A facile one pot route for the synthesis of imide tethered peptidomimetics

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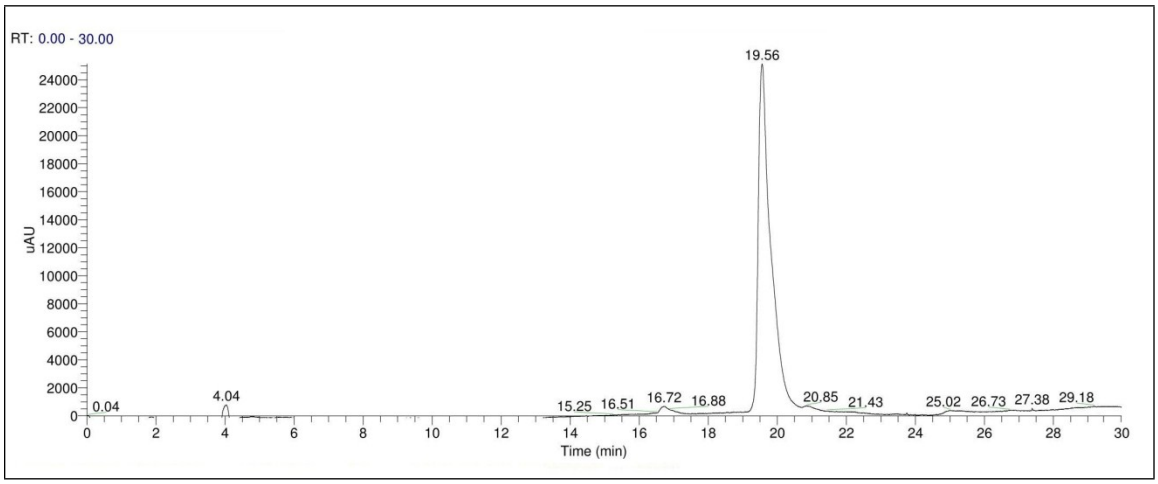
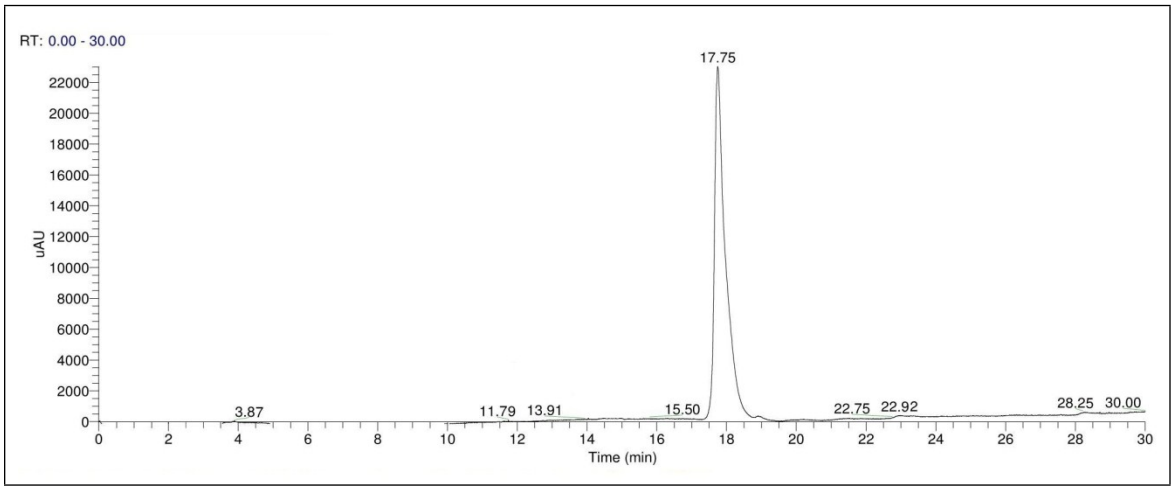
General information

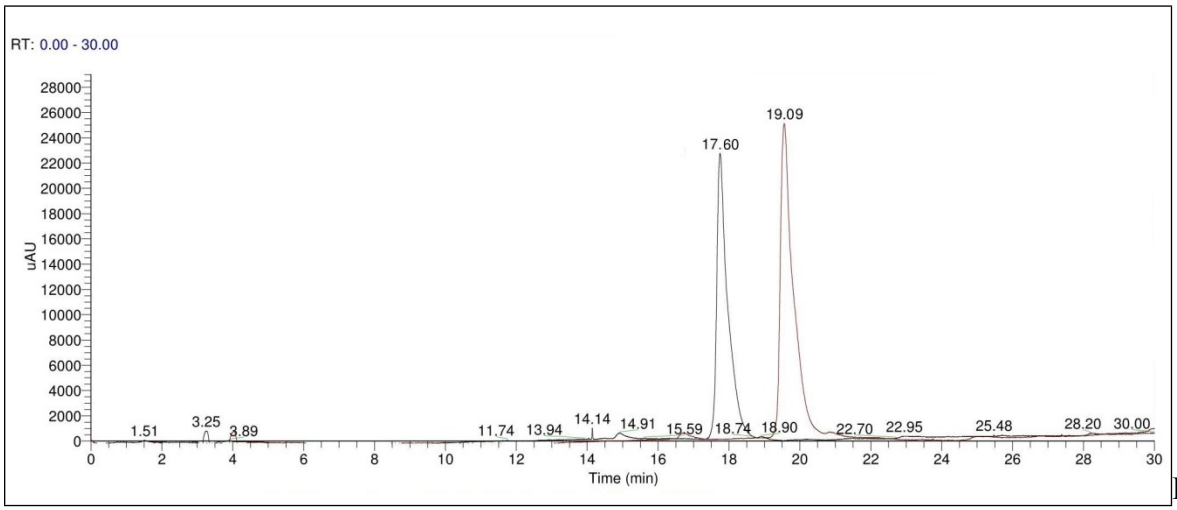
All chemicals were used as obtained from Sigma Aldrich Company, USA. All the solvents were dried and purified using recommended procedures in the literature whenever necessary. High resolution mass spectra were recorded on a Micromass Q-TOF micromass spectrometer and ESMS on LCQ Deca XP MAX using electron spray ionization mode. ^{1}H NMR and ^{13}C NMR spectra were recorded on a Bruker AMX 300 MHz and 100 MHz spectrometer, respectively, at the Indian Institute of Science, Bangalore. RP-HPLC analysis of epimers was carried out by LCQ Deca XP MAX VWD at λ = 272nm; flow rate: 1.0 mL/min; column: Thermoscientific C18 syncronis, pore size-5 μm, diameter x length = 4.6 x 250 nm; method: gradient 0.1% TFA water-acetonitrile; acetonitrile 30-100% in 30 min. Melting points were determined in an open capillary and are uncorrected. TLC experiments were done using MERCK TLC aluminum sheets (silica gel 60 F254) and chromatograms were visualized by exposing in iodine chamber or UV-lamp. Column chromatography was performed on silica gel (100-200 mesh) using ethyl acetate and hexane mixtures as eluent.



Racemization study of Fmoc-L-Phg-ψ[CONHCO]-Ala-Boc



Racemization study of Fmoc-D-Phg-ψ[CONHCO]-Ala-Boc



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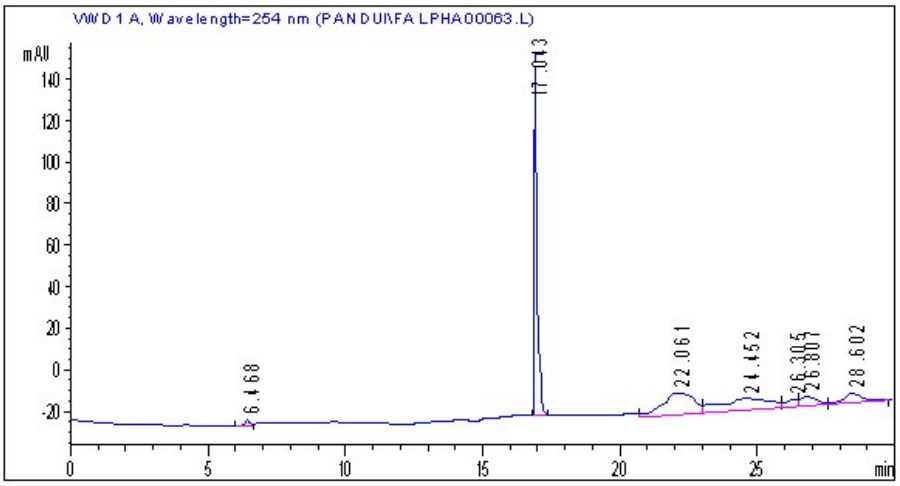

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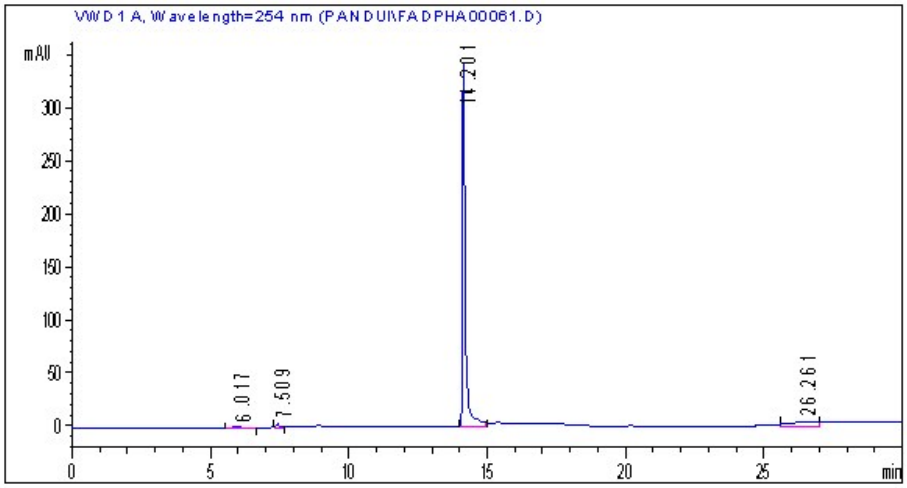

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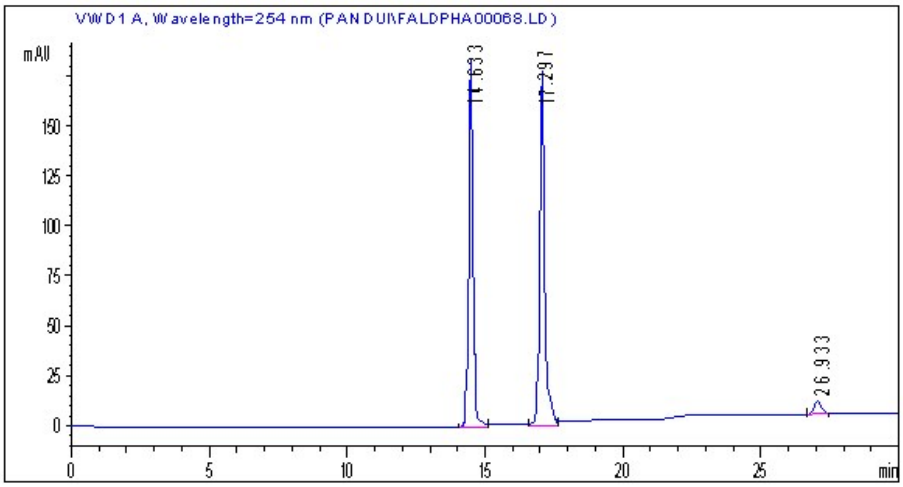

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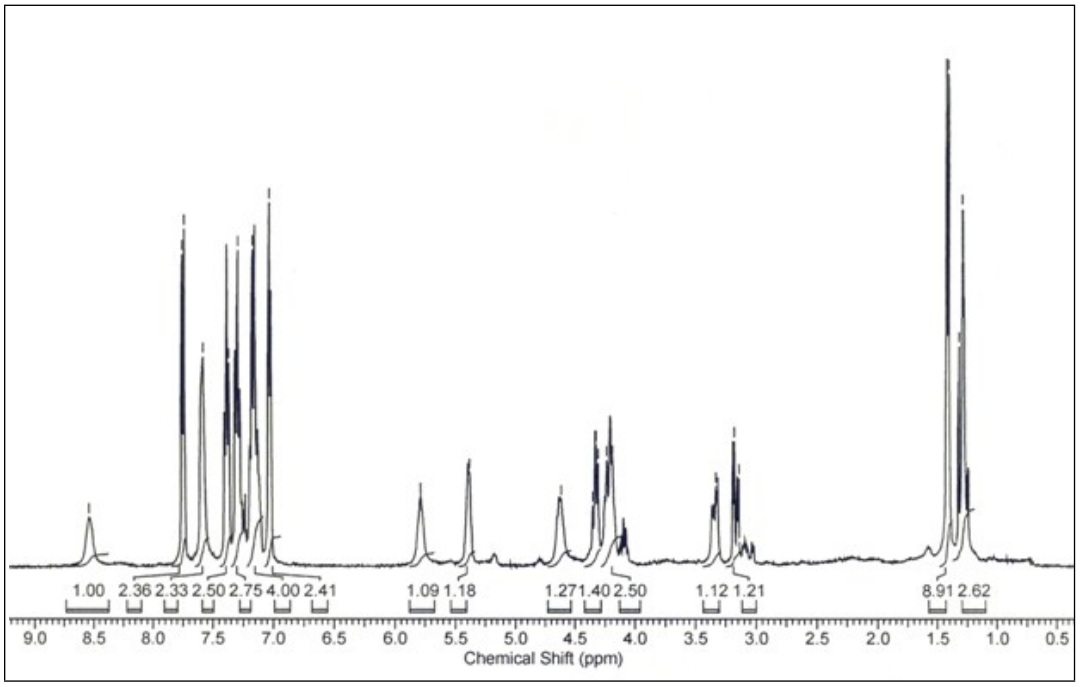
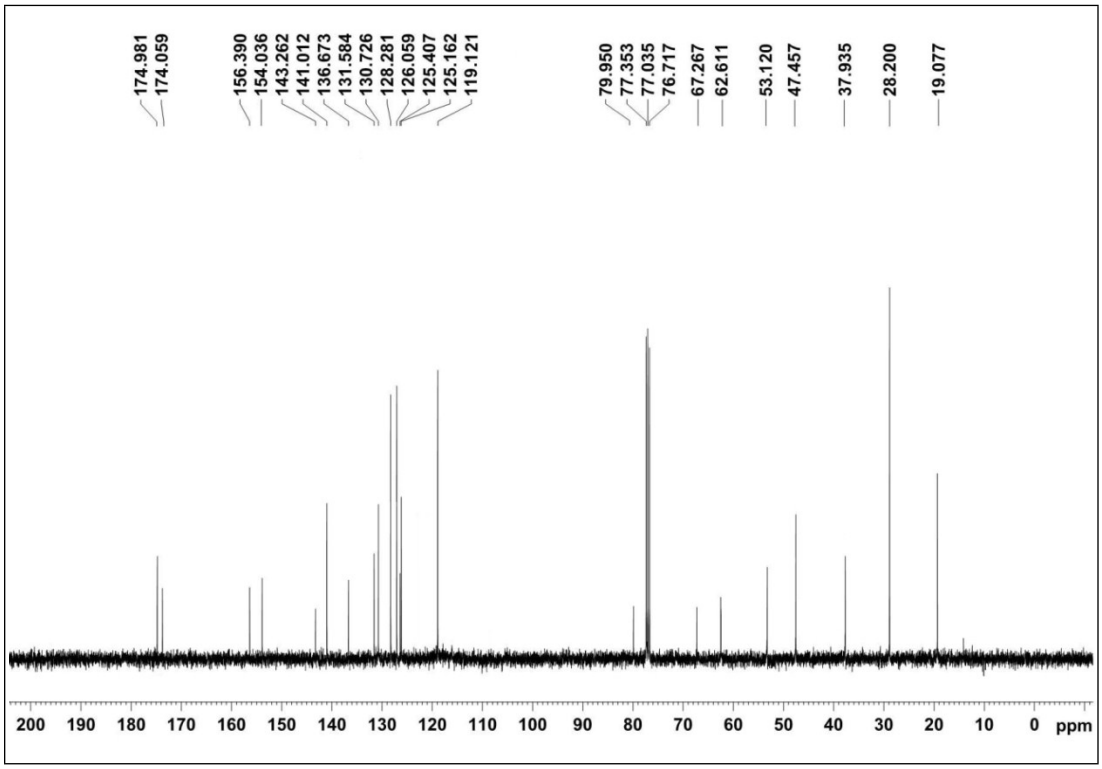


^{1}H NMR Spectrum of Compound Fmoc-Ala-ψ[CONHCO]-Phe-Boc 4a



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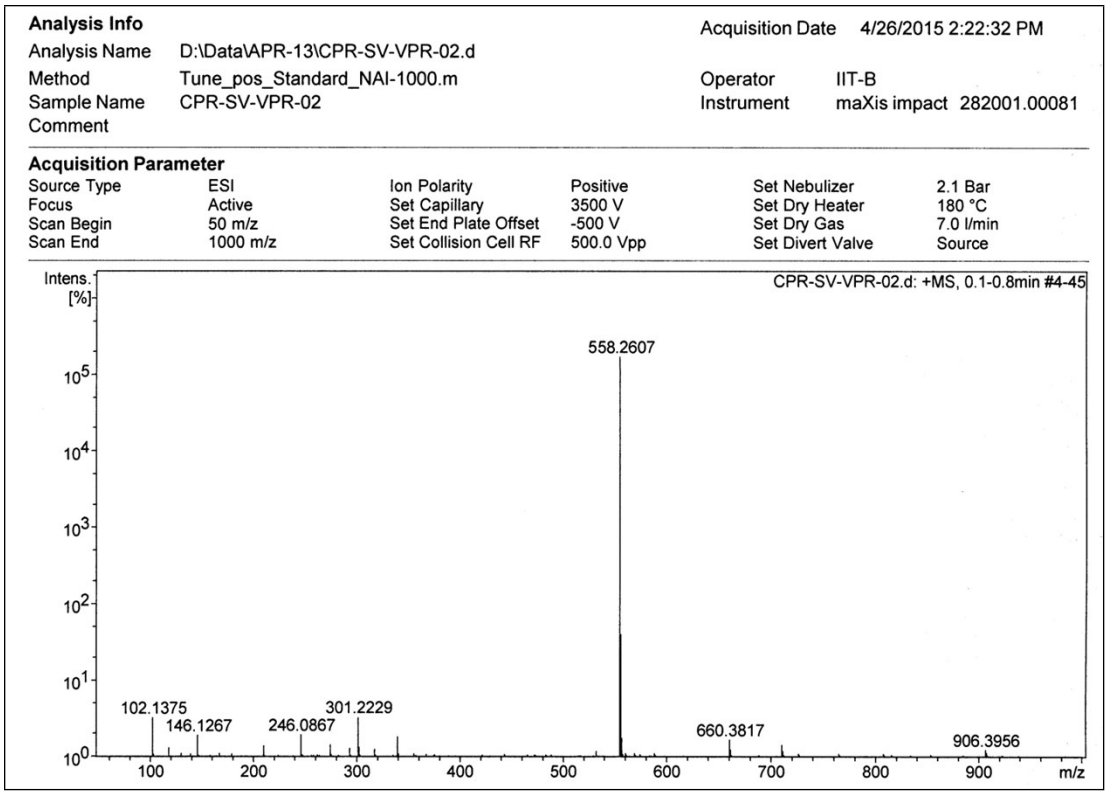
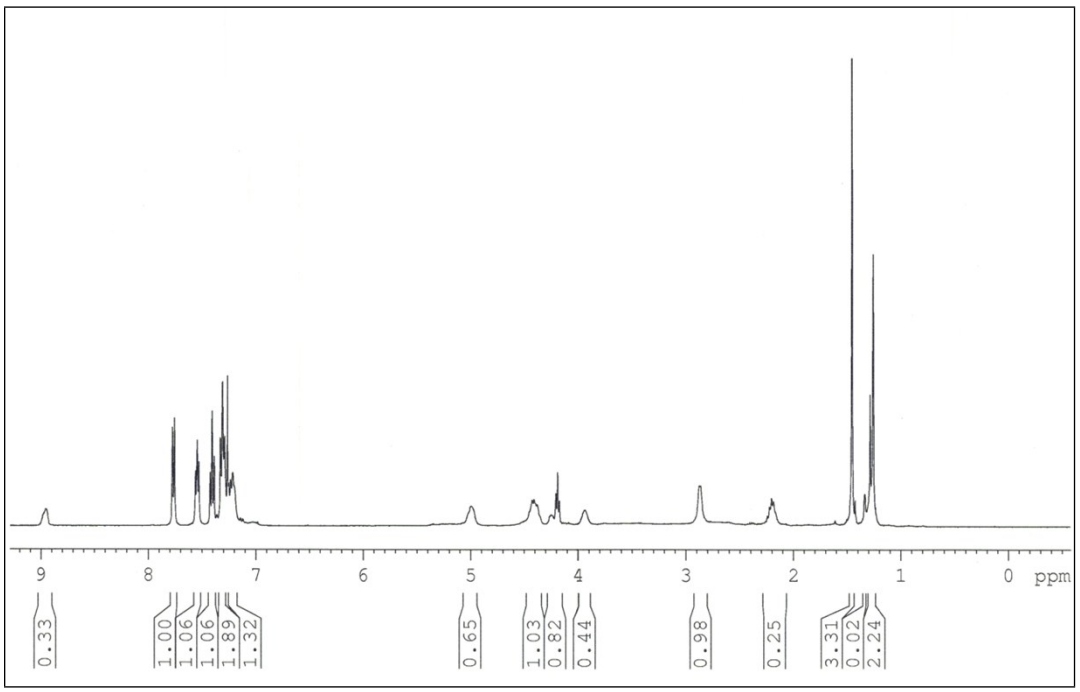


HRMS of Compound Fmoc-Ala-ψ[CONHCO]-Phe-Boc 4a



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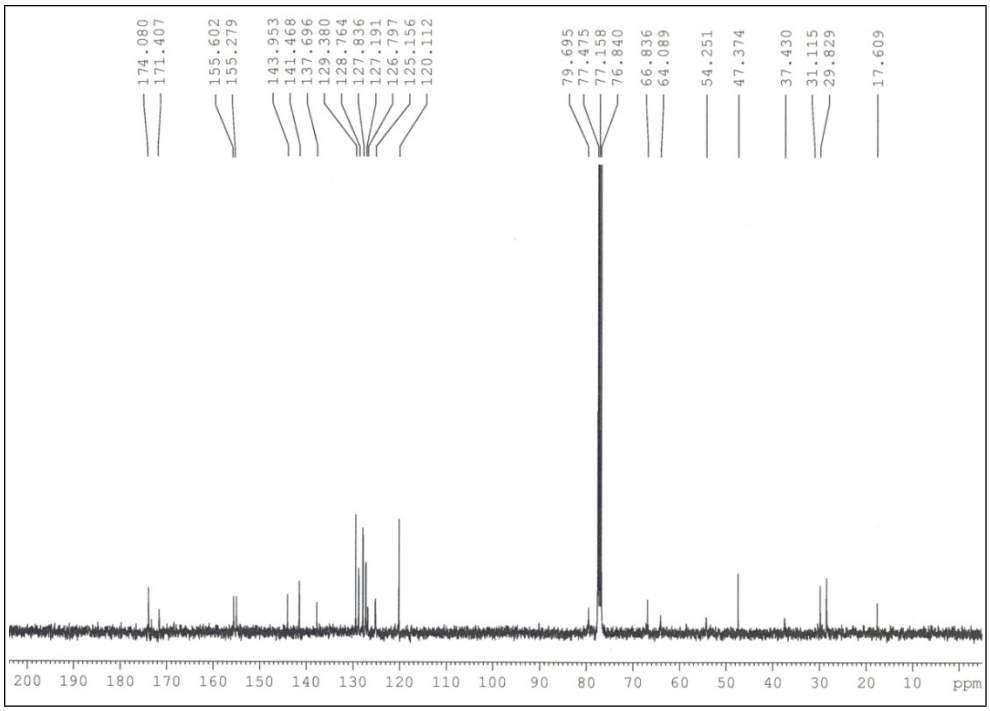

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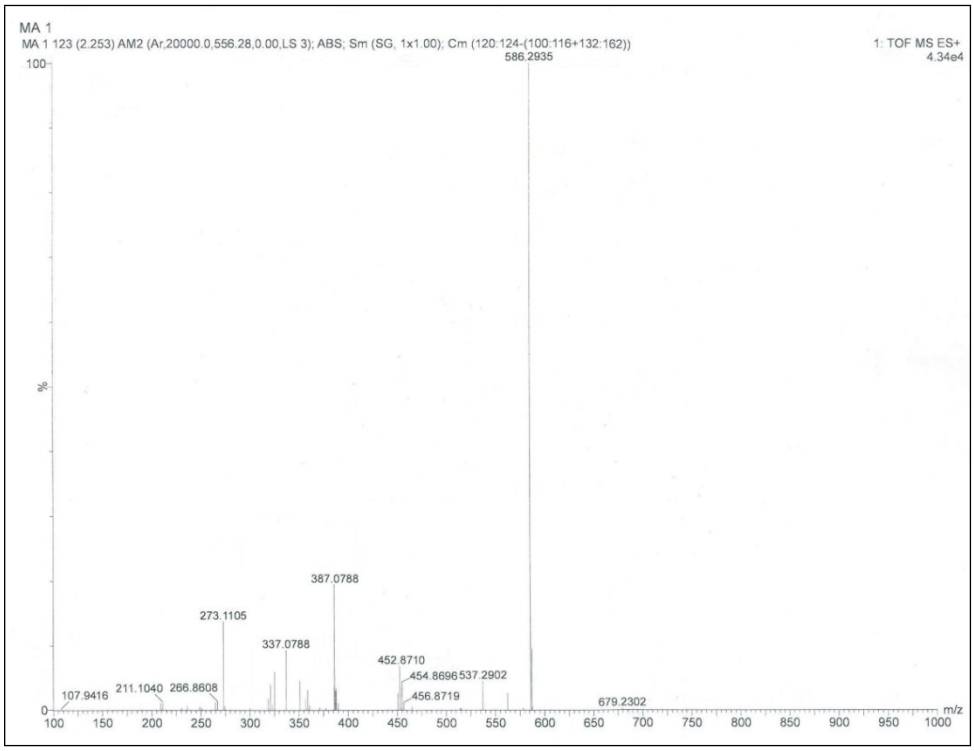

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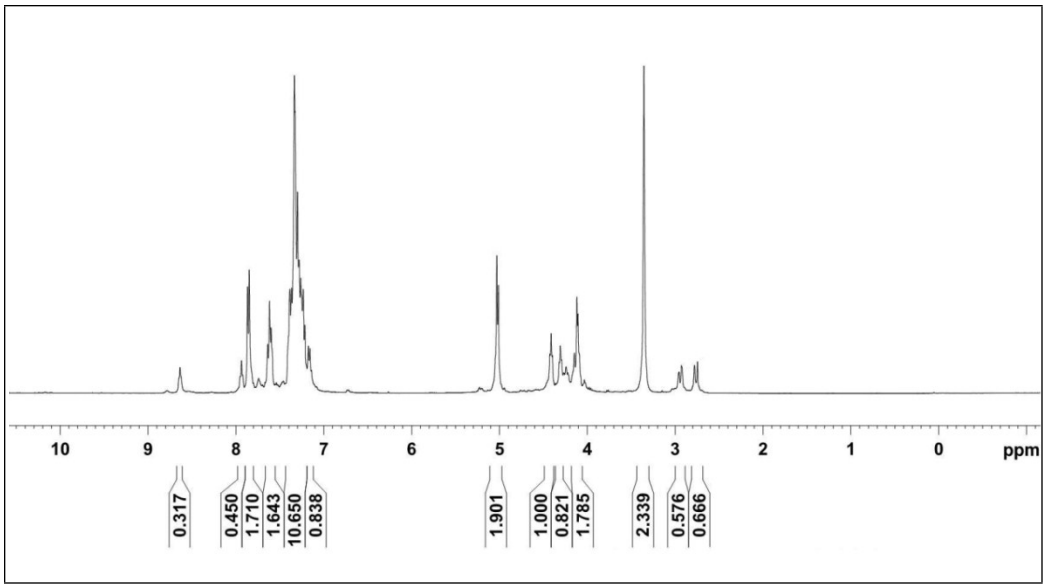

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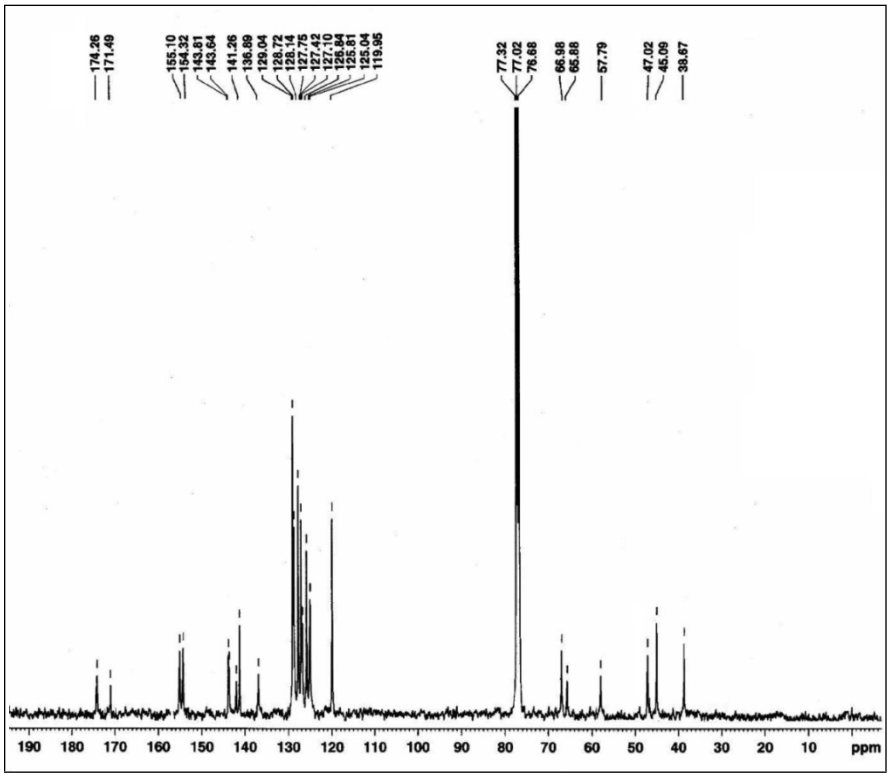

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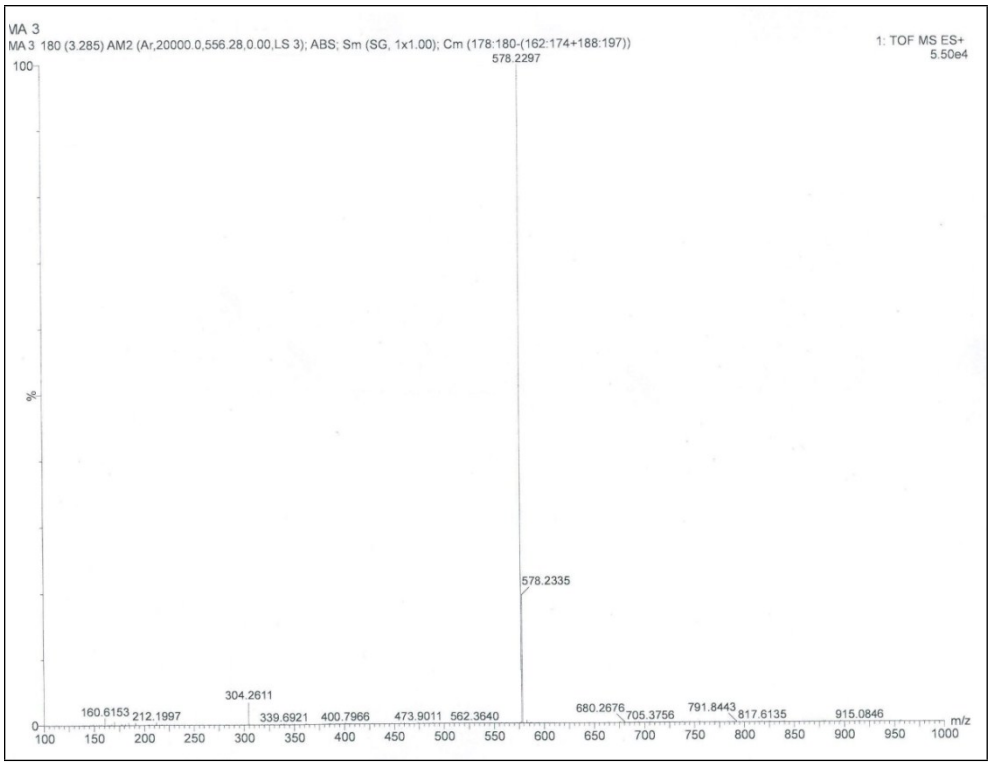

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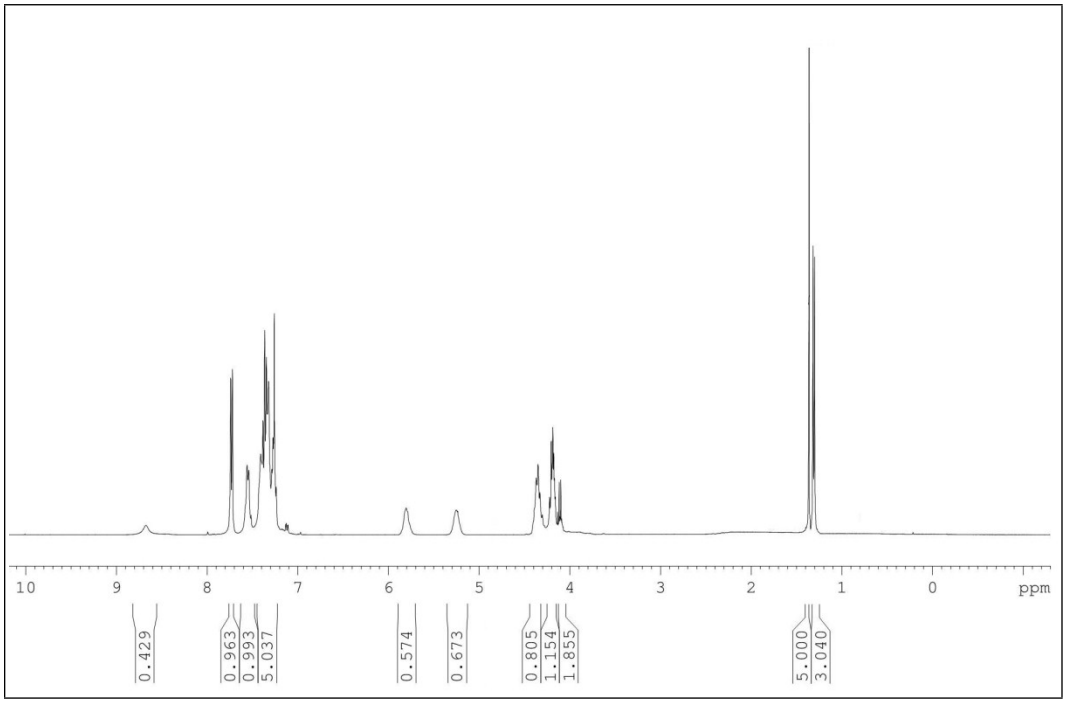

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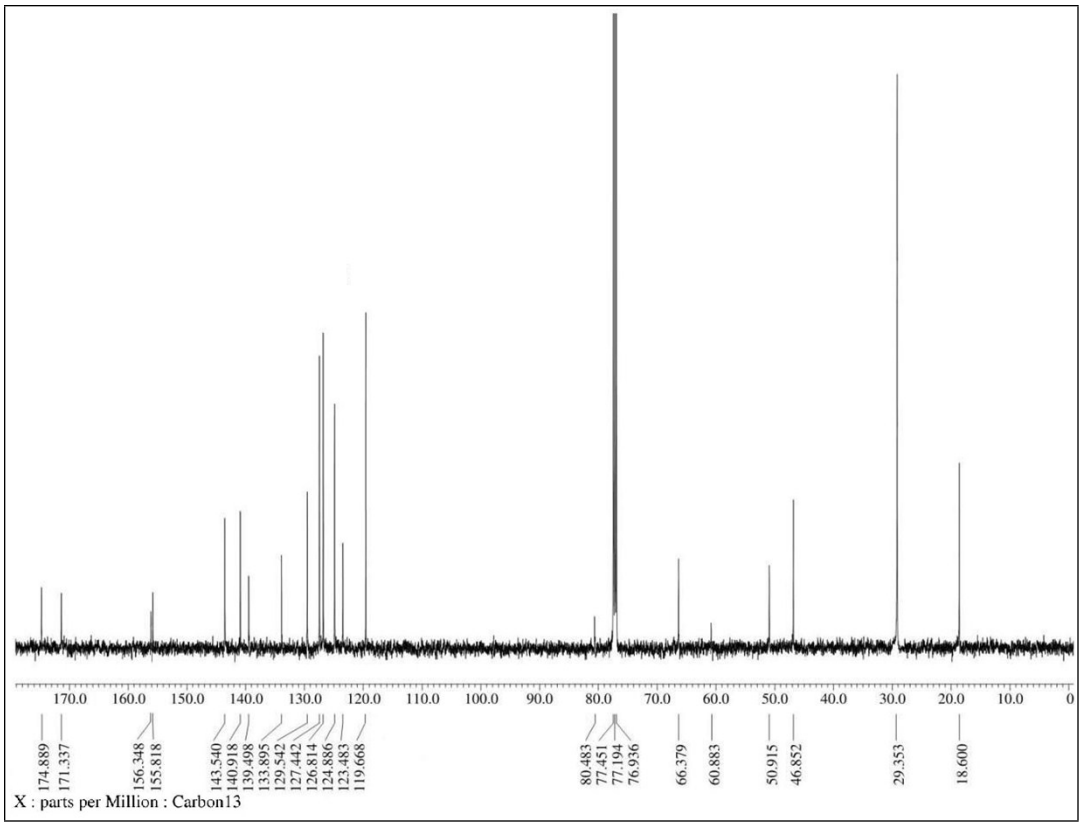

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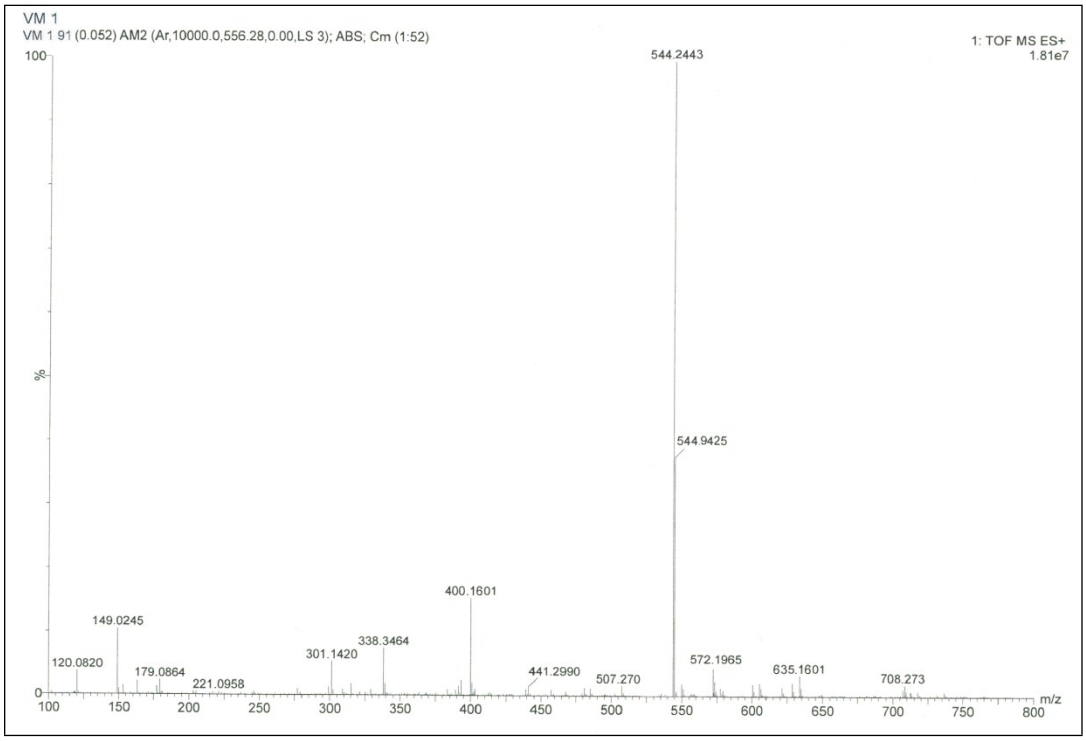

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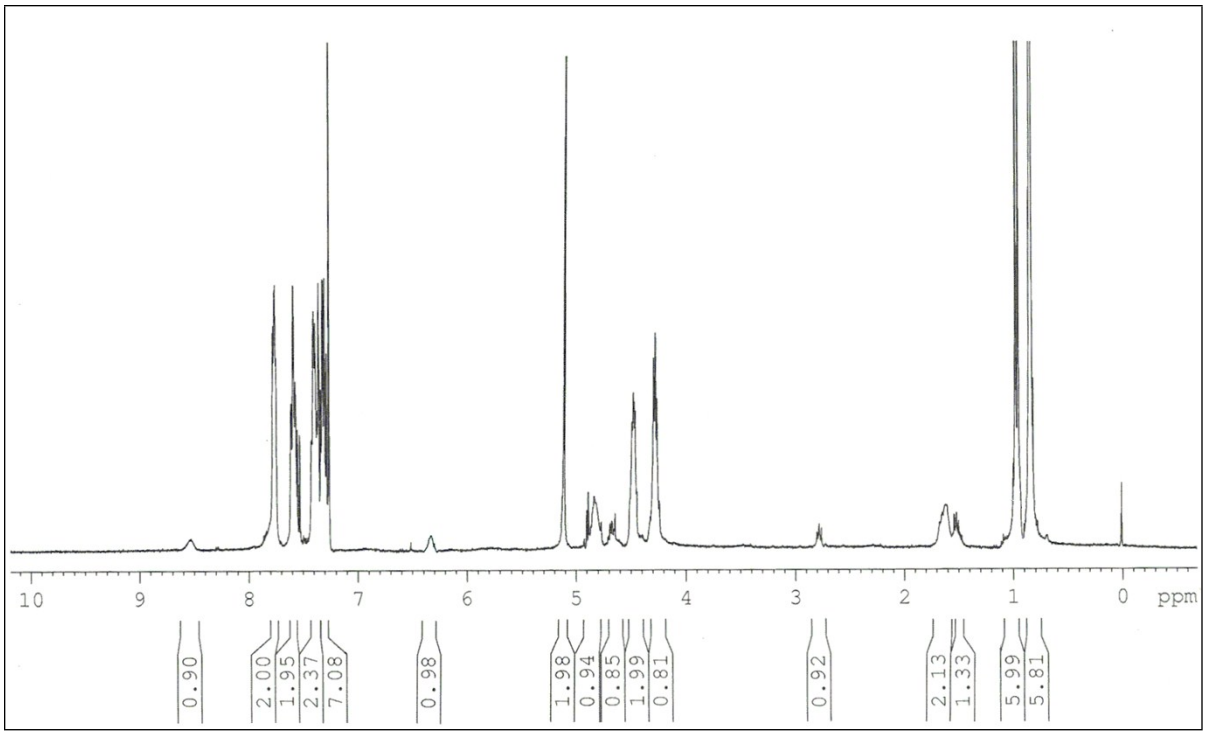

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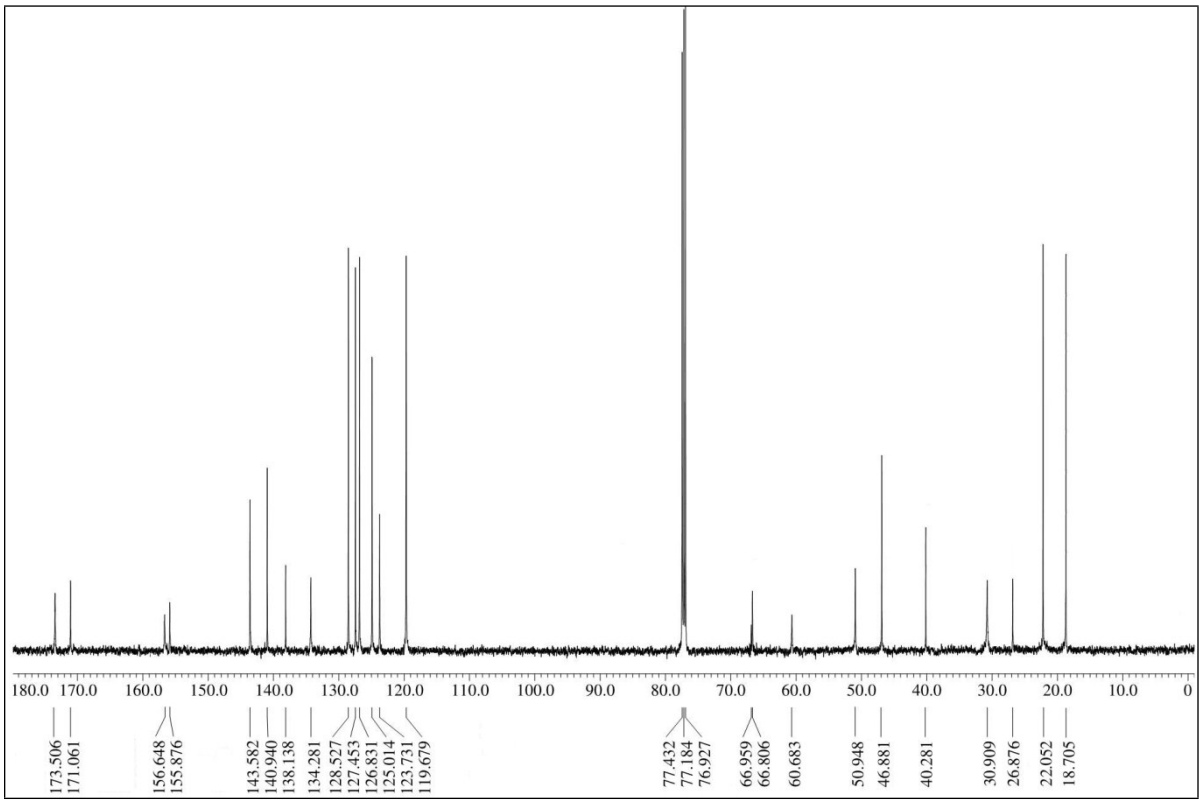

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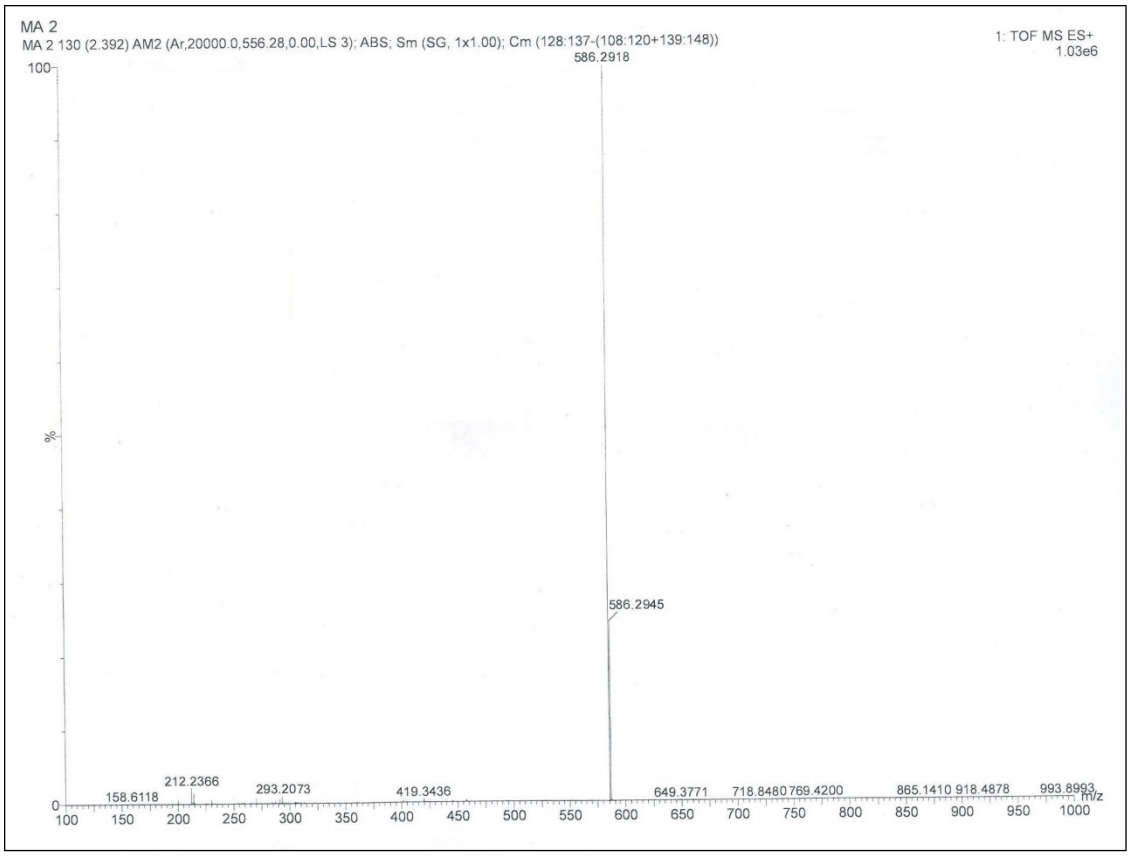

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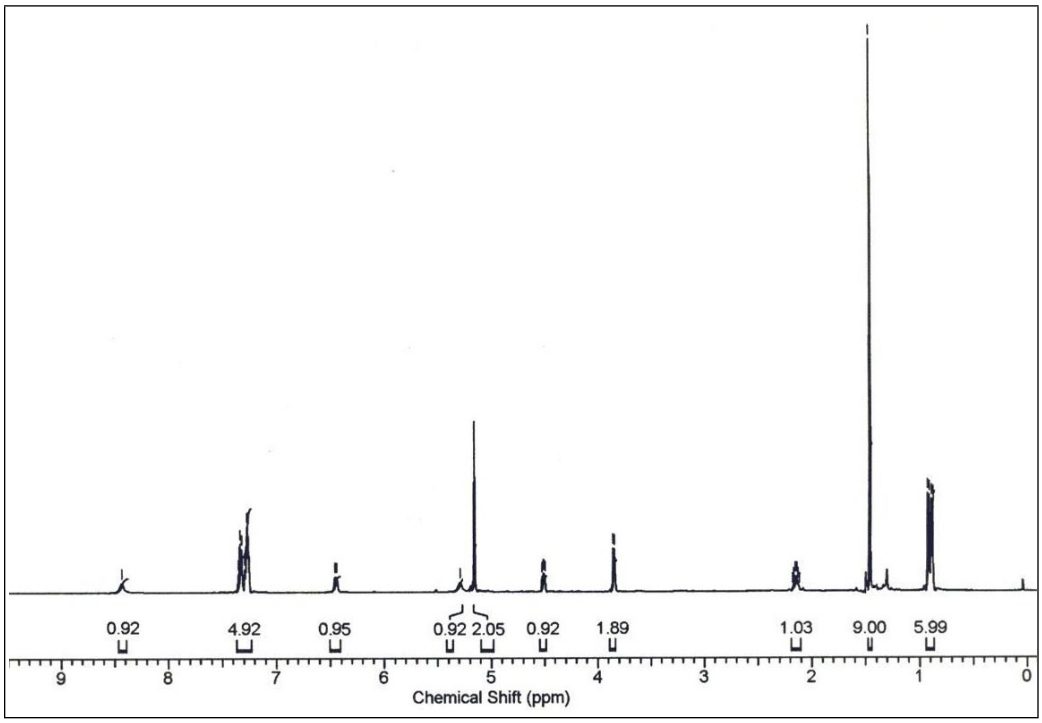
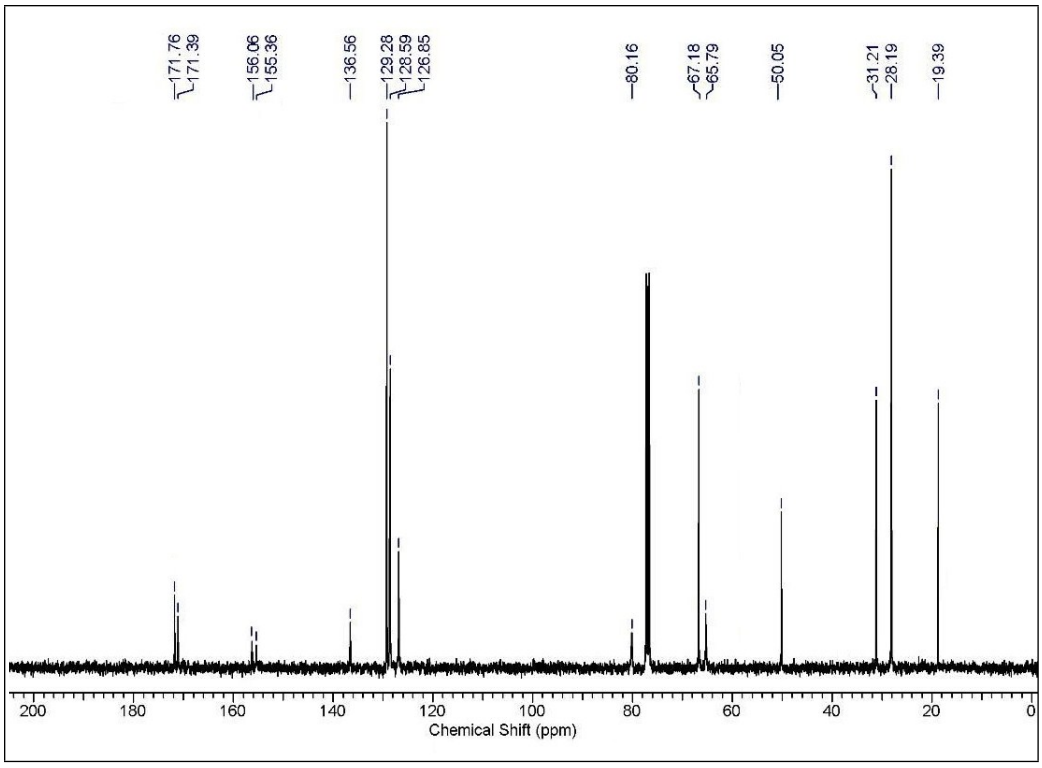


^{1}H NMR Spectrum of Compound Boc-Gly-ψ[CONHCO]-Val-Cbz 4f



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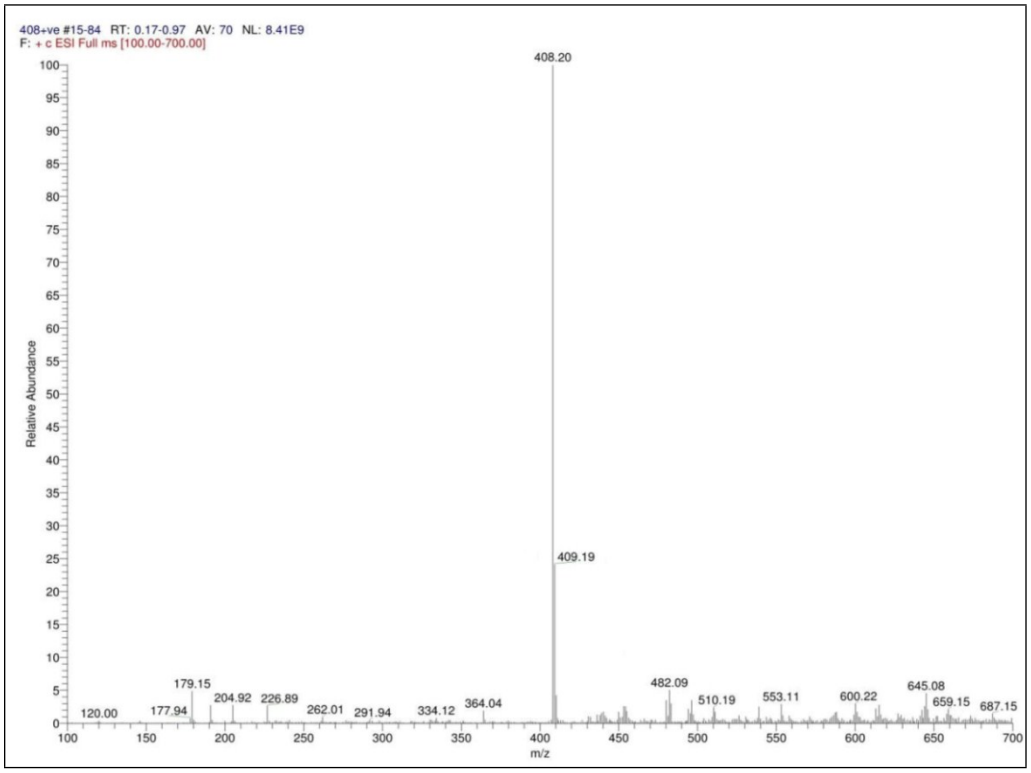

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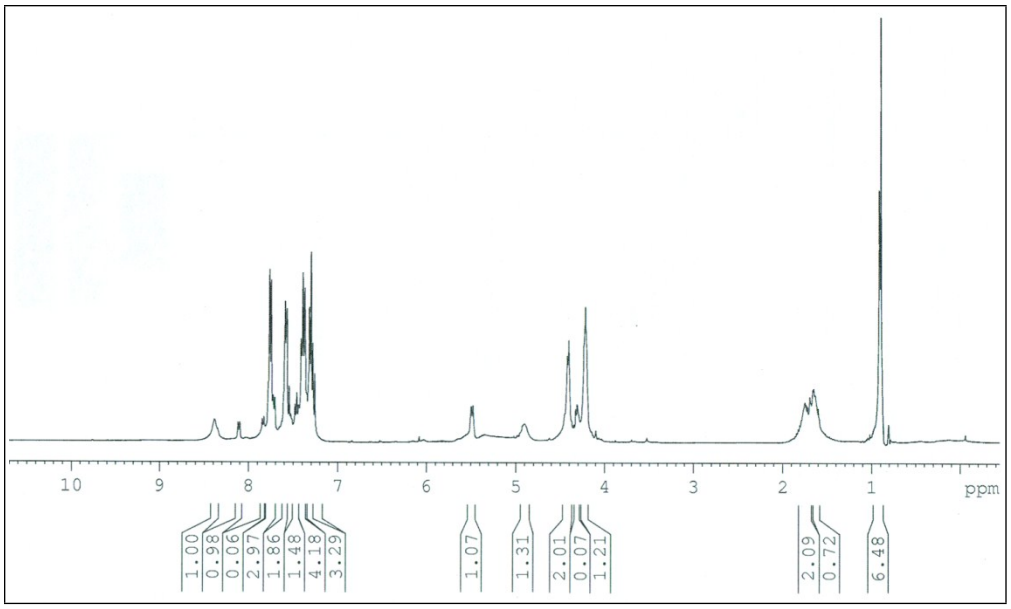

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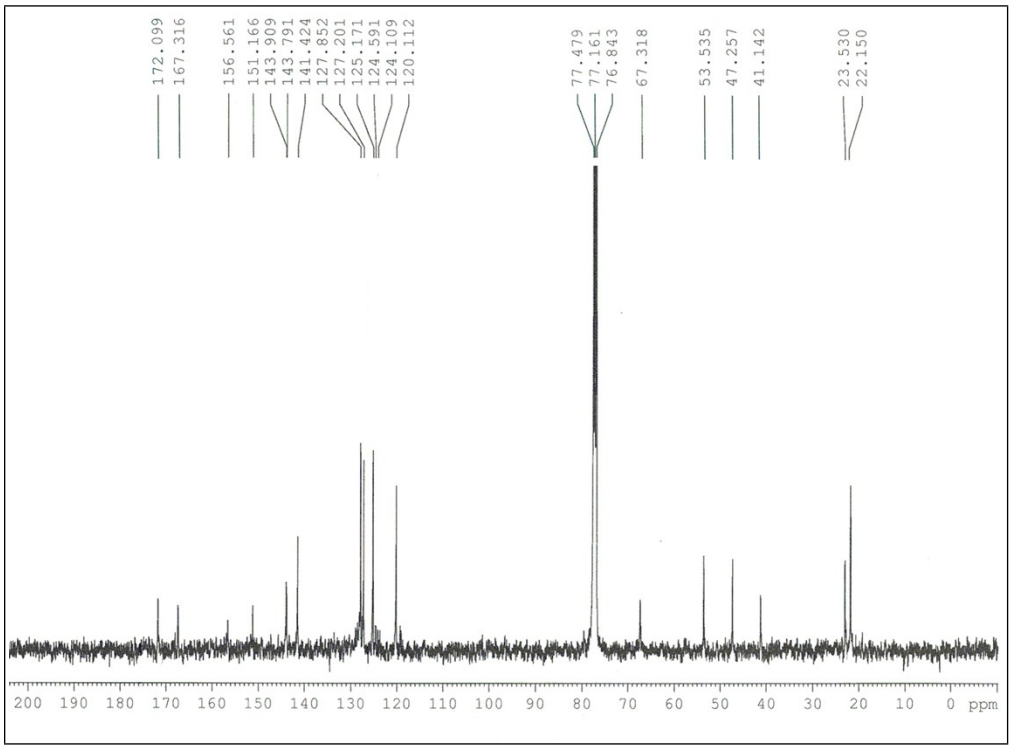

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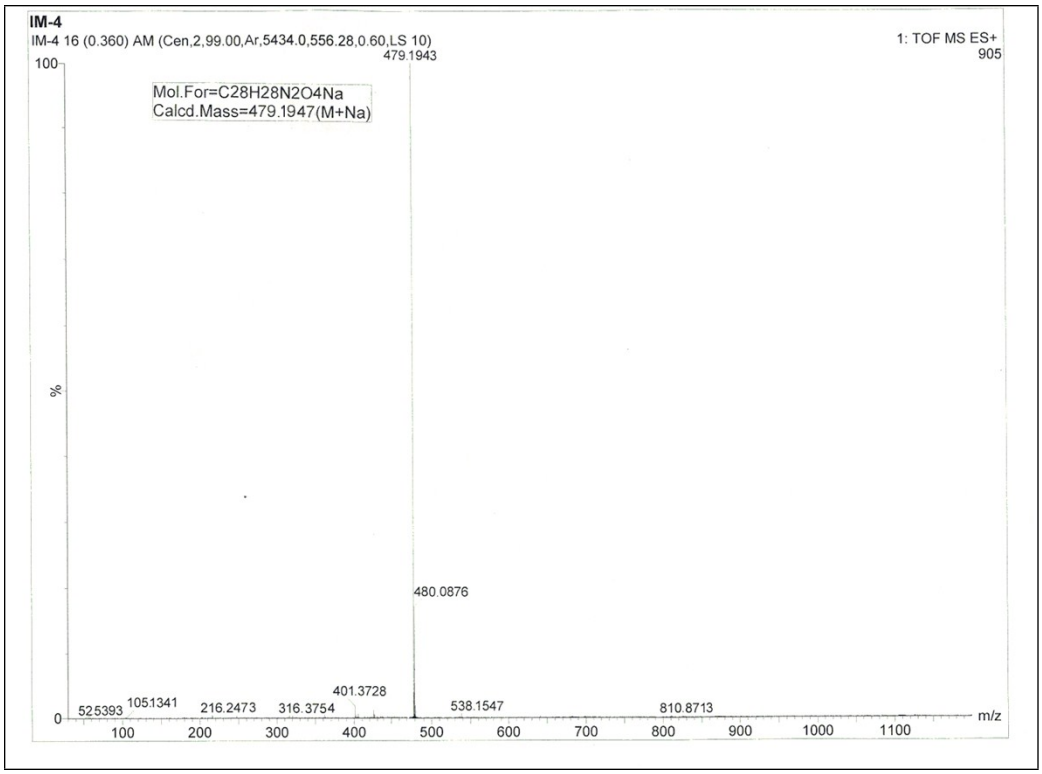

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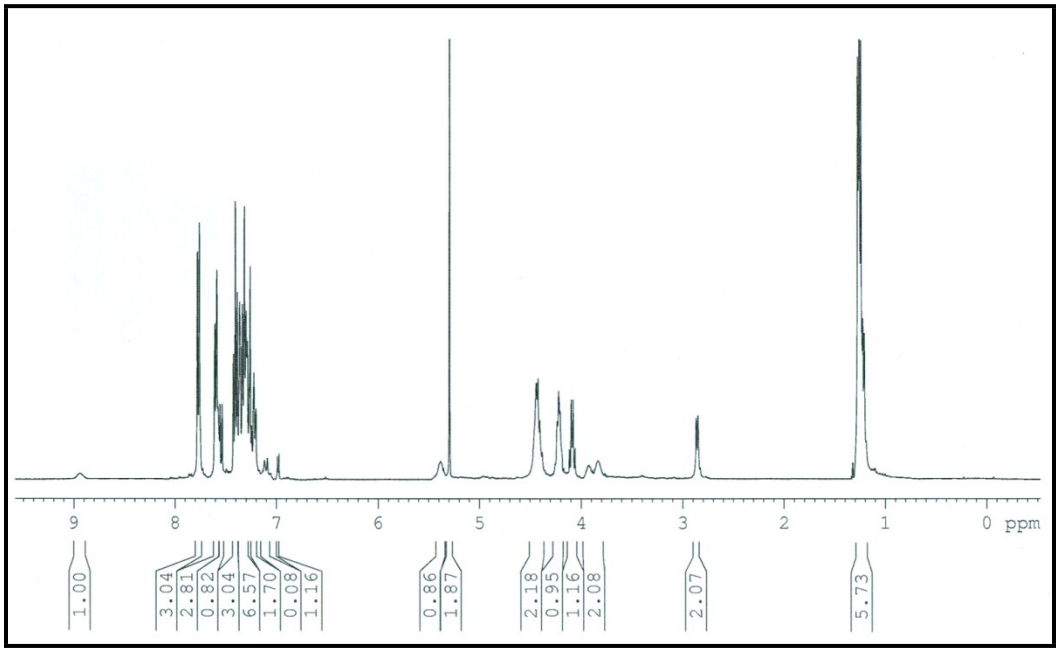
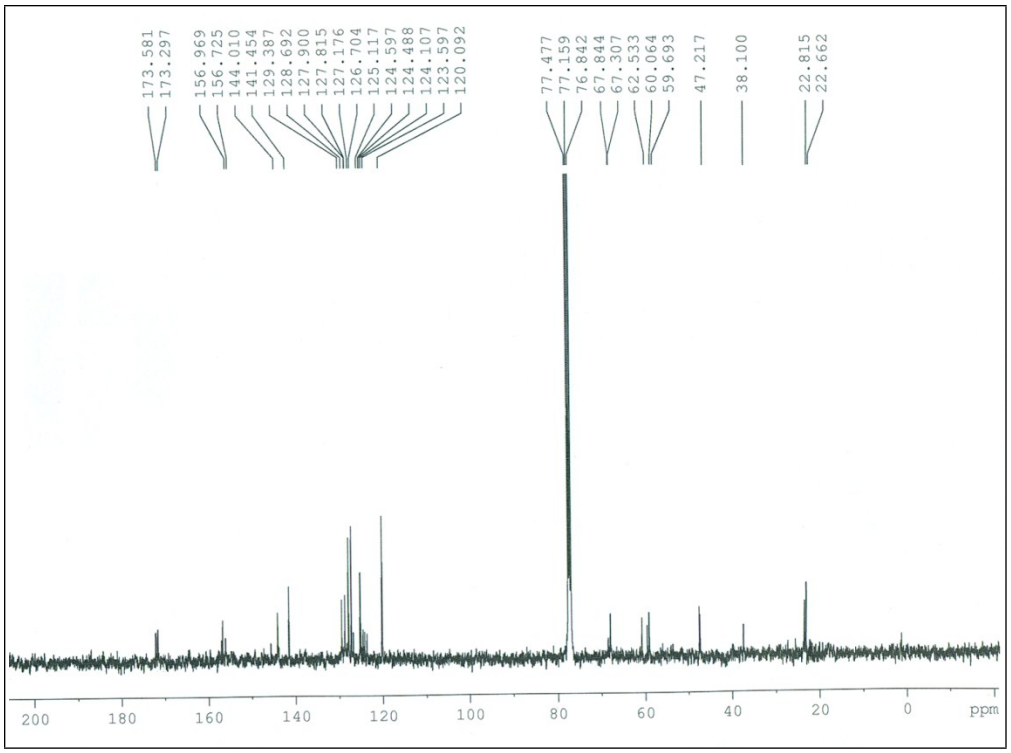


^{1}H NMR Spectrum of Compound Cbz-Ala-Phe-ψ[CONHCO]-Ala-Fmoc 8a



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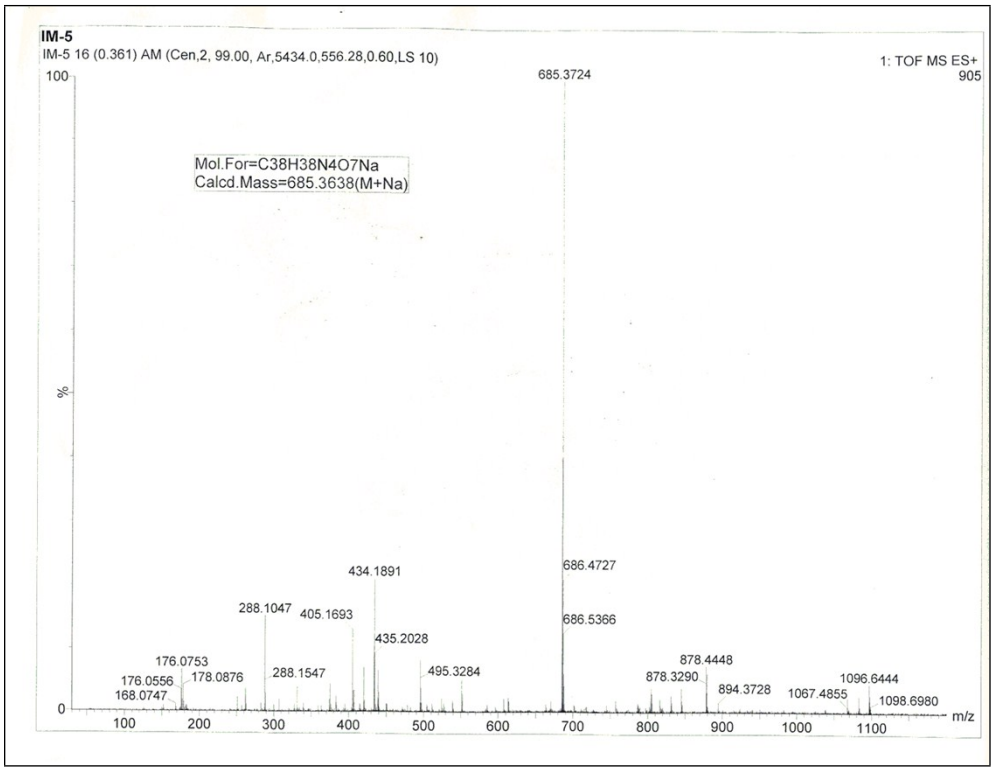

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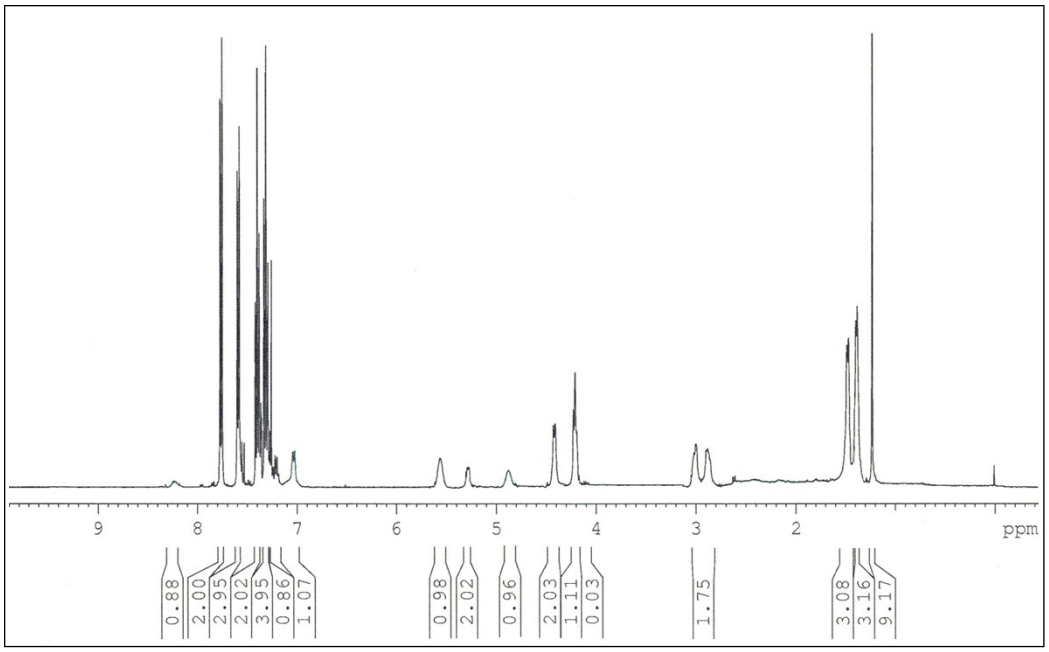

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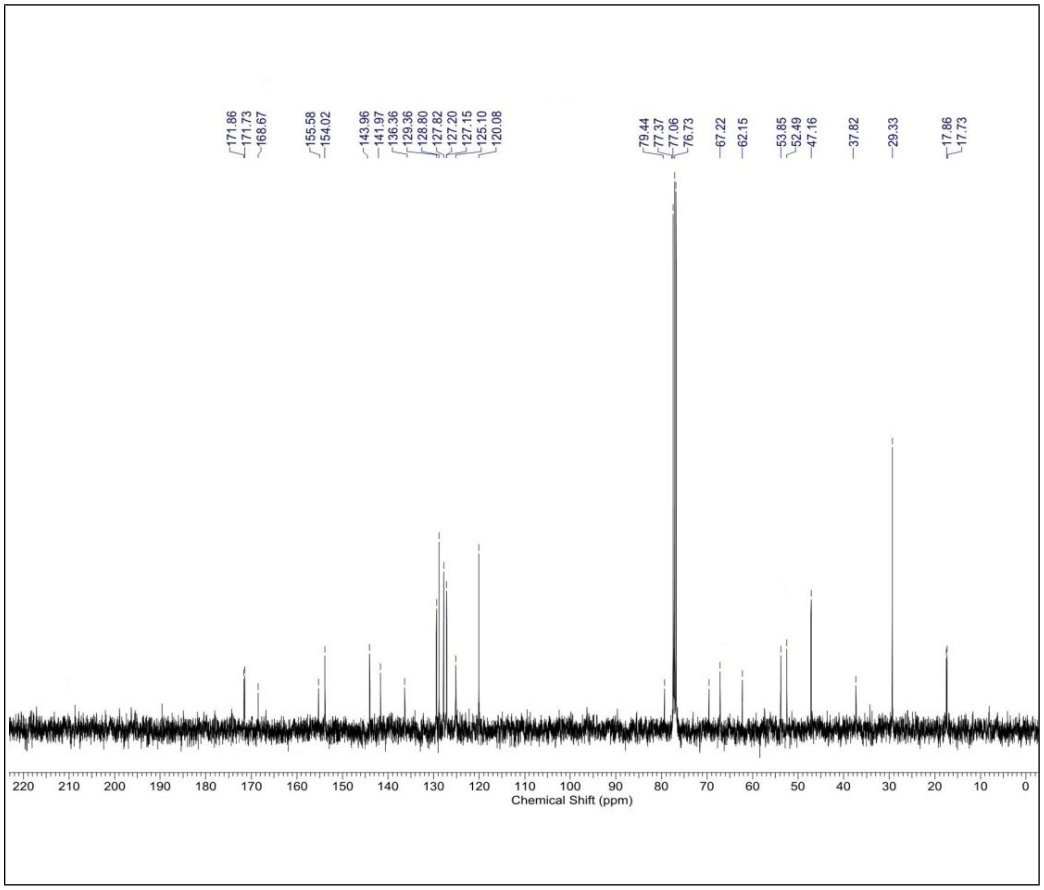

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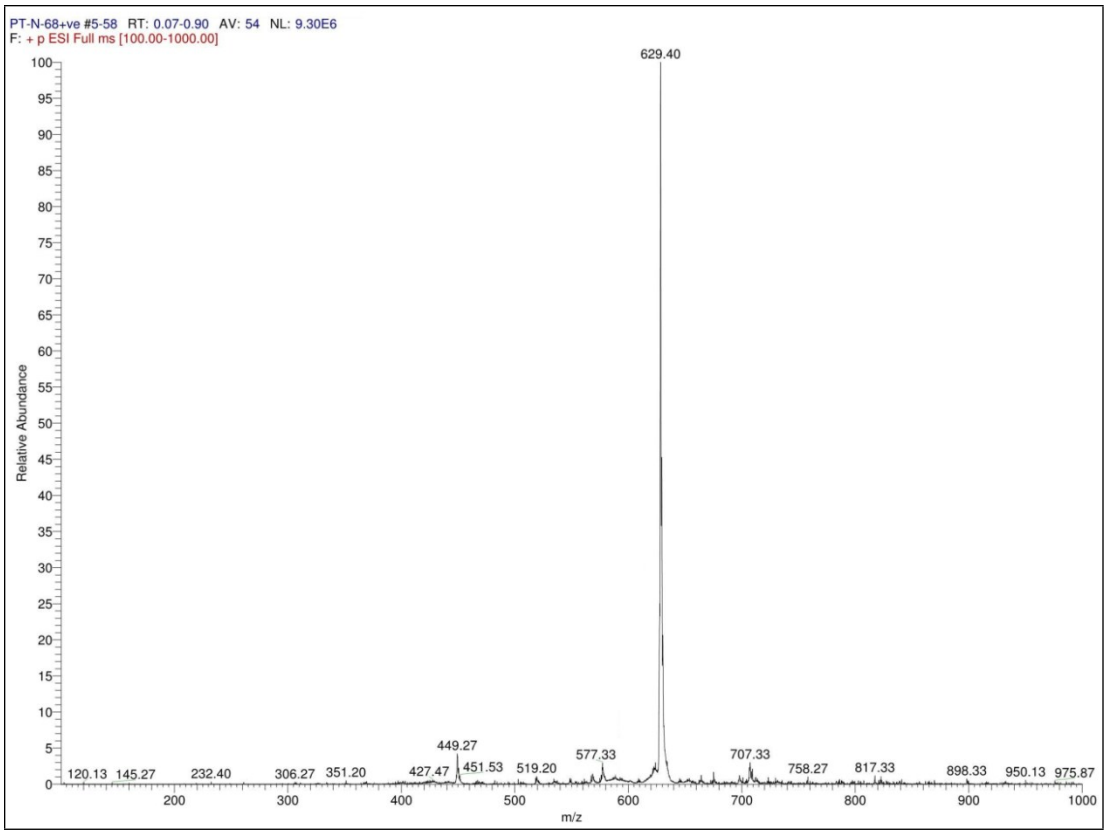

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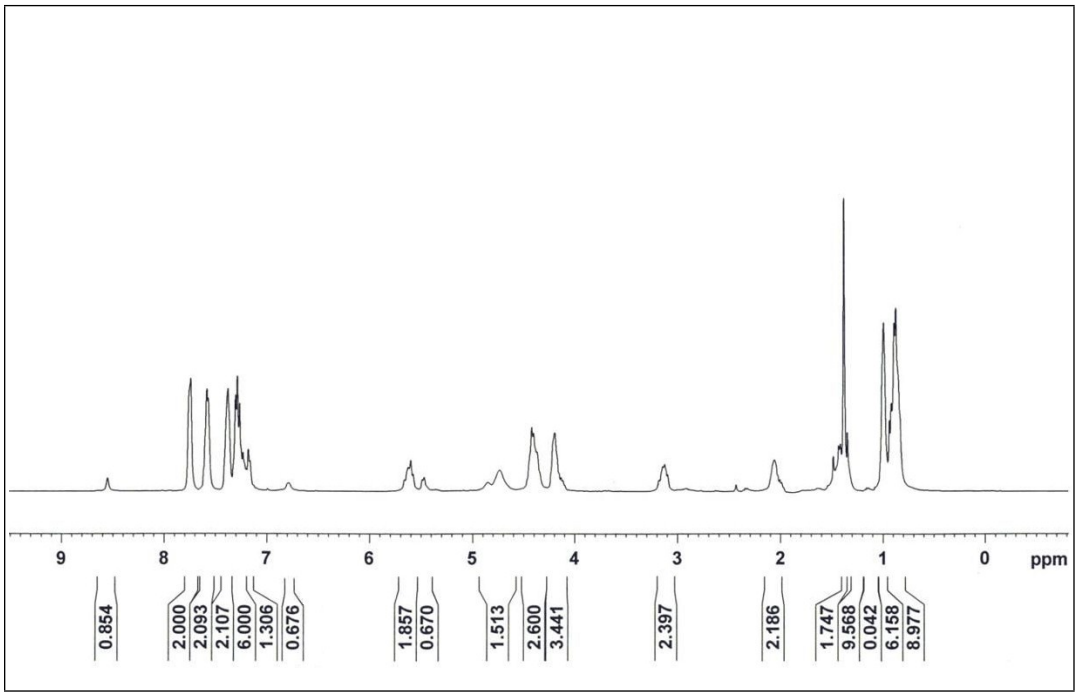

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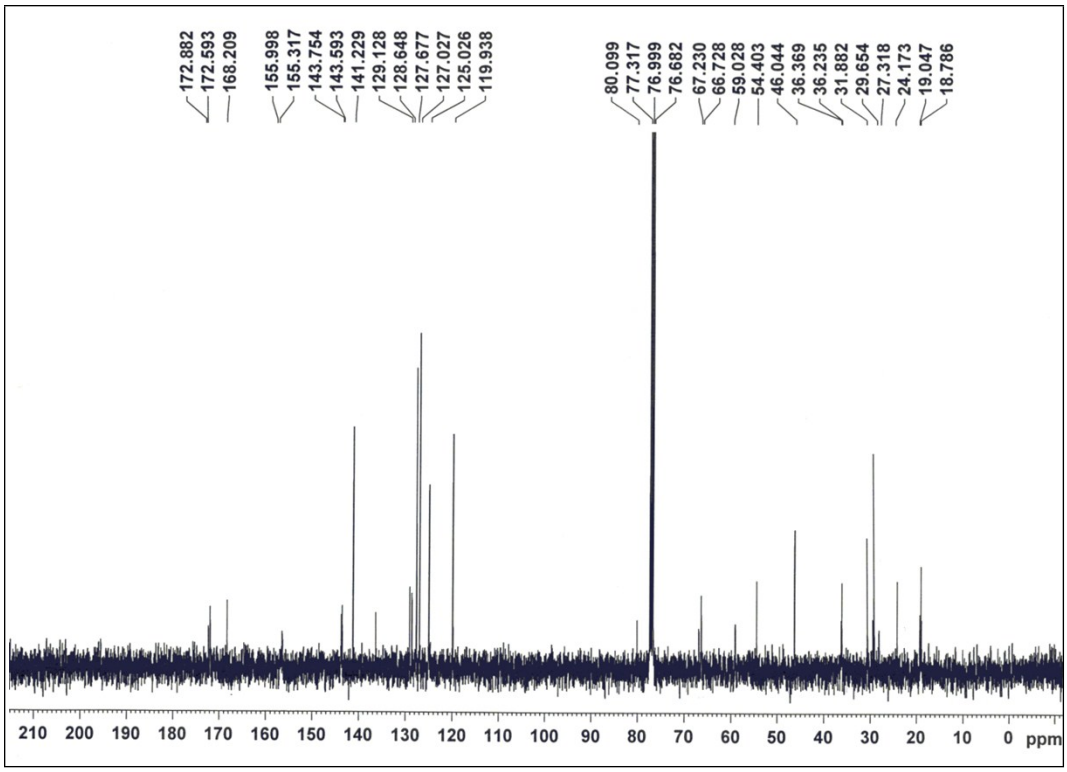

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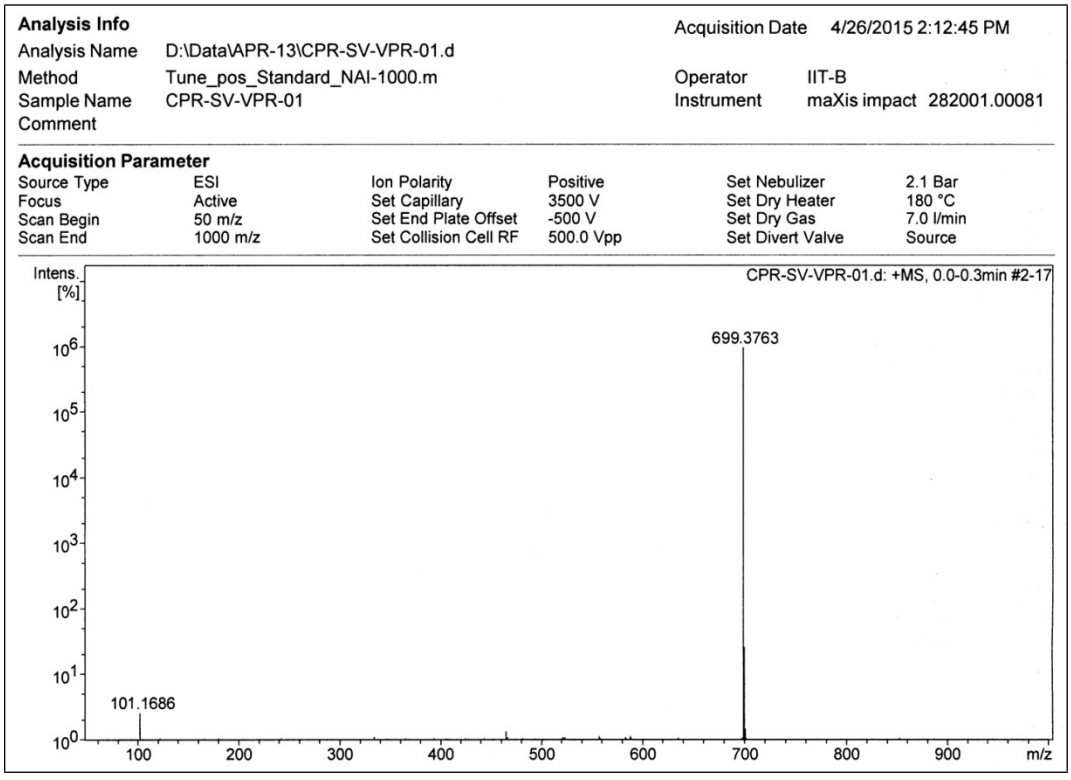

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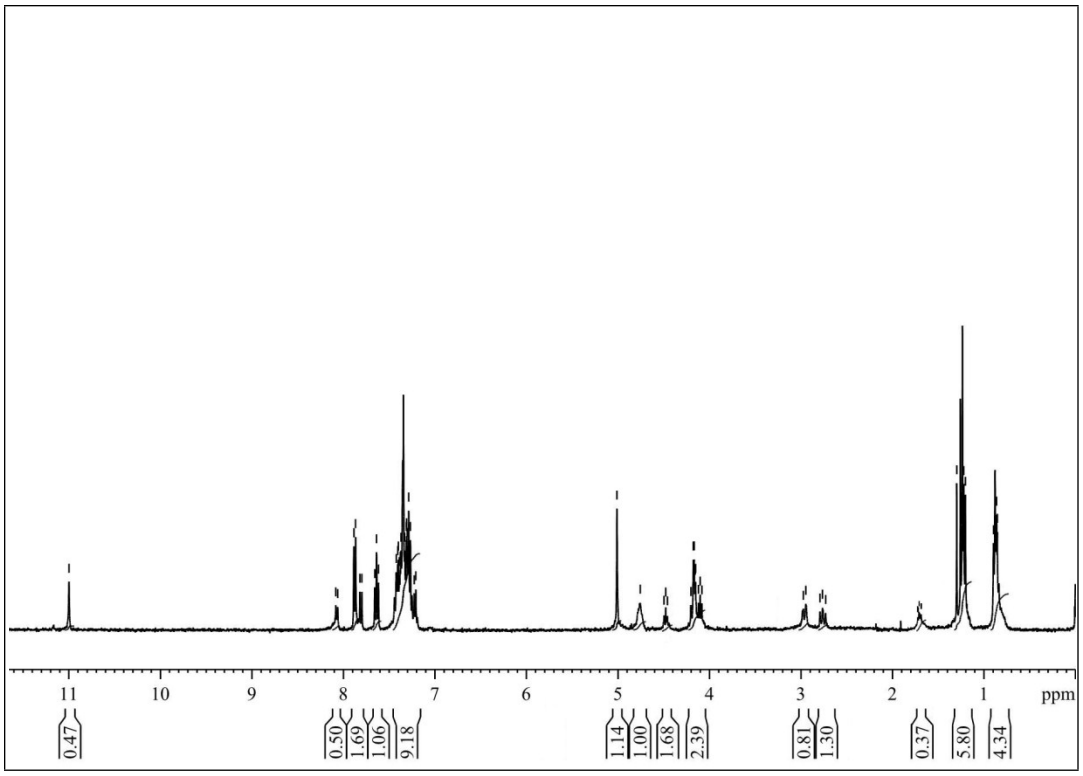

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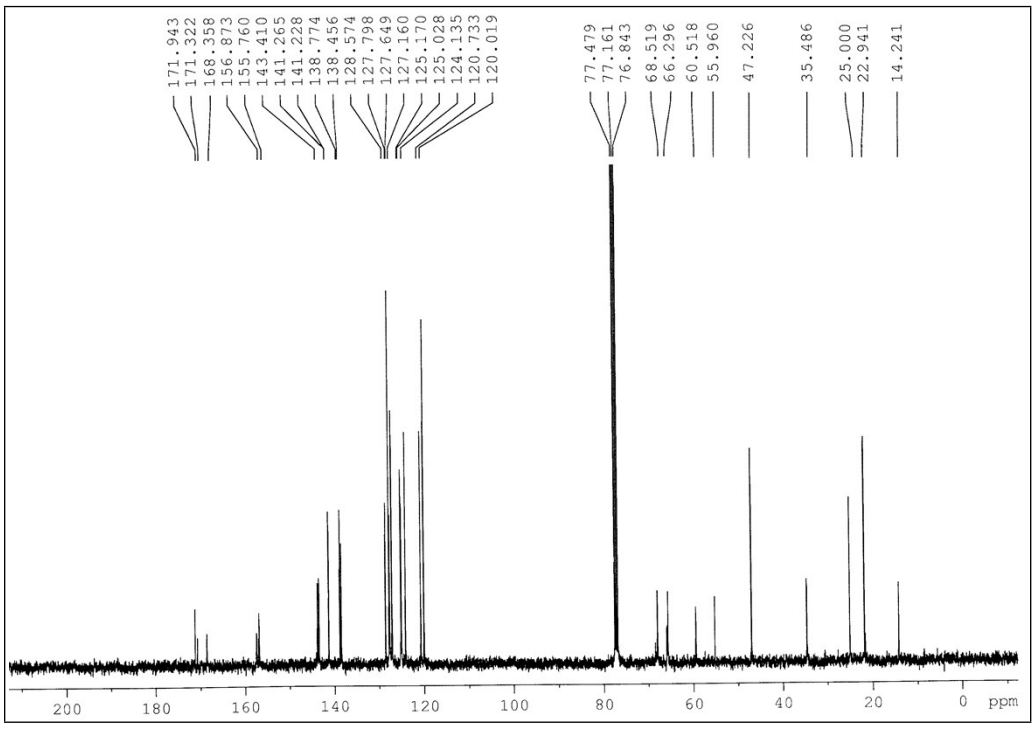

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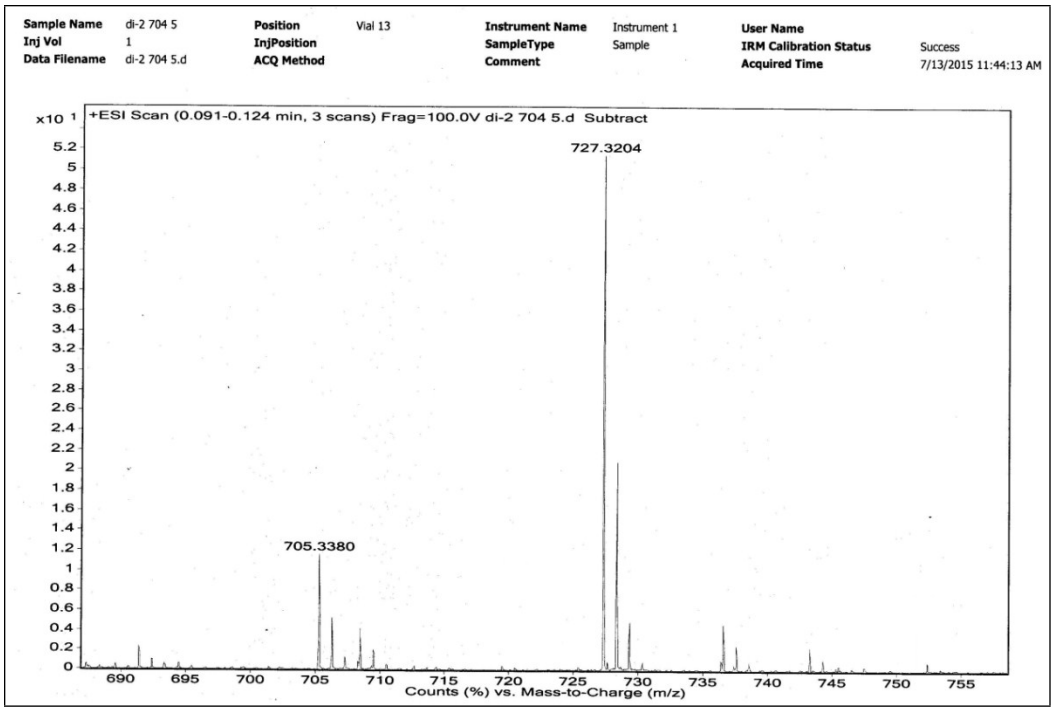

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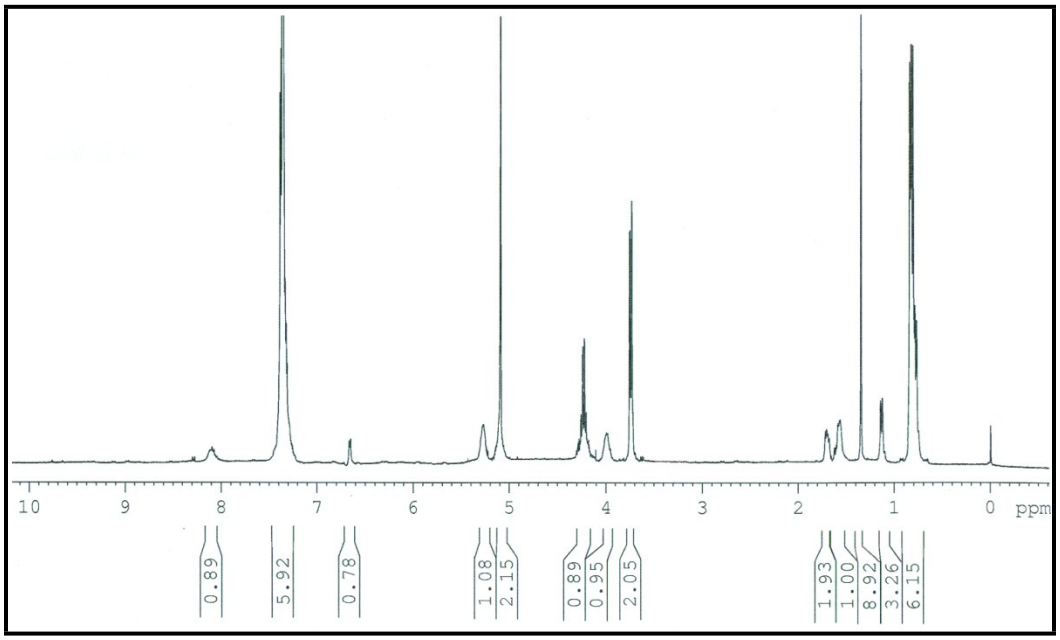

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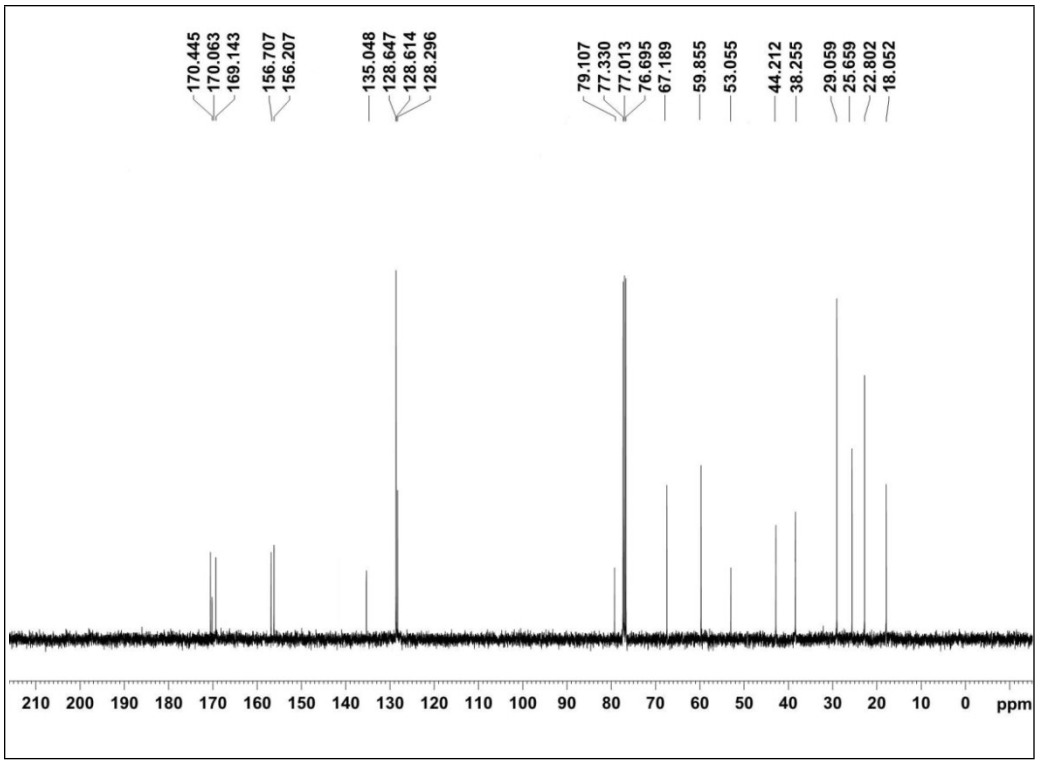

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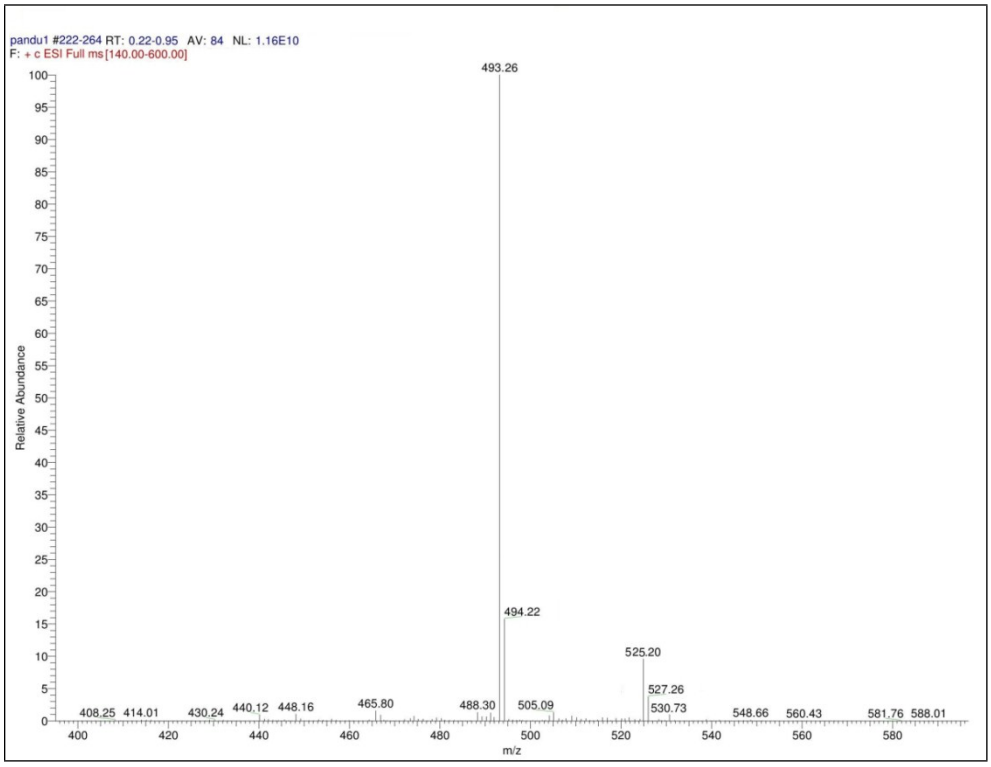

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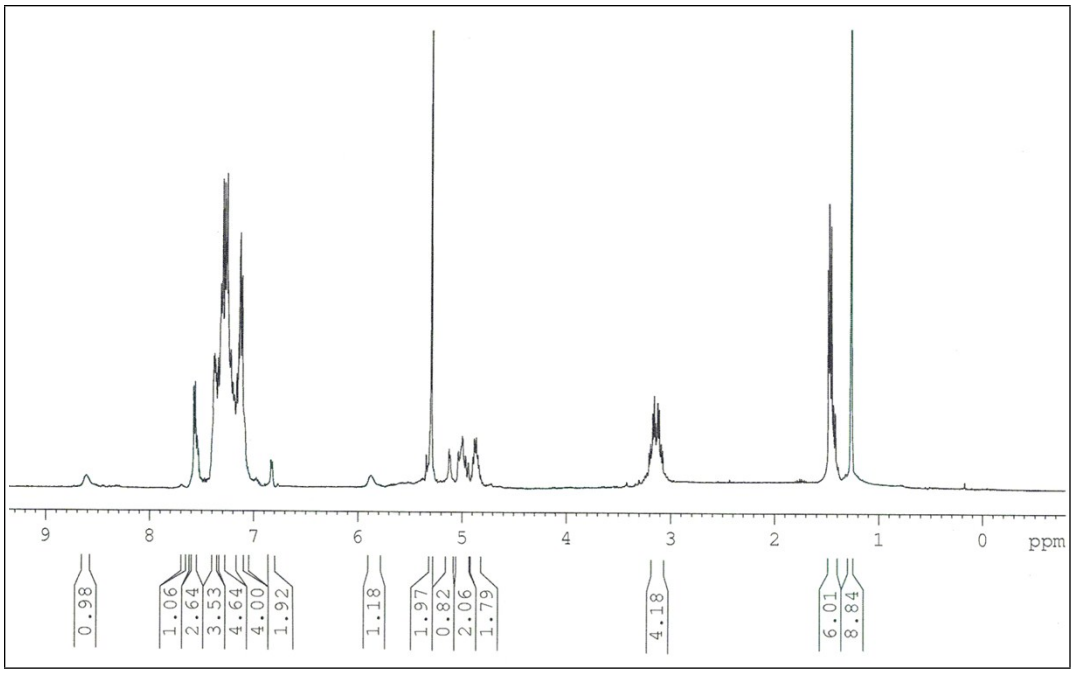

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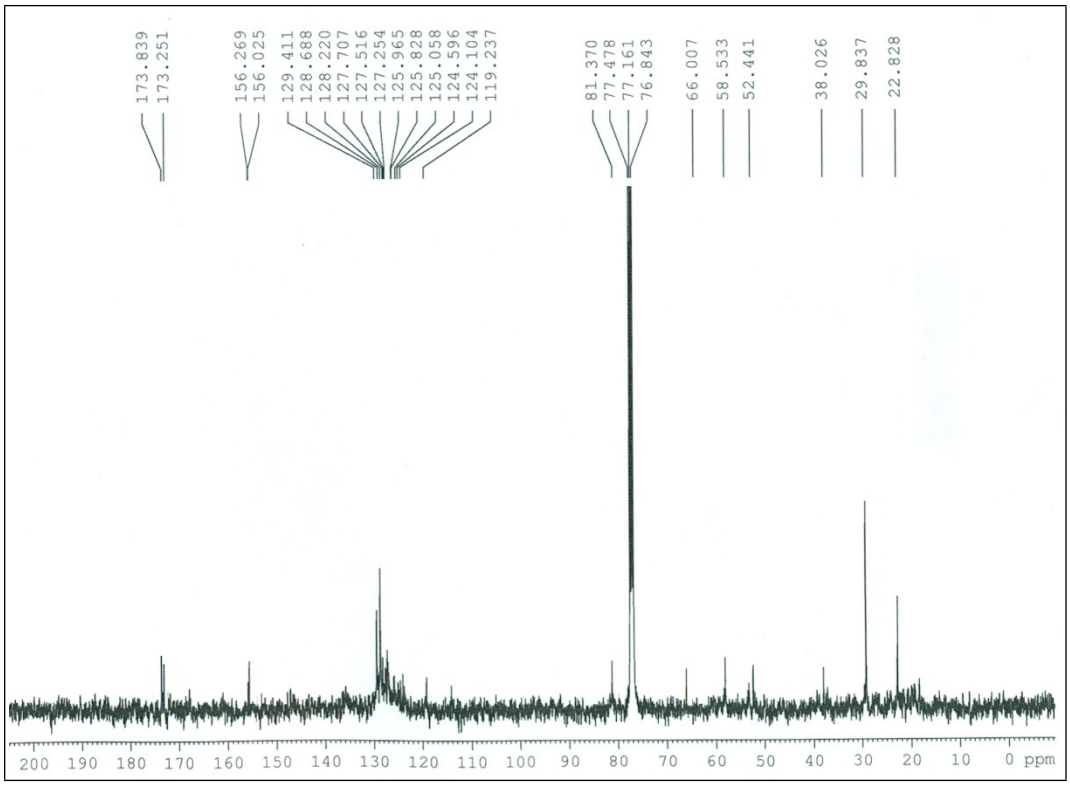

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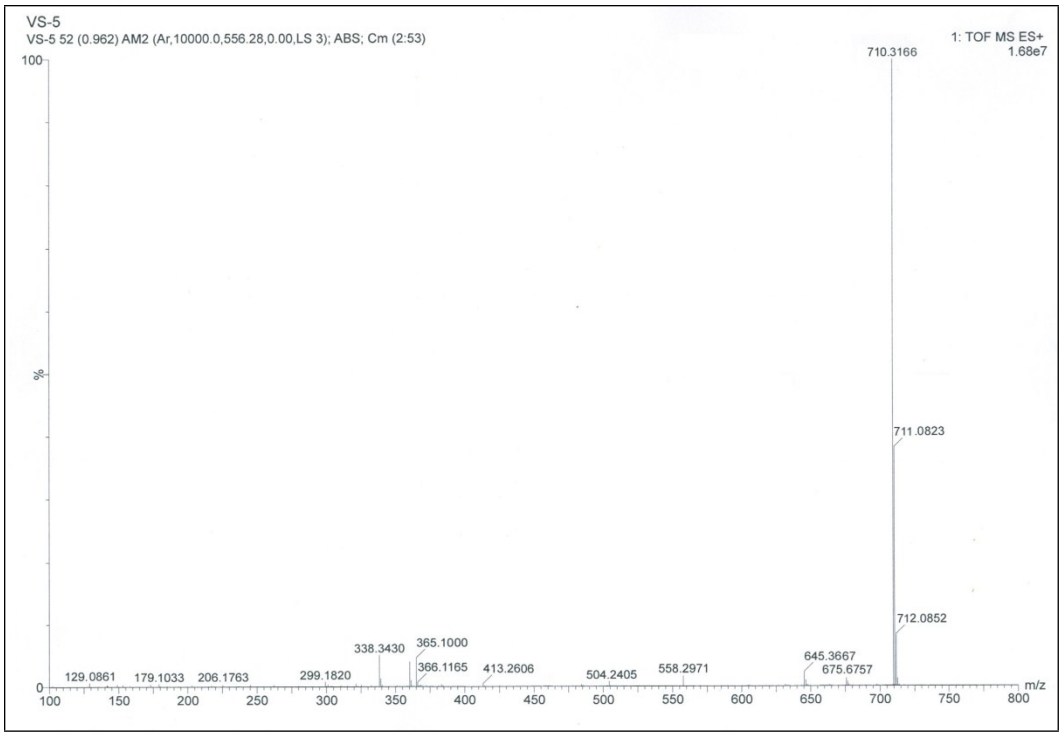

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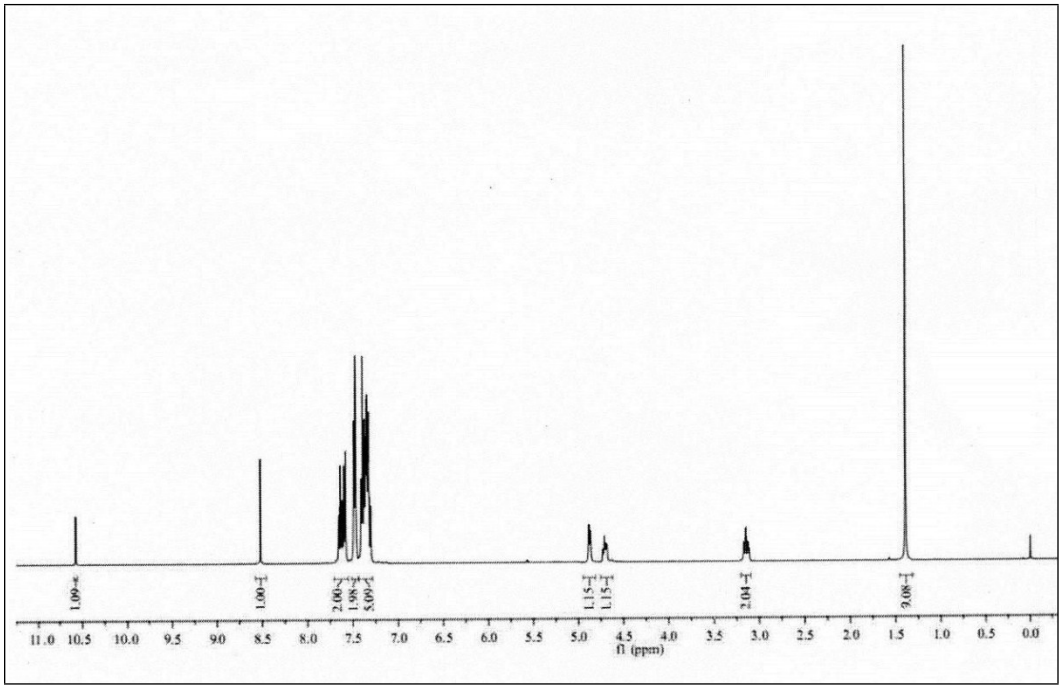
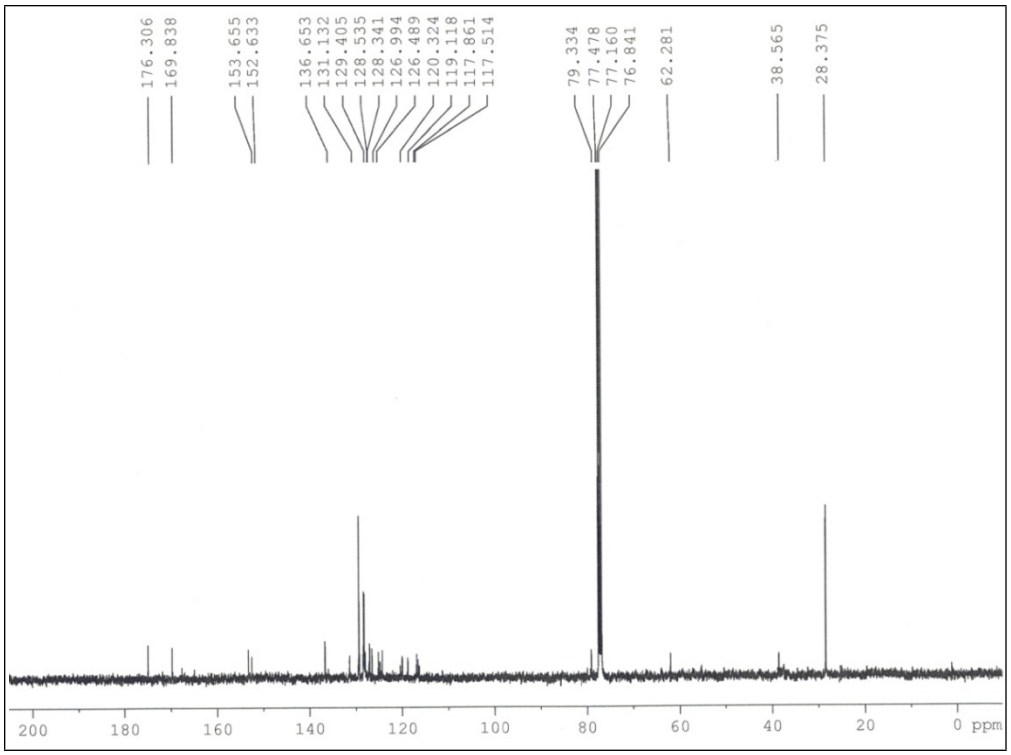
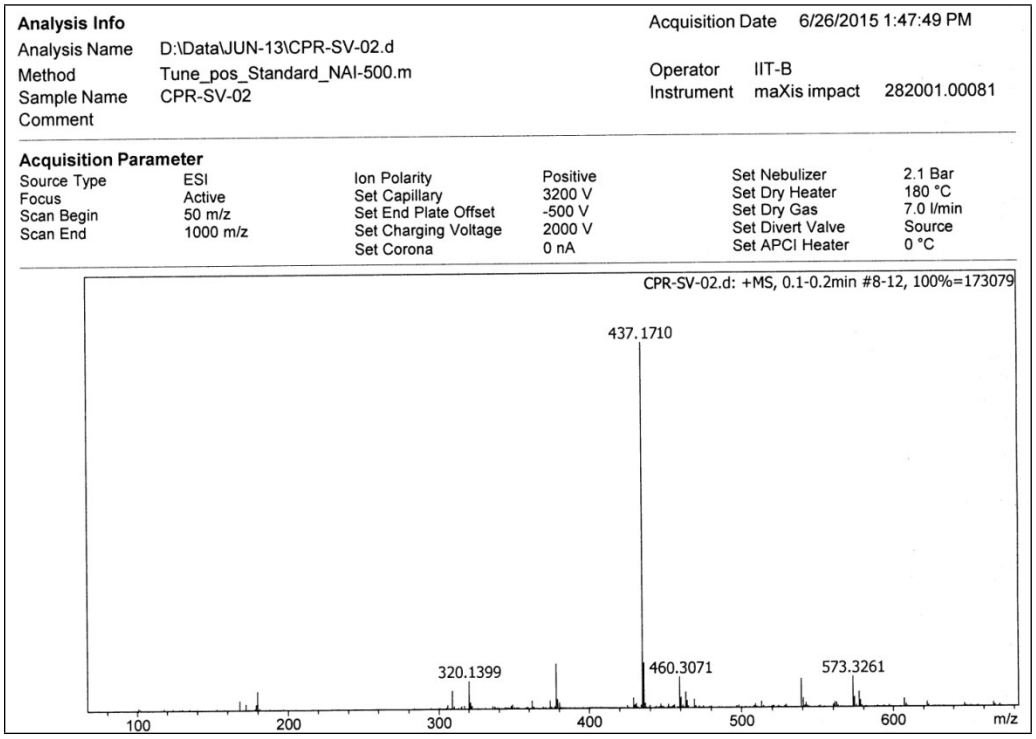


^{1}H NMR Spectrum of Compound (S)-tert-butyl(1-oxo-1-(2-oxo-2H-chromene-3-carboxamido)-3-phenylpropan-2-yl)carbamate 10a



^{13}C NMR Spectrum of Compound (S)-tert-butyl(1-oxo-1-(2-oxo-2H-chromene-3-carboxamido)-3-phenylpropan-2-yl)carbamate 10a



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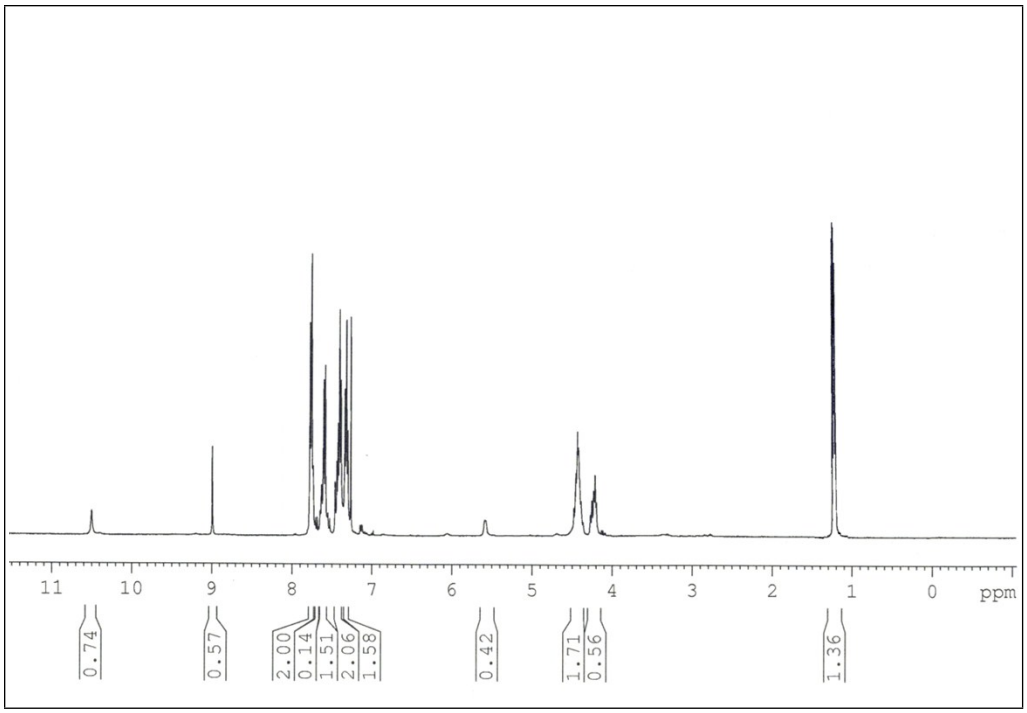
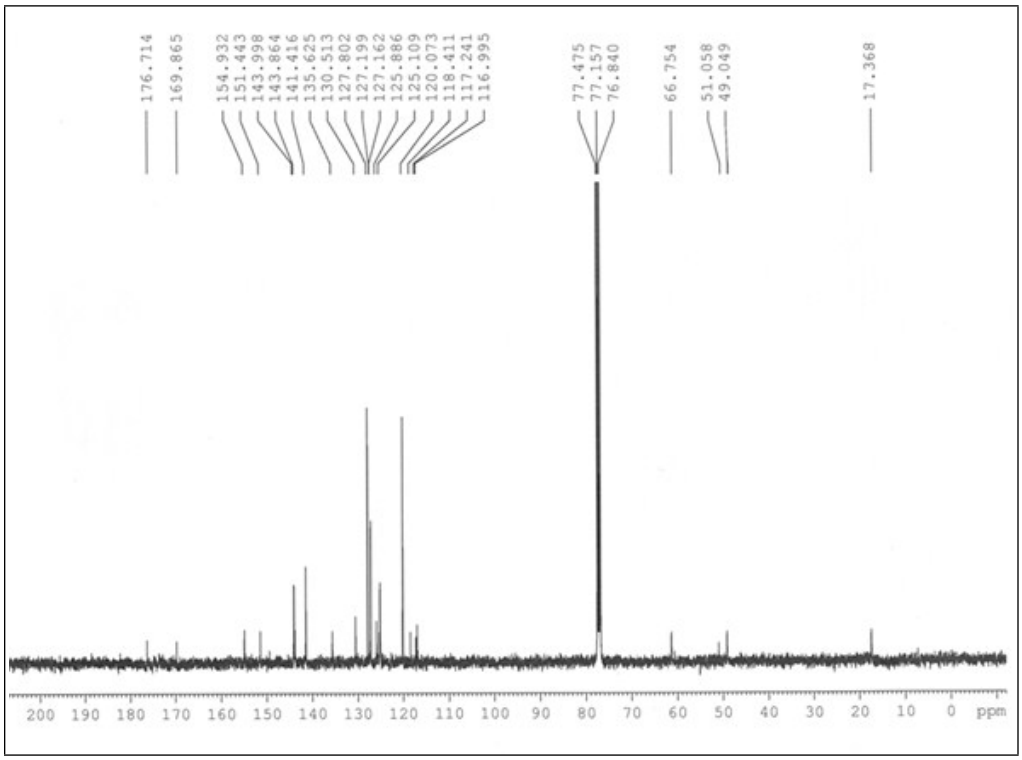
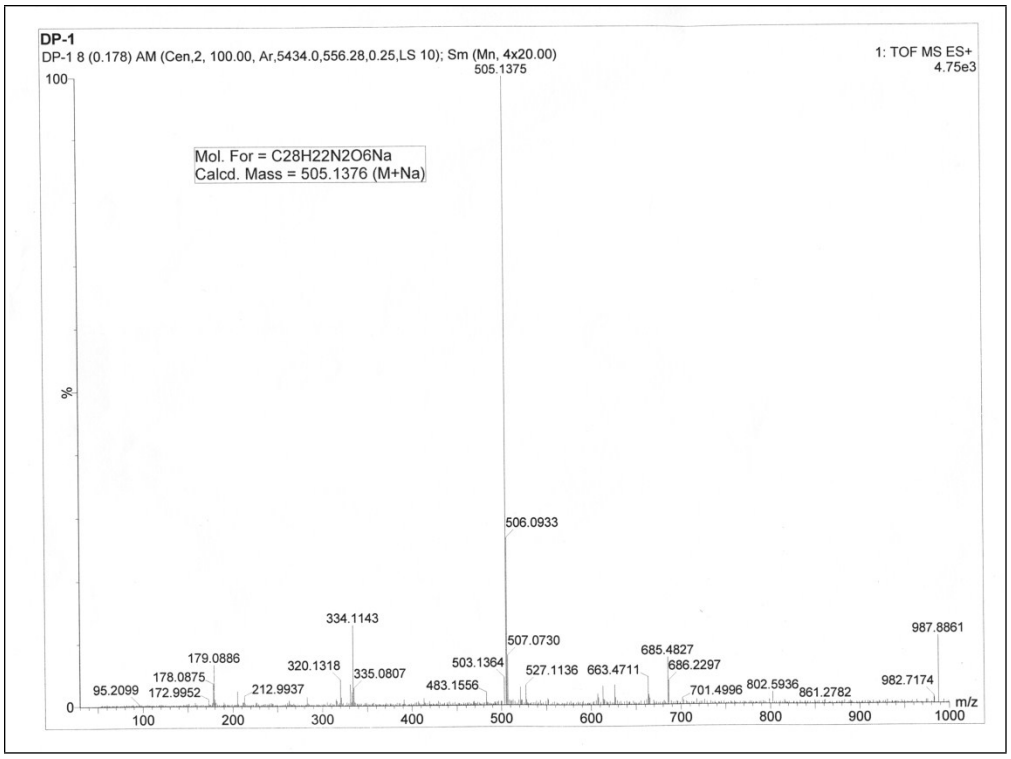


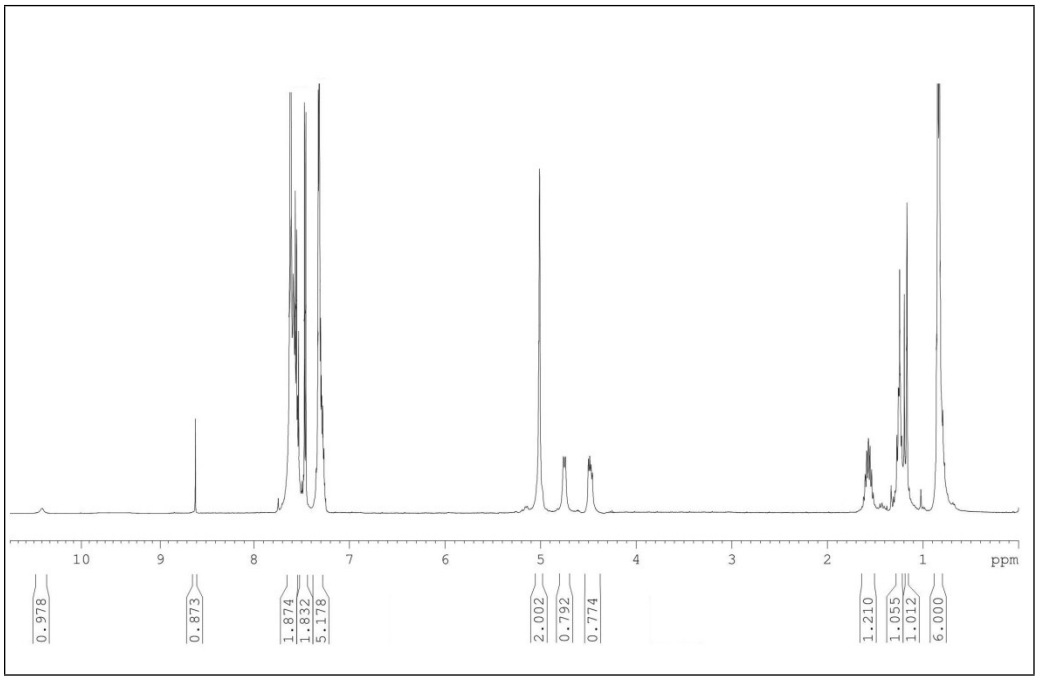
[1]H NMR Spectrum of Compound (S)-(9H-fluoren-9-yl)methyl(1-oxo-1-(2-oxo-2H-chromene-3-carboxamido)propan-2-yl)carbamate 10b



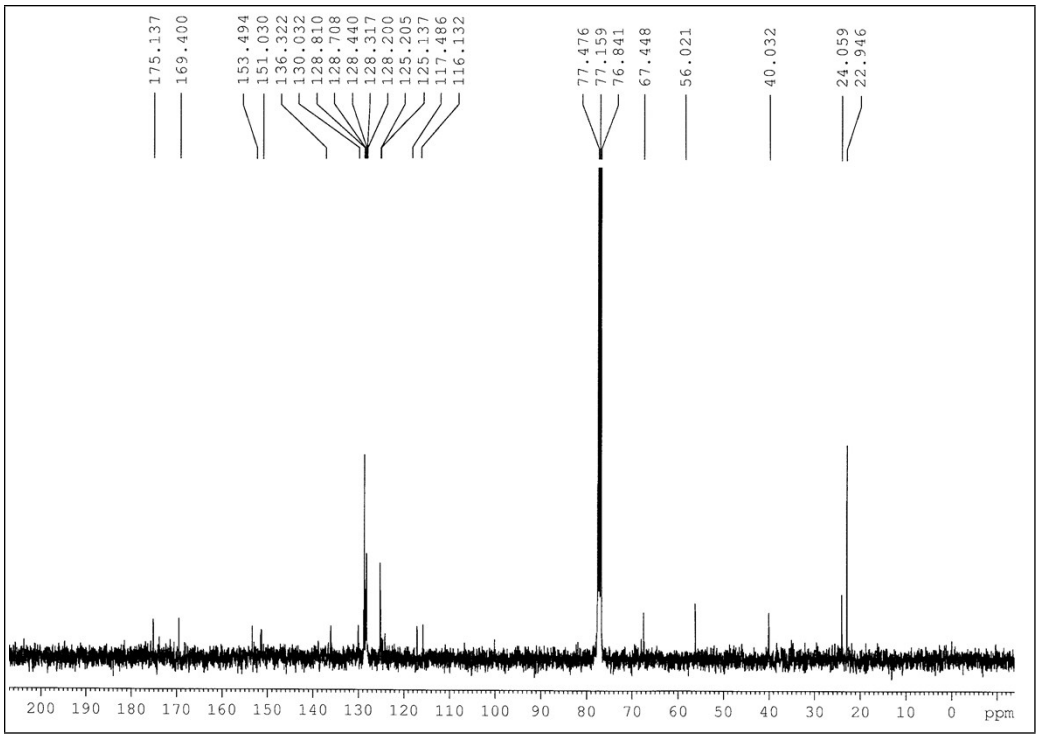
[13]C NMR Spectrum of Compound (S)-(9H-fluoren-9-yl)methyl(1-oxo-1-(2-oxo-2H-chromene-3-carboxamido)propan-2-yl)carbamate 10b



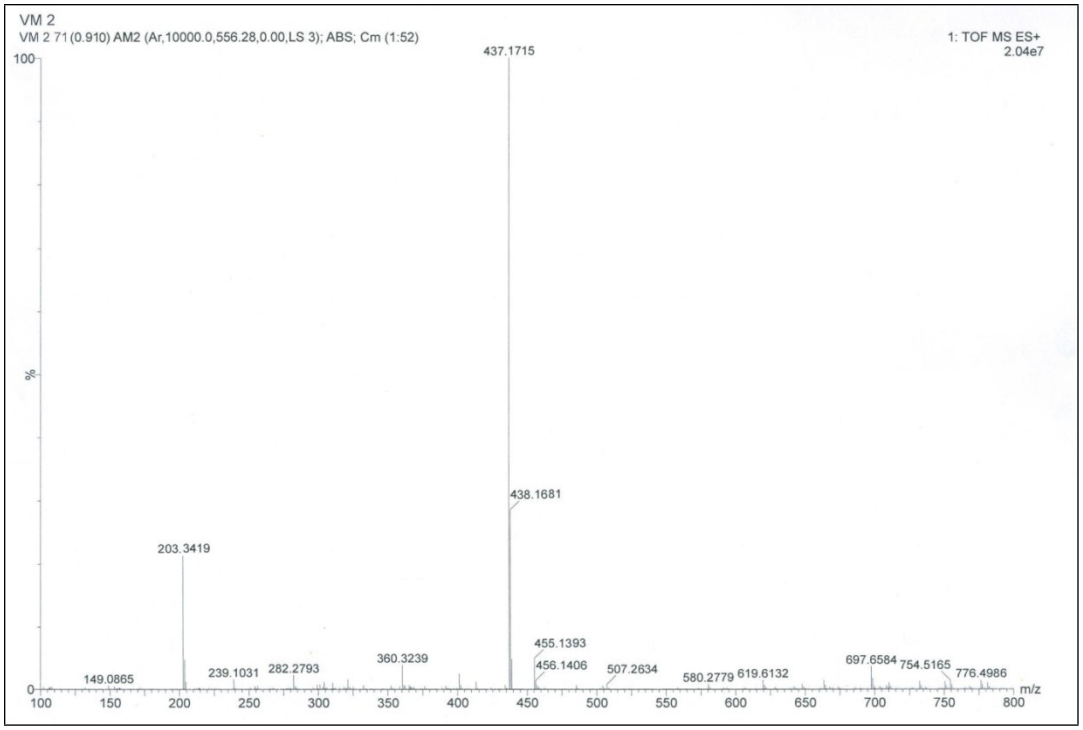
HRMS Spectrum of Compound (S)-(9H-fluoren-9-yl)methyl(1-oxo-1-(2-oxo-2H-chromene-3-carboxamido)propan-2-yl)carbamate 10b



^{1}H NMR Spectrum of Compound (S)-benzyl(4-methyl-1-oxo-1-(2-oxo-2H-chromene-3-carboxamido)pentan-2-yl)carbamate 10c



^{13}C NMR Spectrum of Compound (S)-benzyl(4-methyl-1-oxo-1-(2-oxo-2H-chromene-3-carboxamido)pentan-2-yl)carbamate 10c



HRMS Spectrum of Compound (S)-benzyl(4-methyl-1-oxo-1-(2-oxo-2H-chromene-3-carboxamido)pentan-2-yl)carbamate 10c