Catalyst Free C-N Bond Formation through Addition-Elimination reaction

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Roll No-CY13M1015

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The Degree of Master of Science

Department of Chemistry

April, 2015

Declaration

I declare that this written submission represents my ideas in my own words, and where others' ideas or words have been included, I have adequately cited and referenced the original sources. I also declare that I have adhered to all principles of academic honesty and integrity and have not misrepresented or fabricated or falsified any idea/data/fact/source in my submission. I understand that any violation of the above will be a cause for disciplinary action by the Institute and can also evoke penal action from the sources that have thus not been properly cited, or from whom proper permission has not been taken when needed.

Topi Ghosh

(Signature)

Topi Ghosh

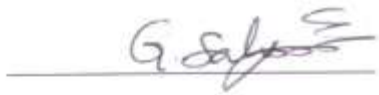
(– Student Name –)

CY13M1015

(Roll No)

Approval Sheet

This thesis entitled – Catalyst free C-N bond formation through Addition-Elimination reaction. – by Topi Ghosh– is approved for the degree of Master of science in chemistry from IIT Hyderabad.



-Name and affiliation-

Examiner

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Examiner

-Name and affiliation-

Adviser

-Name and affiliation-

Co-Adviser

-Name and affiliation-

Chairman

Acknowledgements

I acknowledge my supervisor **Prof. F. A. Khan** for his constant support and encouragement. I also thank to IIT Hyderabad for providing us with the best facilities and equipment's to work. I am grateful to all the faculty members in the department of chemistry at IIT Hyderabad for their guidance. I sincerely thank teaching assistant Raveendra Babu Kaki, Laxmaiah Vasamsetty, and Basavaraj M Budanur, K Sravanthi, Mosim Amin Pathan, K. Sreenivas, Althaf Hussain.Mulla, CH. Narender Reddy, Tapan kumar Jena, shivaji, group members for their constant help and for giving a good working atmosphere. I also thankful to Sami SD for recording of NMR of my compounds. I also thankful to my entire lab mate for their constant help and for giving a good working atmosphere.

Dedicated to

My Parent

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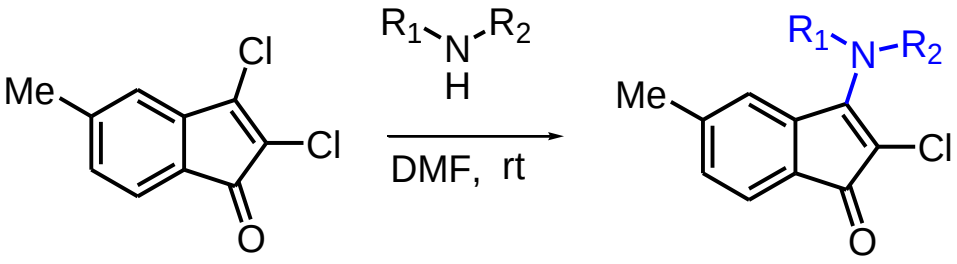
Abstract:

Aliphatic and aromatic amines undergo nucleophilic addition to α-β unsaturated ketone under catalyst free condition to produce β-amino compounds in excellent yield. This method is simple and convenient and works efficiently under mild conditions.

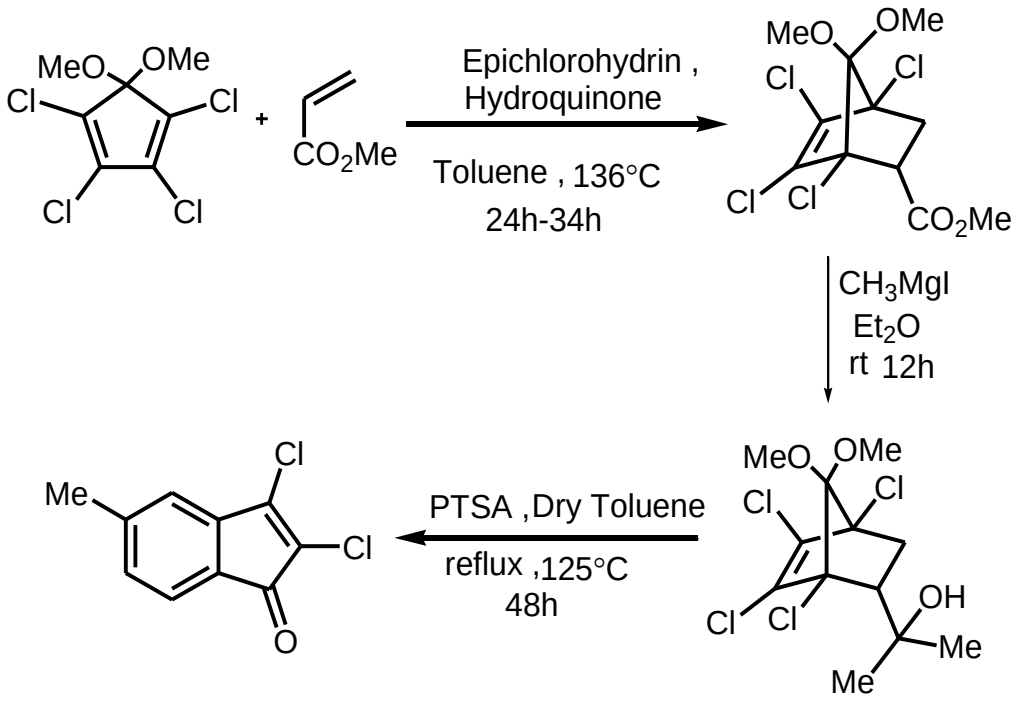


Introduction:

In the recent past, the development of new synthetic routes involve in one pot transformation has been an attractive area of research in organic synthesis. Among these, domino reactions play an important role, because of their ability to build new scaffolds in a single step. The indenone core is an important structural motif in several natural products of biological importance. Taking indinone as a starting material treated with aliphatic or aromatic amines produce 2-chloro-3 amino compounds in excellent yield so efficient reaction catalyst free and non-toxic compound.

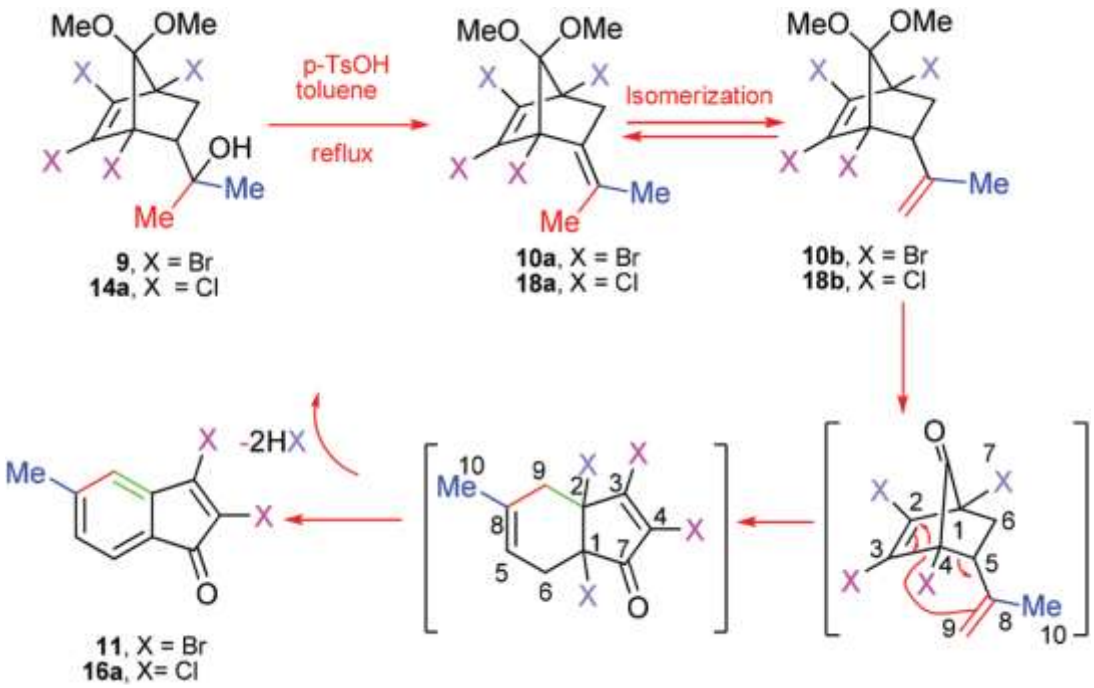
Previous work in our group:

The Diels-Alder adduct of methyl acrylate and 1,2,3,4-tetrachloro-5, 5-dimethoxycyclopentadiene has been utilized to prepare alcohol by treatment with Grignard reagent. Tertiary alcohol having two similar alkyl substituents was treated with PTSA in refluxing toluene to produce starting material indenone (scheme 1).



Scheme 1

Prepared indenones as single regioisomer, the regioselectivity leading to a single indenone with excellent yield (91%) conformed pure compound by ^{1}H NMR, ^{13}C, DEPT, HRMS spectra.

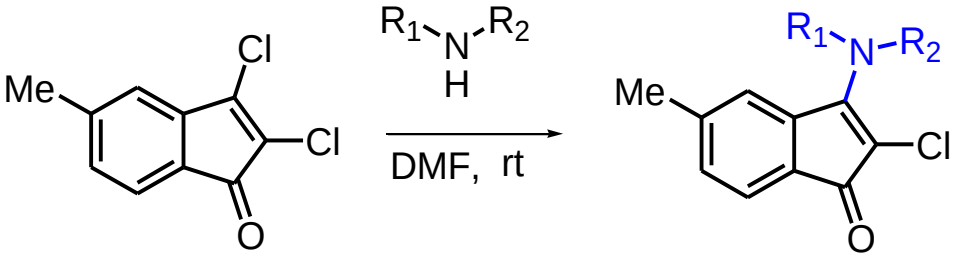


The high regioselectivity leading to a single indenone may be attributed to the preferential formation of more substituted alkenes upon dehydration and isomerization of the initially formed exocyclic alkenes. A plausible mechanism for formation of indenones from alcohols.

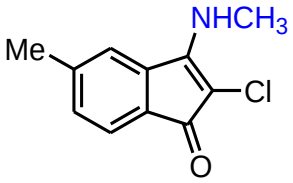
Result and Discussion:

On the basis of Addition–Elimination reaction taking indenone as a starting material produce β-amino compounds. Aromatic amines contain electron donating group (EDG) react with indenone give best yield of product but amines containing electron withdrawing group (EWG) reaction not proceed at all. Two equivalent of amine is used for this reaction. One equivalent of amine is used to activate the indenone and one equivalent of amine is used to react with indenone moiety produce β-amino compound. In this reaction lone pair of nitrogen should be available to form adduct. Among all these reaction o-toluidine gives best yield product, methyl group increases the electron density to nitrogen atom increases the rate of reactions. EWG group containing amines such as 2,6-dibromoaniline, 2,4-dichloroaniline, 2,4-dinitroaniline, 2,4-dinitro-5-fluoroaniline did not give product.

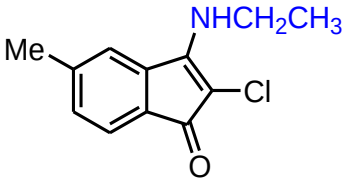
General reaction condition is given below:



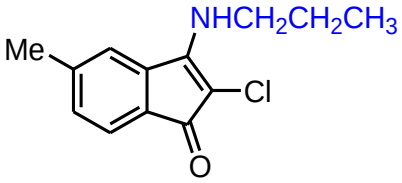
Scope of reaction



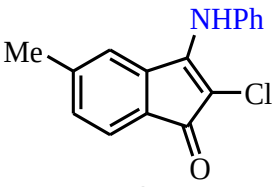
4h, 70%



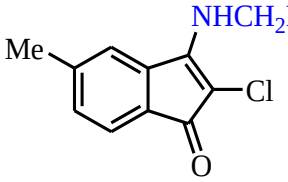
5h, 78%



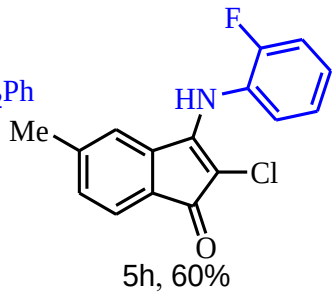
3.5 h, 75%



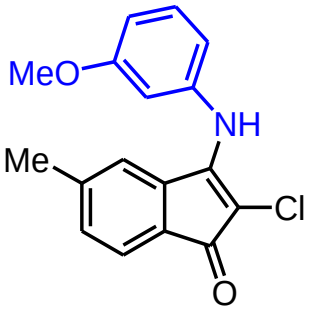
5h, 60%



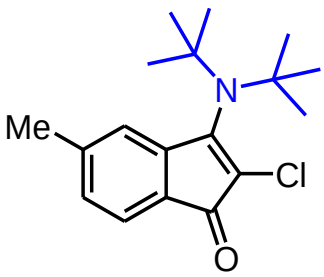
4h, 63%



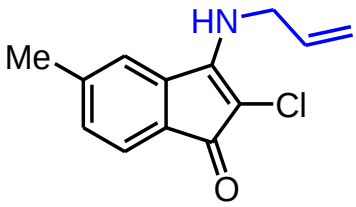
5h, 60%



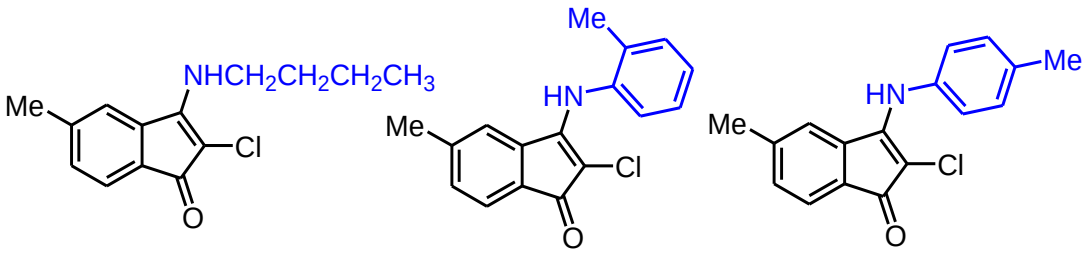
3h, 72%



4h, 68%

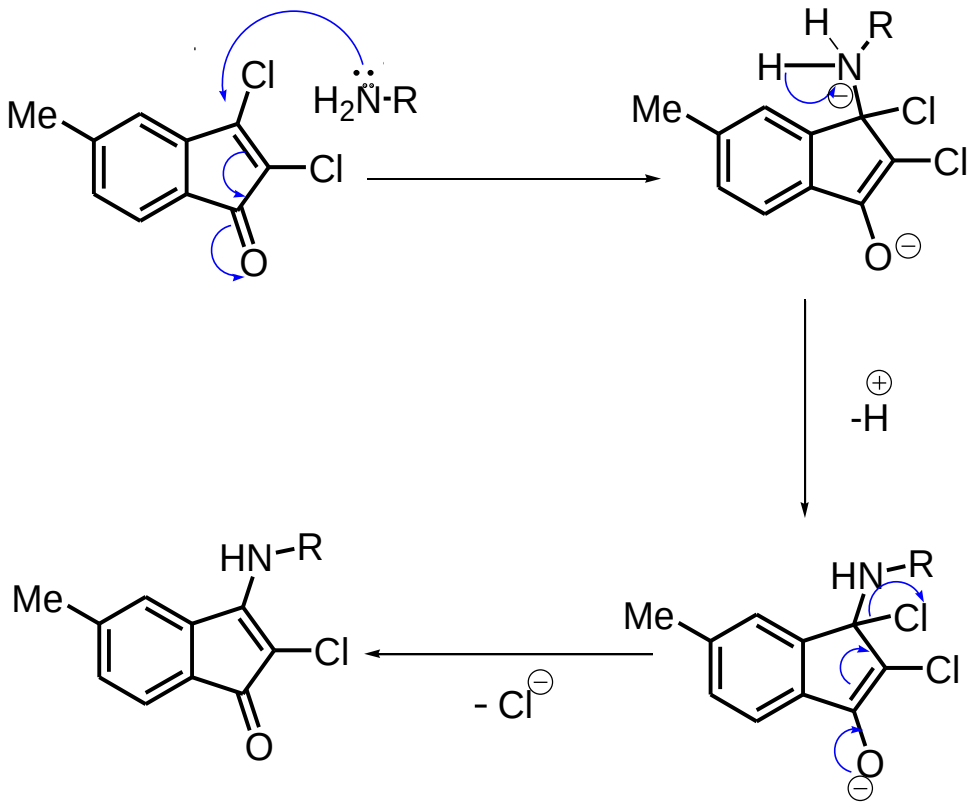


4h, 69%



Recently catalyst free reaction is more efficient. This reaction is two component reactions in DMF solvent .The plausible mechanism of this reaction is given below

Plausible reaction mechanism:



R=Alkyl or Aryl group

Conclusion:

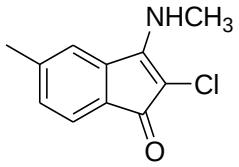
This methodology allows a facile and efficient synthesis of 3-amino-2-chloro-indenone derivatives through addition elimination reaction. In presence of DMF solvent starting material indenone react with aliphatic or aromatic ammine to produce corresponding 3-amino indenones. This reaction catalyst free reaction and efficient, no reflux condition required, good yields are obtained and required less time. EWG group containing aromatic amines does not precede the reaction. EDG affect group containing amines proceeds smoothly.

Experimental section:

In this section the preparation of all the compounds that have been made in the course of synthesis of β-amino compound has written. For experiment all the starting material and the reagents are purchased from standard commercial source or were prepared in laboratory. All the glass wares were cleaned with soap water followed by acetone and the dried in hot air oven at 140^{o}C for 2 hours. Solvents were dried and distilled using standard procedure. For evaporation of solvent Buchi rotary vapor machine was used. NMR data was collected using Bruker 400 MHZ machine; IR data was collected using Bruker machine.

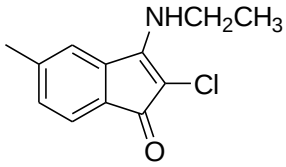
General procedure for the preparation of β-amino compound:

One equivalent of indenone was dissolved in DMF solvent, primary or secondary amine (2 equivalents) was added drop wise, allowed to stirrer at room temperature. After completion of the reaction (monitored by TLC) the reaction mixture was filtered through a short silica gel pad and the residue was washed with ethyl acetate (10ml).The combined filtrate was washed with saturated solution of $NaHCO_3$(3ml) ,brine(2ml),dried over anhydrous Na_2SO4.The crude product was purified by column chromatography.



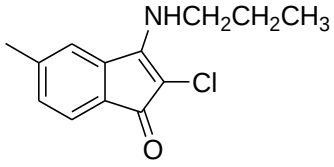
3a

Compound **3a**, red color crystalline solid, Mp 188^{o}C, Yield- 70%, ^{1}H NMR (400 MHz, DMSO-D_6) δ 8.43 (s, 1H), 7.42 (s, 1H), 7.18-7.13 (m, 2H), 3.37 (s, 3H), 2.32 (s, 3H); ^{13}C NMR (100 MHz, DMSO-D_6) δ 21.4, 30.6, 92.4, 119.3, 119.4, 129.8, 130.2, 137.3, 141.2, 156.7, 184.9 ; IR (neat) 3281, 1964, 1685, 1574, 1359, 666 cm^{-1}; HRMS (ESI) m/z calcd for $C_{11}H_{10}ClNO$ $(M+H)^{+}$ 208.0524 found, 208.0524.



3b

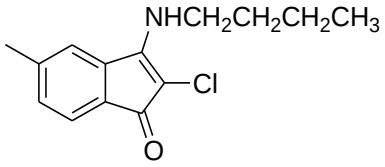
Compound **3b**, red color crystalline solid, Mp 186^{o}C, Yield 78%,^{1}H NMR (400 MHz, DMSO-D_6) δ 0.77 (s, 1H), 7.46 (s, 1H), 7.18-7.13 (m, 2H), 3.75 (q, J = 6.6, 2H) , 2.32 (s, 3H), 1.26 (t, J=8 Hz, 3H); ^{13}C NMR (100 MHz, DMSO-D_6) δ 16.3, 21.4, 37.9, 91.9, 119.4, 129.8, 130.0, 137.5, 141.2, 155.7, 185.0, IR (neat) 3285, 2322, 1570, 898, 662 cm^{-1}; HRMS (ESI) m/z calcd for $C_{12}H_{12}ClNO$ $(M+H)^{+}$ 224.0654 found 224.065.



3c

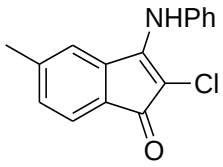
Compound **3c**, red color crystalline solid, Mp 184^{o}C, Yield 75%, ^{1}H NMR (400 MHz, DMSO-D6) δ 8.43 (s, 1H), 7.48 (s, 1H), 7.17-7.12 (m, 2H), 3, 66 (q, J=6.6,

2H) ,2.32 (s, 3H), 1.65 (dt, *J*=6.4, 4, 2H), 0.92 (t, *J* =8, 3H), ^{13}C NMR (100 MHz , DMSO-D_6) δ 10.8, 21.4, 24.0, 40.0, 44.5, 92.0, 119.3, 129.7, 130.0, 137.5, 141.2, 155.9, 185.0, IR (neat) 3282, 2963, 1569, 1311, 663 cm^{-1} ; HRMS (ESI) m/z calcd for $C_{13}H_{14}ClNO$ $(M+H)^+$ 236.0837, found 236.0835.



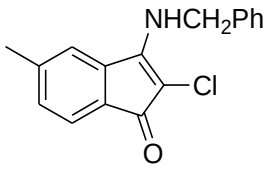
3d

Compound **3d**, red color crystalline solid, Mp 175^{o}C ,Yield 72%, ^{1}H NMR (400MHz , DMSO-D6) δ 8.43 (s, 1H), 7.49(s, 1H), 7.16 (m, 2H), 3.71 (q ,J=6.6 Hz, 2H), 2.33 (s, 3H), 1.65 (m, 2H) 1.38 (m, 2H), 0.91 (m, 3H) ; ^{13}C NMR (100 MHz, DMSO-D_6) δ 13.6, 19.2, 21.4, 32.7, 39.4 ,42.6, 92.0, 119.4, 129.8, 130.0, 137.5 ,141.2, 155.8, 185.0, IR (neat) 3286, 2956, 1570, 1313, 661 cm^{-1} ; HRMS (ESI) m/z calcd for $C_{13}H_{14}ClNO$ $(M+H)^+$ 250.0993, found 250.099.



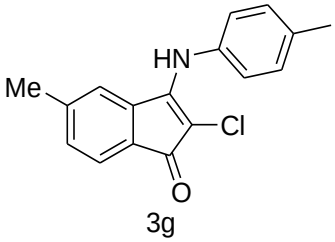
3e

Compound **3e**, crystalline solid, Mp 130^{o}C, Yield 60%, ^{1}H NMR (400 MHz,DMSO-D_6), δ 7.44-7.41, (m, 2H), 7.36-7.32 (m ,3H), 7.28-7.26 (m, 3H), 6.35 (s, 1H) ,2.16 (s, 1H) ,^{13}C NMR (100 MHz, DMSO-D6), δ 21.9, 76.7, 77.3, 101.3, 121.2, 121.7, 125.6, 127.0, 129.2, 129.8, 130.7, 136.5, 137.3, 141.9, 155.7, 185.9, IR (neat) 3056, 2856, 1713, 1599, 1262, 696 cm^{-1}



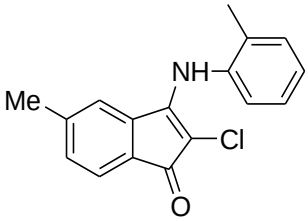
3f

Compound **3f**, reddish brown color crystalline solid, Mp 170^{o}C, ^{1}H NMR (400 MHz, DMSO-D6), δ 7.42-7.26 (m, 7H), 6.92-6.90 (m, 1H), 5.07-4.76 (m, 2H), 2.30 (s, 3H), ^{13}C (100 MHz, DMSO-D6), δ 21.9, 121.1, 127.7, 128.3, 128.8, 129.1, 130.1, 141.1 ; IR (neat) 3228, 1989, 1566, 670, 575 cm^{-1} ; HRMS (ESI) m/z calcd for $C_{17}H_{14}ClNO$ $(M+H)^{+}$ 284.0837, found 284.083.



3g

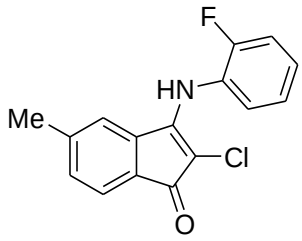
Compound **3g**, semi liquid compound, Yield 78%, ^{1}H NMR (400 MHz (DMSO-D6) δ 10.07 (s, 1H), 7.41 (s, 1H), 7.24-7.13 (m, 3H), 6.94-6.66 (m, 3H), 2.33 (m, 6H), ^{13}C NMR (100 MHz , DMSO-D_6) δ 20.5, 21.5, 95.8, 120.0, 120.5, 125.3, 128.8, 129.6, 129.8, 134.6 ,135.3, 137.8, 141.7, 154.2, 185.6, IR (neat) 3209, 2923, 1734 , 1567, 1216, 711 cm$^{-1;}$ HRMS (ESI) m/z calcd for $C_{17}H_{14}ClNO$ $(M+H)^{+}$ 284.0837, found 284.0845.



3h

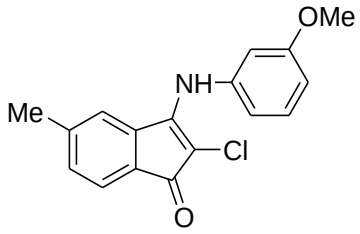
Compound **3h**, reddish brown crystalline solid, ^{1}H NMR (400 MHz, DMSO-D_6) δ 9.96 (s, 1H), 7.33-7.22 (m ,6H), 7.17-7.15 (m, 1H), 2.50 (s, 3H), 2.26 (s, 3H), ^{13}C

NMR (100 MHz, DMSO-D_6) δ 17.5, 21.5, 95.1, 119.9, 120.3, 126.1, 127.6, 127.9, 129.8, 130.0, 130.1, 135.0, 136.0, 137.3, 141.6, 155.5, 185.3, IR (neat) 3209, 2923, 1567,998,711 cm^{-1} ; HRMS (ESI) m/z calcd for $C_{17}H_{14}ClNO$ $(M+H)^+$ 284.0837, found 284.0837.



3i

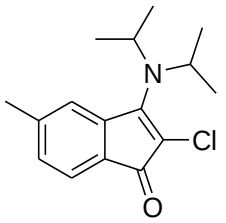
Compound **3i**, red color crystalline solid, Yield 60%, ^1H NMR (400 MHz, DMSO-D_6) δ 9.98 (s, 1H), 7.39-7.26 (m, 4H), 7.22-7.18 (m ,2H), 7.13-7.11 (m, 1H), 2.26 (s, 3H), ^{13}C NMR (100 MHz, DMSO-D_6) δ 19.3, 21.5, 27.5, 59.3, 100.5 ,120.1, 123.7, 129.6, 130.1, 138.1, 142.6, 160.7, 185.2 , IR (neat) 3208, 1676, 1022,668 cm^{-1}



3j

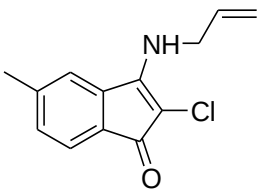
Compound **3j**, reddish black crystalline solid, Yield 72%,^1H NMR (400 MHz, DMSO-D_6), δ 10.08 (s, 1H), 7.39-7.24 (m, 3H), 7.24-7.18 (m, 1H), 6.85-6.83 (m,

3H), 3.78 (s, 3H), 2.33 (s, 3H) ,^{13}C NMR (100 MHz, DMSO-D_6) δ 21.5, 55.1, 67, 110.7, 111.6, 117.4, 120.1, 120.5, 129.0, 129.4, 129.8, 138.0, 138.3 ,141.9, 153.9, 159.1, 185.8, IR (neat) 3363, 2929, 1706, 1602, 1258, 1024,999,730 cm^{-1}



3k

Compound **3k**, red crystalline solid, Yield 68%, ^{1}H NMR (400 MHz, DMSO-D_6) δ 7.52 (s, 1H), 7.36-7.35 (m, 1H), 7.30-7.28 (m, 1H), 3.75 (d, J= 8, Hz, 4H) ,2.65 (s, 3H) ,1.02 (d , J=8, 12H), ^{13}C NMR (100 MHz, DMSO-D_6), δ 21.5,96.1,115.7, 115.8, 120.2, 120.3, 124.3, 124.3, 125.1, 125.2, 128.9, 129.2, 129.9, 137.6, 142.1, 154.9, 155.8, 158.3, IR (neat) 3246, 2290, 1200,686 cm^{-1}; ; HRMS (ESI) m/z calcd for $C_{17}H_{14}ClNO$ $(M+H)^+$ 306.1619, found 306.1621.



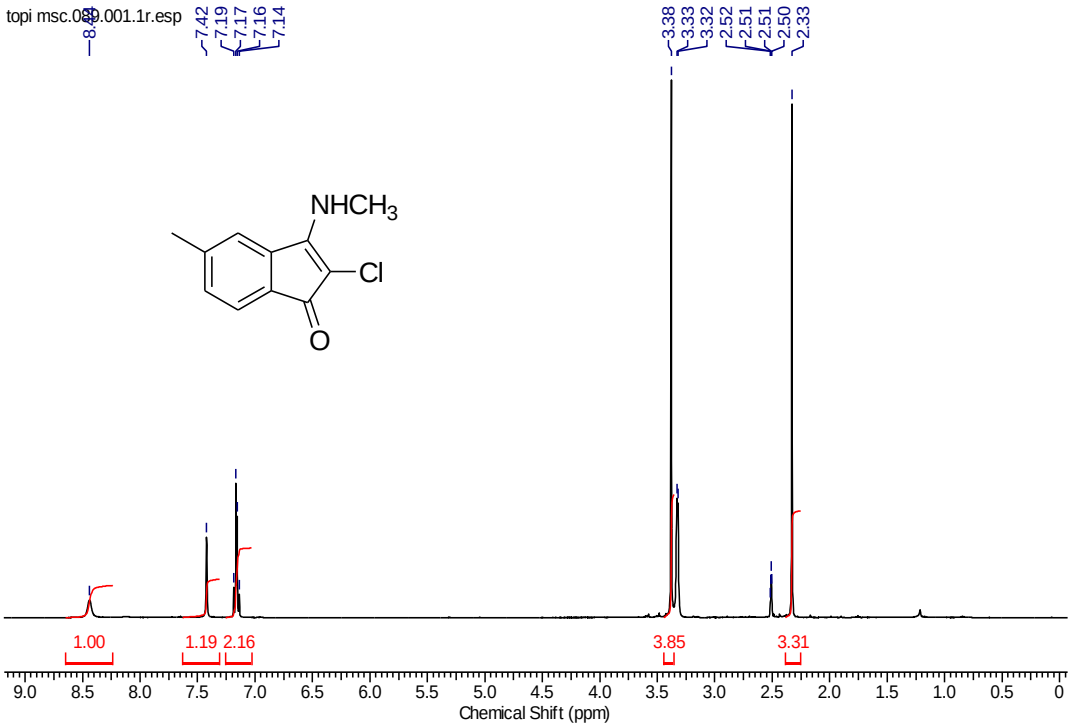
3l

Compound **3l**, red color crystalline solid , Mp 168^{o}C ,Yield 64%,^{1}H NMR (400 MHz, DMSO-D_6), δ 8.58 (s, 1H), 7.50 (s, 1H) ,7.19-7.13 (m, 2H), 6.05-5.95 (m, J=8, 1H), 5.21-5.16 (m, 2H), 4.36-4.33 (m, 2H), 2.33 (s, 3H); ^{13}C NMR (100 MHz, DMSO-D6), δ 21.4, 44.8, 92.6, 115.9, 119.5, 129.8, 135.0, 137.6, 141.4, 155.9,

185.2, IR (neat) 3279, 1563, 1205 ,776 ,659 cm^{-1} ; HRMS (ESI) m/z calcd for $C_{13}H_{12}ClNO$ $(M+H)^+$ 234.068, found 234.0677.

Spectral data

^{1}H NMR (400 MHz), ^{13}C NMR (100 MHz) in (DMSO-D_6) compounds (3a-l)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Cl
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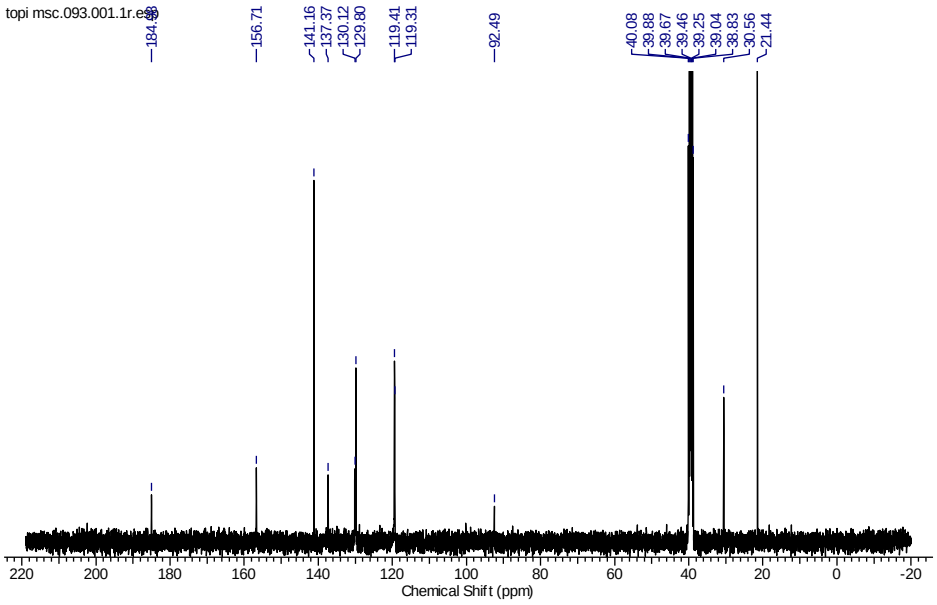
3.31
Chemical Shift (ppm)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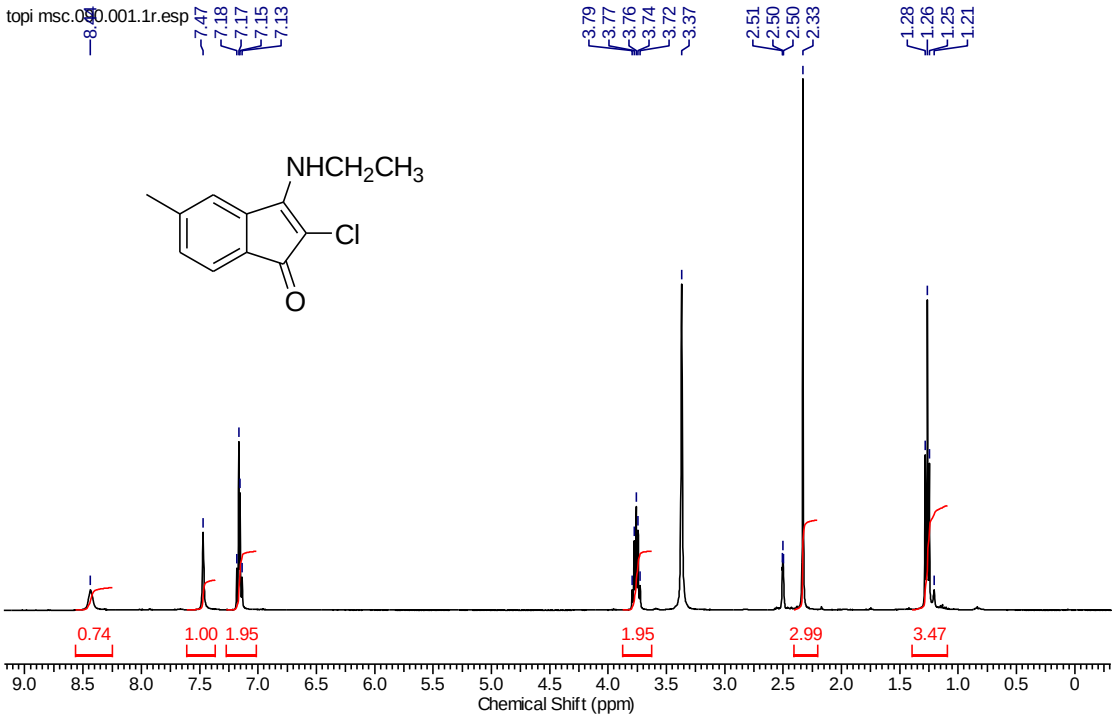
Chemical Shift (ppm)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2.50
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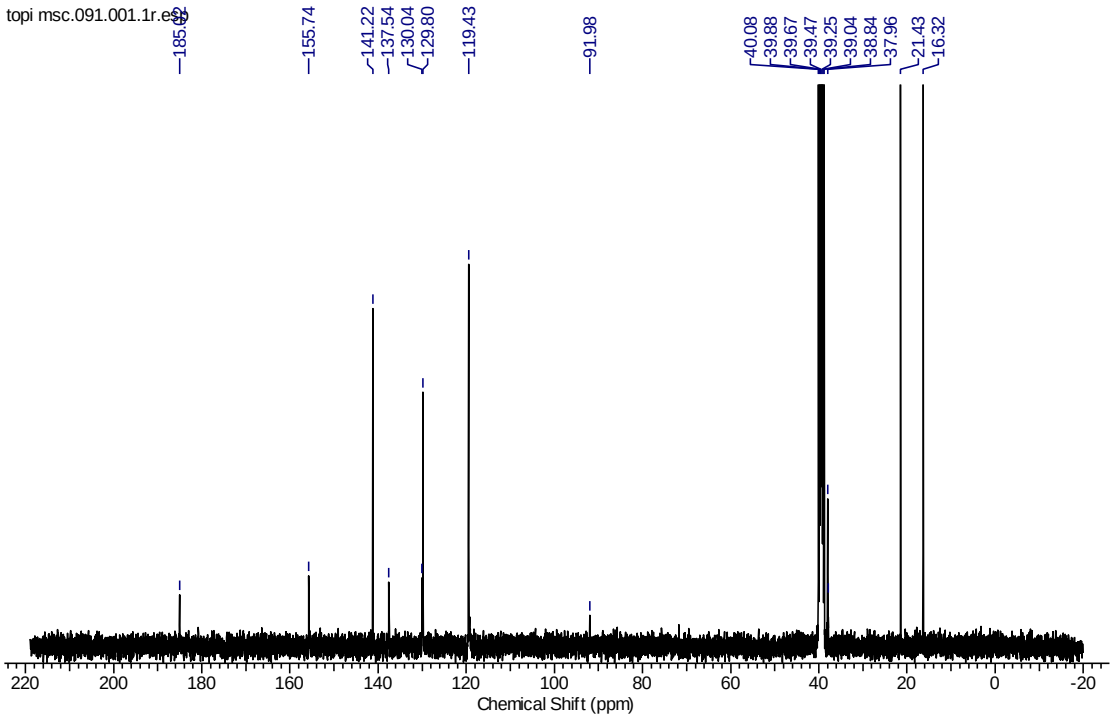
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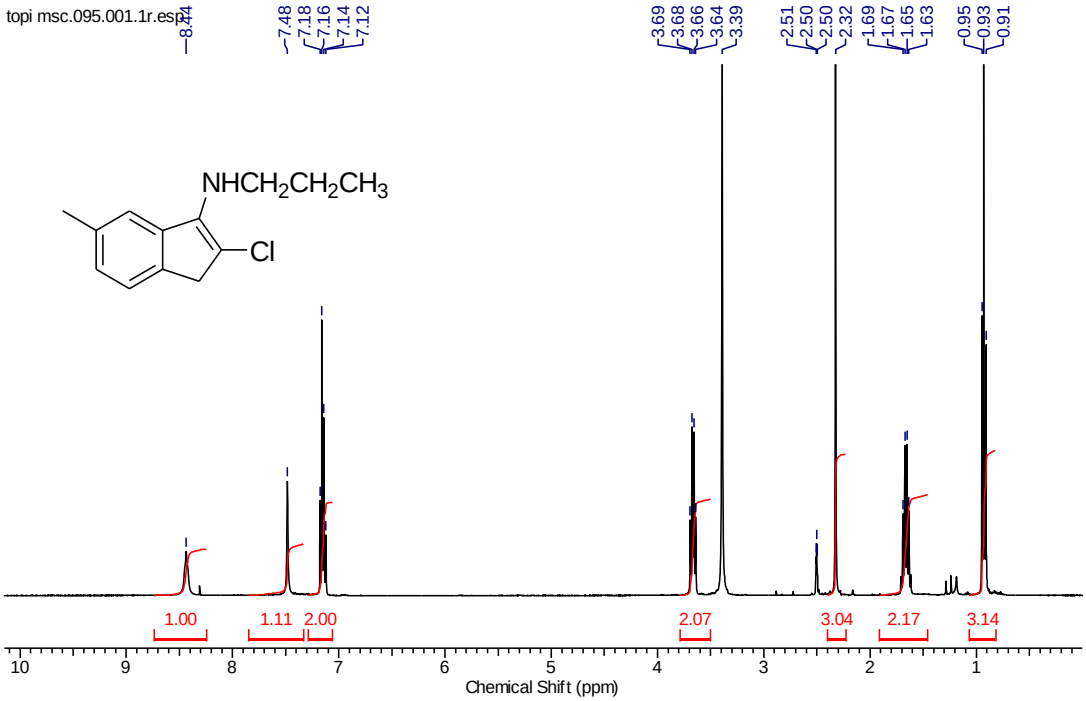
Chemical Shift (ppm)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1.63
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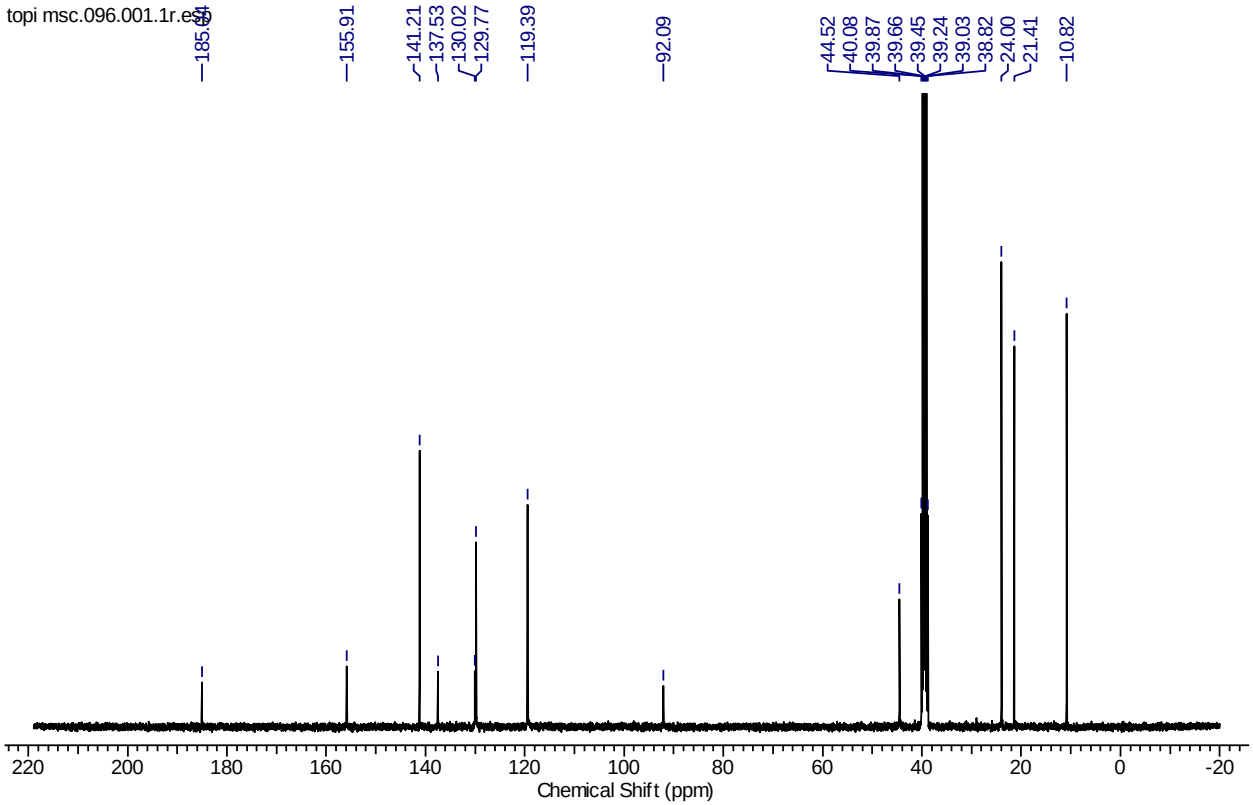
Chemical Shift (ppm)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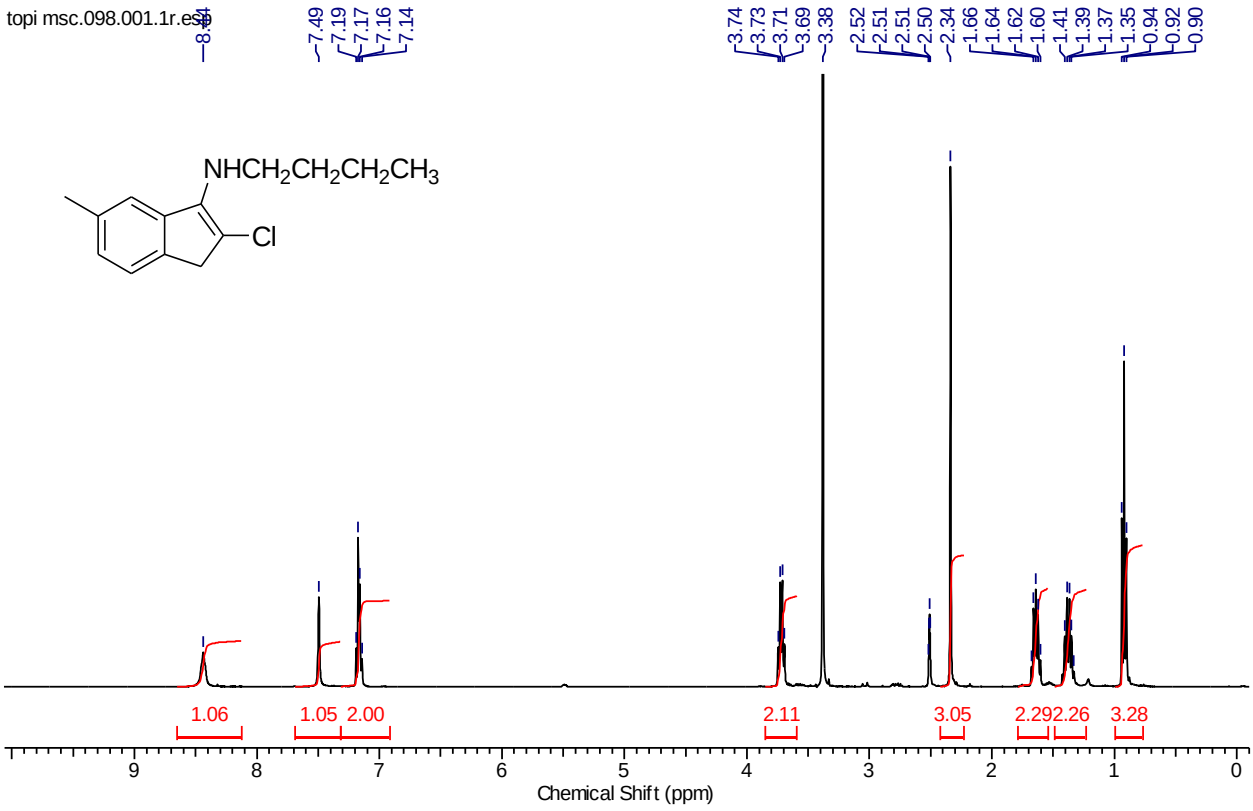
Chemical Shift (ppm)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2.50
2.34
1.66
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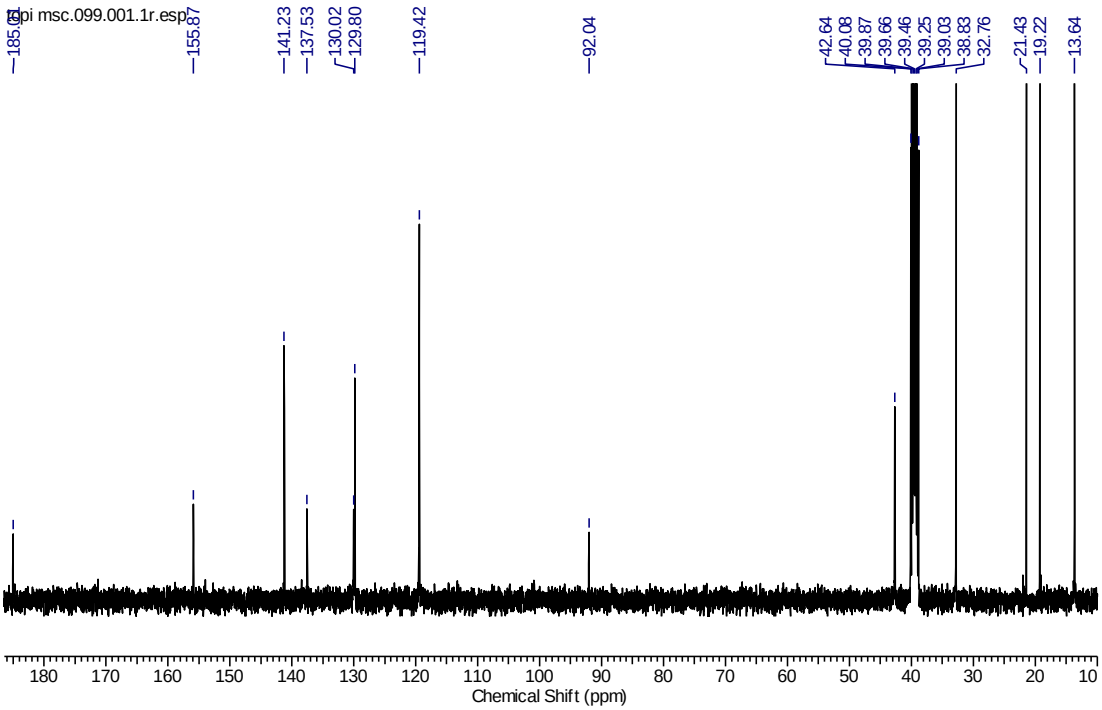
Chemical Shift (ppm)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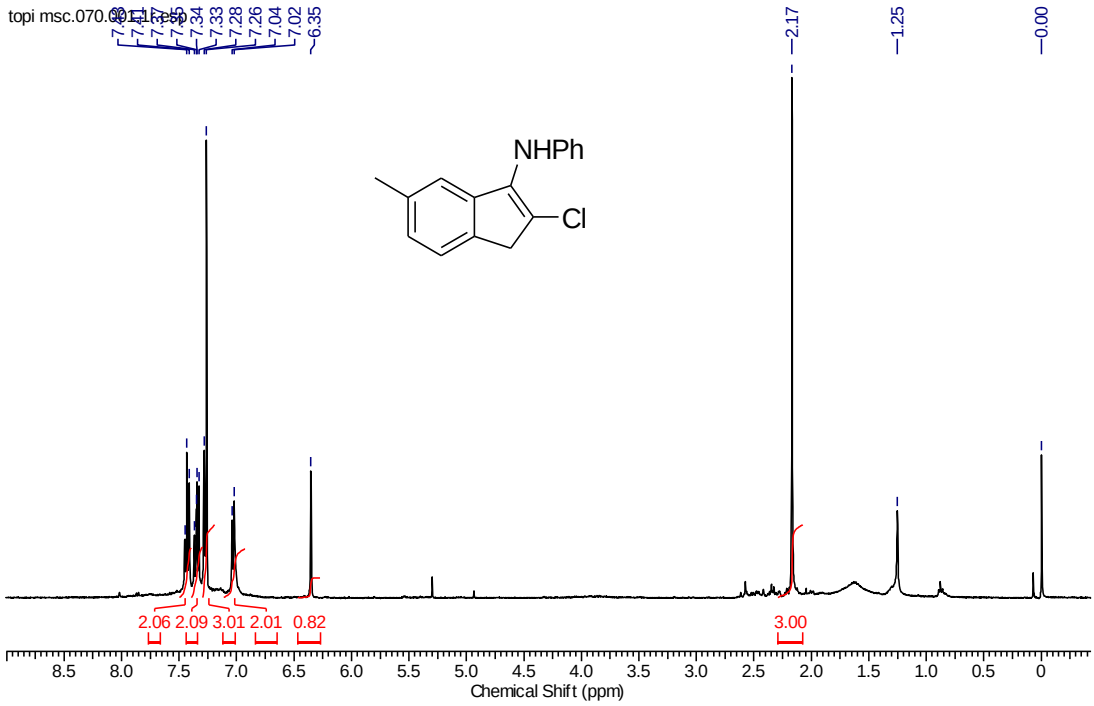
13.64
Chemical Shift (ppm)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Cl
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2.09
3.01
2.01
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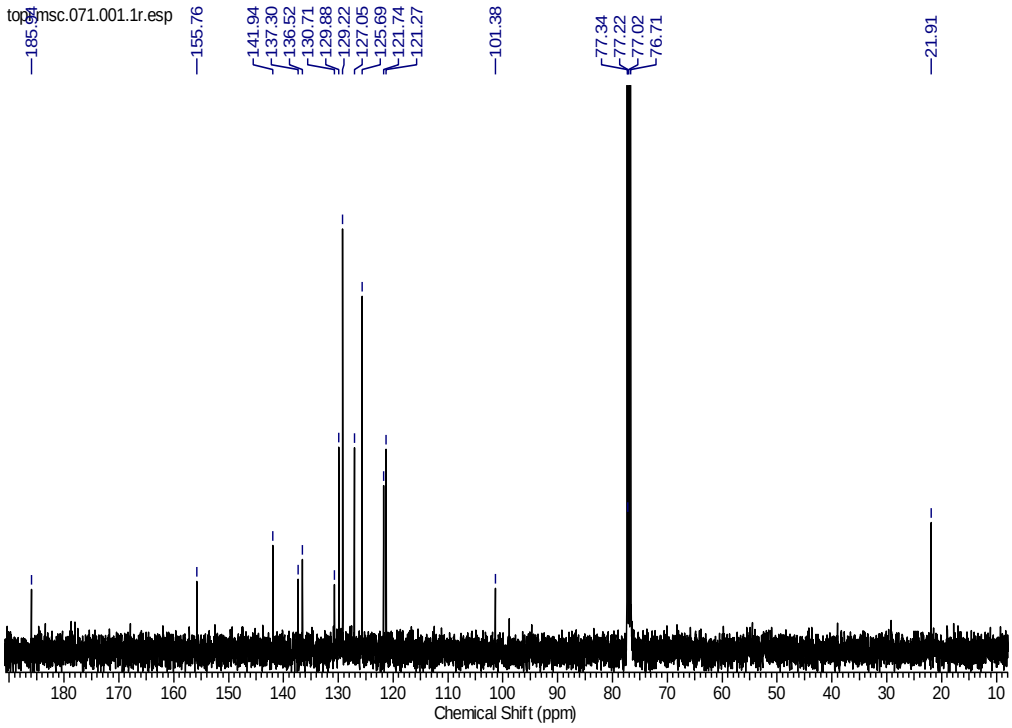
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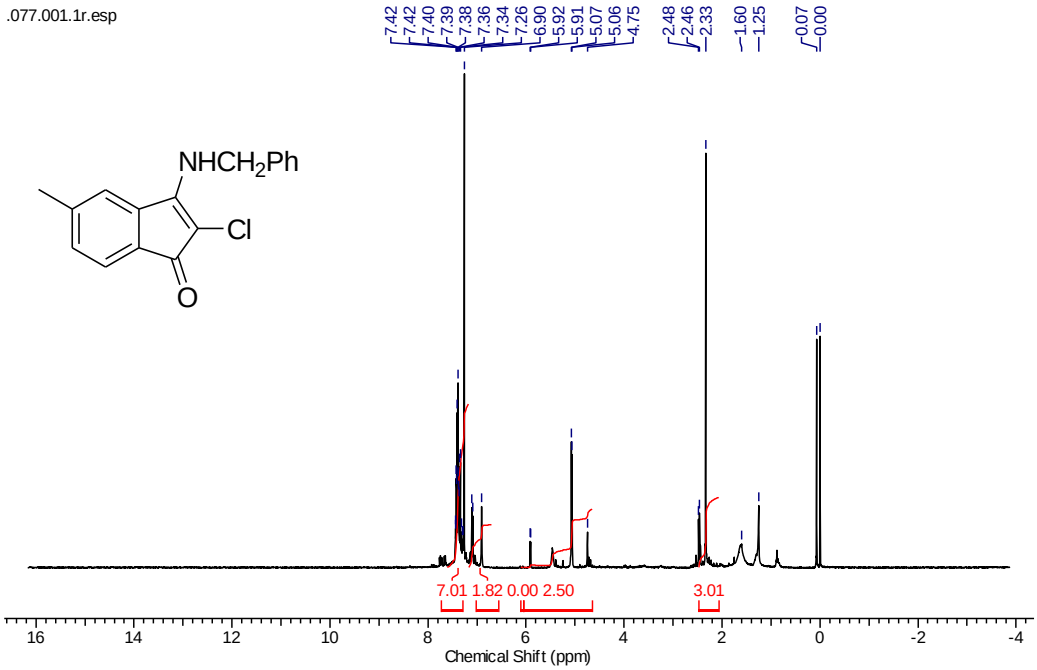
Chemical Shift (ppm)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1.25
0.07
0.00
NHCH2Ph
Cl
O
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4
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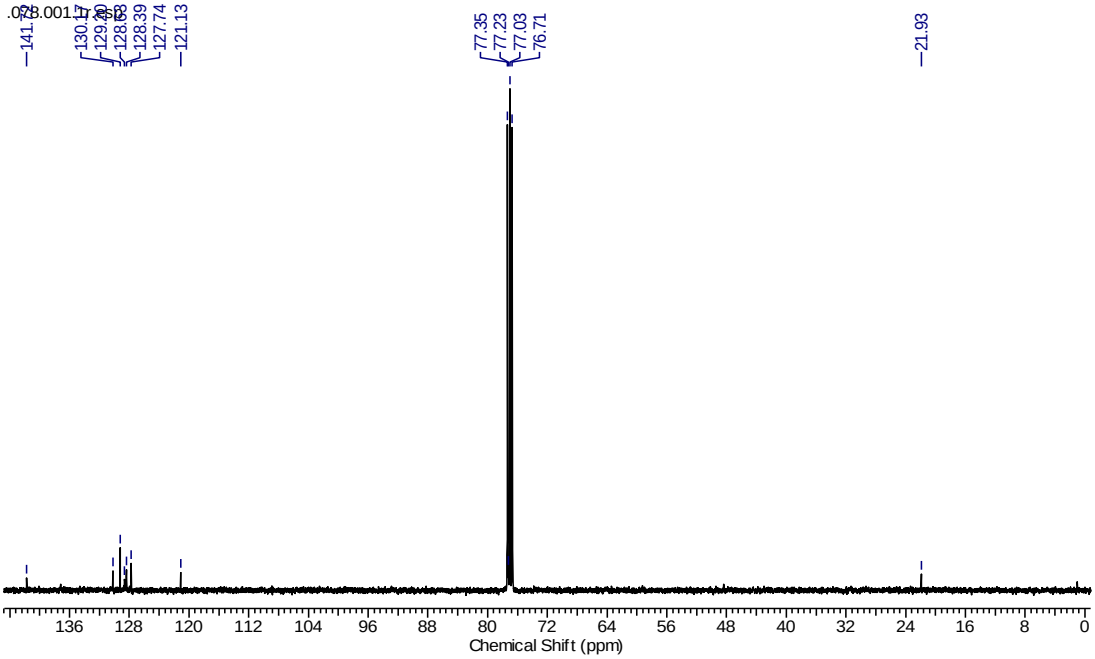
Chemical Shift (ppm)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32
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8
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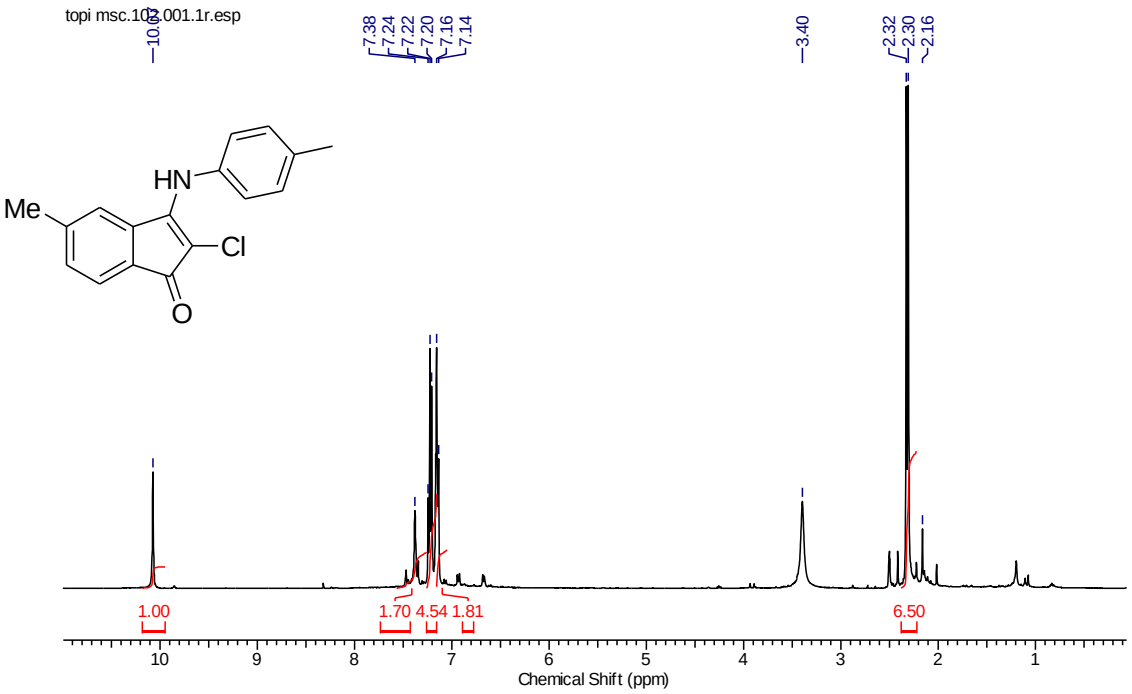
Chemical Shift (ppm)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Me
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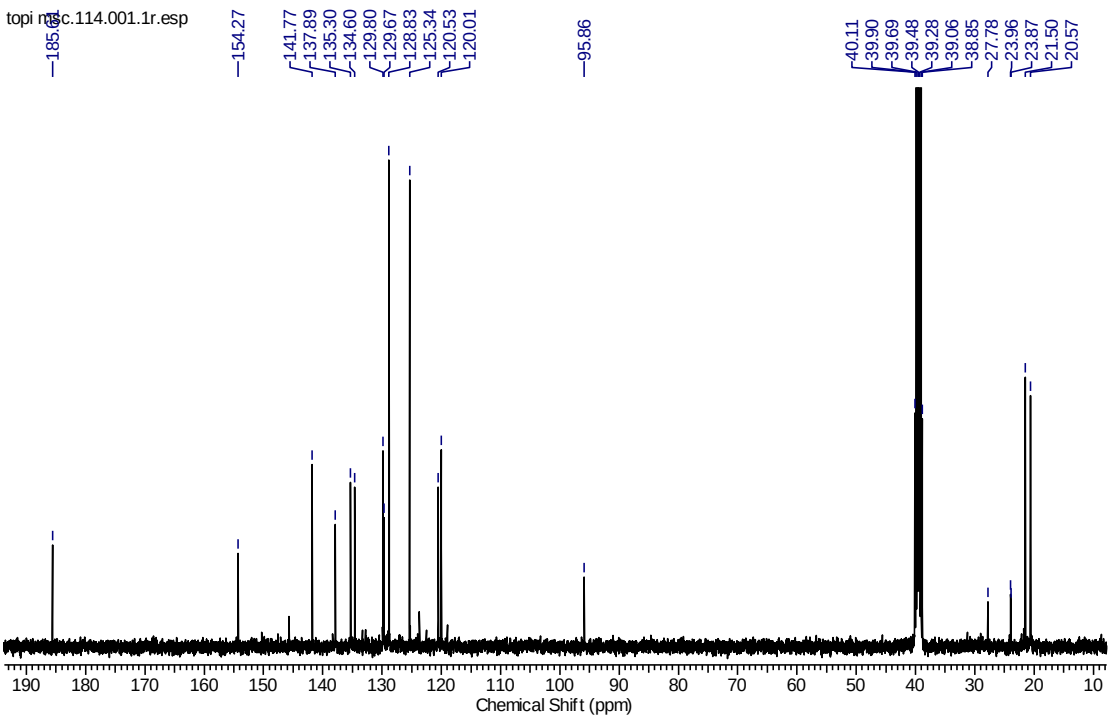
Chemical Shift (ppm)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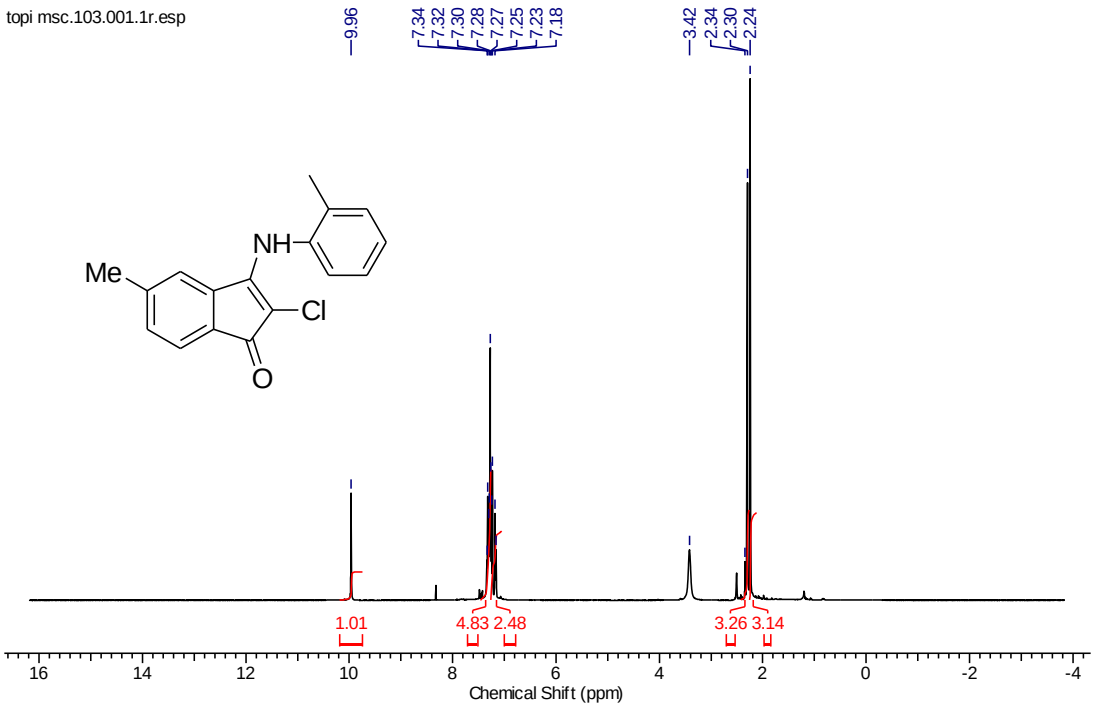
Chemical Shift (ppm)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Me
Cl
O
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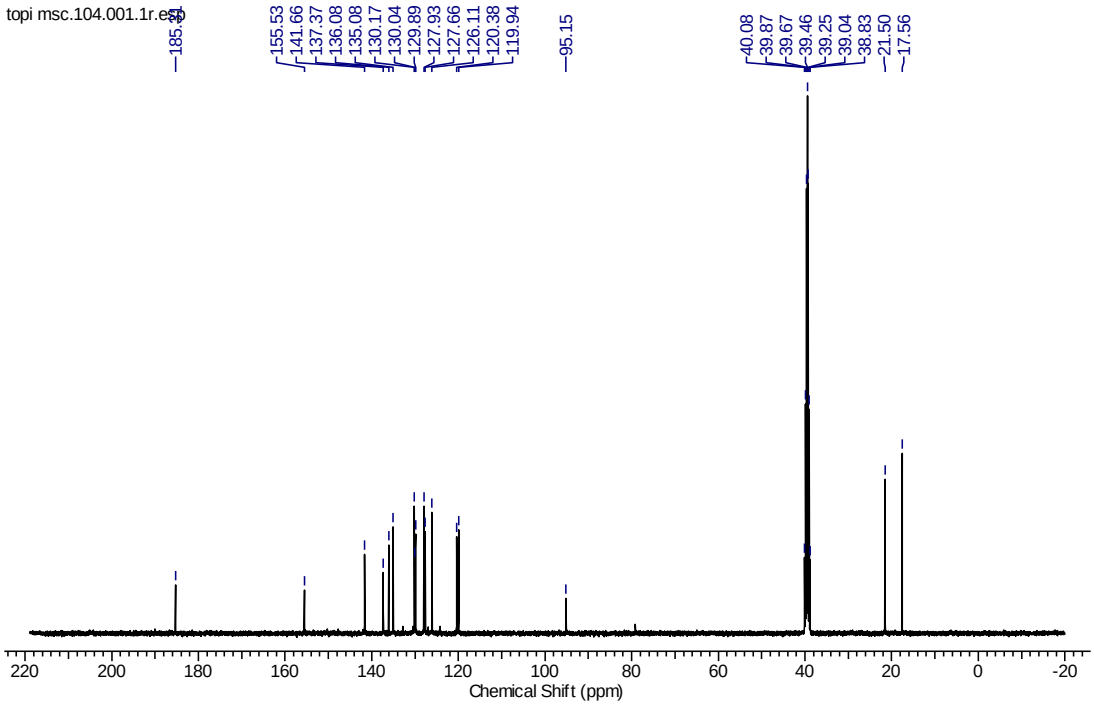
Chemical Shift (ppm)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60
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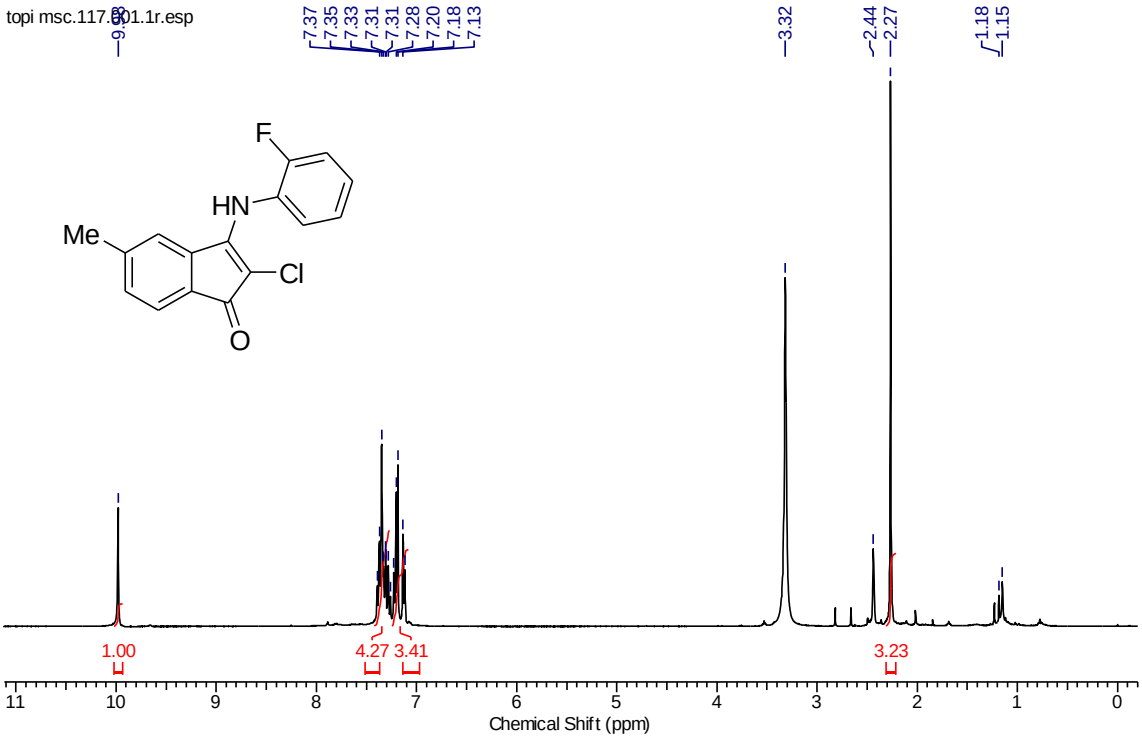
Chemical Shift (ppm)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Me
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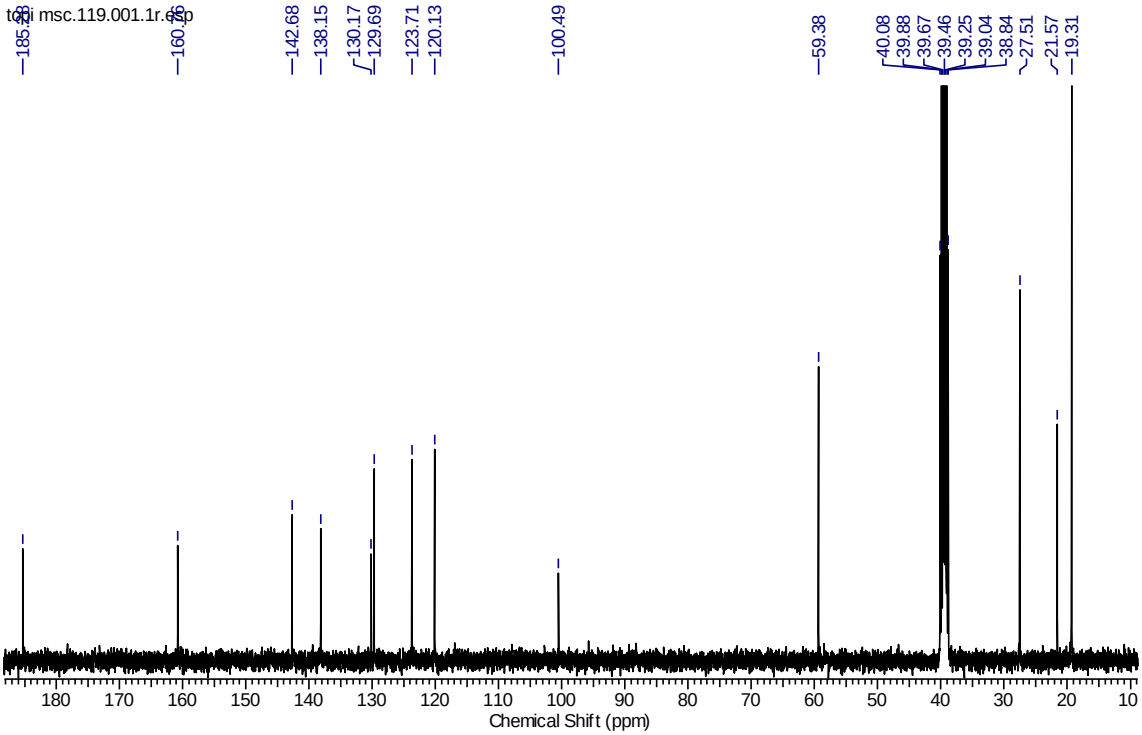
Chemical Shift (ppm)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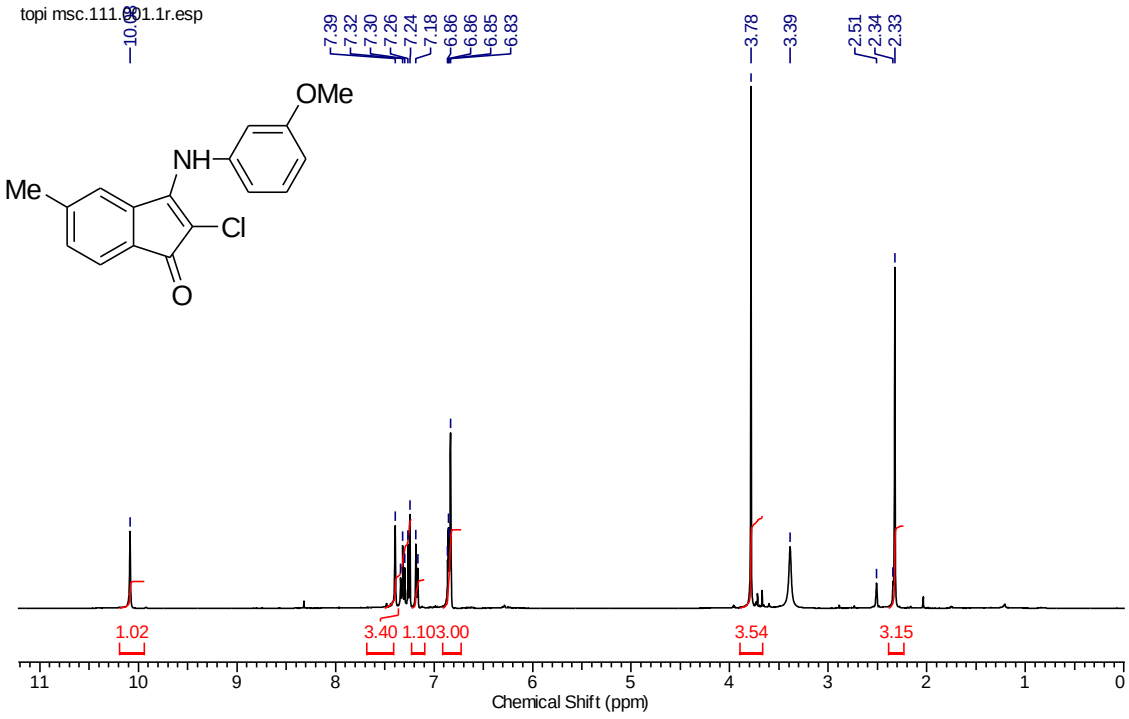
Chemical Shift (ppm)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Cl
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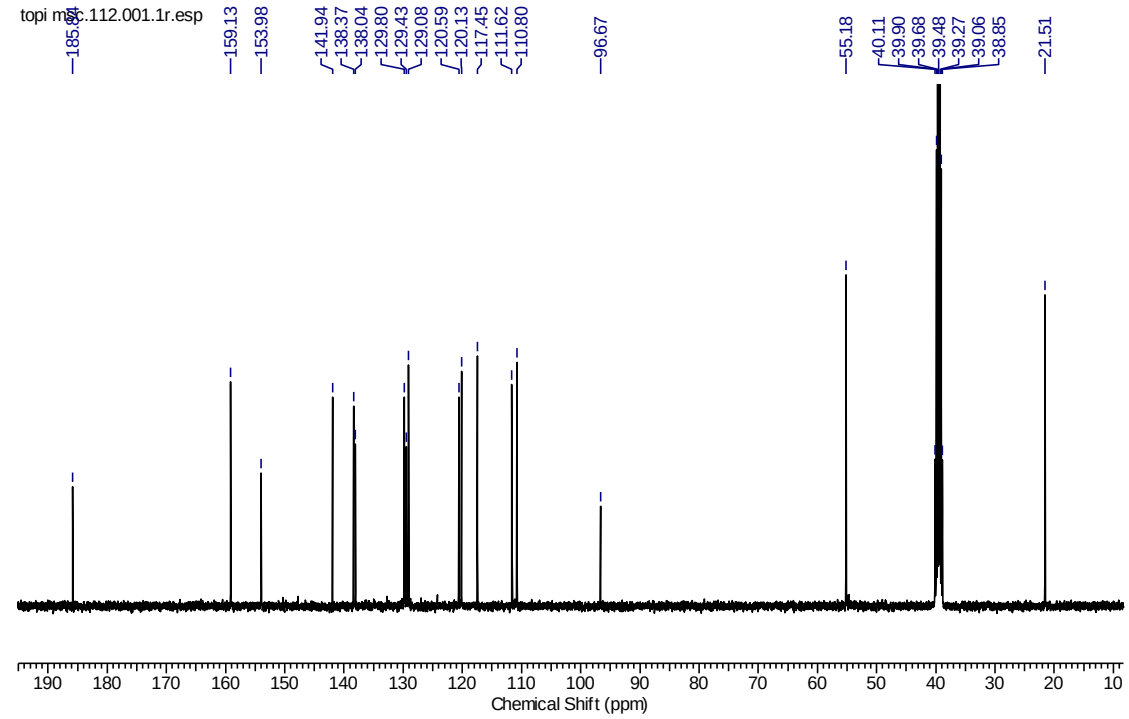
Chemical Shift (ppm)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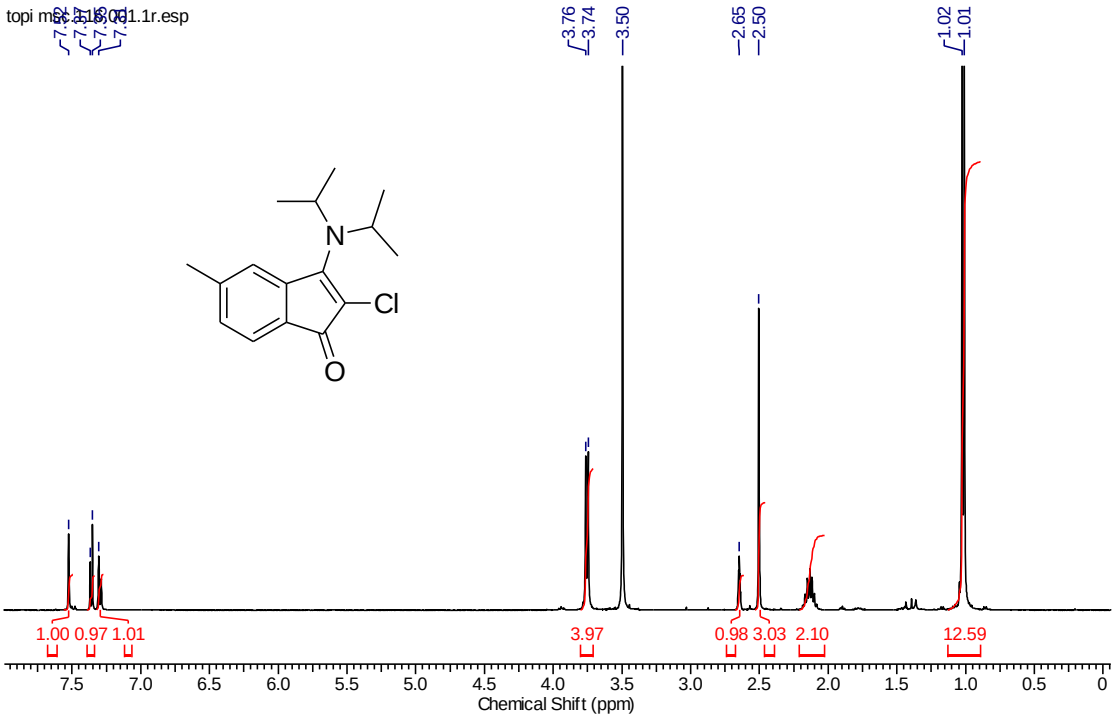
Chemical Shift (ppm)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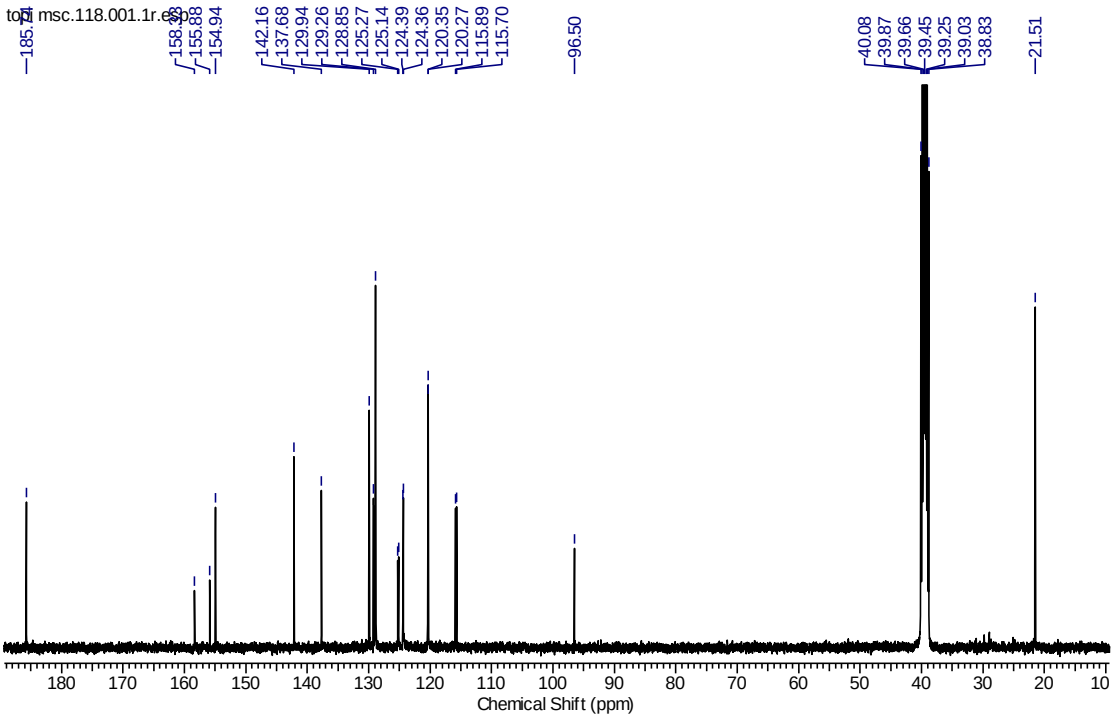
0.98 3.03 2.10
12.59
Chemical Shift (ppm)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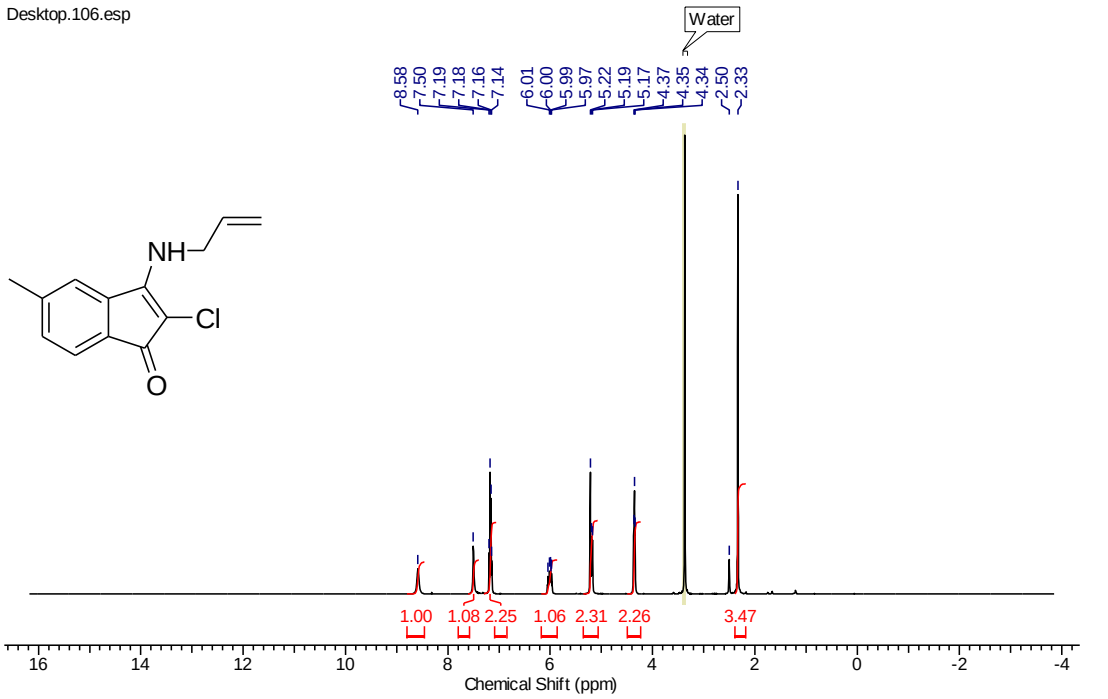
Chemical Shift (ppm)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4.35
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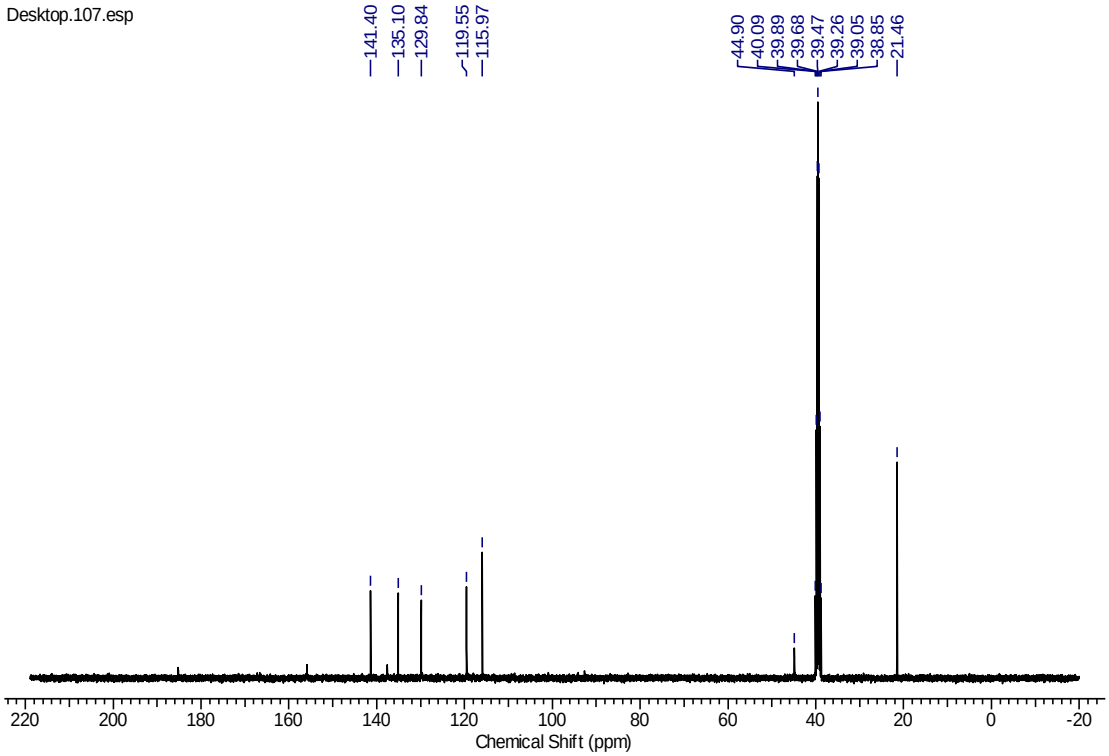
Chemical Shift (ppm)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39.68
39.47
39.26
39.05
38.85
21.46
Chemical Shift (ppm)

Reference

1) Babu, K. R; Khan, F. A. *Org. Biomol.Chem*, 2015, *13*, 299.

2) (a) Khan, F. A; Sudheer, Ch. *Org. Lett*, 2008, *10*, 3029. (b) Khan F. A; Rout, B. *Tetrahedron Lett*, 2006, *47*, 5251.

3) (a) Khan F. A ; Dash, J ; Jain, D ; Das, B. P. *J. Chem. Soc.,Perkin Trans. 1*, 2001, 3132. (b) Khan, F. A; Soma, L. *Tetrahedron Lett*, 2007, *48*, 85.

4) Khan, F. A; Choudhury, S. *Eur. J. Org. Chem*, 2006, 672.

5) *J. Braz. Chem. Soc.*, Vol. 22, No. 3, 598-603, 2011.