

Ultrasound-promoted synthesis of Bi, Tri and Tetrapodal Polyhydroquinolines, 1,4-Dihydropyridines and their pyridines

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Centre for Organic and Medicinal Chemistry,

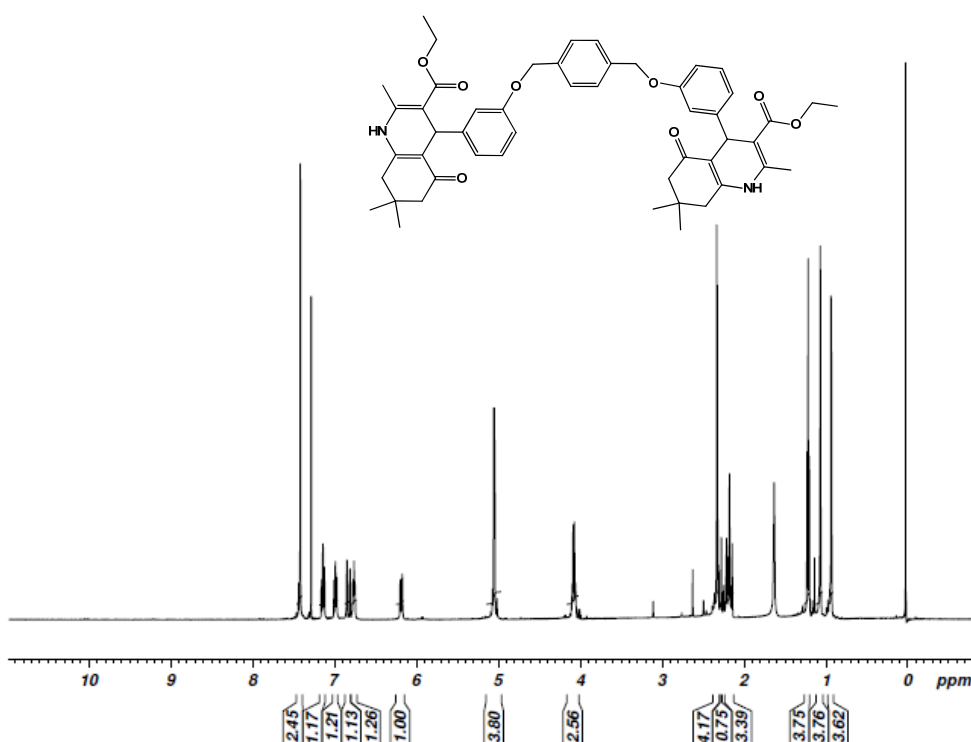
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Supporting Information

¹H NMR of 3.3 Diethyl 4, 4'-(3, 3'-(1,4-phenylenebis(methylene)bis(oxy)bis(3,1-phenylene))bis(2,7,7-trimethyl -5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate) [9]

S-3.....Balaji, VIT



Current Data Parameters
NAME Sep23-2010
EXPNO 9
PROCNO 1

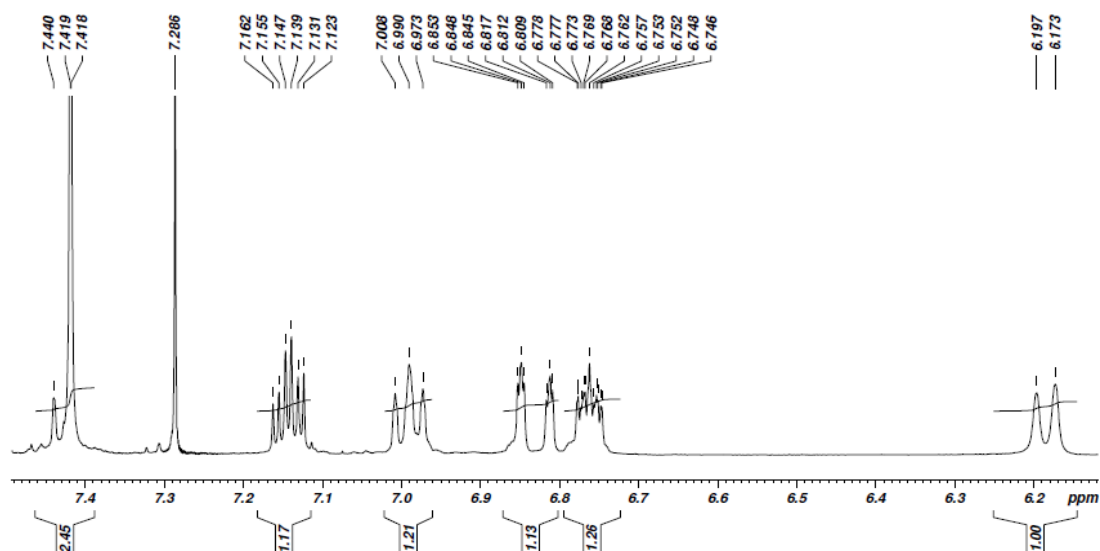
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FIDRES 0.315264 Hz
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DE 6.50 usec
TE 299.8 K
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TD0 1

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PL1 0.00 dB
PL1W 23.53637505 W
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F2 - Processing parameters
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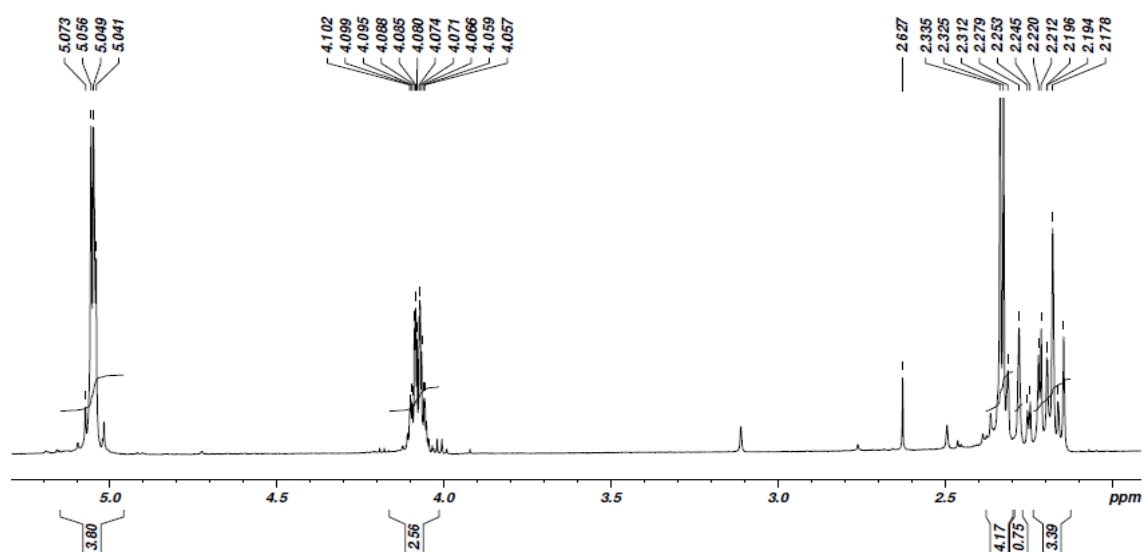
¹H NMR (Enlarged) of 3.3 Diethyl 4, 4'-(3, 3'-(1,4-phenylenebis(methylene)bis(oxy)bis(3,1-phenylene))bis(2,7,7-trimethyl -5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate) [9]

S-3.....Balaji, VIT



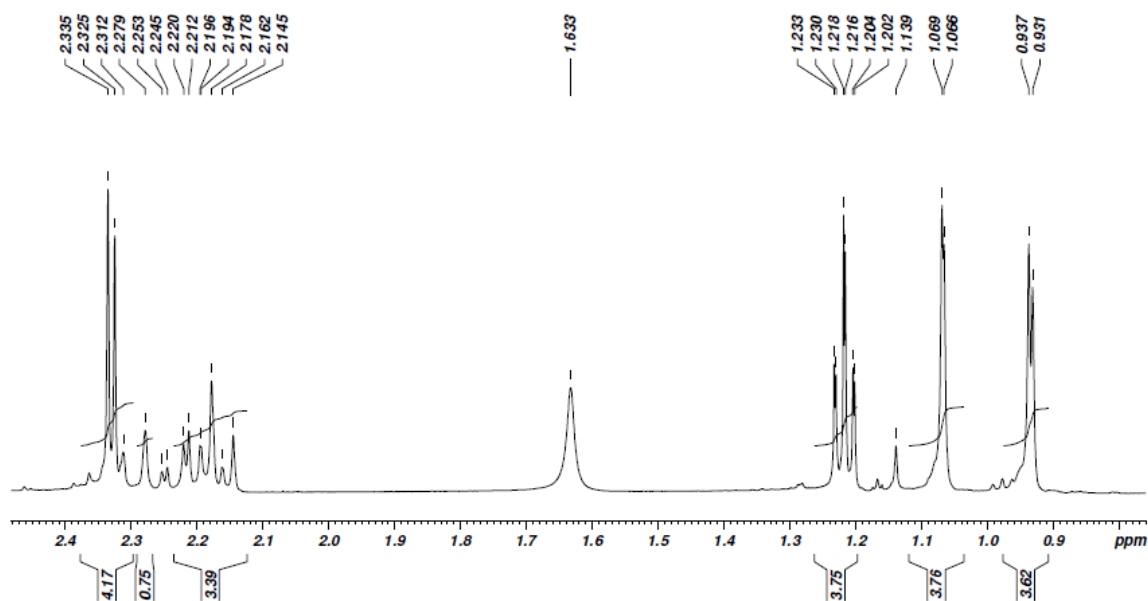
¹H NMR (Enlarged) of 3.3 Diethyl 4, 4'-(3, 3'-(1,4-phenylenebis(methylene)bis(oxy)bis(3,1-phenylene))bis(2,7,7-trimethyl -5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate) [9]

S-3.....Balaji, VIT



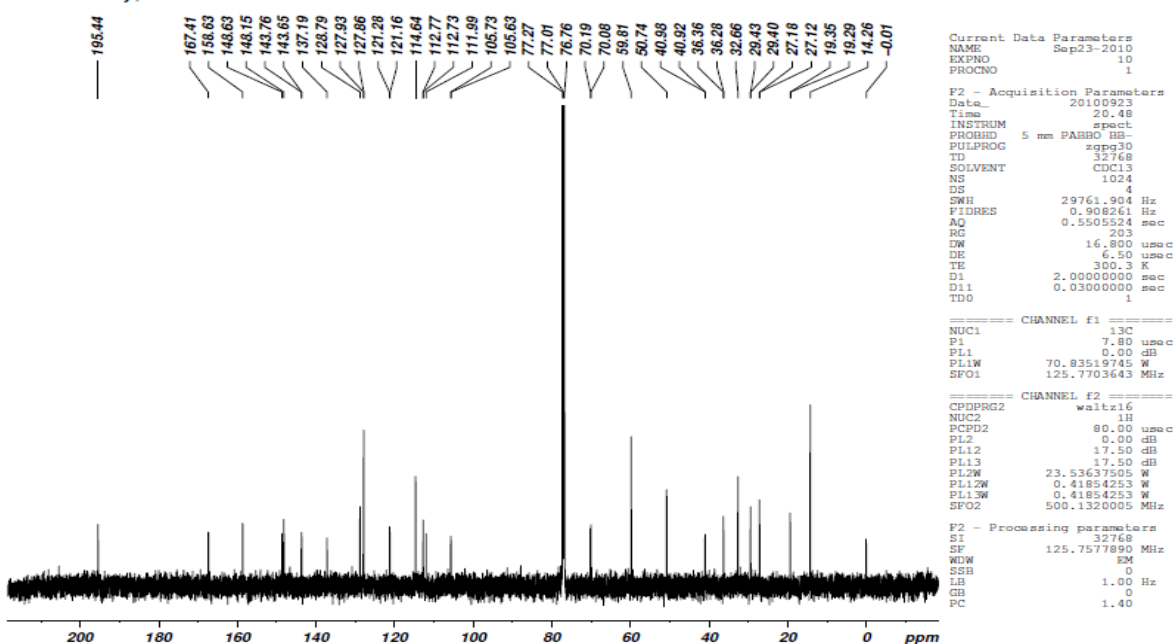
¹H NMR (Enlarged) of 3.3 Diethyl 4, 4'-(3, 3'-(1,4-phenylenebis(methylene)bis(oxy)bis(3,1-phenylene))bis(2,7,7-trimethyl -5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate) [9]

S-3.....Balaji, VIT

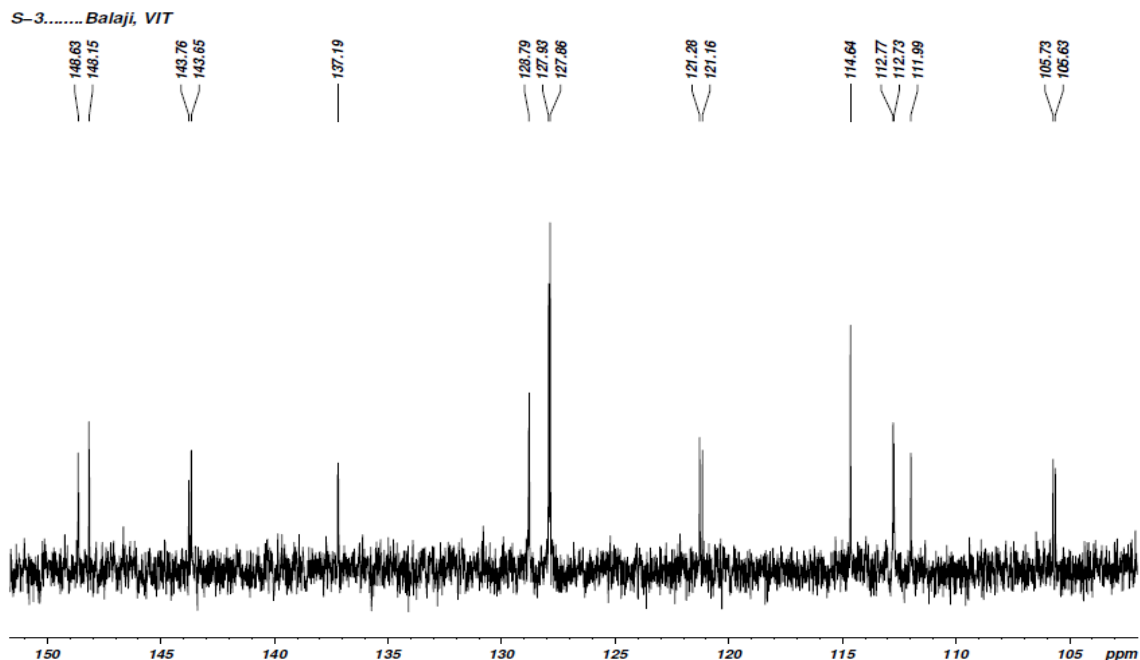


¹³C NMR of 3.3 Diethyl 4, 4'-(3, 3'-(1,4-phenylenebis(methylene)bis(oxy)bis(3,1-phenylene))bis(2,7,7-trimethyl -5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate) [9]

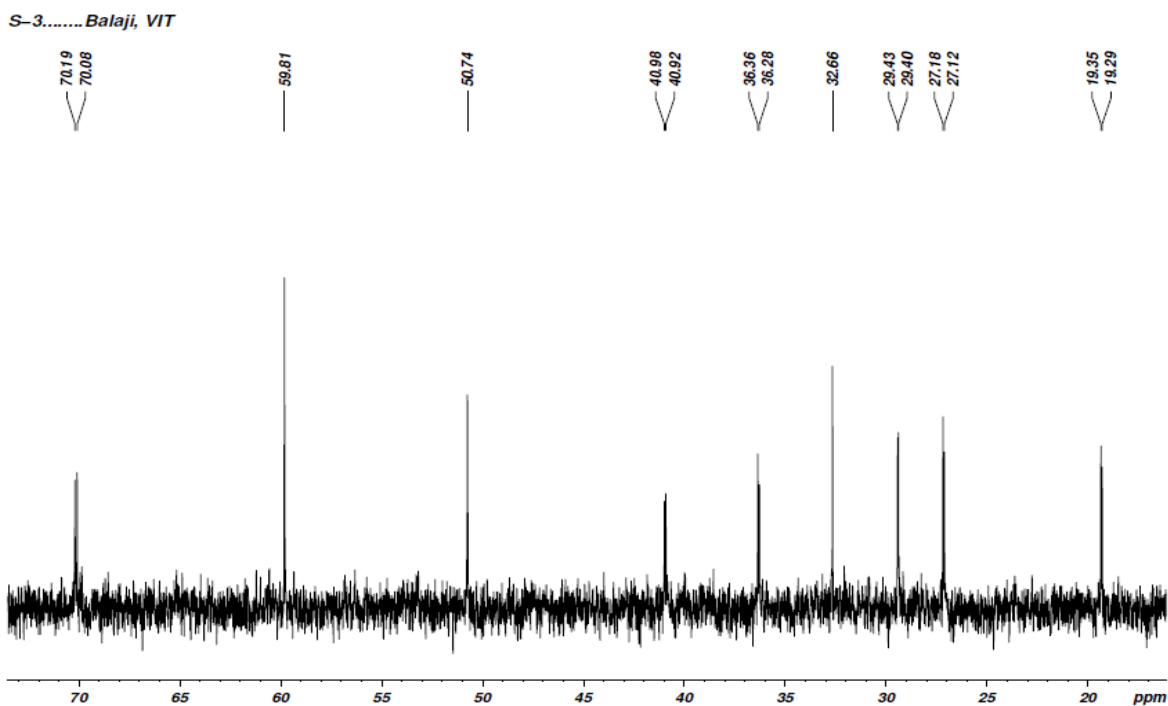
S-3.....Balaji, VIT



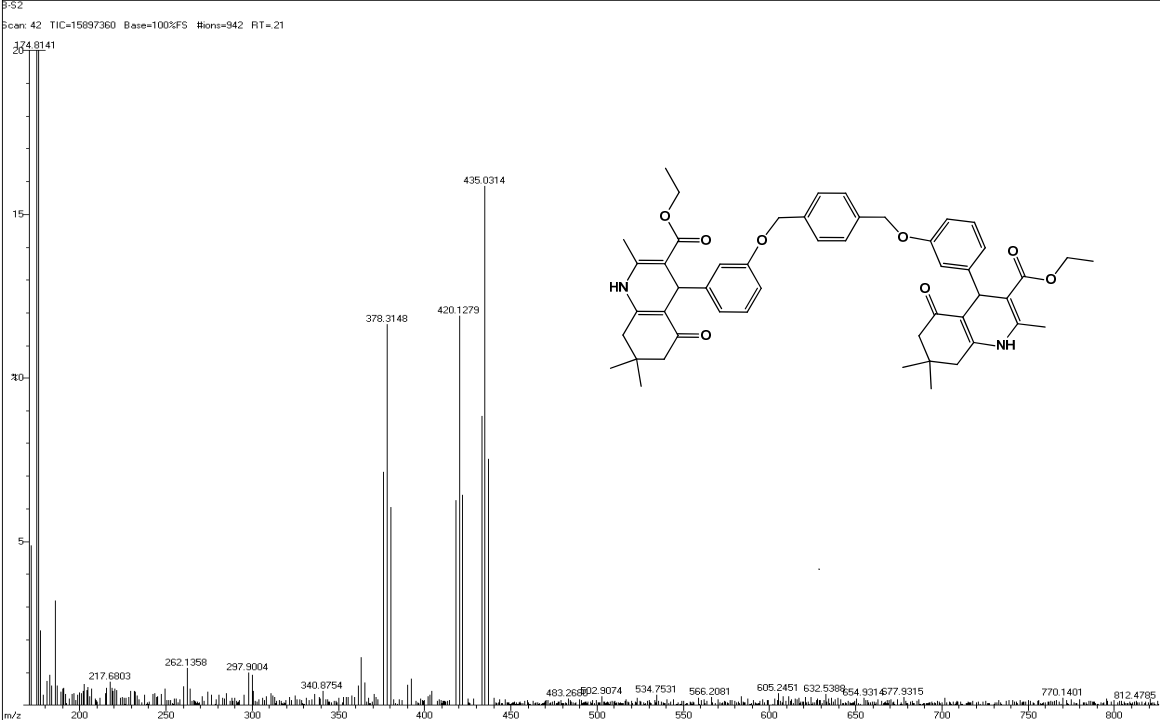
^{13}C NMR (Enlarged) of 3.3 Diethyl 4, 4'-(3, 3'-(1,4-phenylenebis(methylene)bis(oxy)bis(3,1-phenylene))bis(2,7,7-trimethyl -5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate) [9]



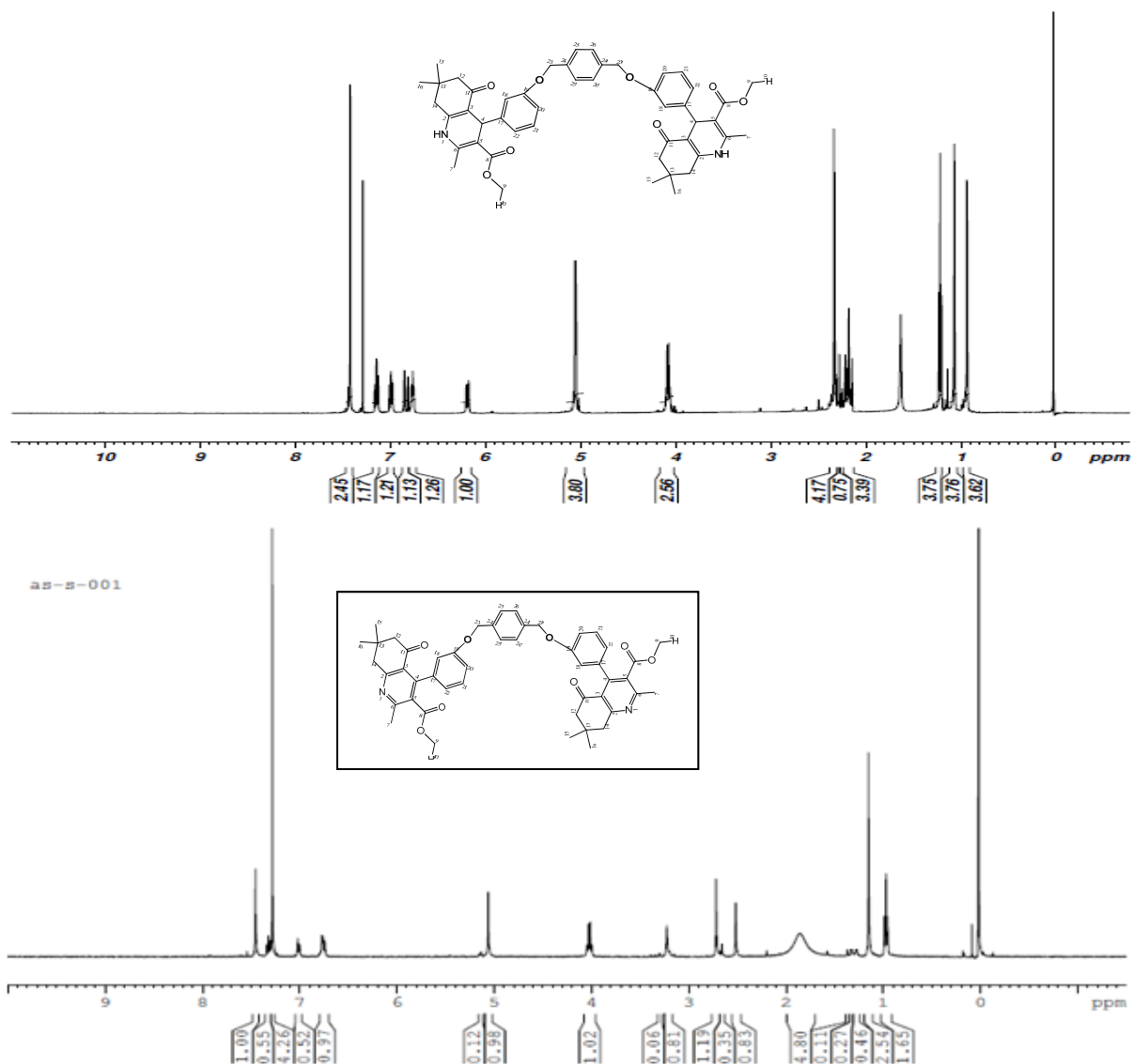
^{13}C NMR (Enlarged) of 3.3 Diethyl 4, 4'-(3, 3'-(1,4-phenylenebis(methylene)bis(oxy)bis(3,1-phenylene))bis(2,7,7-trimethyl -5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate) [9]



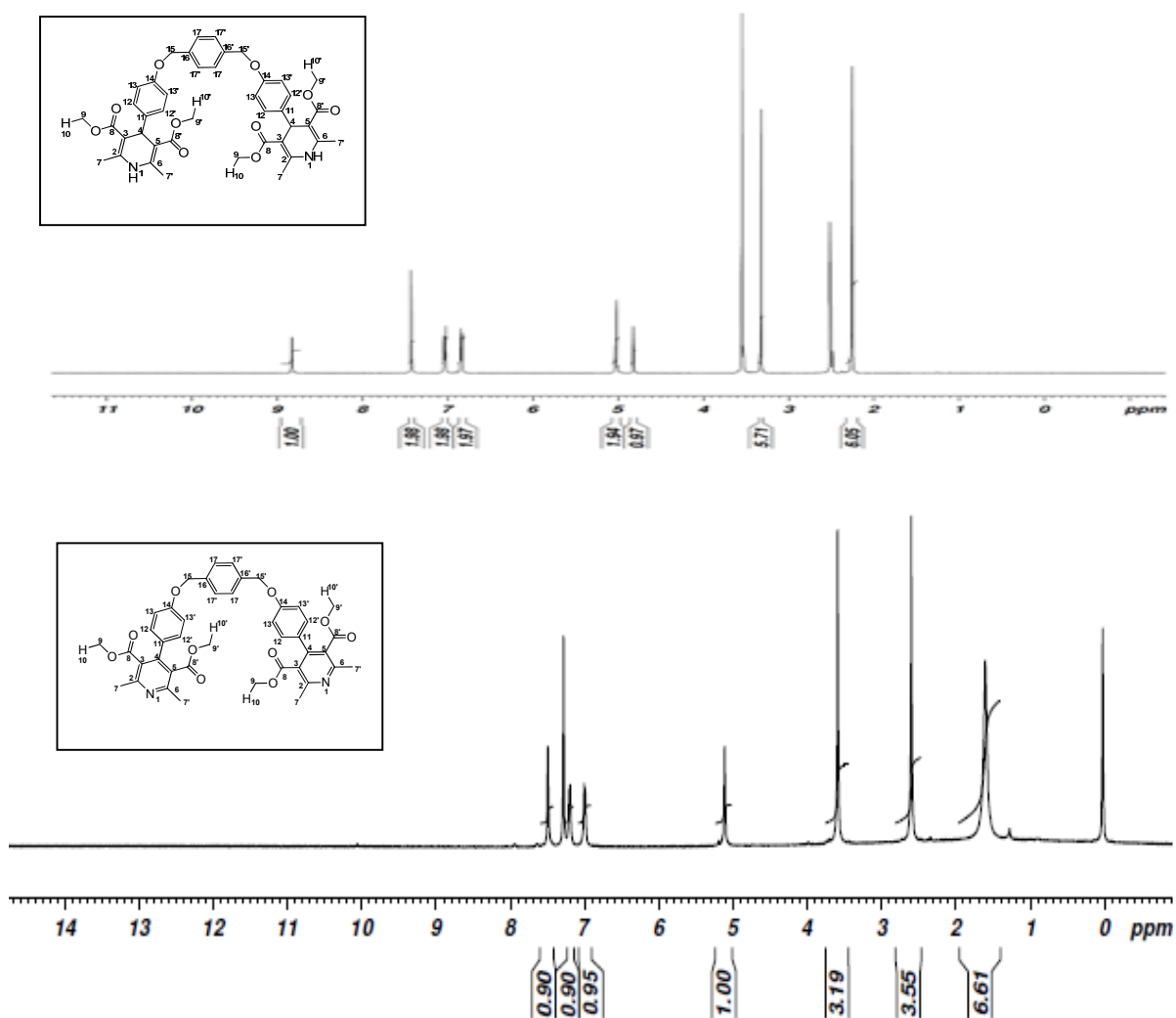
HR-MS of 3.3 Diethyl 4, 4'-(3, 3'-(1,4-phenylenebis(methylene)bis(oxy)bis(3,1-phenylene))bis(2,7,7-trimethyl -5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate) [9]



Comparative ^1H -NMR Spectra of Diethyl 4,4'-(((1,4-phenylenebis(methylene)))bis(oxy))bis(3,1-phenylene))bis(2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate) (**9**) and Diethyl 4,4'-(((1,4-phenylenebis(methylene)))bis(oxy))bis(3,1-phenylene))bis(2,7,7-trimethyl-5-oxo-5,6,7,8-tetrahydroquinoline-3-carboxylate) (**28**).

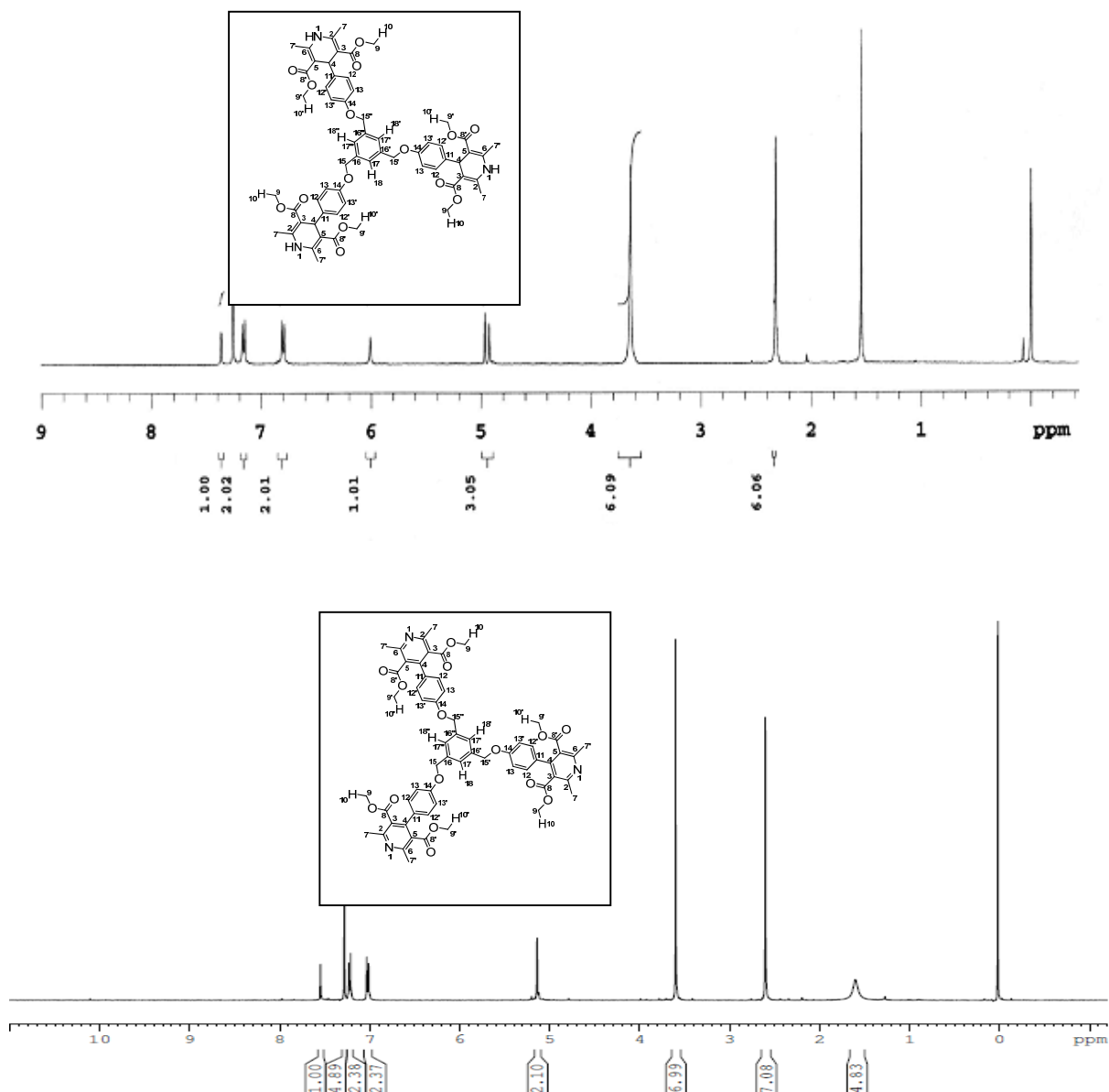


Comparative ^1H -NMR spectra of Tetramethyl 4,4'-(((1,4-phenylenebis(methylene)))bis(oxy))bis(4,1-phenylene))bis(2,6-dimethyl-1,4-dihydropyridine-3,5-dicarboxylate) (**15**) and Tetramethyl 4,4'-(((1,4-phenylenebis(methylene)))bis(oxy)) bis(4,1-phenylene))bis(2,6-dimethylpyridine-3,5-dicarboxylate) (**33**).

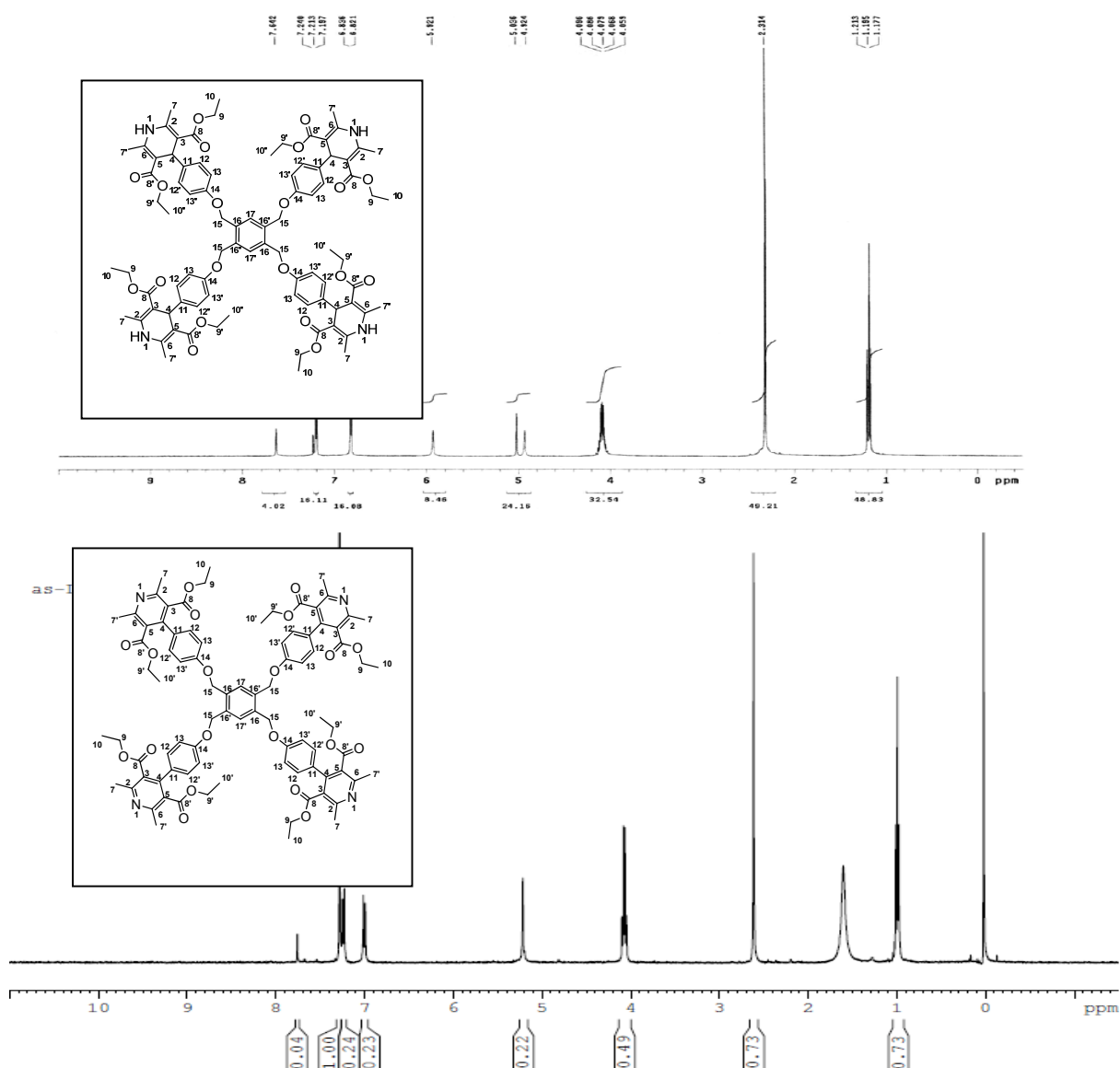


Though the ^1H -NMR spectrum of **33** is similar to **15** the observation of slight difference in chemical shift values along with the absence of signals corresponds to the protons at -NH as well as at C-4, 4' supports the conversion of **15** into Tetramethyl 4,4'-(((1,4-phenylenebis(methylene)))bis(oxy))bis(4,1-phenylene))bis(2,6-dimethyl pyridine-3,5-dicarboxylate) (**33**).

Comparative ^1H -NMR Spectra of Hexamethyl 4,4',4''-(((benzene-1,3,5-triyl tris(methylene))tris(oxy)))tris(benzene-4,1-diyl))tris(2,6-dimethyl-1,4-dihydropyridine-3,5-dicarboxylate) (**24**) and Hexamethyl 4,4',4''-(((benzene-1,3,5-triyl tris(methylene))tris(oxy)))tris(benzene-4,1-diyl))tris(2,6-dimethylpyridine-3,5-dicarboxylate) (**38**).

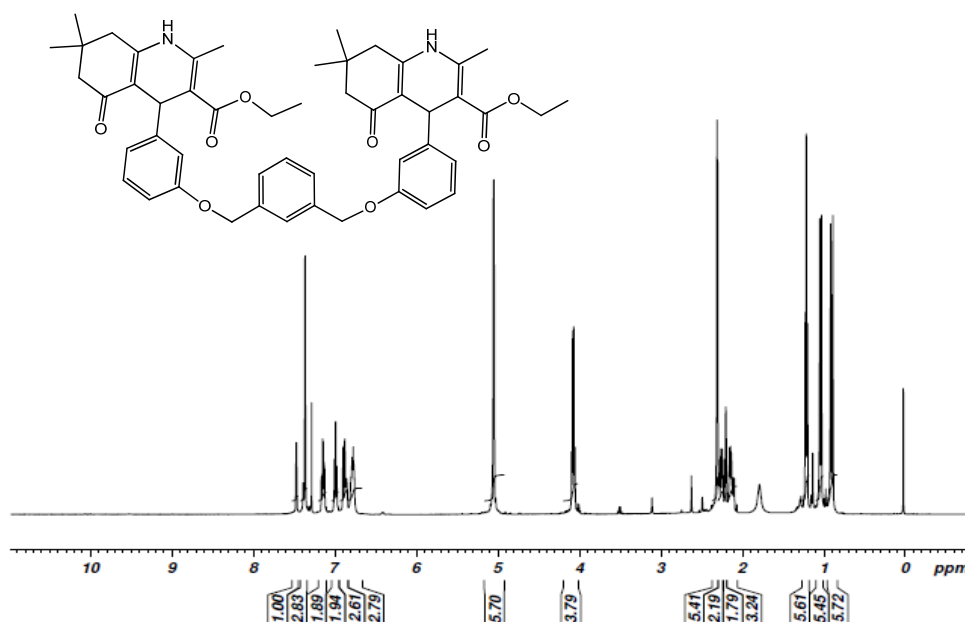


Comparative ^1H -NMR spectra of Octaethyl 4,4',4'',4'''-(((benzene-1,2,4,5-tetrayltetrakis(methylene))tetrakis(oxy))tetrakis(benzene-4,1-diyl))tetrakis(2,6-dimethyl-1,4-dihydropyridine-3,5-dicarboxylate) (**26**) and Octaethyl 4,4',4'',4'''-(((benzene-1,2,4,5-tetrayltetrakis(methylene))tetrakis(oxy))tetrakis(benzene-4,1-diyl))tetrakis(2,6-dimethylpyridine-3,5-dicarboxylate) (**39**).



^1H NMR of Diethyl 4,4'-(3,3'-(1,3-phenylenebis(methylene)bis(oxy))bis(3,1-phenylene))bis(2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate) [10a]

S-2.....Balaji, VIT



Current Data Parameters
NAME Sep23-2010
EXPNO 23
PROCNO 1

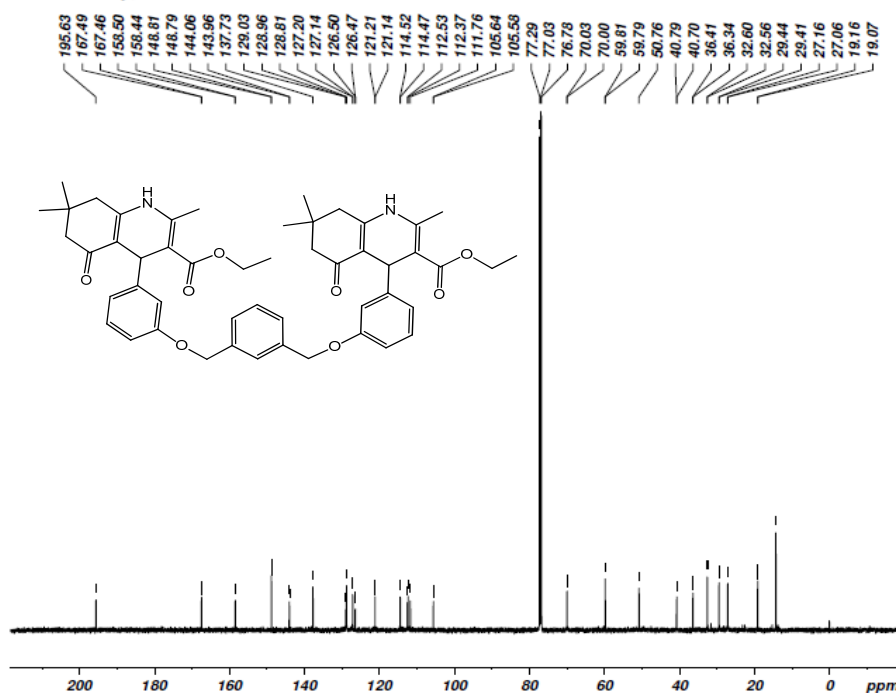
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FIDRES 0.315264 Hz
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RG 1
DW 48.400 usec
DE 6.50 usec
TE 300.0 K
D1 1.0000000 sec
TD0 1

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PL1 0.00 dB
PL1W 23.53637505 W
SFO1 500.1330885 MHz

F2 - Processing parameters
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SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40

^{13}C NMR of Diethyl 4,4'-(3,3'-(1,3-phenylenebis(methylene)bis(oxy))bis(3,1-phenylene))bis(2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate) [10a]

S-2.....Balaji, VIT



Current Data Parameters
NAME Sep23-2010
EXPNO 24
PROCNO 1

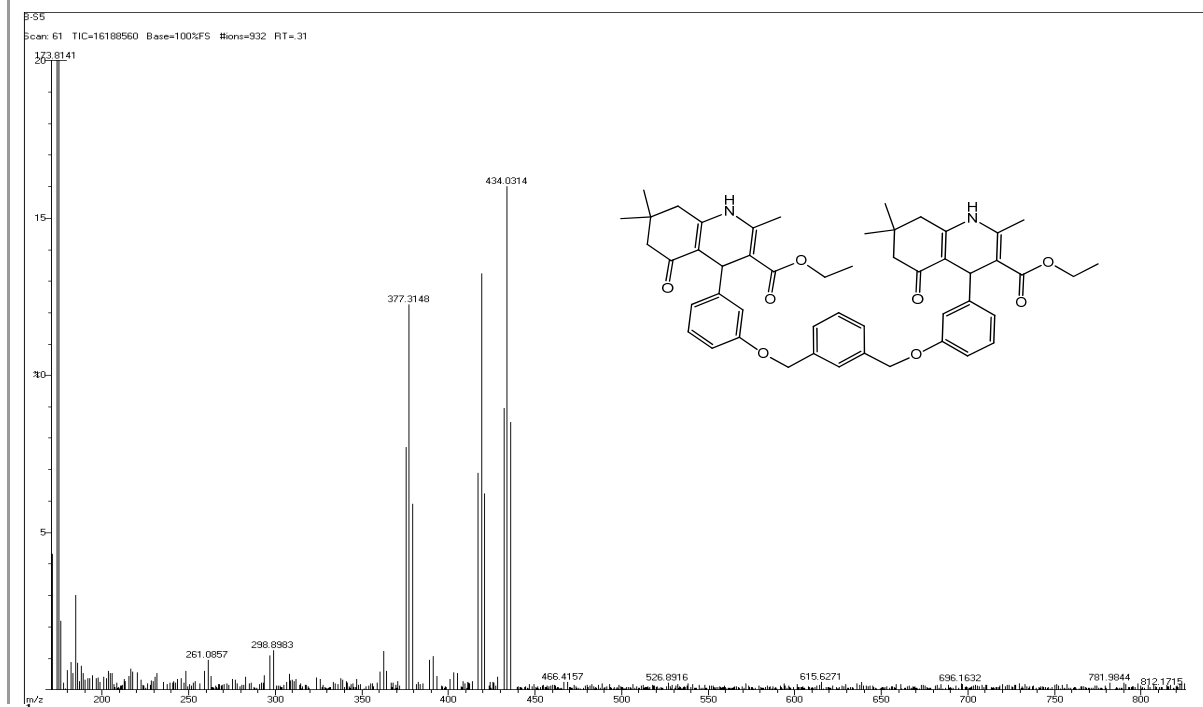
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SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 0.5505524 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 300.2 K
D1 2.0000000 sec
D11 0.0300000 sec
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PL1W 70.83519745 W
SFO1 125.7703643 MHz

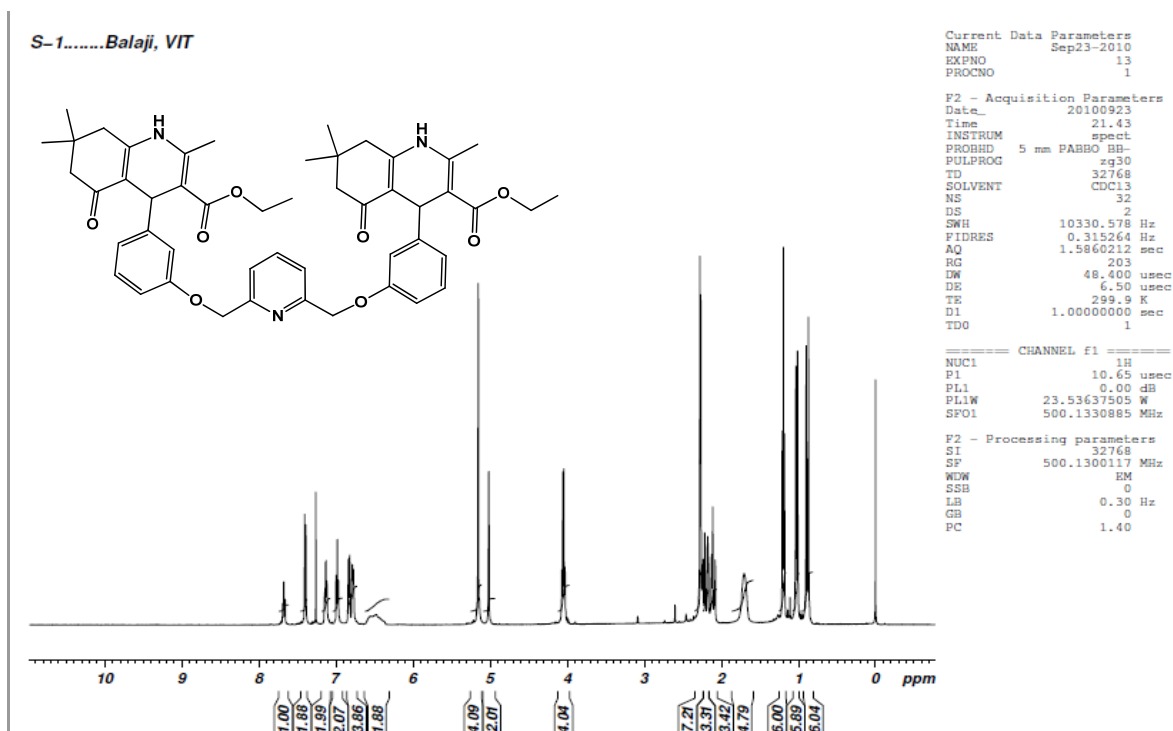
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PL12 17.50 dB
PL13 17.50 dB
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PL12W 0.41854253 W
PL13W 0.41854253 W
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F2 - Processing parameters
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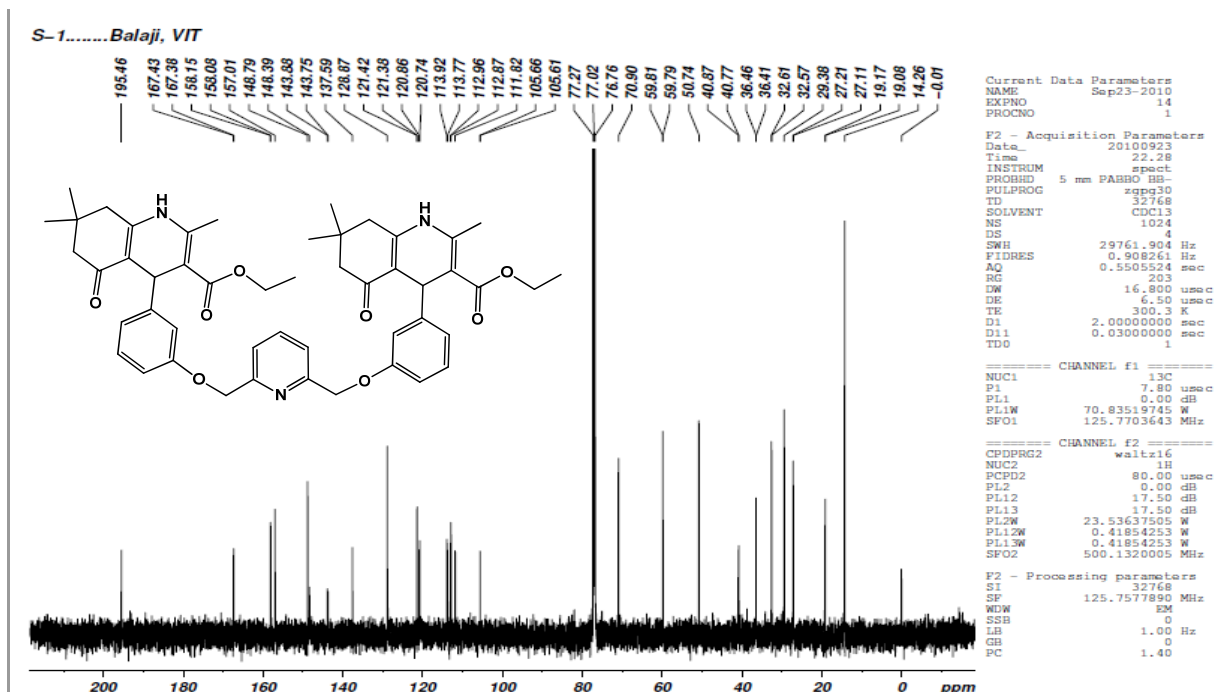
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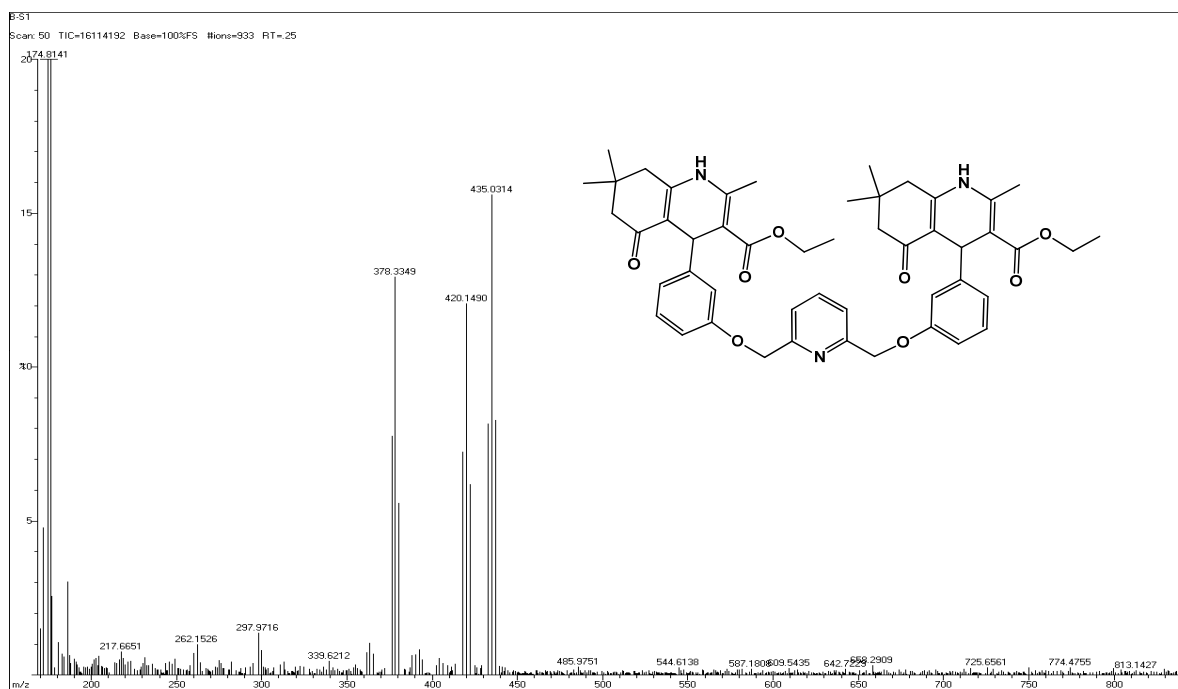
¹H NMR of Diethyl 4,4'-(3,3'-(pyridine-2,6-diylbis(methylene)bis(oxy)bis(3,1-phenylene))bis(2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate) [**10b**]



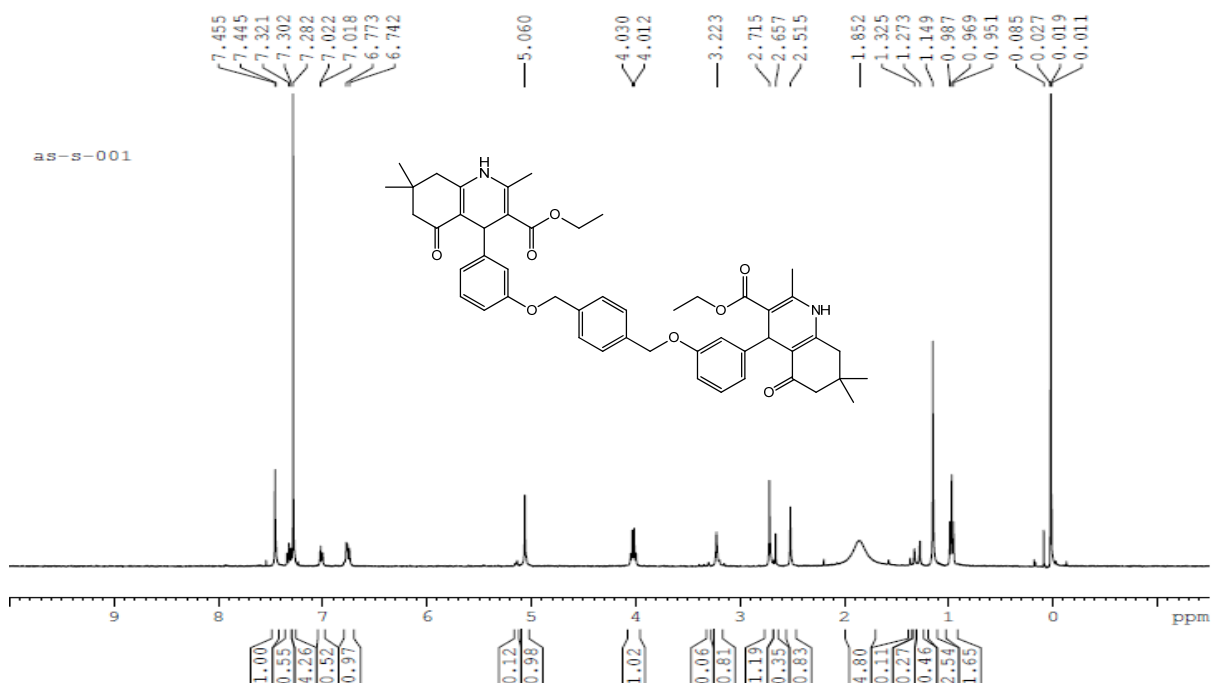
¹³C NMR of Diethyl 4,4'-(3,3'-(pyridine-2,6-diylbis(methylene)bis(oxy)bis(3,1-phenylene))bis(2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate) [**10b**]



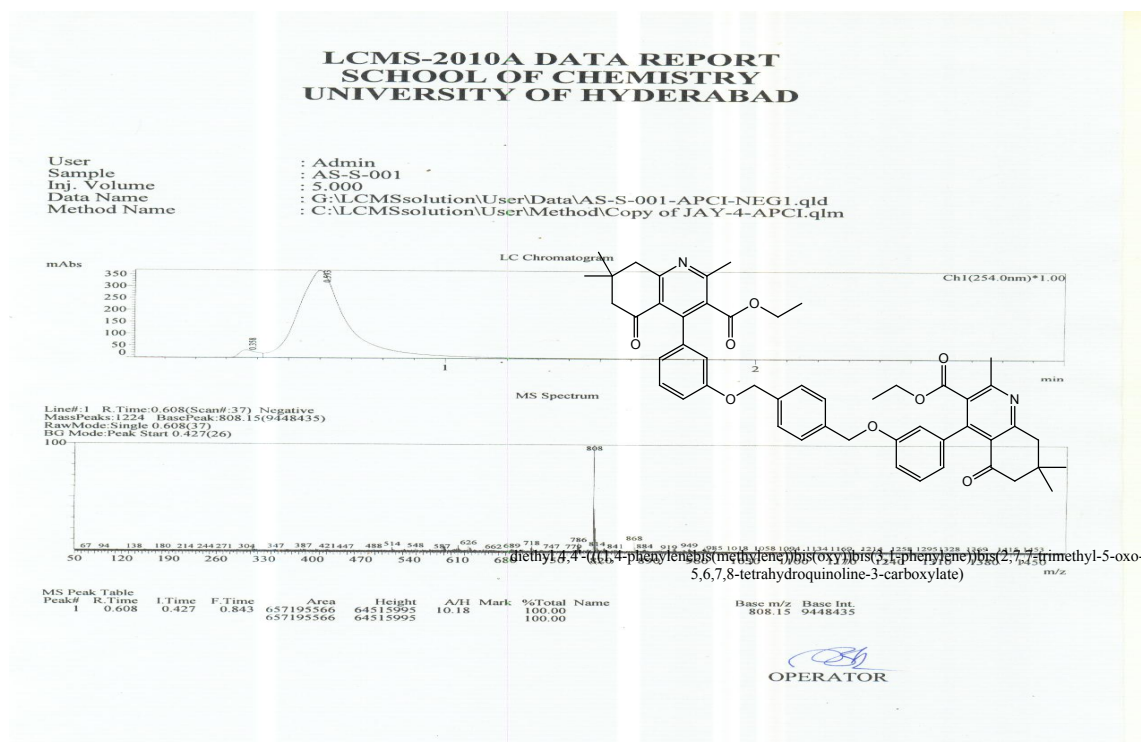
HR-MS of Diethyl 4,4'-(3,3'-(pyridine-2,6-diylbis(methylene)bis(oxy)bis(3,1-phenylene))bis(2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate) [**10b**]



^1H NMR of Diethyl 4,4'-(((1,4-phenylenebis(methylene))bis(oxy))bis(3,1-phenylene))bis(2,7,7-trimethyl-5-oxo-5,6,7,8-tetrahydroquinoline-3-carboxylate) [28]

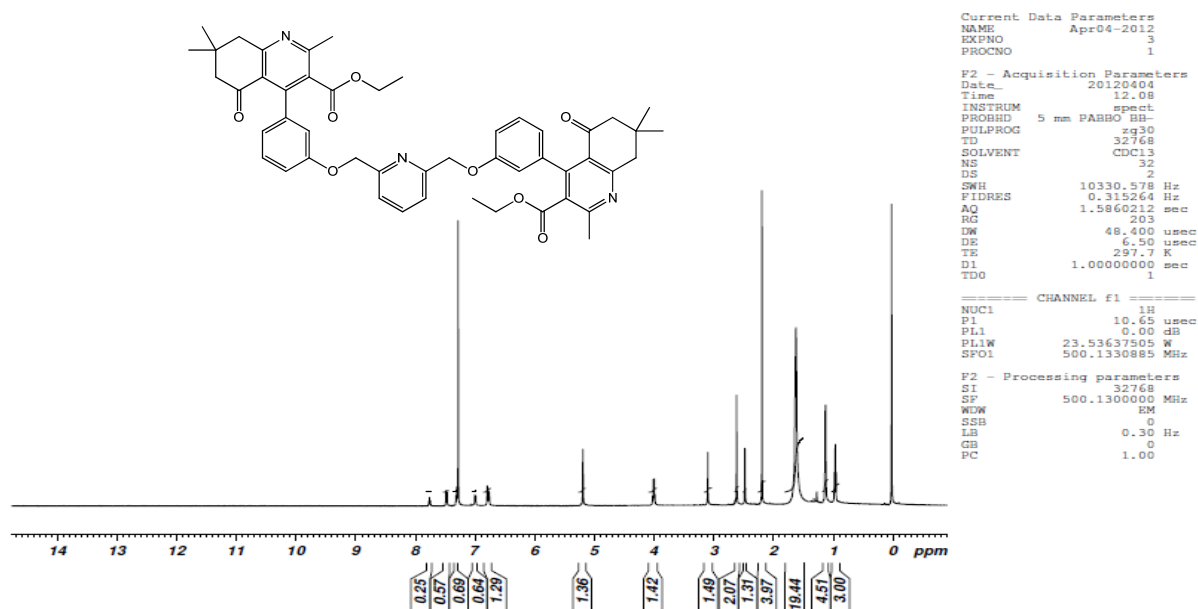


LC-MS of Diethyl 4,4'-(((1,4-phenylenebis(methylene))bis(oxy))bis(3,1-phenylene))bis(2,7,7-trimethyl-5-oxo-5,6,7,8-tetrahydroquinoline-3-carboxylate) [28]



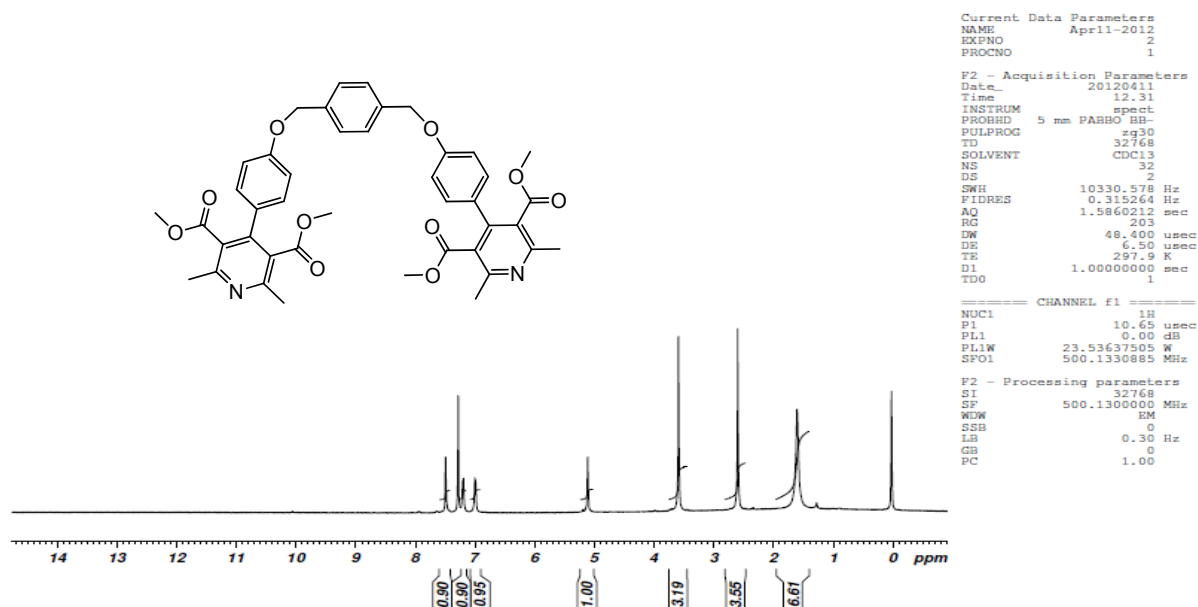
¹H NMR of Diethyl 4,4'-(((pyridine-2,6-diylbis(methylene))bis(oxy))bis(3,1-phenylene))bis(2,7,7-trimethyl-5-oxo-5,6,7,8-tetrahydroquinoline-3-carboxylate) [29a]

S-4.....Balaji.

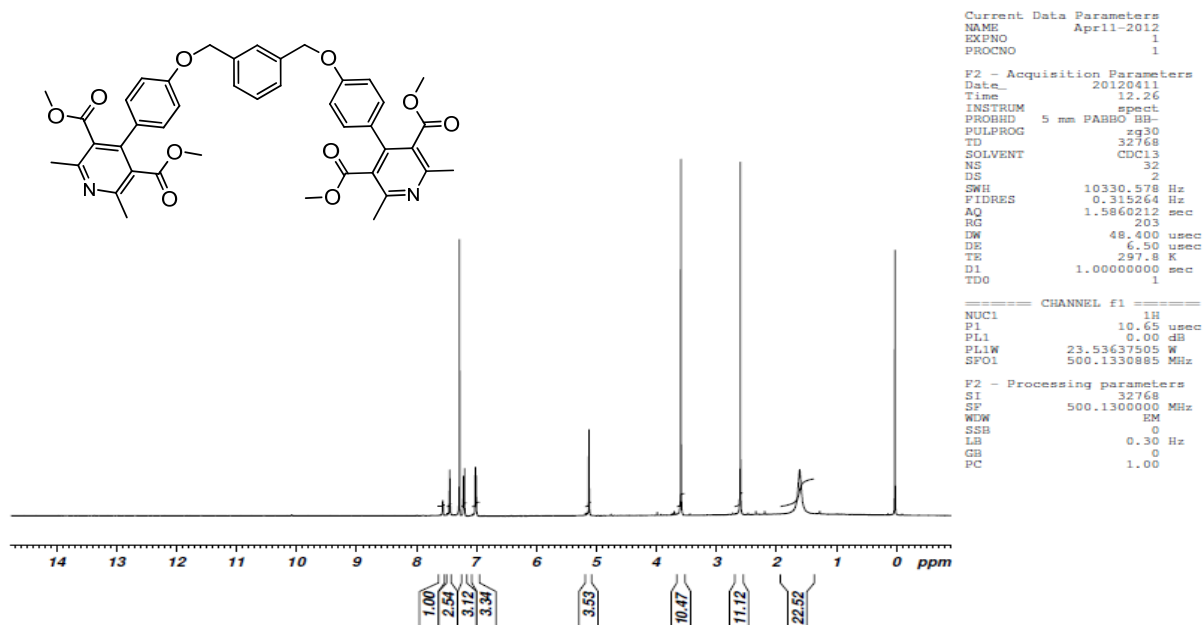


¹H NMR of Tetramethyl 4,4'-(((1,4-phenylenebis(methylene))bis(oxy))bis(4,1-phenylene))bis(2,6-dimethylpyridine-3,5-dicarboxylate) [33]

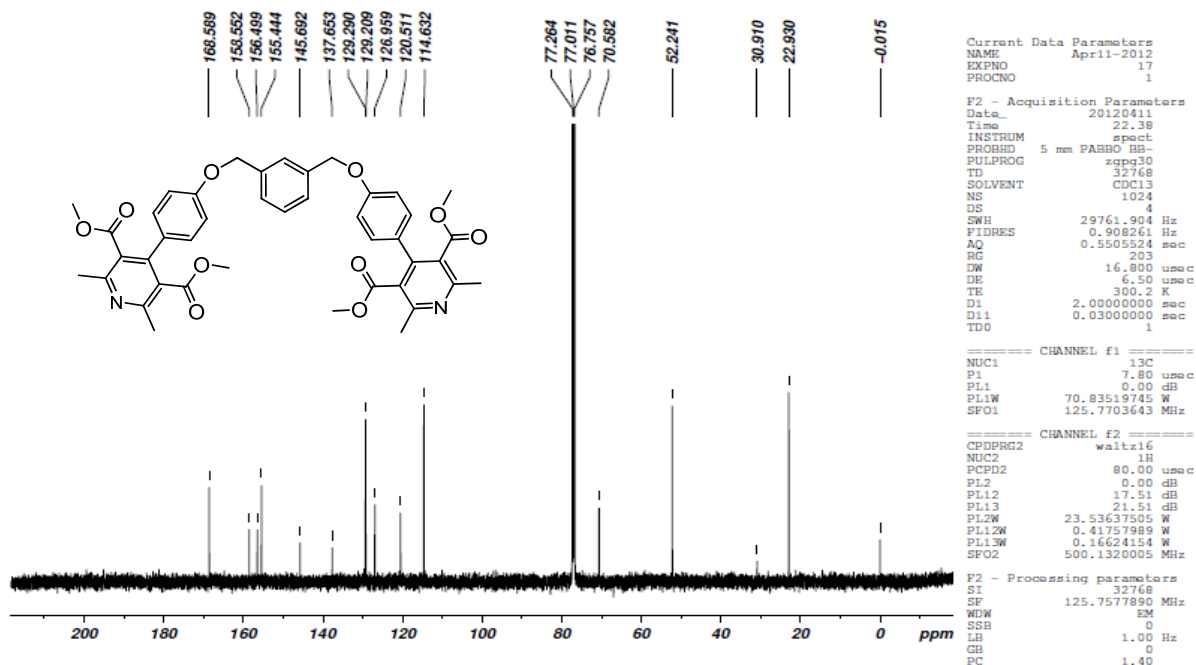
L-7.....Balaji.



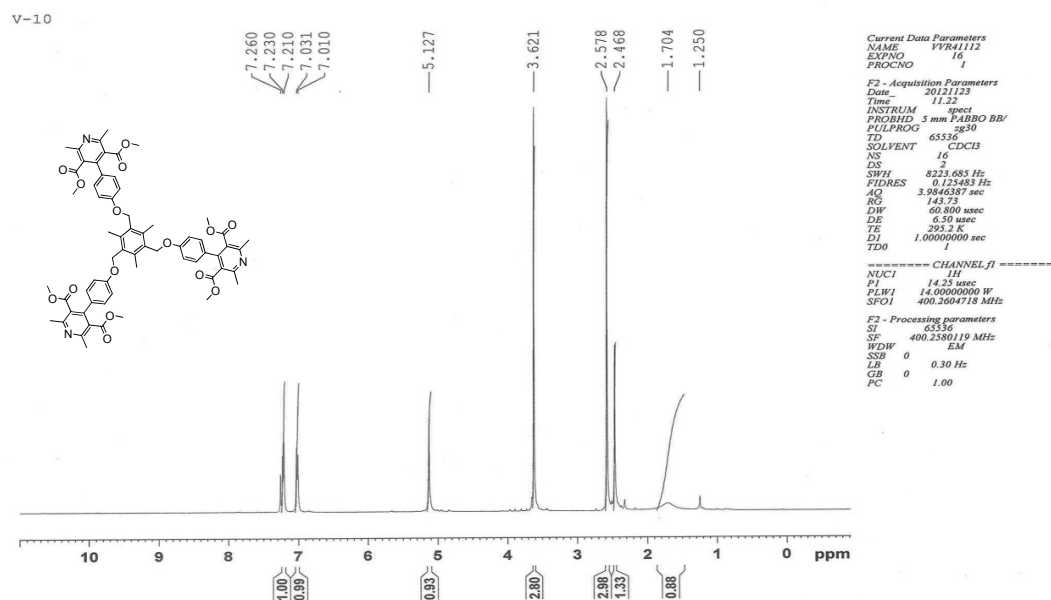
¹H NMR of Tetramethyl 4,4'-(((1,3-phenylenebis(methylene)))bis(oxy))bis(4,1-phenylene))bis(2,6-dimethylpyridine-3,5-dicarboxylate) [34]
L-4.....Balaji.



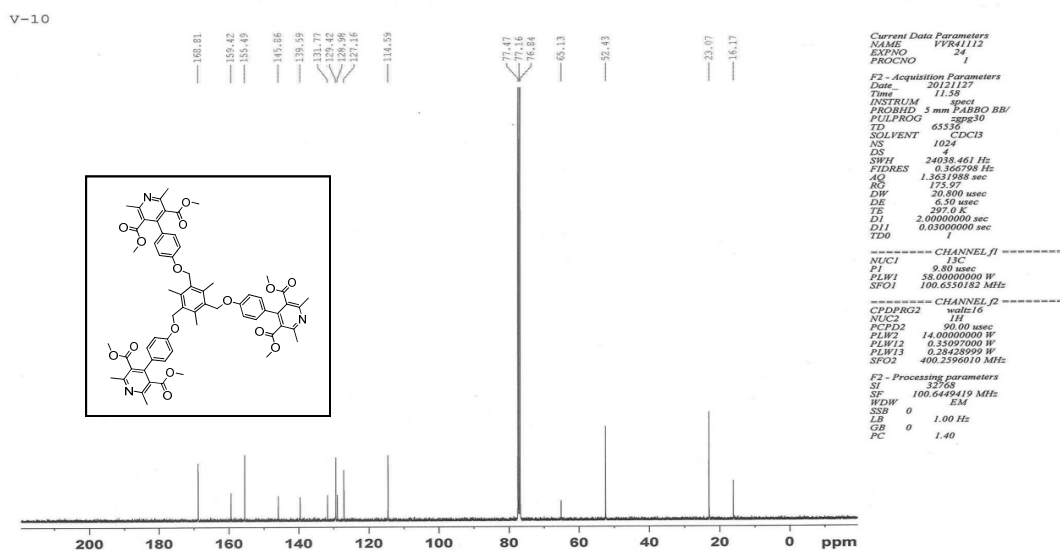
¹³C NMR of Tetramethyl 4,4'-(((1,3-phenylenebis(methylene)))bis(oxy))bis(4,1-phenylene))bis(2,6-dimethylpyridine-3,5-dicarboxylate) [34]
L-9.....Balaji.



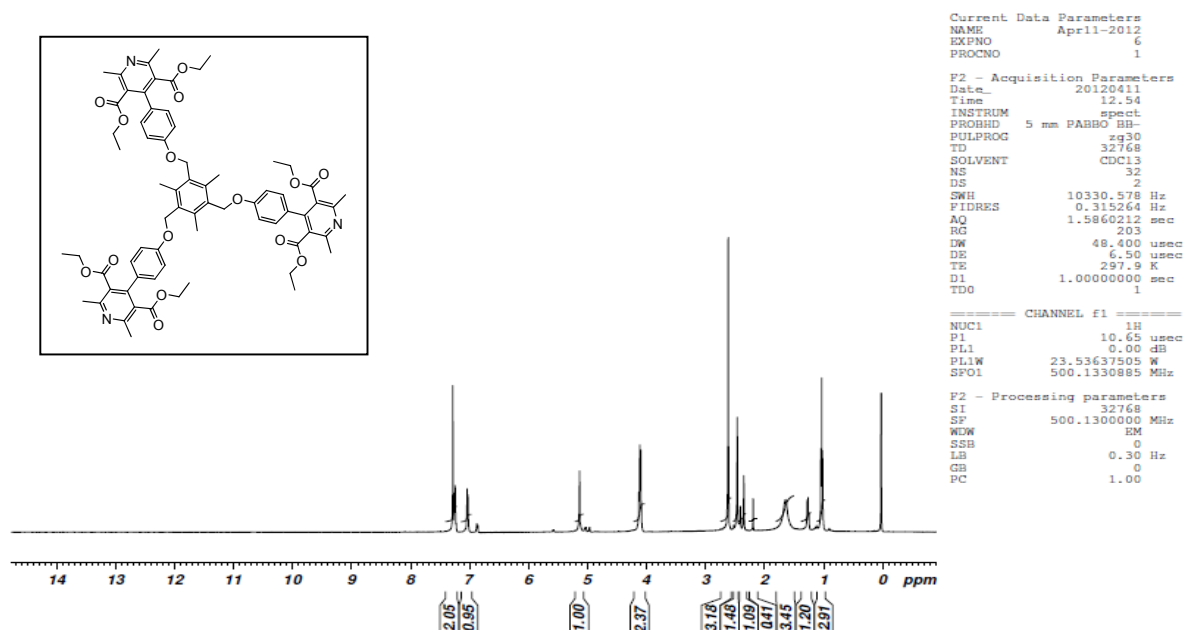
^1H NMR spectrum of Hexamethyl 4,4',4''-(((2,4,6-trimethylbenzene-1,3,5-triyl)tris(methylene))tris(oxy))tris(benzene-4,1-diyl))tris(2,6-dimethylpyridine-3,5-dicarboxylate) (**36**).



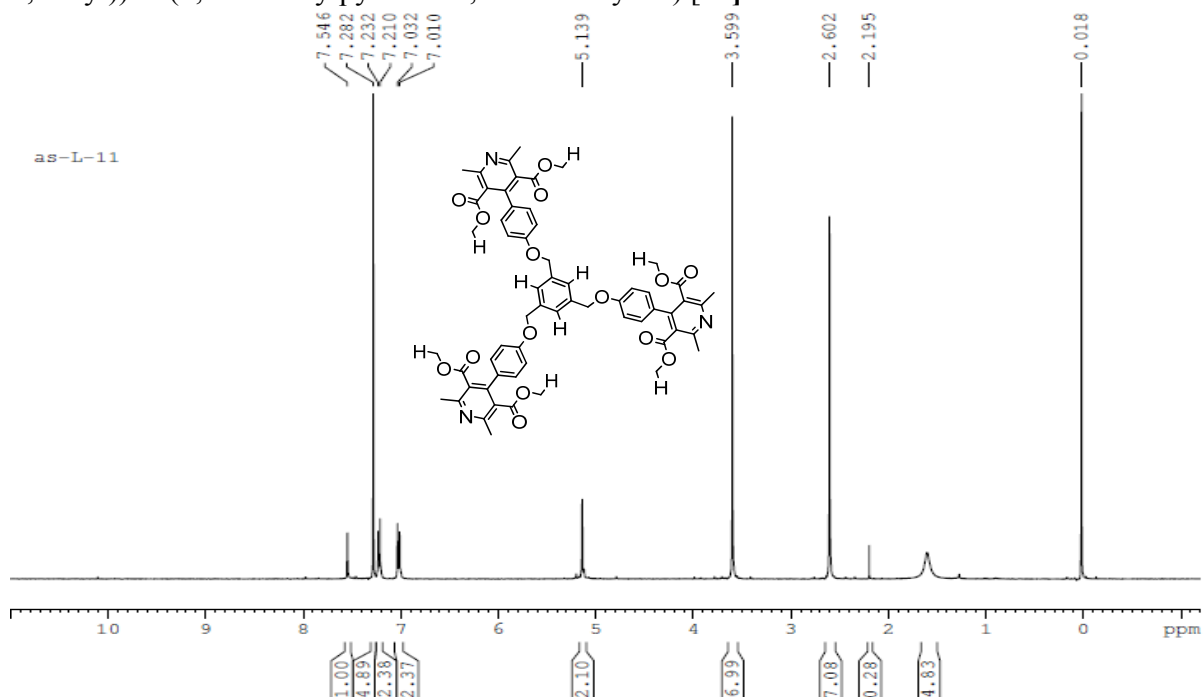
^{13}C NMR spectrum of Hexamethyl 4,4',4''-(((2,4,6-trimethylbenzene-1,3,5-triyl)tris(methylene))tris(oxy))tris(benzene-4,1-diyl))tris(2,6-dimethylpyridine-3,5-dicarboxylate) (**36**).



^1H NMR spectrum of Hexaethyl 4,4',4''-(((2,4,6-trimethylbenzene-1,3,5-triyl)tris(methylene))tris(oxy))tris(benzene-4,1-diyl))tris(2,6-dimethylpyridine-3,5-dicarboxylate) (**37**).

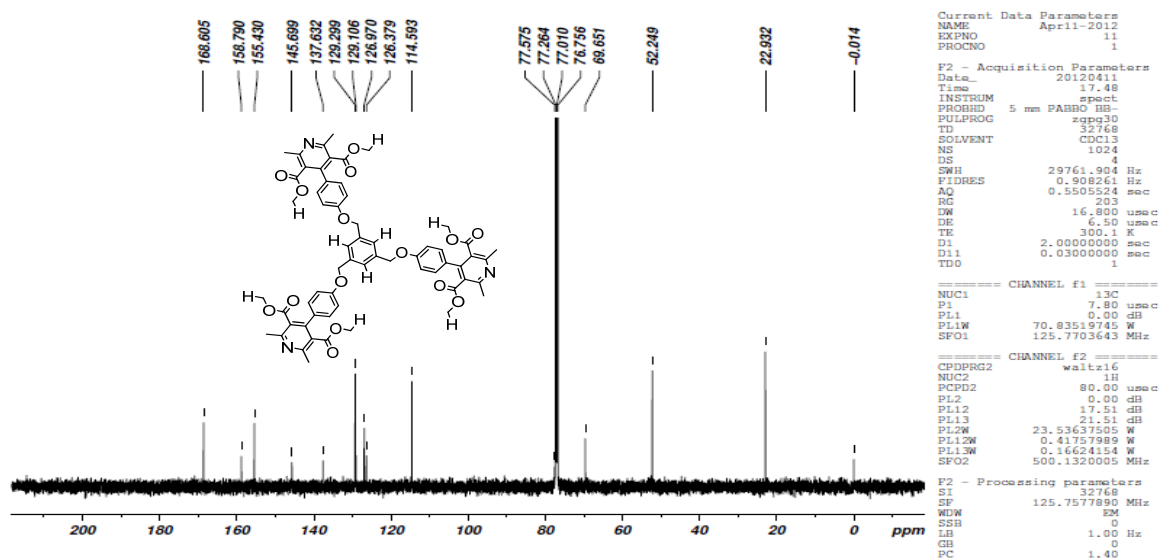


^1H NMR of Hexamethyl 4,4',4''-(((benzene-1,3,5-triyl)tris(methylene))tris(oxy))tris(benzene-4,1-diyl))tris(2,6-dimethylpyridine-3,5-dicarboxylate) [**38**]



¹³C NMR of Hexamethyl 4,4',4''-((((benzene-1,3,5-triyl)tris(methylene))tris(oxy))tris(benzene-4,1-diyl))tris(2,6-dimethylpyridine-3,5-dicarboxylate) [38]

L-11.....Balaji.



¹H NMR of Octaethyl 4,4',4'',4'''-(4,4',4'',4'''-(benzene-1,2,4,5-tetrayltetrakis(methylene))tetrakis(oxy) tetrakis(benzene-4,1-diyl))tetrakis(2,6-dimethyl-1,4-dihydropyridine-3,5-dicarboxylate) [39]

