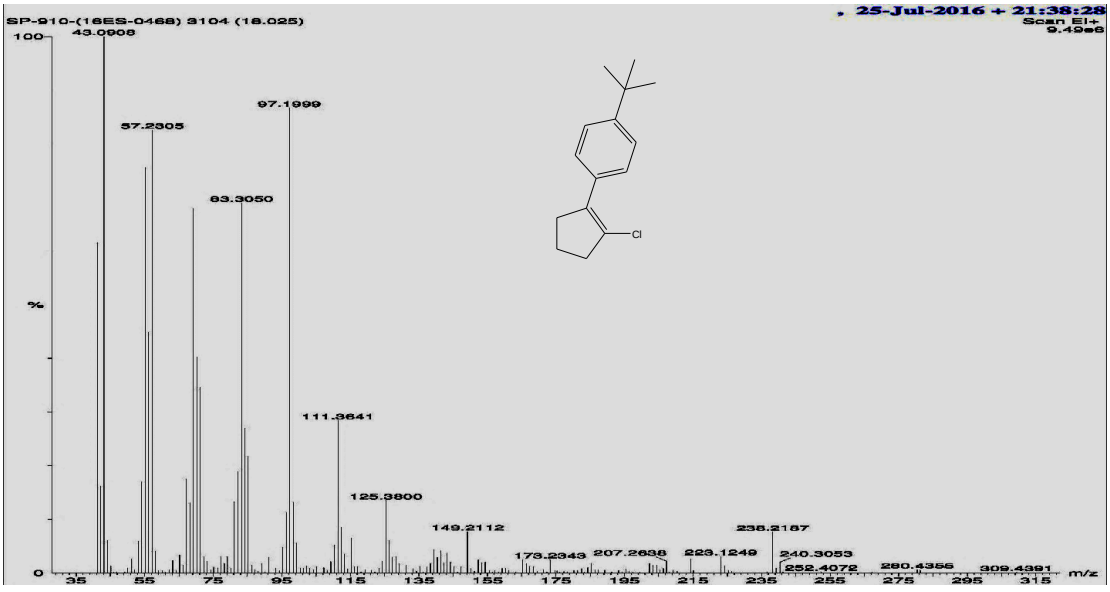

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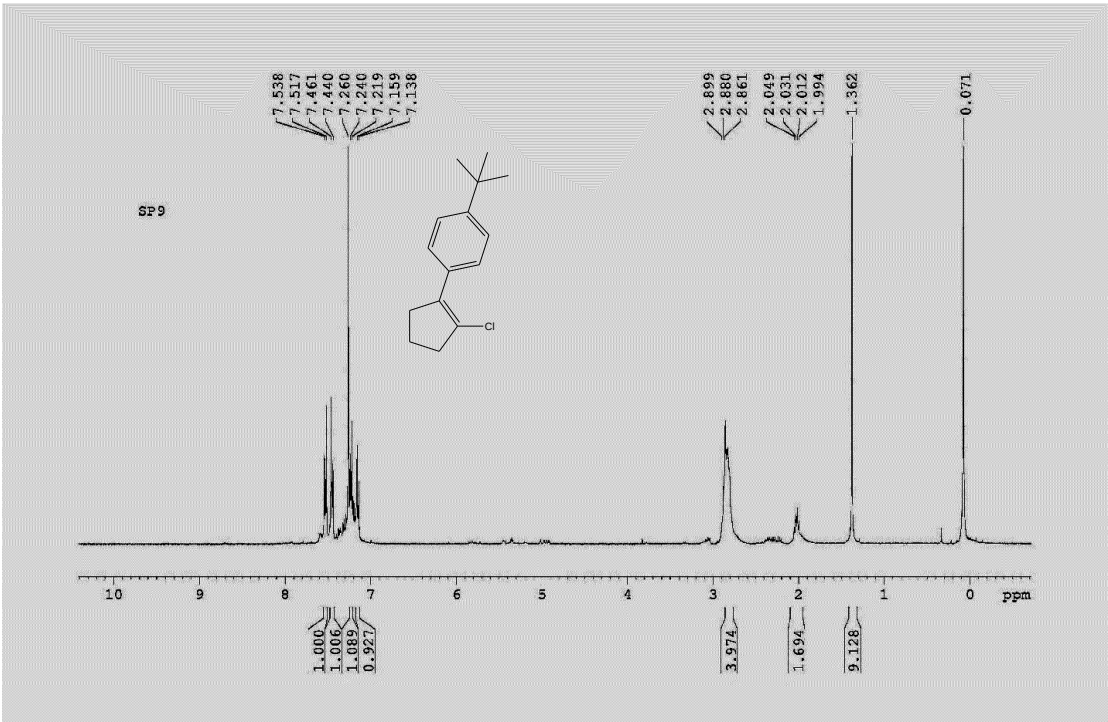

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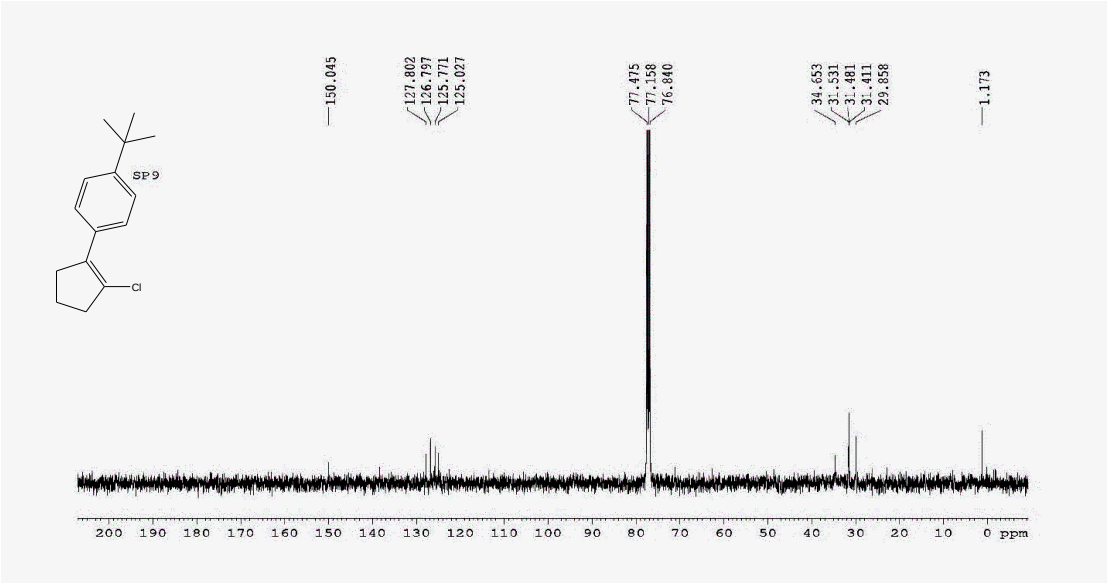

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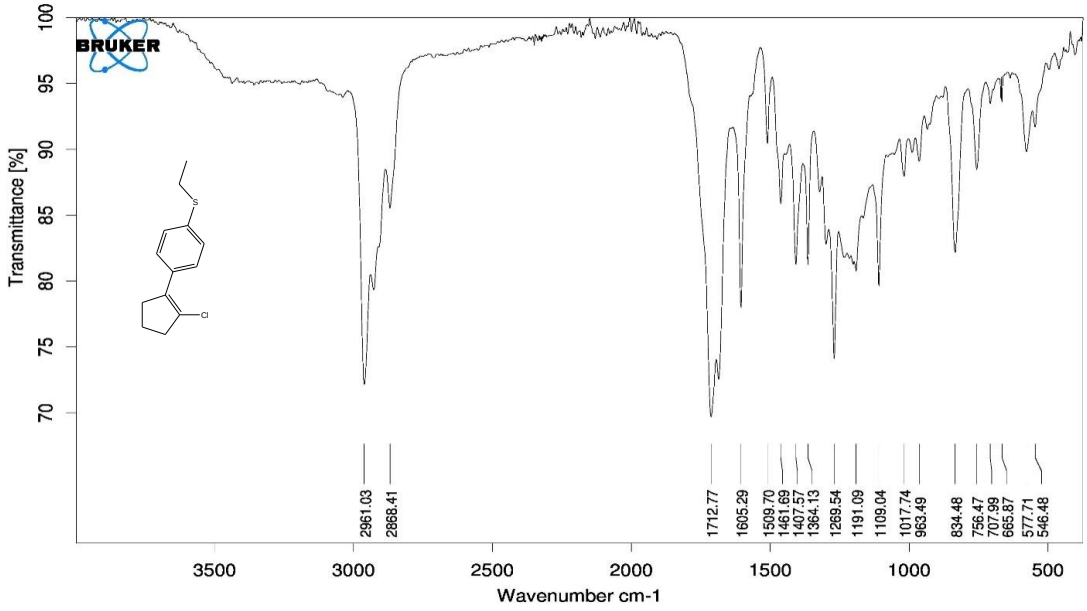

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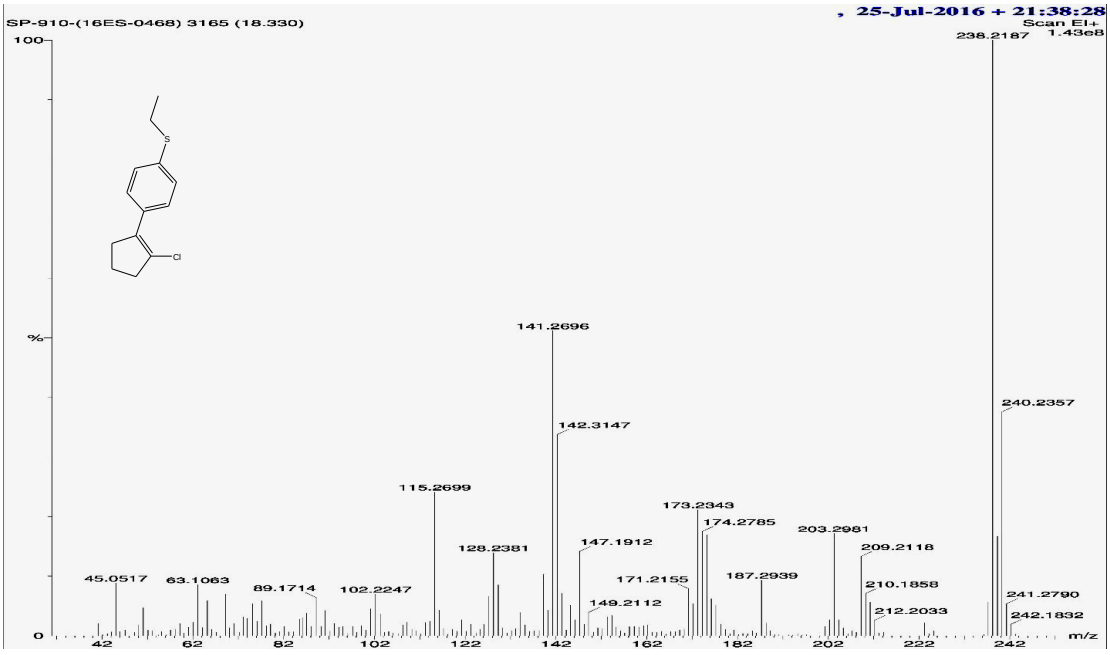

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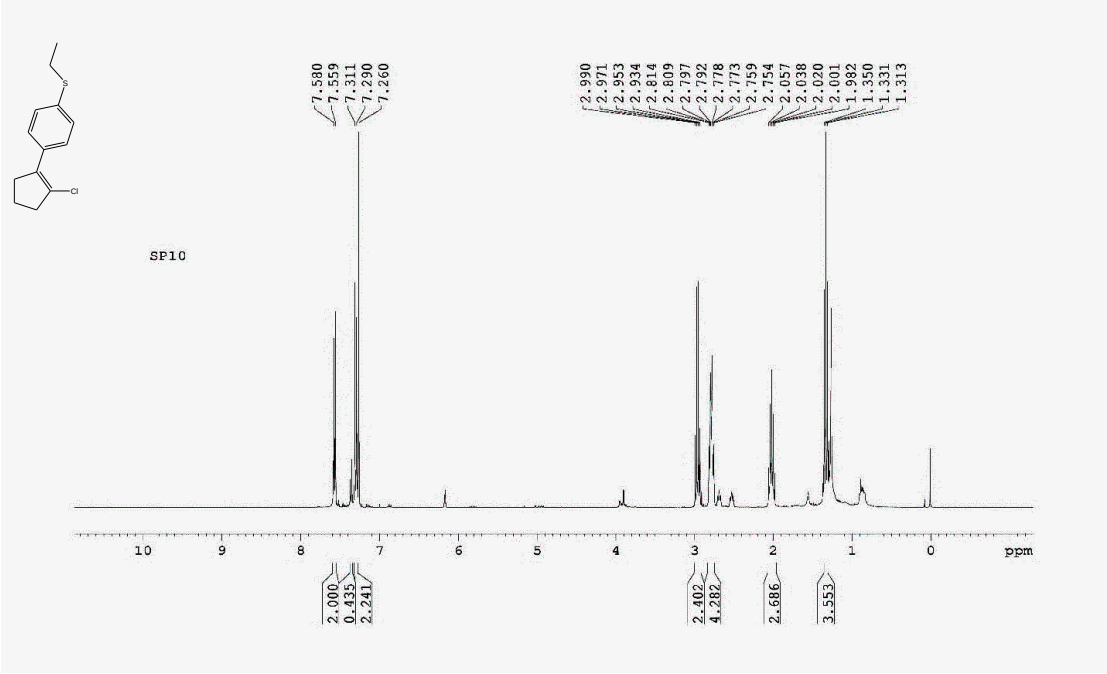

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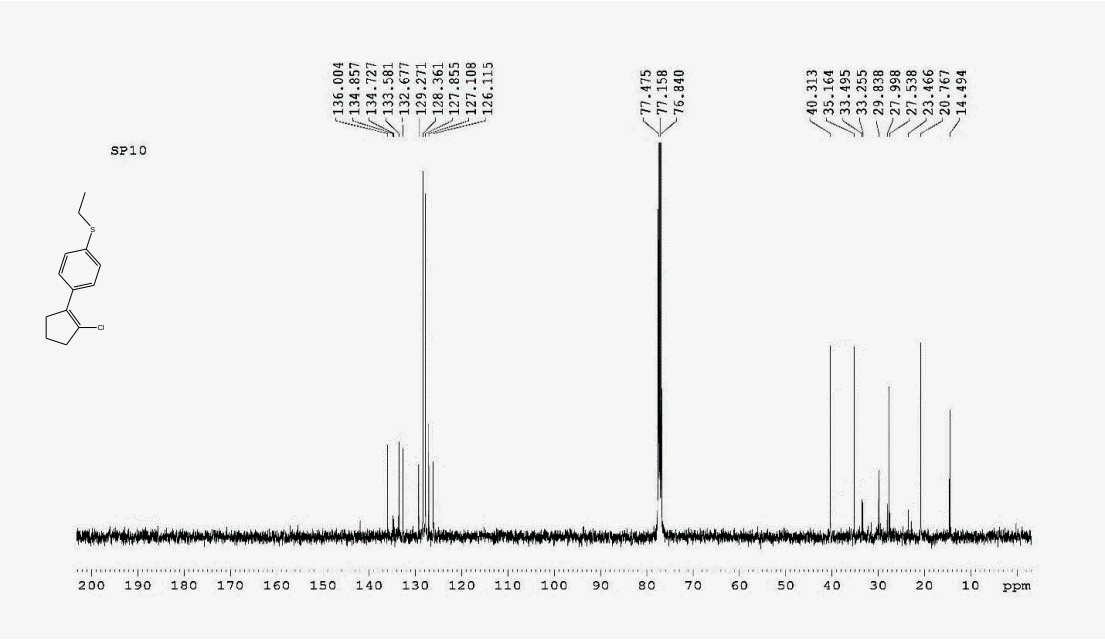

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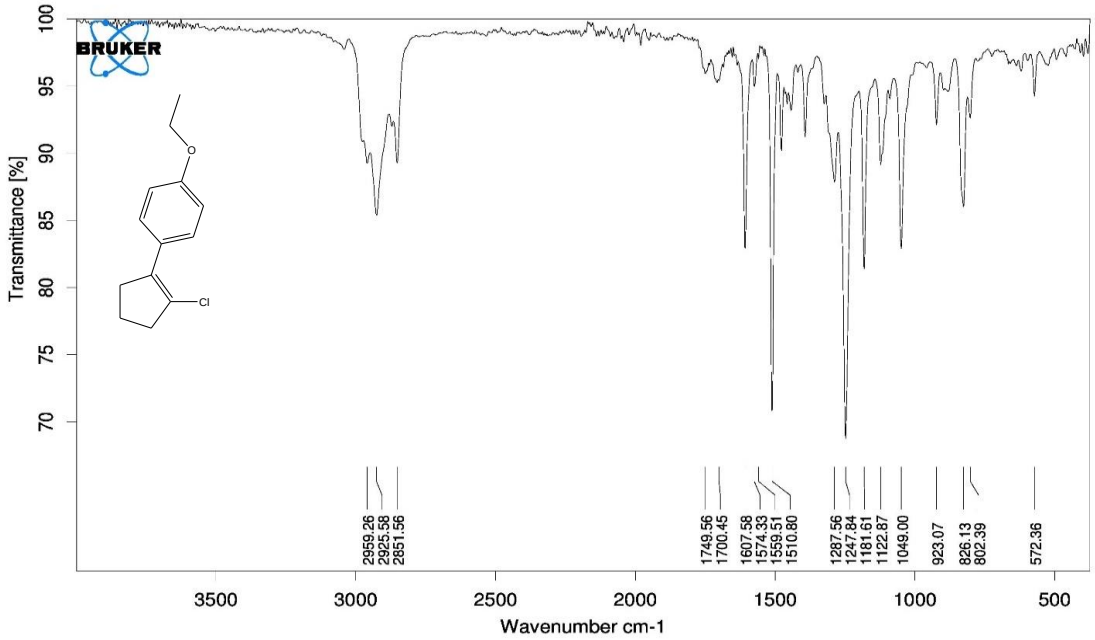

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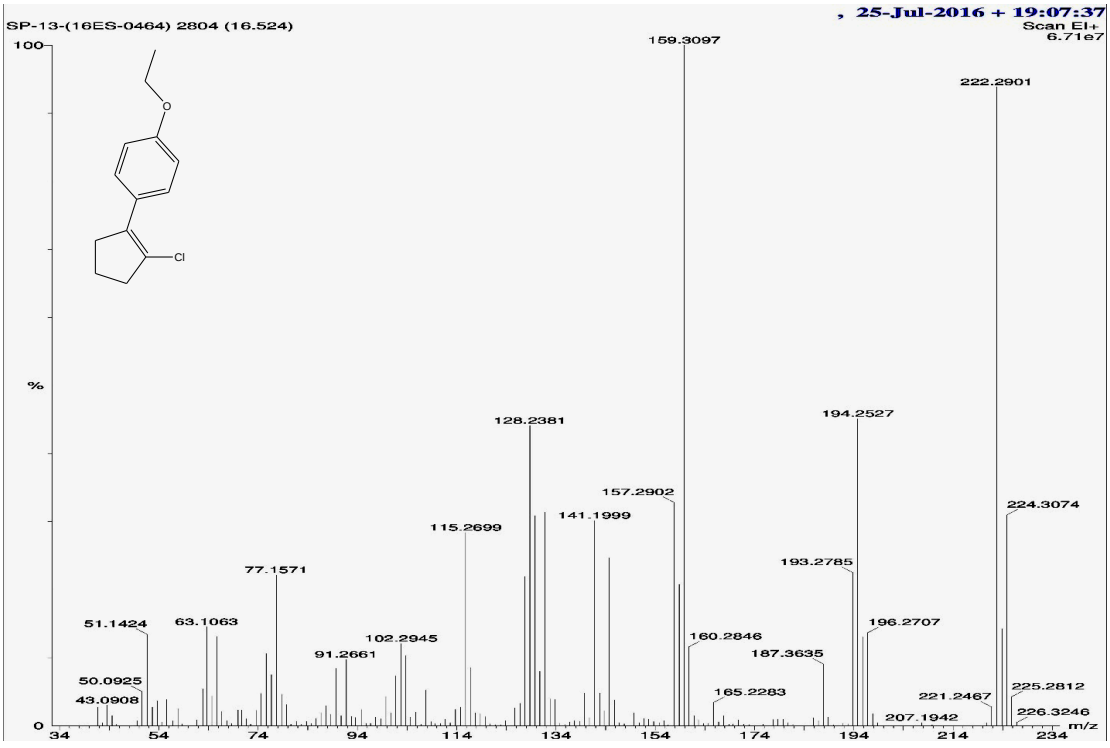

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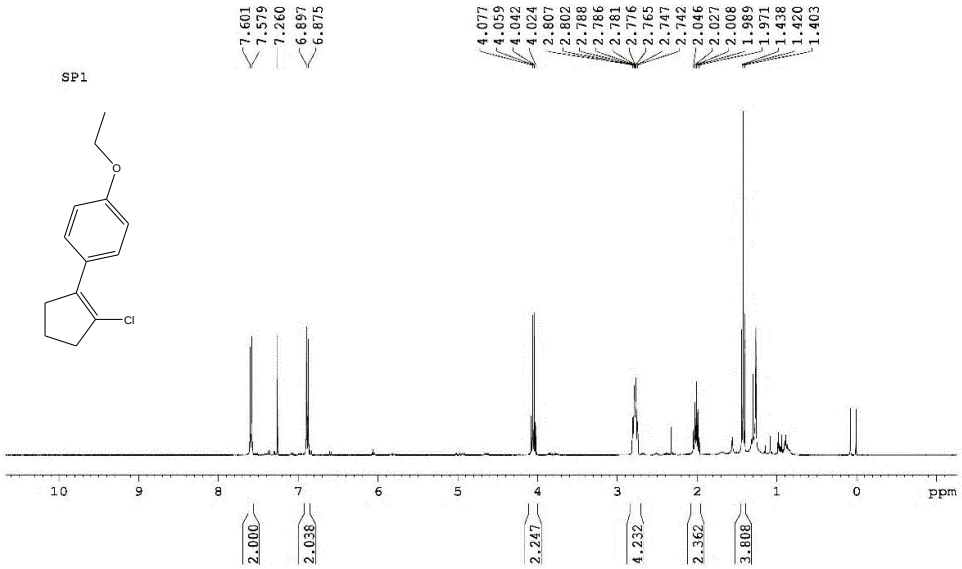

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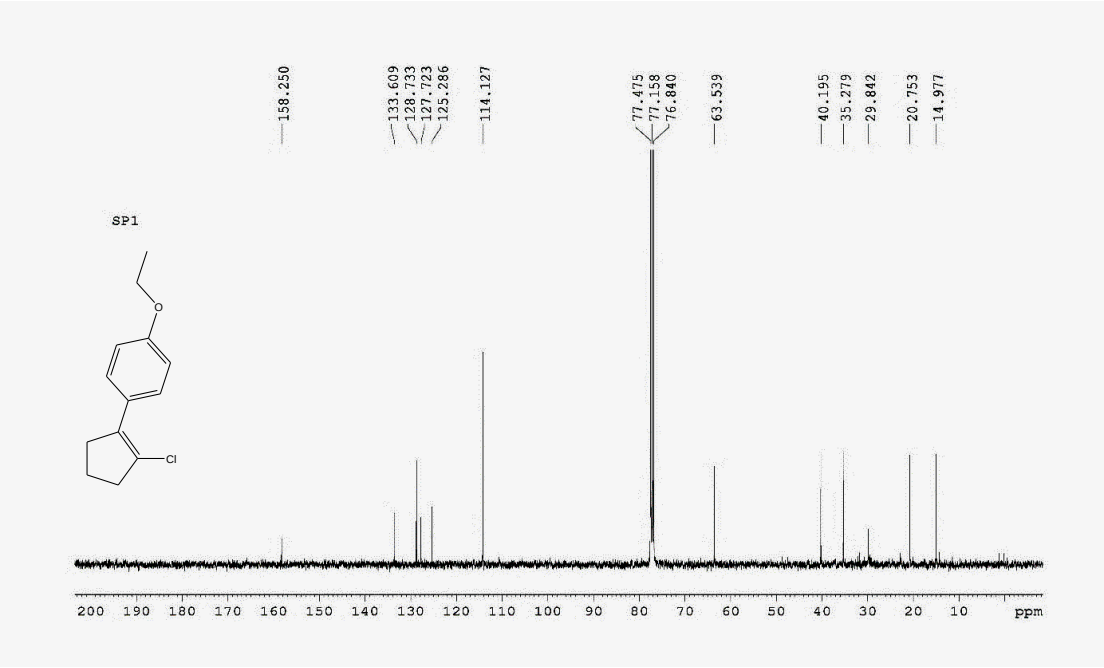
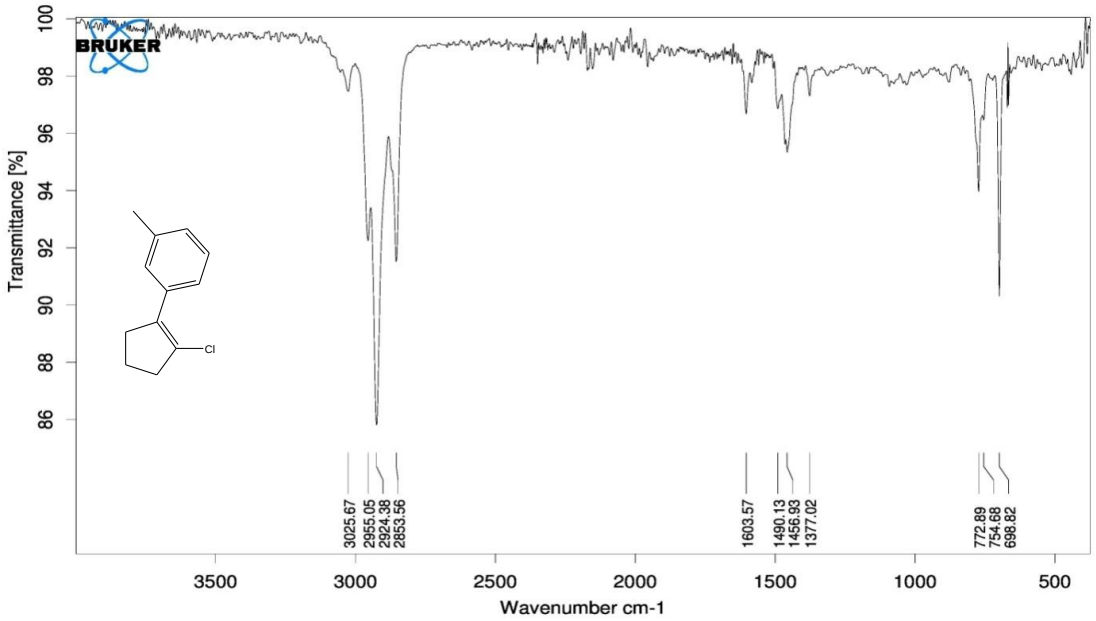


IR of 1-chloro-2-(3′-methylphenyl)cyclopentene 3h:



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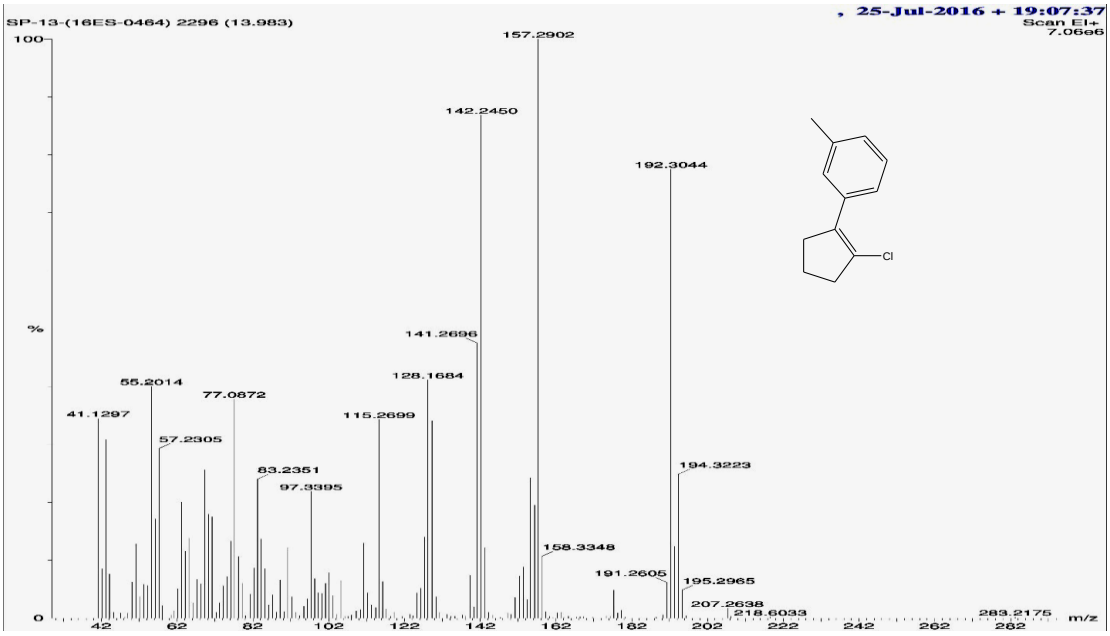

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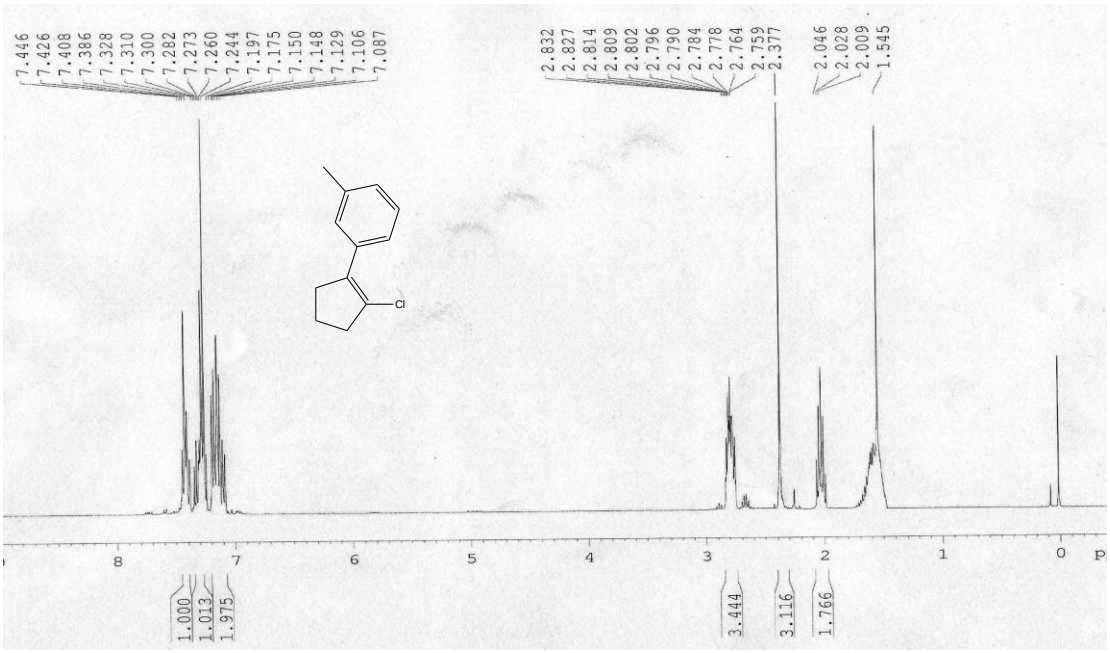

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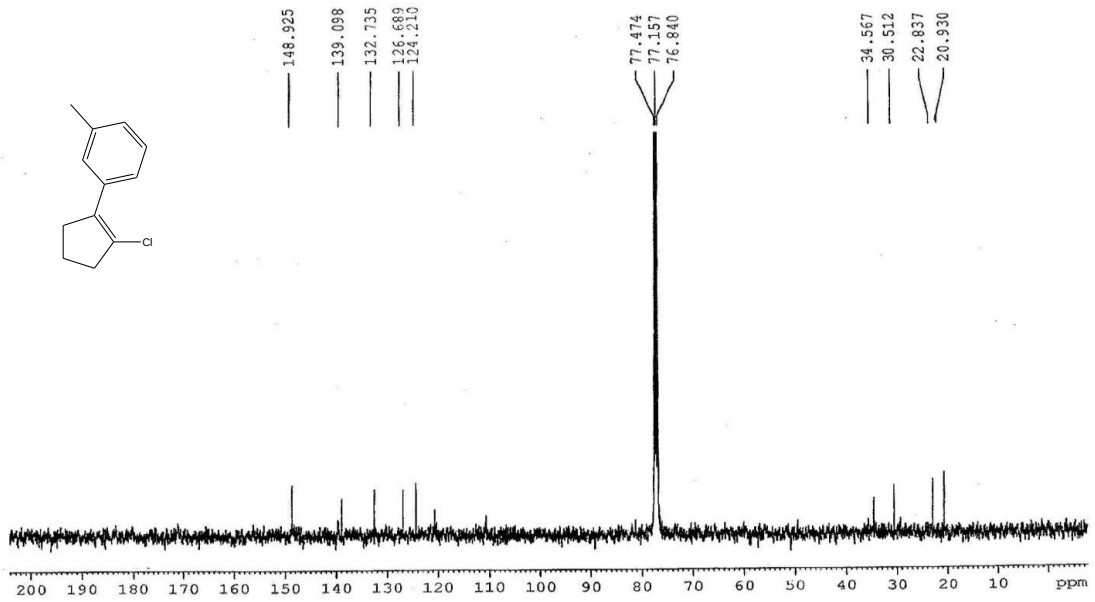

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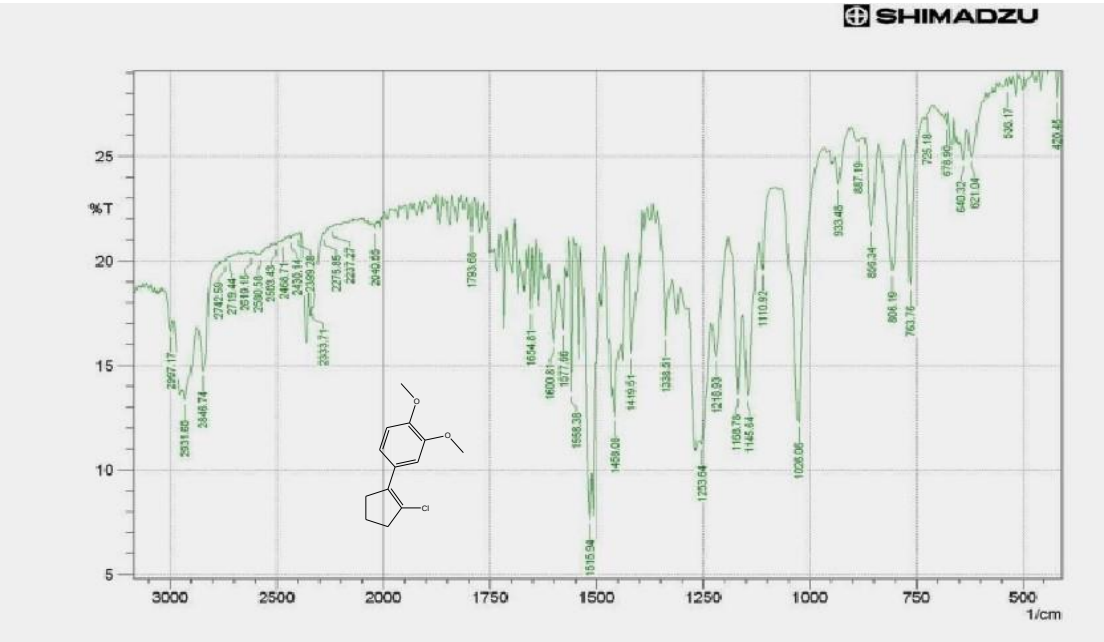

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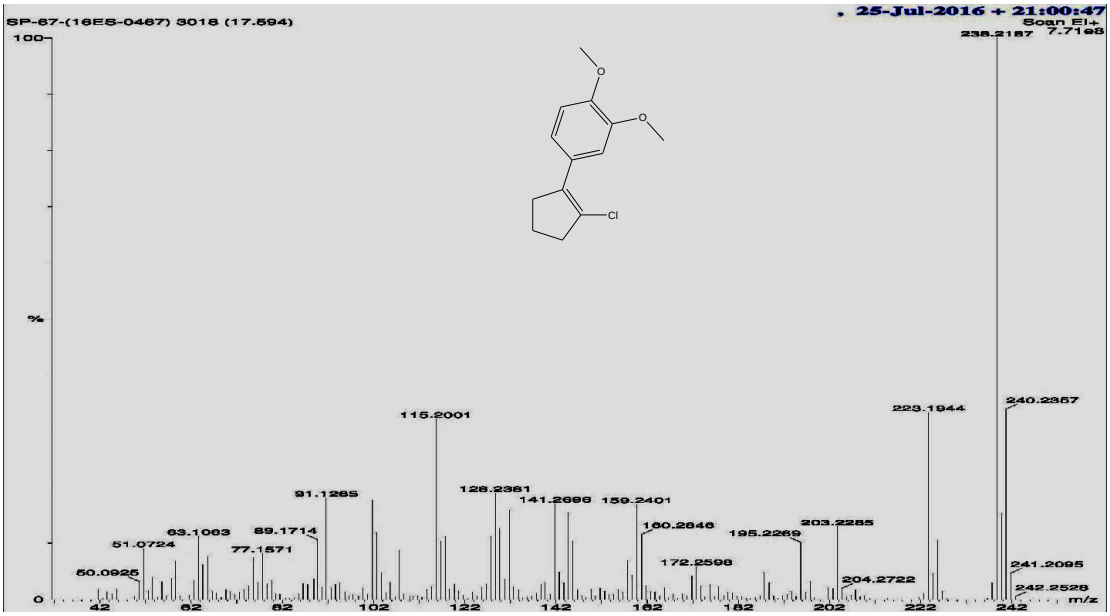

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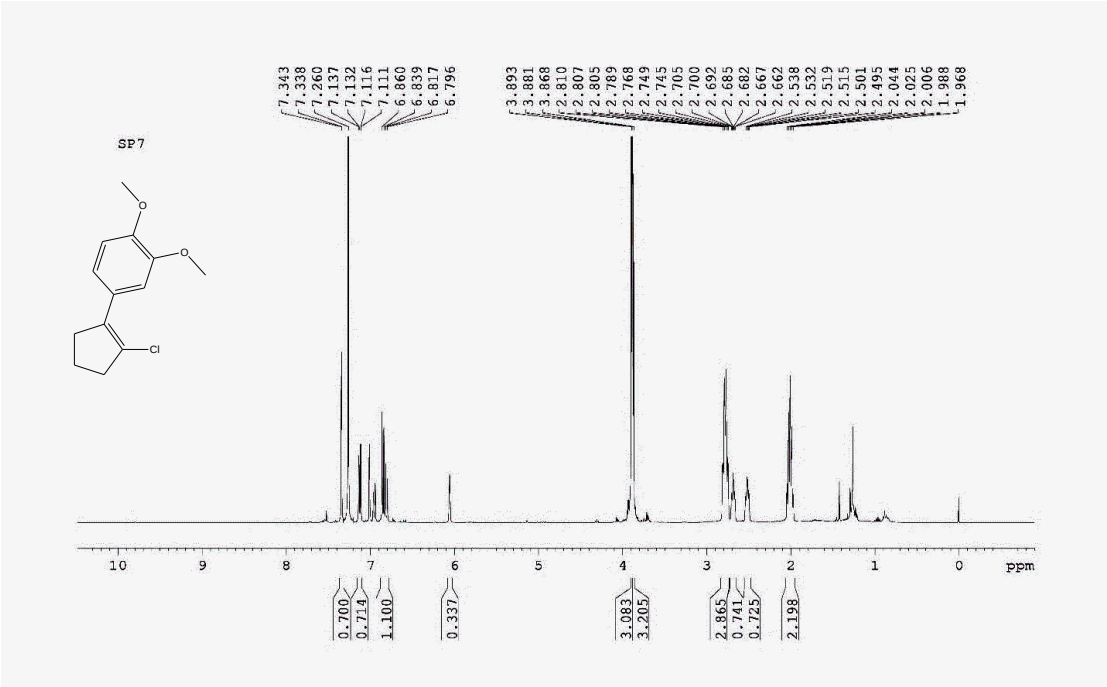

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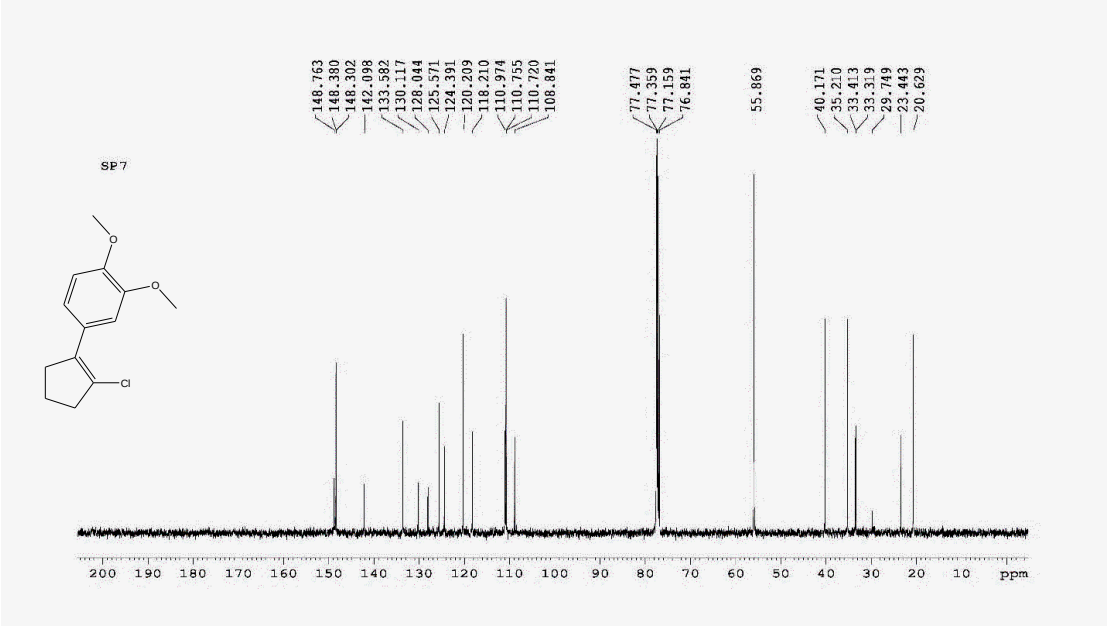

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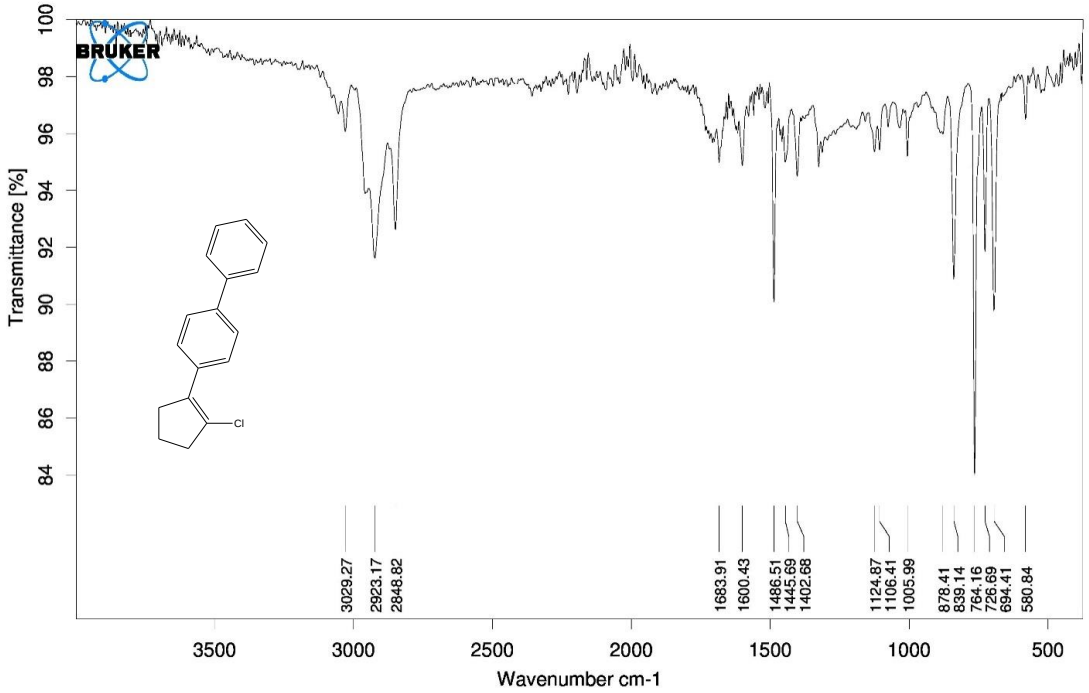

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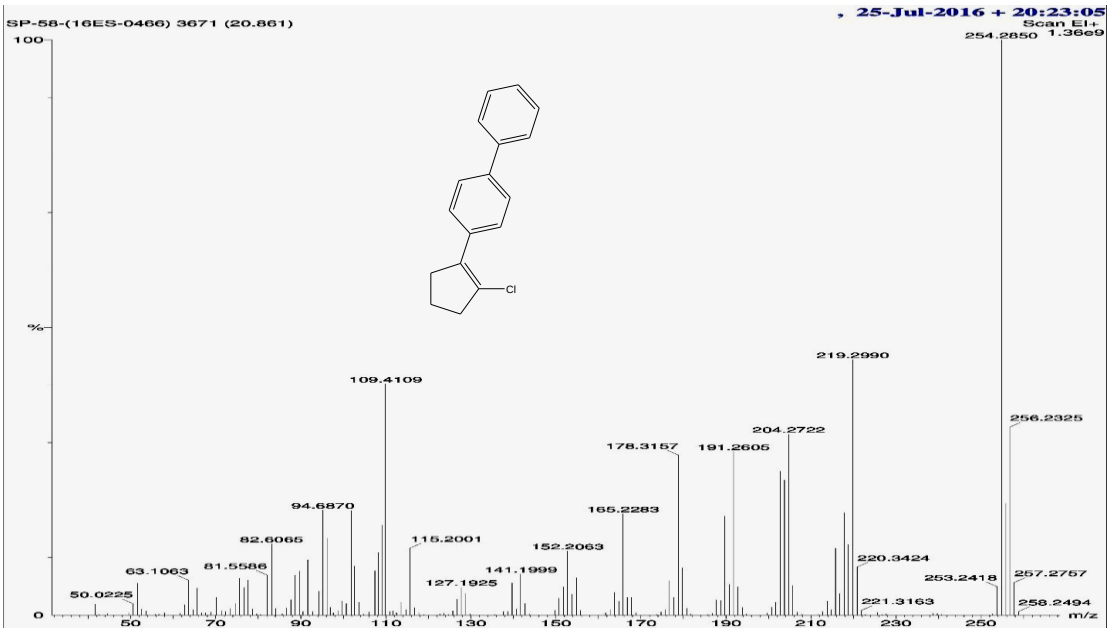

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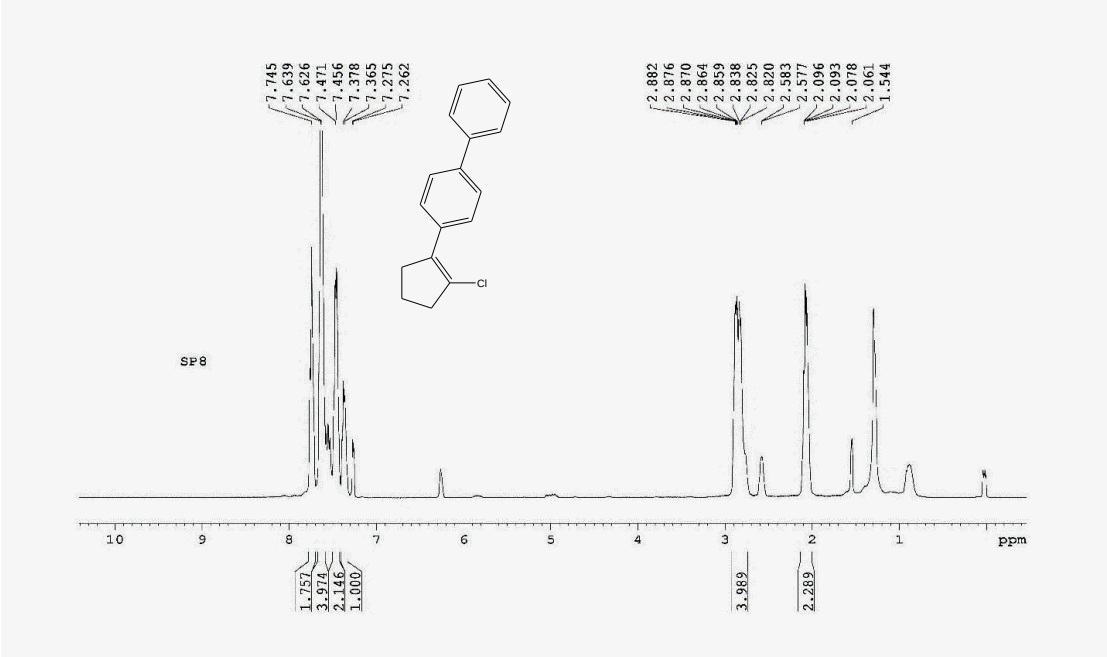

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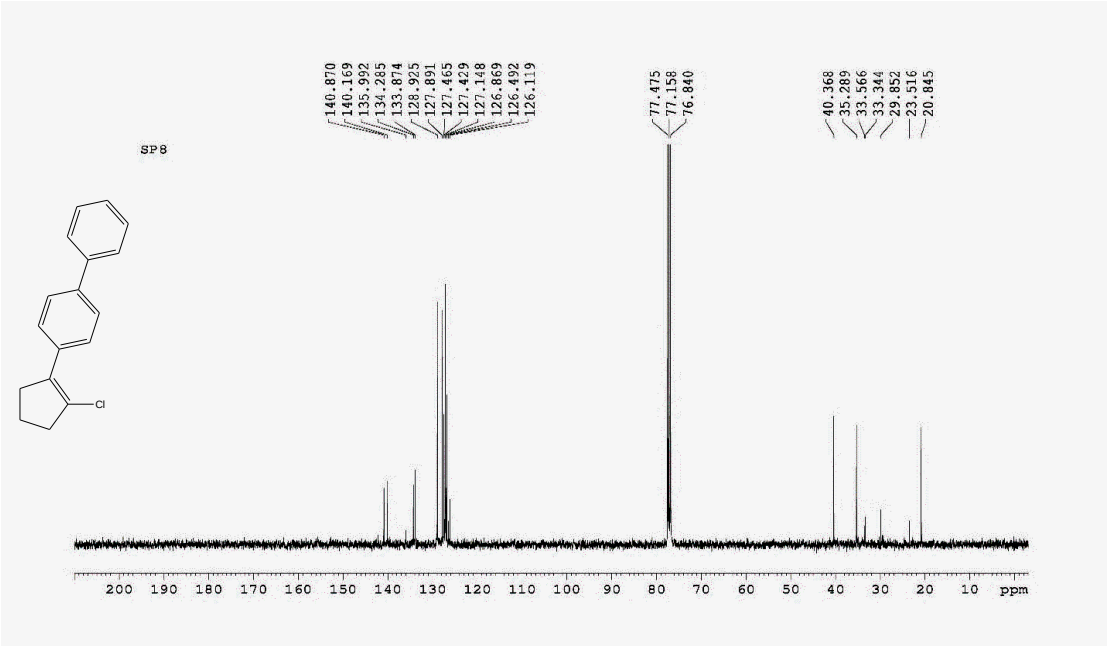

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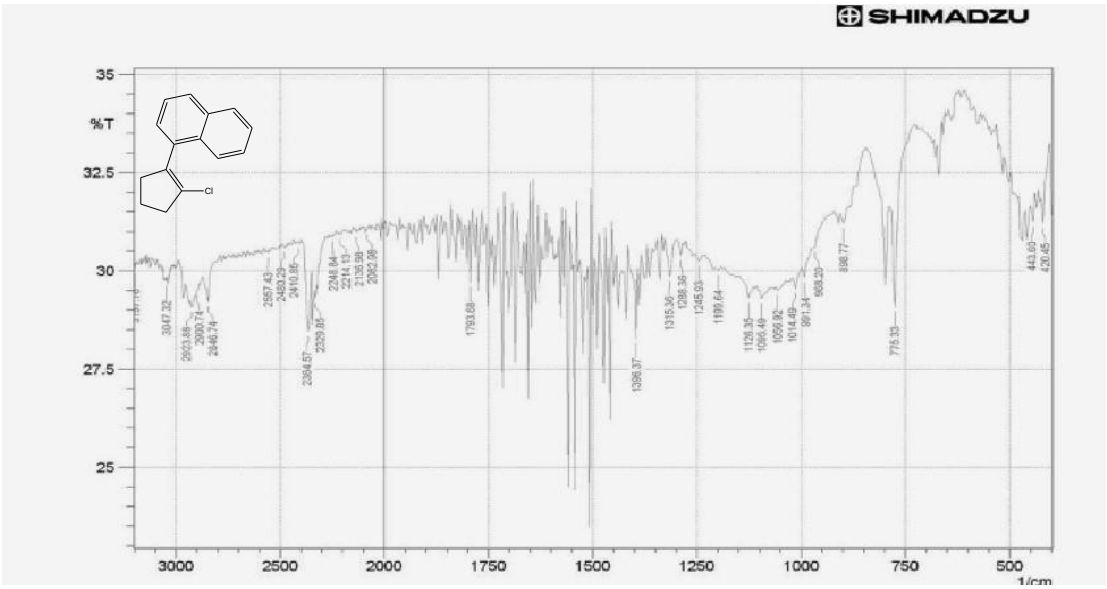

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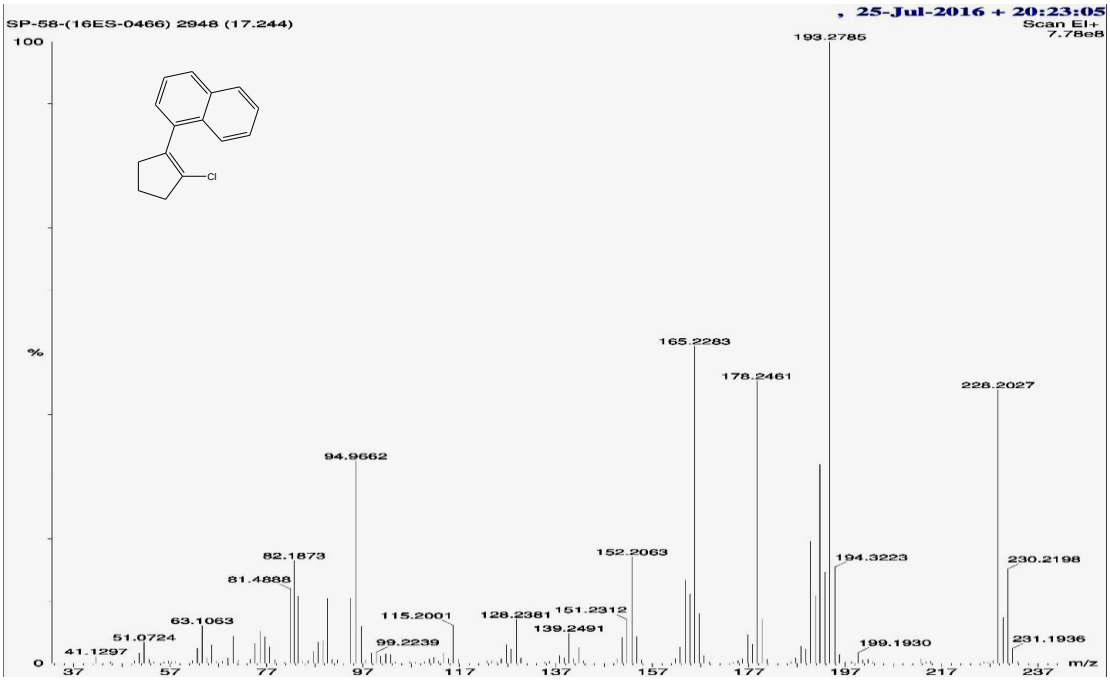

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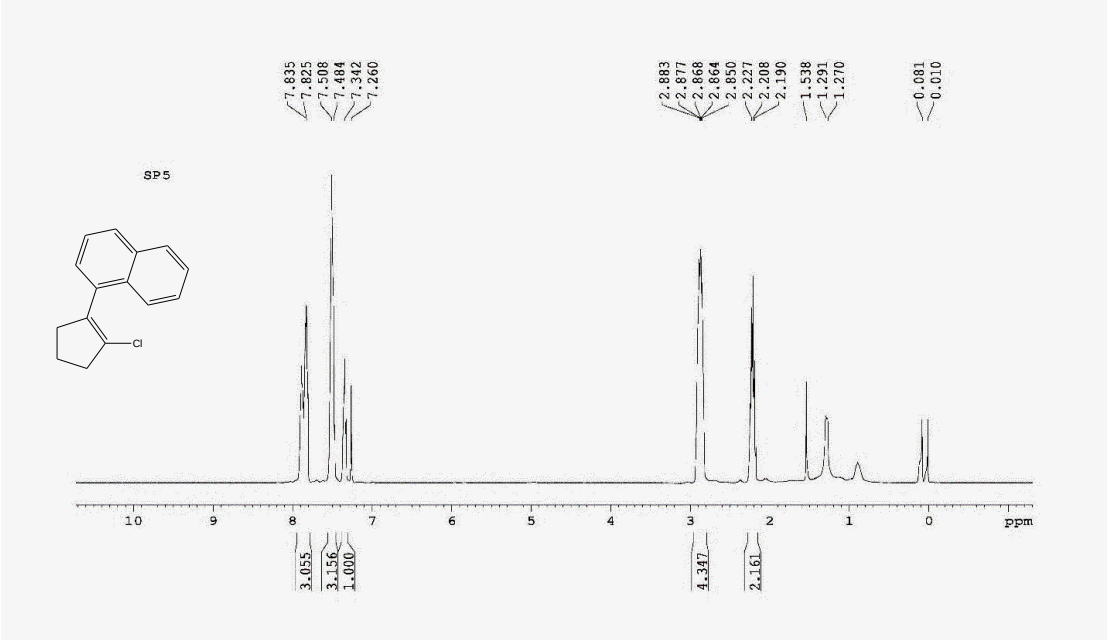

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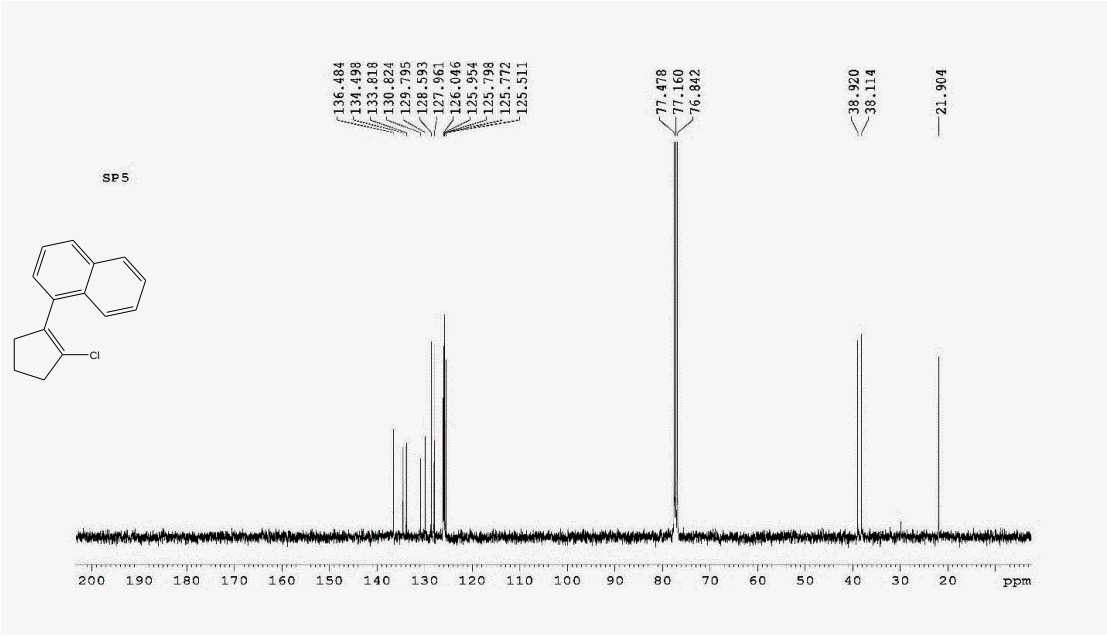


X-ray structure analysis

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

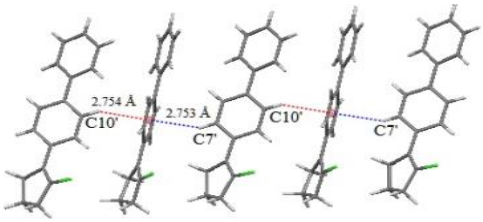
The compounds **3j** and **3k** are not prospective candidates for any robust weak interactions, as all the molecules are devoid of O, N, F, S atoms that would result in hydrogen bonds and other weak interactions . The only weak interactions observed are C-H···Cl and C-H···Cg (**Table 2**).

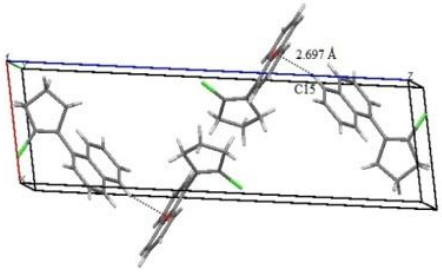
Compound **3j** is stabilized by, C10'-H10'…Cg and C7'-H7'…Cg (Cg is the centroid of aryl ring C6-C11) which forms a chain along 'a' axis with a distance of 2.754 Å and 2.753 Å respectively. The C-H…π interactions is seen in compounds **3k.** This interaction, in compound **3k**, C15-H15…Cg (Cg is the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

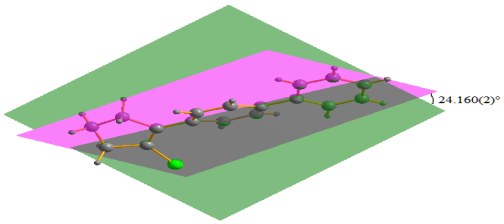
Figure 3: Packing of the compound **3j** showing intermolecular C-H···π interactions and **3k** showing intermolecular C-H···π

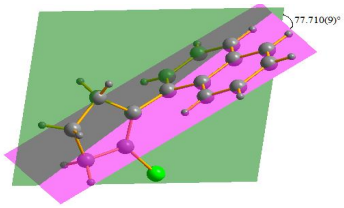




3j **3k**

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	$-11 \le h \le 11$, $-12 \le k \le 12$, $-16 \le l \le 16$	$-8 \le h \le 8$, $-9 \le k \le 9$, $-27 \le l \le 27$
Reflections collected	15246	13789
Independent reflections	4503 [R(int) = 0.0869]	4008 [R(int) = 0.0496]
Completeness to theta	99.8% θ = 25.00°	99.8 % 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
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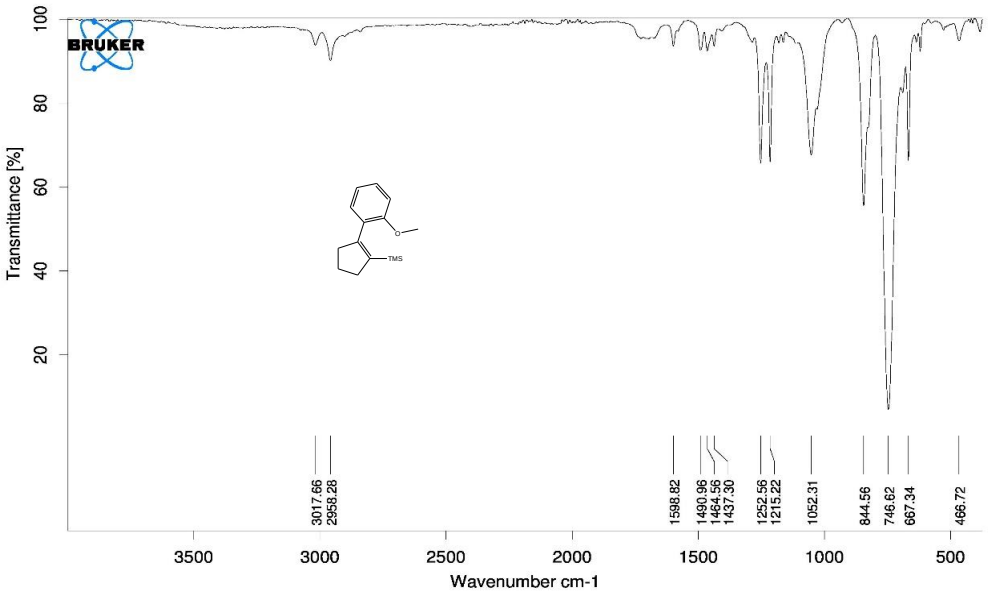
IR of 1-Trimethylsilyl-2-(2′-methoxyphenyl)cyclopenetene 4a:



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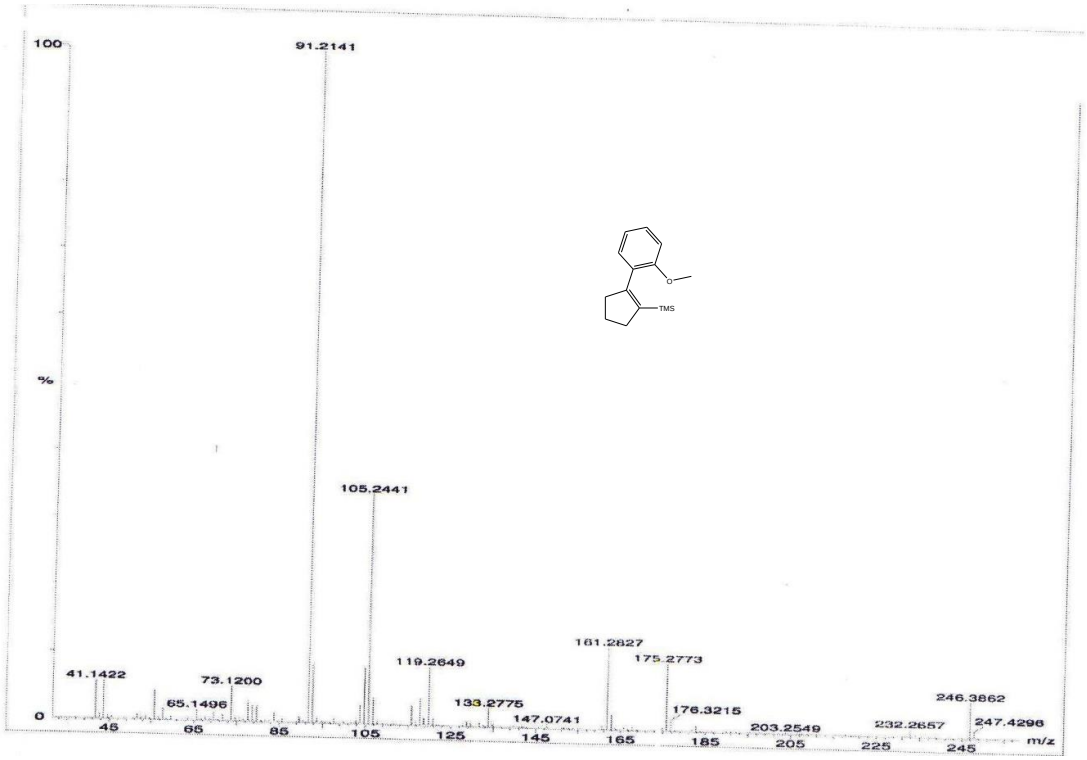

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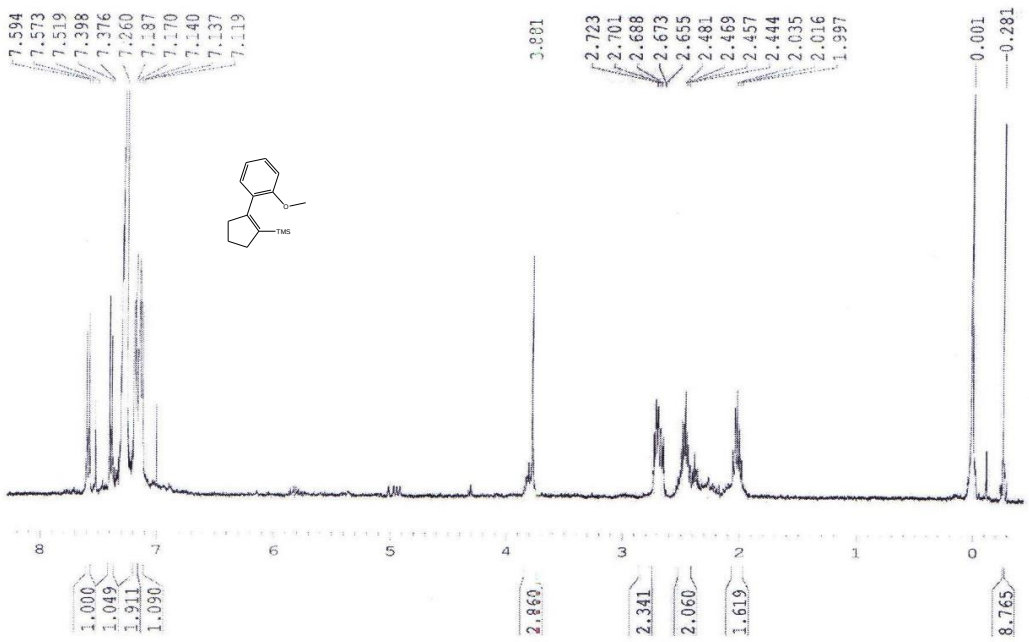

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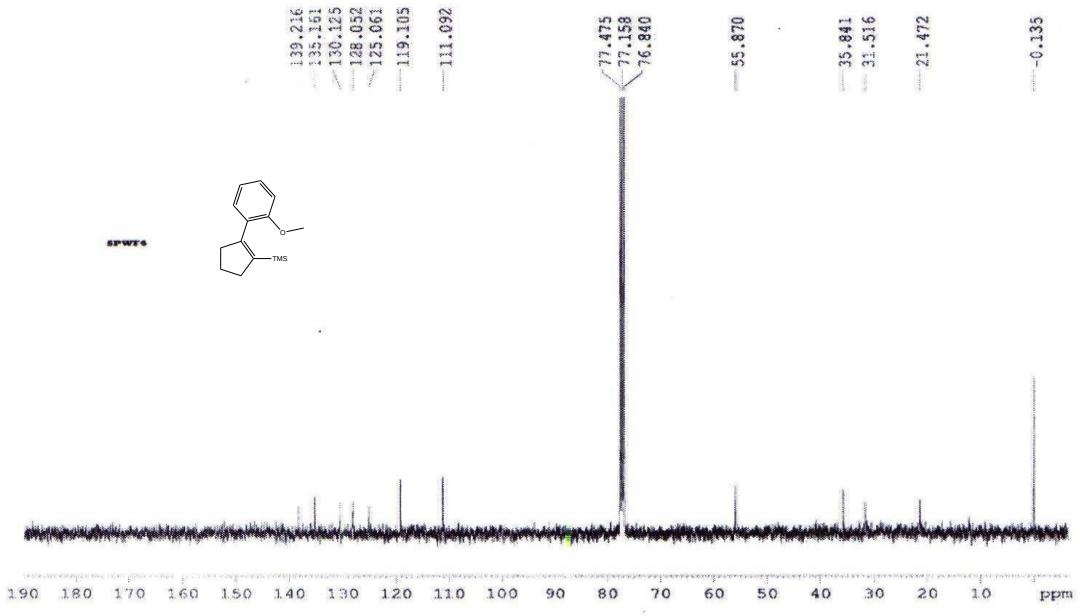

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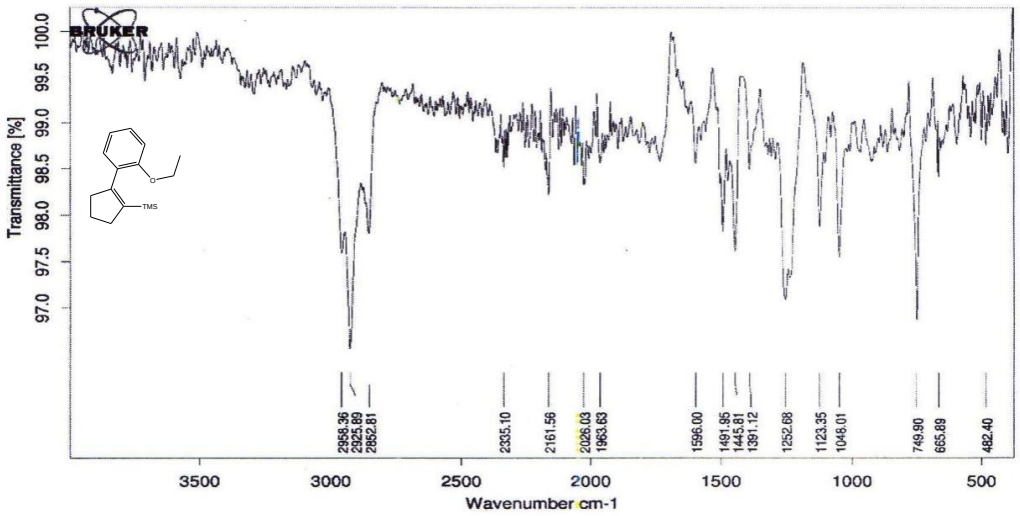

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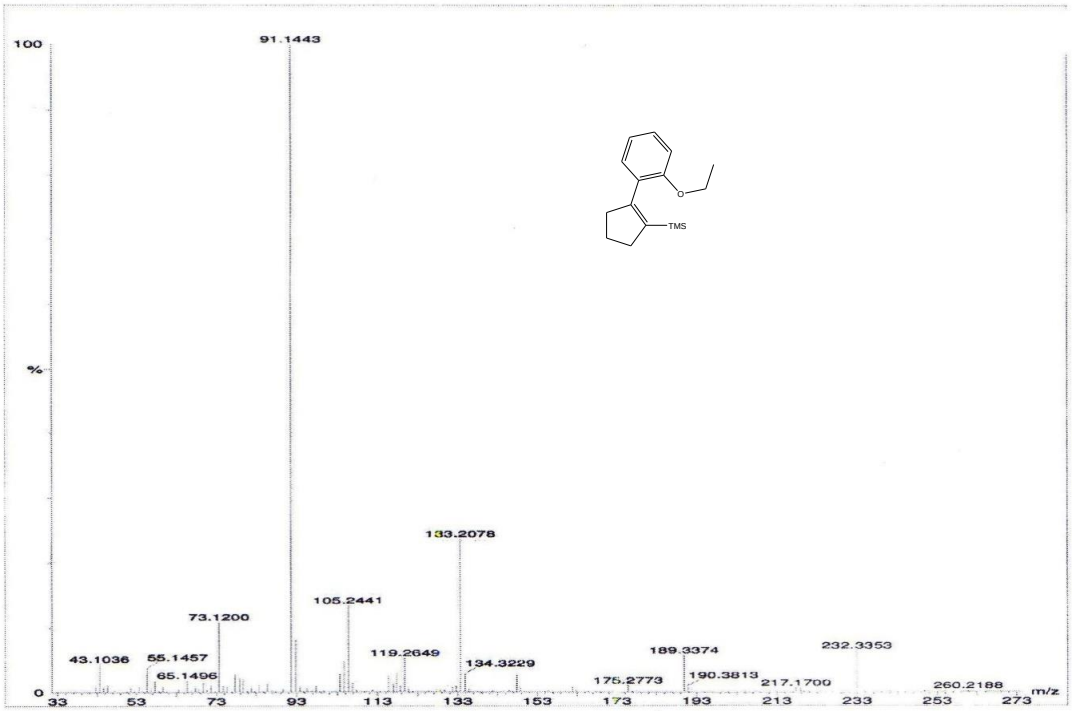

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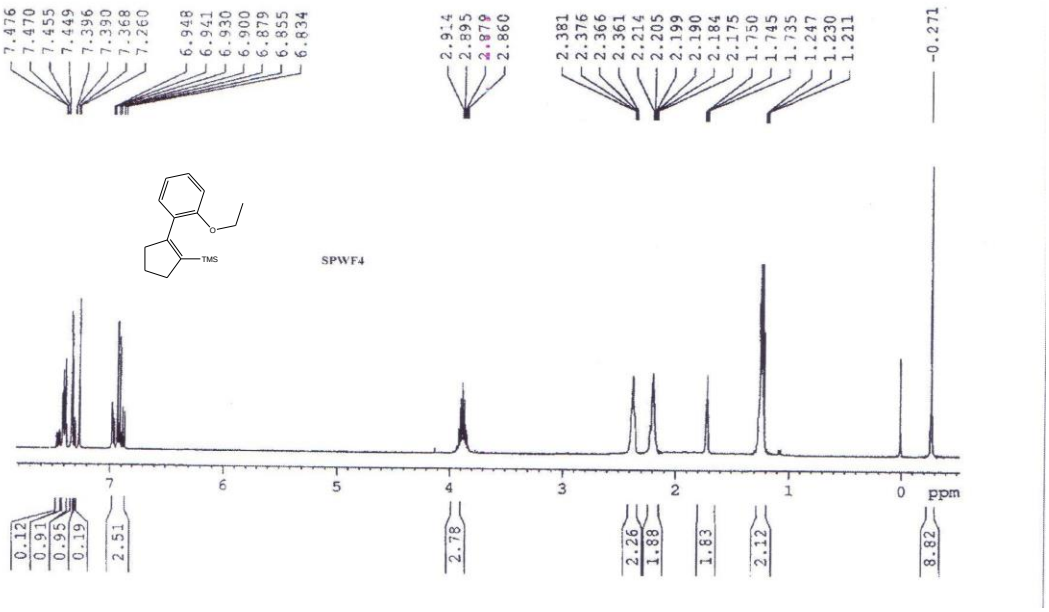

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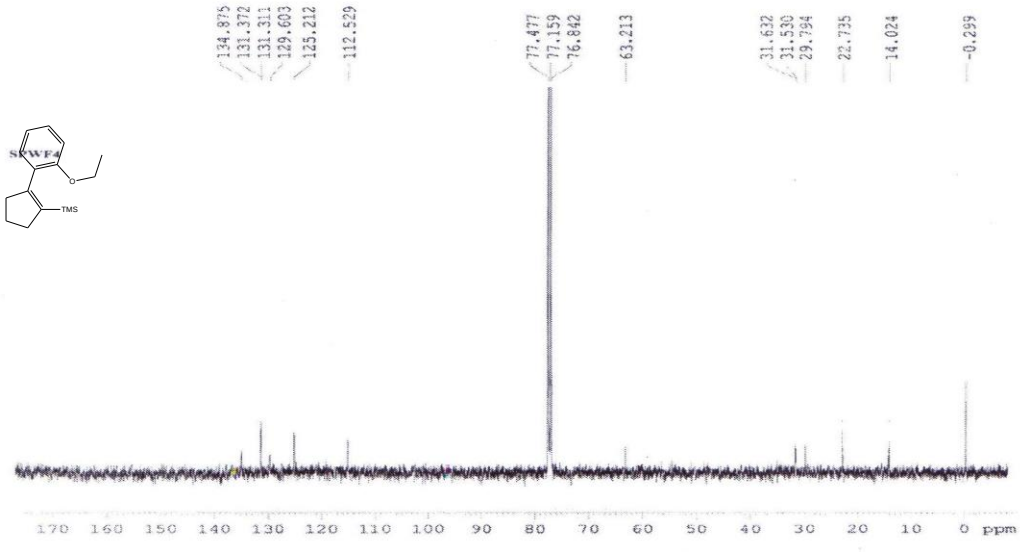

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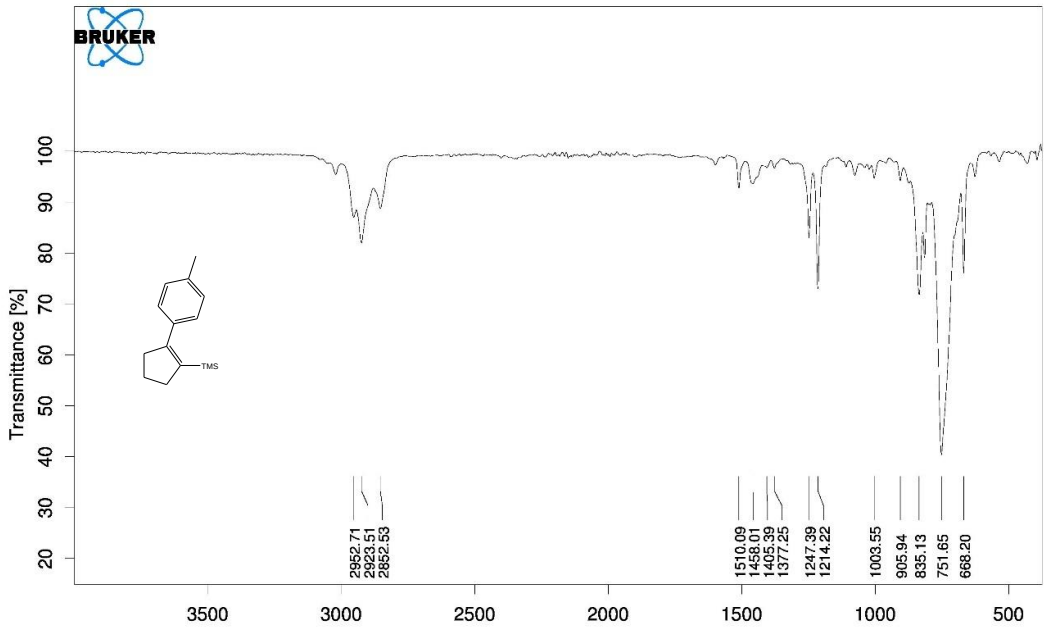

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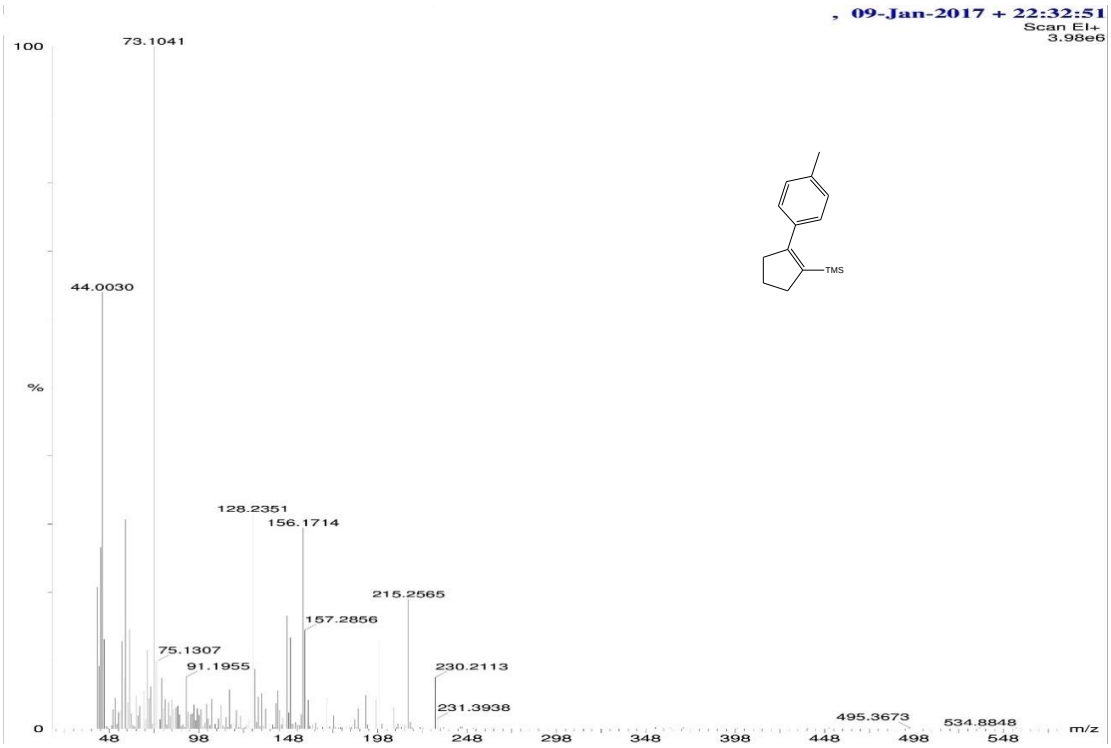

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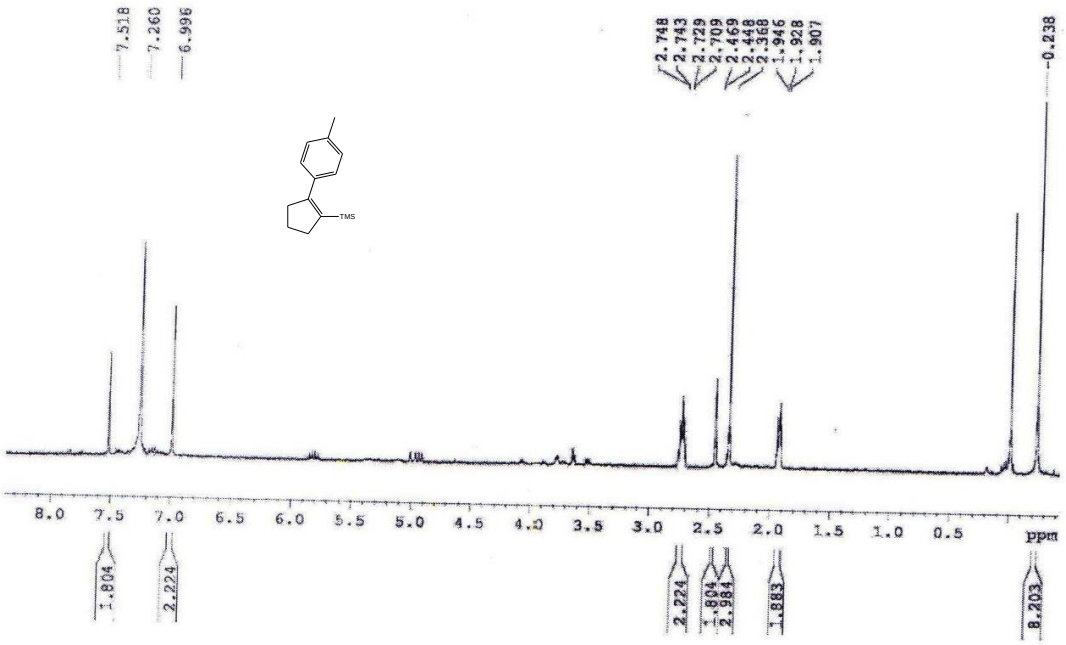

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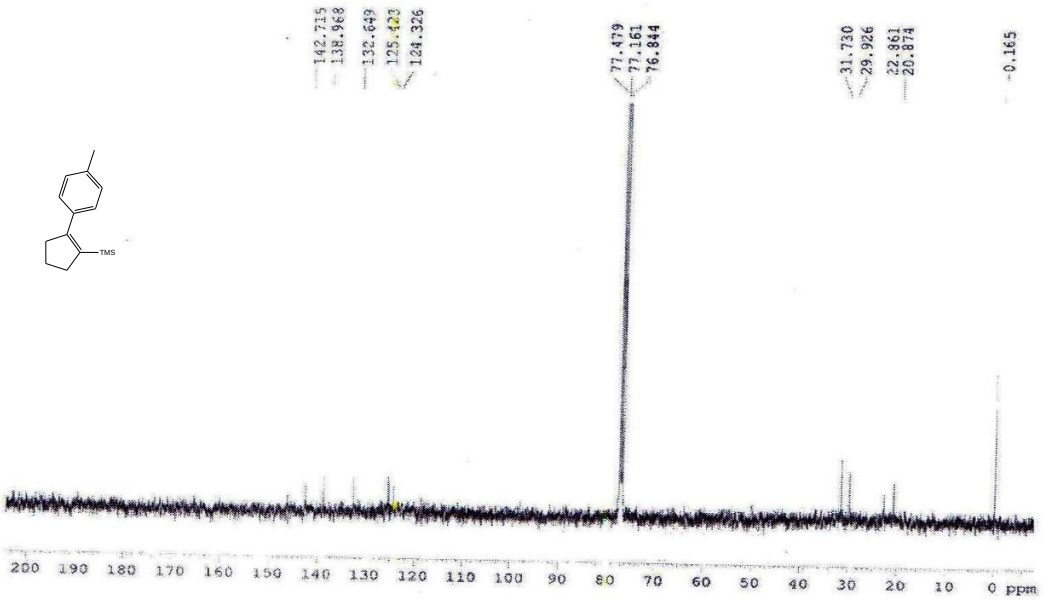

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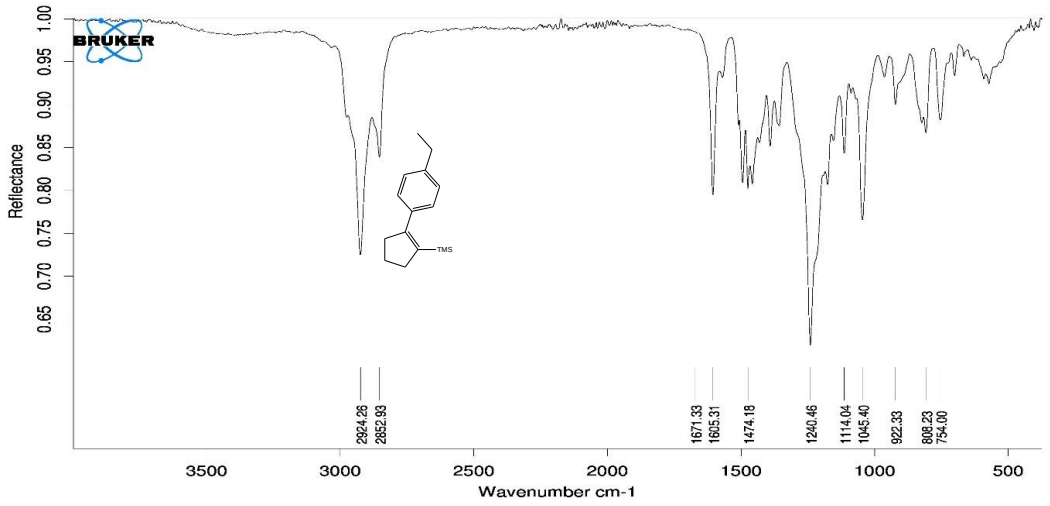

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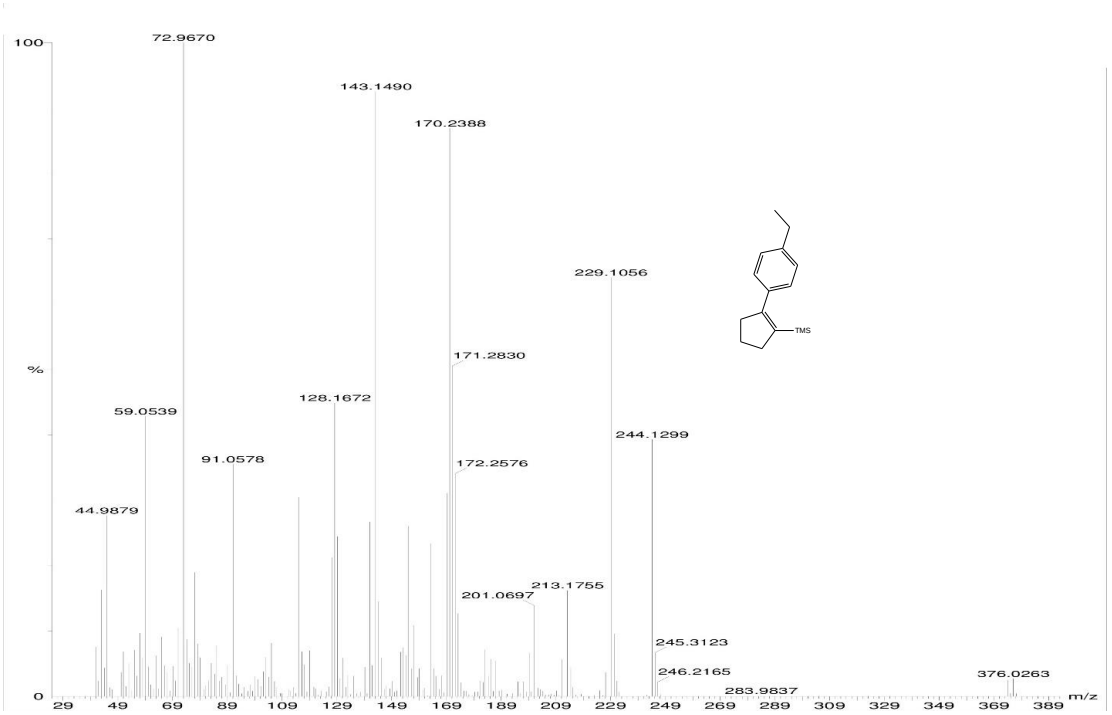

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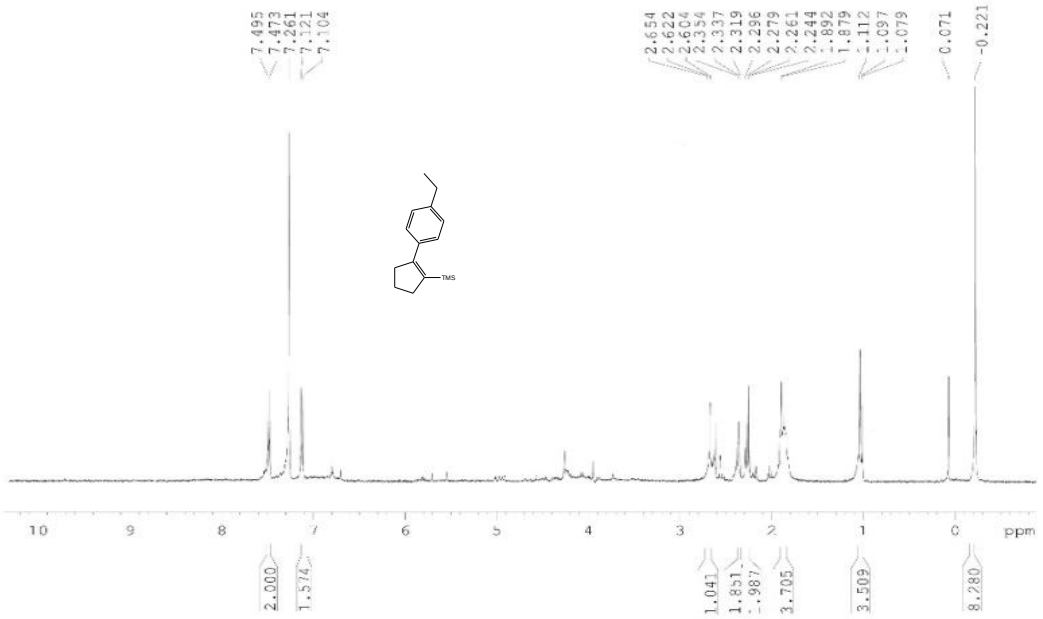

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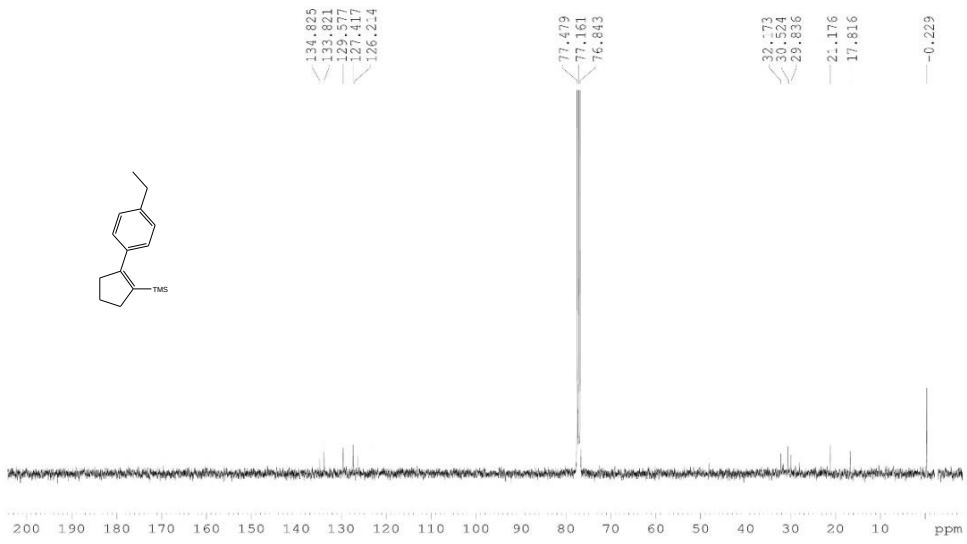

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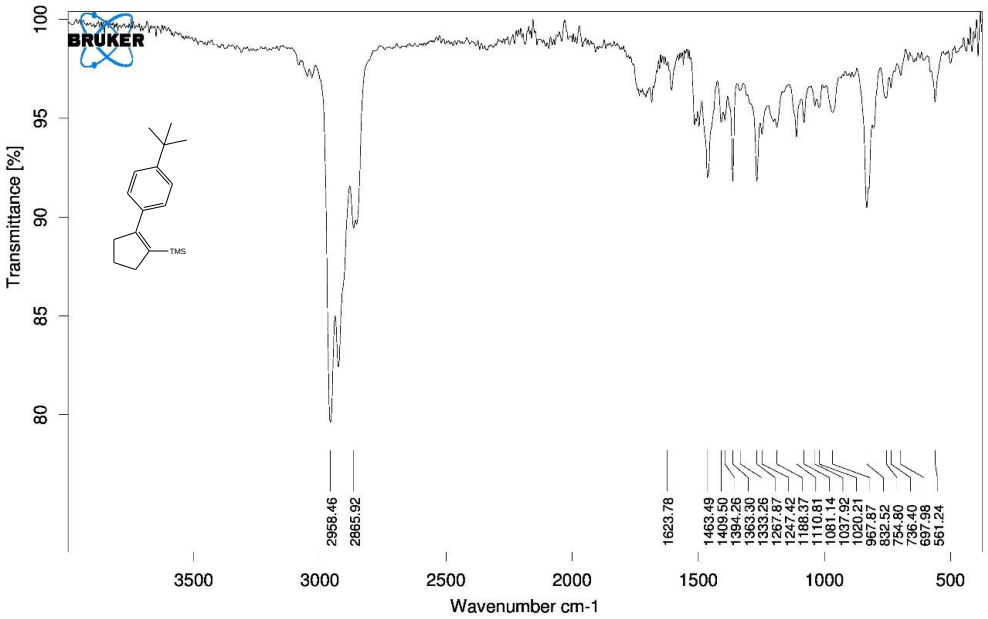

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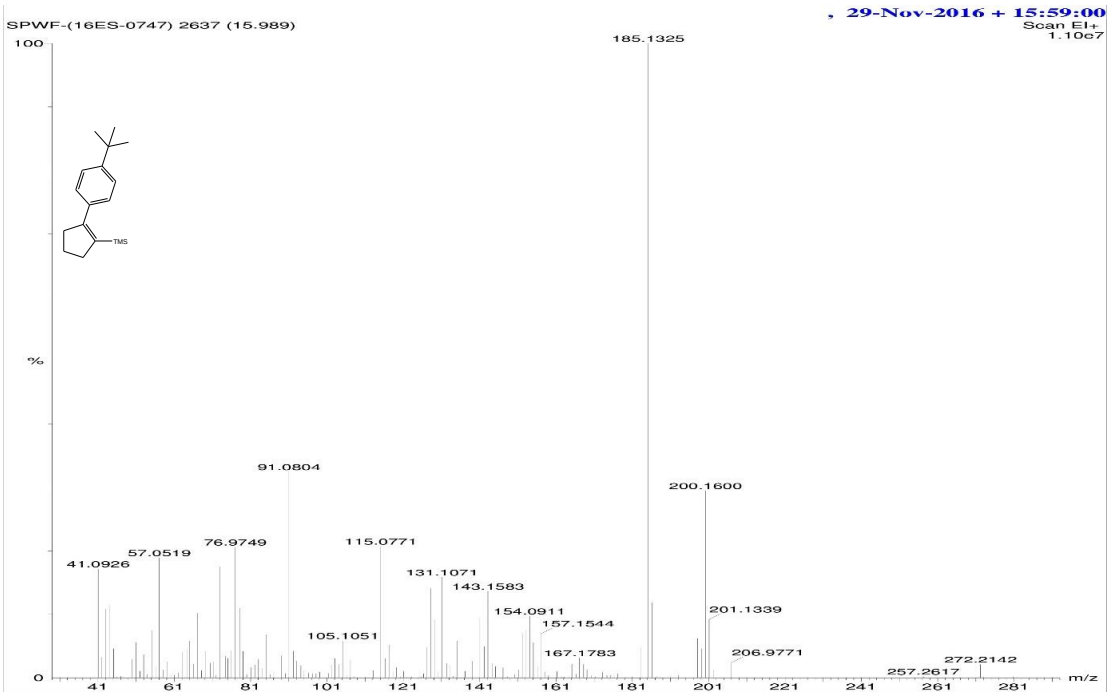

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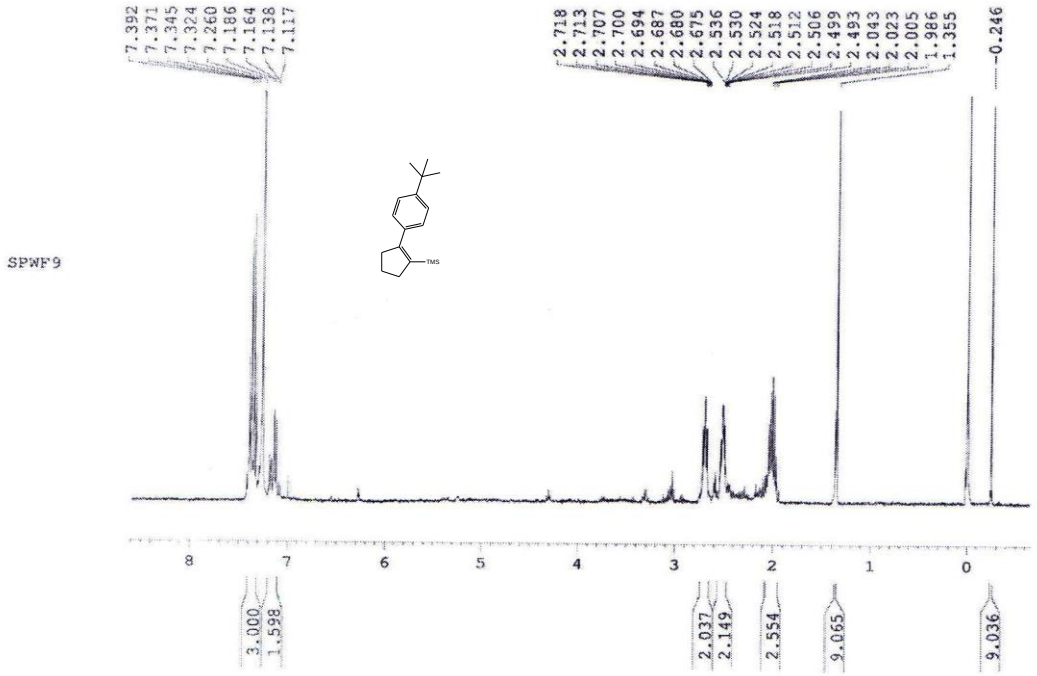

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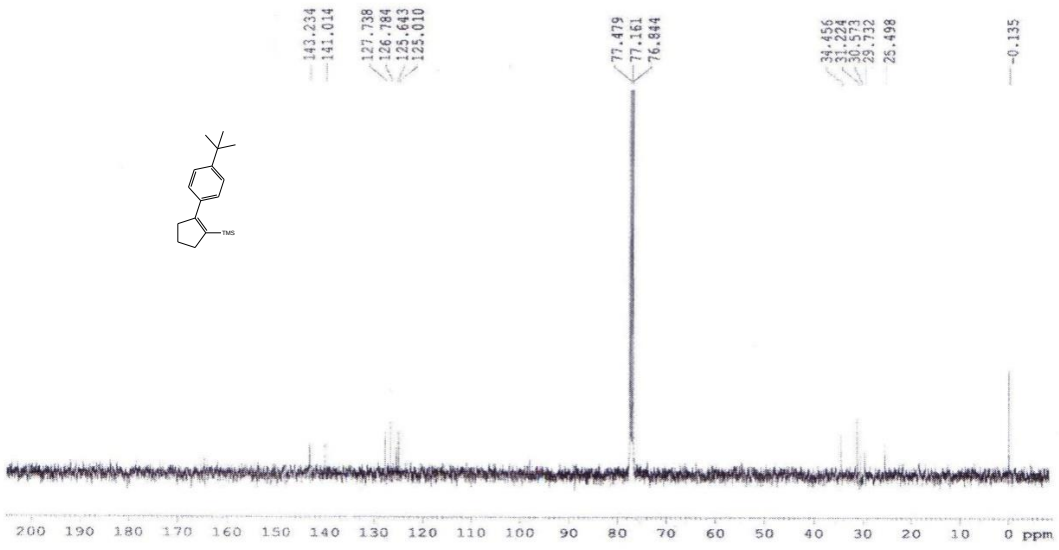

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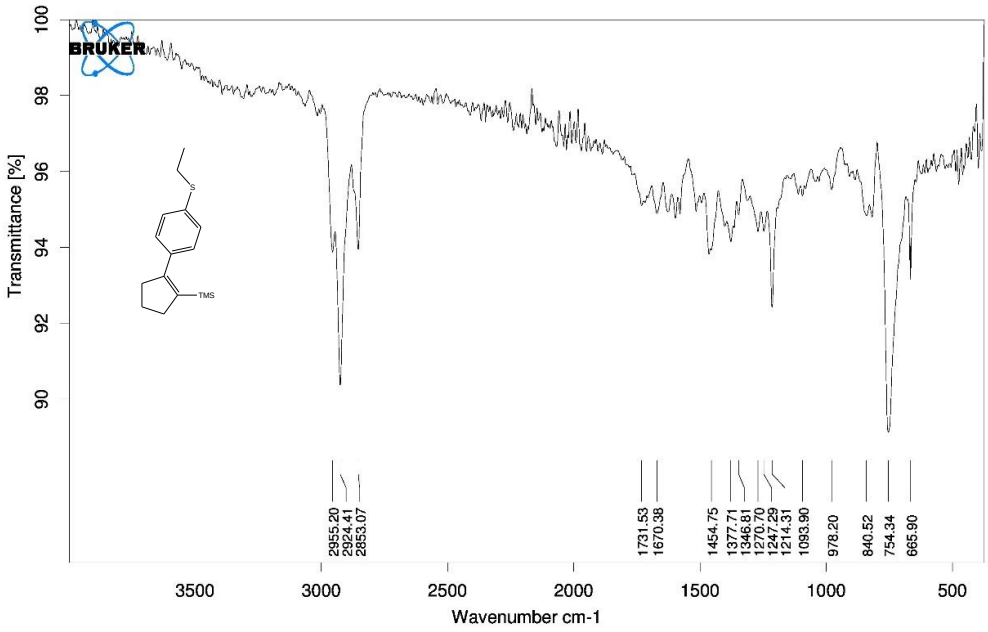

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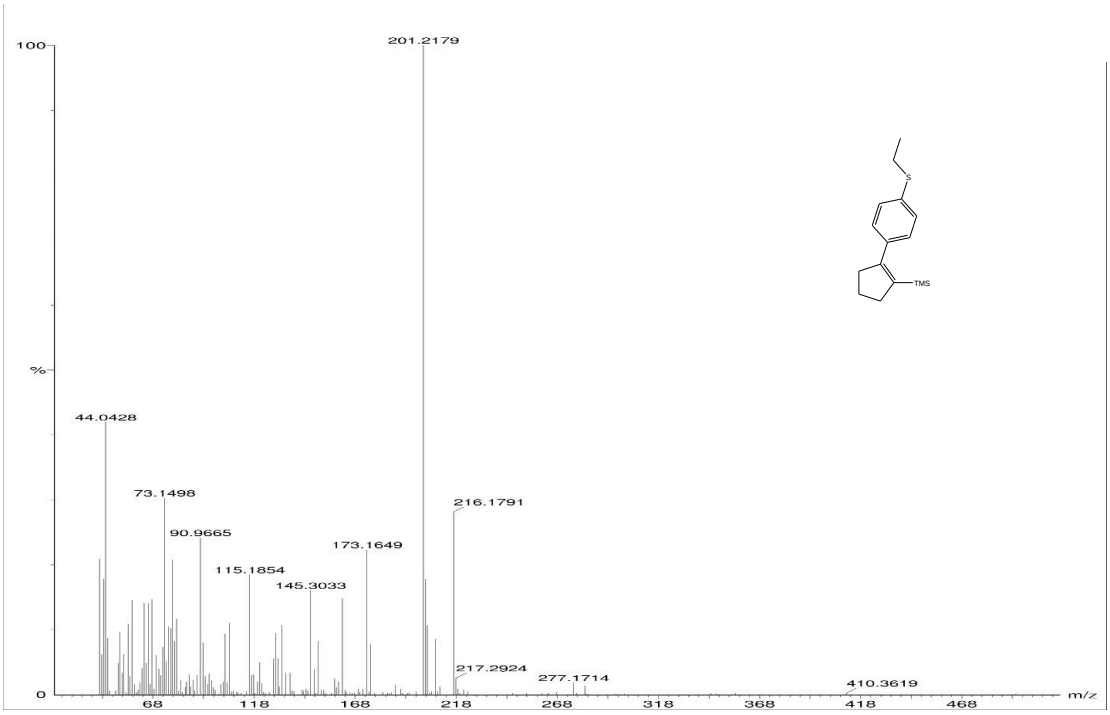

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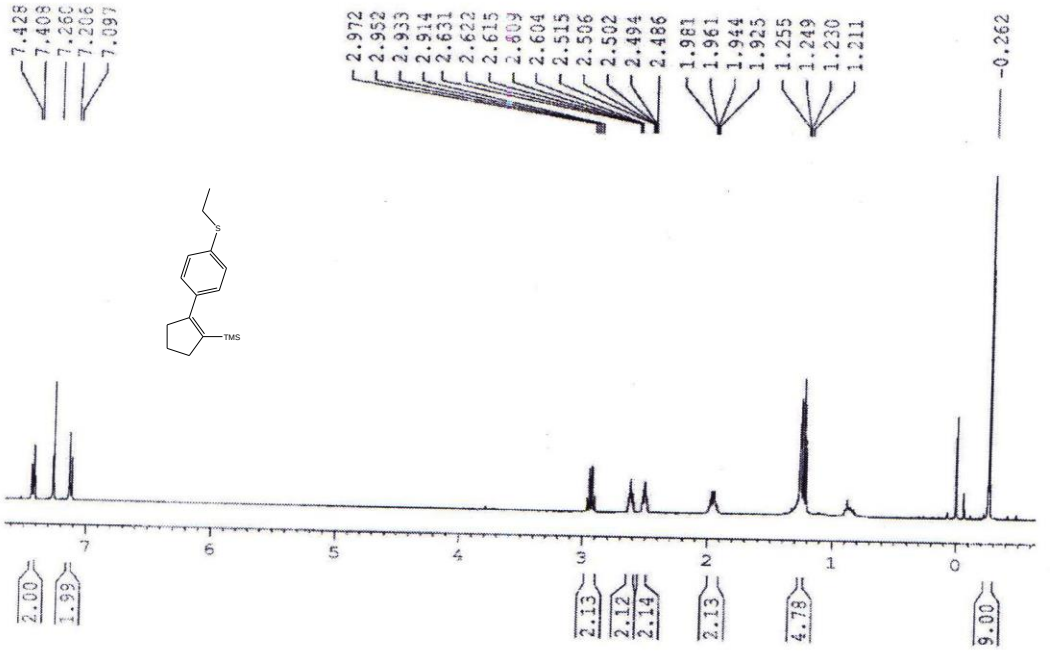

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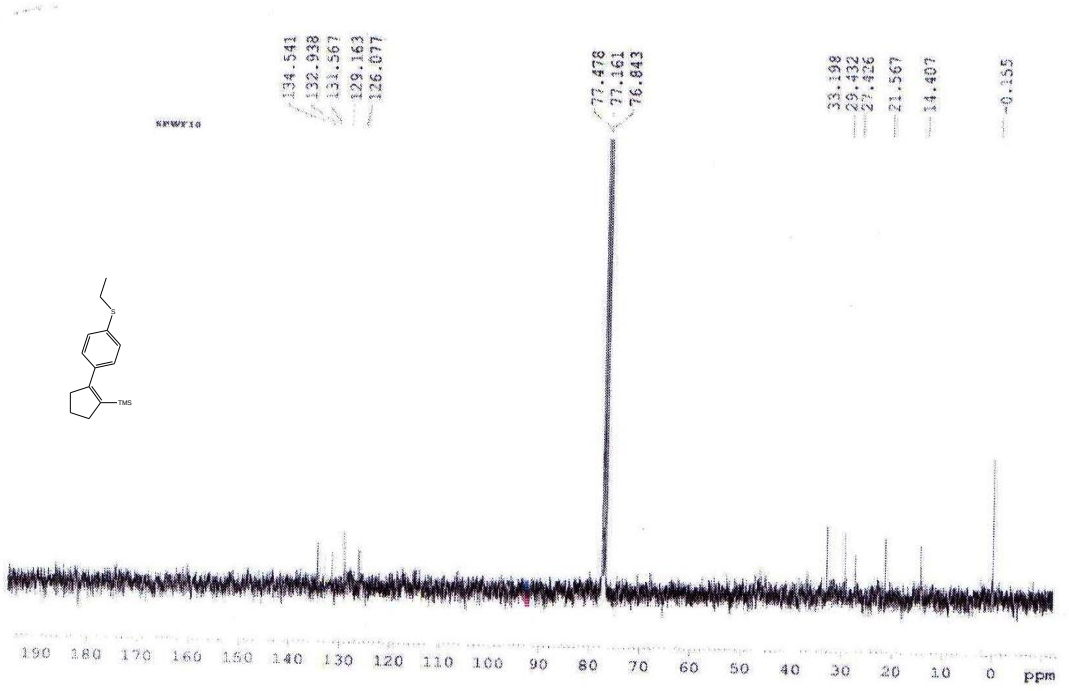

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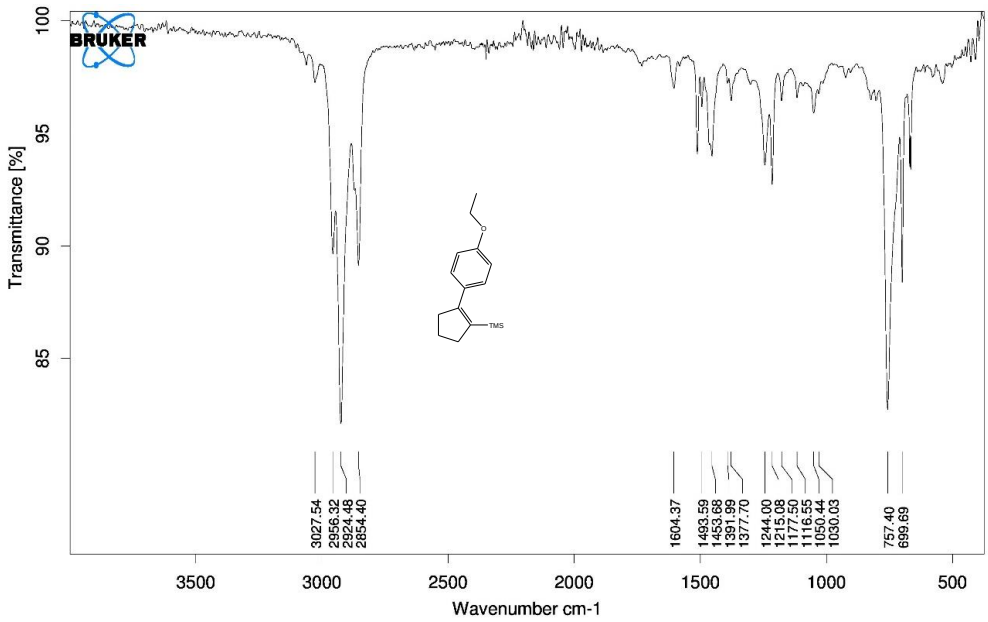

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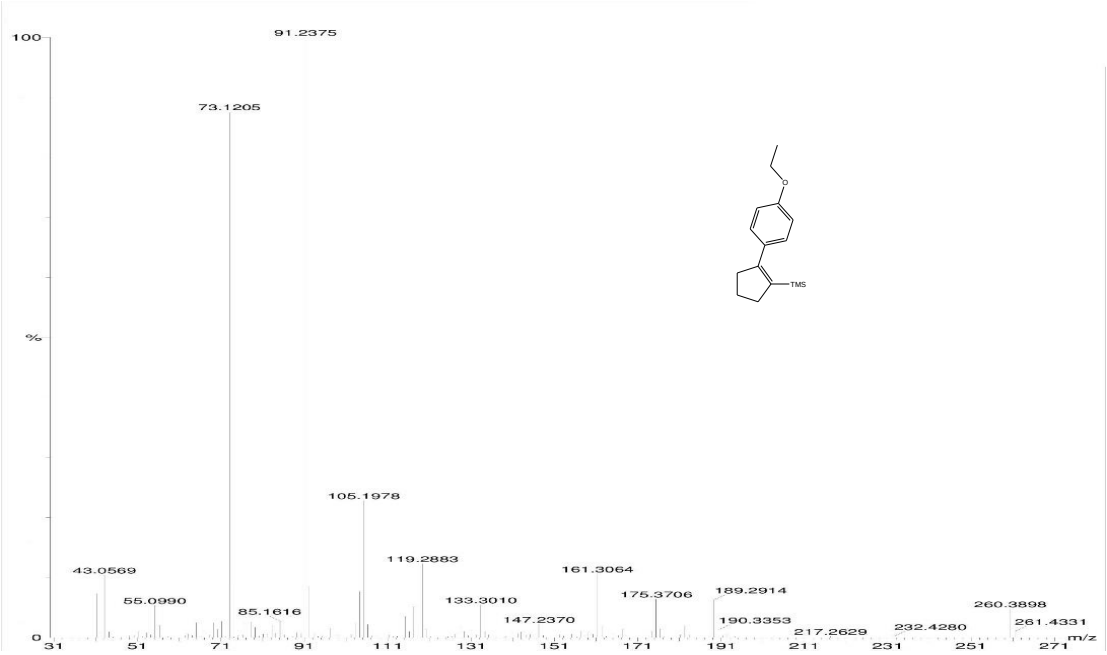

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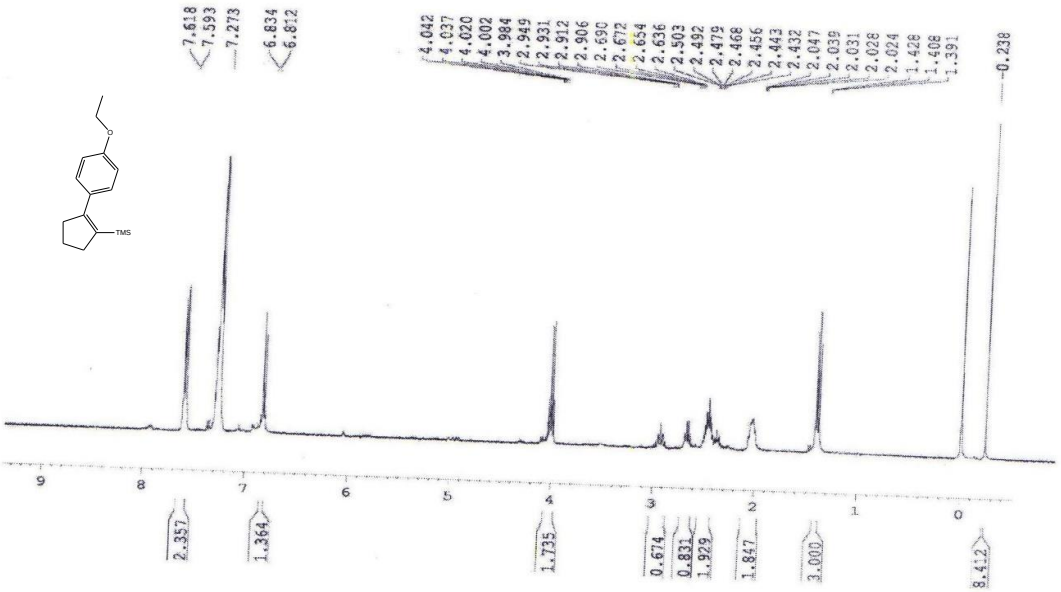

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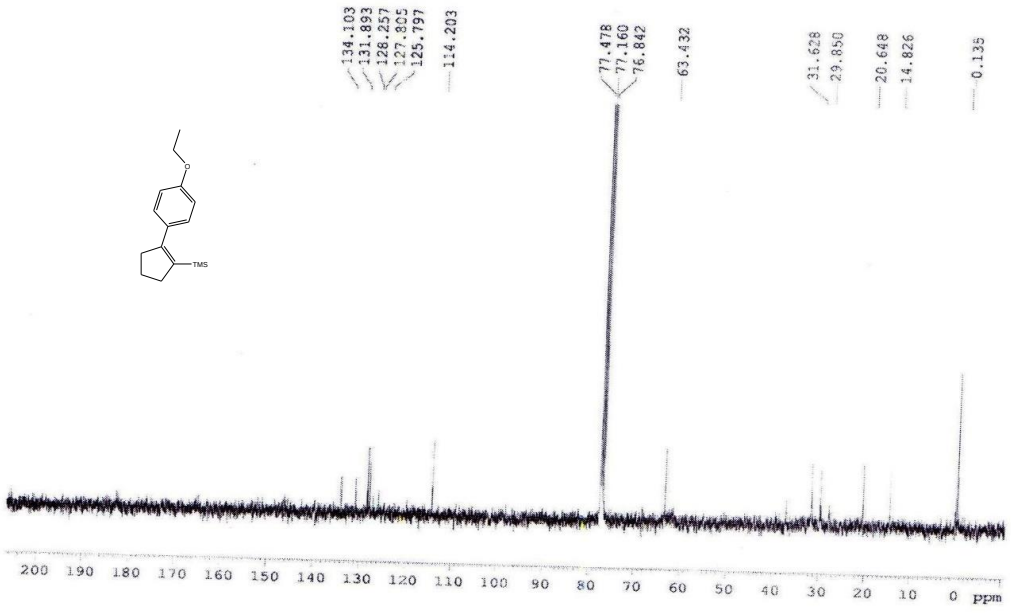
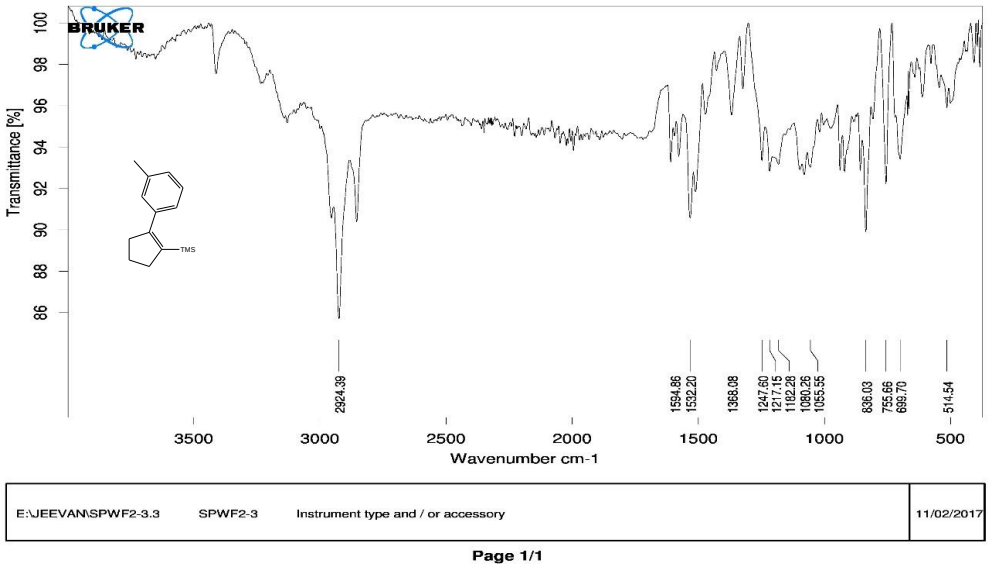


IR of 1-Trimethylsilyl-2-(3′-methylphenyl)cyclopentene 4h:



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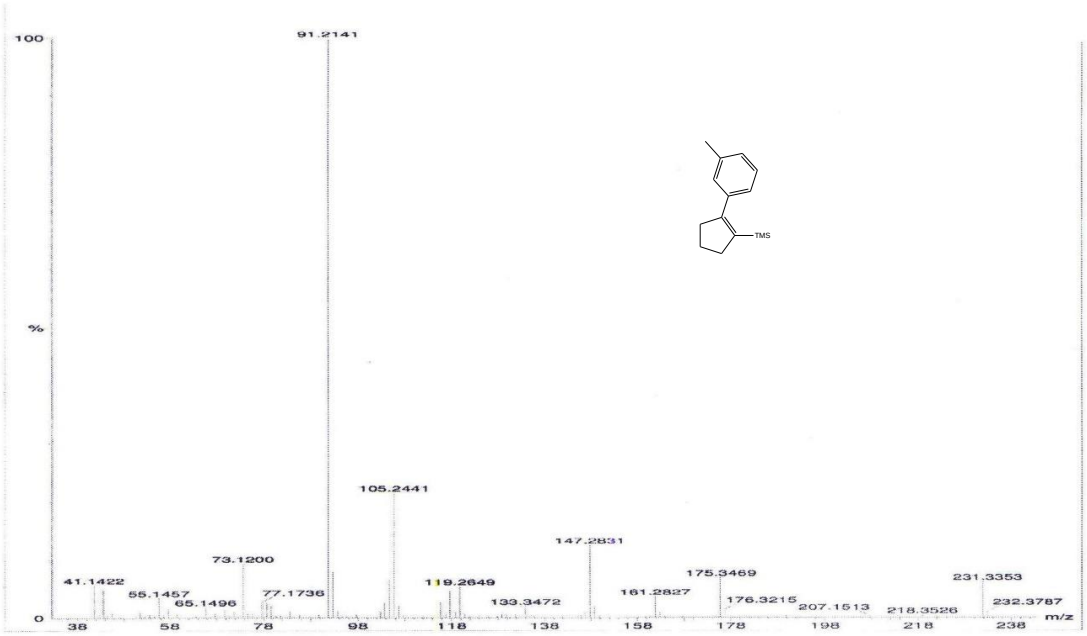

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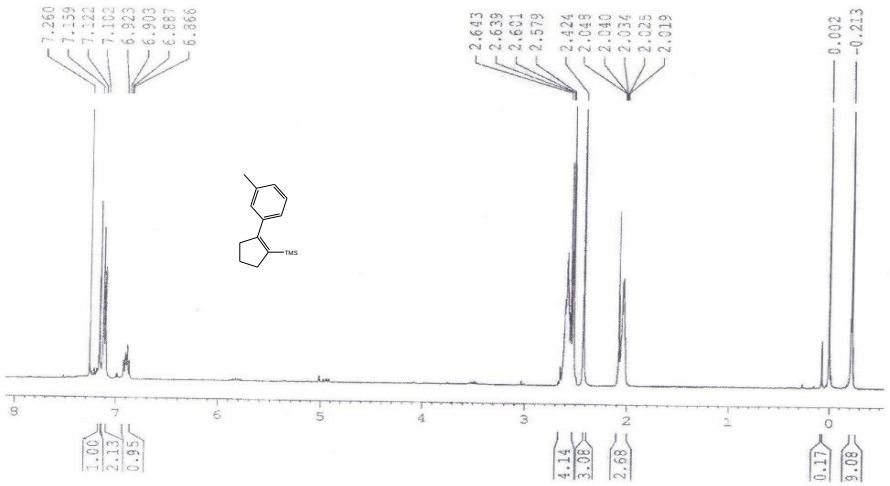

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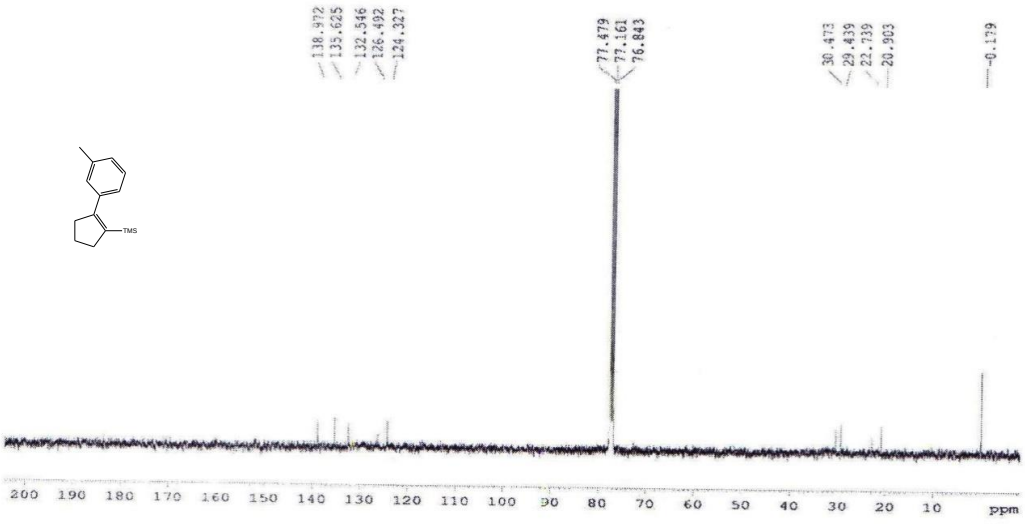

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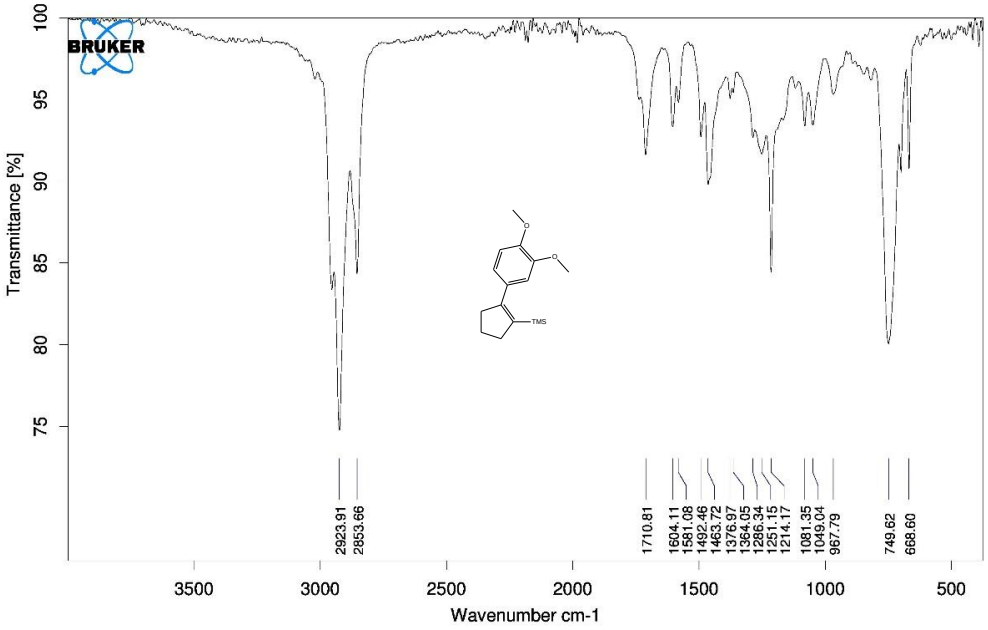

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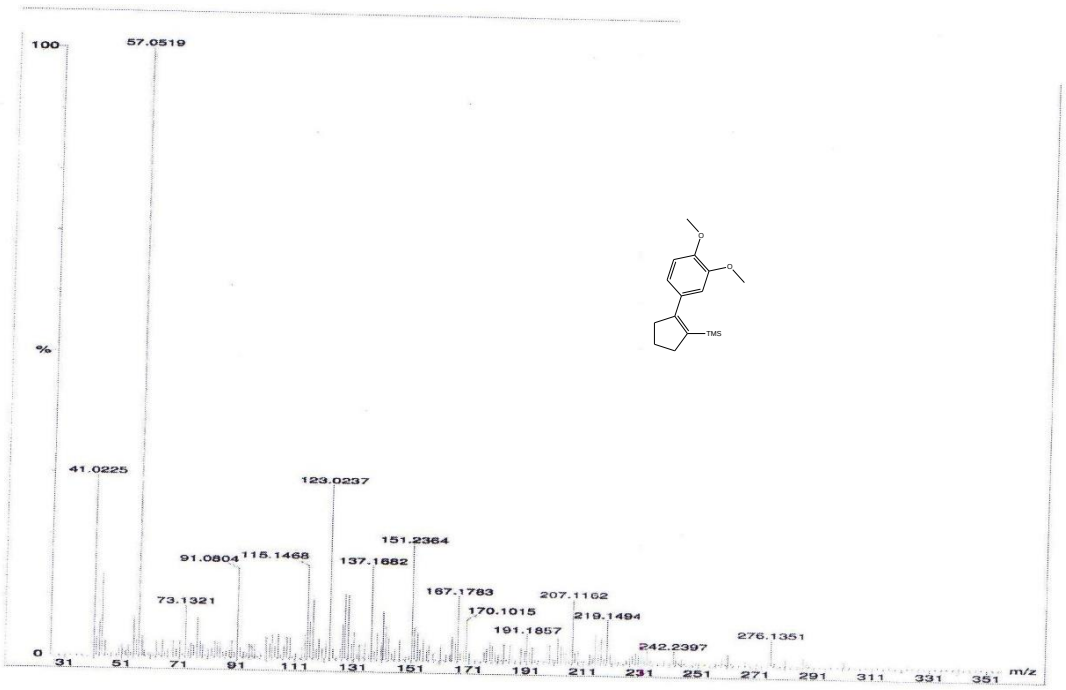

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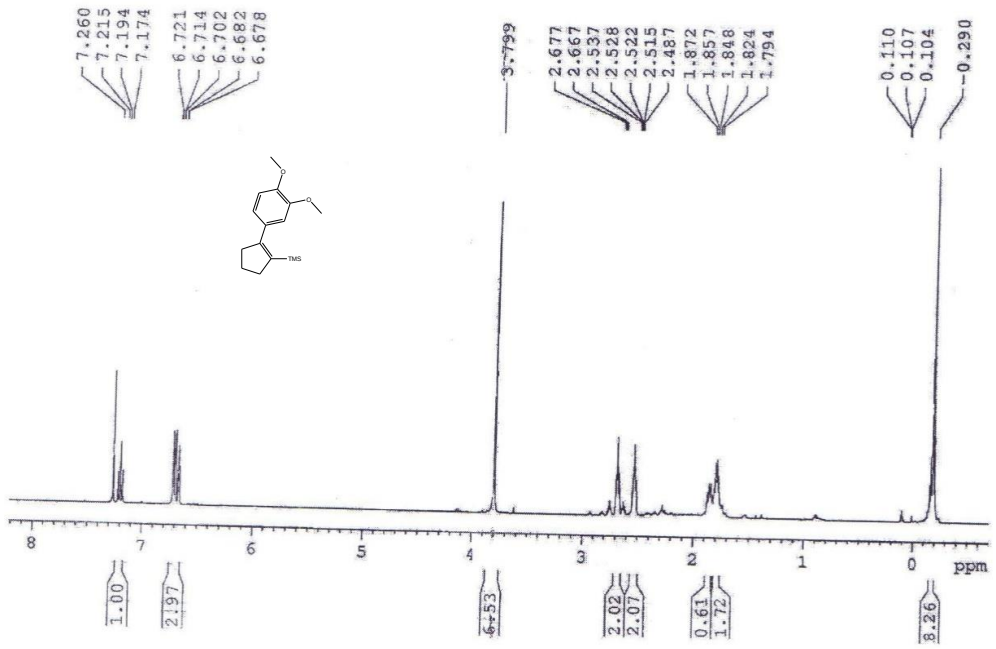

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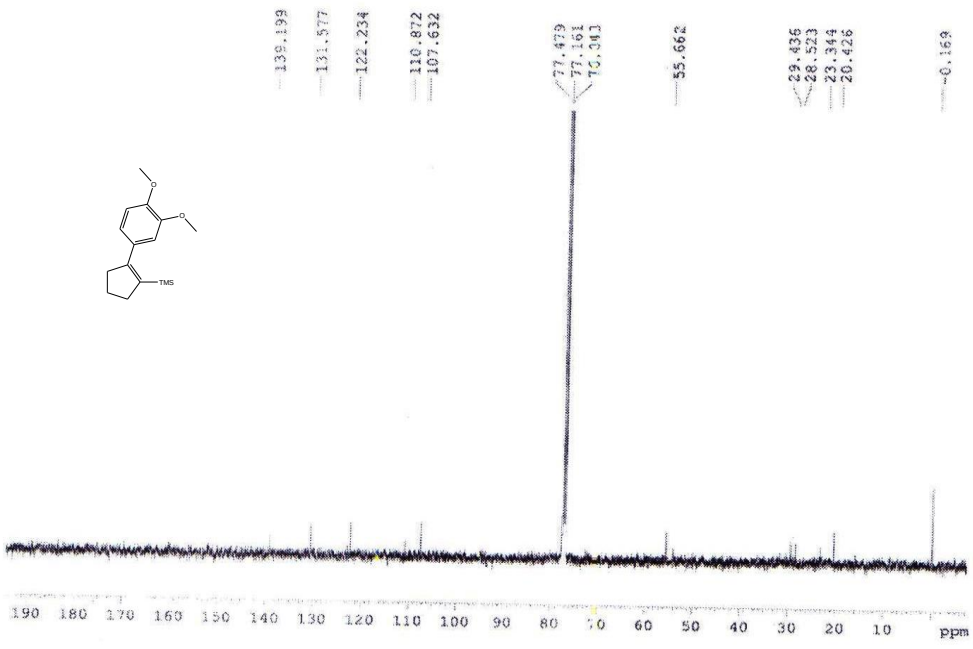

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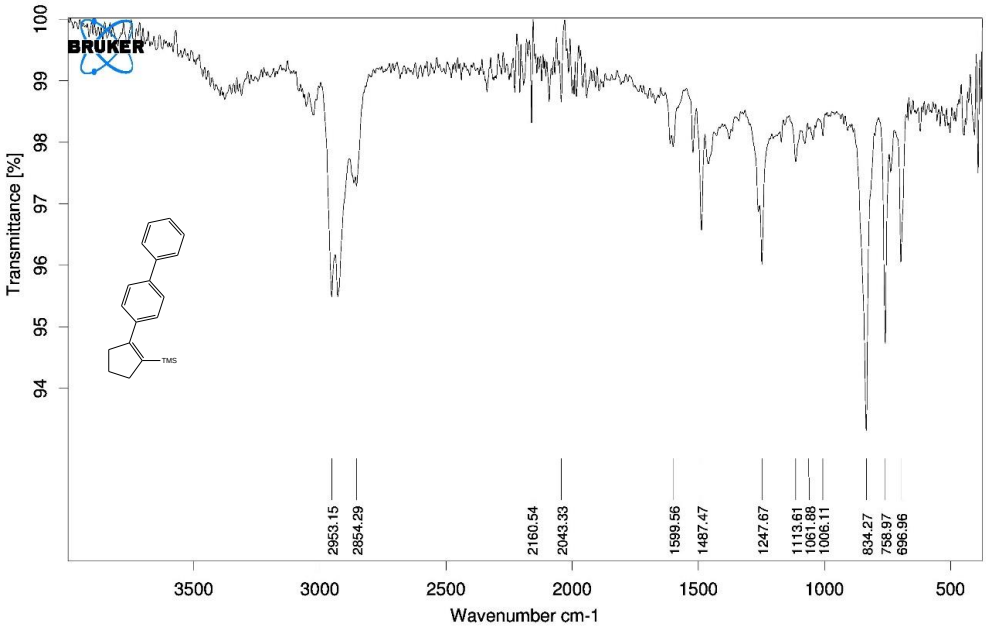

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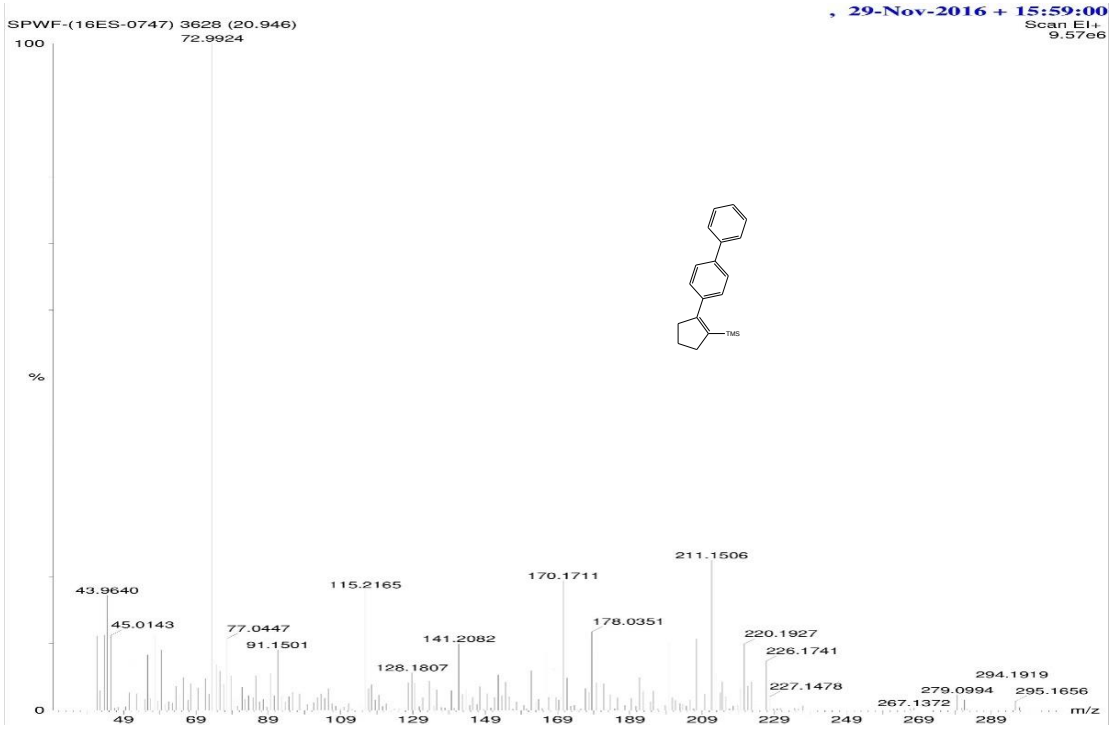

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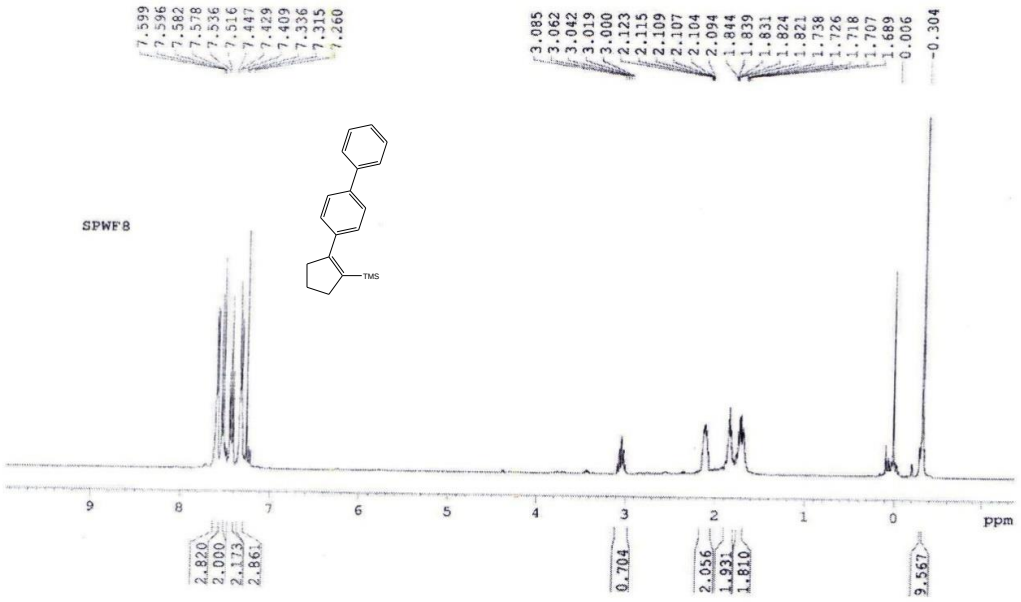

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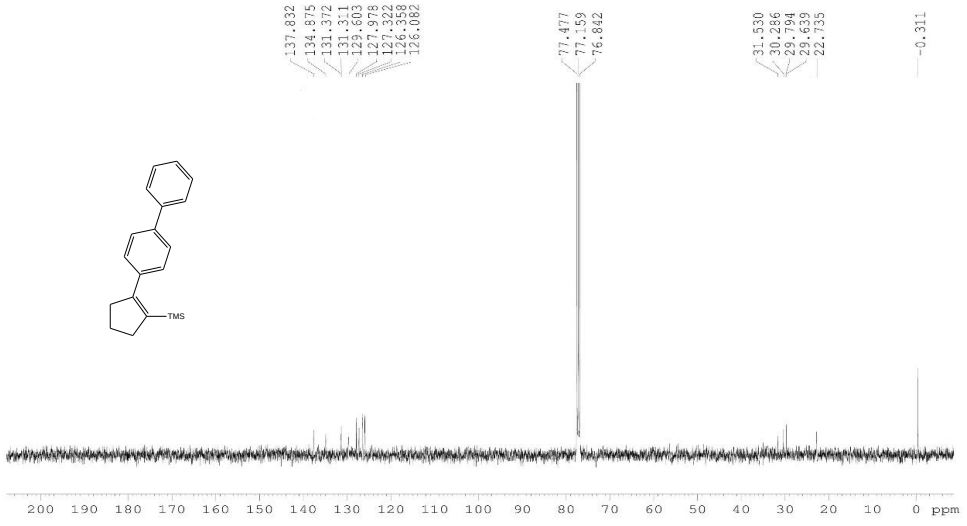
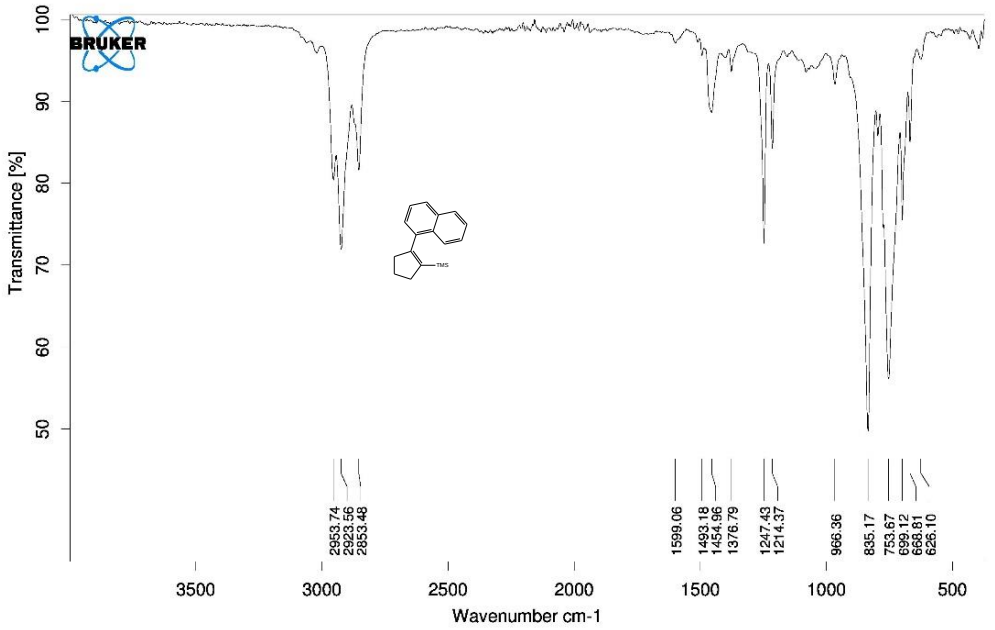
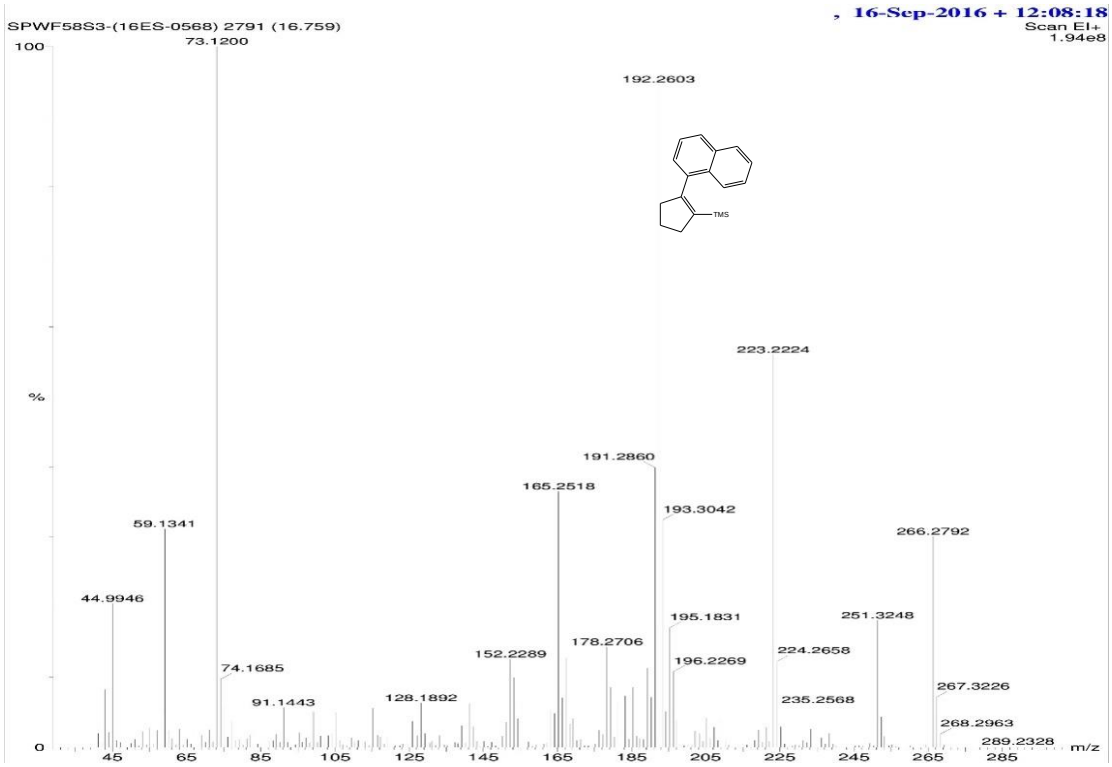


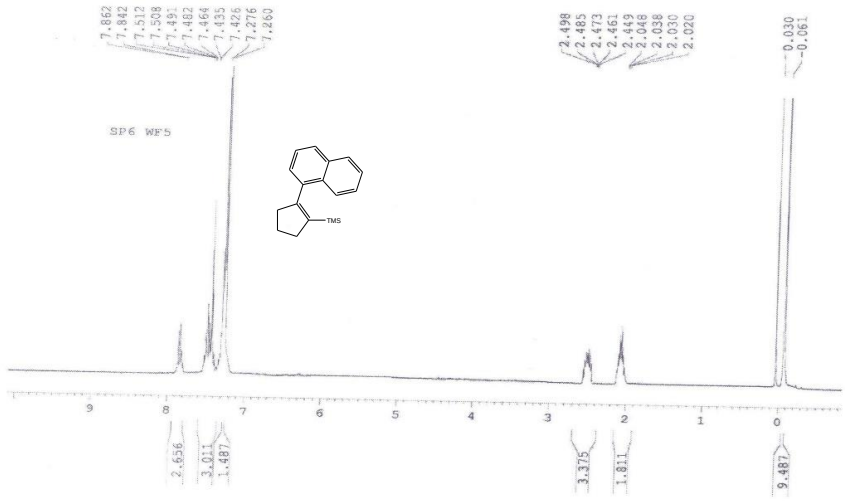
IR of 1-Trimethylsilyl-2-(1-napthyl)cyclopentene 4k:



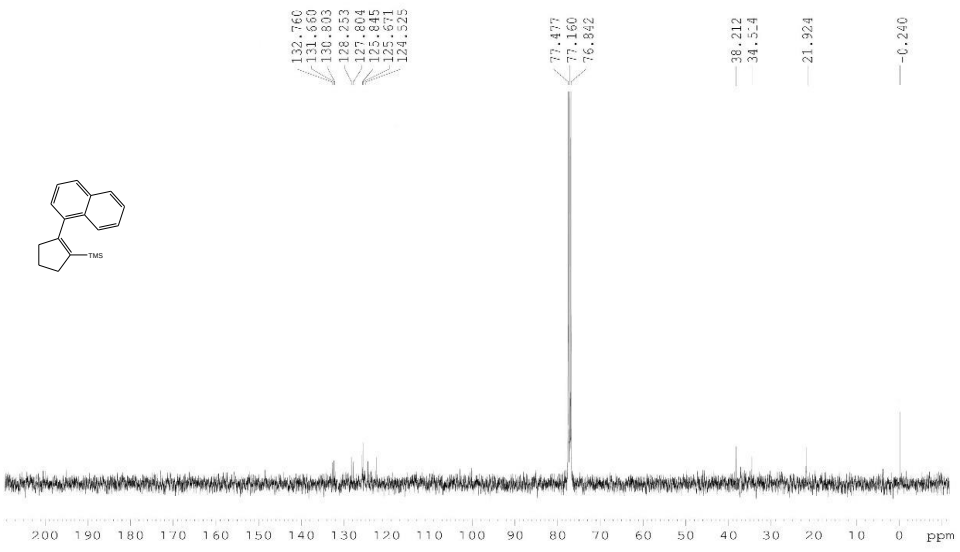
MS of 1-Trimethylsilyl-2-(1-napthyl)cyclopentene 4k:



^{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**).

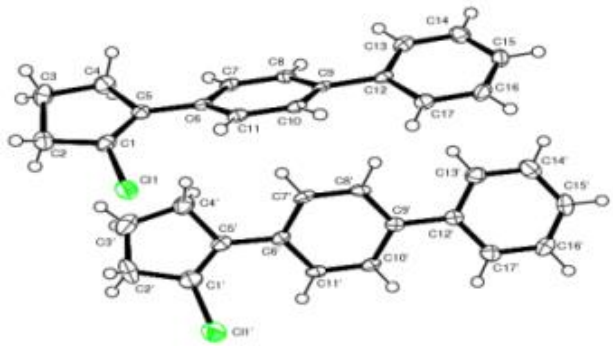
We now report the XRD crystal structures of some representative 1-chloro-2-arylcyclopentens. Very good quality crystals of the 1-chloro-2-aryl compounds **3j**, and **3k** were obtained by slow evaporation technique.

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 ω – ϕ 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

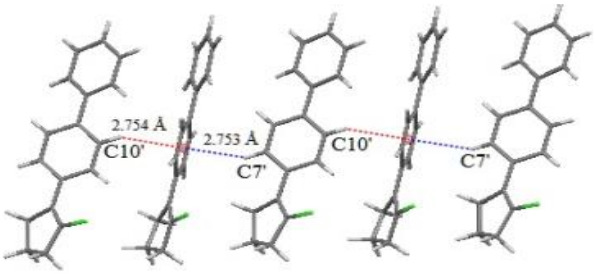


Figure 4: Packing of tl :

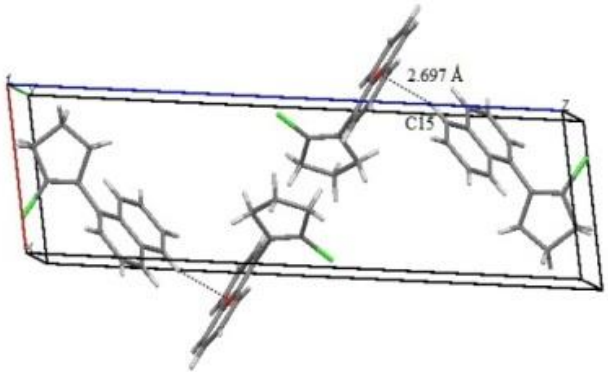
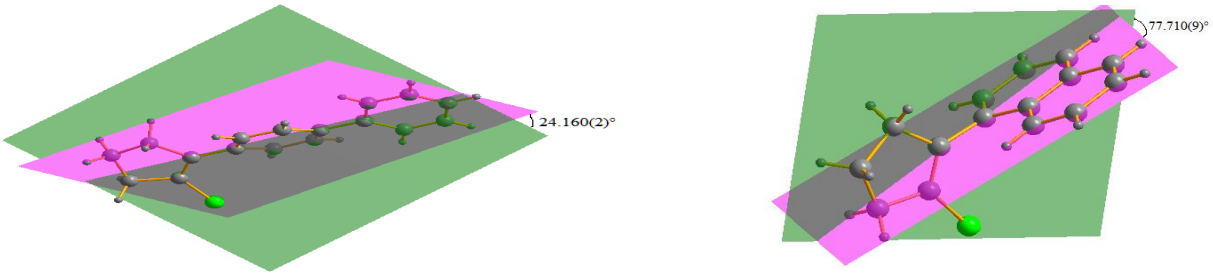


Figure 5: The orientation of the ring structures present in the compounds **3j** and **3k.**



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
	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	*Pī*	*Pī*
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	$-11 \le h \le 11$, $-12 \le k \le 12$, $-16 \le l \le 16$	$-8 \le h \le 8$, $-9 \le k \le 9$, $-27 \le l \le 27$
Reflections collected	15246	13789
Independent reflections	4503 [R(int) = 0.0869]	4008 [R(int) = 0.0496]
Completeness to theta	99.8% θ = 25.00°	99.8 % 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

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