

Electronic Supplementary Information (ESI)

AlCl₃@ZnO nanostructured material: an efficient green catalyst to one-pot solvent-free synthesis of 1, 4 dihydropyridines

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Table 6: Synthesis of 1, 4 dihydropyridine (4a–n) using 20% AlCl₃@ZnO catalyst at solvent free condition.

Entry	Ar 1(a-n)	Compound 4(a-n)	Yield*	Melting Point (°C)[ref.]	
				Reported	Observed
1	C ₆ H ₅	4a	92	158-160 ¹	158
2	4-ClC ₆ H ₄	4b	93	147-148 ¹	146
3	4-FC ₆ H ₄	4c	90	155-157 ²	154
4	4-BrC ₆ H ₄	4d	88	158-160 ³	160
5	4-OMeC ₆ H ₄	4e	92	161-163 ¹	162
6	4-NO ₂ C ₆ H ₄	4f	80	129-131 ¹	128
7	2-NO ₂ C ₆ H ₄	4g	75	-	168
8	4-OHC ₆ H ₄	4h	78	226-228 ³	130
9	2-Cl, 6-FC ₆ H ₃	4i	78	-	142
10	2-ClC ₆ H ₄	4j	86	80-82 ³	82
11	3-ClC ₆ H ₄	4k	88	245-147 ²	148
12	2-FC ₆ H ₄	4l	88	155-157 ²	154
13	4-CH ₃ C ₆ H ₄	4m	84	162-164 ³	162
14	3-CH ₃ C ₆ H ₄	4n	82	122-124 ⁴	122

Reaction conditions: 1(a-n) substituted benzaldehydes (4.71 mmol), 2 Ethyl acetoacetate (9.42 mmol), 3 ammonium acetate (5.66 mmol), and 20% AlCl₃@ZnO (1.17 mmol) under solvent-free conditions, room temperature, 2 h.

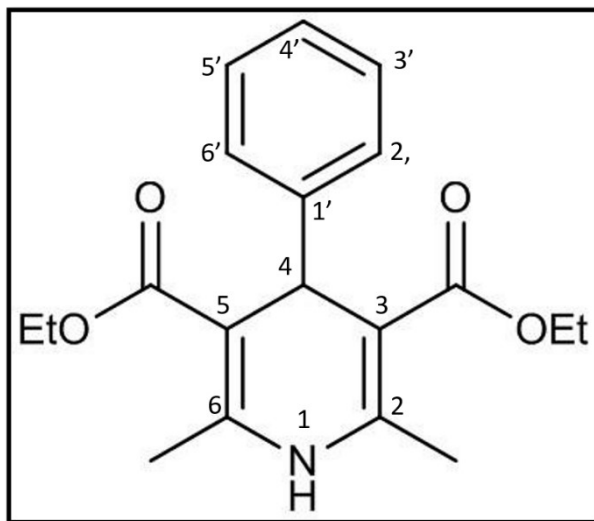
*Isolated Yield.

References

- 1 Abdolhamid Bamoniri and Sara Fouladgar, *RSC Adv*, 2014, DOI:10.1039/x0xx00000x.
- 2 A. Kuraitheerthakumaran, S. Pazhamalai and M. Gopalakrishnan, *Chinese Chemical Letters*, 2011, **22**, 1199–1202.
- 3 A. Teimouri, L. Ghorbanian and A. Moatari, *Bull Chem Soc Ethiop*, 2013, **27**, 427–437.
- 4 A. Debache, R. Boulcina, A. Belfaitah, S. Rhouati and B. Carboni, *Synlett*, 2008, 509–512.

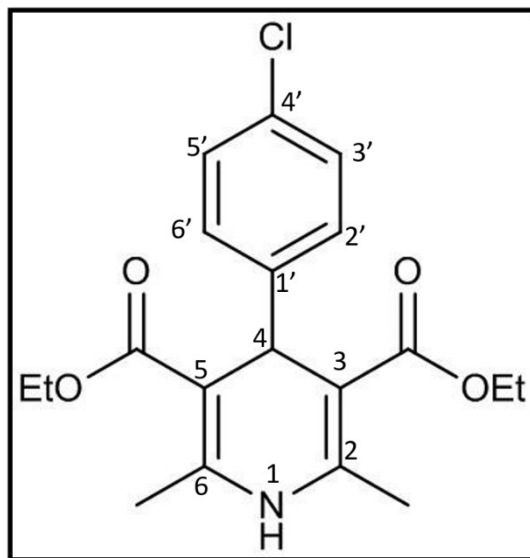
I. Spectral data of as-synthesized 1, 4 dihydropyridines derivatives (Table 3, entries 1 to 9)

2, 6-Dimethyl-4-phenyl-1,4-dihydropyridine-3,5-diethylcarboxylate [Compound (a) Entry 1, Table 6].



White solid; FT-IR 3338, 2981, 1683, 1653, 1558, 1207 cm^{-1} ; $^1\text{H-NMR}$ (CDCl_3) δ 1.22 (t, $J = 7.0$ Hz, 6H, 2, $\text{OCH}_2\text{-CH}_3$), 2.33 (s, 6H, 2, CH_3), 4.08 (q, $J = 7.0$ Hz, 4H, 2, $\text{OCH}_2\text{-CH}_3$), 4.99 (s, 1H, CH), 5.64 (s, 1H, NH), 7.13 (t, $J = 2.0$ and 8.6 Hz, 1H, H-4'), 7.21 (t, $J = 2$ and 8.6 Hz, 2H, H-2' & H-6'), 7.27 (t, $J = 8.6$ Hz, 2H, H-3' & H-5') ppm; $^{13}\text{C-NMR}$ (CDCl_3) δ 14.26 (2, $\text{OCH}_2\text{-CH}_3$), 19.63 (2, CH_3), 39.63 (C-4), 59.74 (2, $\text{OCH}_2\text{-CH}_3$), 104.21 (C-3 & C-5), 126.11 (C-4'), 127.84 (C-2' & C-6'), 128.03 (C-3' & C-5'), 143.82 (C-2 & C-6), 147.76 (C-1'), 167.64 (2, C=O) ppm; HRMS m/z Calcd. for $\text{C}_{19}\text{H}_{24}\text{NO}_4$ [$\text{M}+\text{H}$]: 330.1705, Found: 330.1703.

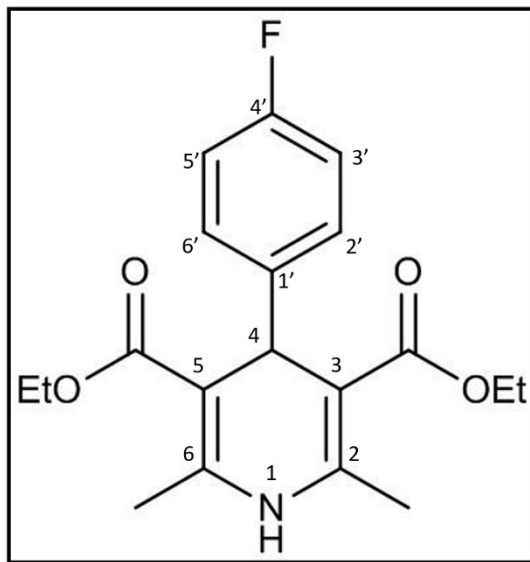
2, 6-Dimethyl-4-(4-chlorophenyl)-1, 4-dihydropyridine-3, 5-diethylcarboxylate [Compound (b) Entry 2, Table 6].



White solid; FT-IR 3354, 2981, 1693, 1651, 1485, 1209, 829 cm^{-1} ; $^1\text{H-NMR}$ (CDCl_3) δ 1.23 (t, $J = 7.0$ Hz, 6H, 2, $\text{OCH}_2\text{-CH}_3$), 2.32 (s, 6H, 2, CH_3), 4.08 (q, $J = 7.0$ Hz, 4H, 2, $\text{OCH}_2\text{-CH}_3$), 4.95 (s, 1H, CH), 5.68 (s, 1H, NH), 7.17 (d, $J = 8.0$ Hz, 2H, H-2' & H-6'), 7.22 (d, $J = 8.0$ Hz, 2H, H-3' & H-5') ppm; $^{13}\text{C-NMR}$ (CDCl_3) δ 14.27 (2, $\text{OCH}_2\text{-CH}_3$), 19.63 (2, CH_3), 39.26 (C-4), 59.84 (2, $\text{OCH}_2\text{-CH}_3$), 103.90 (C-3 & C-5), 127.95 (C-3' & C-5'), 129.44 (C-2' & C-6'), 131.70 (C-4'), 143.99 (C-2 & C-6), 146.34 (C-1'), 167.44 (2, C=O) ppm; HRMS m/z Calcd. for $\text{C}_{19}\text{H}_{23}\text{NO}_4\text{Cl}$ [M+H]: 364.1316, Found: 364.1310.

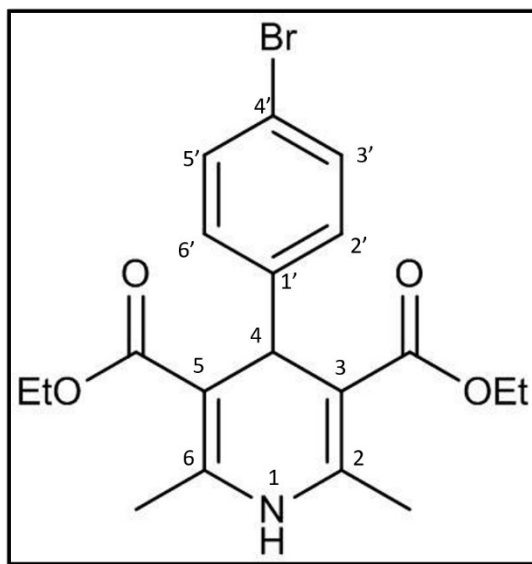
2,6-Dimethyl-4-(4-fluorophenyl)-1,4-dihydropyridine-3,5-diethylcarboxylate [Compound

(c) Entry 3, Table 6]



White solid; FT-IR 3340, 2981, 1685, 1649, 1506, 1207 cm^{-1} ; $^1\text{H-NMR}$ (CDCl_3) δ 1.22 (t, $J = 7.2$ Hz, 6H, 2, $\text{OCH}_2\text{-CH}_3$), 2.33 (s, 6H, 2, CH_3), 4.10 (q, $J = 7.2$ Hz, 4H, 2, $\text{OCH}_2\text{-CH}_3$), 4.96 (s, 1H, CH), 5.66 (s, 1H, NH), 6.88 (t, $J = 8.0$ Hz, 2H, H-3' & 5'), 7.21 (dd, $J = 1.5$ and 8.0 Hz, 2H, H-2' & 6') ppm; $^{13}\text{C-NMR}$ (CDCl_3) δ 14.27 (2, $\text{OCH}_2\text{-CH}_3$), 19.622 (2, CH_3), 39.05 (C-4), 59.79 (2, $\text{OCH}_2\text{-CH}_3$), 104.19 (C-3 & C-5), 114.50 (d, $J = 21.2$ Hz, C-3' & C-5'), 129.46 (d, $J = 7.5$ Hz, C-2' & C-6'), 143.67 (d, $J = 2.5$ Hz, C-1'), 143.80 (C-2 & C-6), 161.36 (d, $J = 242.5$ Hz, C-4'), 167.53 (2, C=O) ppm; HRMS m/z Calcd. for $\text{C}_{19}\text{H}_{23}\text{NO}_4\text{F}$ [$\text{M}+\text{H}$]: 348.1611 Found: 348.1605.

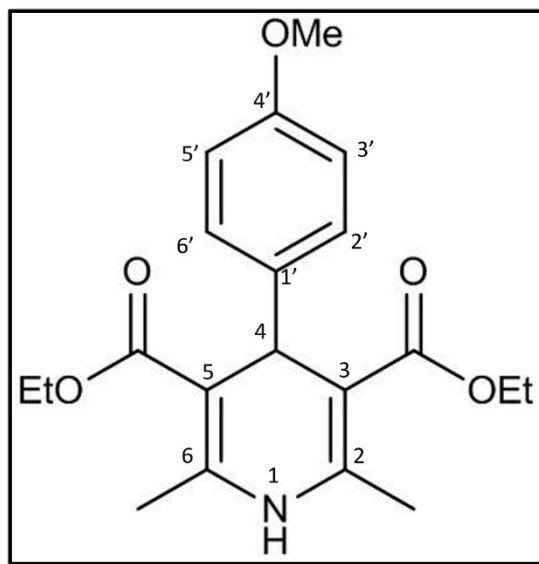
2, 6-Dimethyl-4-(4-bromophenyl)-1, 4-dihydropyridine-3, 5-diethylcarboxylate [Compound (d) Entry 4, Table 6]



White solid; FT-IR 3358, 2981, 1689, 1647, 1481, 1209, 1116 cm^{-1} ; $^1\text{H-NMR}$ (CDCl_3) δ 1.24 (t, $J = 7.2$ Hz, 6H, 2, $\text{OCH}_2\text{-CH}_3$), 2.32 (s, 6H, 2, CH_3), 4.07 (q, $J = 7.2$ Hz, 4H, 2, $\text{OCH}_2\text{-CH}_3$), 4.94 (s, 1H, CH), 5.69 (s, 1H, NH), 7.15 (d, $J = 8.0$ Hz, 2H, H-2 & H-6), 7.32 (d, $J = 8.0$ Hz, 2H, H-3 & H-5) ppm; $^{13}\text{C-NMR}$ (CDCl_3) δ 14.28 (2, $\text{OCH}_2\text{-CH}_3$), 19.63 (2, CH_3), 39.34 (C-4), 59.85 (2, $\text{OCH}_2\text{-CH}_3$), 103.82 (C-3 & C-5), 119.89 (C-4'), 129.85 (C-2' & C-6'), 130.90 (C-3' & C-5'), 144.02 (C-2 & C-6), 146.83 (C-1'), 167.42 (2, C=O) ppm; HRMS m/z Calcd. for $\text{C}_{19}\text{H}_{23}\text{NO}_4\text{Br}$ [M+H]: 408.0810, Found: 408.0807.

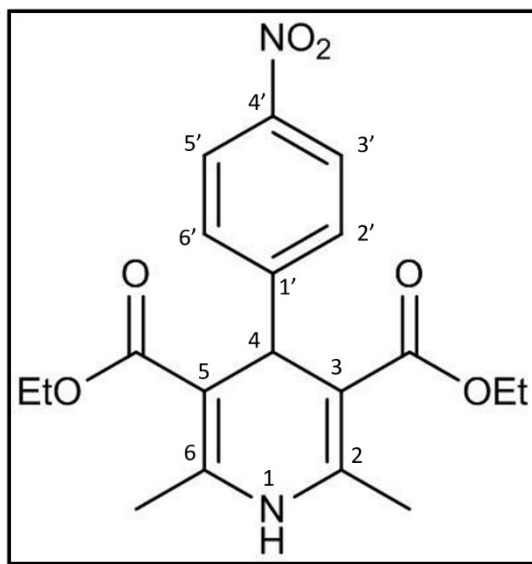
2,6-Dimethyl-4-(4-methoxyphenyl)-1,4-dihydropyridine-3,5-diethylcarboxylate [Compound

(e) Entry 5, Table 6]



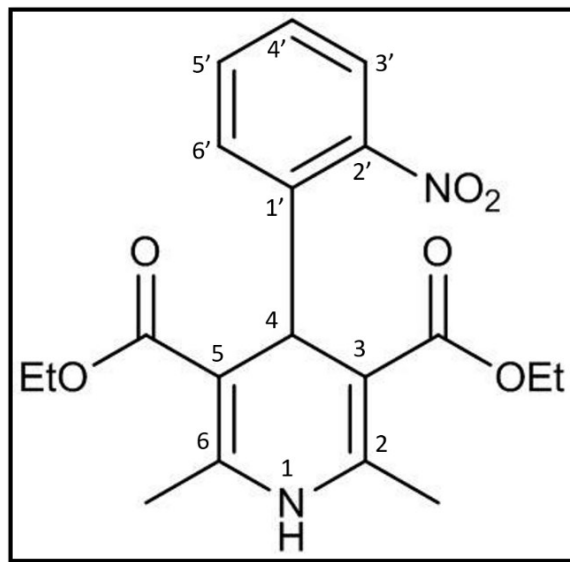
White solid; FT-IR 3340, 2982, 1688, 1646, 1206, 1116, 1024, 832 cm^{-1} ; $^1\text{H-NMR}$ (CDCl_3) δ 1.22 (t, $J = 7.0$ Hz, 6H, 2, O-CH₂-CH₃), 2.31 (s, 6H, 2, CH₃), 3.748 (s, 3H, OCH₃), 4.09 (q, $J = 7.0$ Hz, 4H, 2, OCH₂-CH₃), 4.93 (s, 1H, CH), 5.76 (s, 1H, NH), 6.75 (d, $J = 8.0$ Hz, 2H, H-3' & H-5'), 7.20 (d, $J = 8.0$ Hz, 2H, H-2' & H-6') ppm; $^{13}\text{C-NMR}$ (CDCl_3) δ 14.29 (2, OCH₂-CH₃), 19.56 (2, CH₃), 39.04 (C-4), 55.15 (OCH₃), 59.72 (2, OCH₂-CH₃), 104.34 (C-3 & C-5), 113.31 (C-3' & C-5'), 128.97 (C-2' & C-6'), 140.37 (C-1'), 143.67 (C-2 & C-6), 157.88 (C-4'), 167.75 (2, C=O) ppm; HRMS m/z Calcd. for $\text{C}_{20}\text{H}_{26}\text{NO}_5[\text{M}+\text{H}]$: 360.1811, Found: 360.1807.

2, 6-Dimethyl-4-(4-nitrophenyl)-1, 4-dihydropyridine-3, 5-diethylcarboxylate [Compound (f) Entry 6, Table 6]



Yellow solid; FT-IR 3307, 2981, 1687, 1598, 1479, 1211, 856 cm^{-1} ; $^1\text{H-NMR}$ (CDCl_3) δ 1.21 (t, $J = 7.0$ Hz, 6H, 2, $\text{OCH}_2\text{-CH}_3$), 2.36 (s, 6H, 2, CH_3), 4.10 (q, $J = 7.0$ Hz, 4H, 2, $\text{CH}_2\text{-CH}_3$), 5.09 (s, 1H, CH), 5.70 (s, 1H, NH), 7.45 (d, $J = 8.0$ Hz, 2H, H-2' & H-6'), 8.08 (d, $J = 8.0$ Hz, 2H, H-3' & H-5') ppm; $^{13}\text{C-NMR}$ (CDCl_3) δ 14.27(2, $\text{OCH}_2\text{-CH}_3$), 19.72 (2, CH_3), 40.14 (C-4), 60.03 (2, $\text{OCH}_2\text{-CH}_3$), 103.27 (C-3 & C-5), 123.31 (C-3' & C-5'), 128.92 (C-2' & C-6'), 144.57 (C-2 & C-6), 146.36 (C-1'), 155.08 (C-4'), 167.05 (2, C=O) ppm; HRMS m/z Calcd. for $\text{C}_{19}\text{H}_{23}\text{N}_2\text{O}_6$ [M+H]: 375.1556, Found: 375.1551.

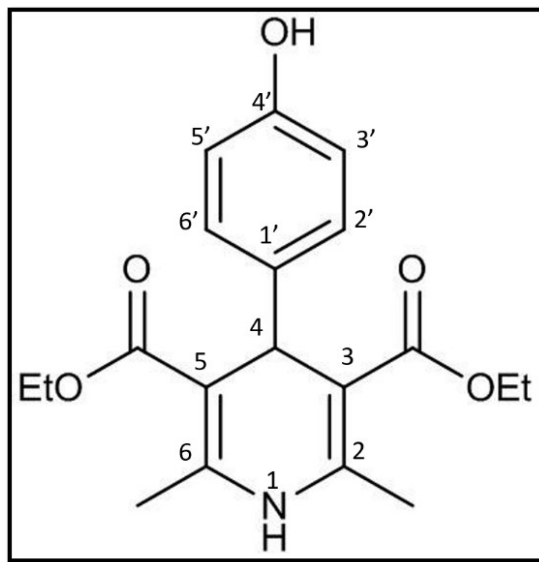
2, 6-Dimethyl-4-(2-nitrophenyl)-1, 4-dihydropyridine-3, 5-diethylcarboxylate [Compound (g) Entry 7, Table 6]



Yellow solid; FT-IR 3331, 2983, 1693, 1670, 1525, 1487, 1352, 1207, 756 cm^{-1} ; $^1\text{H-NMR}$ (CDCl_3) δ 1.16 (t, $J = 7.0$ Hz, 6H, 2, $\text{OCH}_2\text{-CH}_3$), 2.33 (s, 6H, 2, CH_3), 4.00 (q, $J = 7.0$ Hz, 2H, $\text{OCH}_2\text{-CH}_3$), 4.12 (q, $J = 7.0$ Hz, 2H, $\text{OCH}_2\text{-CH}_3$), 5.60 (s, 1H, CH), 5.85 (s, 1H, NH), 7.25 (dd, $J = 2.0$ and 8.0 Hz, 1H, H-6'), 7.46 (dt, $J = 2$ and 8.0 Hz, 1H, H-4'), 7.54 (dd, $J = 2$ and 8.0 Hz, 1H, 5'), 7.74 (dd, $J = 2$ and 8.0 Hz, 1H, H-3') ppm; $^{13}\text{C-NMR}$ (CDCl_3) δ 14.15 (2, $\text{OCH}_2\text{-CH}_3$), 19.73 (2, CH_3), 34.67 (C-4), 60.07 (2, $\text{OCH}_2\text{-CH}_3$), 104.08 (C-3 & C-5), 124.03 (C-3'), 126.95 (C-4'), 131.36 (C-6'), 132.77 (C-5'), 142.58 (C-1'), 144.37 (C-2 & C-6), 147.82 (C-2'), 167.22 (2, C=O) ppm; HRMS m/z Calcd. for $\text{C}_{19}\text{H}_{23}\text{N}_2\text{O}_6$ [$\text{M}+\text{H}$]: 375.1556, Found: 375.1550.

2,6-Dimethyl-4-(4-hydroxyphenyl)-1,4-dihydropyridine-3,5-diethylcarboxylate [Compound

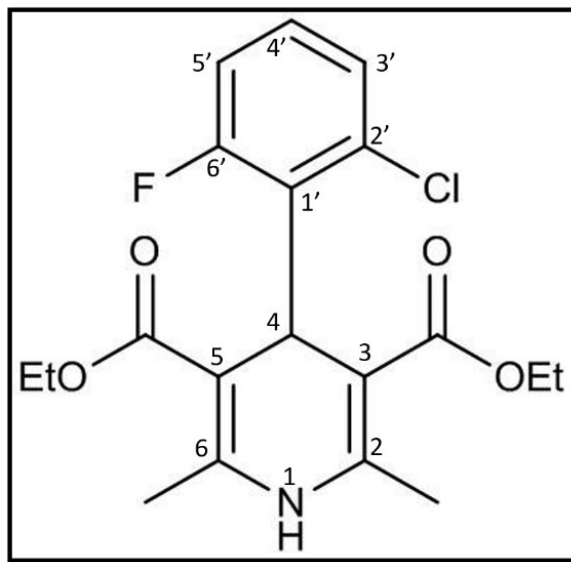
(h) Entry 8, Table 6]



Off-white solid; FT-IR 3342, 1656, 1633, 1487, 1222, 840 cm^{-1} ; $^1\text{H-NMR}$ (CDCl_3) δ 1.22 (t, $J = 7.0$ Hz, 6H, 2, $\text{OCH}_2\text{-CH}_3$), 2.32 (s, 6H, 2, CH_3), 4.09 (q, $J = 7.0$ Hz, 4H, 2, $\text{OCH}_2\text{-CH}_3$), 4.91 (s, 1H, CH), 5.51 (s, 1H, NH), 6.66 (d, $J = 8.0$ Hz, 2H, H-3' & H-5'), 7.13 (d, $J = 8.0$ Hz, 2H, H-2' & H-6') ppm; $^{13}\text{C-NMR}$ (CDCl_3) δ 14.28 (2, $\text{OCH}_2\text{-CH}_3$), 19.37 (2, CH_3), 38.79 (C-4), 59.74 (2, $\text{OCH}_2\text{-CH}_3$), 104.08 (C-3 & C-5), 114.03 (C-3' & C-5'), 129.23 (C-2' & C-6'), 137.36 (C-1'), 143.26 (C-2 & C-6), 155.50 (C-4'), 167.70 (2, C=O) ppm; HRMS m/z Calcd. for $\text{C}_{19}\text{H}_{24}\text{NO}_5$ [M+H]: 346.1654, Found: 346.1653.

2,6-Dimethyl-4-(2-chloro-6-fluorophenyl)-1,4-dihydropyridine-3,5-diethylcarboxylate

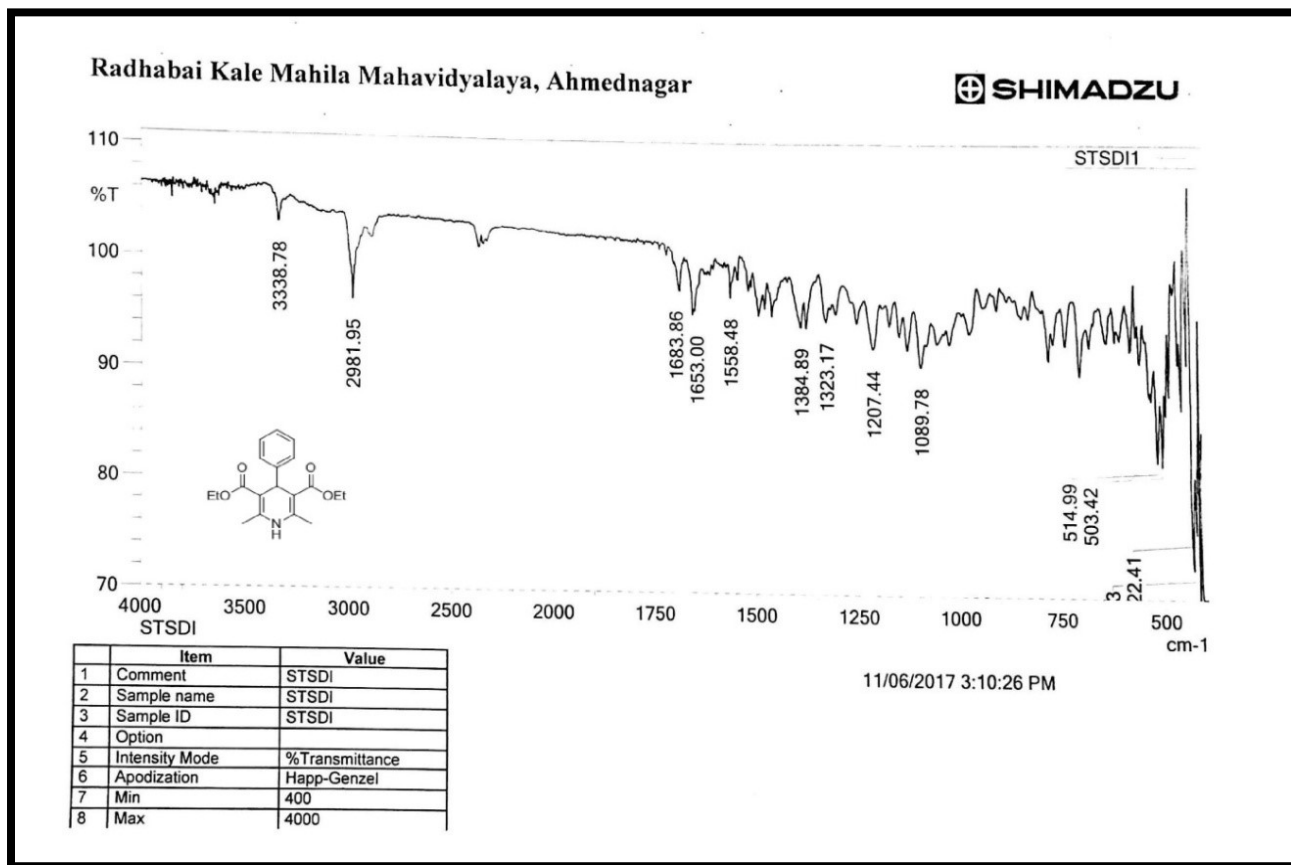
[Compound (i) Entry 9, Table 6]



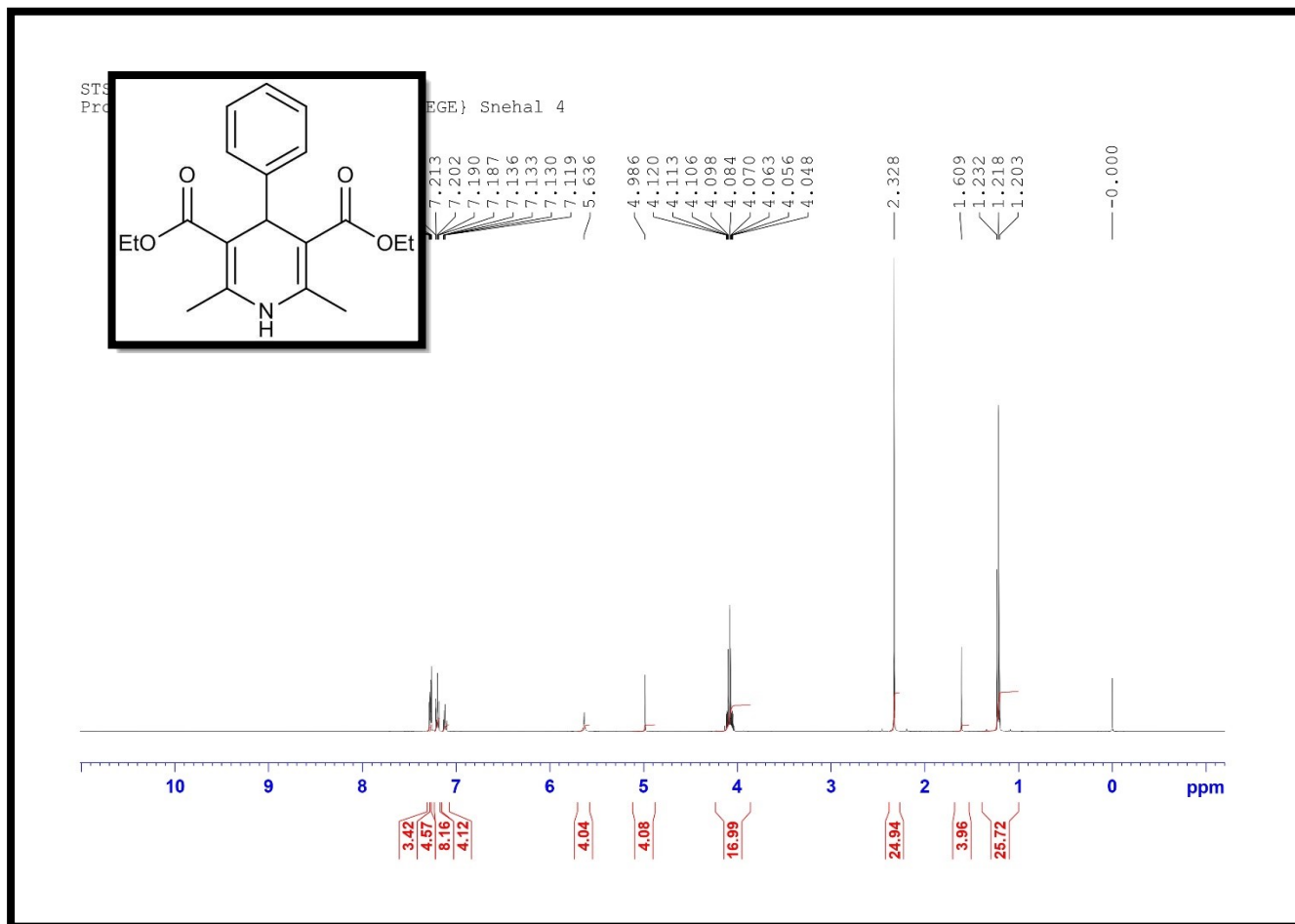
Pale yellow solid; FT-IR 3330, 2984, 1691, 1673, 1487, 1222, 892, 787 cm^{-1} ; $^1\text{H-NMR}$ (CDCl_3) δ 1.15 (t, $J = 7.0$ Hz, 6H, 2, $\text{OCH}_2\text{-CH}_3$), 2.27 (s, 6H, 2, CH_3), 4.04 (q, $J = 7.0$ Hz, 4H, 2, $\text{OCH}_2\text{-CH}_3$), 5.66 (s, 1H, CH), 5.76 (s, 1H, NH), 6.87 (dt, $J = 9.5$ Hz & 1.15 Hz, 1H, H-4'), 7.03 (m, 1H, H-5'), 7.09 (d, $J = 8.0$ Hz, 1H, H-3') ppm; $^{13}\text{C-NMR}$ (CDCl_3) δ 14.12 (2, $\text{OCH}_2\text{-CH}_3$), 19.60 (2, CH_3), 33.68 (C-4), 59.63 (2, $\text{OCH}_2\text{-CH}_3$), 100.12 (C-3 & C-5), 113.96 (d, $J = 31.3$ Hz, C-5'), 125.77 (d, $J = 3.7$ Hz, C-3'), 127.47 (d, $J = 12.5$ Hz, C-4'), 131.57 (d, $J = 18.7$ Hz, C-1'), 135.54 (d, $J = 7.5$ Hz, C-2'), 145.34 (C-2 & C-6), 162.60 (d, $J = 310.0$ Hz, C-6'), 167.64 (2, C=O) ppm; HRMS m/z Calcd. for $\text{C}_{19}\text{H}_{22}\text{NO}_5\text{ClF}$ [$\text{M}+\text{H}$]: 382.1221, Found: 382.1217.

II. FT-IR, ¹H-NMR, D₂O exchange ¹H-NMR, ¹³C NMR and HRMS spectra of
1, 4 dihydropyridines derivatives (Table 6, Entries 1 to 9)

FTIR (Table 6, Entry 1)

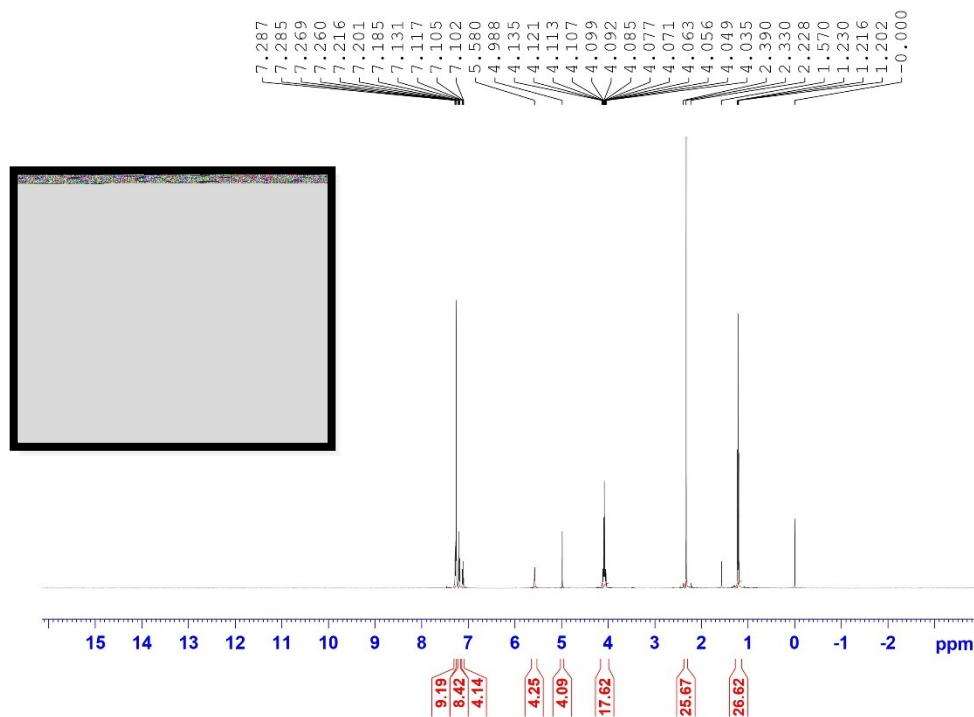


¹H-NMR spectra (Table 6, Entry 1)



¹H-NMR spectra without D₂O exchange (Table 6, Entry 1)

STSD-I
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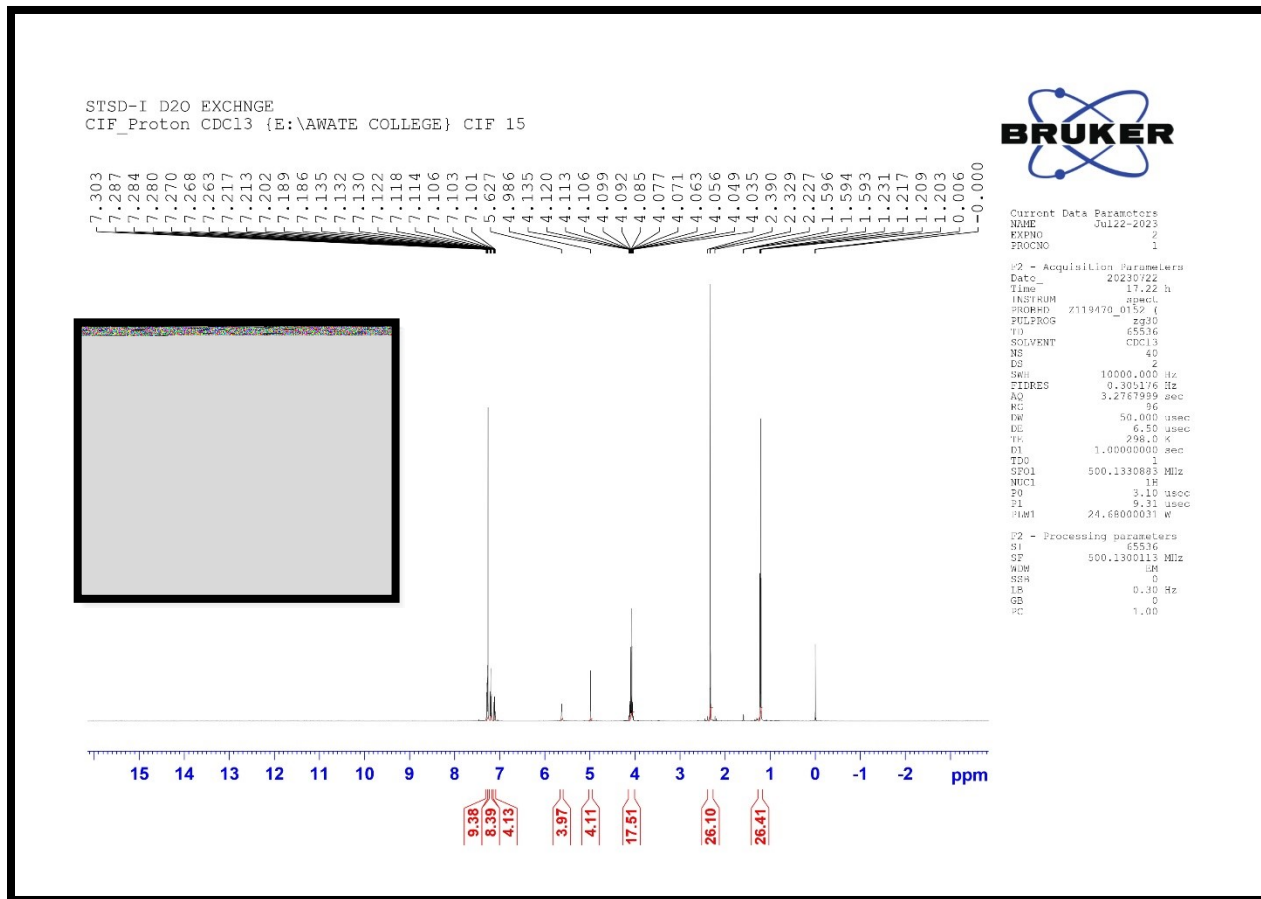


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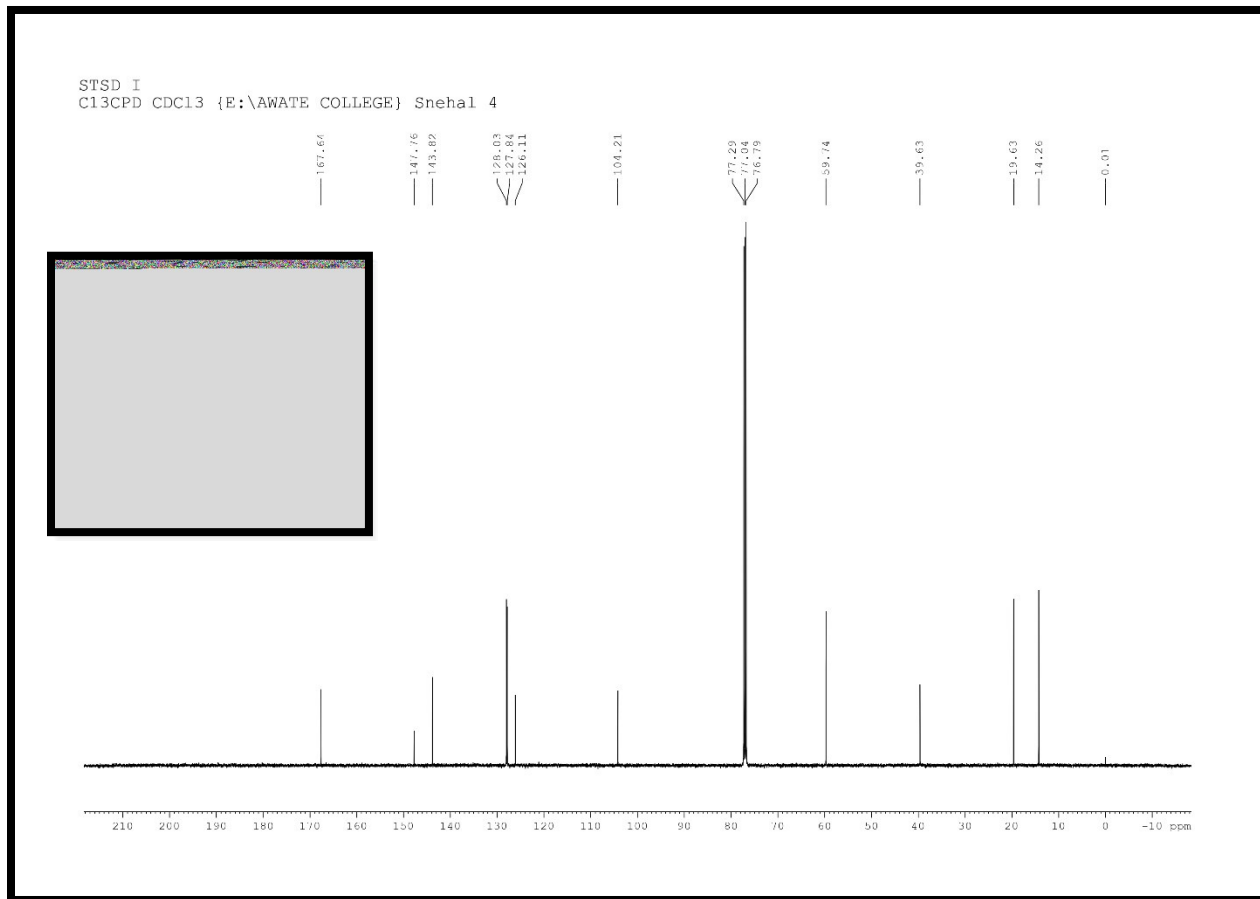
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¹H-NMR spectra with D₂O exchange (Table 6, Entry 1)



¹³C NMR spectra (Table 6, Entry 1)

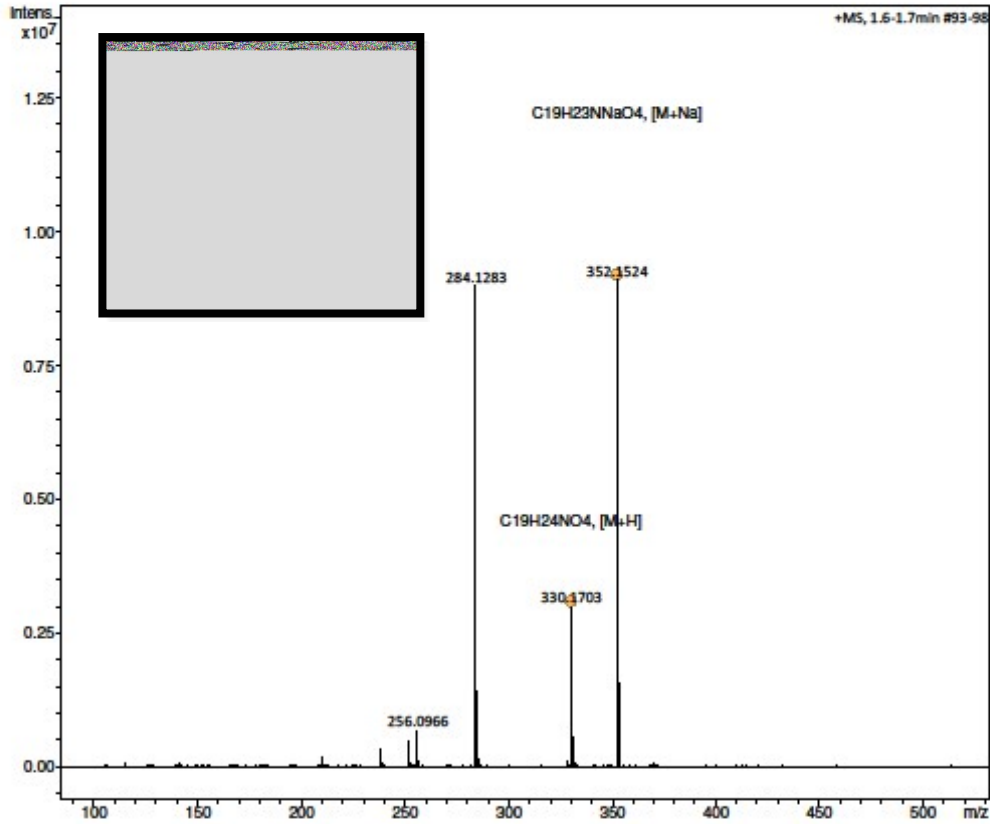


HRMS (Table 6, Entry 1)

Savitribai Phule Pune University - Central Instrumentation Facility

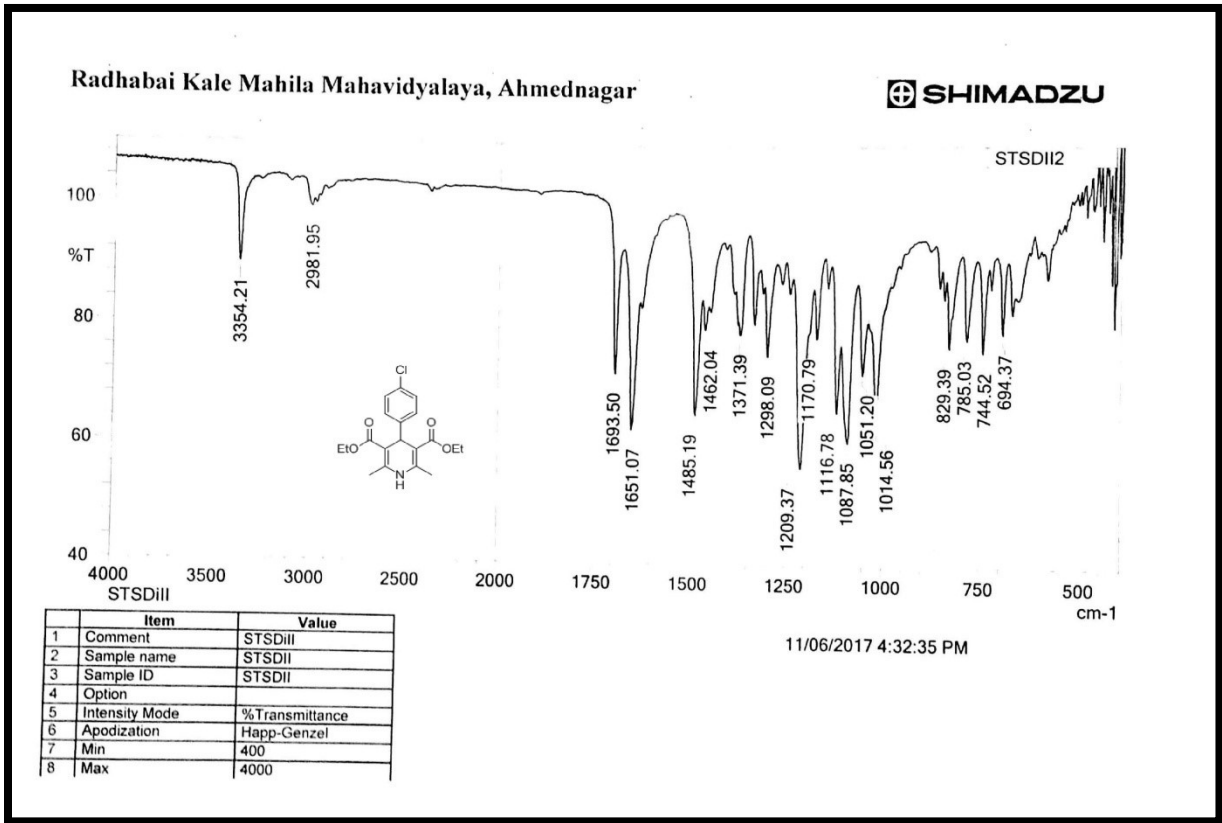
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Method	dlc-ms600mz_10min.m	Operator	CIF
Sample Name	STSD-I	Instrument	impact HD 1819696.00184
Comment			

Acquisition Parameter					
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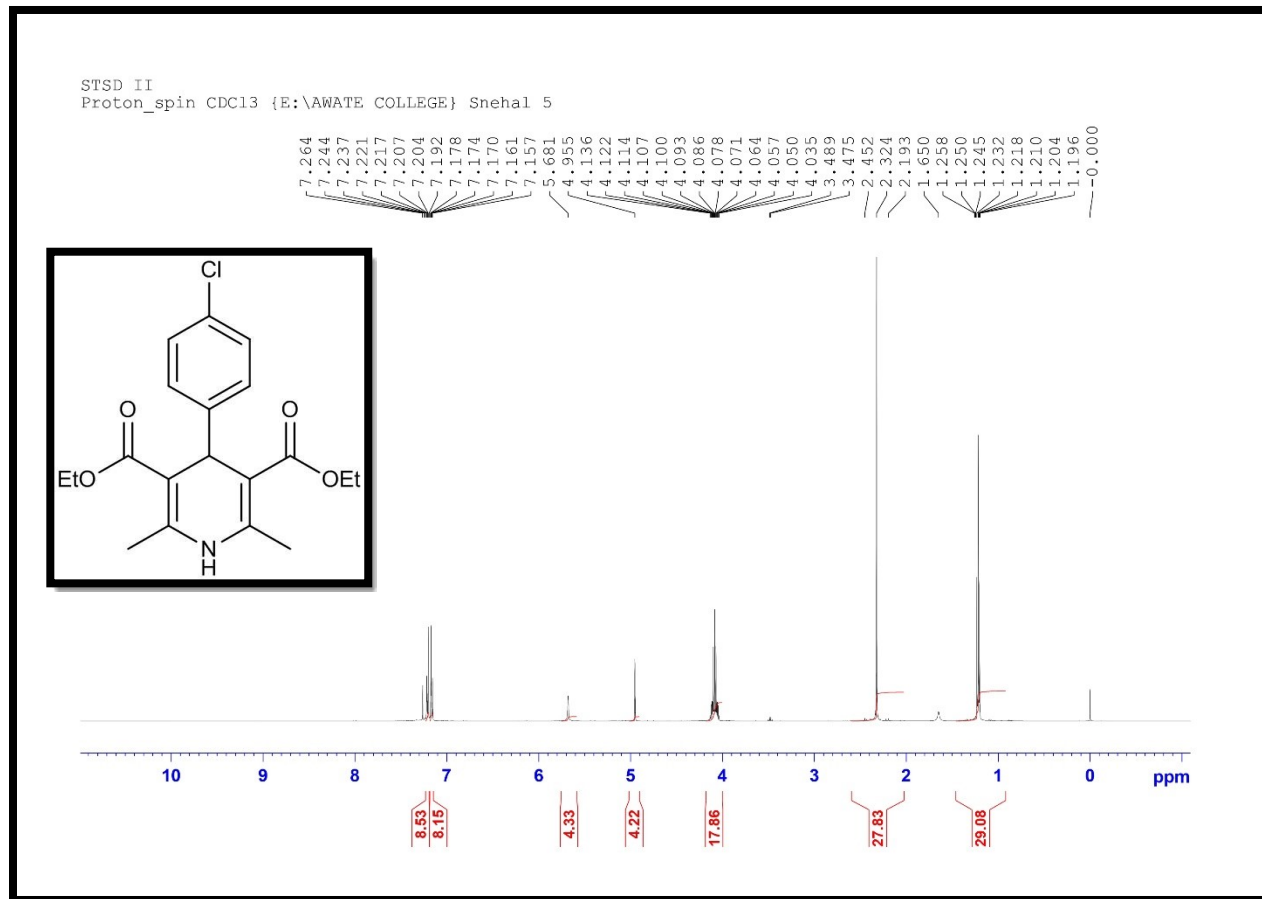


Meas. m/z	#	Ion Formula	m/z	err [ppm]	mSigma	# mSigma	Score	rdB	e ⁻	Conf	N-Rule	Adduct
330.1703	1	C19H24NO4	330.1700	-1.0	17.9	1	100.00	8.5	even		ok	M+H
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FTIR (Table 6, Entry 2)

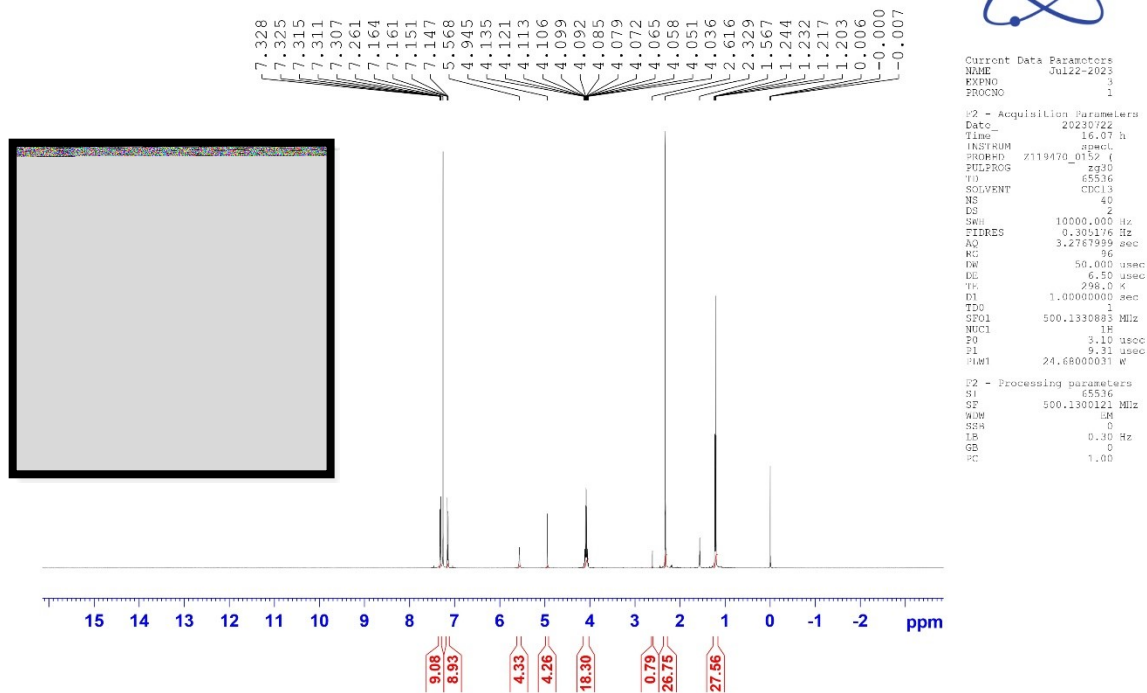


¹H-NMR spectra (Table 6, Entry 2)

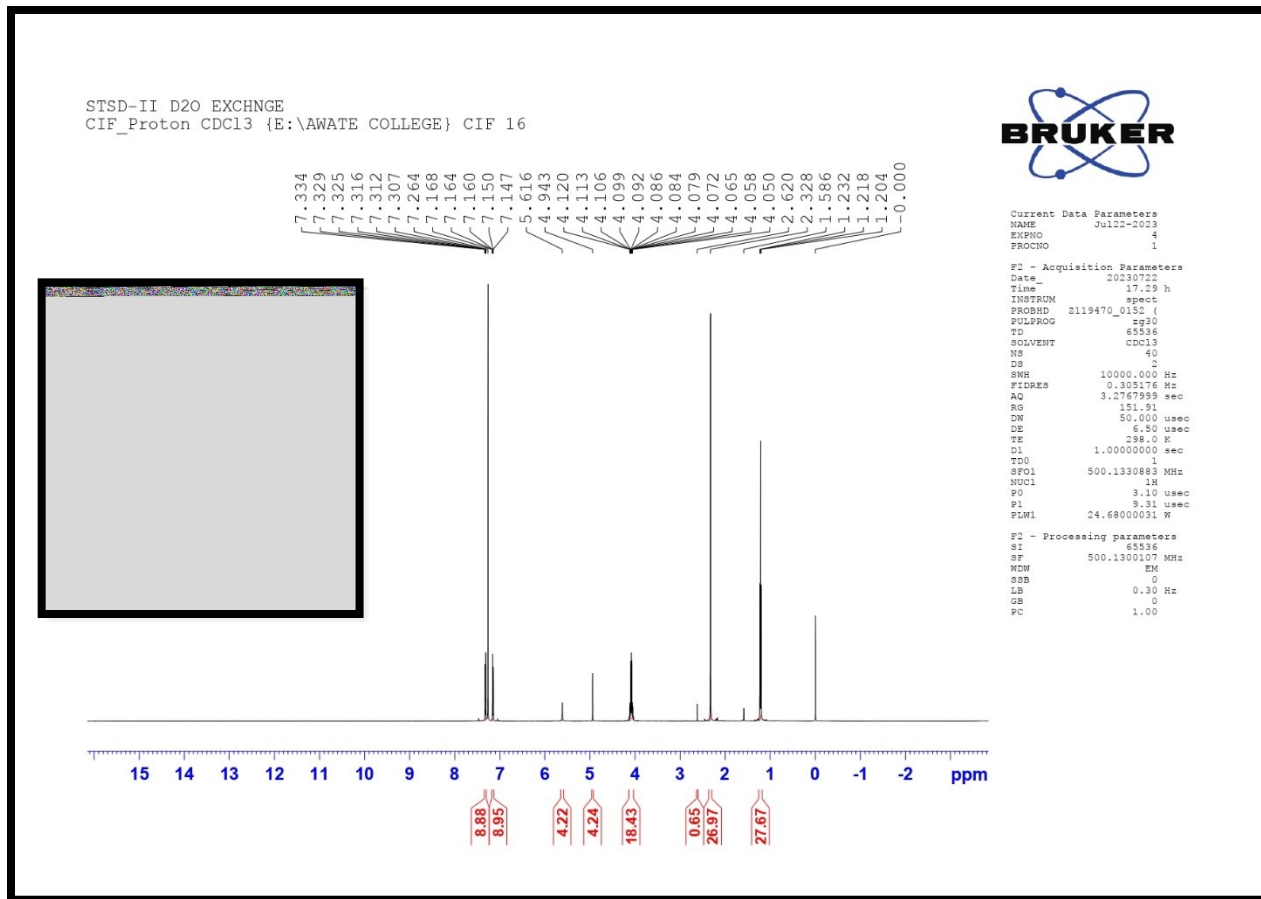


¹H-NMR spectra without D₂O exchange (Table 6, Entry 2)

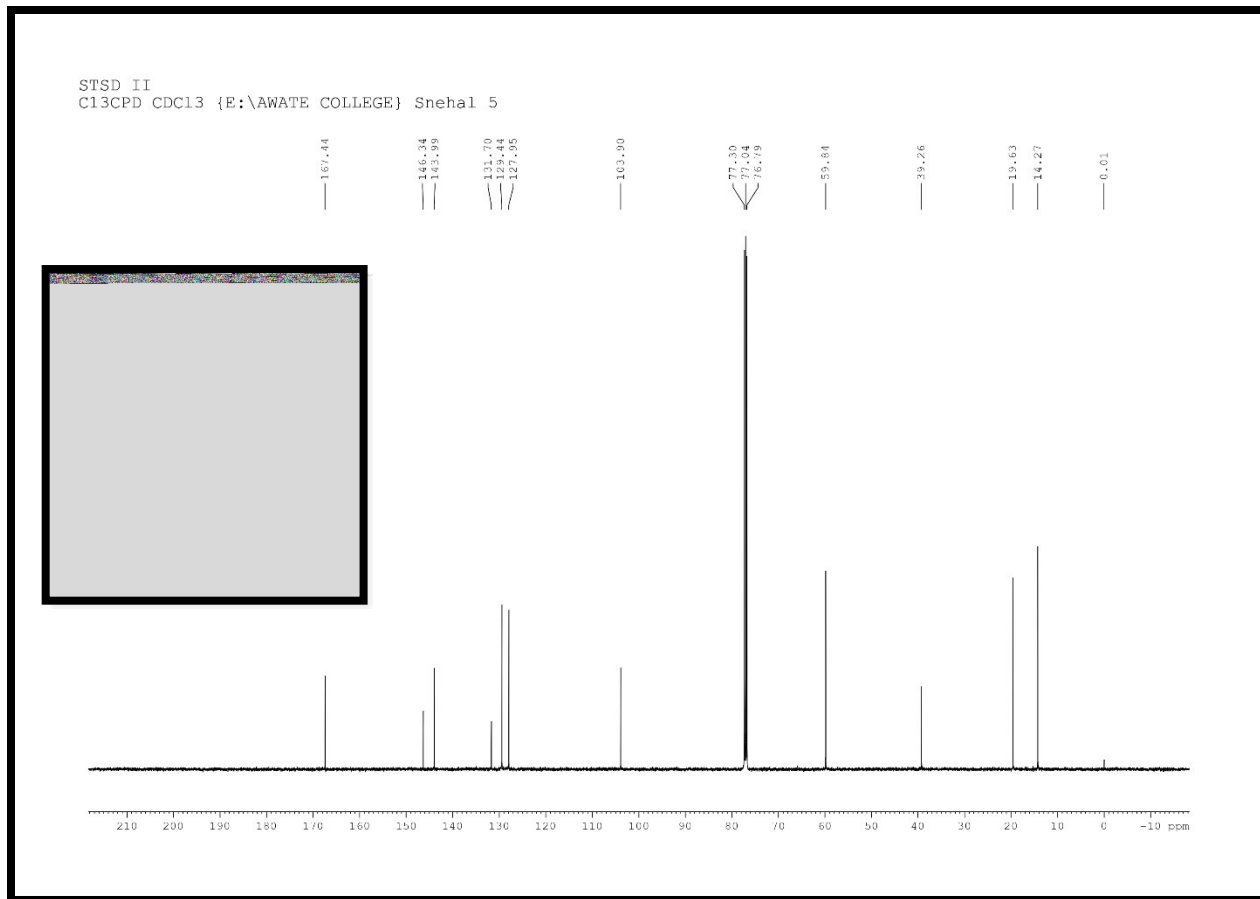
STSD-II
CIF_Proton CDC13 {E:\AWATE COLLEGE} CIF 16



¹H-NMR spectra with D₂O exchange (Table 6, Entry 2)



¹³C-NMR spectra (Table 6, Entry 2)

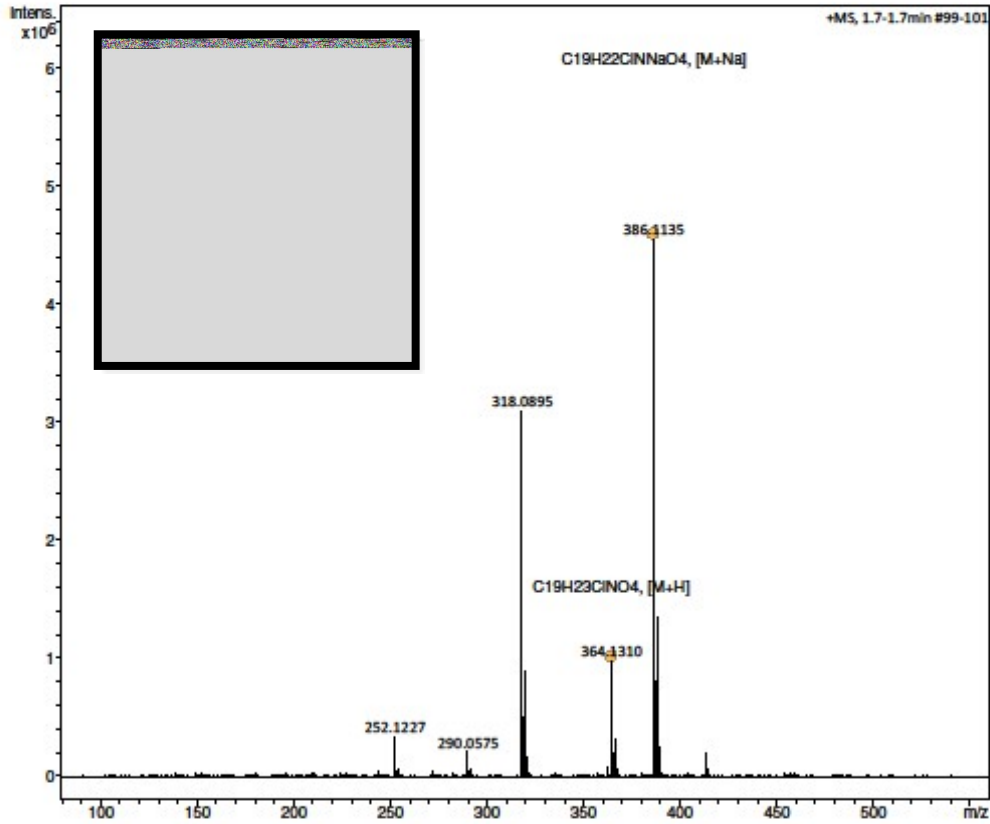


HRMS (Table 6, Entry 2)

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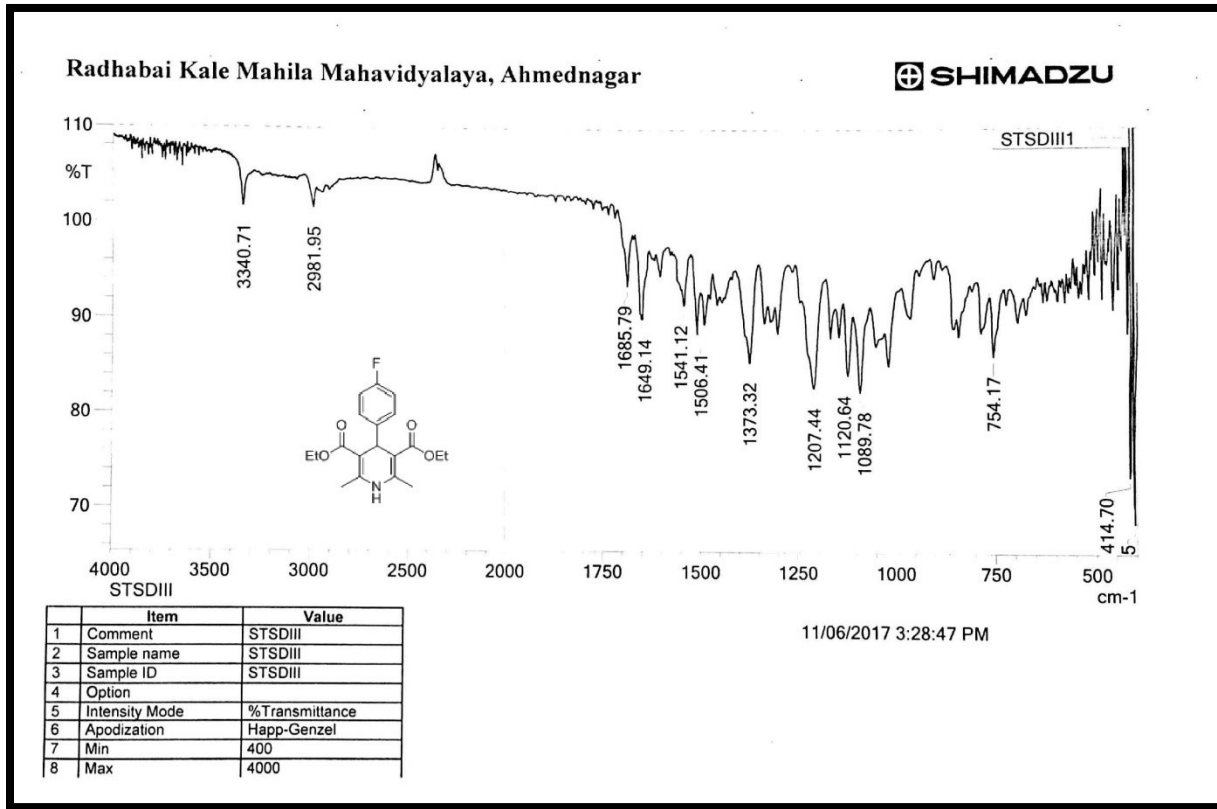
Analysis Info		Acquisition Date 7/5/2017 7:23:41 PM	
Analysis Name	D:\Data\2017\JUNE\SPPU AFFILIATED\VA AWATE COLLEGE, MANCHAR\KG KANADE\DB BANKAR\STSD-II_RB1_01_3975.d		
Method	dlc-ms600mz_10min.m	Operator	CIF
Sample Name	STSD-II	Instrument	impact HD 1819696.00184
Comment			

Acquisition Parameter					
Source Type	ESI	Ion Polarity	Positive	Set Nebulzer	0.3 Bar
Focus	Active	Set Capillary	3500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	600 m/z	Set Charging Voltage	2000 V	Set Divert Valve	Waste
		Set Corona	0 nA	Set APCI Heater	0 °C



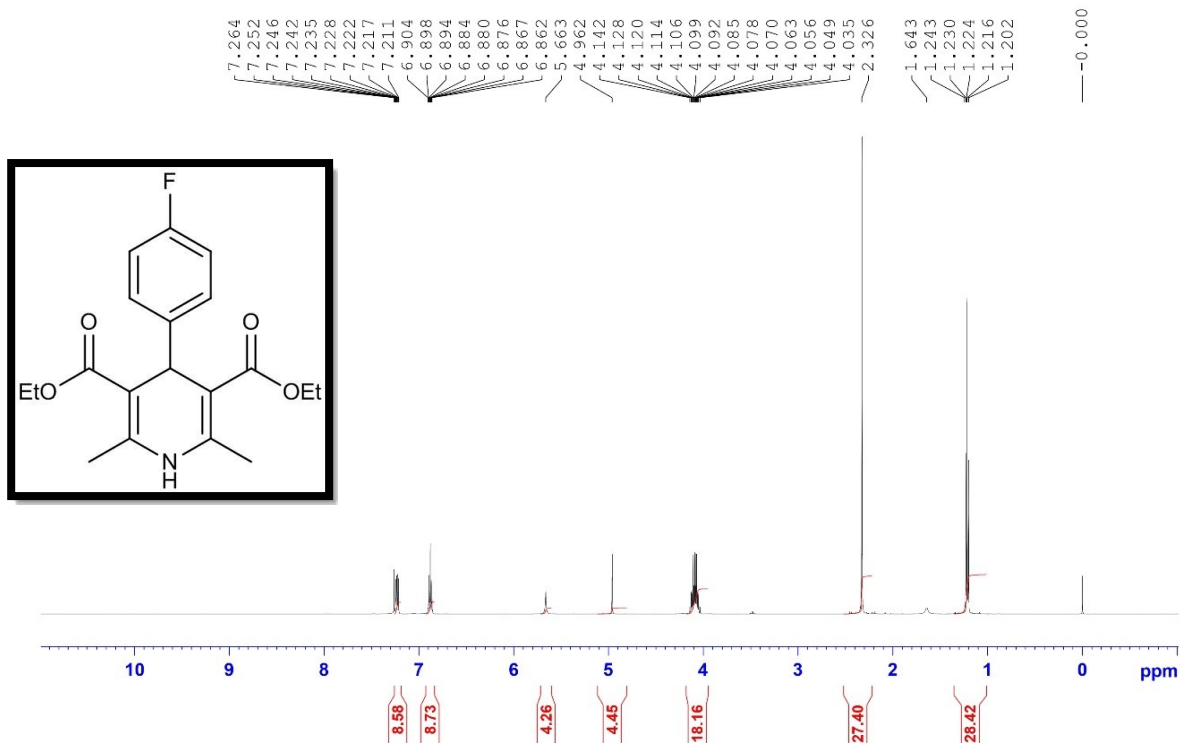
Meas. m/z	#	Ion Formula	m/z	err [ppm]	mSigma	# mSigma	Score	rdb	e ⁻	Conf	N-Rule	Adduct
364.1310	1	C19H23ClNO4	364.1310	-0.1	12.0	1	100.00	8.5	even		ok	M+H
386.1135	1	C19H22CINNaO4	386.1130	-1.5	30.1	1	100.00	8.5	even		ok	M+Na

FTIR (Table 6, Entry 3)

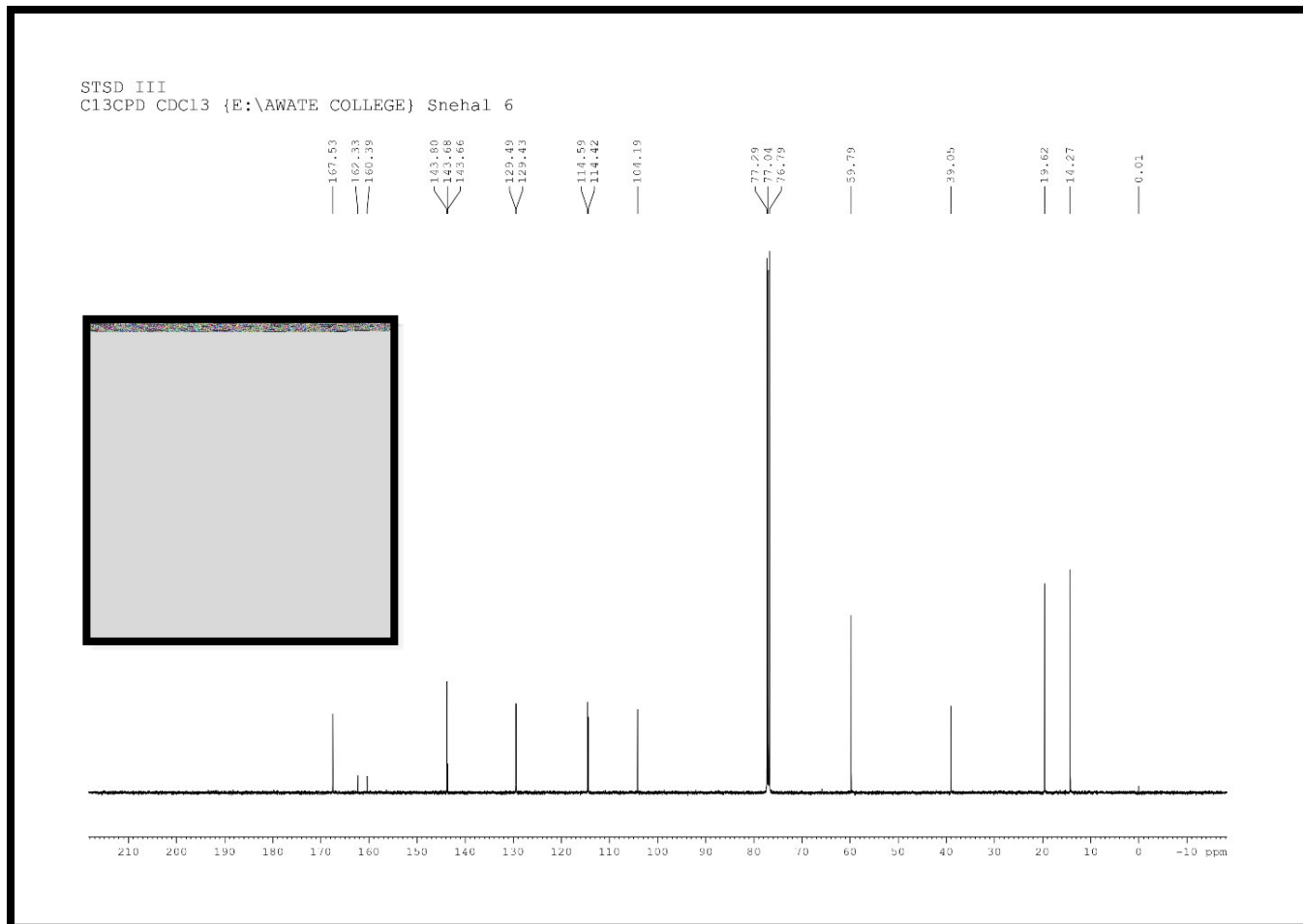


¹H-NMR spectra (Table 6, Entry 3) at 500 MHz

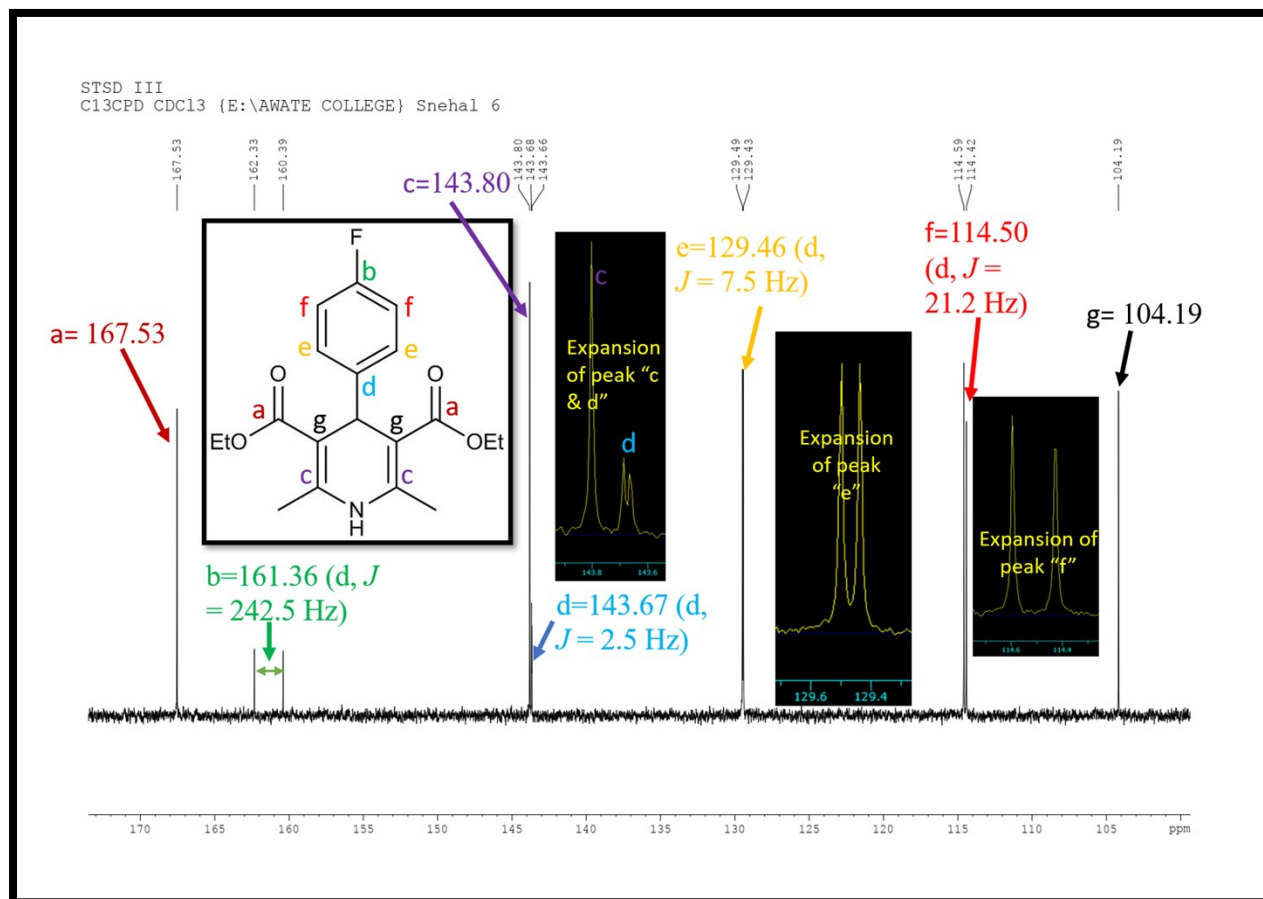
STSD III
Proton_spin CDC13 {E:\AWATE COLLEGE} Snehal 6



¹³C-NMR spectra (Table 6, Entry 3)



Expansion image of ^{13}C -NMR (Table 6, Entry 3)

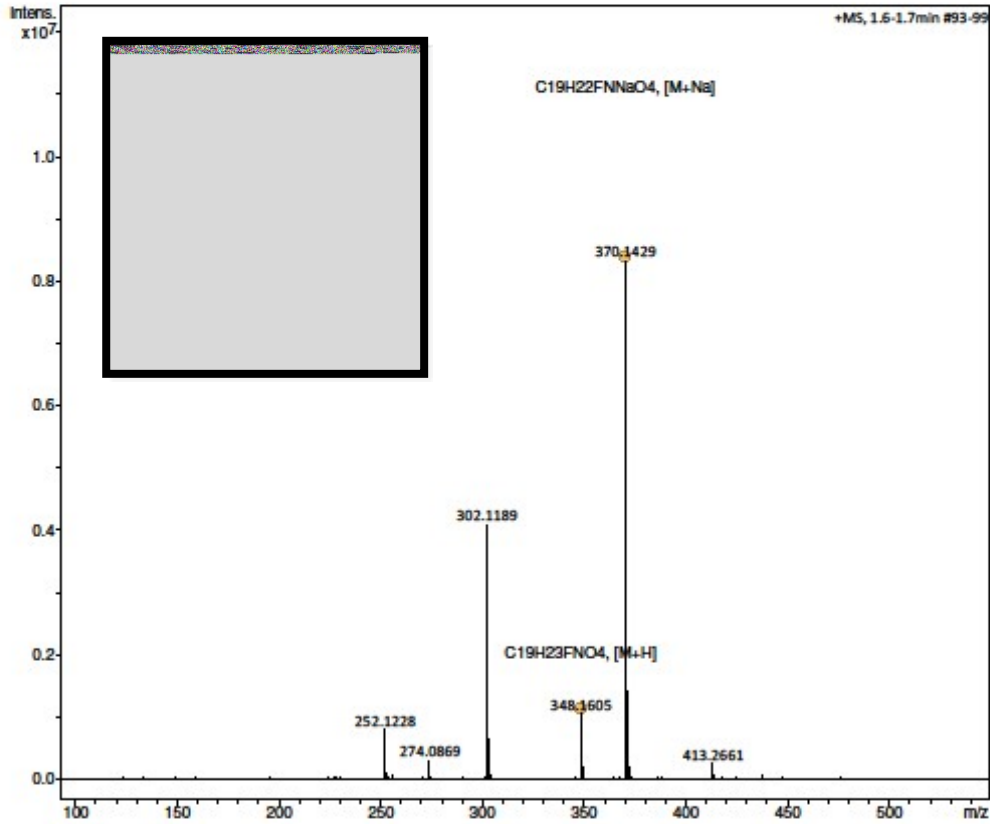


HRMS (Table 6, Entry 3)

Savitribai Phule Pune University - Central Instrumentation Facility

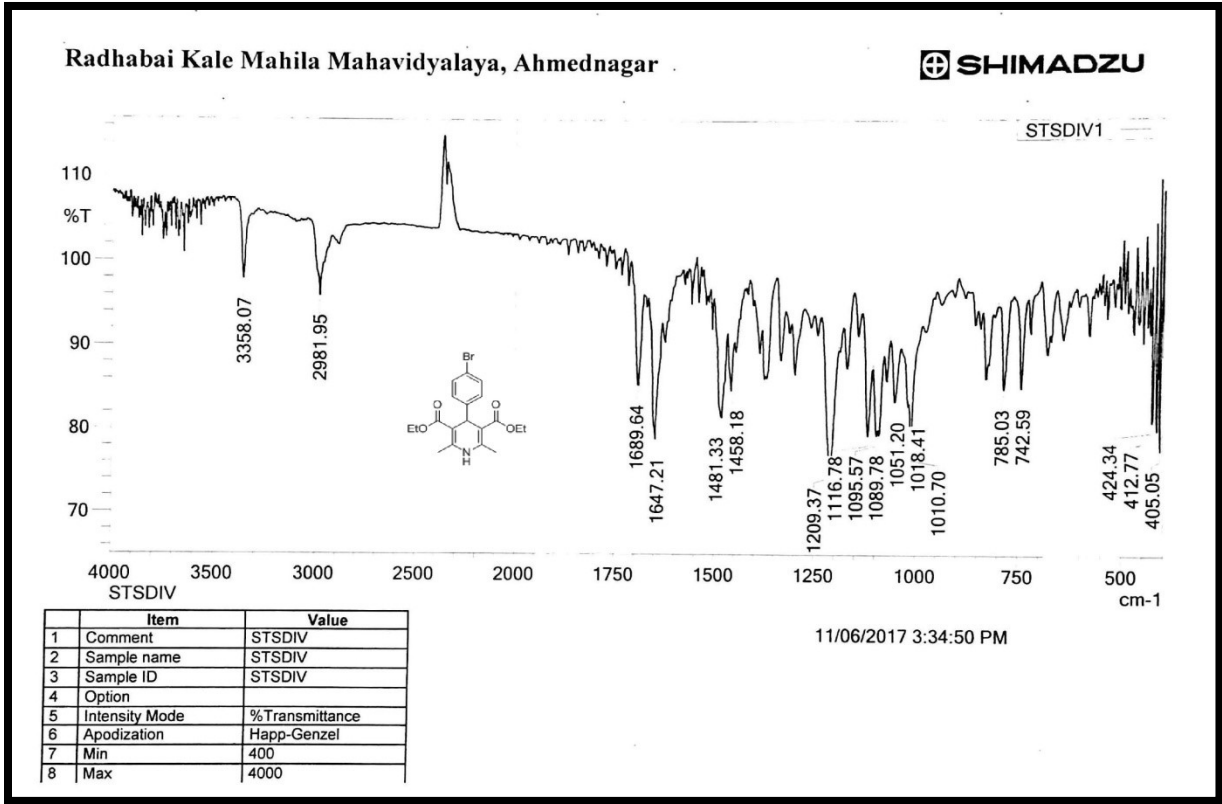
Analysis Info		Acquisition Date 7/5/2017 7:37:10 PM	
Analysis Name	D:\Data\2017\JUNE\SPPU AFFILIATED\A AWATE COLLEGE, MANCHAR\KG KANADE\DB BANKAR\STSD-III_RB2_01_3976.d		
Method	dlc-ms600mz_10min.m	Operator	CIF
Sample Name	STSD-III	Instrument	impact HD 1819696.00184
Comment			

Acquisition Parameter					
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.3 Bar
Focus	Active	Set Capillary	3500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	600 m/z	Set Charging Voltage	2000 V	Set Divert Valve	Waste
		Set Corona	0 nA	Set APCI Heater	0 °C

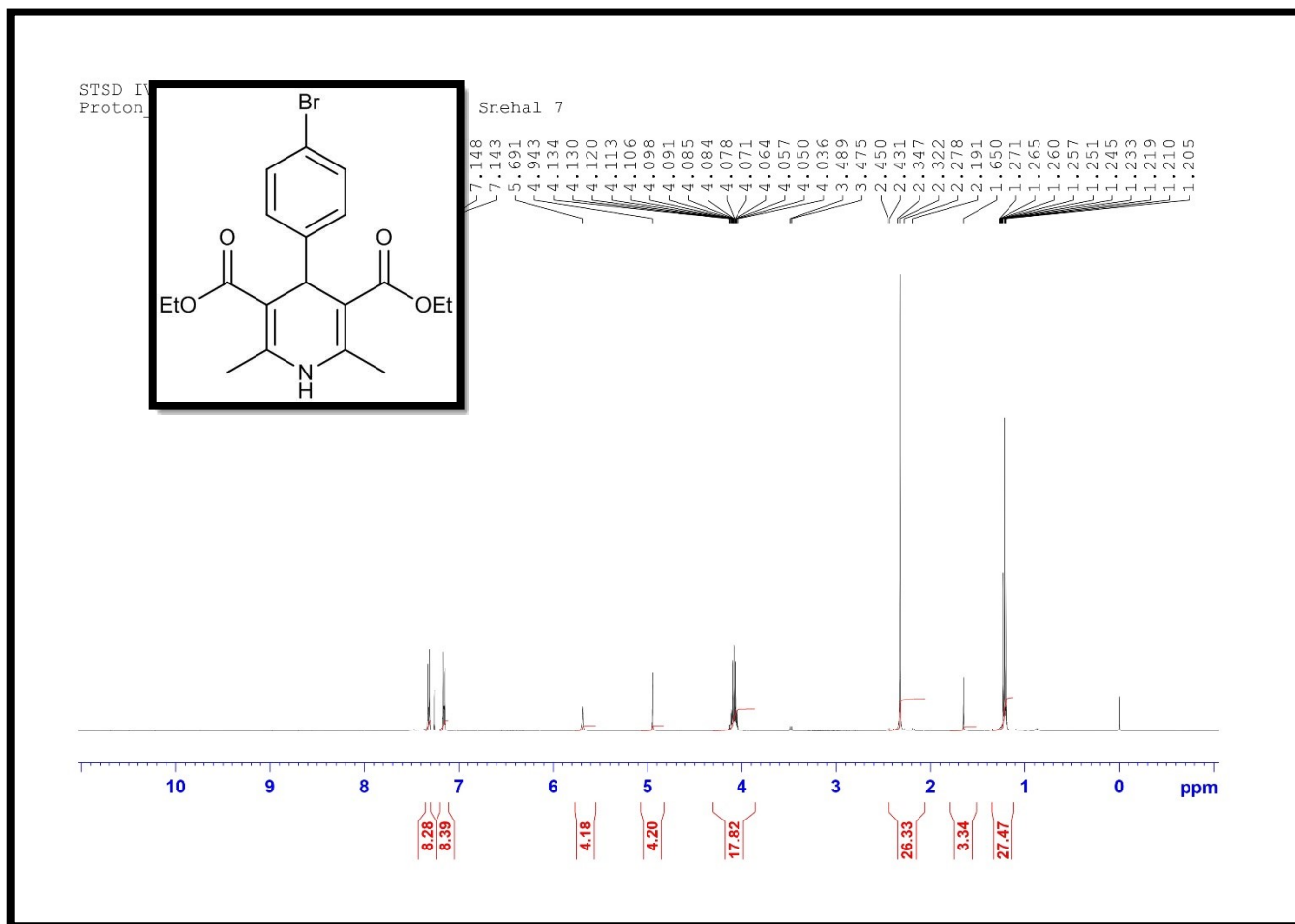


Meas. m/z	#	Ion Formula	m/z	err [ppm]	mSigma	# mSigma	Score	rdb	e ⁻	Conf	N-Rule	Adduct
348.1605	1	C19H23FO4	348.1606	0.0	12.2	1	100.00	8.5	even		ok	M+H
370.1429	1	C19H22FNNaO4	370.1425	-1.2	25.0	1	100.00	8.5	even		ok	M+Na

FTIR (Table 6, Entry 4)

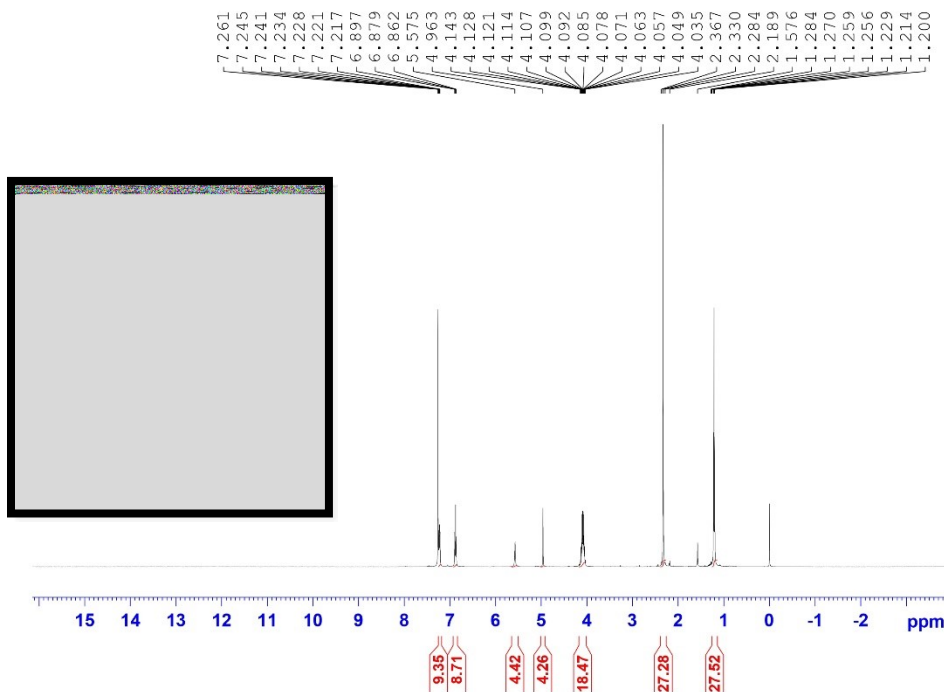


¹H-NMR spectra (Table 6, Entry 4)



¹H-NMR spectra without D₂O exchange (Table 6, Entry 4)

STSD-IV
CIF_Proton CDCl₃ (E:\AWATE COLLEGE) CIF 17

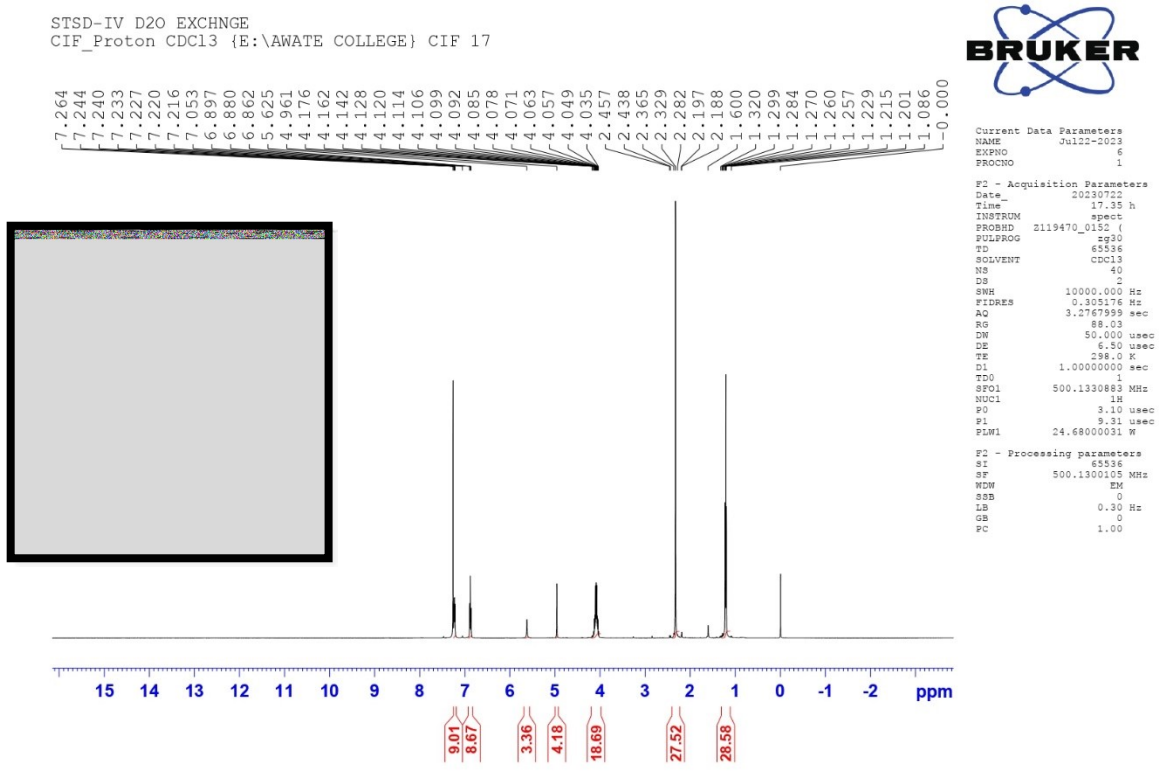


Current Data Parameters
NAME Jul22-2023
EXNO 5
PROCNO 1

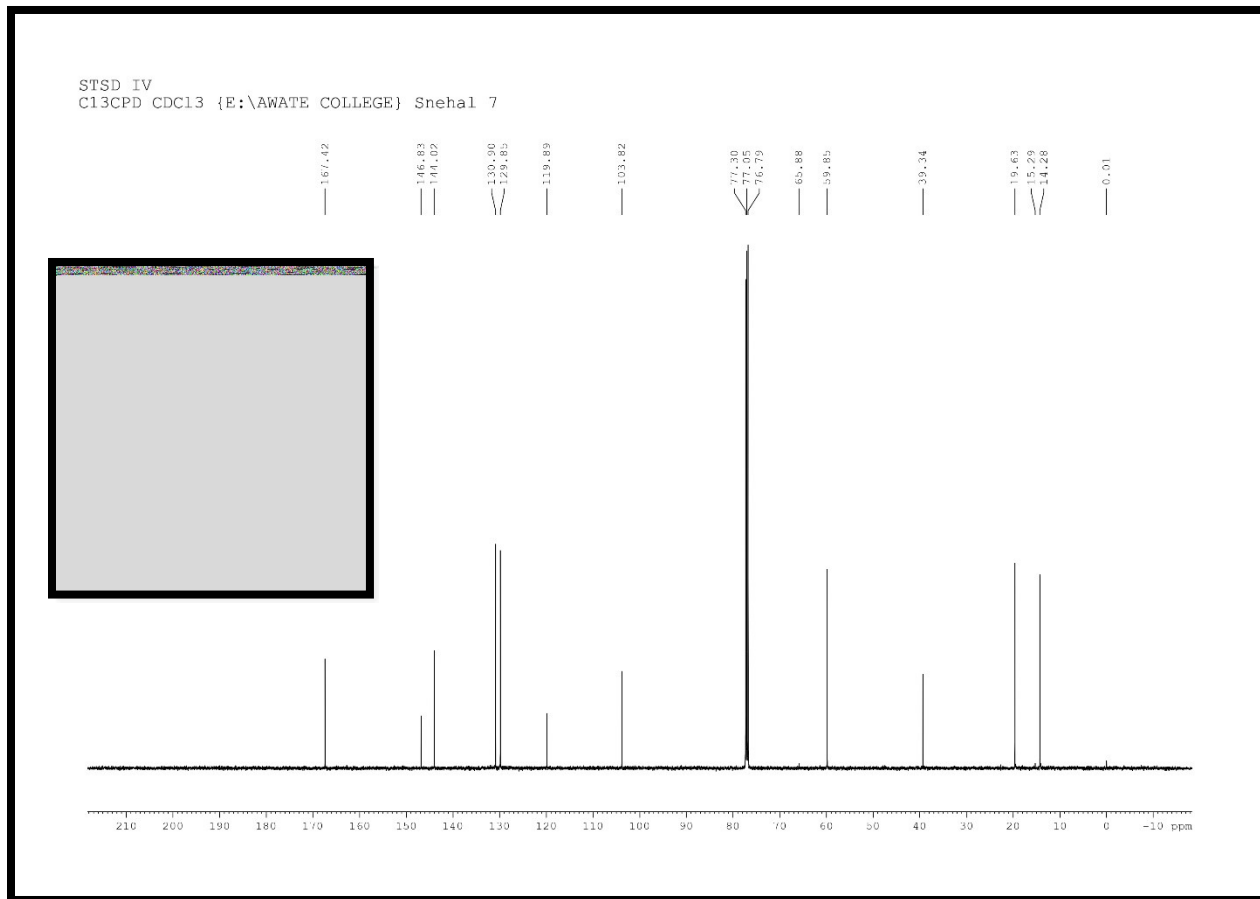
F2 - Acquisition Parameters
Date 20230722
Time 16.15 h
INSTRUM spect
PROBHD Z119470.0152 (
PULPROG zg30
TD 65536
SOLVENT CDCl₃
NS 40
DS 2
SMU 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767399 sec
RG 96
ZM 50.000 usoc
DE 6.50 usoc
TE 298.0 K
D1 1.00000000 sec
TDO 1
SFO1 500.1330863 MHz
RUC1 1H
PD 3.10 usoc
P1 9.31 usoc
PLW1 24.68000031 W

F2 - Processing parameters
SI 65536
SF 500.1300119 MHz
NUC 1H
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹H-NMR spectra with D₂O exchange (Table 6, Entry 4)



¹³C-NMR spectra (Table 6, Entry 4)

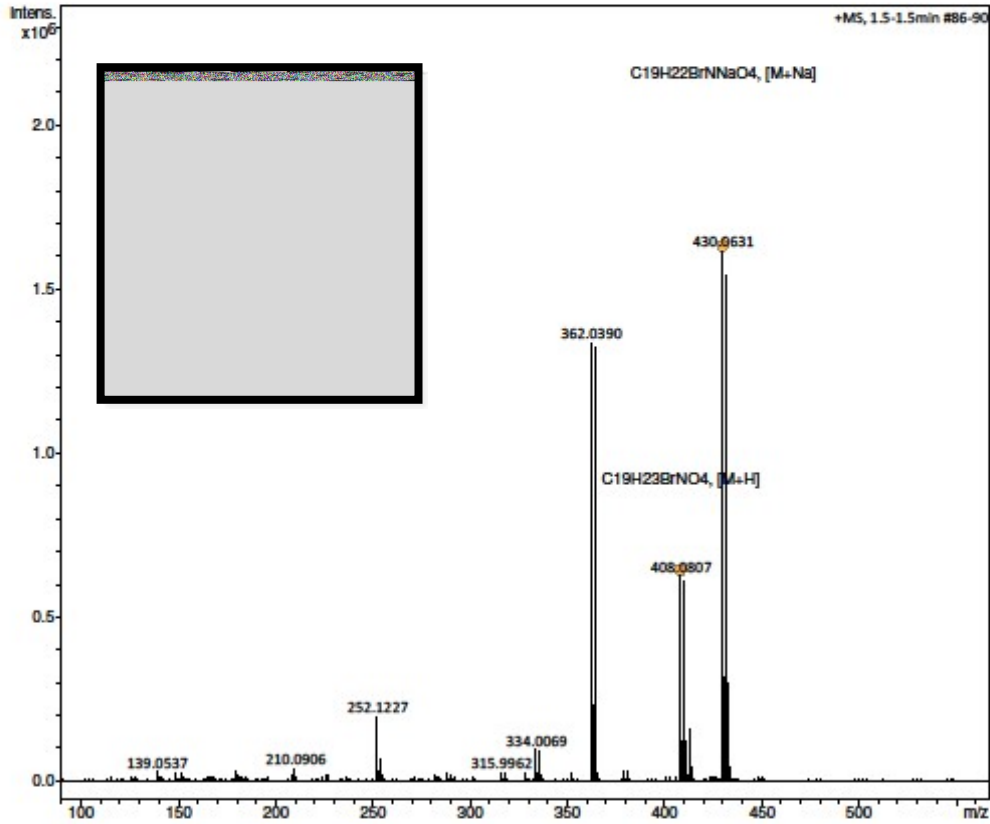


HRMS (Table 6, Entry 4)

Savitribai Phule Pune University - Central Instrumentation Facility

Analysis Info		Acquisition Date 7/5/2017 7:47:51 PM	
Analysis Name	D:\Data\2017\JUNE\SPPU AFFILIATED\A AWATE COLLEGE, MANCHAR\KG KANADE\DB BANKAR\STSD-IV_RB3_01_3977.d		
Method	dlc-ms600mz_10min.m	Operator	CIF
Sample Name	STSD-IV	Instrument	impact HD 1819696.00184
Comment			

Acquisition Parameter					
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.3 Bar
Focus	Active	Set Capillary	3500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	600 m/z	Set Charging Voltage	2000 V	Set Divert Valve	Waste
		Set Corona	0 nA	Set APCI Heater	0 °C



Meas. m/z	#	Ion Formula	m/z	err [ppm]	mSigma	# mSigma	Score	rdb	e ⁻	Conf	N-Rule	Adduct
408.0807	1	C19H23BrNO4	408.0805	-0.5	14.7	1	100.00	8.5	even		ok	M+H
430.0631	1	C19H22BrNNO4	430.0624	-1.6	24.2	1	100.00	8.5	even		ok	M+Na

STSD-IV_RB3_01_3977.d

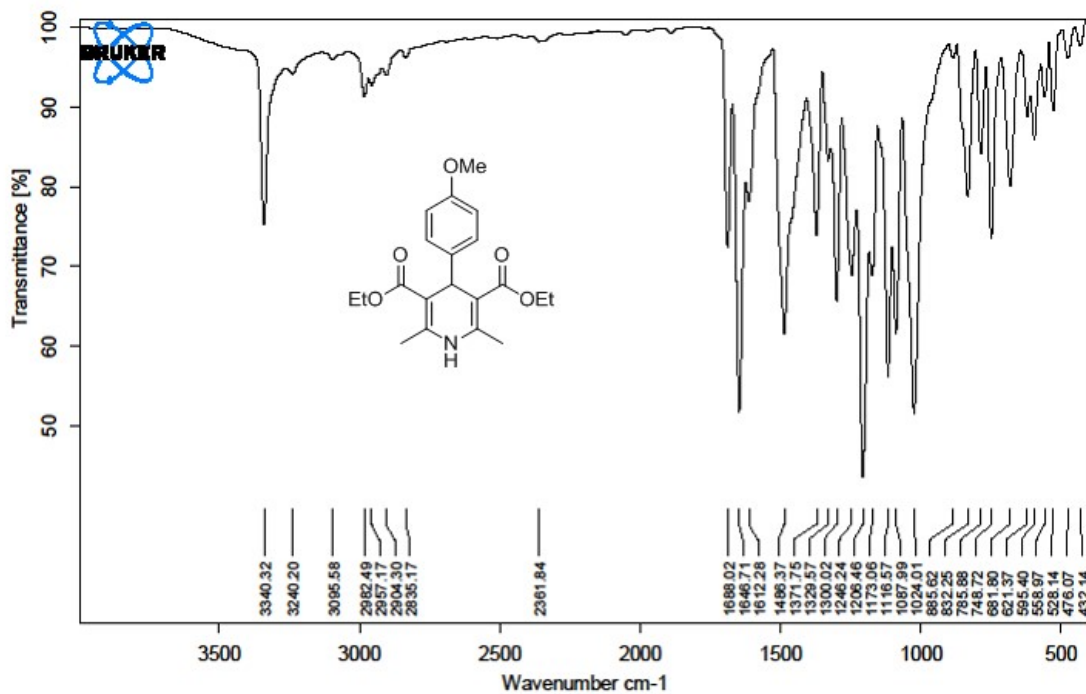
Bruker Compass DataAnalysis 4.2

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by: CIF

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FTIR (Table 6, Entry 5)



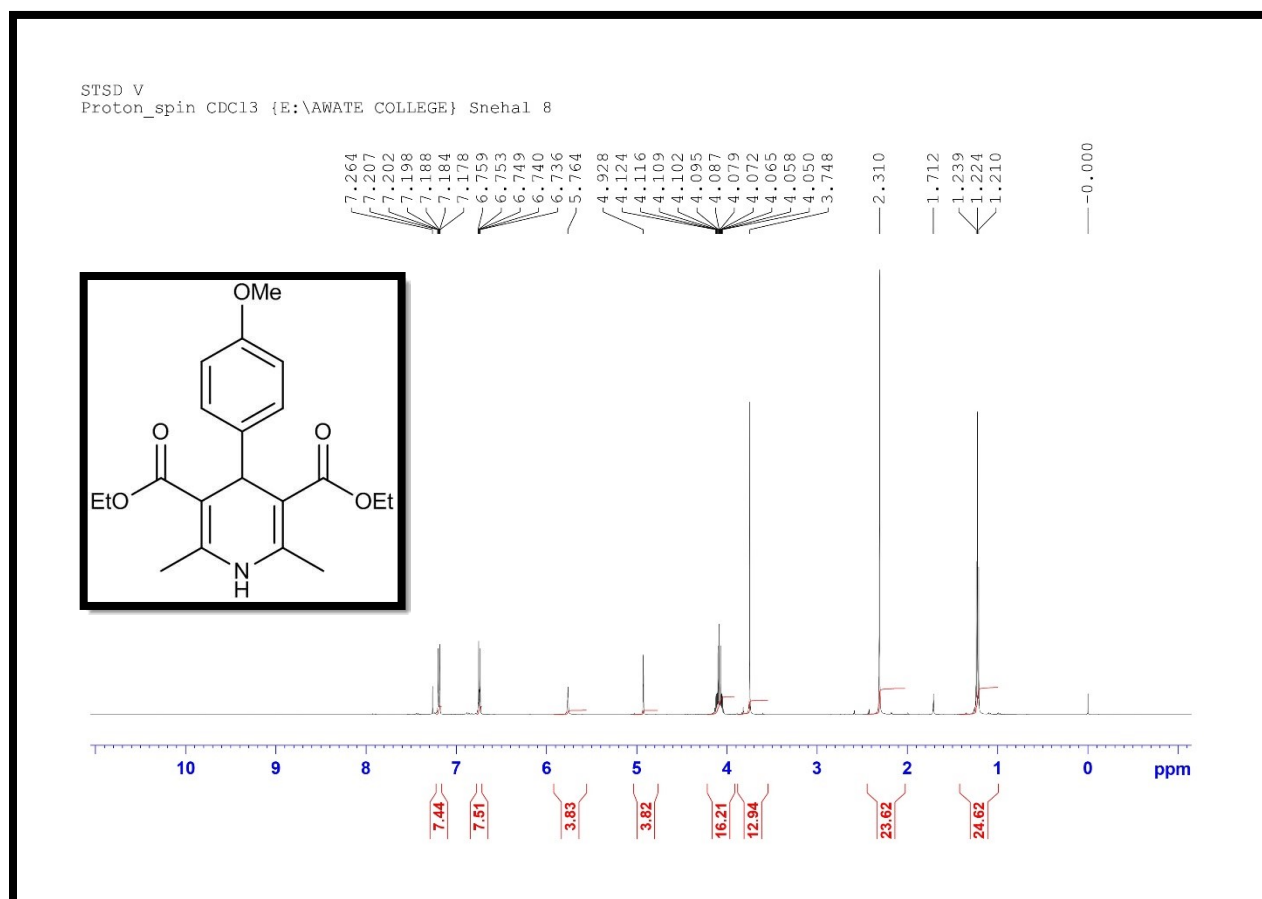
D:\OPUS\FTIR DATA 2017\JUNE\14 JUNE 2017\AAC.MANCHAR\STS-DV.0

STS-DV

SOLID

14/08/2017

¹H-NMR spectra (Table 6, Entry 5)



¹³C-NMR spectra (Table 6, Entry 5)

STSD V
C13CPD CDC13 {E:



114.29
113.31
113.19

104.34

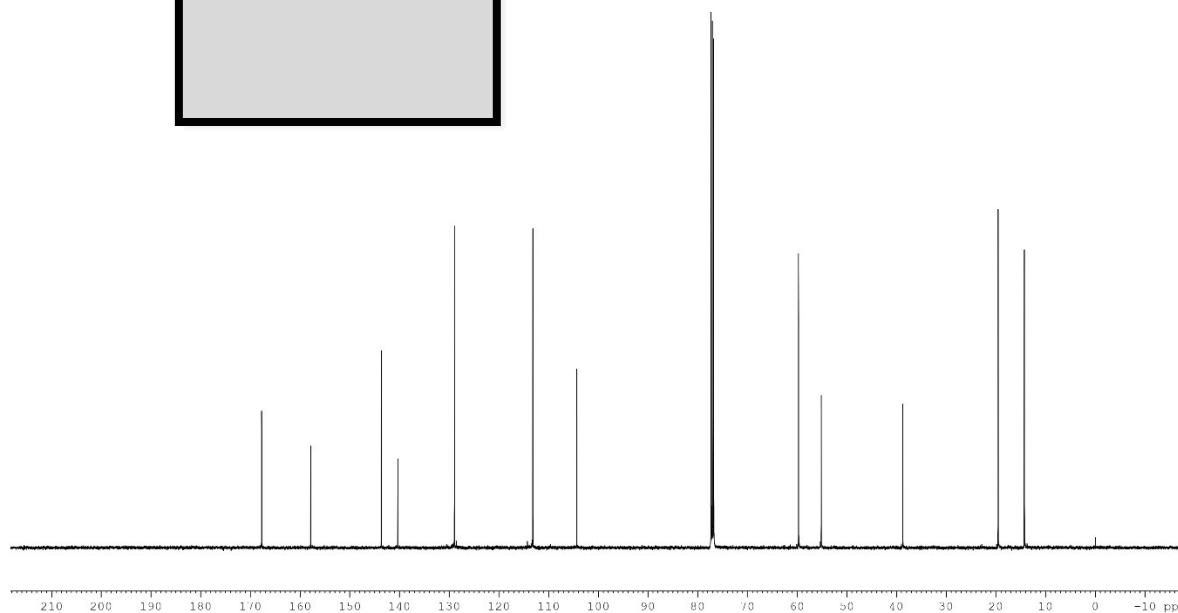
77.32
77.06
76.81

59.72
55.35
55.15

39.64
38.74

19.36
14.29
13.75

0.01

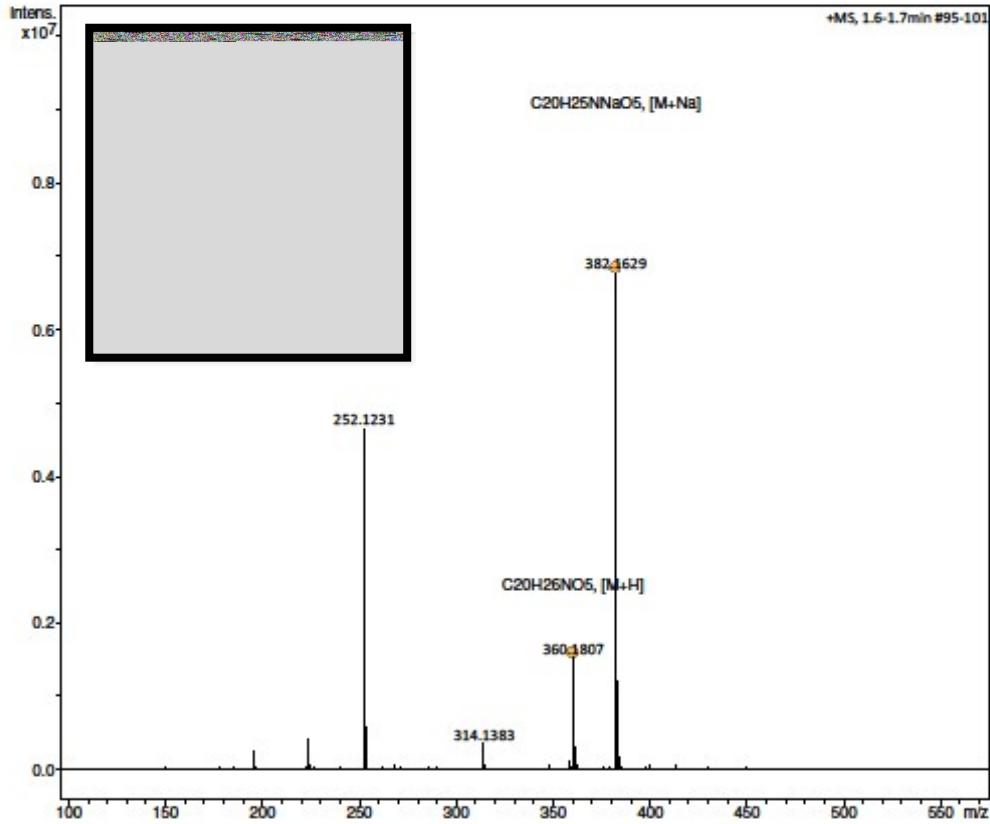


HRMS (Table 6, Entry 5)

Savitribai Phule Pune University - Central Instrumentation Facility

Analysis Info		Acquisition Date 7/5/2017 7:58:32 PM	
Analysis Name	D:\Data\2017\JUNE\SPPU AFFILIATED\VA AWATE COLLEGE, MANCHAR\KG KANADE\DB BANKAR\STSD-V_RB4_01_3978.d		
Method	dlc-ms600mz_10min.m	Operator	CIF
Sample Name	STSD-V	Instrument	impact HD 1819696.00184
Comment			

Acquisition Parameter					
Source Type	ESI	Ion Polarity	Positive	Set Nebulzer	0.3 Bar
Focus	Active	Set Capillary	3500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	600 m/z	Set Charging Voltage	2000 V	Set Divert Valve	Waste
		Set Corona	0 nA	Set APCI Heater	0 °C



Meas. m/z	#	Ion Formula	m/z	err [ppm]	mSigma	# mSigma	Score	rdB	e ⁻	Conf	N-Rule	Adduct
360.1807	1	C ₂₀ H ₂₆ NO ₅	360.1805	-0.5	14.7	1	100.00	8.5	even		ok	M+H
382.1629	1	C ₂₀ H ₂₅ NNaO ₅	382.1625	-1.0	27.1	1	100.00	8.5	even		ok	M+Na

STSD-V_RB4_01_3978.d

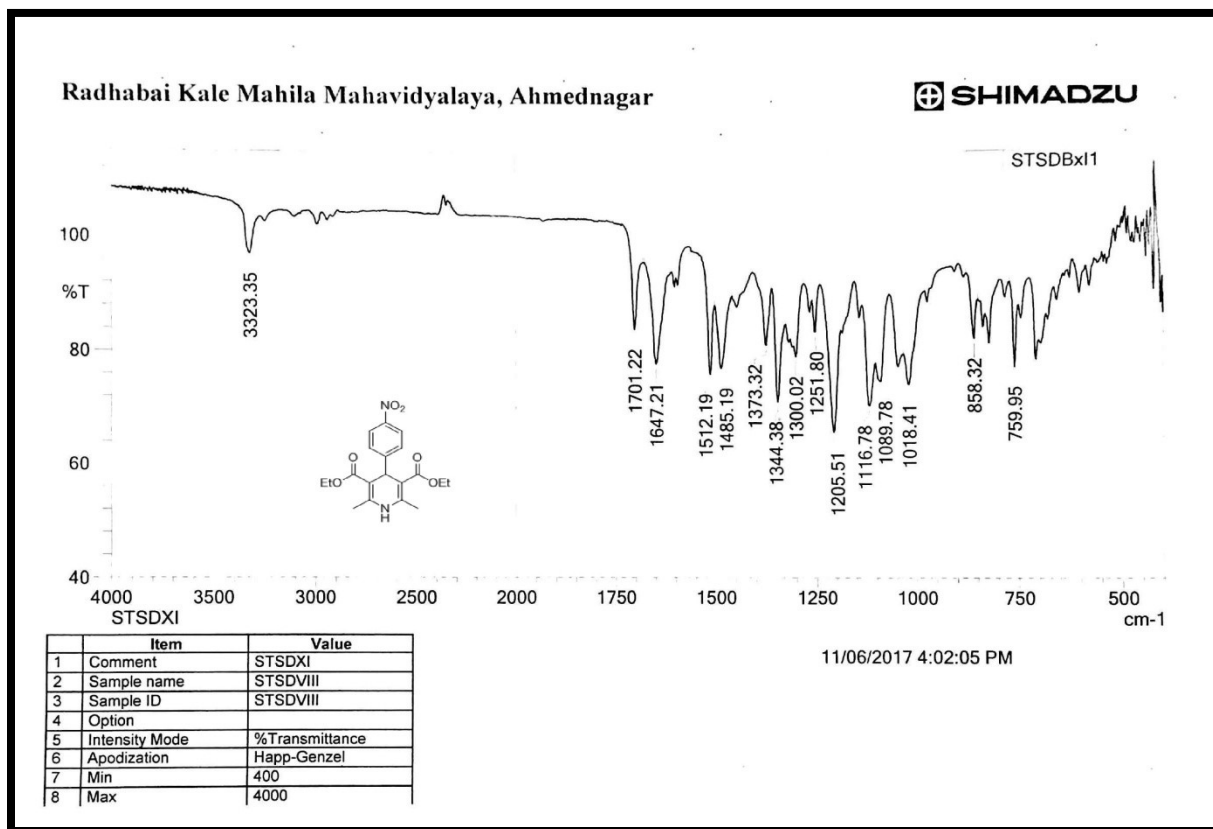
Bruker Compass DataAnalysis 4.2

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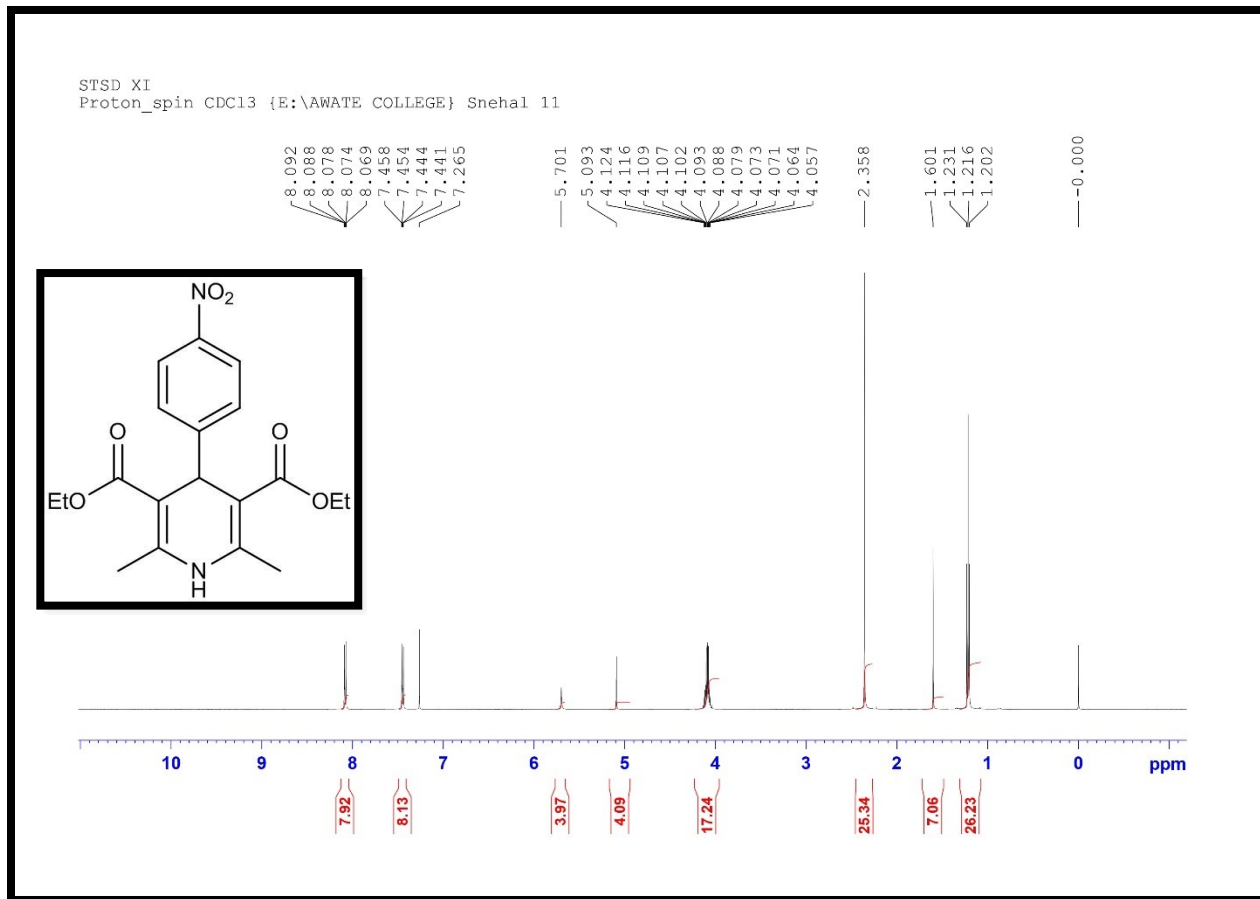
by: CIF

Page 1 of 1

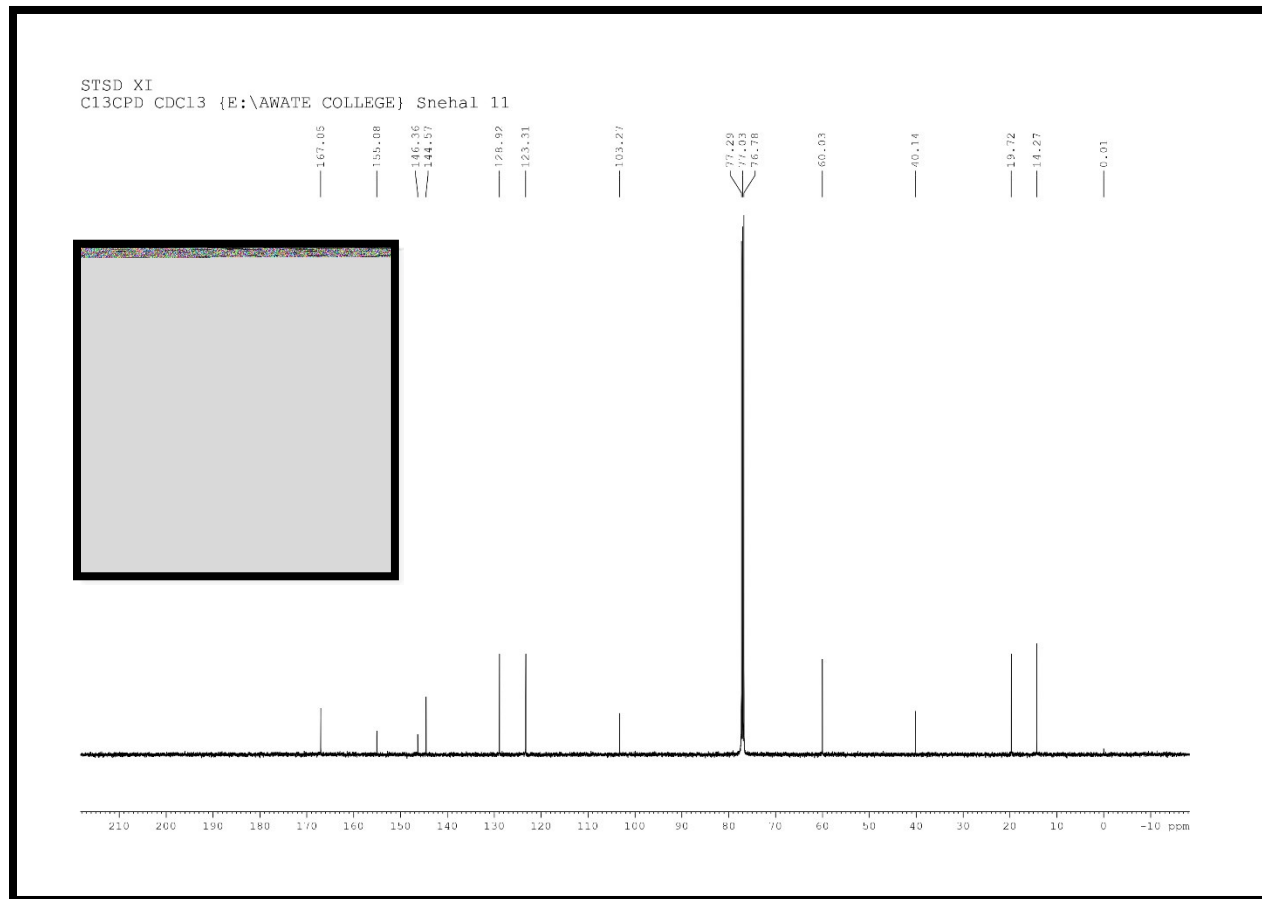
FTIR (Table 6, Entry 6)



¹H-NMR spectra (Table 6, Entry 6)



¹³C-NMR spectra (Table 6, Entry 6)

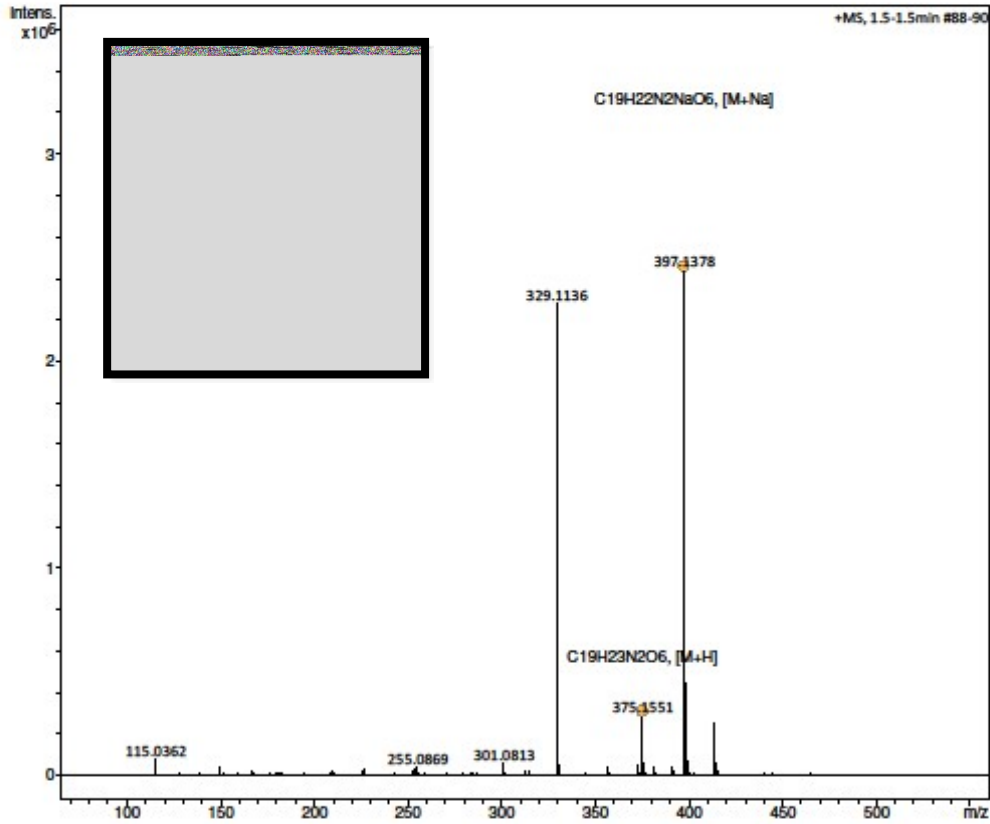


HRMS (Table 6, Entry 6)

Savitribai Phule Pune University - Central Instrumentation Facility

Analysis Info		Acquisition Date 7/5/2017 8:30:47 PM	
Analysis Name	D:\Data\2017\JUNE\SPPU AFFILIATED\VA AWATE COLLEGE, MANCHAR\KG KANADE\DB BANKAR\STSD-XI_RB7_01_3981.d		
Method	dlc-ms600mz_10min.m	Operator	CIF
Sample Name	STSD-XI	Instrument	impact HD 1819696.00184
Comment			

Acquisition Parameter					
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.3 Bar
Focus	Active	Set Capillary	3500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	600 m/z	Set Charging Voltage	2000 V	Set Divert Valve	Waste
		Set Corona	0 nA	Set APCI Heater	0 °C



Meas. m/z	#	Ion Formula	m/z	err [ppm]	mSigma	# mSigma	Score	rdb	e ⁻	Conf	N-Rule	Adduct
375.1551	1	C ₁₉ H ₂₃ N ₂ O ₆	375.1551	-0.0	3.4	1	100.00	9.5	even		ok	M+H
397.1378	1	C ₁₉ H ₂₂ N ₂ NaO ₆	397.1370	-1.9	20.8	1	100.00	9.5	even		ok	M+Na

STSD-XI_RB7_01_3981.d

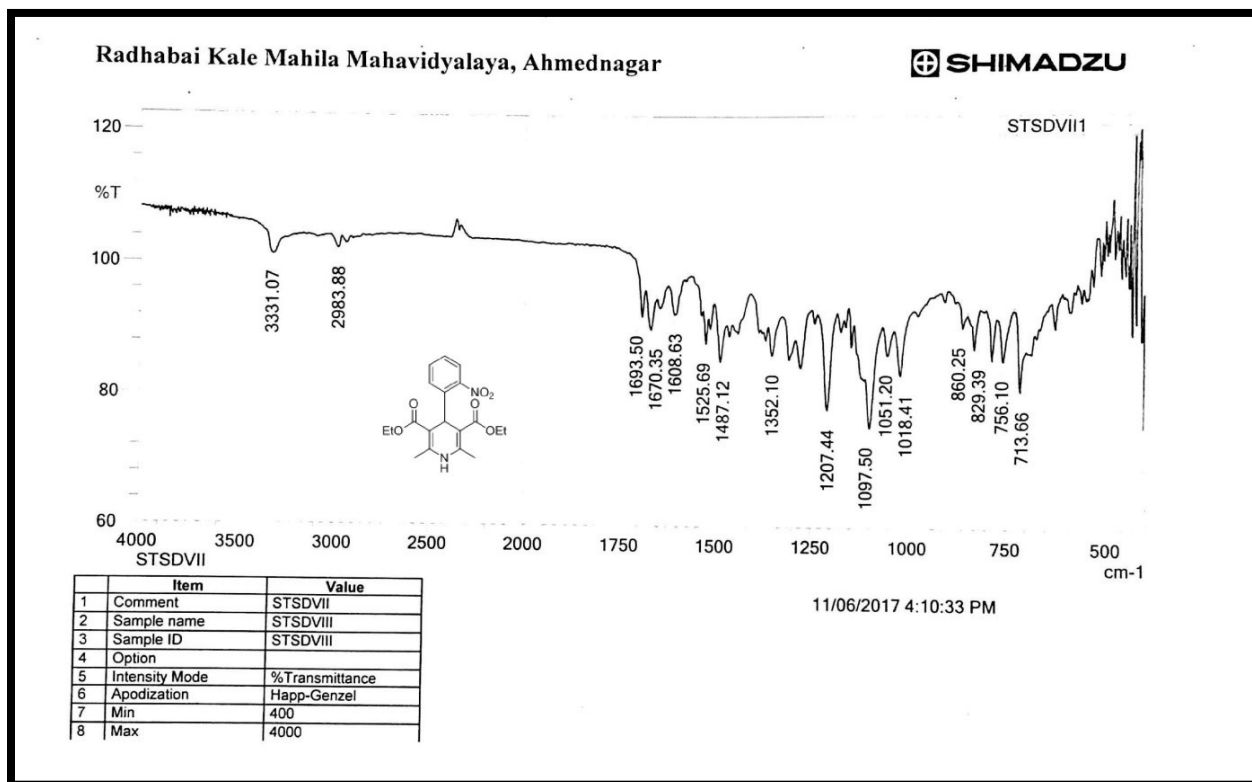
Bruker Compass DataAnalysis 4.2

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by: CIF

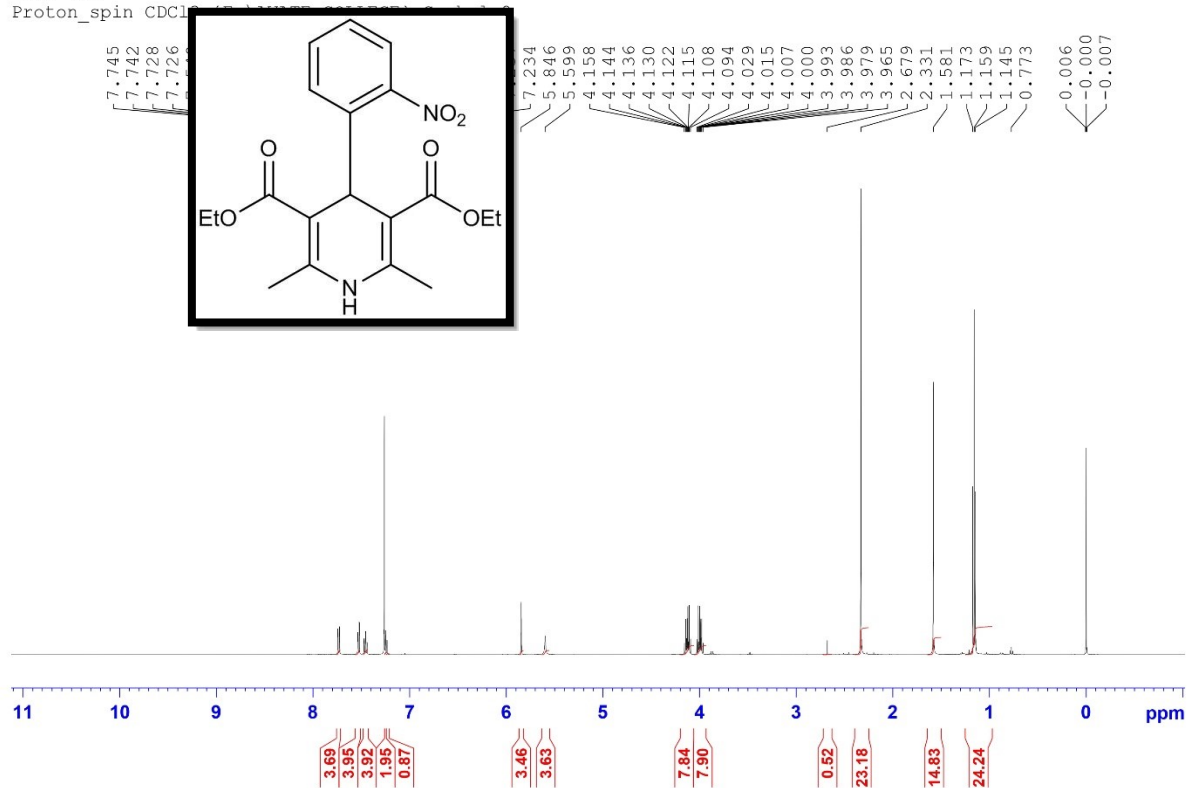
Page 1 of 1

FTIR (Table 6, Entry 7)

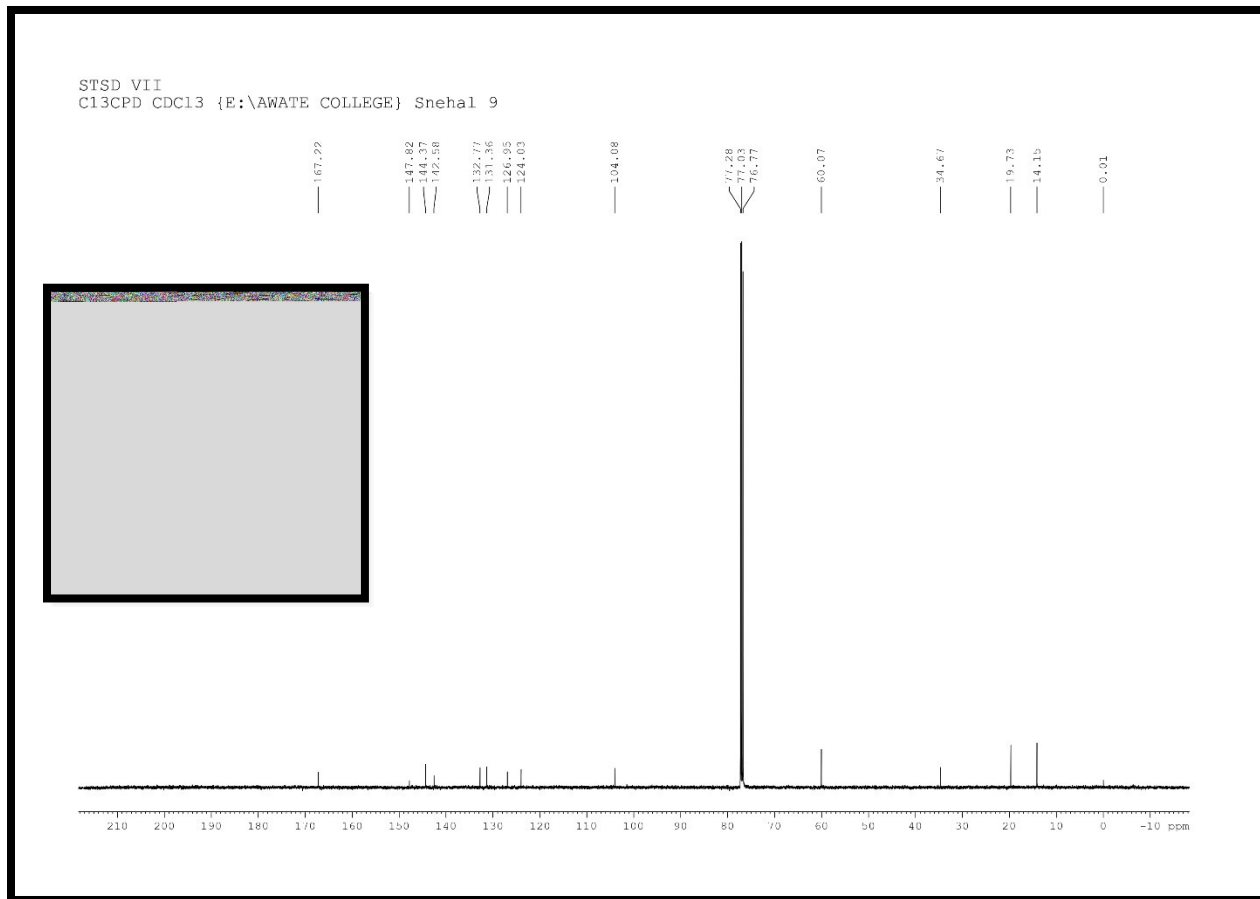


¹H-NMR spectra (Table 6, Entry 7)

STSD VII
Proton_spin CDCl₃



¹³C-NMR spectra (Table 6, Entry 7)

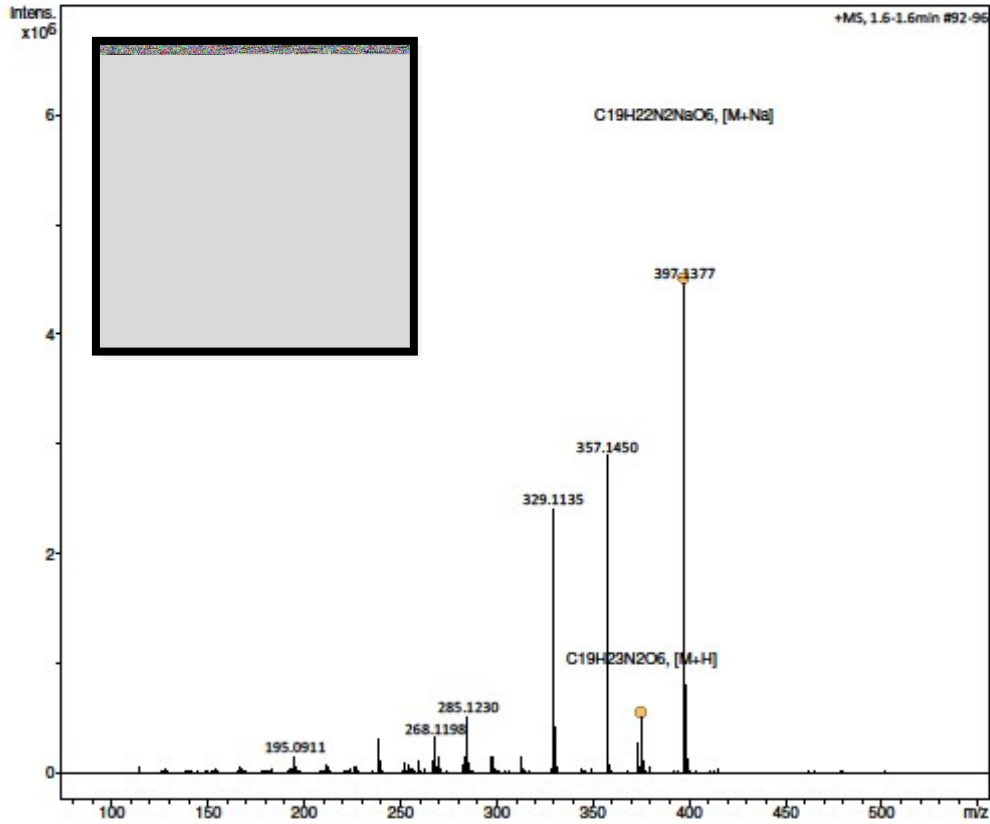


HRMS (Table 6, Entry 7)

Savitribai Phule Pune University - Central Instrumentation Facility

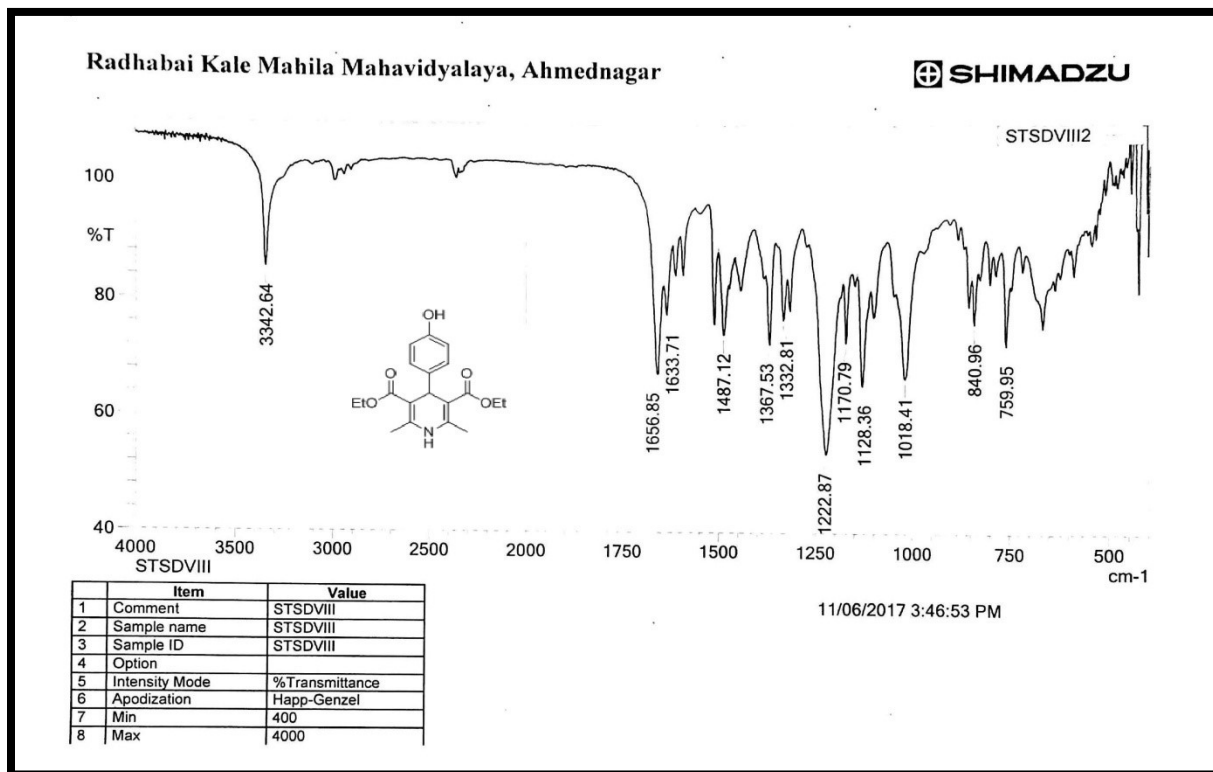
Analysis Info		Acquisition Date 7/5/2017 8:09:14 PM	
Analysis Name	D:\Data\2017\JUNE\SPPU AFFILIATED\VA AWATE COLLEGE, MANCHAR\KG KANADE\DB BANKAR\STSD-VII_RB5_01_3979.d		
Method	dlc-ms600mz_10min.m	Operator	CIF
Sample Name	STSD-VII	Instrument	impact HD 1819696.00184
Comment			

Acquisition Parameter					
Source Type	ESI	Ion Polarity	Positive	Set Nebulzer	0.3 Bar
Focus	Active	Set Capillary	3500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	600 m/z	Set Charging Voltage	2000 V	Set Divert Valve	Waste
		Set Corona	0 nA	Set APCI Heater	0 °C

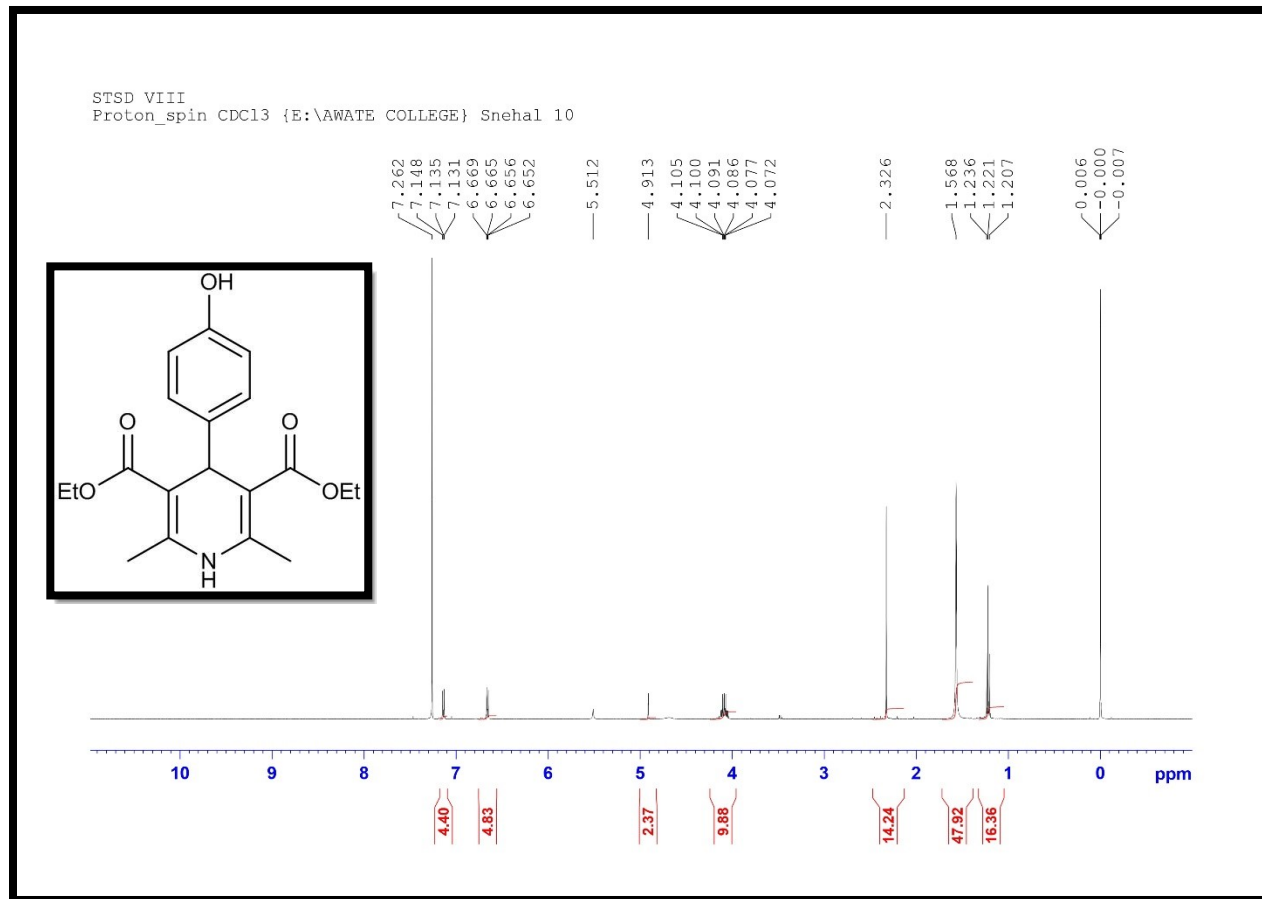


Meas. m/z	#	Ion Formula	m/z	err [ppm]	mSigma	# mSigma	Score	rdb	e ⁻	Conf	N-Rule	Adduct
375.1550	1	C19H23N2O6	375.1551	0.2	7.8	1	100.00	9.5	even		ok	M+H
397.1377	1	C19H22N2NaO6	397.1370	-1.8	22.4	1	100.00	9.5	even		ok	M+Na

FTIR (Table 6, Entry 8)



¹H-NMR spectra (Table 6, Entry 8)

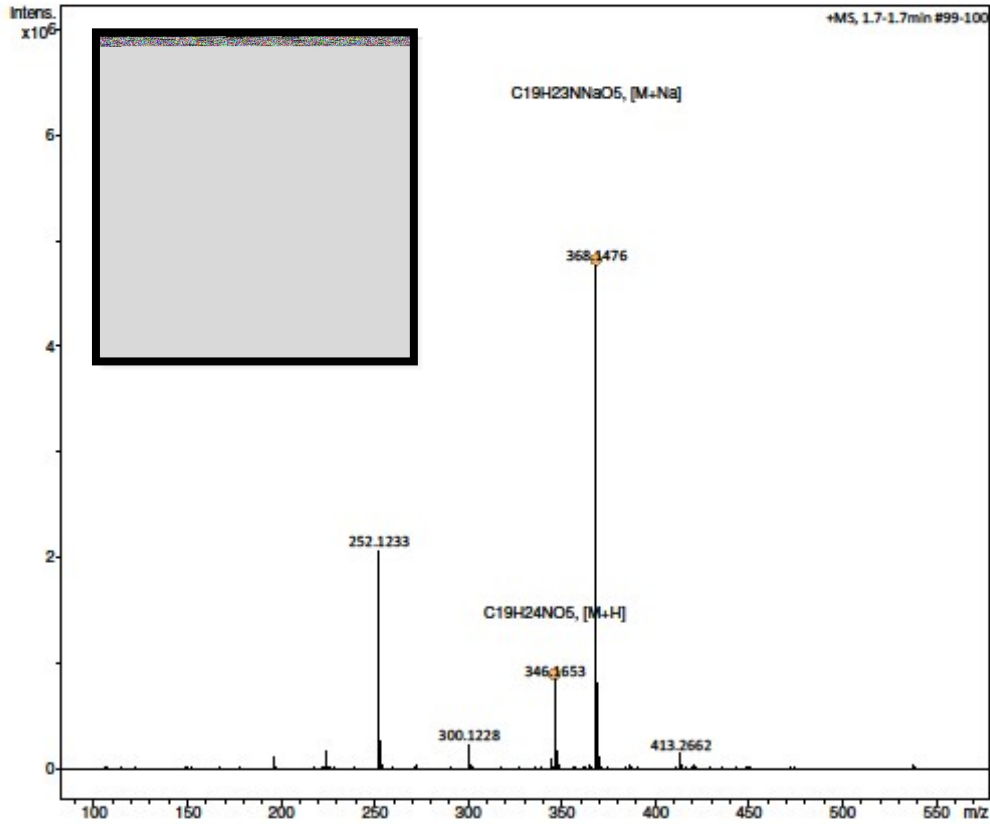


HRMS (Table 6, Entry 8)

Savitribai Phule Pune University - Central Instrumentation Facility

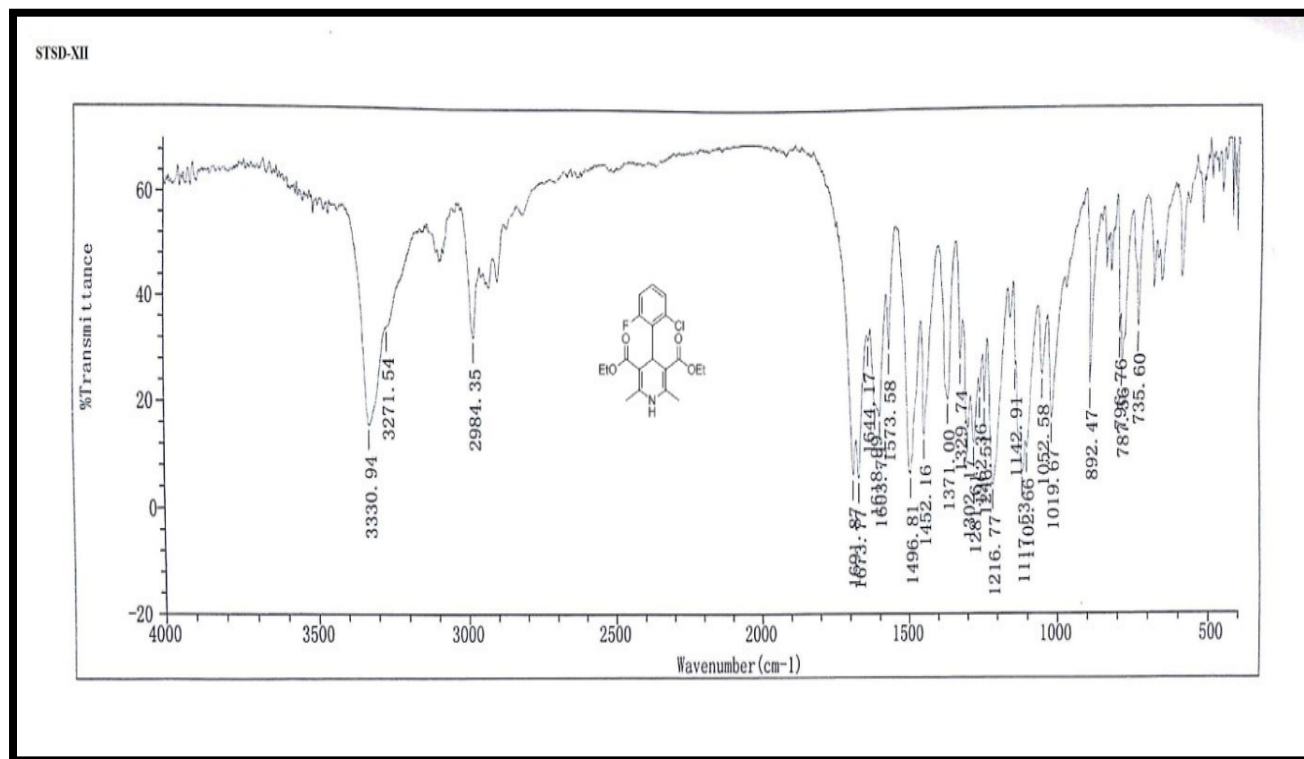
Analysis Info		Acquisition Date 7/5/2017 8:20:07 PM	
Analysis Name	D:\Data\2017\JUNE\SPPU AFFILIATED\VA AWATE COLLEGE, MANCHAR\KG KANADE\DB BANKAR\STSD-VIII_RB6_01_3980.d		
Method	dlc-ms600mz_10min.m	Operator	CIF
Sample Name	STSD-VIII	Instrument	impact HD 1819696.00184
Comment			

Acquisition Parameter					
Source Type	ESI	Ion Polarity	Positive	Set Nebulzer	0.3 Bar
Focus	Active	Set Capillary	3500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	600 m/z	Set Charging Voltage	2000 V	Set Divert Valve	Waste
		Set Corona	0 nA	Set APCI Heater	0 °C

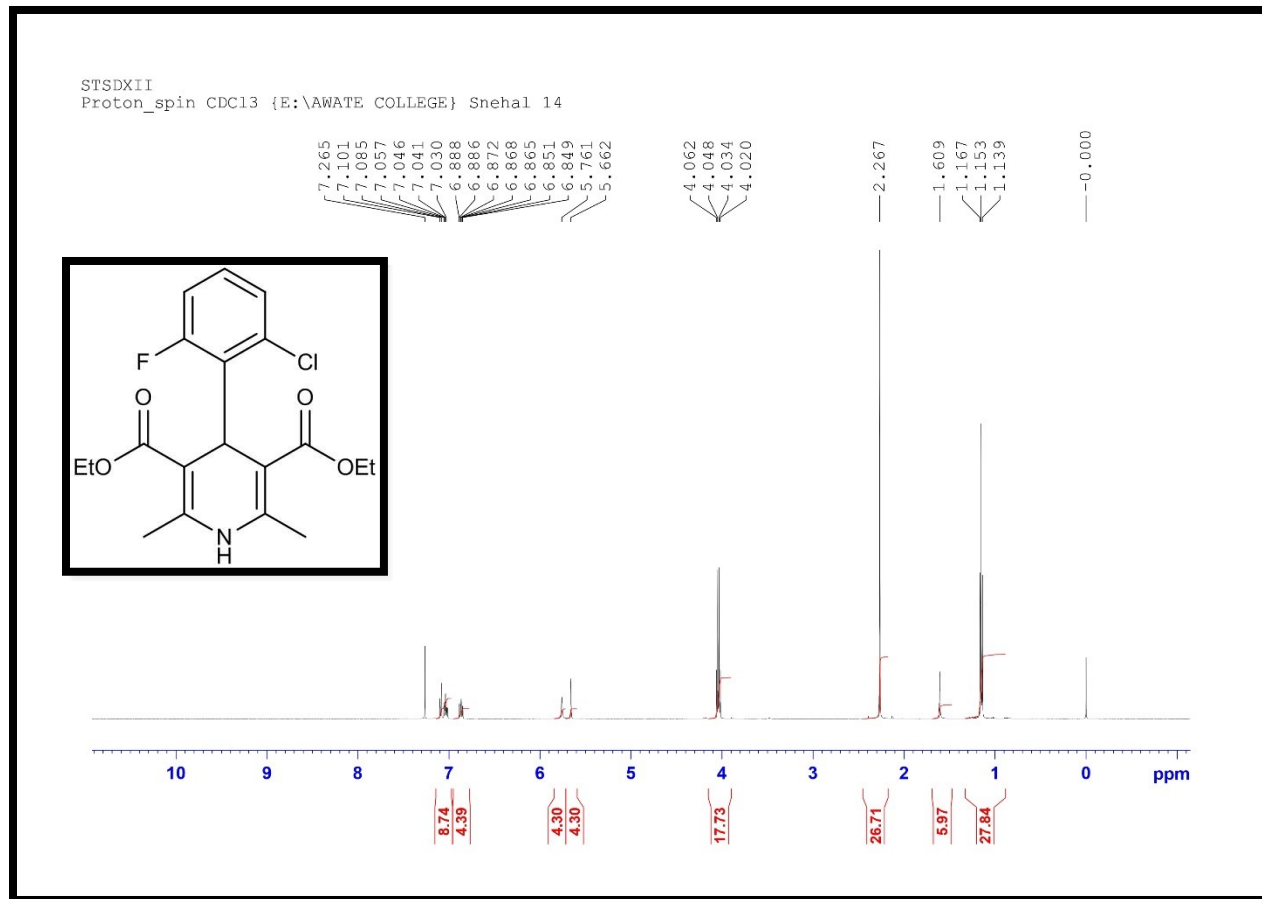


Meas. m/z	#	Ion Formula	m/z	err [ppm]	mSigma	#	mSigma	Score	rdB	e ⁻	Conf	N-Rule	Adduct
346.1653	1	C19H24NO5	346.1649	-1.0	7.0	1	100.00	8.5	even			ok	M+H
368.1476	1	C19H23NNaO5	368.1468	-2.2	25.1	1	100.00	8.5	even			ok	M+Na

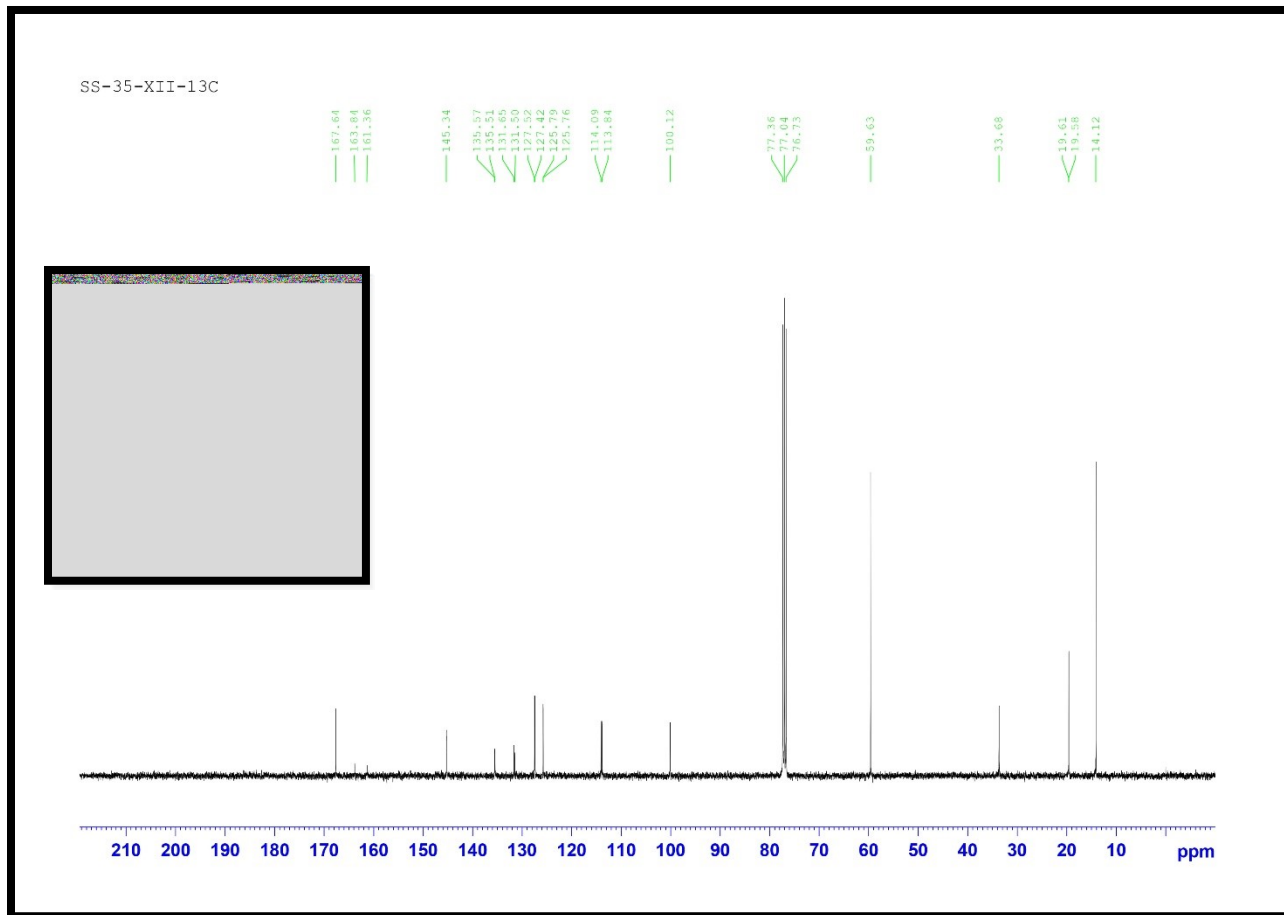
FTIR (Table 6, Entry 9)



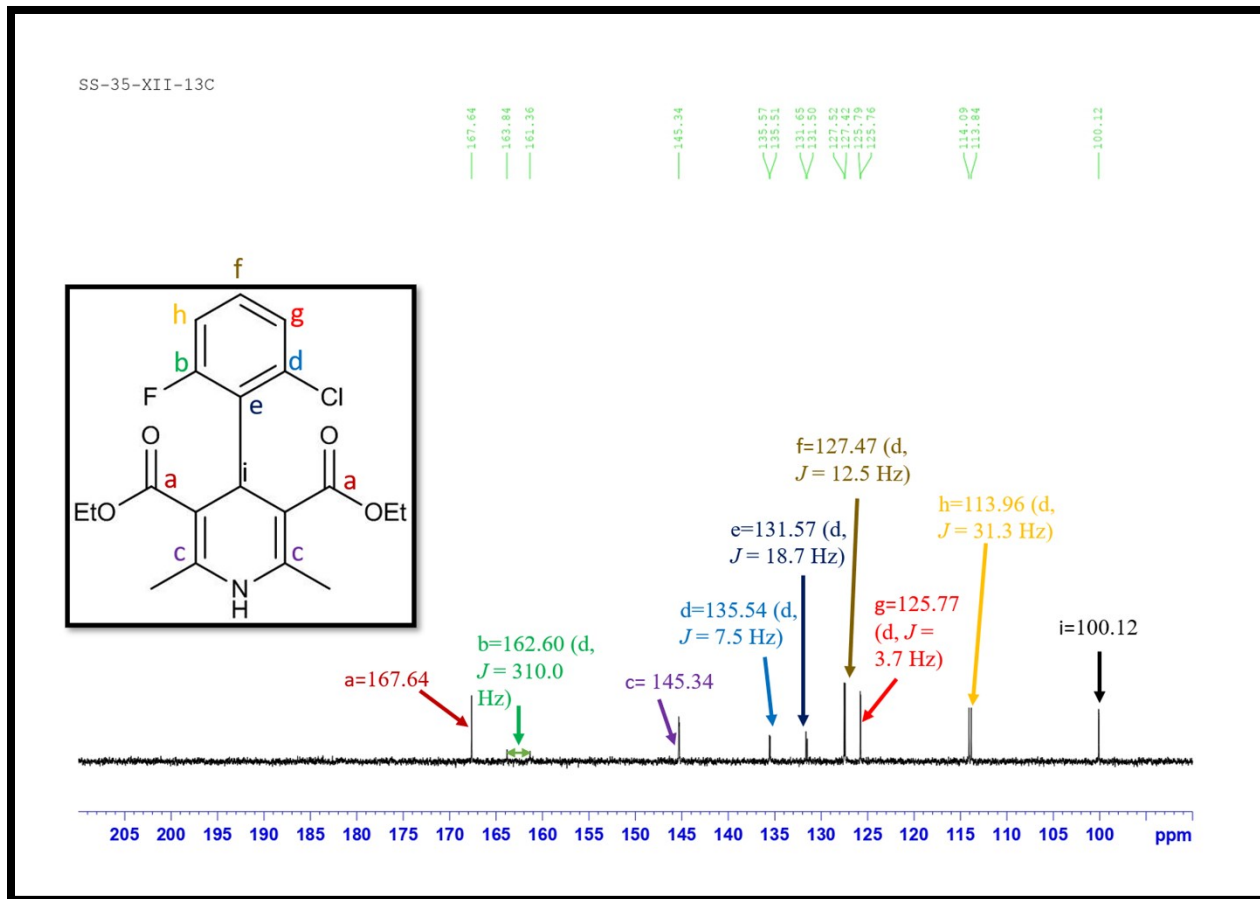
¹H-NMR spectra (Table 6, Entry 9)



¹³C-NMR spectra (Table 6, Entry 9)



¹³C-NMR Expansion image (Table 6, Entry 9)

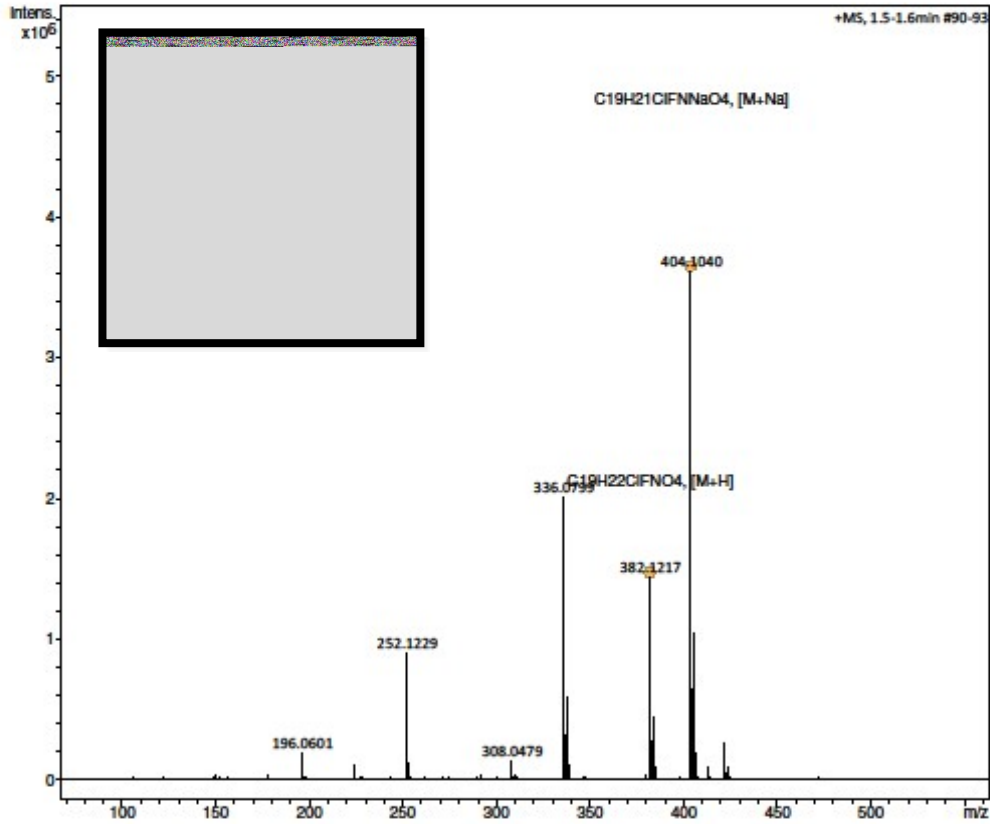


HRMS (Table 6, Entry 9)

Savitribai Phule Pune University - Central Instrumentation Facility

Analysis Info		Acquisition Date 7/5/2017 8:41:30 PM	
Analysis Name	D:\Data\2017\JUNE\SPPU AFFILIATED\VA AWATE COLLEGE, MANCHAR\KG KANADE\DB BANKAR\STSD-XII_RB8_01_3982.d		
Method	dlc-ms600mz_10min.m	Operator	CIF
Sample Name	STSD-XII	Instrument	impact HD 1819696.00184
Comment			

Acquisition Parameter					
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.3 Bar
Focus	Active	Set Capillary	3500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	600 m/z	Set Charging Voltage	2000 V	Set Divert Valve	Waste
		Set Corona	0 nA	Set APCI Heater	0 °C



Meas. m/z	#	Ion Formula	m/z	err [ppm]	mSigma	# mSigma	Score	rdb	e ⁻	Conf	N-Rule	Adduct
382.1217	1	C19H22ClFNO4	382.1216	-0.4	19.1	1	100.00	8.5	even		ok	M+H
404.1040	1	C19H21ClFNNaO4	404.1035	-1.2	32.7	1	100.00	8.5	even		ok	M+Na

STSD-XII_RB8_01_3982.d

Bruker Compass DataAnalysis 4.2

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