Supplementary Information (SI) for RSC Advances.
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Supporting Information

Iodine-Catalyzed Green and Efficient Synthesis of Secondary and *Tert*-Esters of *N*-Acetyl-Protected Amino Acids

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1 Material and instrumentation

Acetyl chloride, glycine, leucine, phenylalanine, methionine, tryptophan and proline were purchased from Sigma-Aldrich, Merck, and Fluka. All commercial solvents were distilled and dried prior to use. Thin layer chromatography (TLC) with silica gel-coated plates (0.5 mm thick, Merck) was used to monitor the reaction progress, and spots were visualized under a UV lamp and ninhydrine solution. Synthesized compounds were recrystallized using ethyl acetate and ethanol. Physical properties such as the melting point were determined using the Gallen Kamp melting point apparatus. An FT-IR 6300 SHIMADZU spectrophotometer was used to record the IR spectrum of synthesized compounds. A Bruker Advance nuclear magnetic resonance spectrometer resonating at 400 MHz was used for the structural elucidation of targeted compounds in $CDCl_3$ as the solvent and TMS as the internal standard.

1.1 Synthesis of Acetylated Amino Acids (3a-f)

5-7 mmol of L-amino acids (1a-f) were dissolved in water, and an equimolar amount of acetyl chloride was added, heating it for 2 hours at 70ºC. *N*-acetyl amino acid formation was monitored by TLC and then by ninhydrin test, and the solvent was evaporated using a rotary evaporator. The crude product was dissolved in methanol and filtered to remove any unreacted amino acid. Methanol was removed till dryness under vacuum to get pure precipitates of *N*-acetyl amino acids.

Table 1: Physical and Chemical Parameters of Acetylation of Amino Acids

Compound	Yield %	m.p (°C)	[α] D20 (C=0.5g. 50mL Methanol)
3a	58	208-210	-
3b	94	190-195	-24.32
3c	96	176-178	+39.47
3d	93	103-105	-8.2
3e	96	182-188	+27.46
3f	91	117-120	-86

1.2 Synthesis of *N*-Protected Amino Esters:

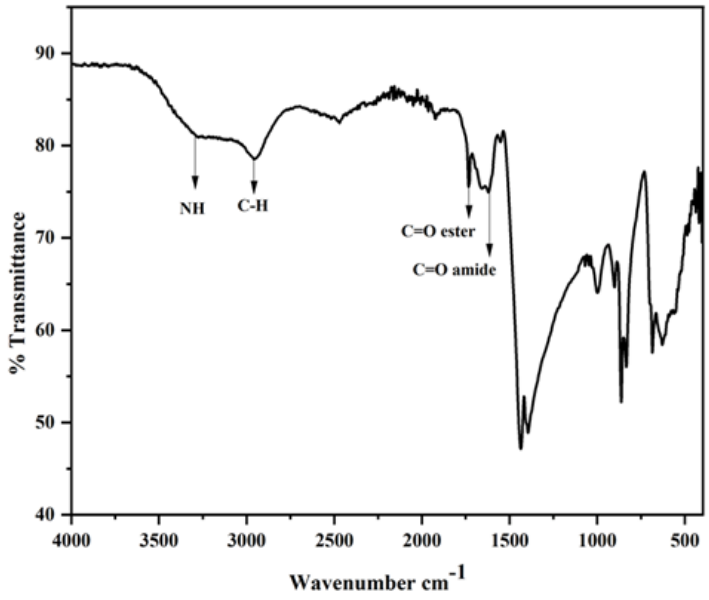
N-Protected amino acids (0.5g, 2.88 mmol) were dissolved in excess alcohol, followed by the addition of I_2 (25 mg, 0.01 equiv; doubled for *tert*- butyl esters) under stirring. The reaction mixture was refluxed for 6-8 hours and monitored by TLC. After completion, excess alcohol was removed under reduced pressure. The residue was extracted with diethyl ether or DCM, and the organic layer was washed successively with sodium thiosulfate, sodium bicarbonate, and water. It was then dried over anhydrous Na_2SO_4 or $MgSO_4$ and concentrated under vacuum. This general procedure was applied to all substrates for esterification. In case of DMAP, 1N HCl was used during work.

Table 2: Physical and chemical parameters of N-Acetylated Amino Esters

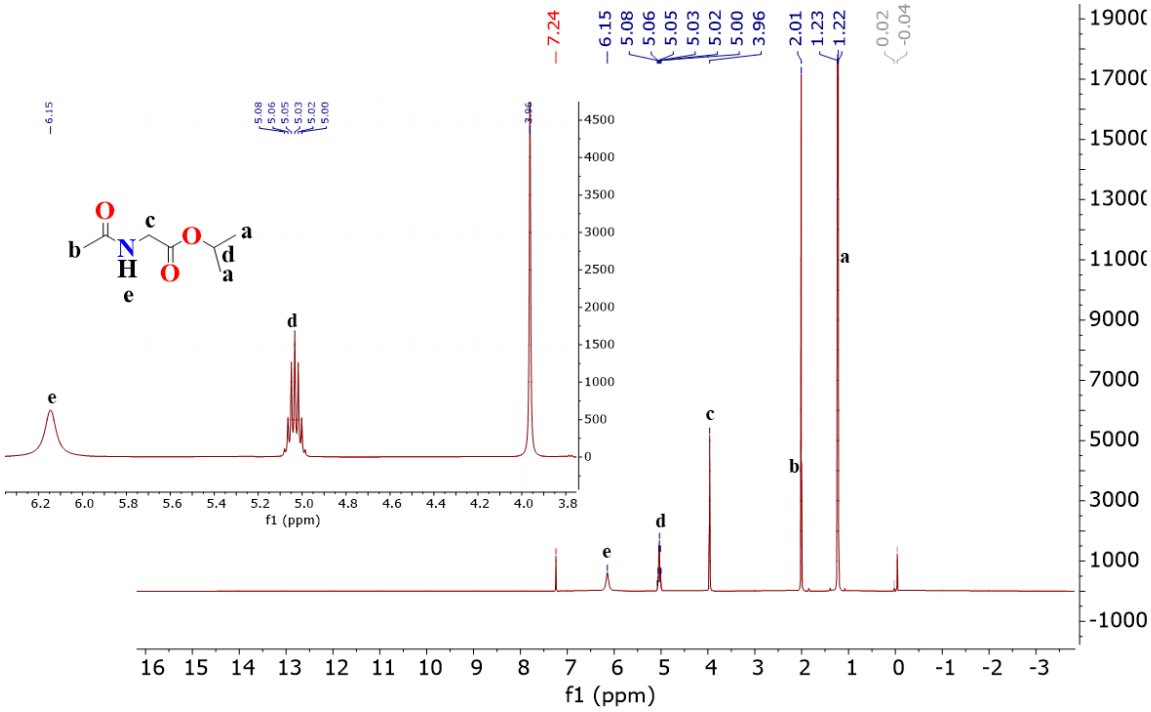
Compound	Melting Point °C	Yield %	[α] D^{20} (C=0.05g. 50mL Methanol)
$5a_1$	58-60	75	-
$5a_2$	47-50	80	-54
$5a_3$	68-70	77	+40.1
$5a_4$	80-82	80	-28.03
$5a_5$	208-214	80	+18.98
$5a_6$	72-73	70	-35.23
$5b_1$	47-50	78	-
$5b_2$	60-63	85	+22.34

5b$_3$	85-90	78	+13.2
5b$_4$	102-104	72	+25.15
5b$_5$	109-111	78	+22.40
5b$_6$	64-65	69	-9.3
5c$_1$	Oily liq.	76	-
5c$_2$	Semi-solid liq.	80	+9.27
5c$_3$	62-64	78	-0.20
5c$_4$	Oily liq.	80	-0.50
5c$_5$	150-152	74	-25.50
5c$_6$	Oily liq.	68	+11.62
5d$_1$	Oily liq.	70	-
5d$_2$	Oily liq.	75	+13.22
5d$_3$	Oily liq.	73	-1.23
5d$_4$	Oily liq.	75	+20.28
5d$_5$	Oily liq.	70	+9.33
5d$_6$	Oily liq.	60	-32.32

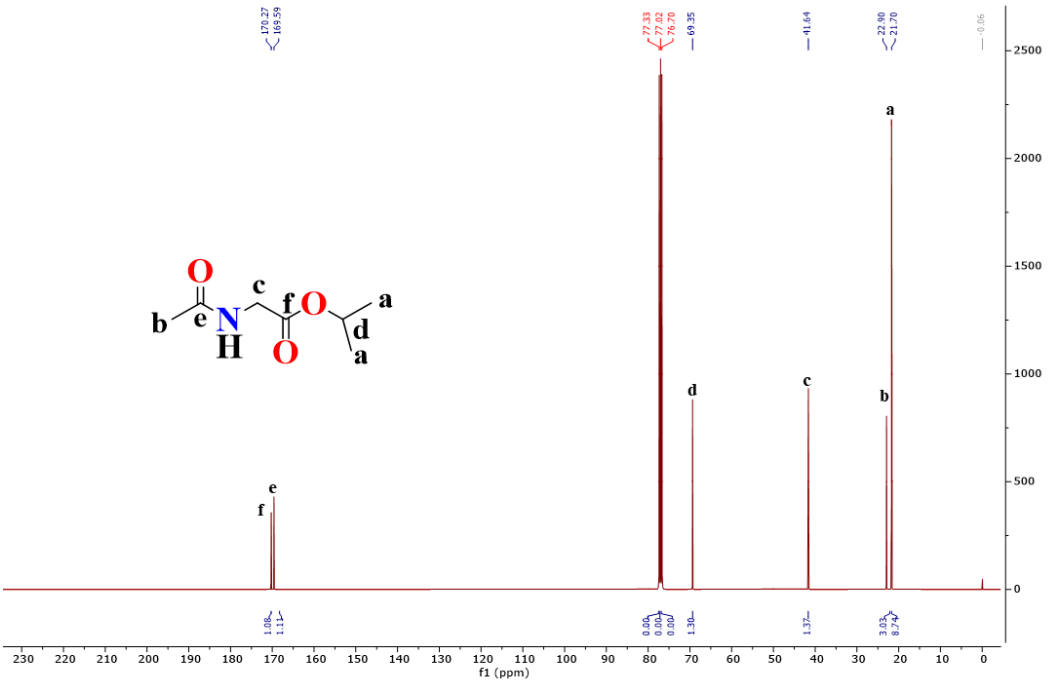
Spectroscopic Data of Secondary Esters of *N*-Acetylated Amino Acids



S 1: FT-IR of 5c1



S1. ^{1}HNMR 5c1



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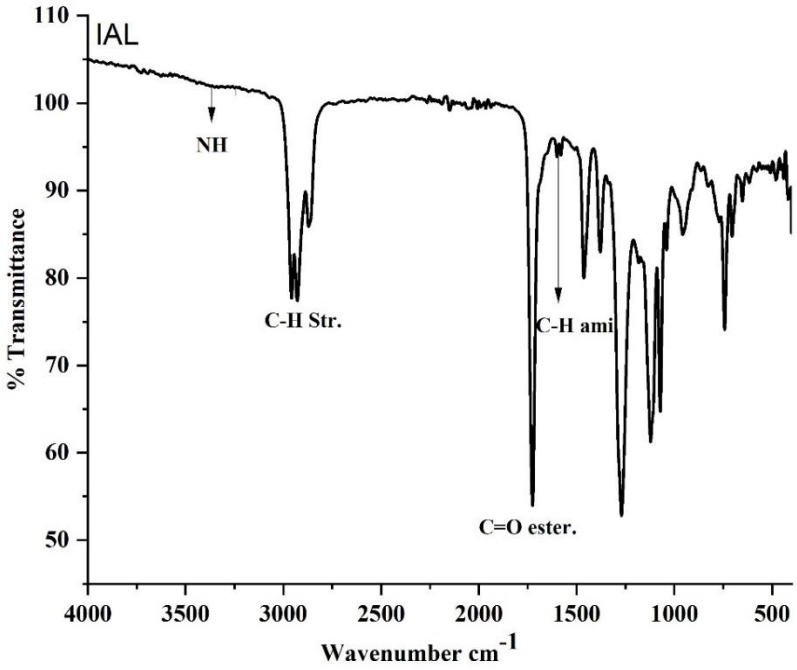

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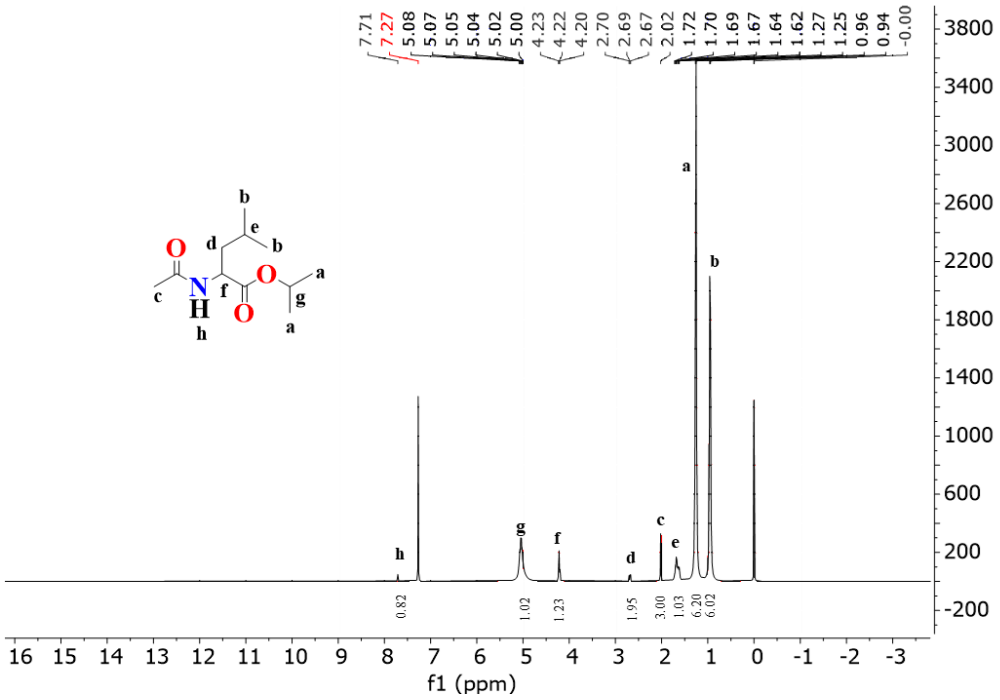
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7.27
5.08
5.07
5.05
5.04
5.02
5.00
4.23
4.22
4.20
2.70
2.69
2.67
2.02
1.72
1.70
1.69
1.67
1.64
1.62
1.27
1.25
0.96
0.94
-0.00
f1 (ppm)

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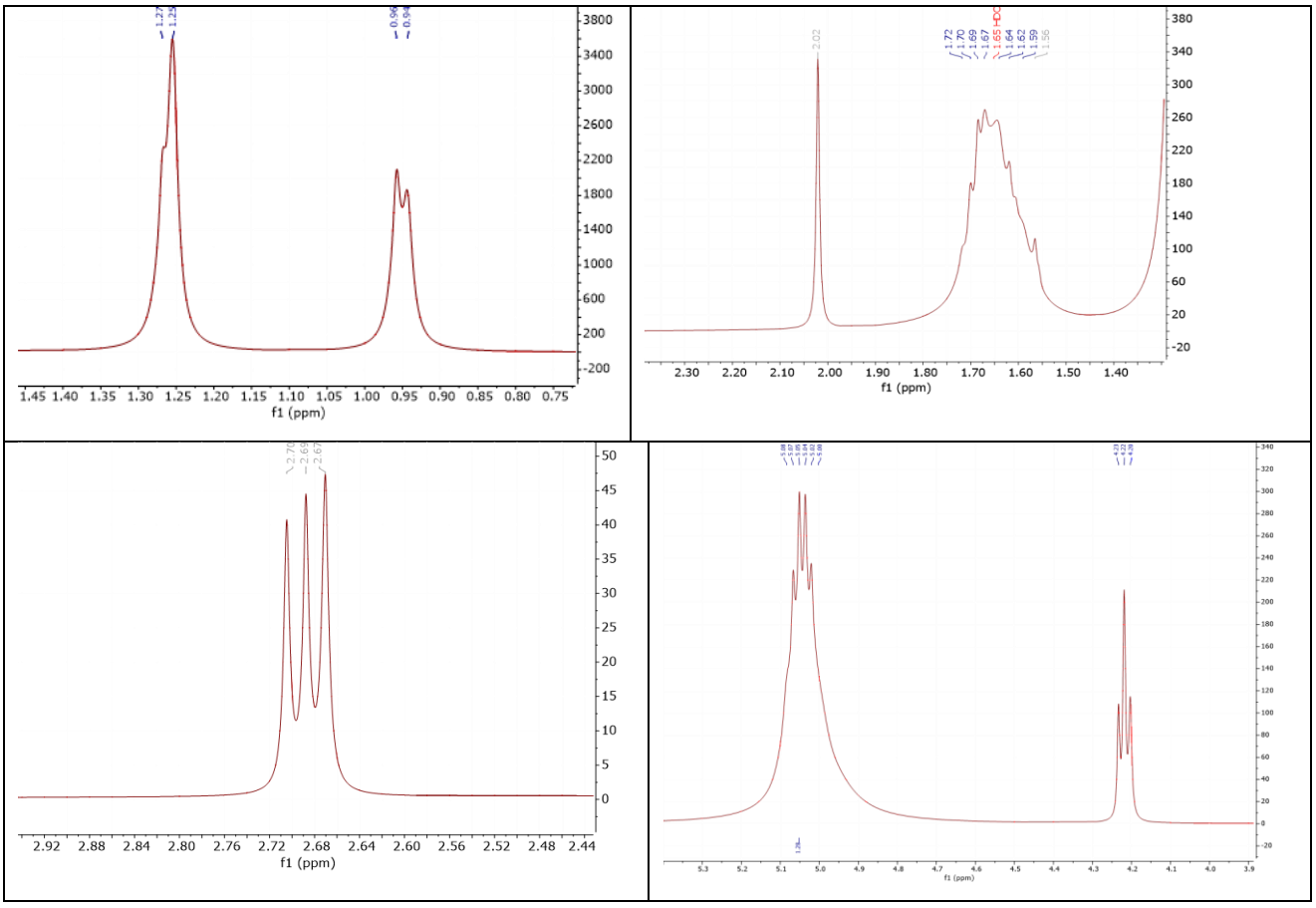
f1 (ppm)
f1 (ppm)
f1 (ppm)

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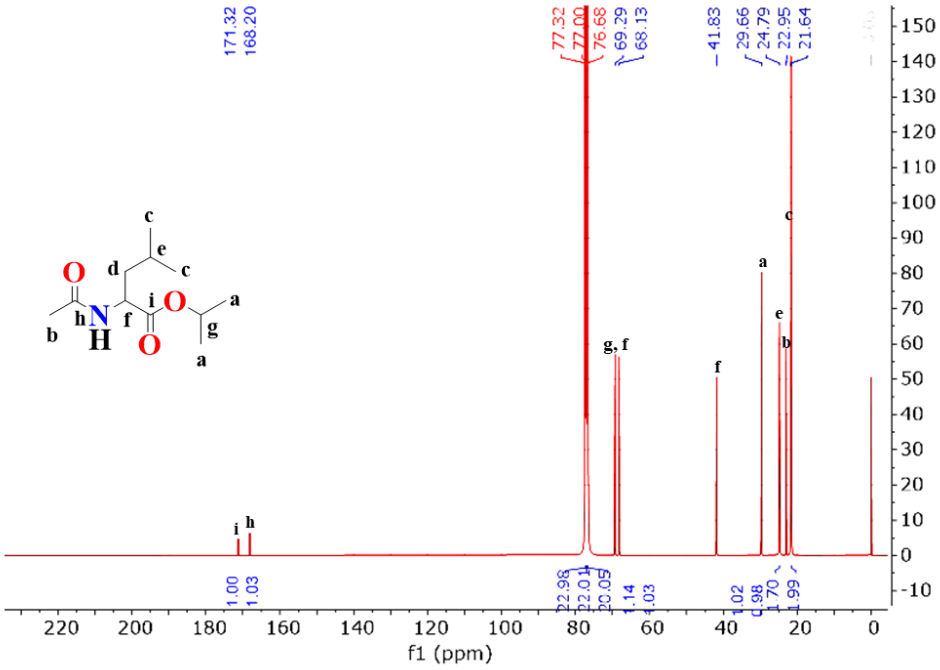

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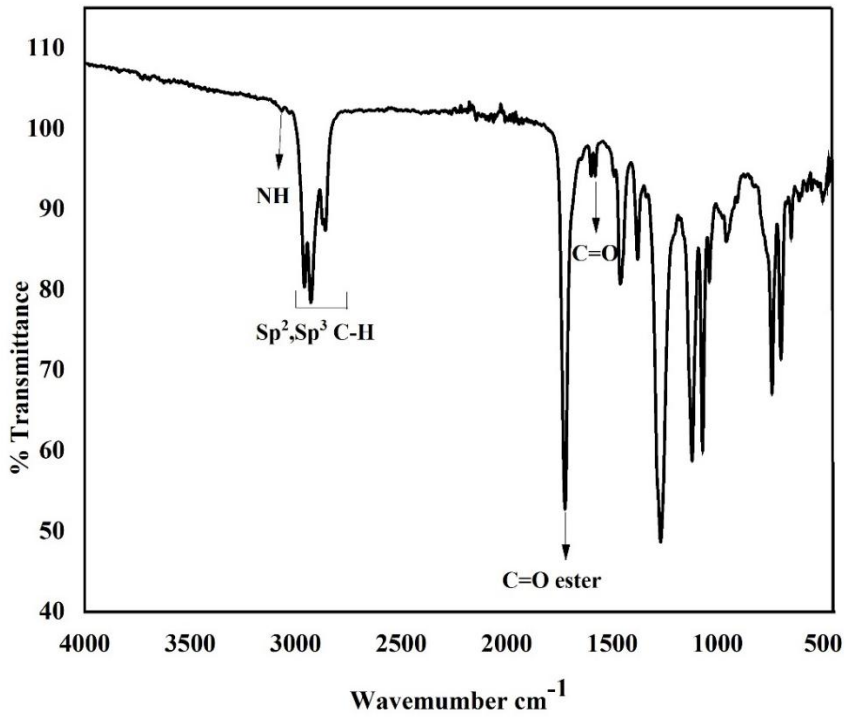

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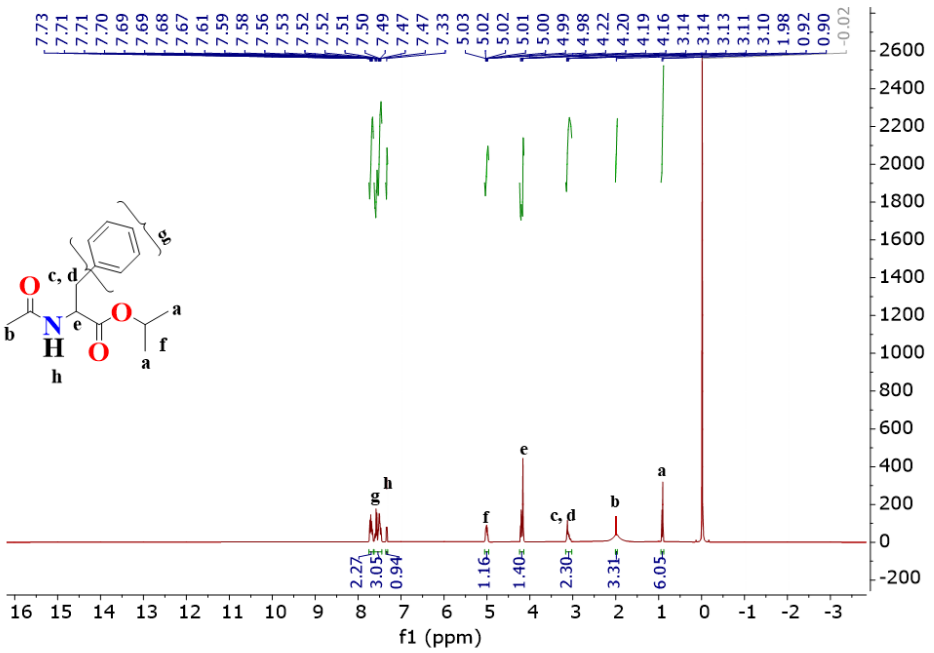

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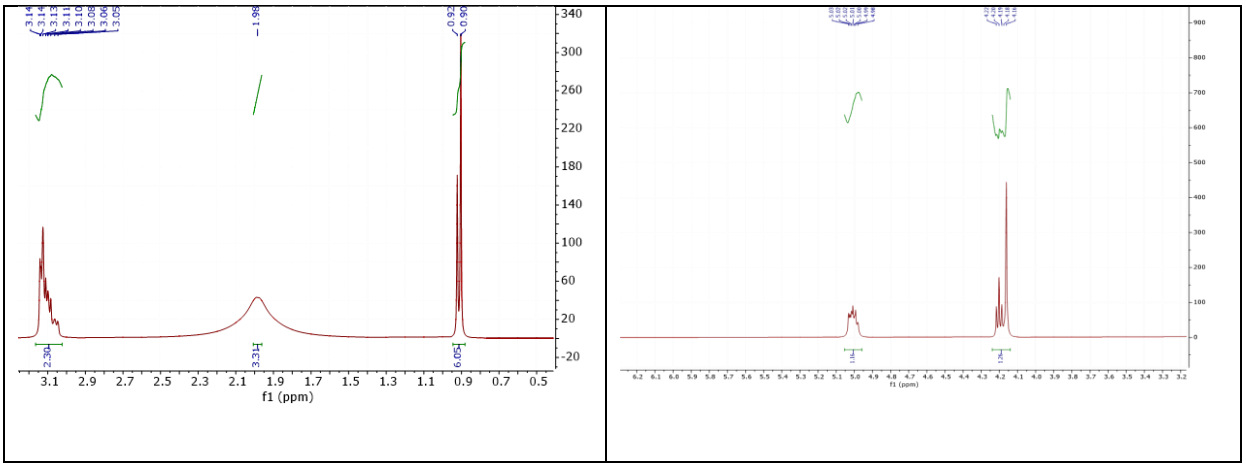

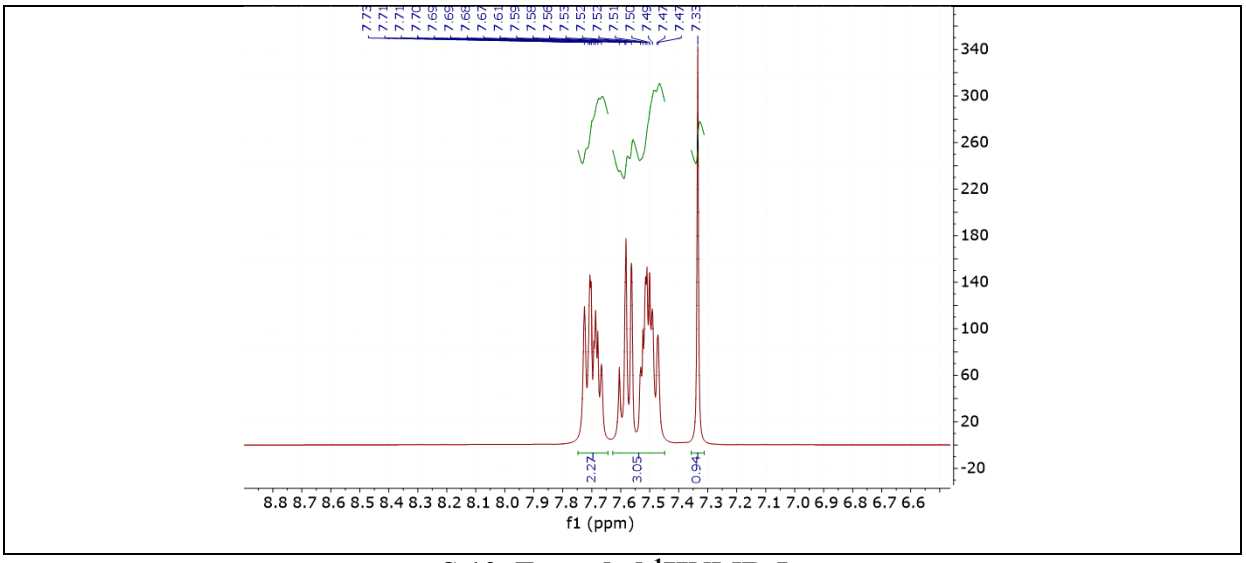

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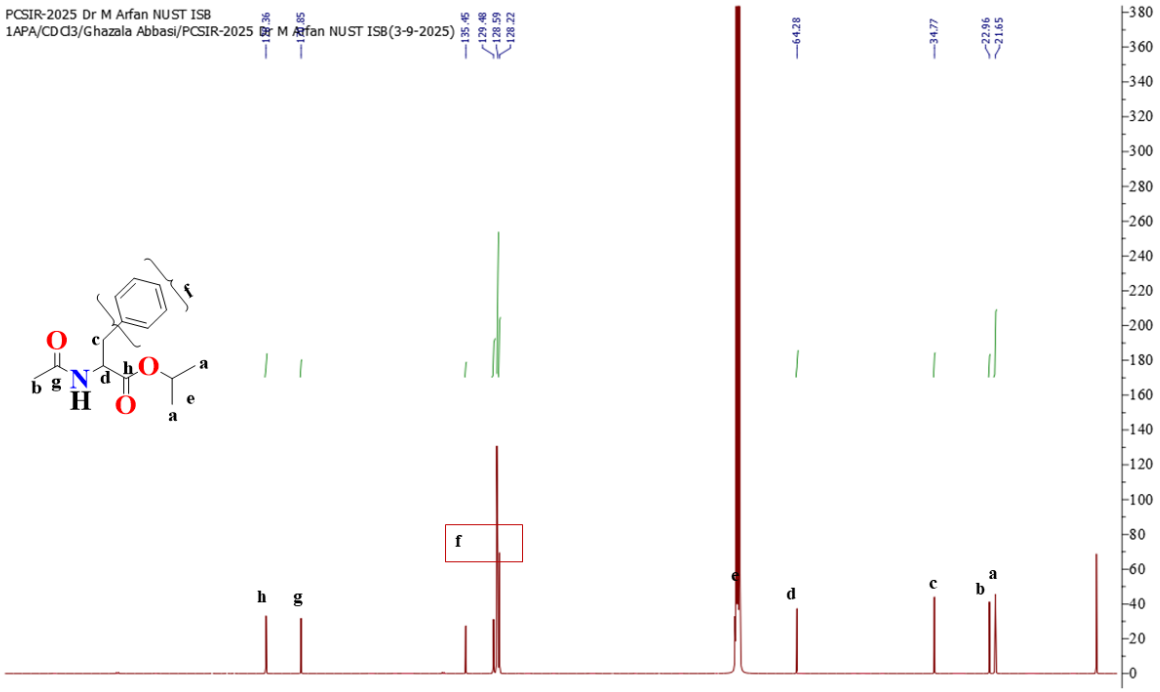

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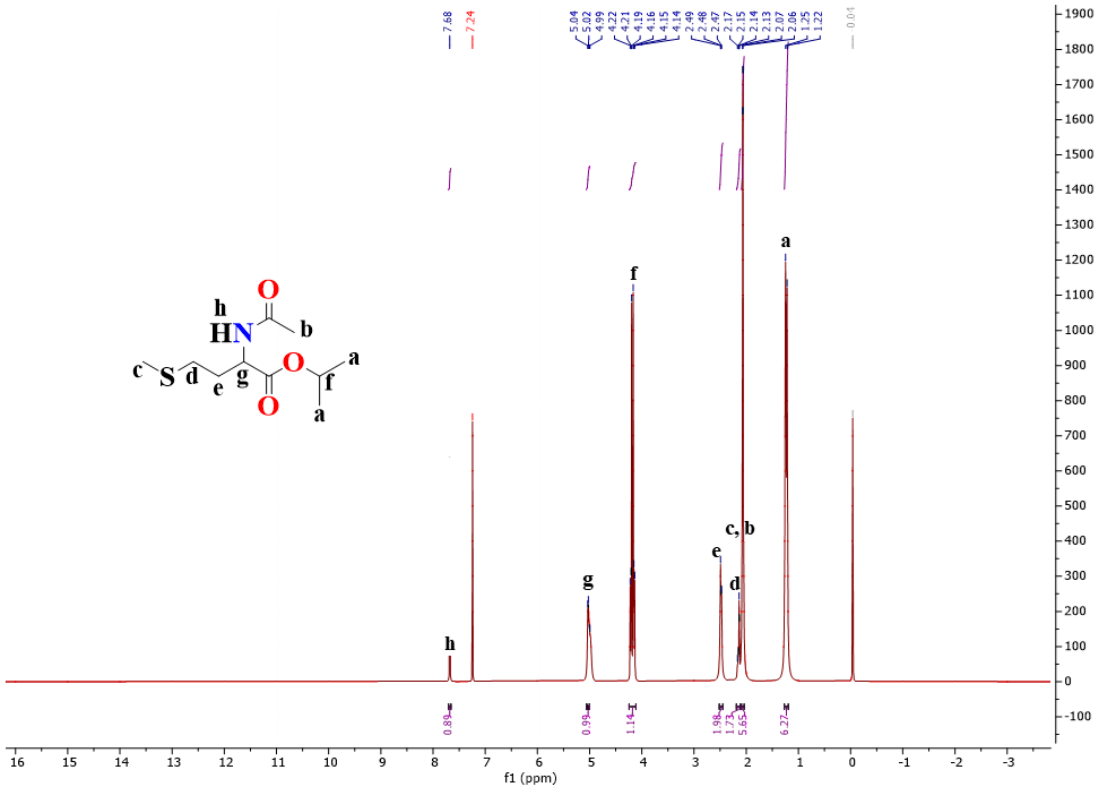

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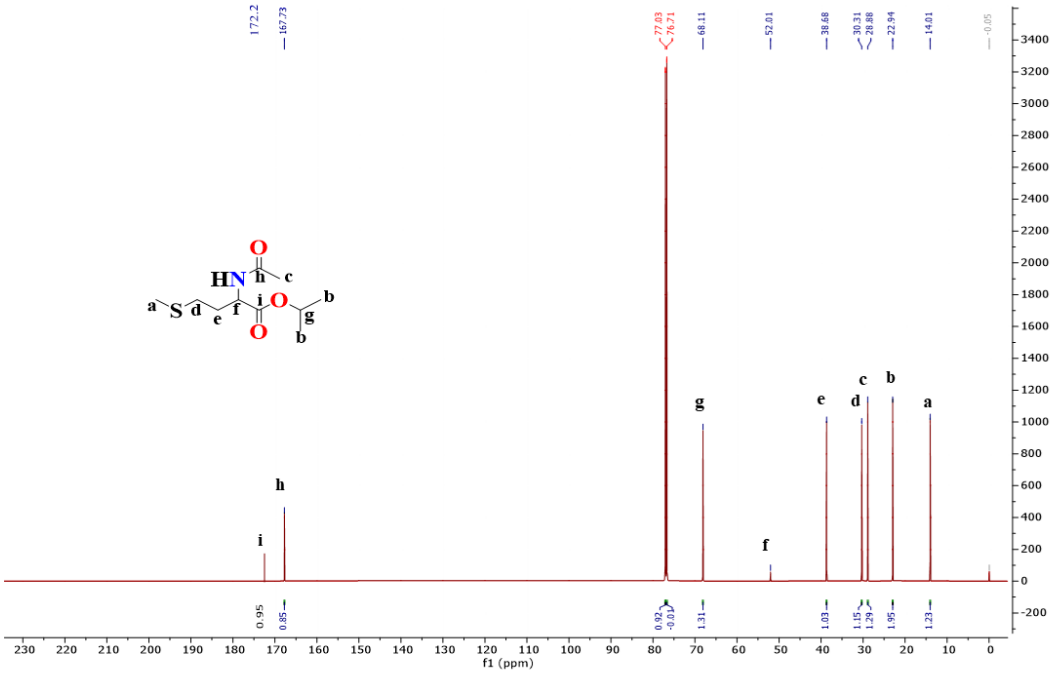

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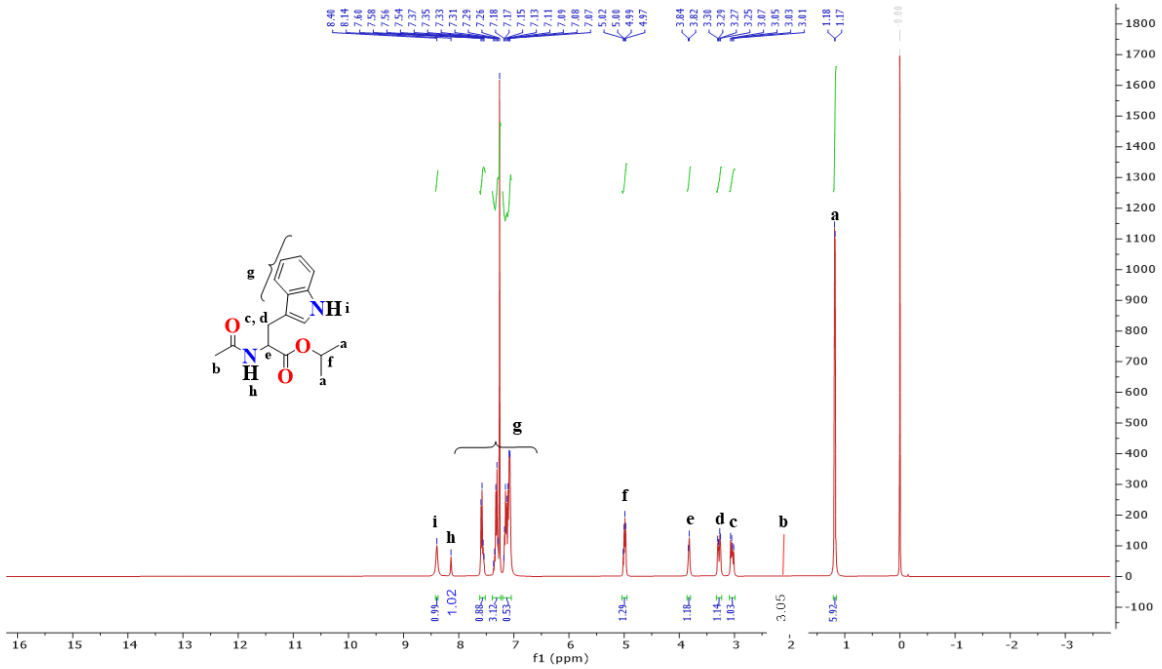

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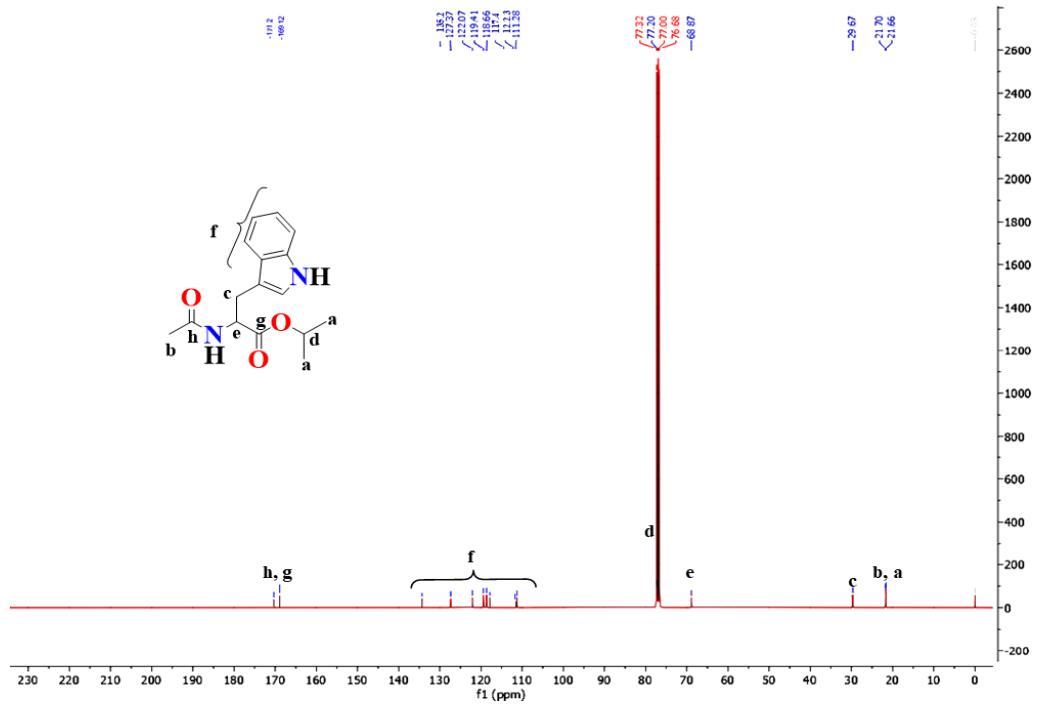

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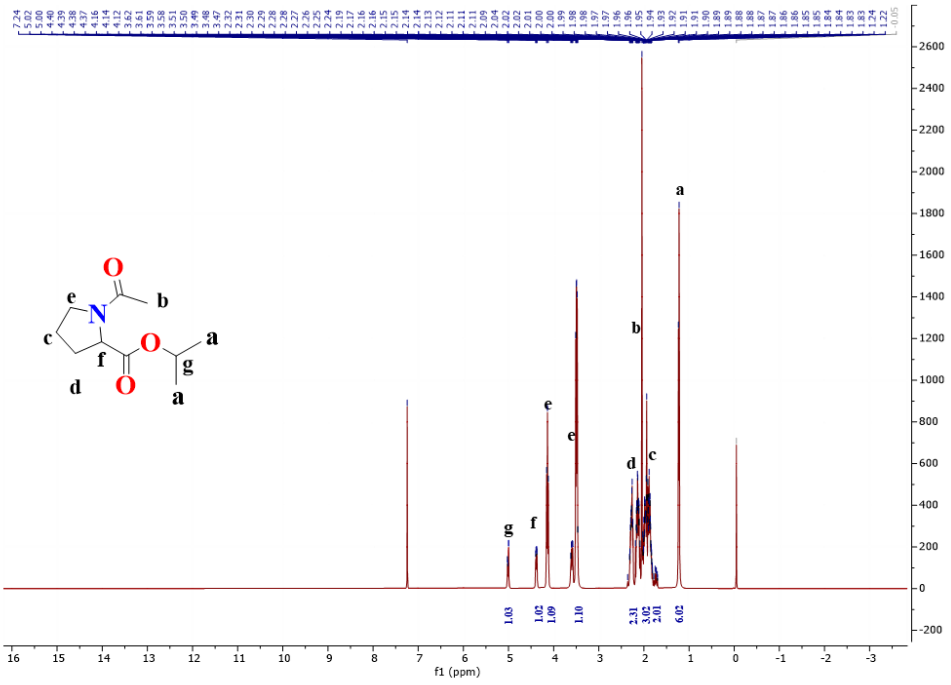

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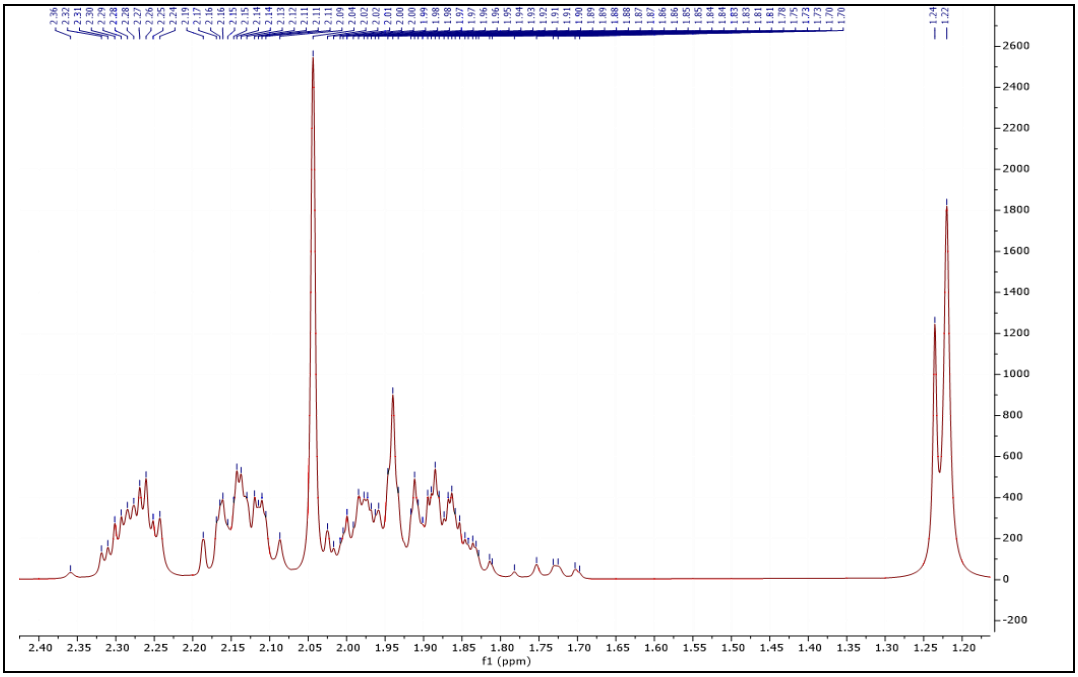

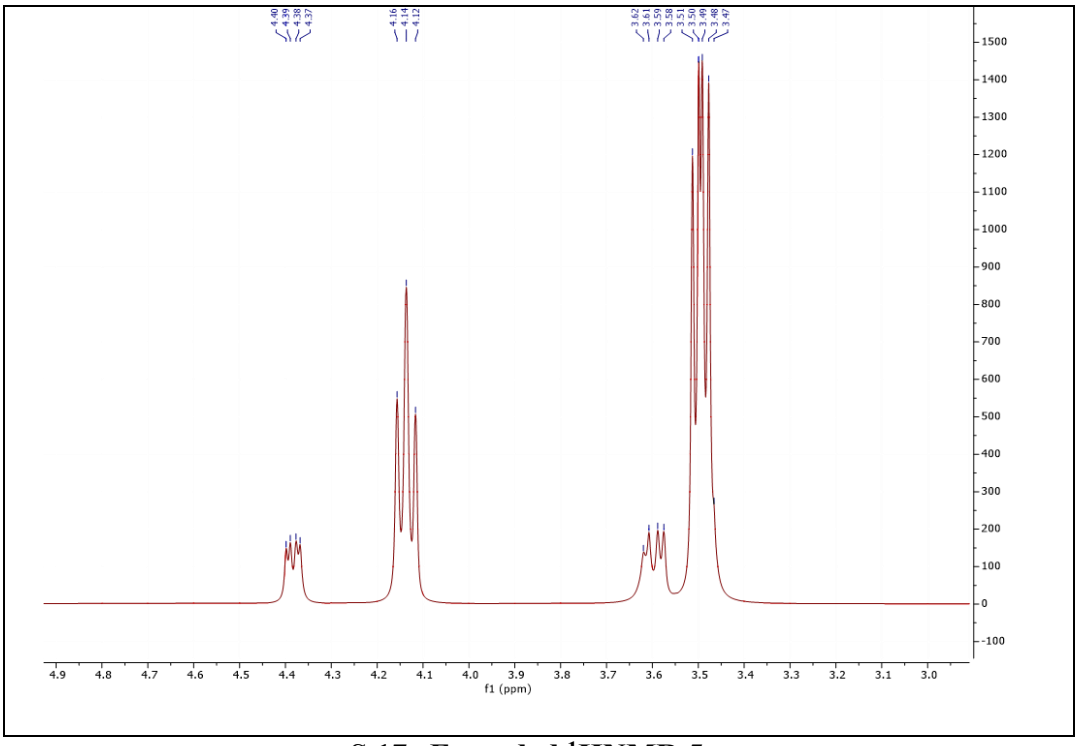

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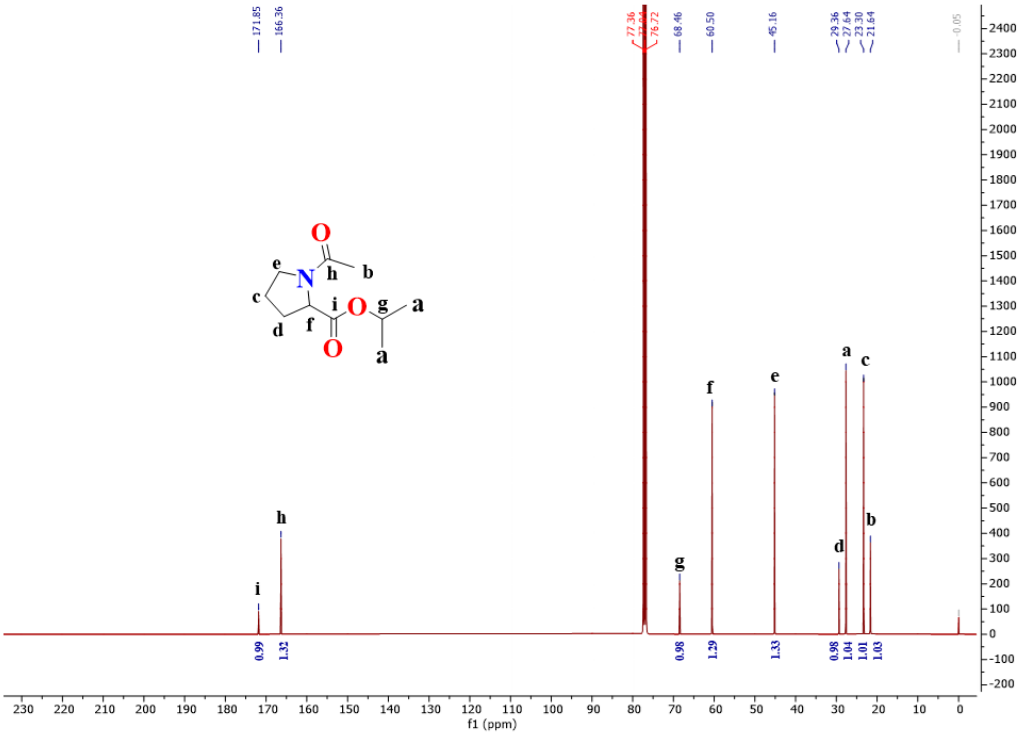
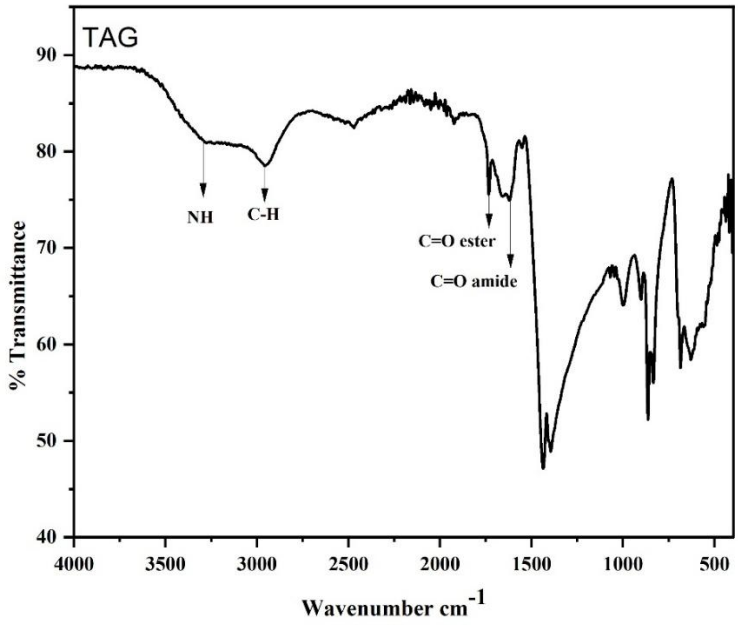
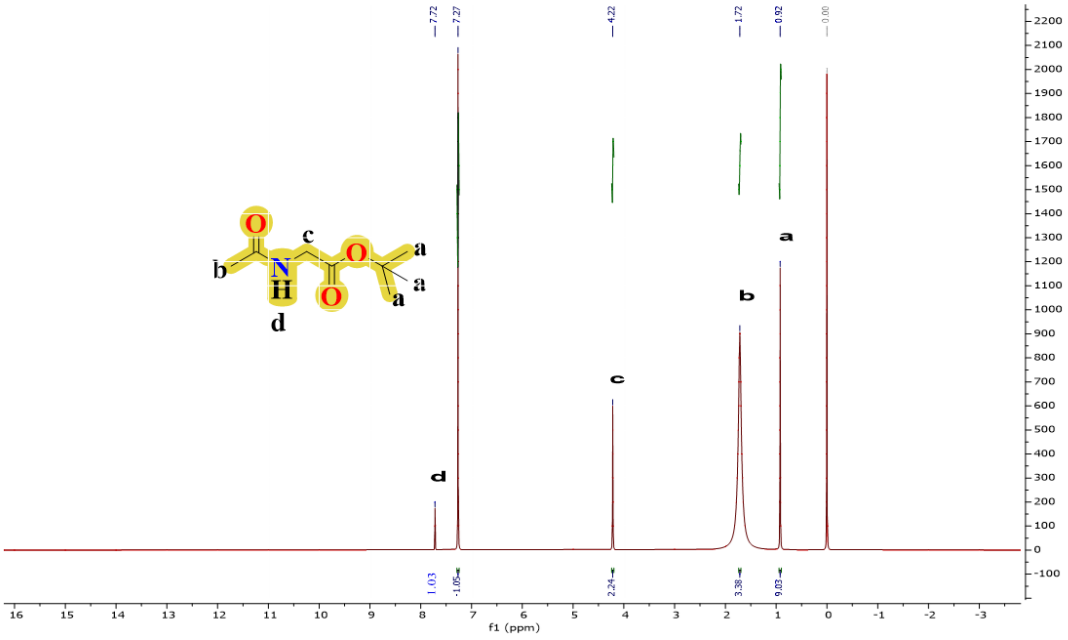


S 18: ^{13}CNMR 5c$_6$

Spectroscopic Data of *Tert*-butyl Esters of *N*-Acetylated Amino Acids



S 19: FT-IR of 5d$_1$



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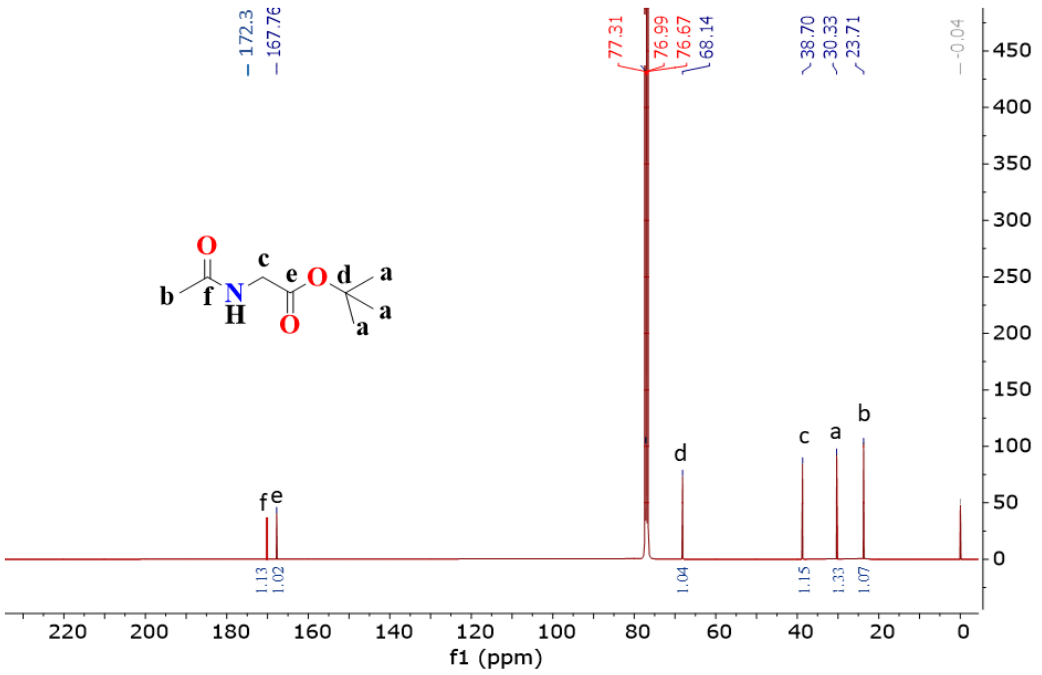

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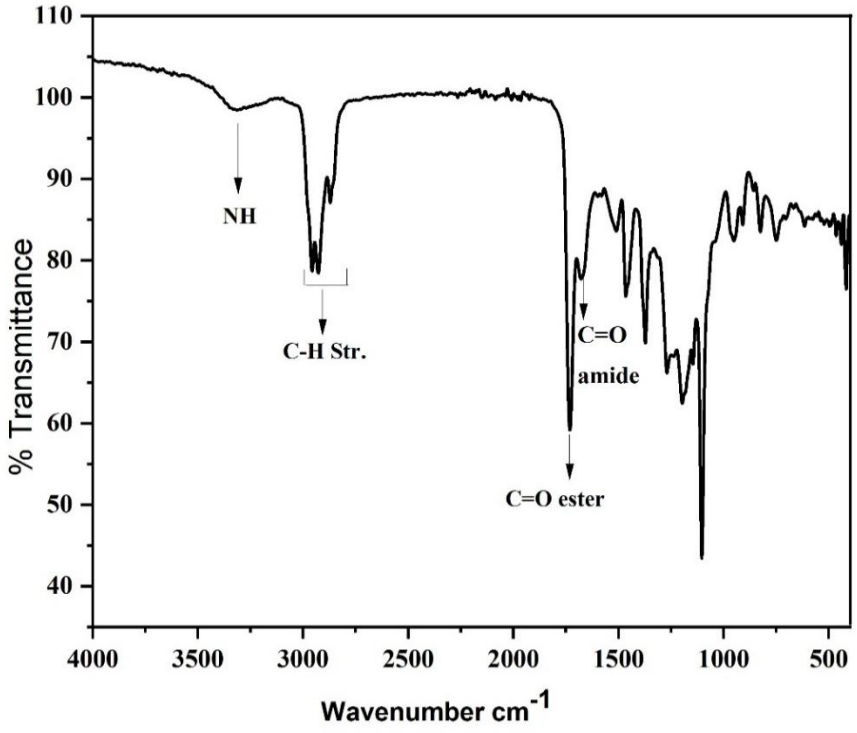

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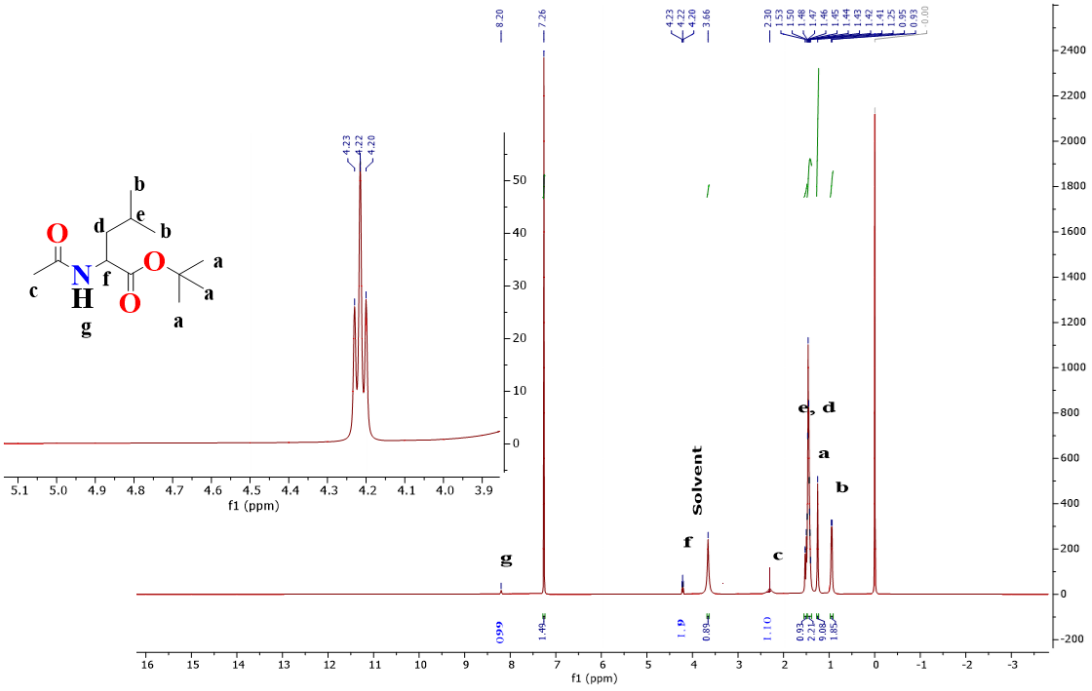

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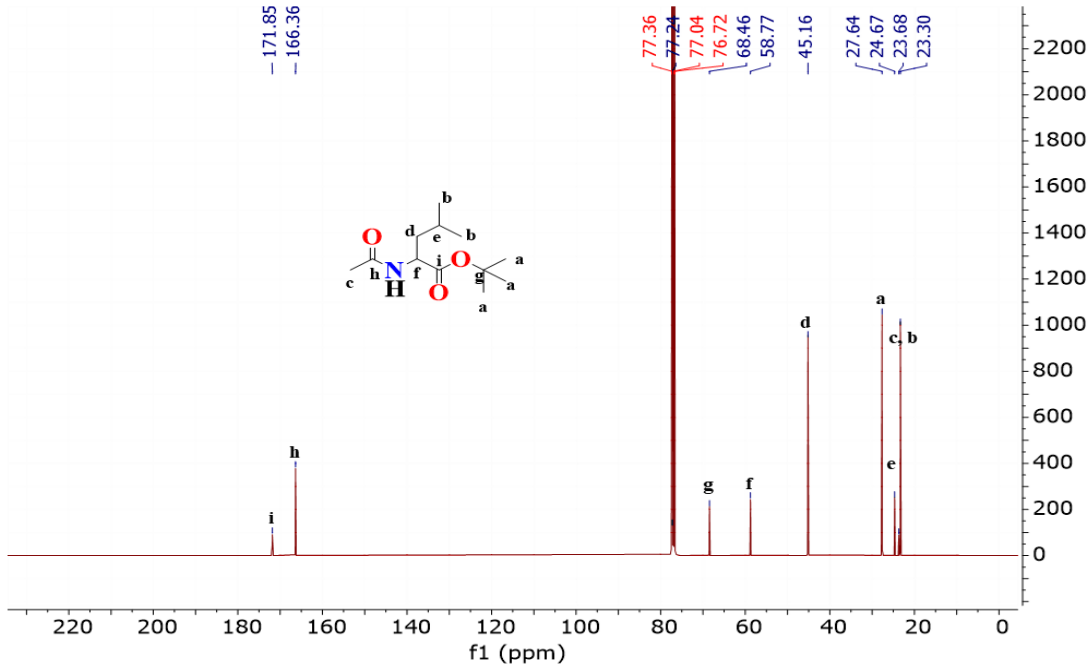

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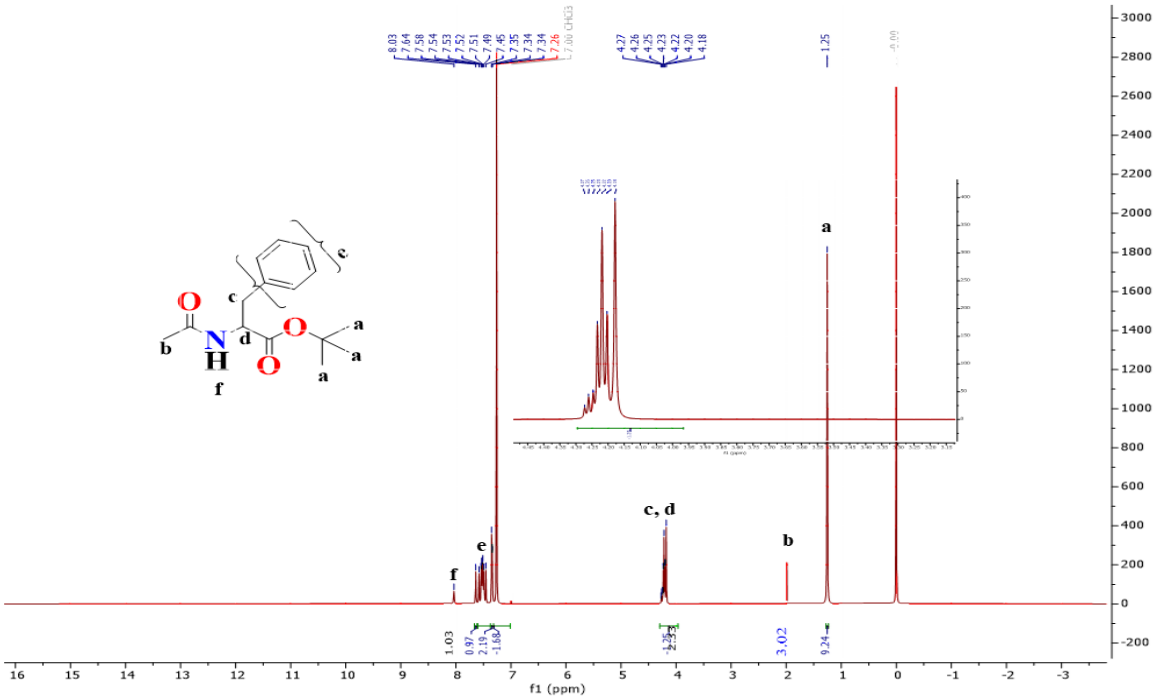

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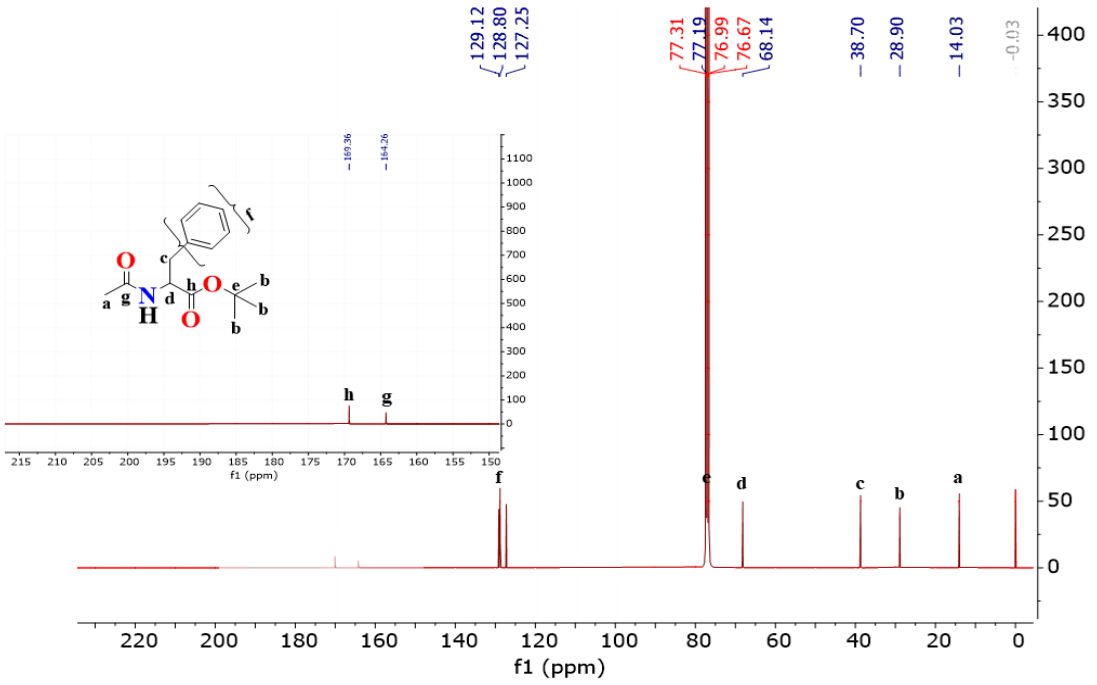

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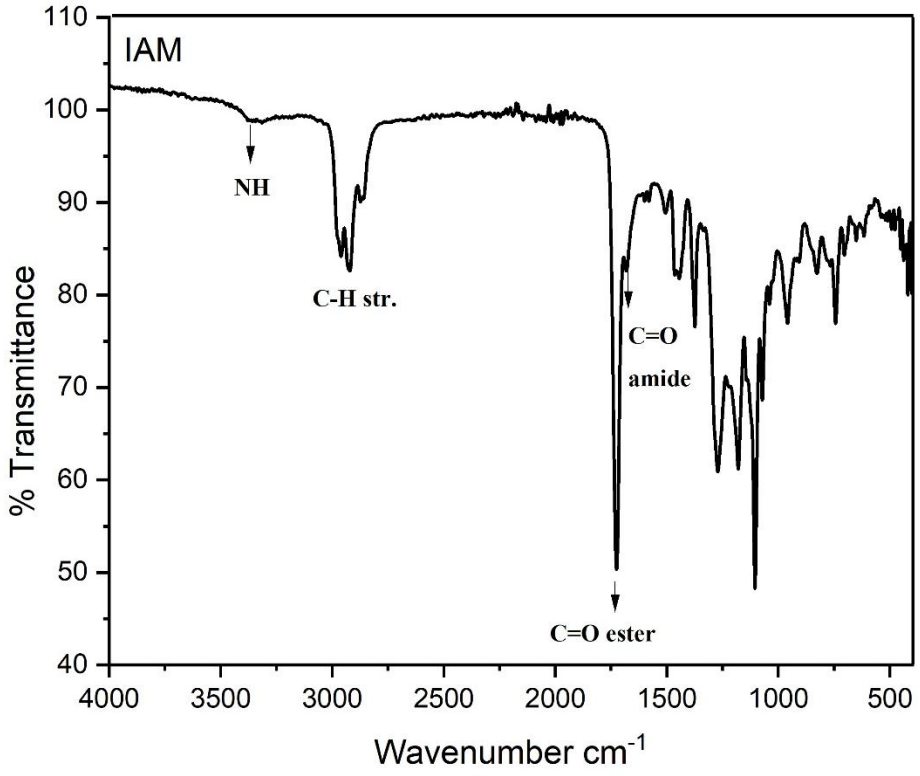
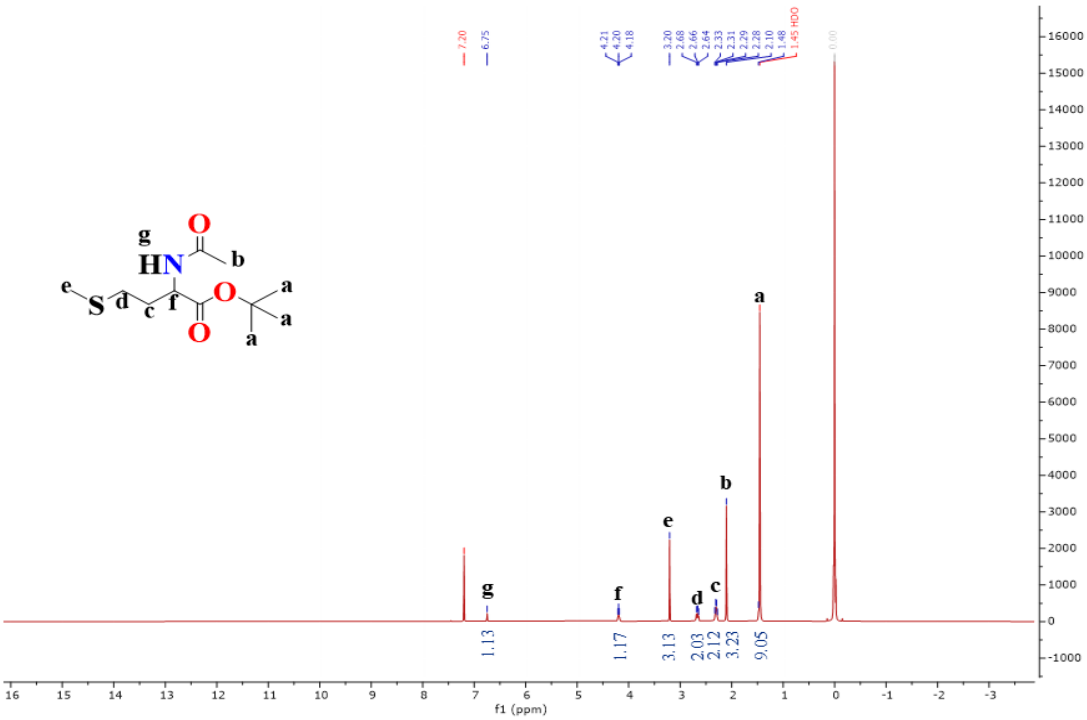
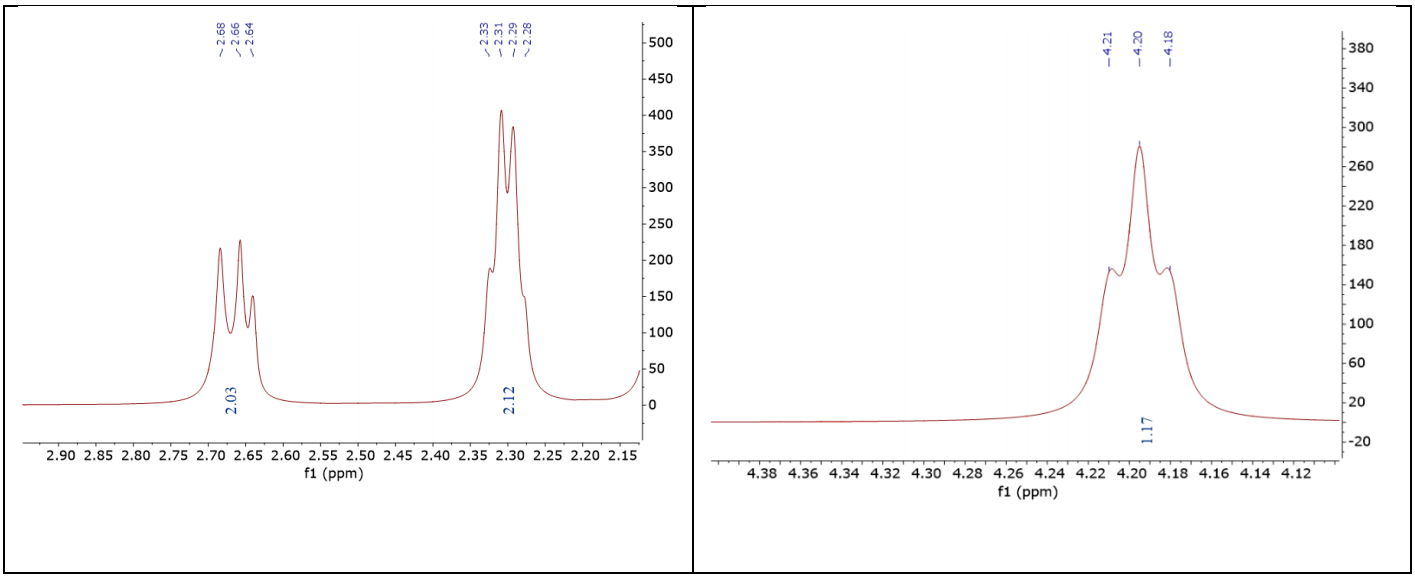


S 27: FT-IR of 5d$_4$



S 28: ^{1}HNMR *5d*$_{4}$



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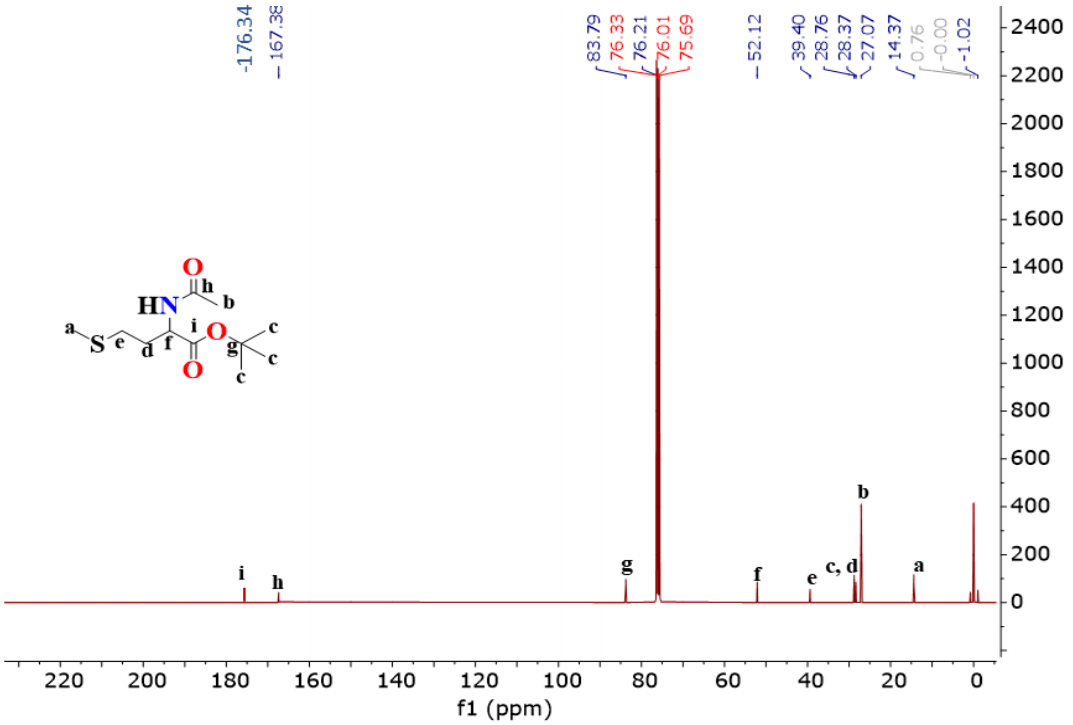

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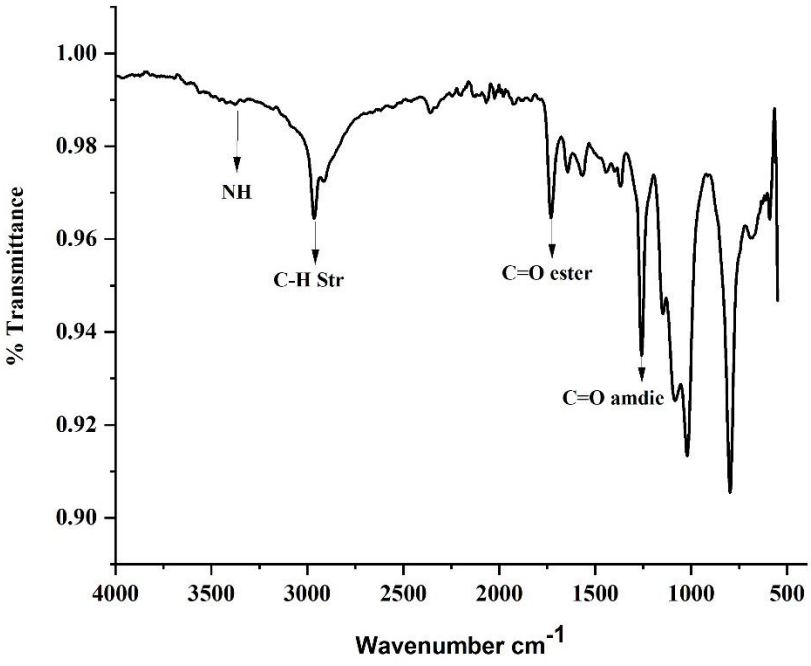

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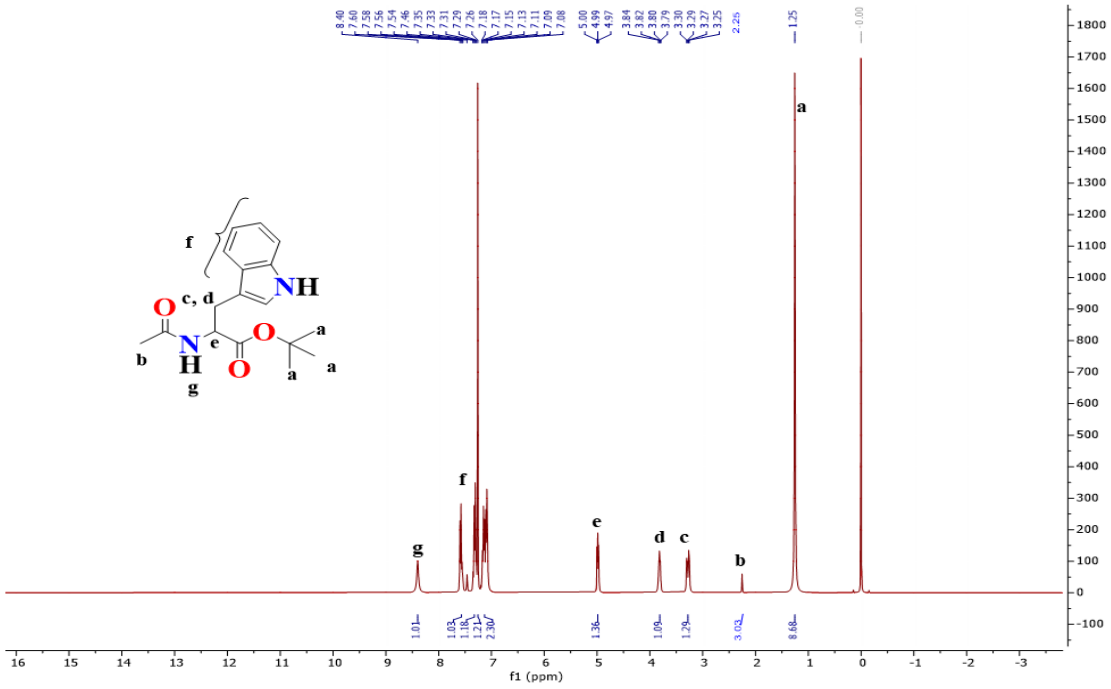

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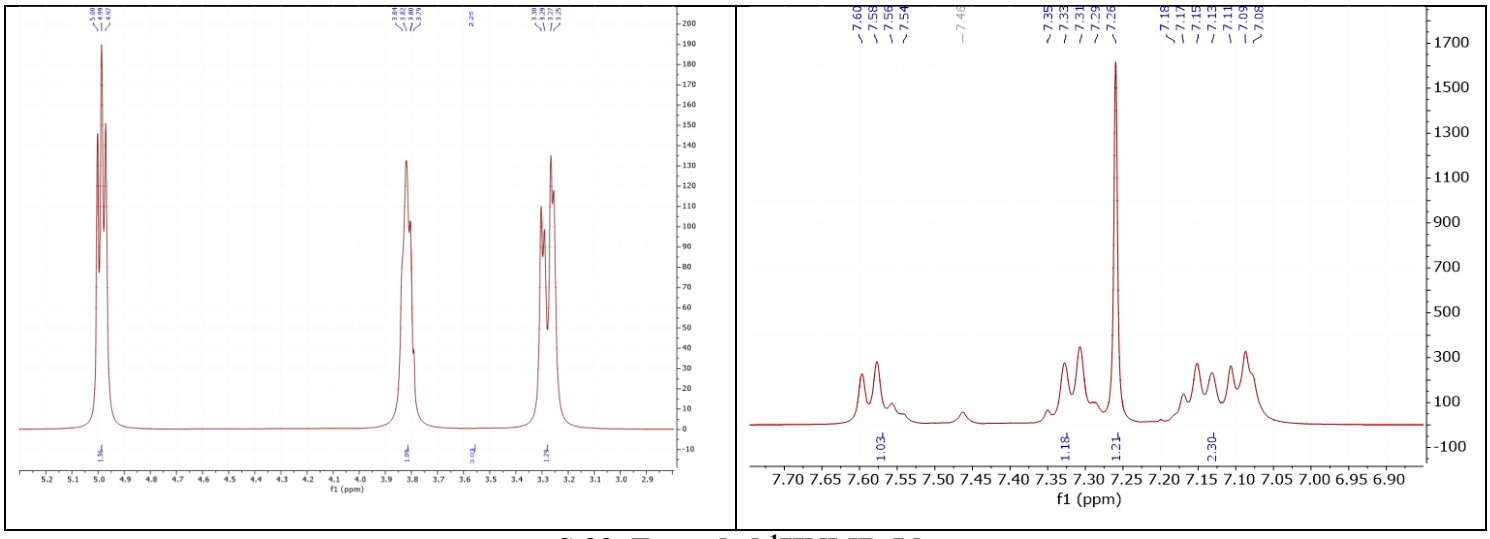
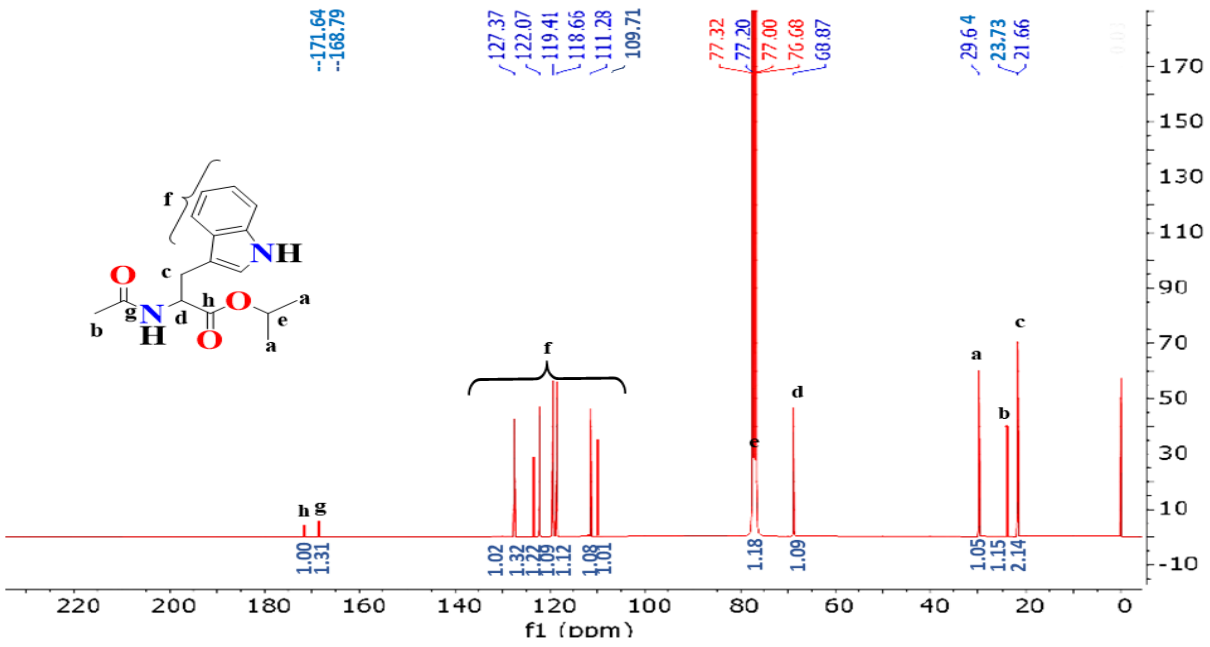


S 33: Extended ^{1}HNMR 5d$_5$



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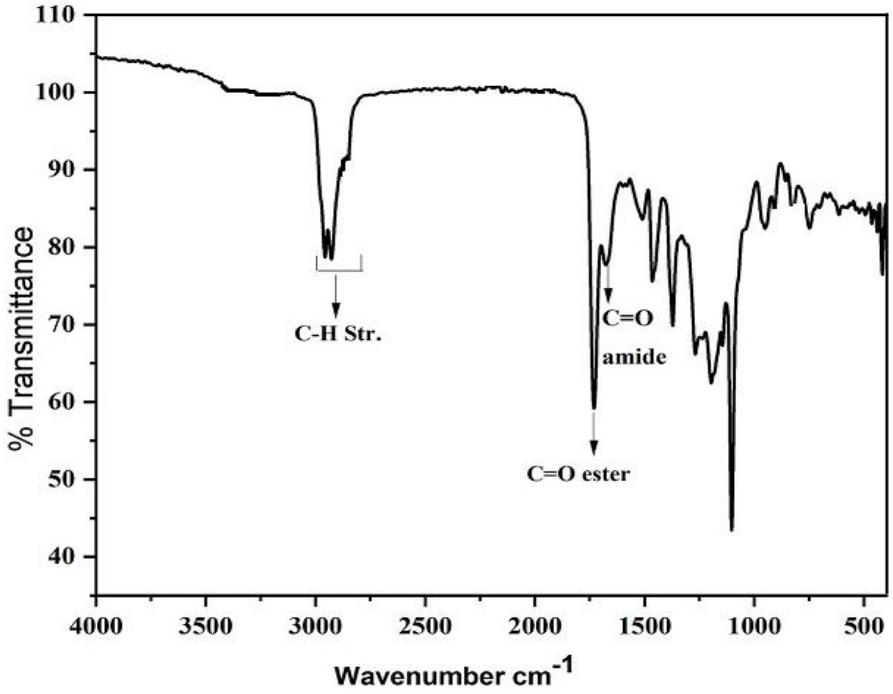

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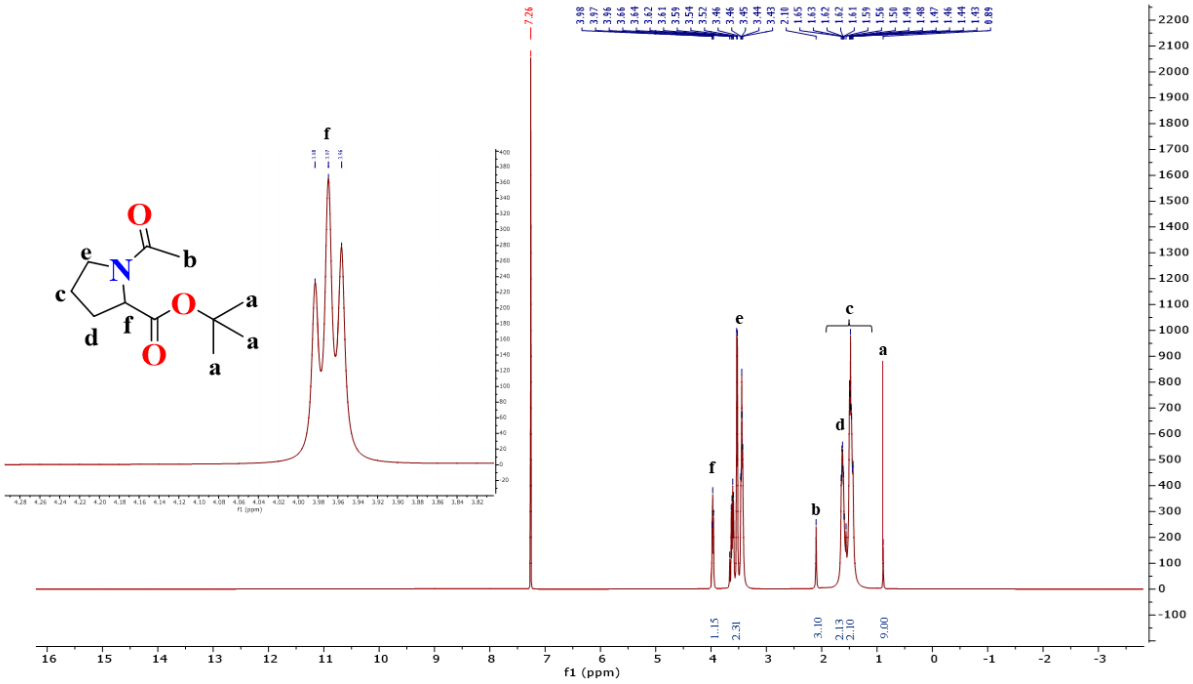

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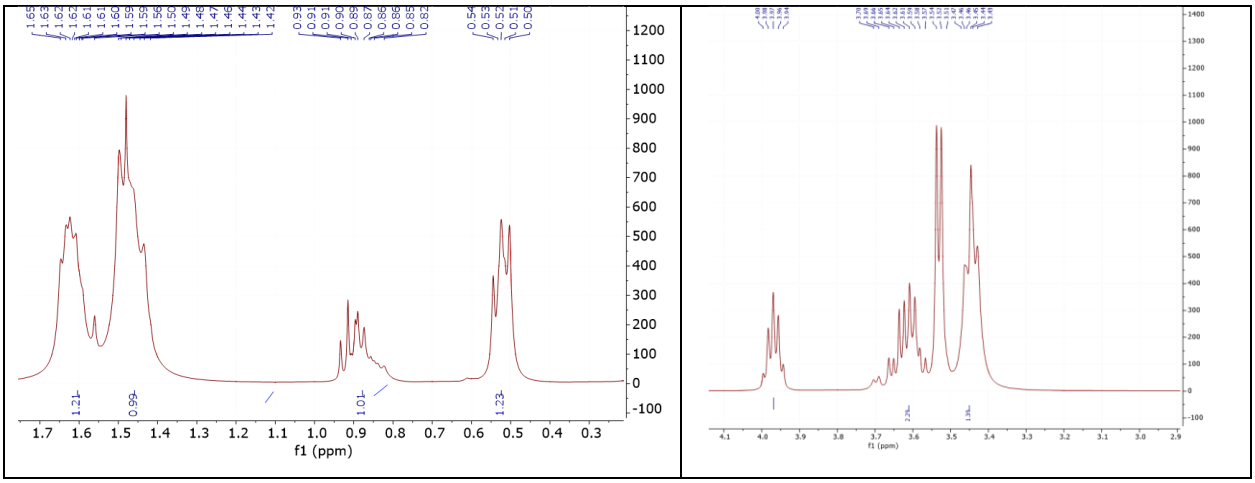
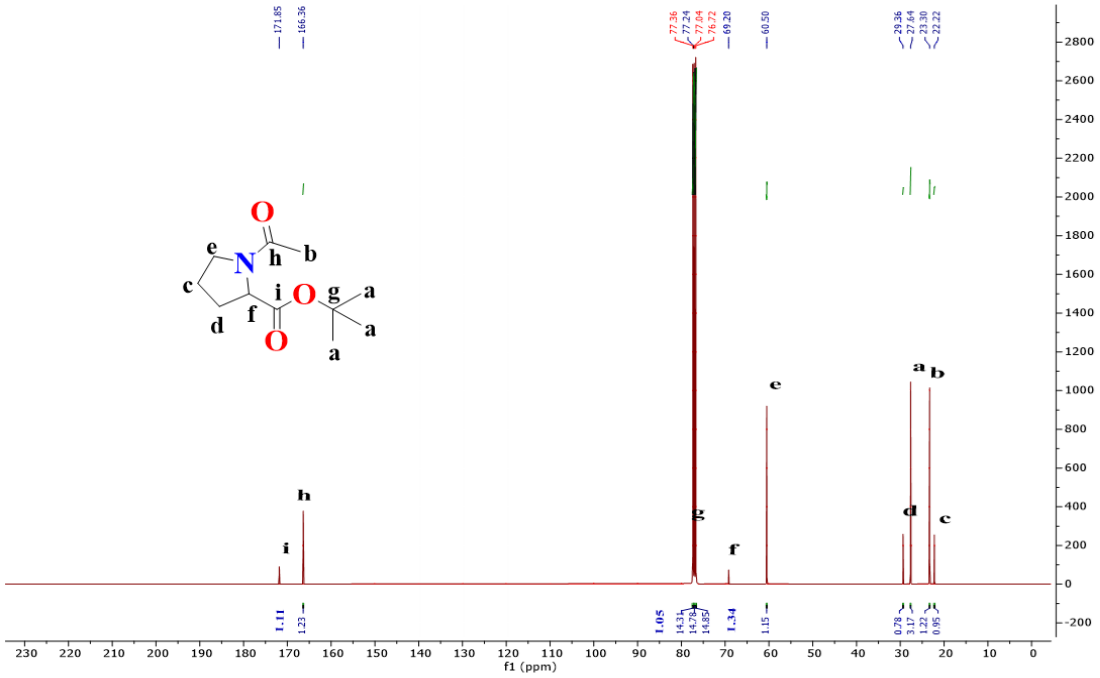
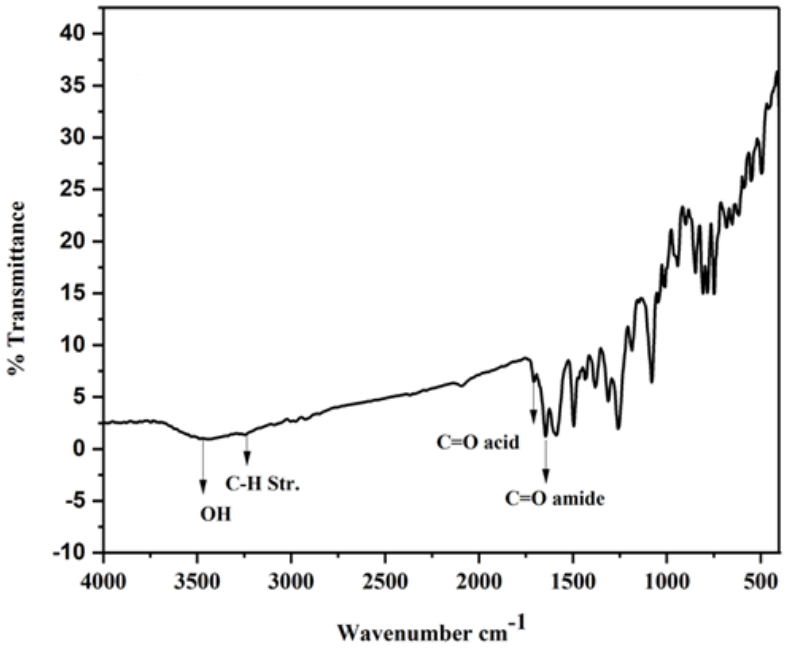


S 37: Extended ^{1}HNMR 5d$_6$



S 38: ^{13}CNMR 5d$_6$

FT-IR Spectroscopic Data of Acetylated Acids (De-esterified *tert*-butyl esters of *N*-acetylated amino acids)



S2. FT-IR of 6d1

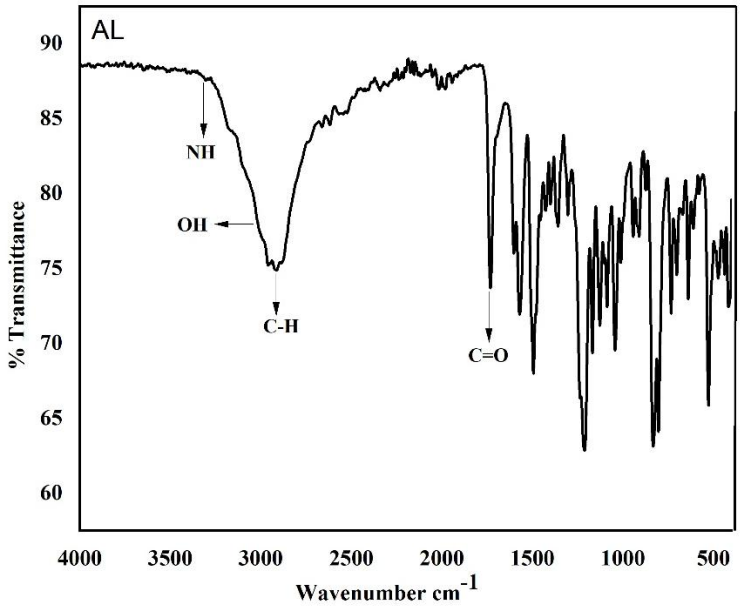
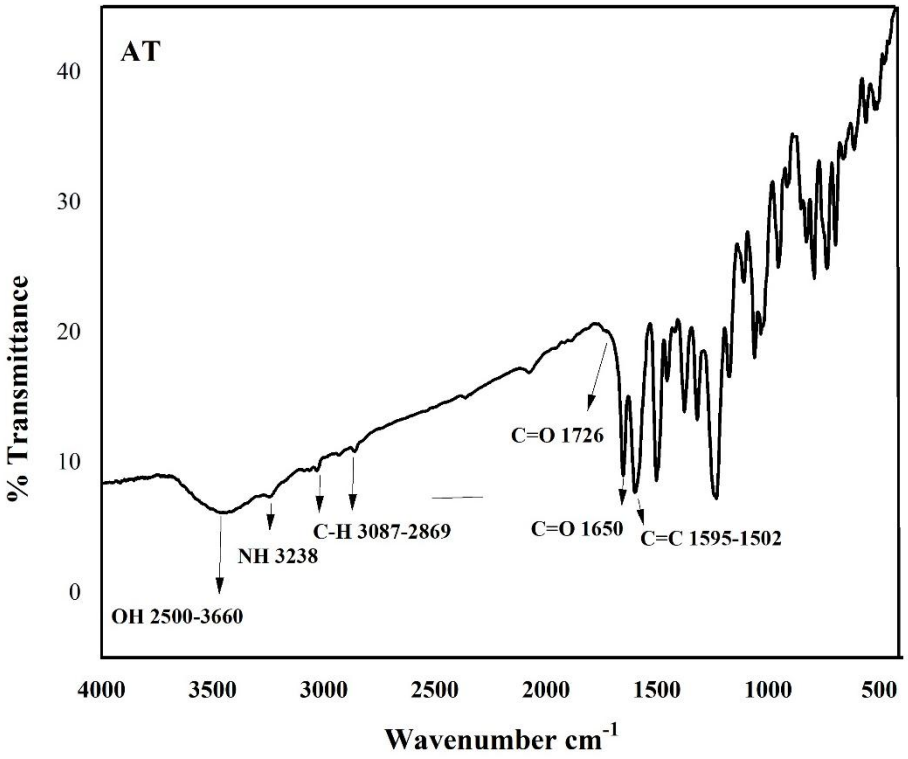
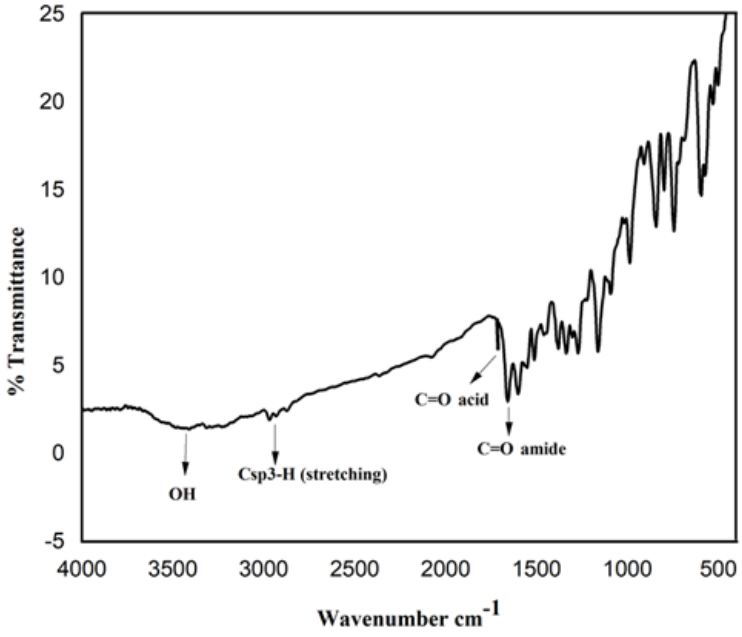


Fig. 3: FT-IR of 6d2



S4. FT-IR of 6d$_5$



S5. FT-IR of 6d$_6$