An efficient green chemistry protocol for the synthesis of novel spiropyrrolizidine compounds

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Supplementary data

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General remarks

Chalcones have been prepared by the condensation of ketones with aldehydes in mild basic medium at cold conditions. Isatin, acenaphthenequinone, L-Proline and L-Thiaproline were purchased from Sigma-Aldrich and were used as such without further purification. The melting points of all compounds were determined on Guna apparatus using capillary tube and are uncorrected. The purities of the compounds were checked by TLC using precoated silica gel plates with n-hexane:ethyl acetate (6:4) as eluent. ^{1}H, ^{13}C and 2D NMR spectra were recorded on a Bruker Avance spectrophotometer at 400, 100 and 500/125 respectively using TMS as reference. High Resolution Mass Spectra of representative compounds were recorded on maXis 10138 Mass spectrometer at 70 eV. Elemental microanalyses were carried out on a Perkin-Elmer elemental analyzer Model 240C and a Thermo Finnigan analyser series Flash EA1112. Single crystal X-ray diffraction was performed on a Bruker-Nonius SMART APEX CCD area detector system using graphite monochromated, Mo-Kα (λ=0.71073) radiation.

Synthesis of spiro pyrrolizidine 6a-6n:

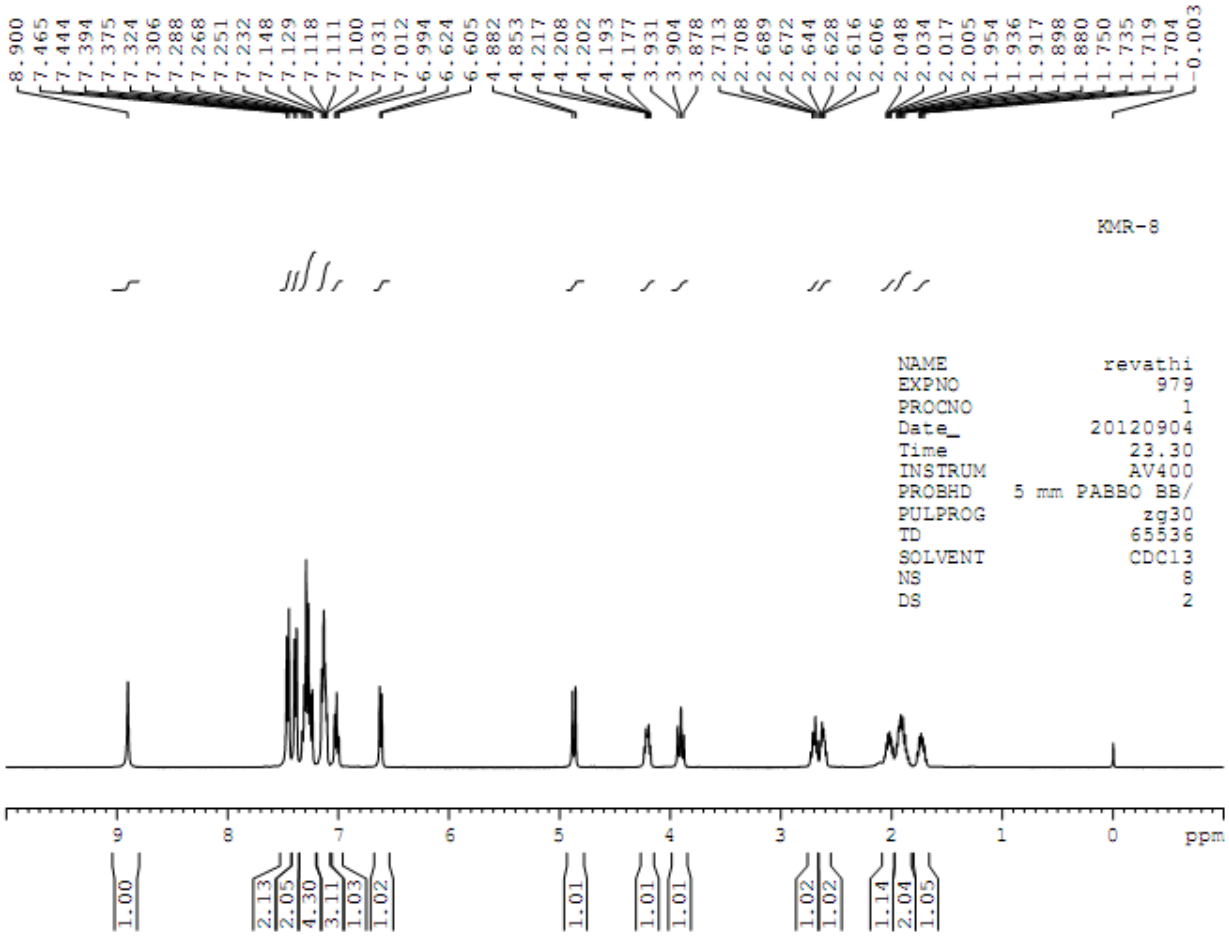
To the solution of 0.0005mol of isatin and 0.0005mol of L-Proline/ L-Thiaproline/ Sarcosine in 10 ml of methanol, 0.0005 mol of chalcone was added and stirred at room temperature until the completion of reaction as indicated by TLC. After the completion of reaction, solvent was removed in a rotovapor and the crystallized product was obtained in excellent yields and purity.

Synthesis of spiro pyrrolizidine 8a and 8e:

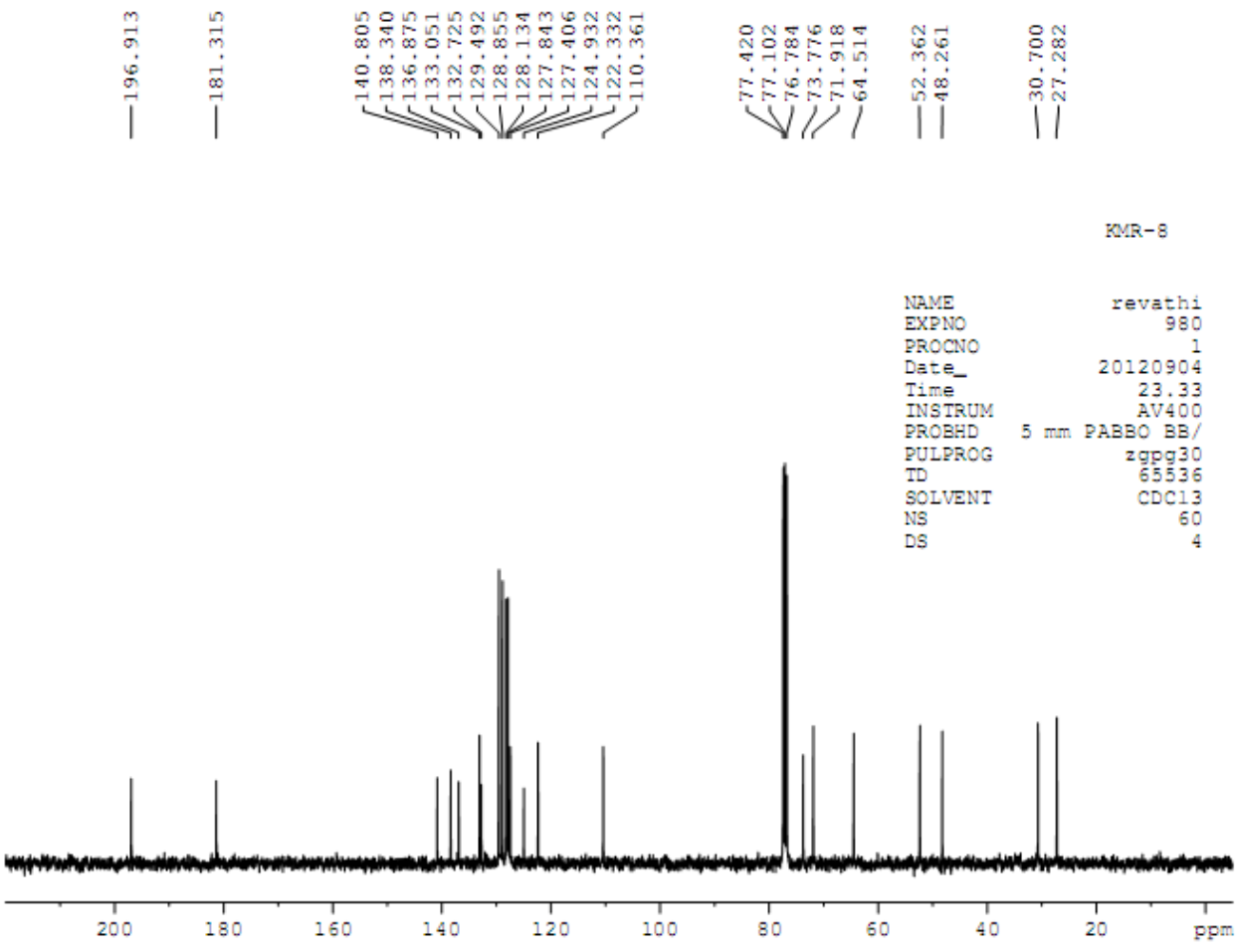
To the solution of 0.0005mol of isatin and 0.0005mol of L-Proline in 10 ml of methanol, 0.0005 mol of chalcone was added and stirred at reflux temperature until the completion of reaction as indicated by TLC. After the completion of reaction, solvent was removed in a rotovapor and the crystallized product was obtained in excellent yields and purity.

Synthesis of spiro pyrrolizidine 10a-10f:

To the solution of 0.0005mol of acenaphthenequinone and 0.0005mol of L-Proline/ L-Thiaproline in 10 ml of methanol, 0.0005 mol of chalcone was added and stirred at reflux temperature until the completion of reaction as indicated by TLC. After the completion of reaction, solvent was removed in a rotovapor and the crystallized product was obtained in excellent yields and purity.



^{1}H NMR Spectrum of **6b**



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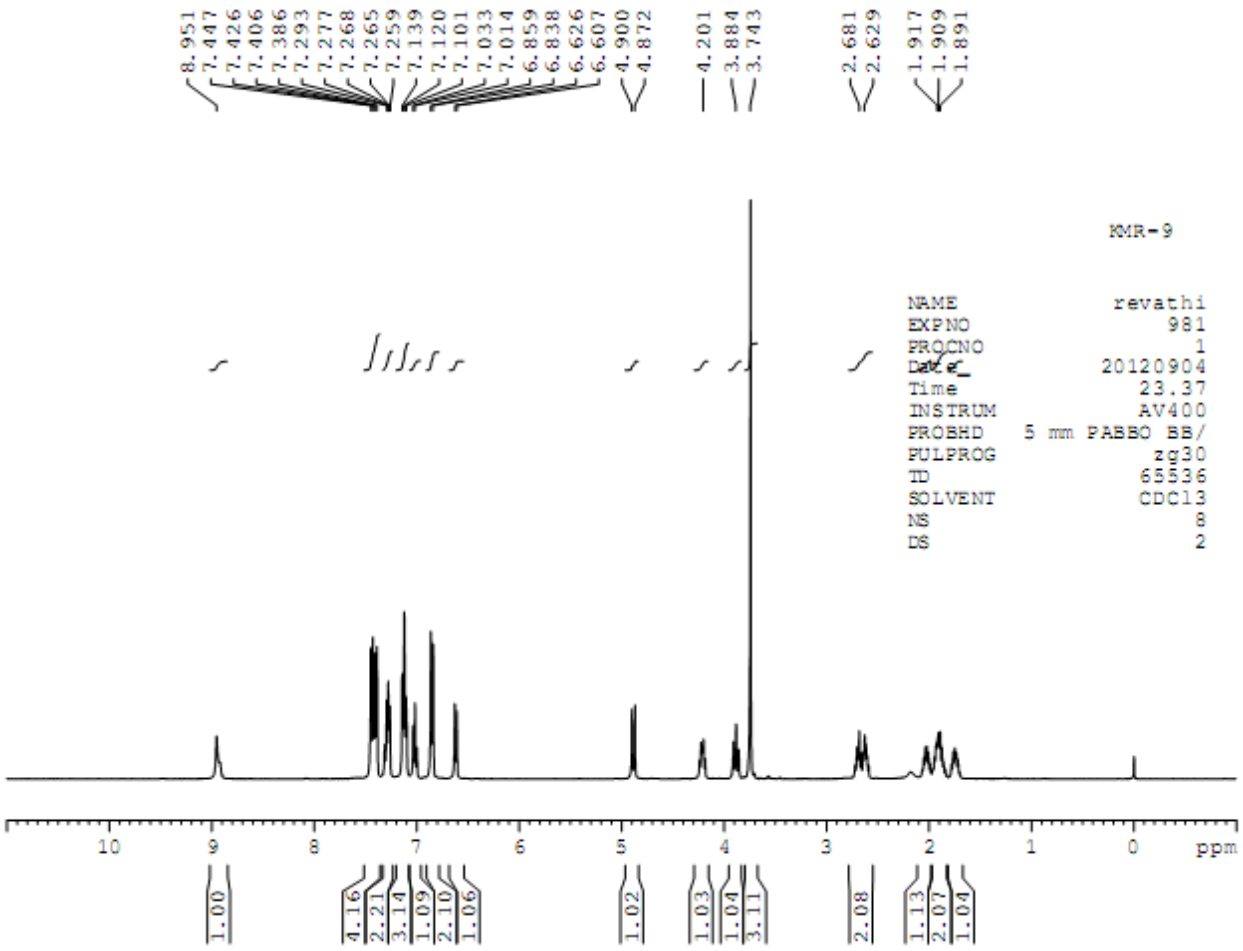
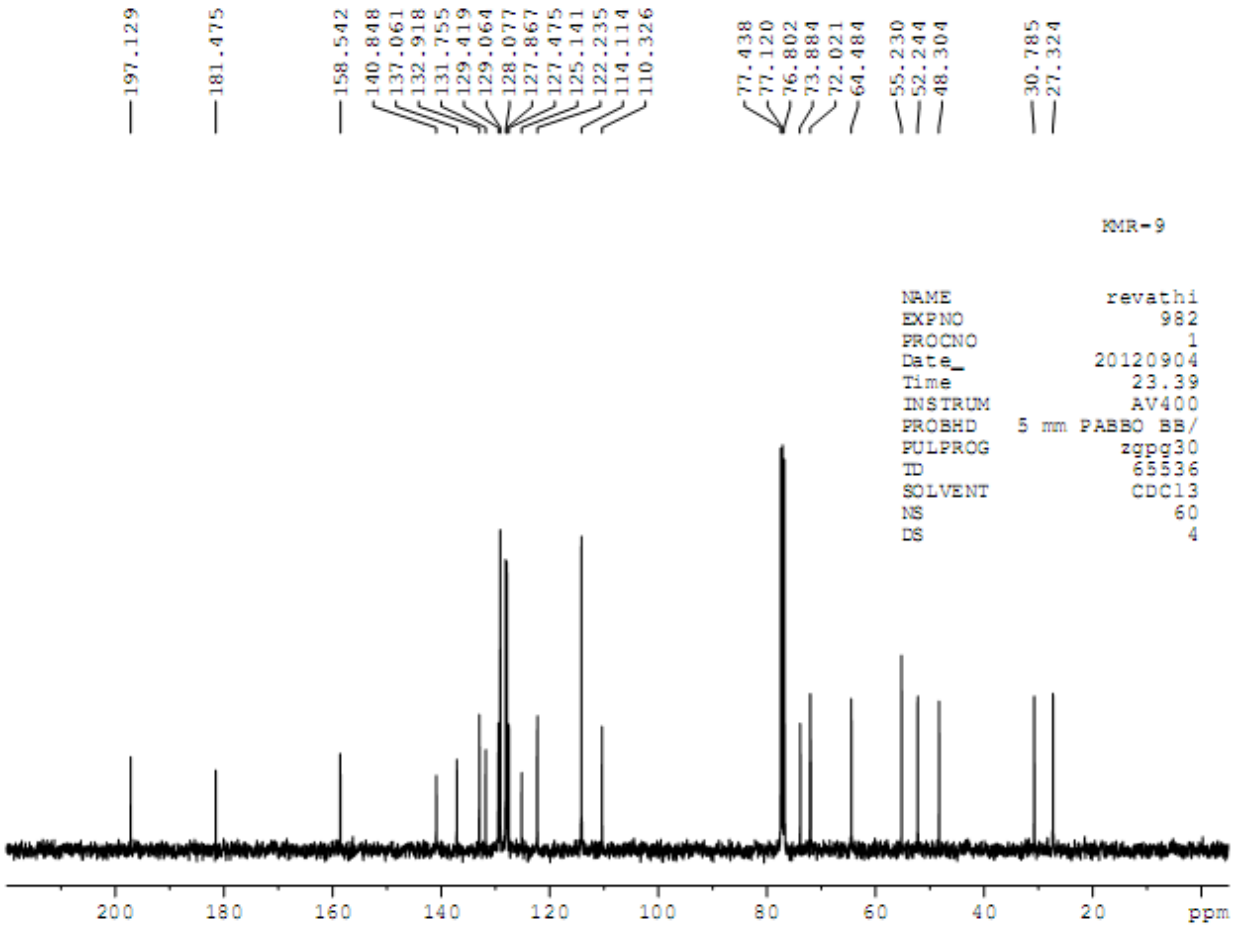
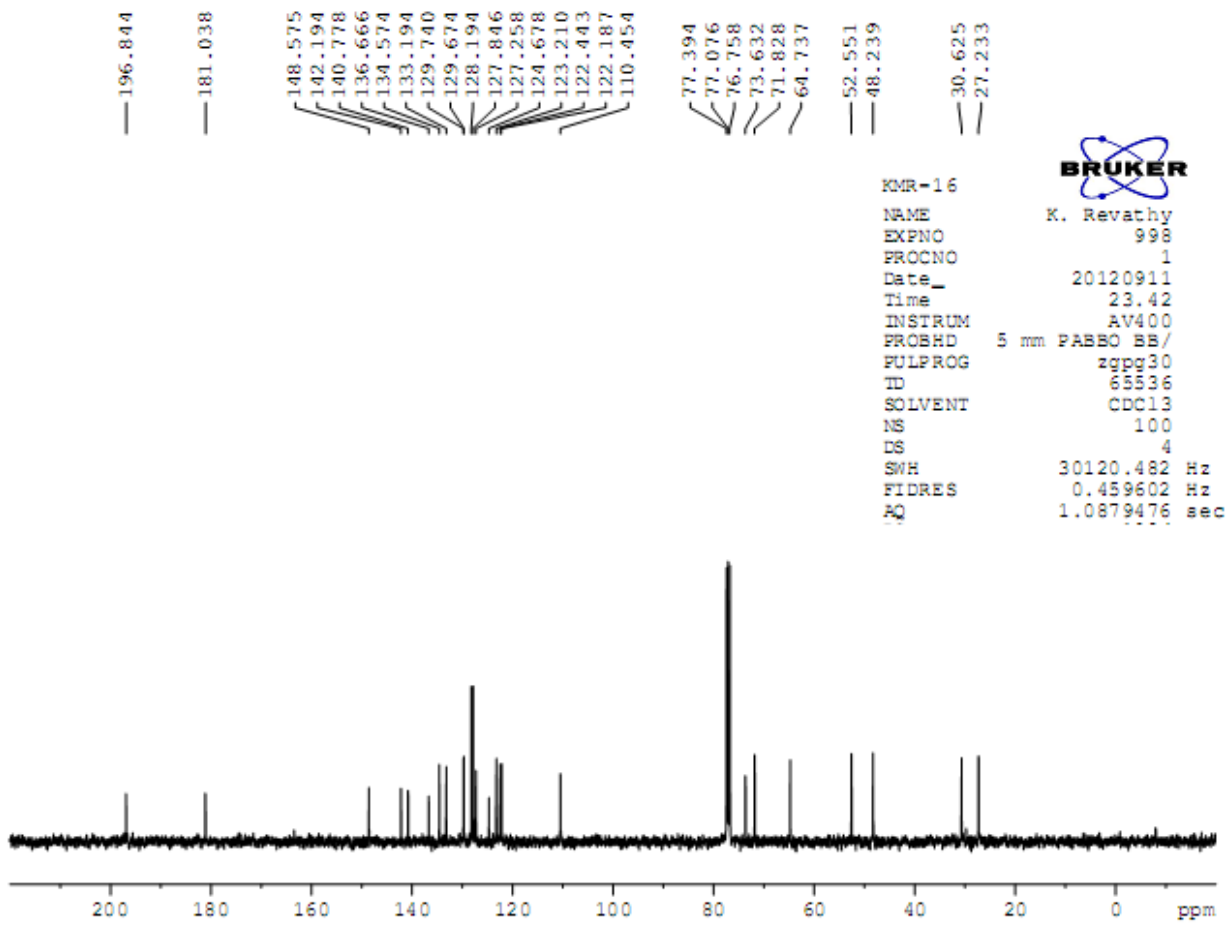


^{1}H NMR Spectrum of **6d**



^{13}C NMR Spectrum of **6d**



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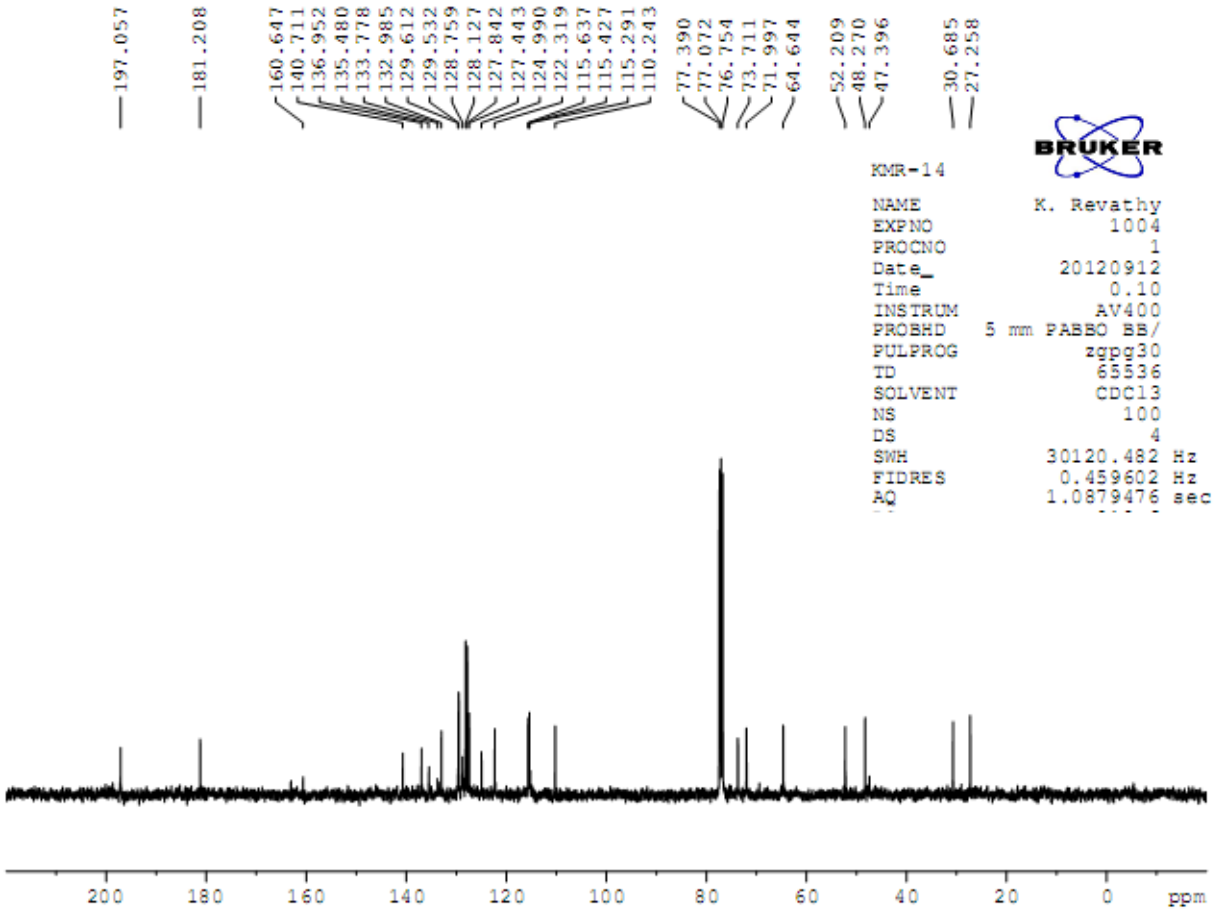

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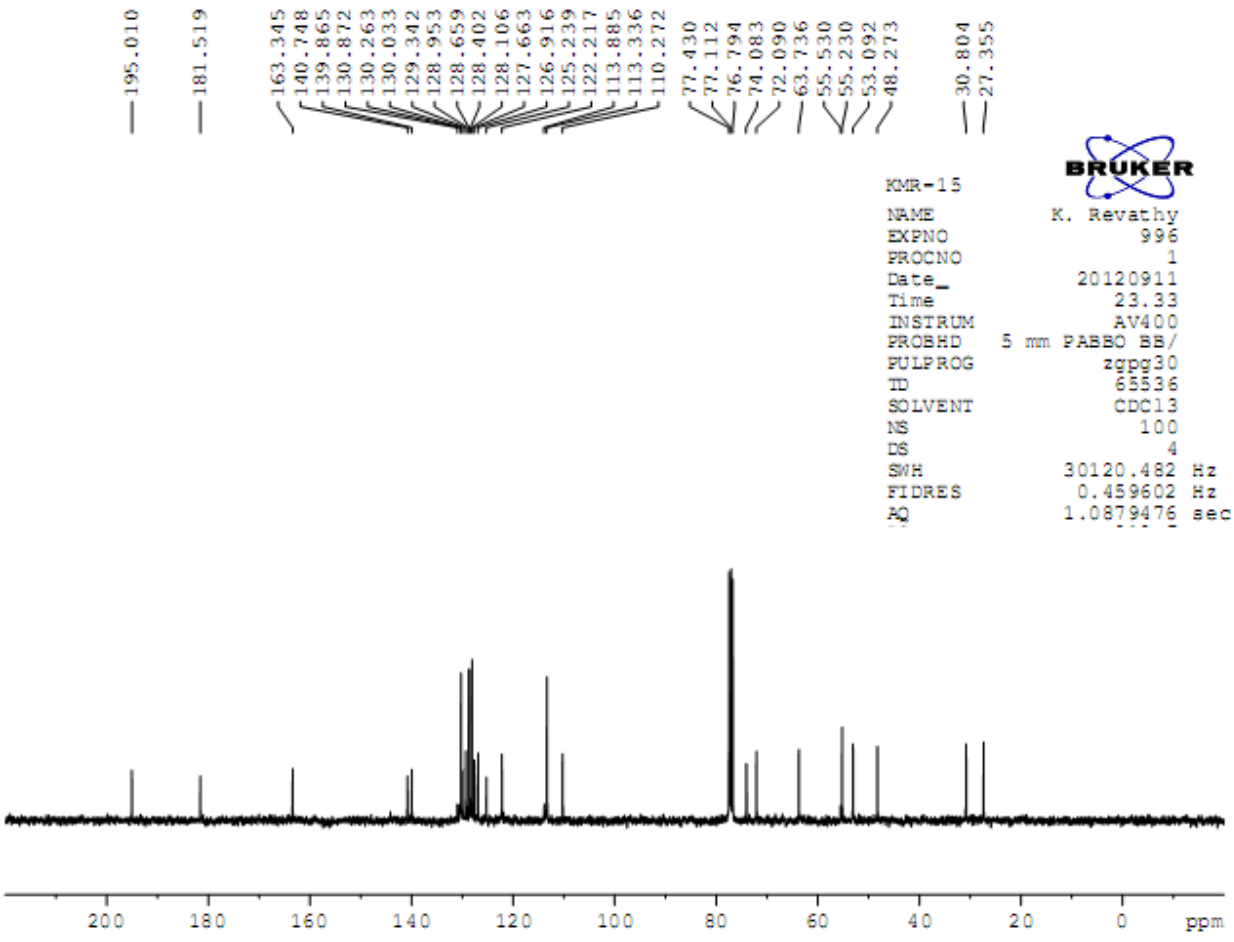

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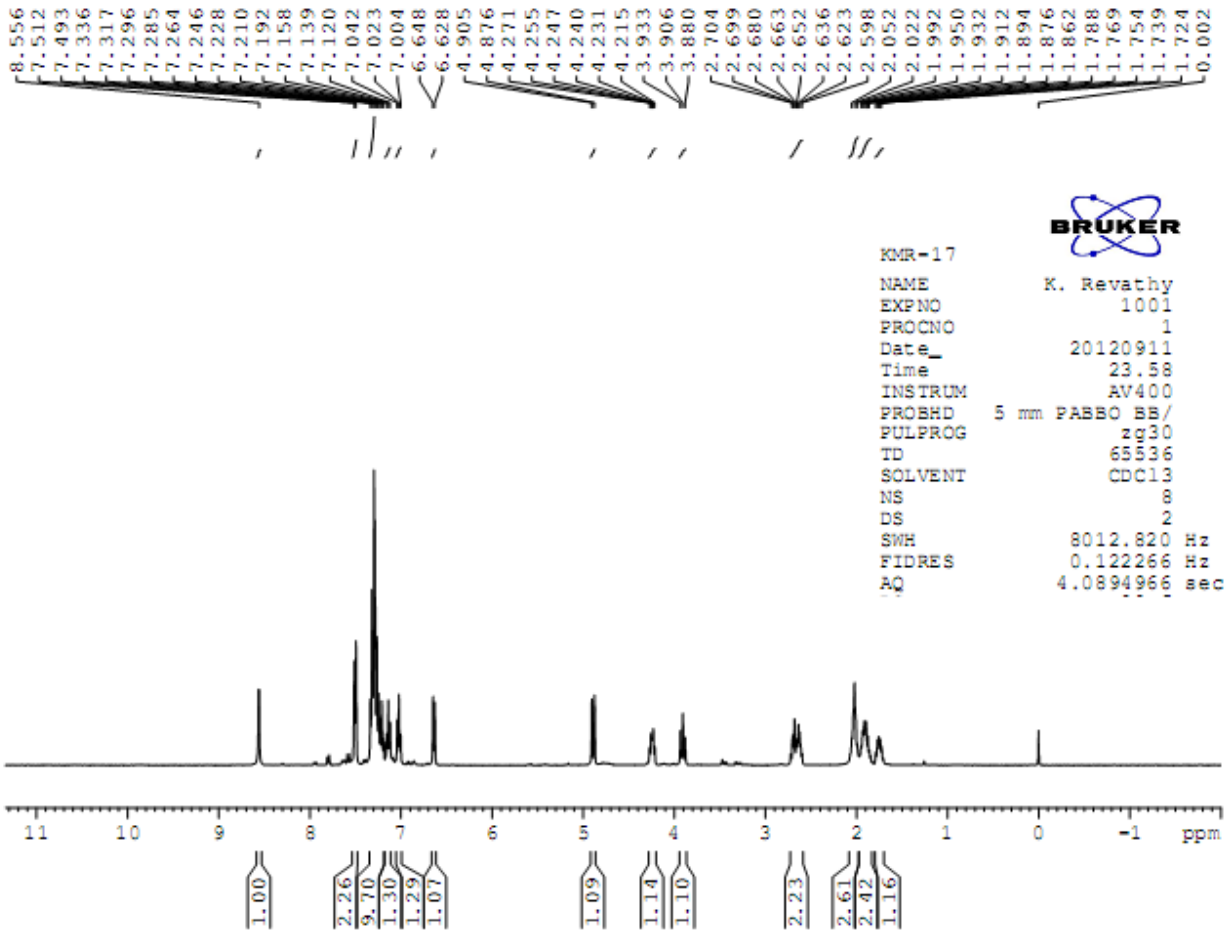

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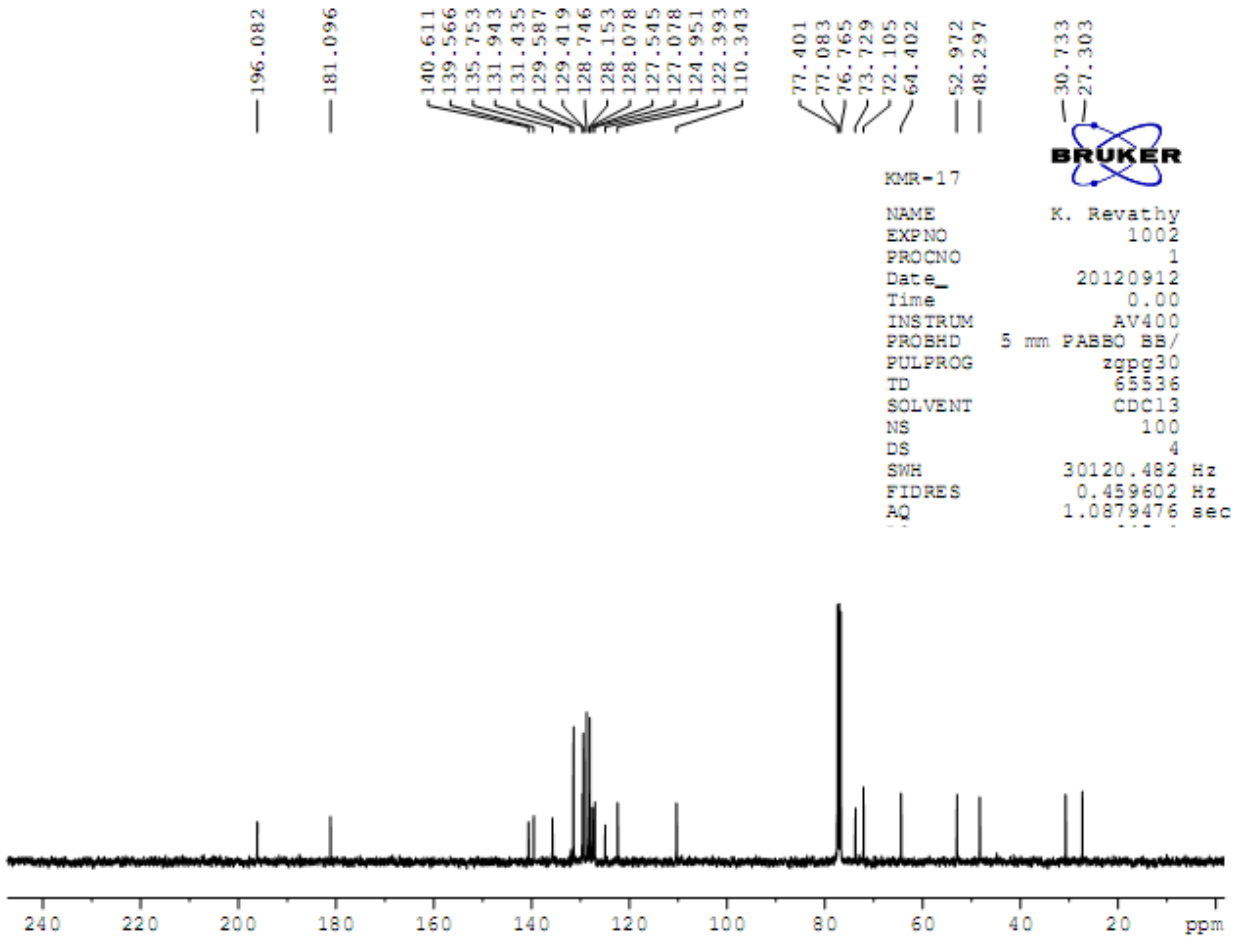
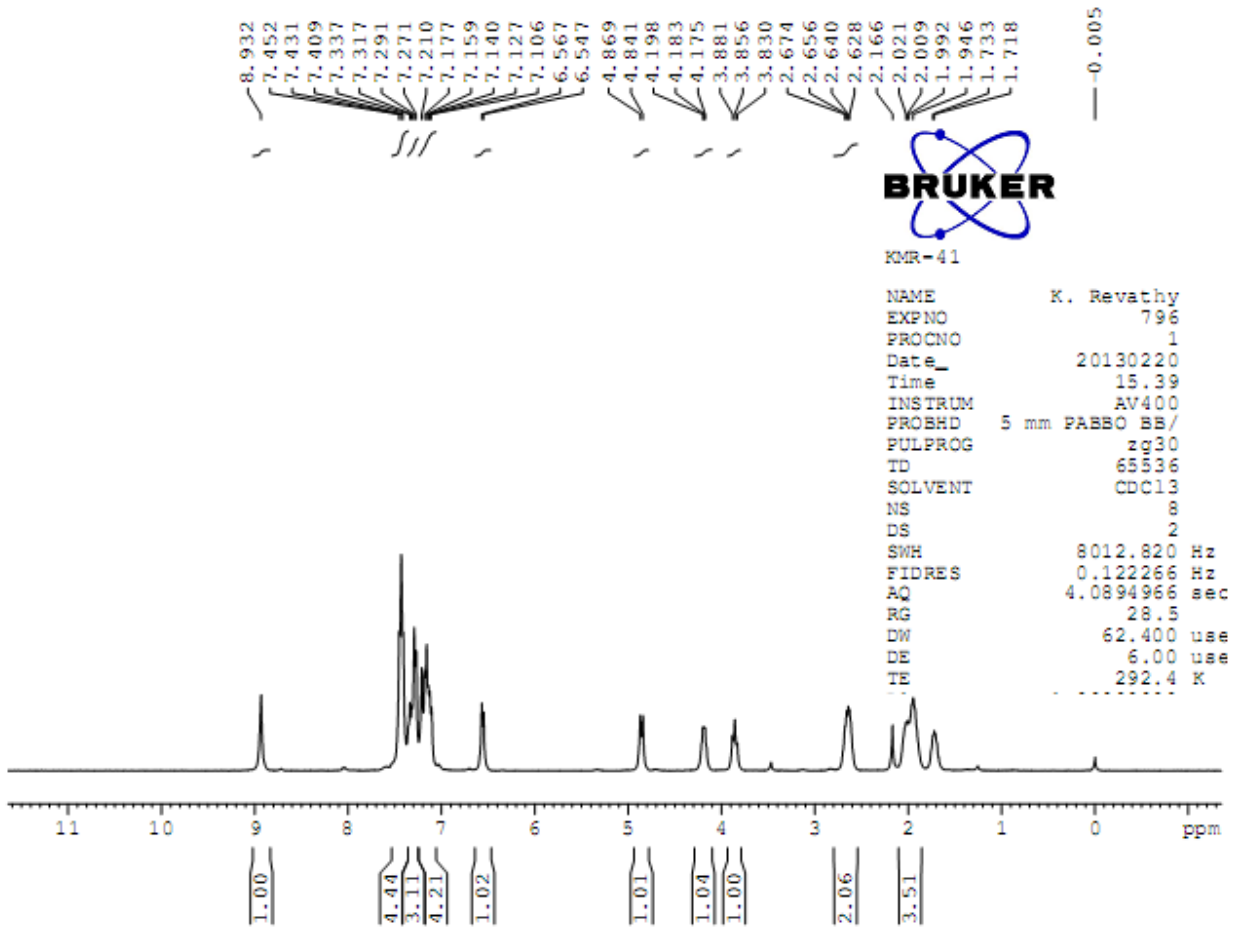
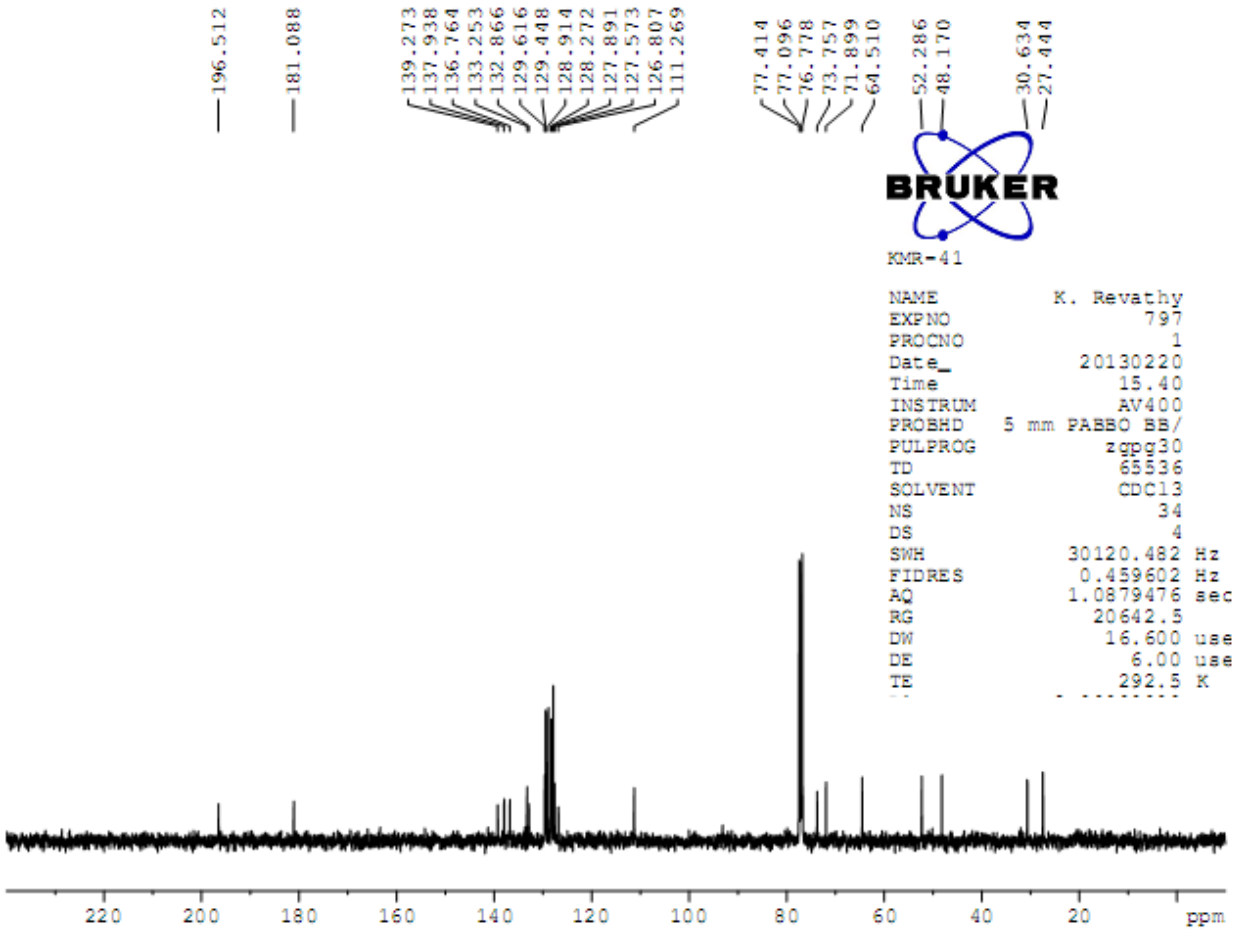


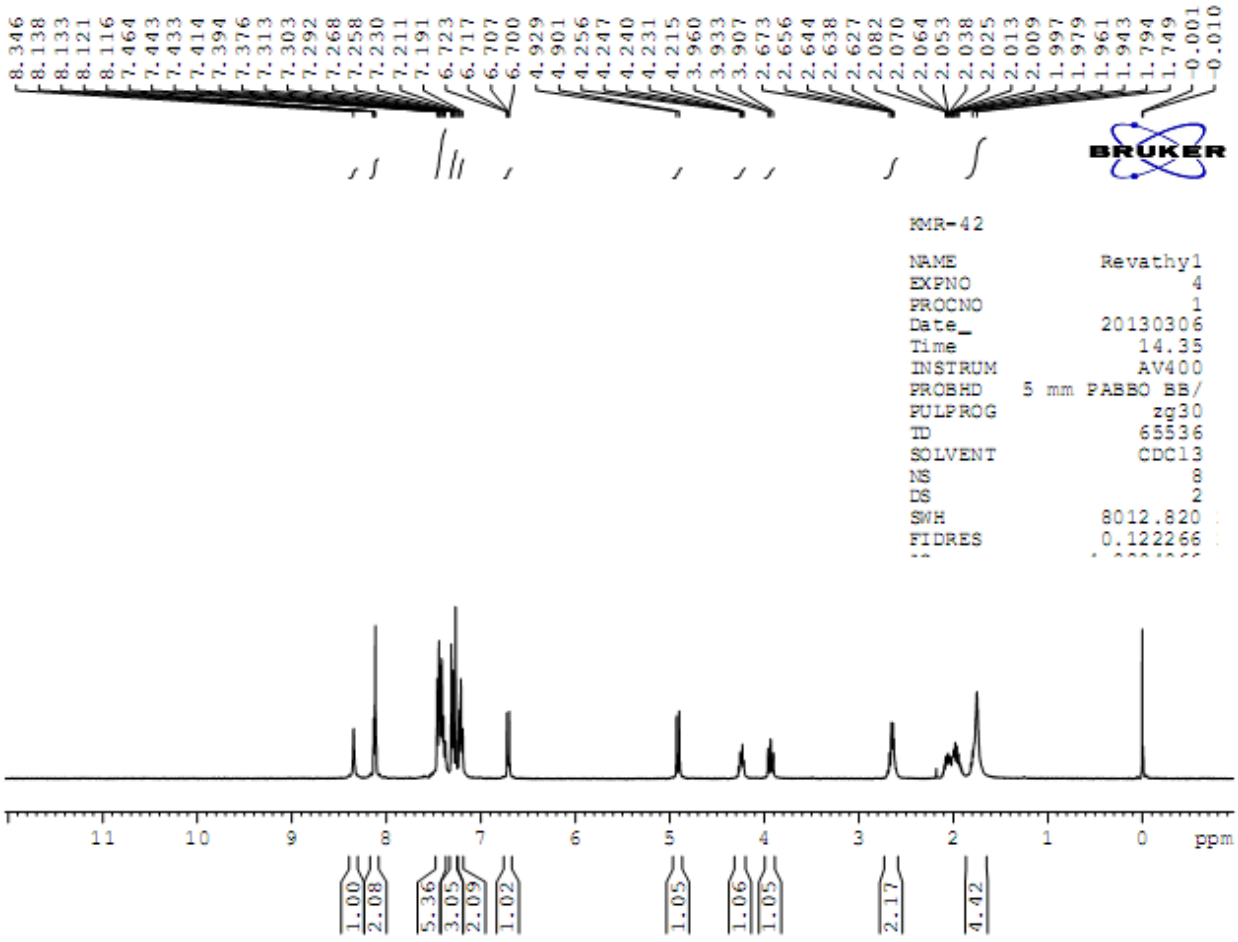
^{13}C NMR Spectrum of **6h**



^{1}H NMR Spectrum of **6i**



^{13}C NMR Spectrum of **6i**



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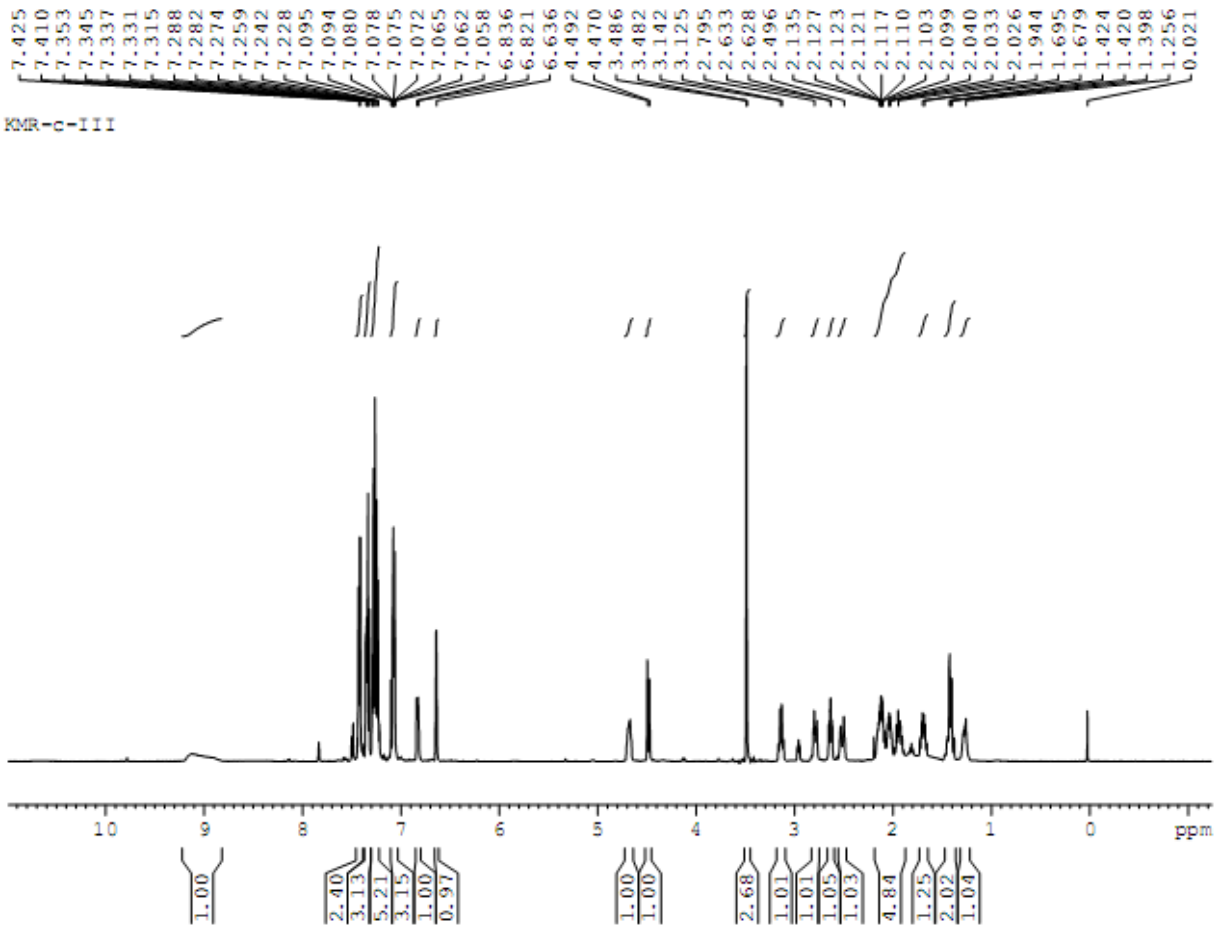

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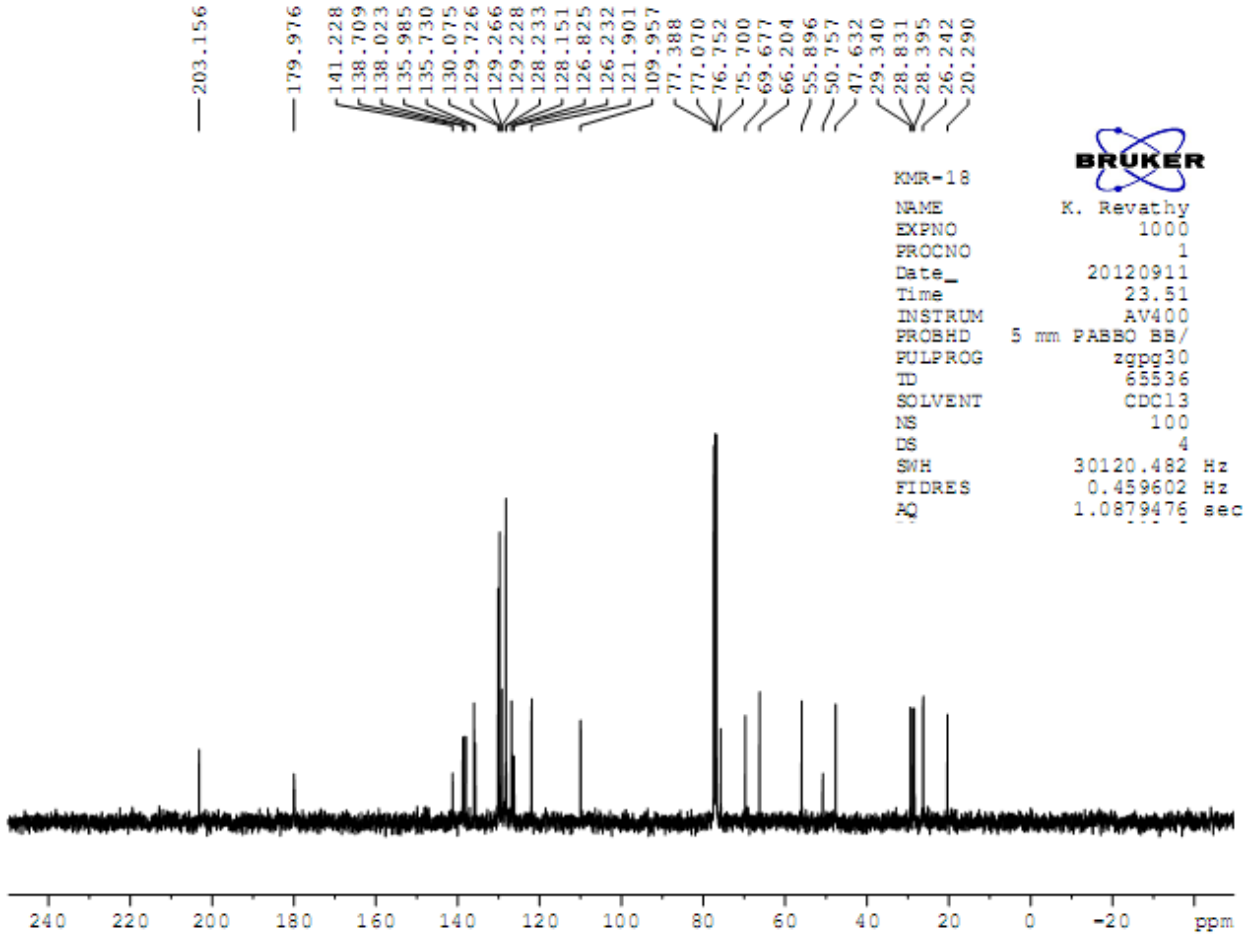

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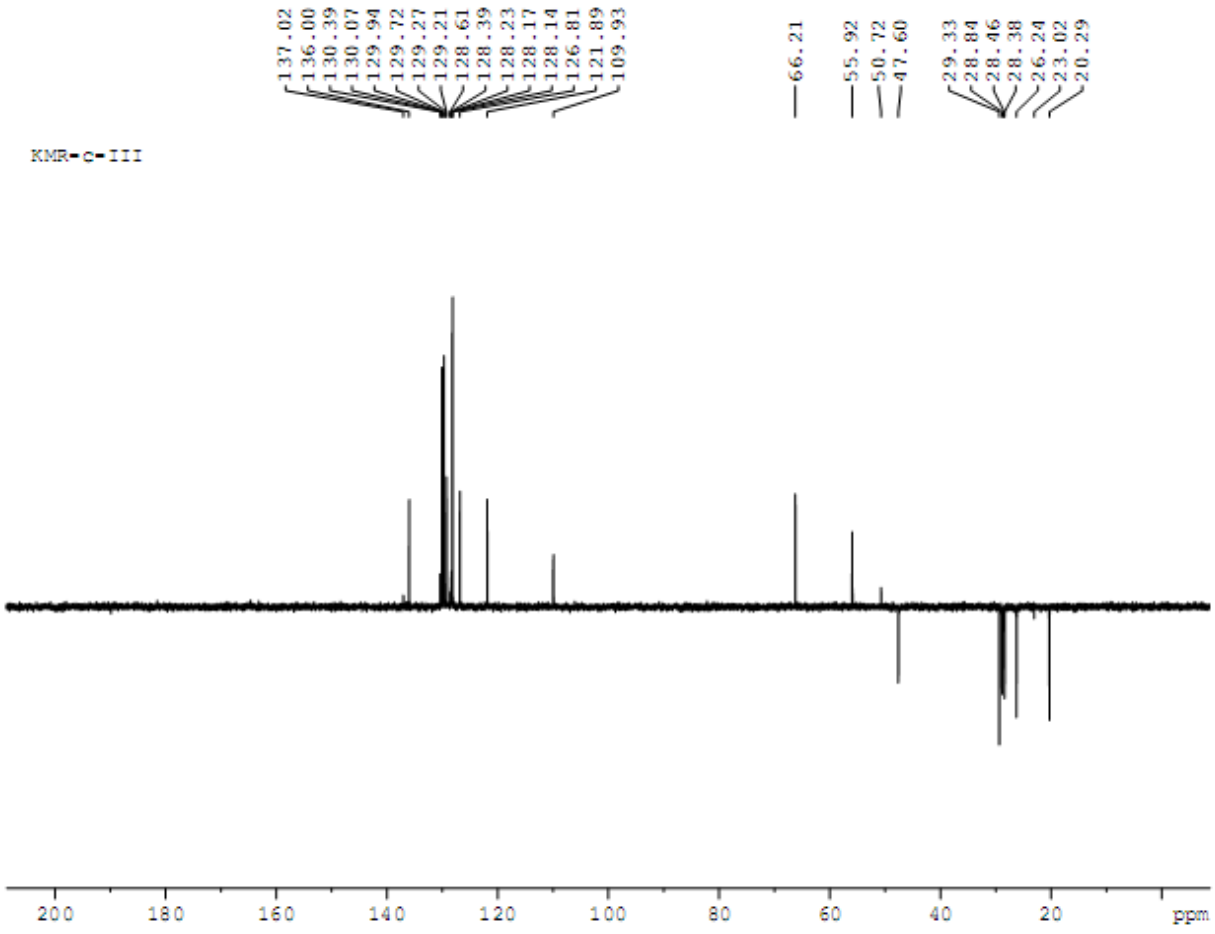
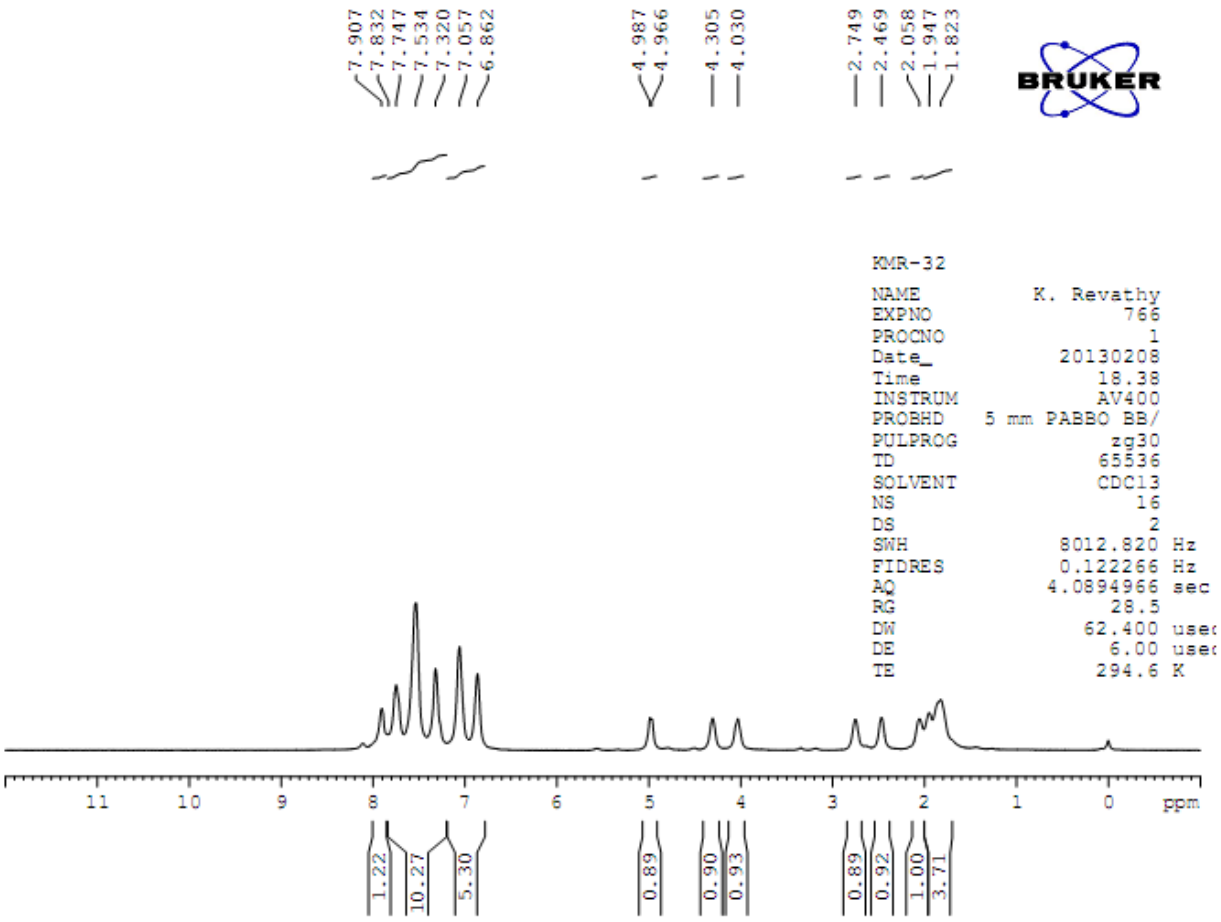
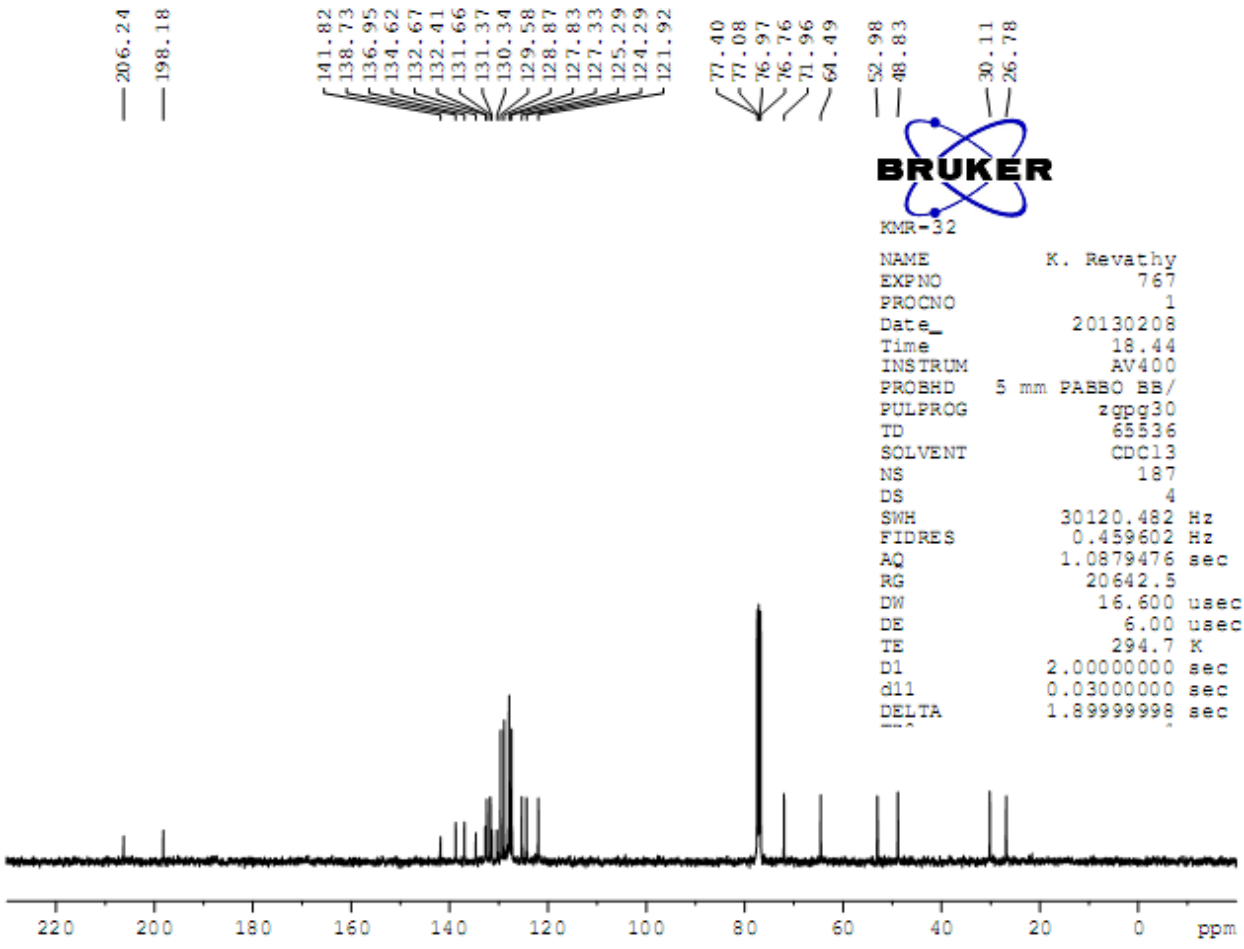


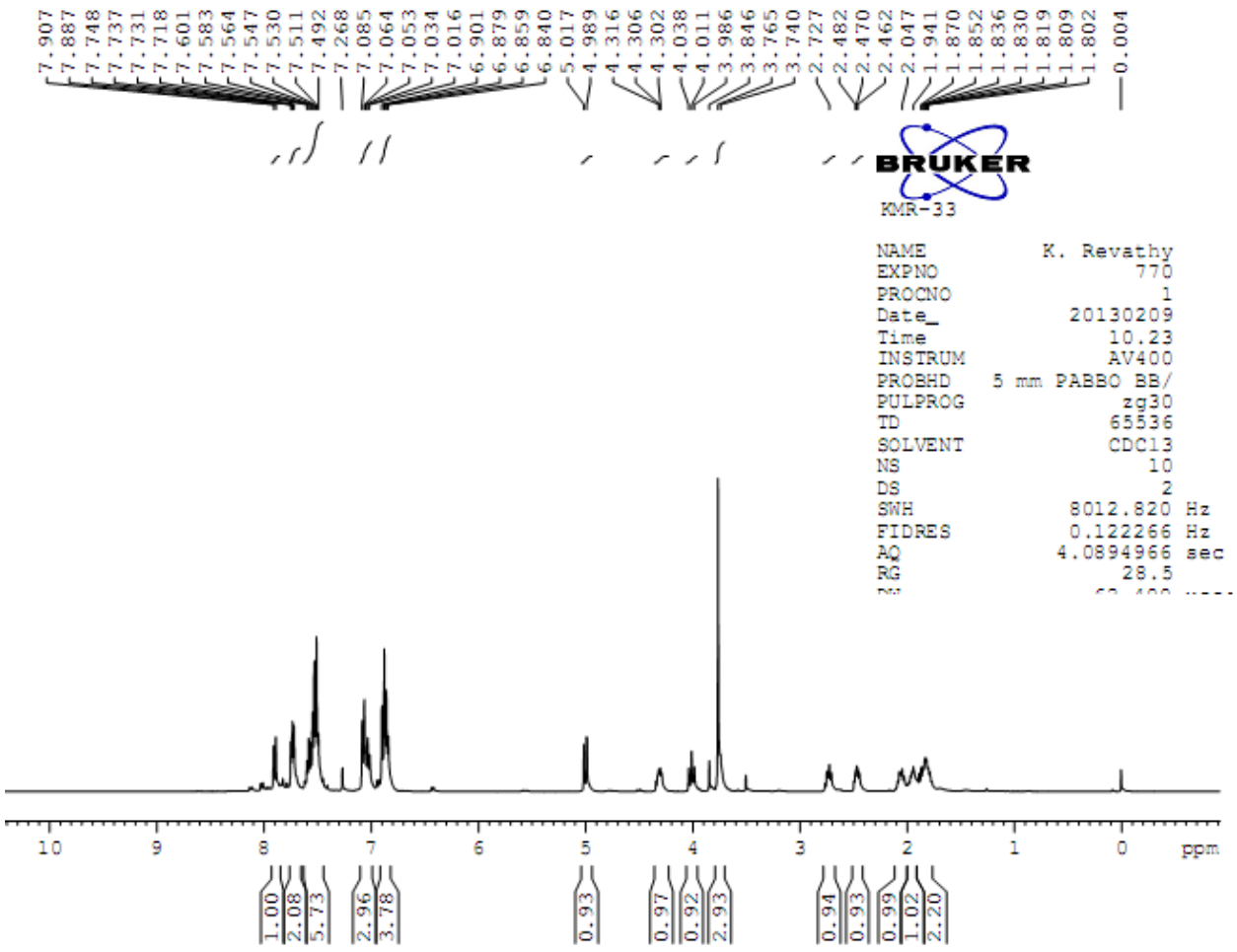
DEPT ^{13}C NMR Spectrum of **8a**



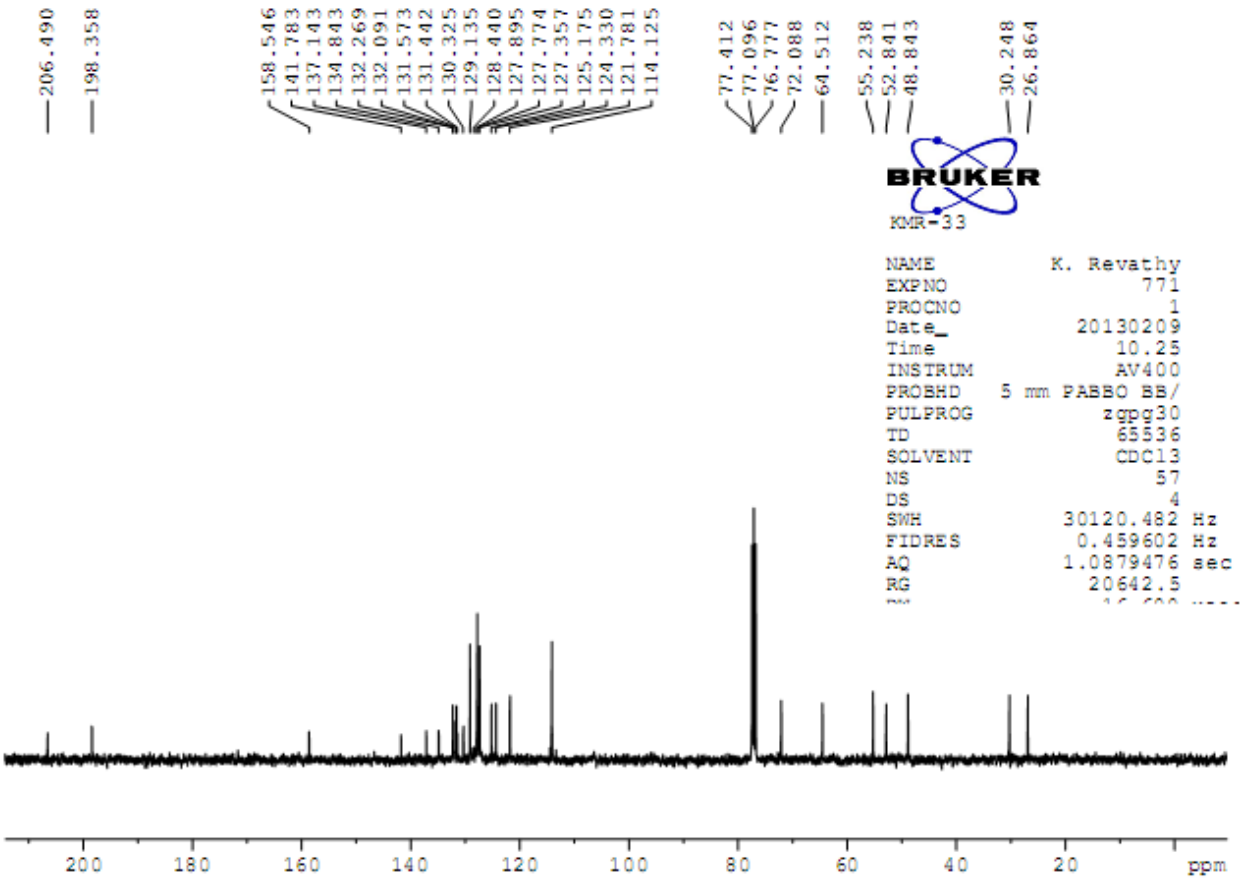
^{1}H NMR Spectrum of **10a**



^{13}C NMR Spectrum of **10a**



^{1}H NMR Spectrum of **10b**



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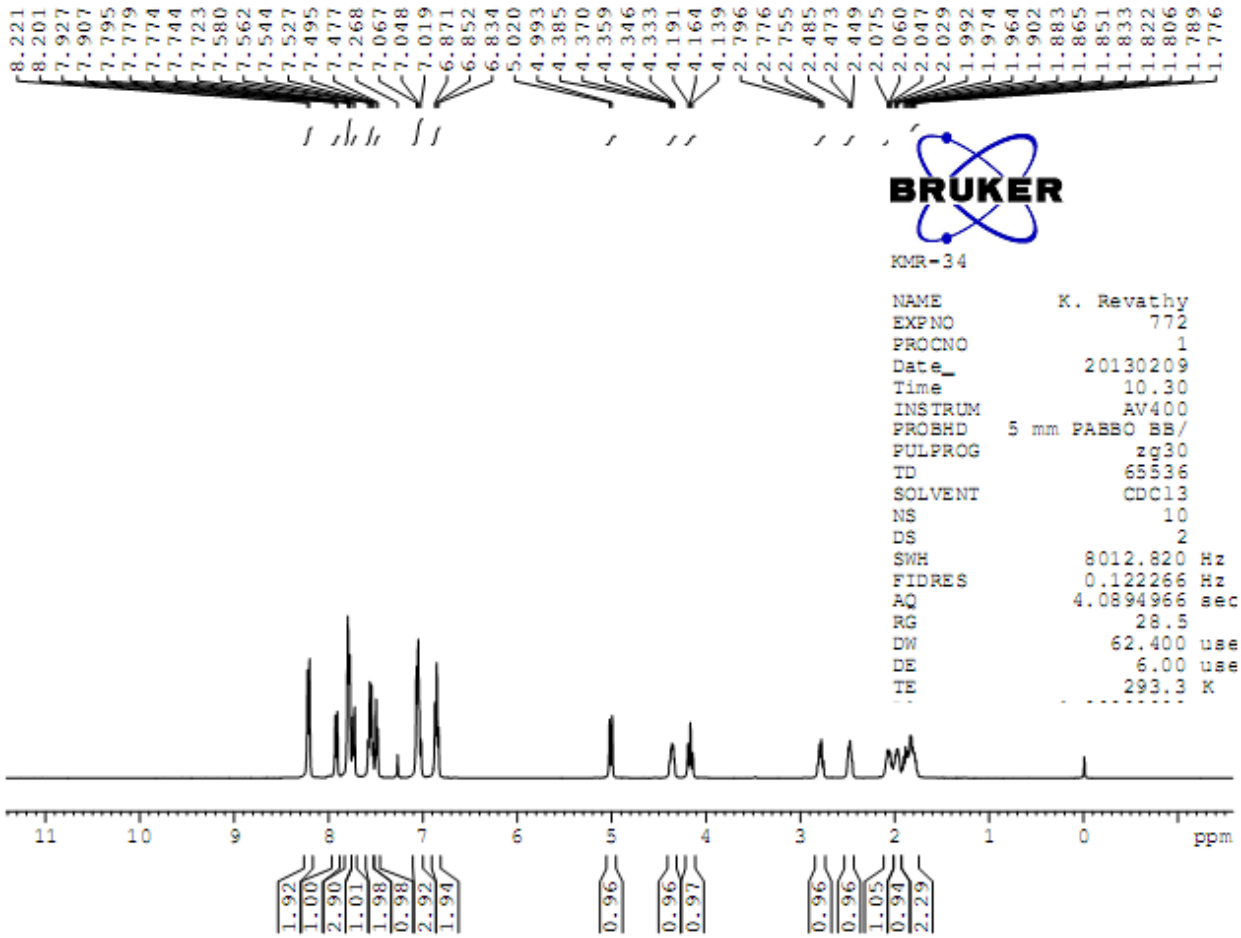

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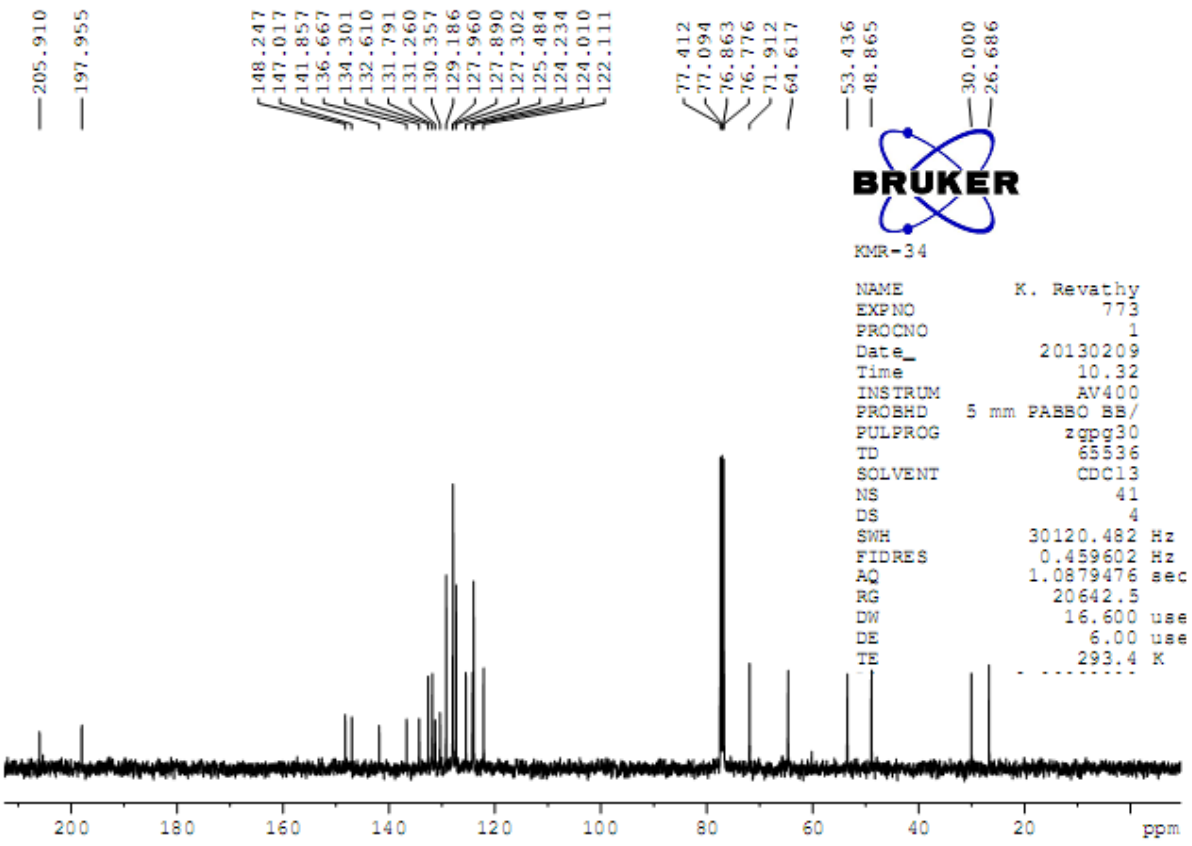

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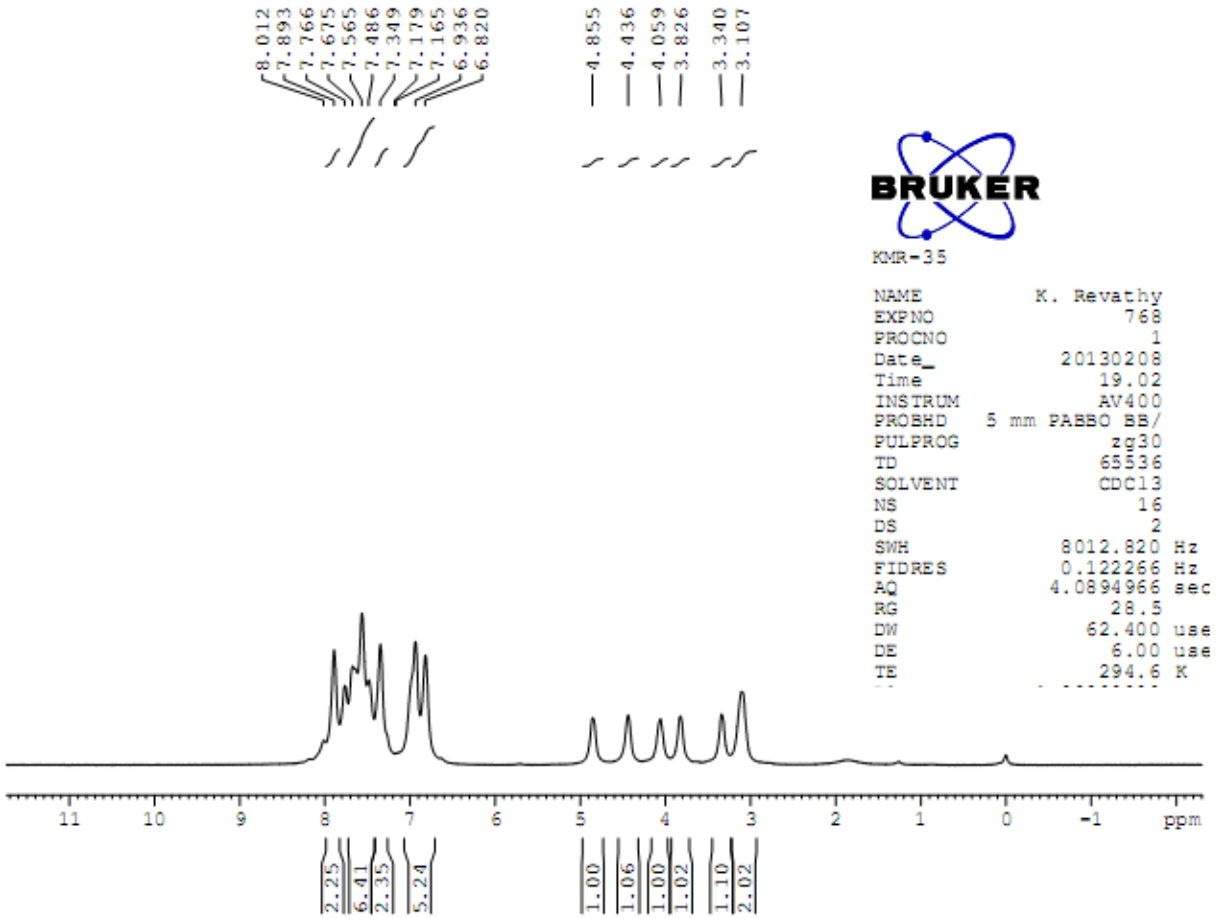

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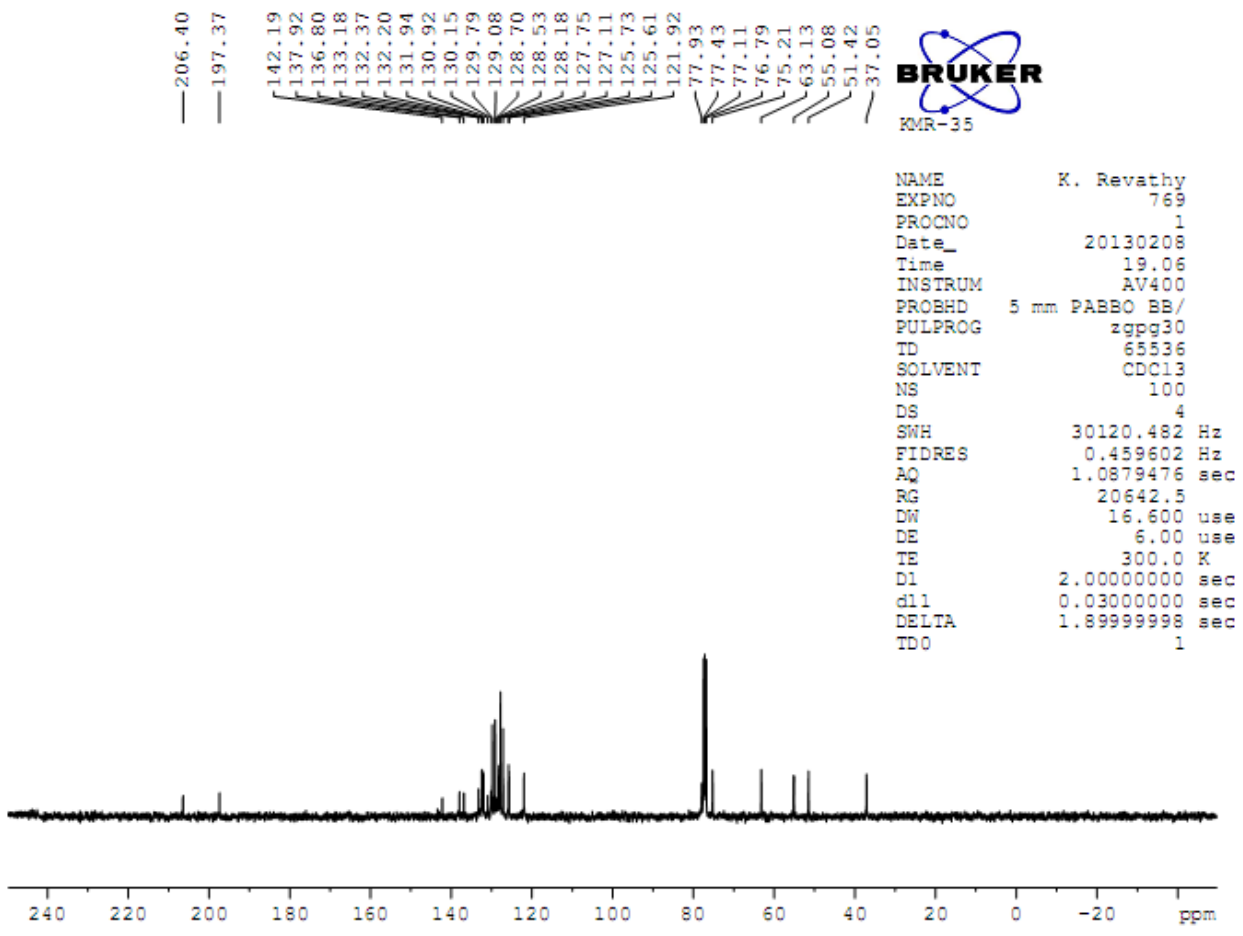

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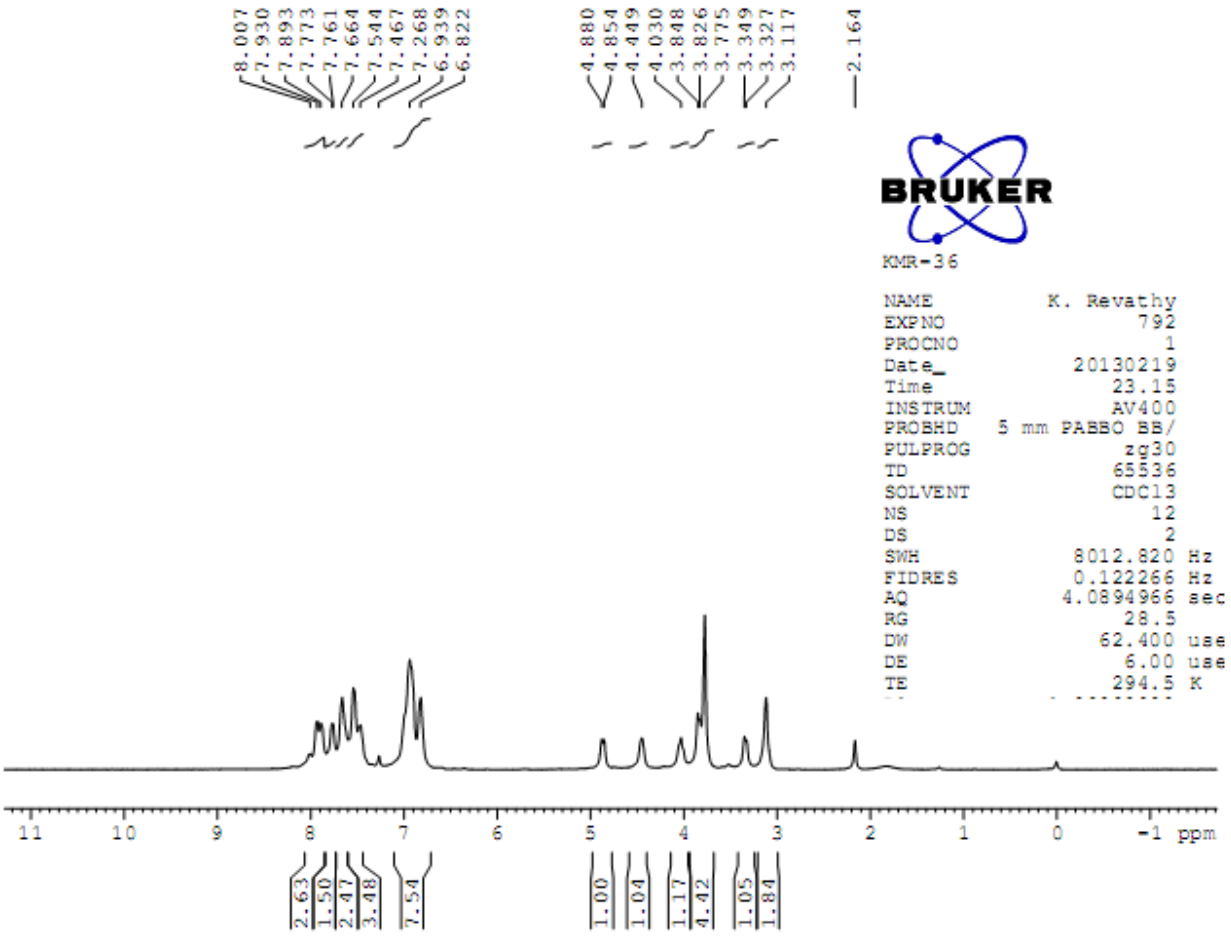

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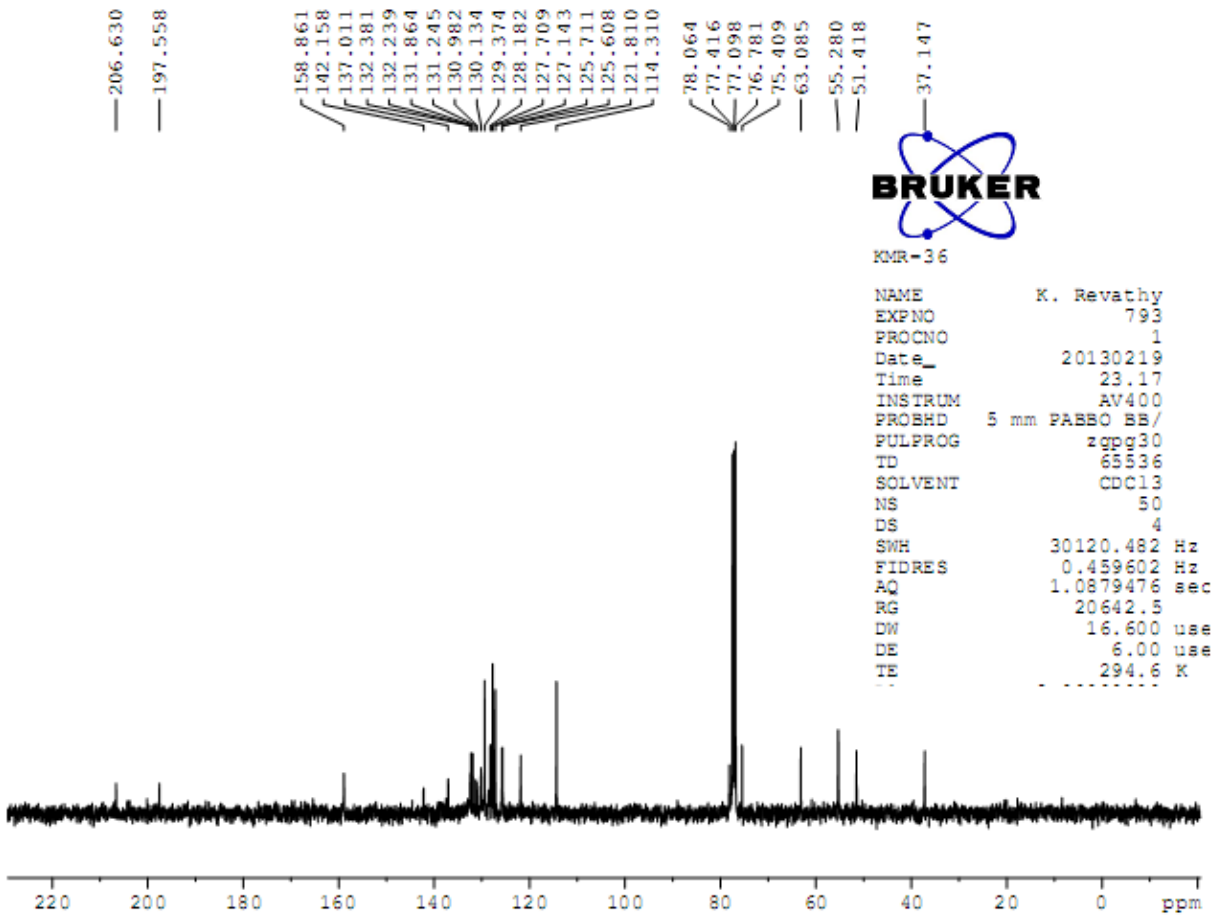

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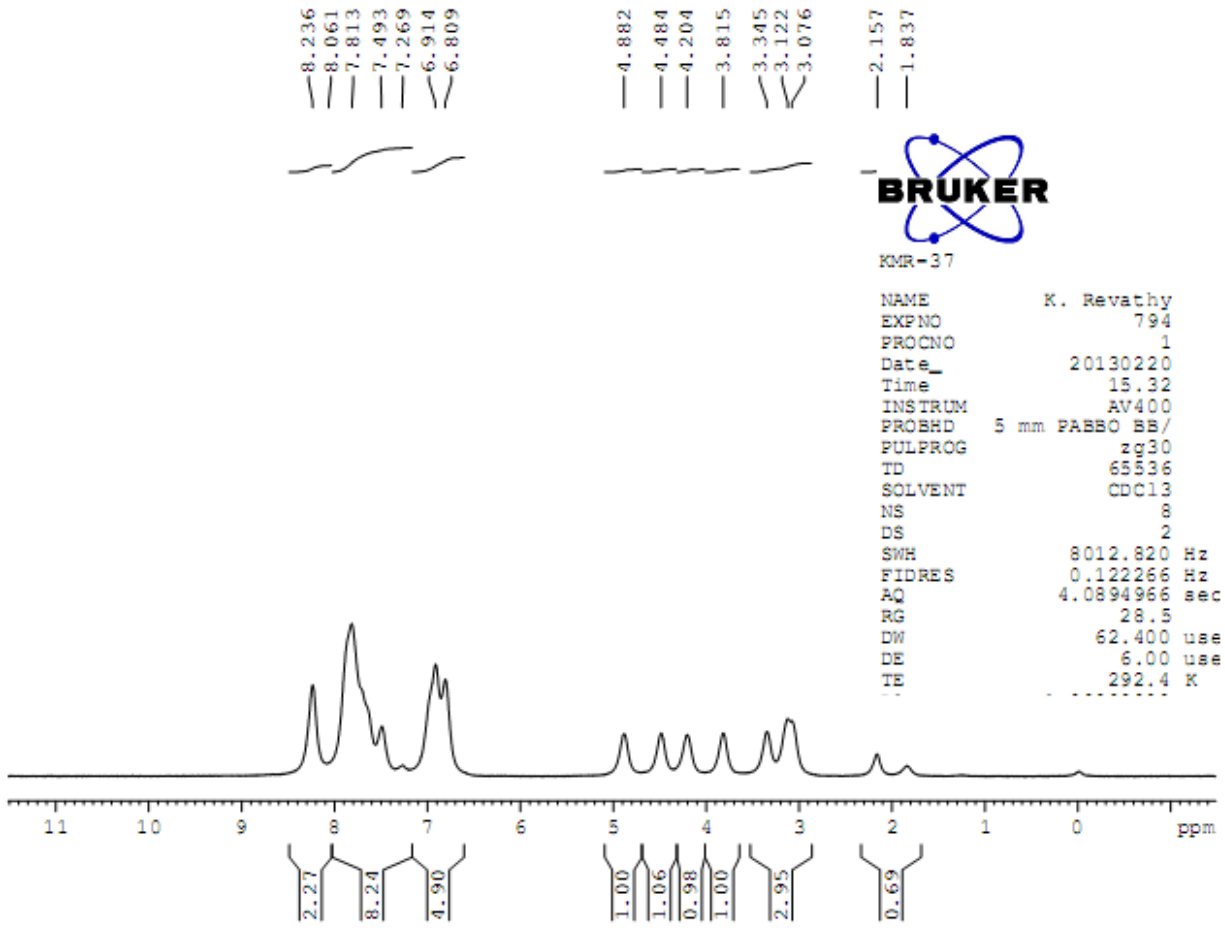

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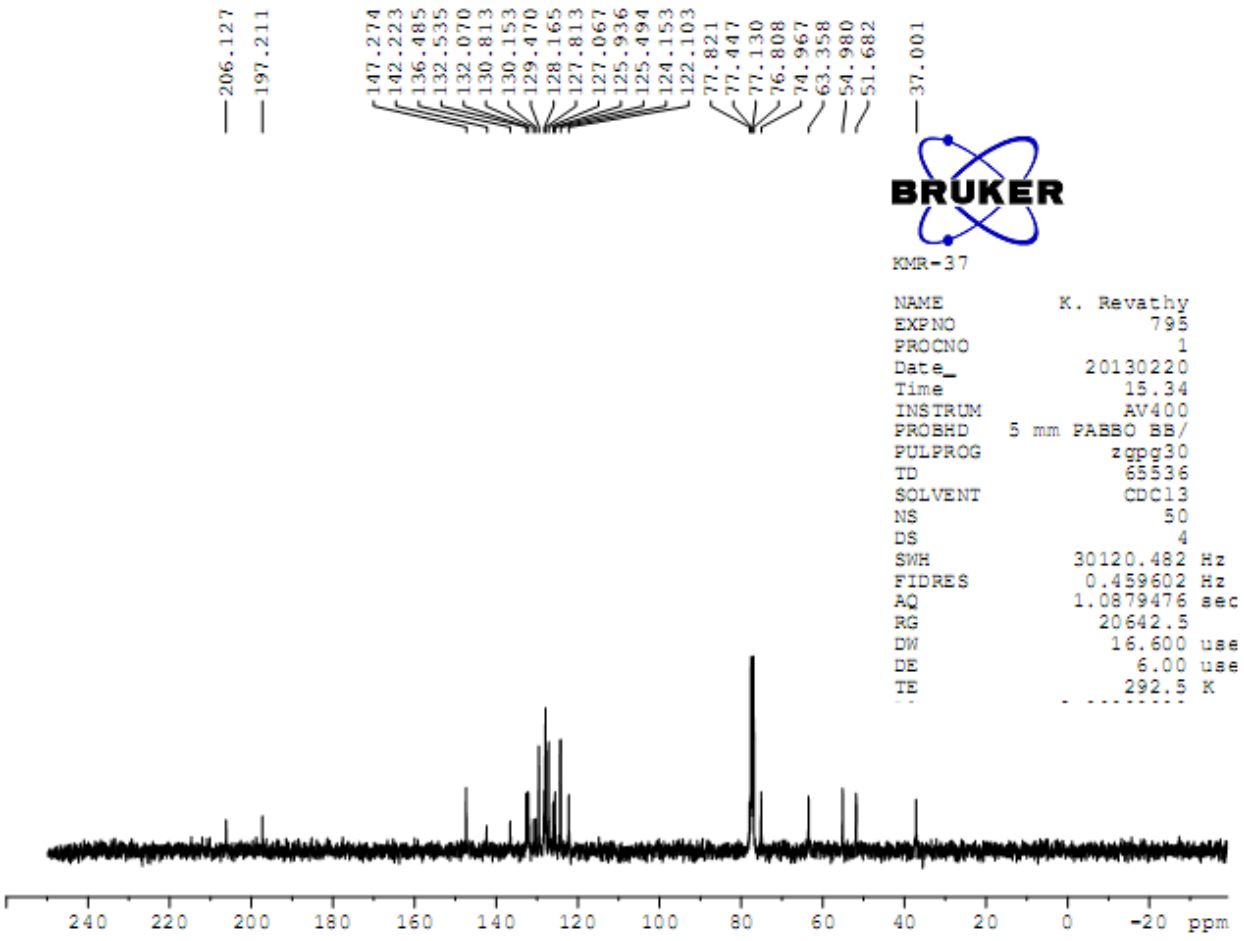
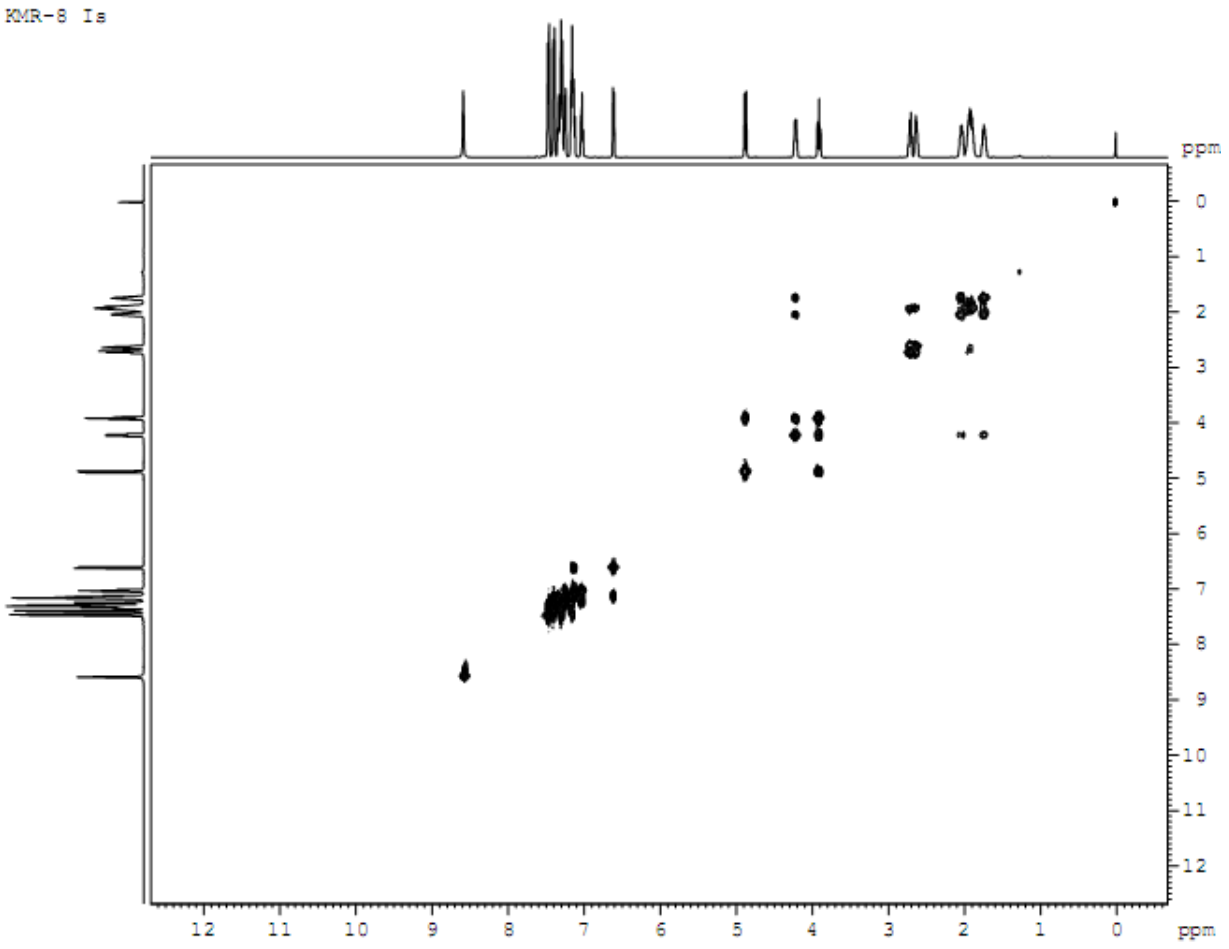
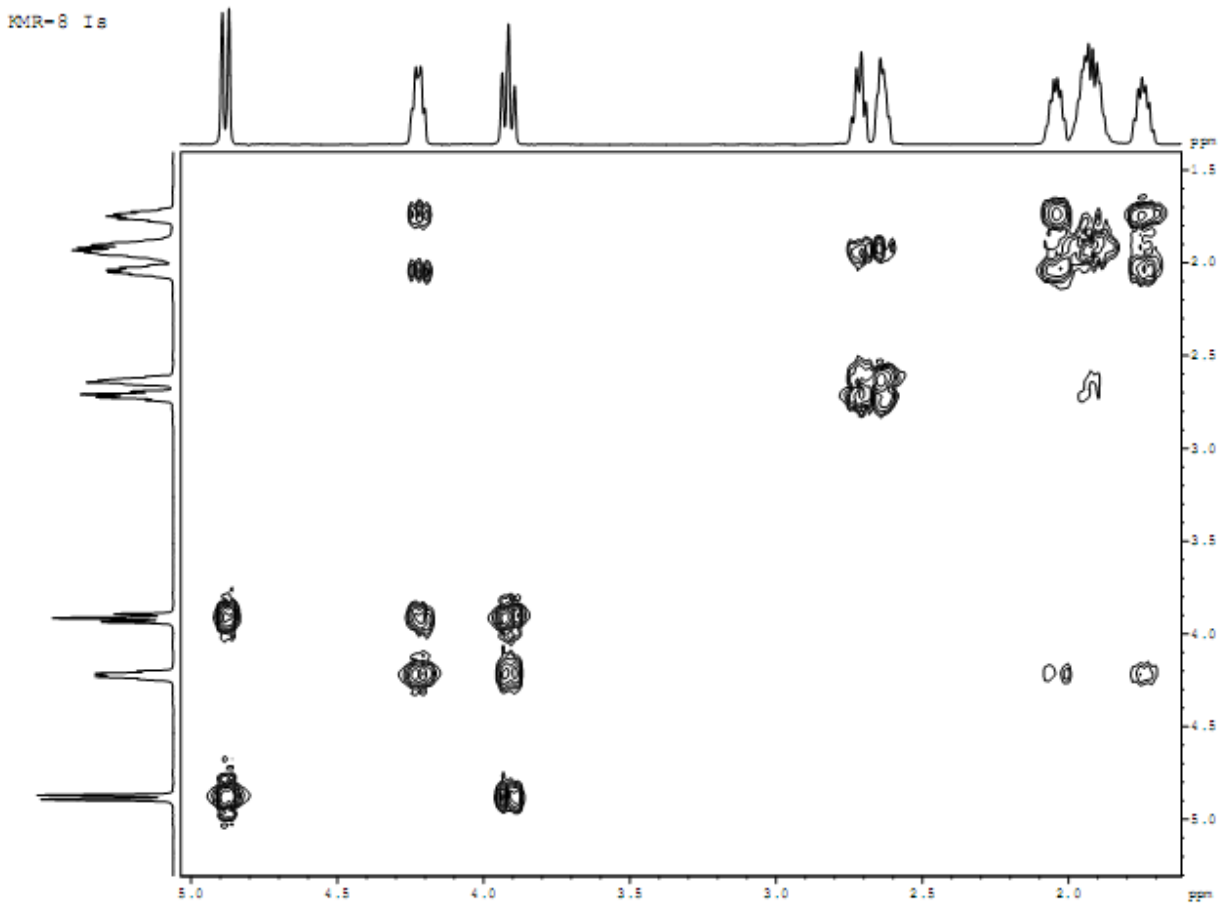


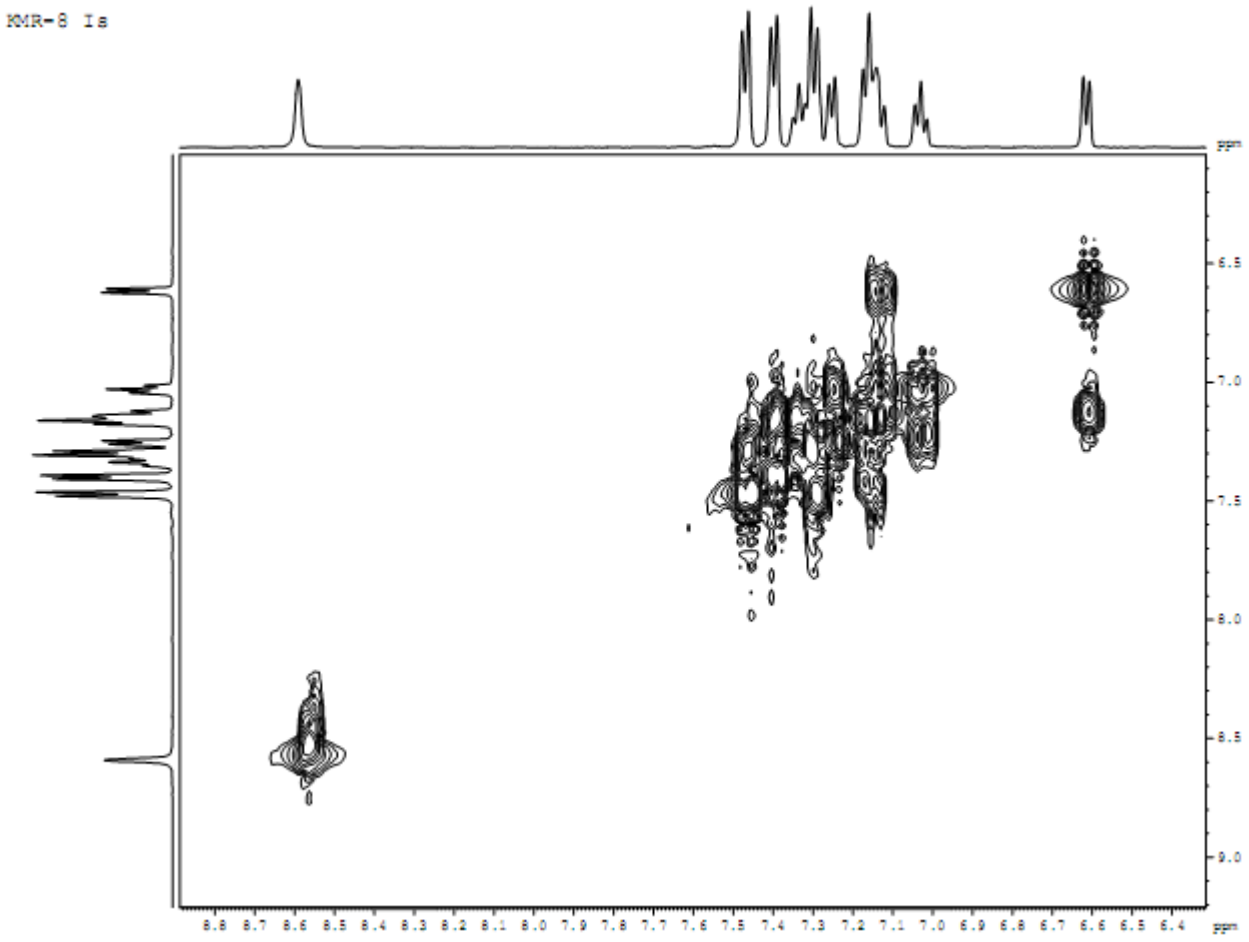
^{13}C NMR Spectrum of **10f**



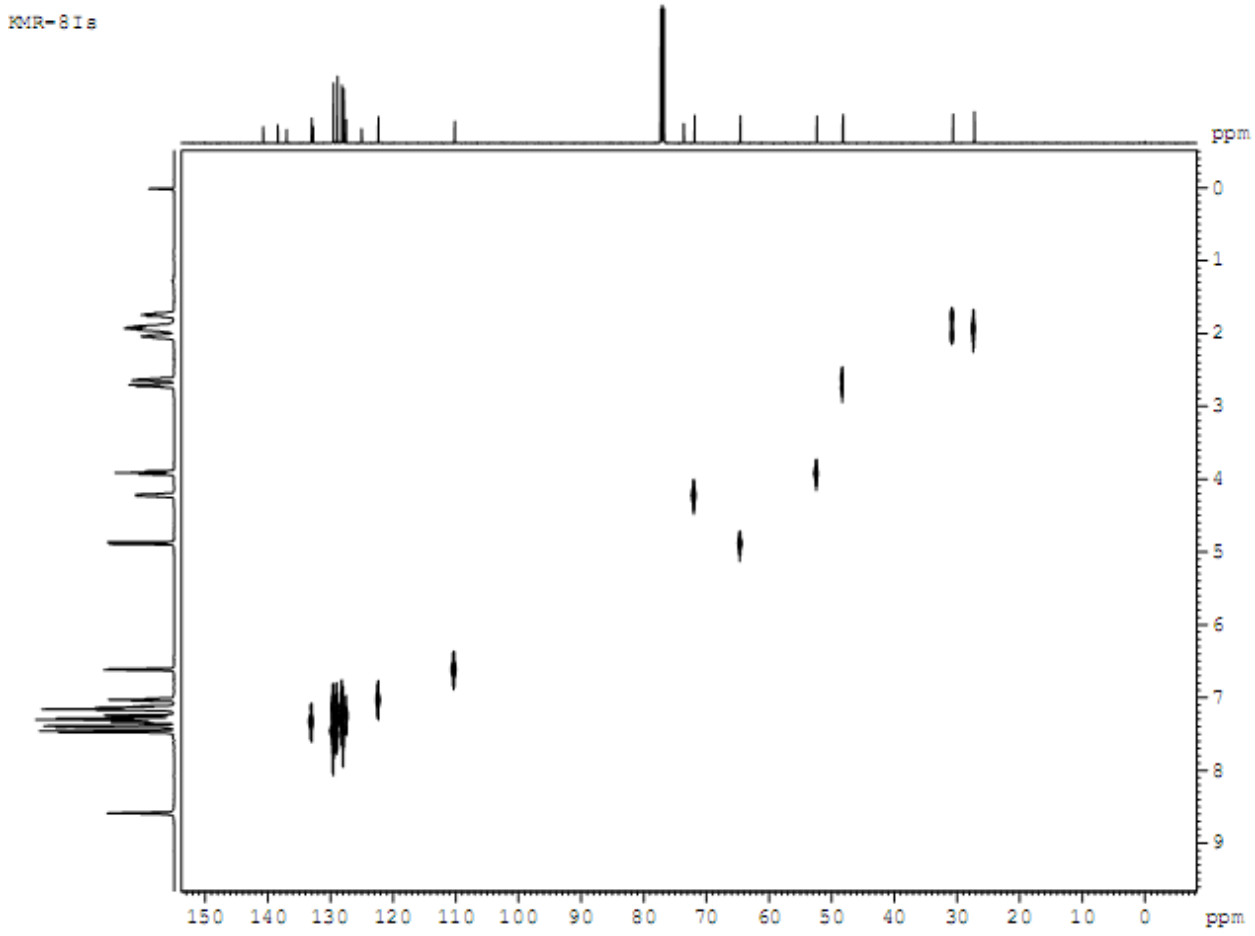
^{1}H-^{1}HCOSY spectrum of compound 6b



Expanded ^{1}H-^{1}HCOSY spectrum of compound 6b



Expanded ^{1}H-^{1}HCOSY spectrum of compound 6b



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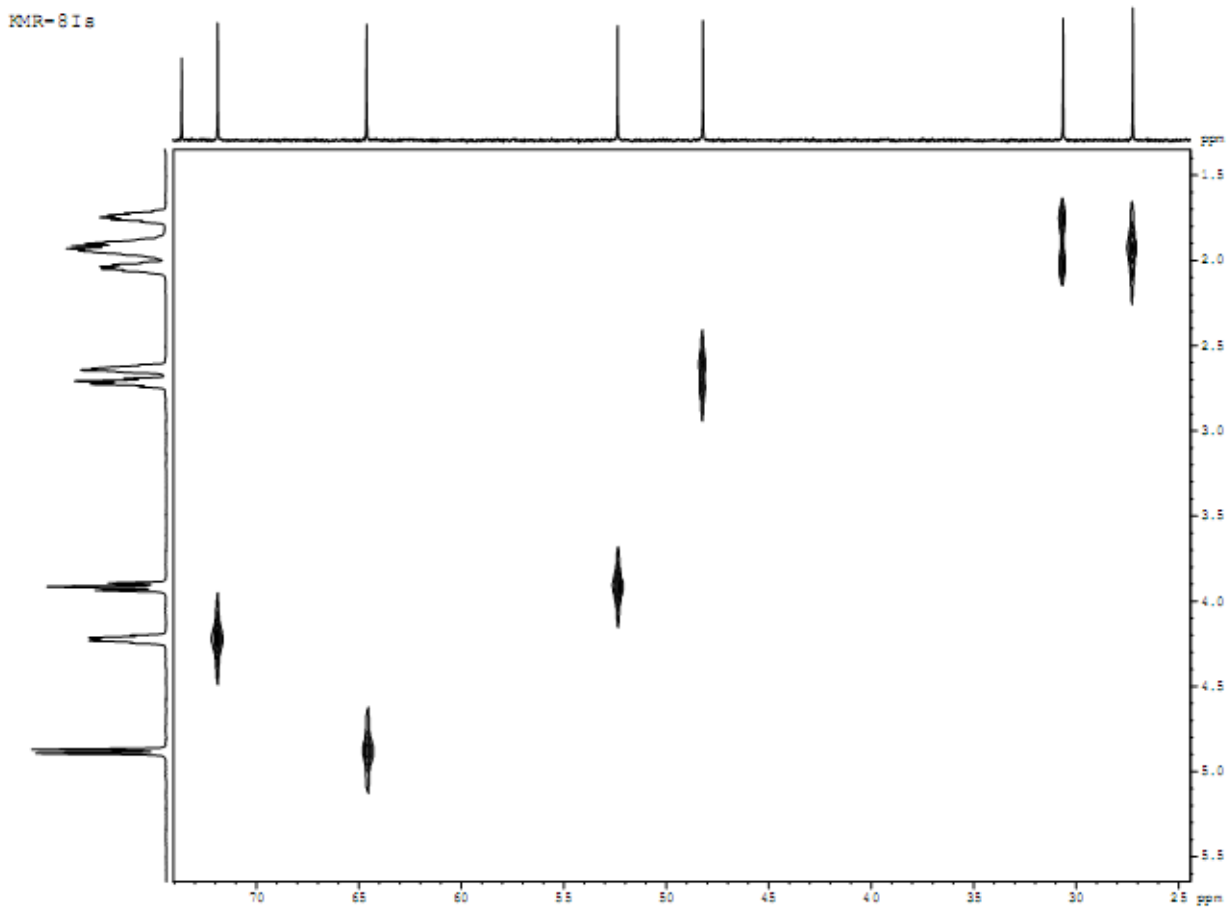
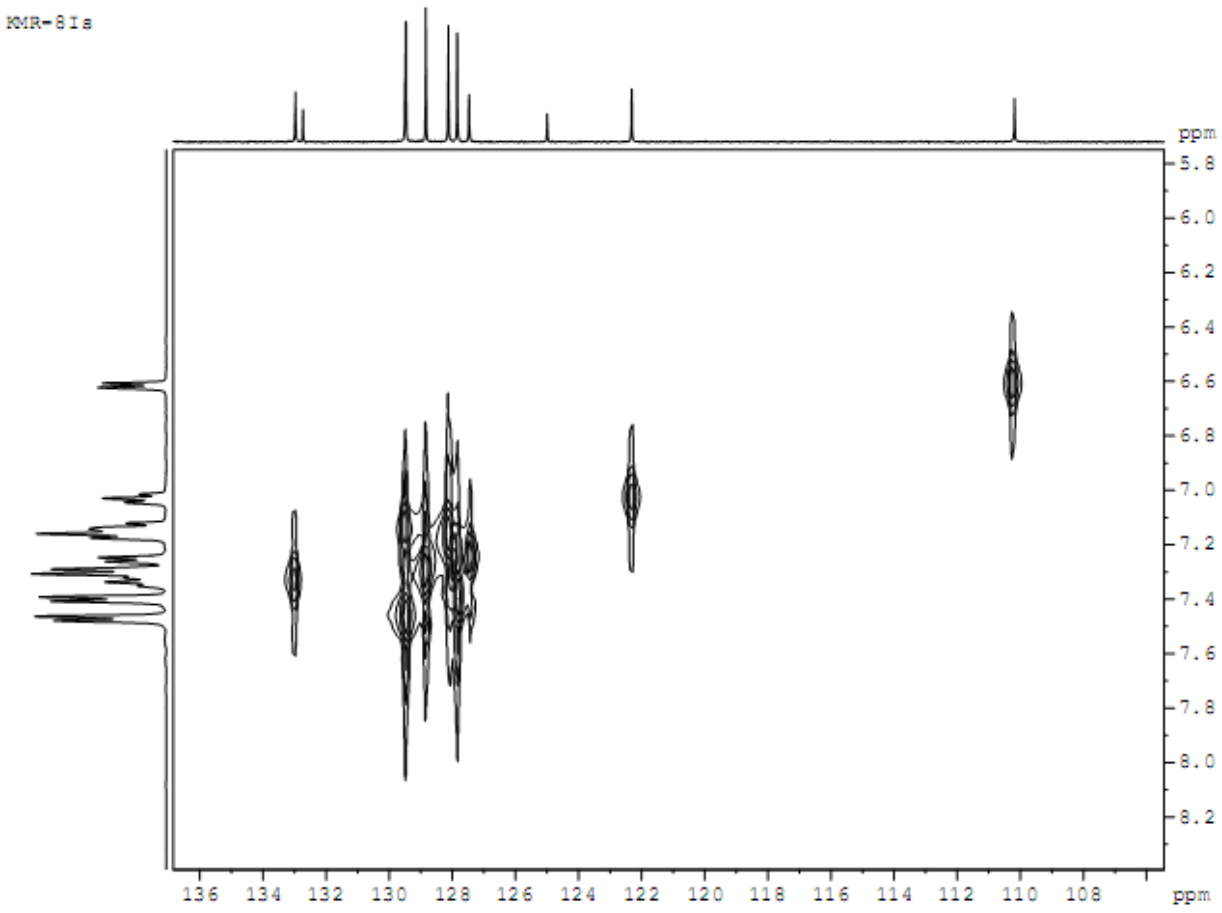
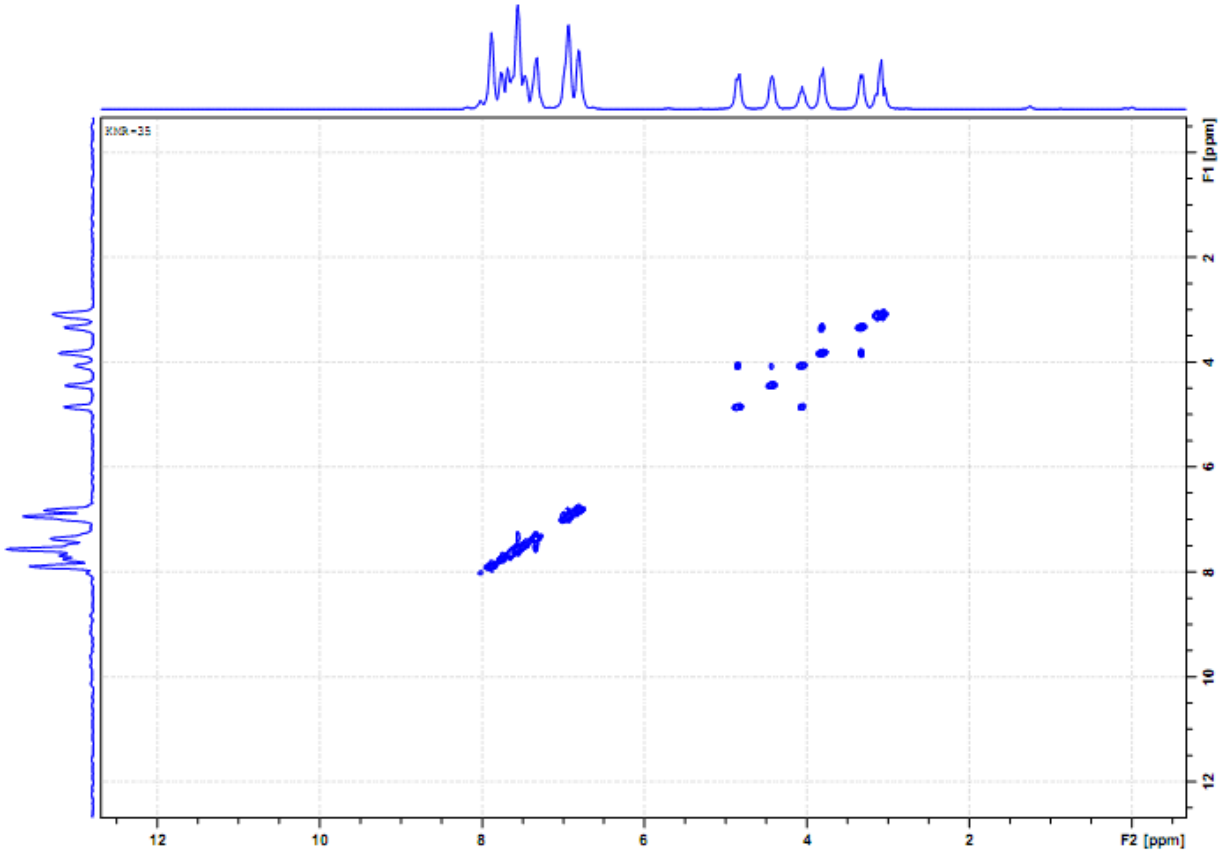


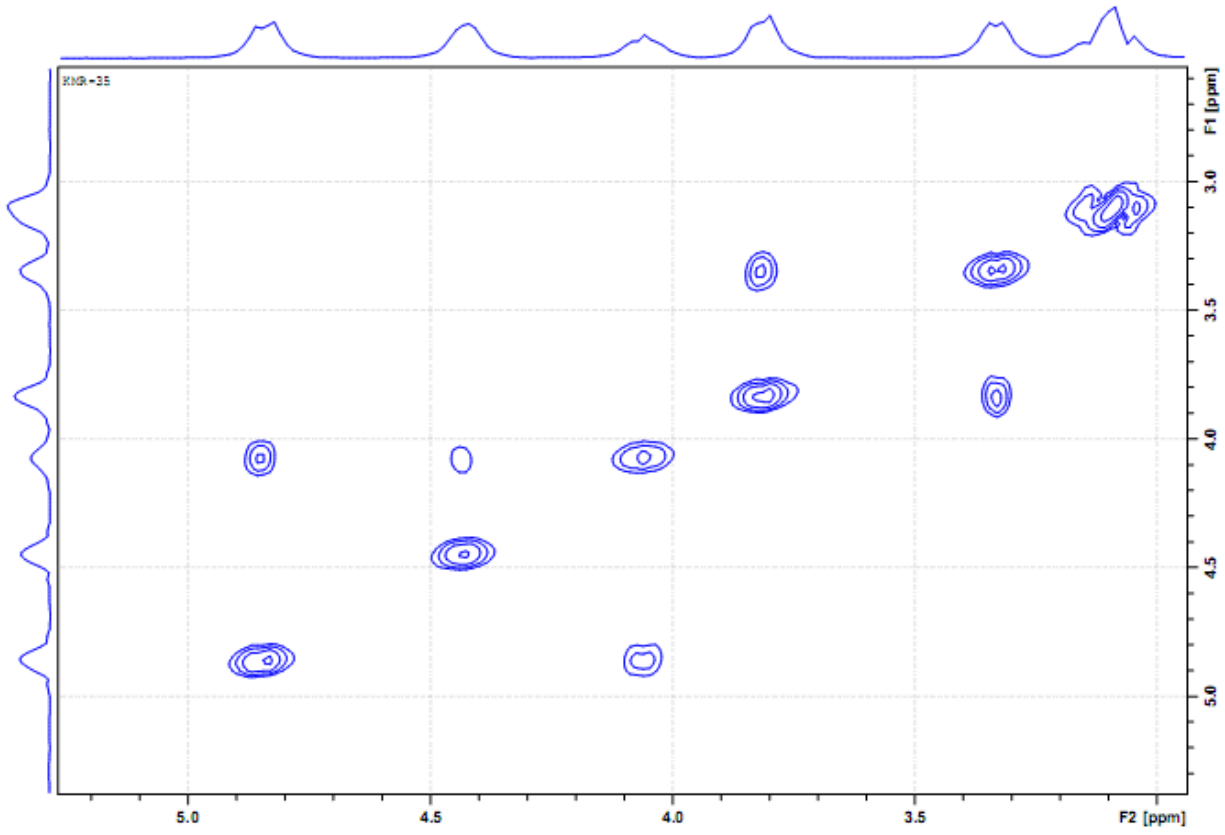
Expanded ^{1}H-^{13}C COSY spectrum of compound 6b



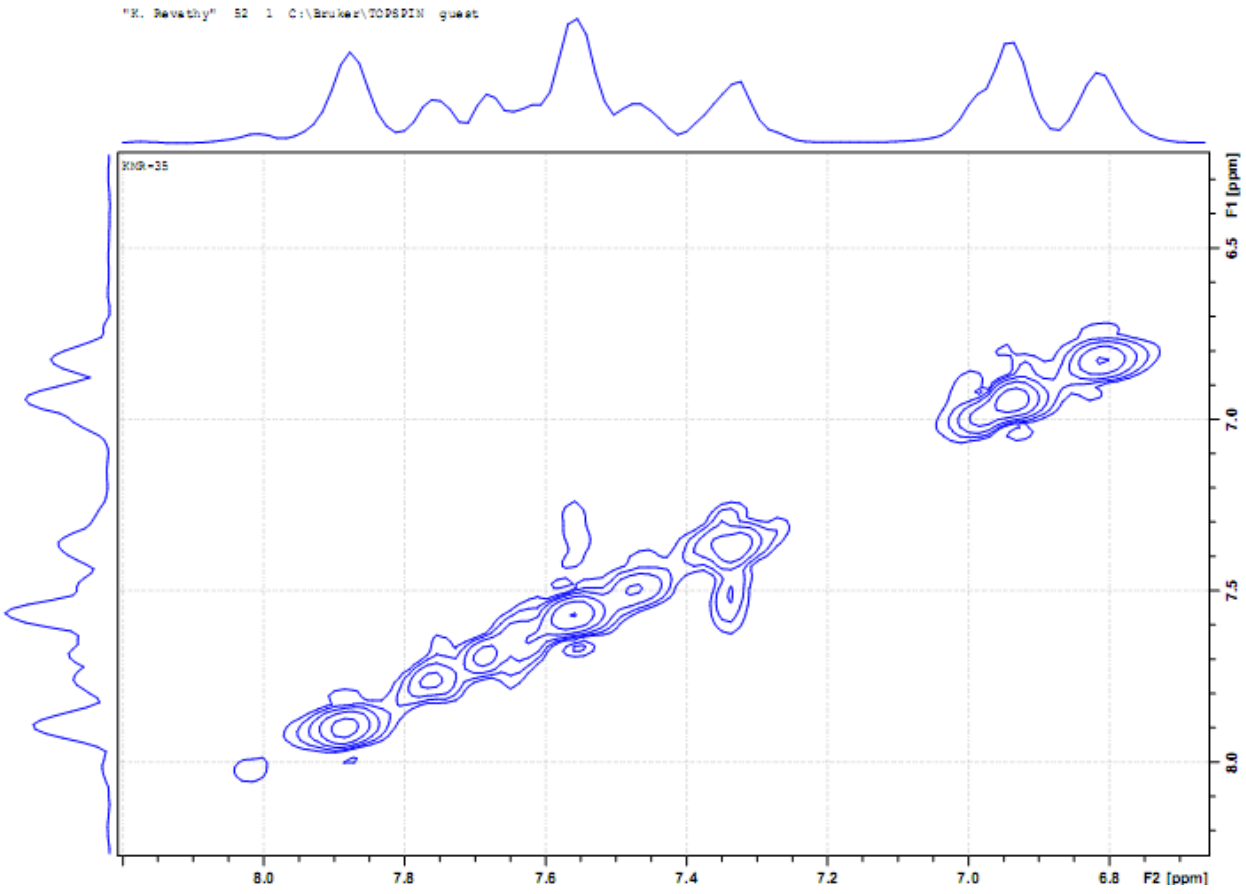
Expanded ^{1}H-^{13}C COSY spectrum of compound 6b



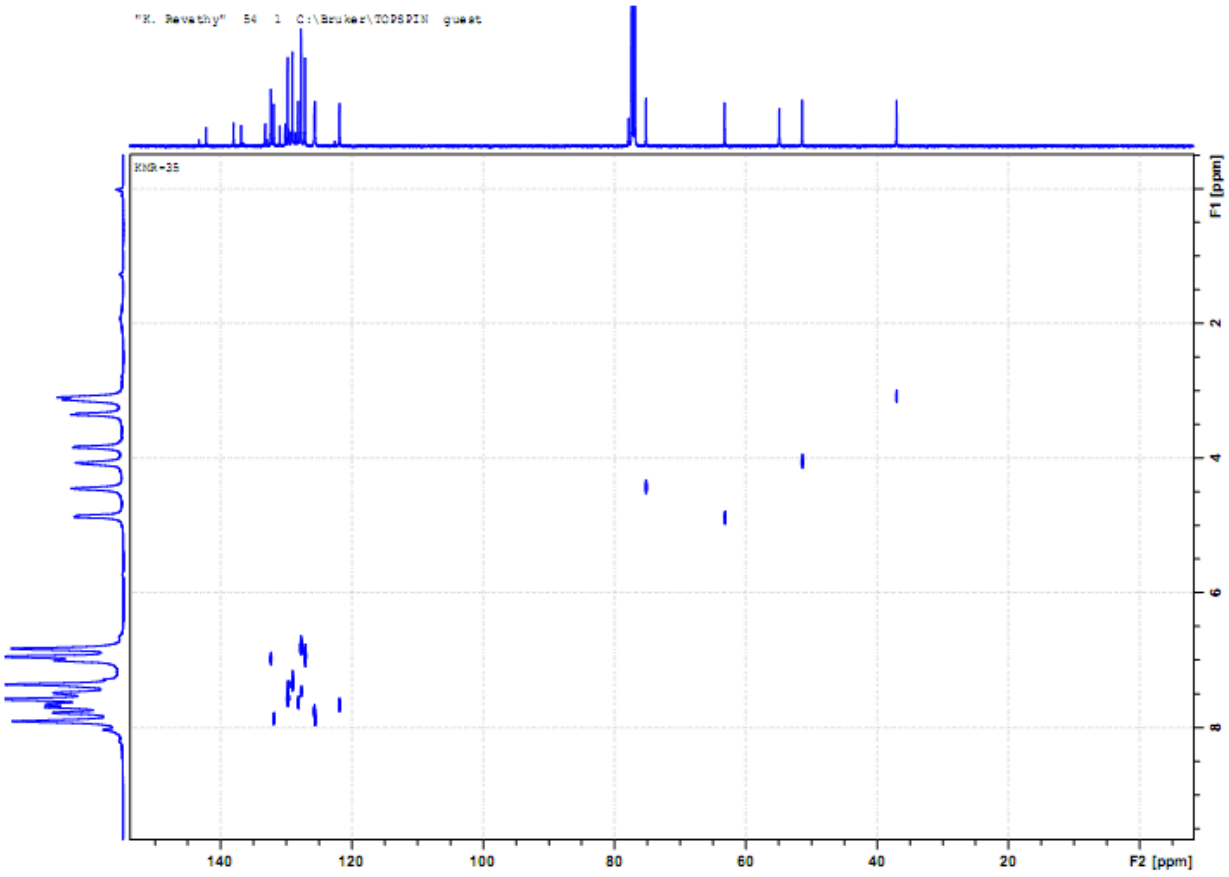
^{1}H-^{1}H COSY spectrum of compound 10d



Expanded ^{1}H-^{1}H COSY spectrum of compound 10d



Expanded ^{1}H-^{1}H COSY spectrum of compound 10d



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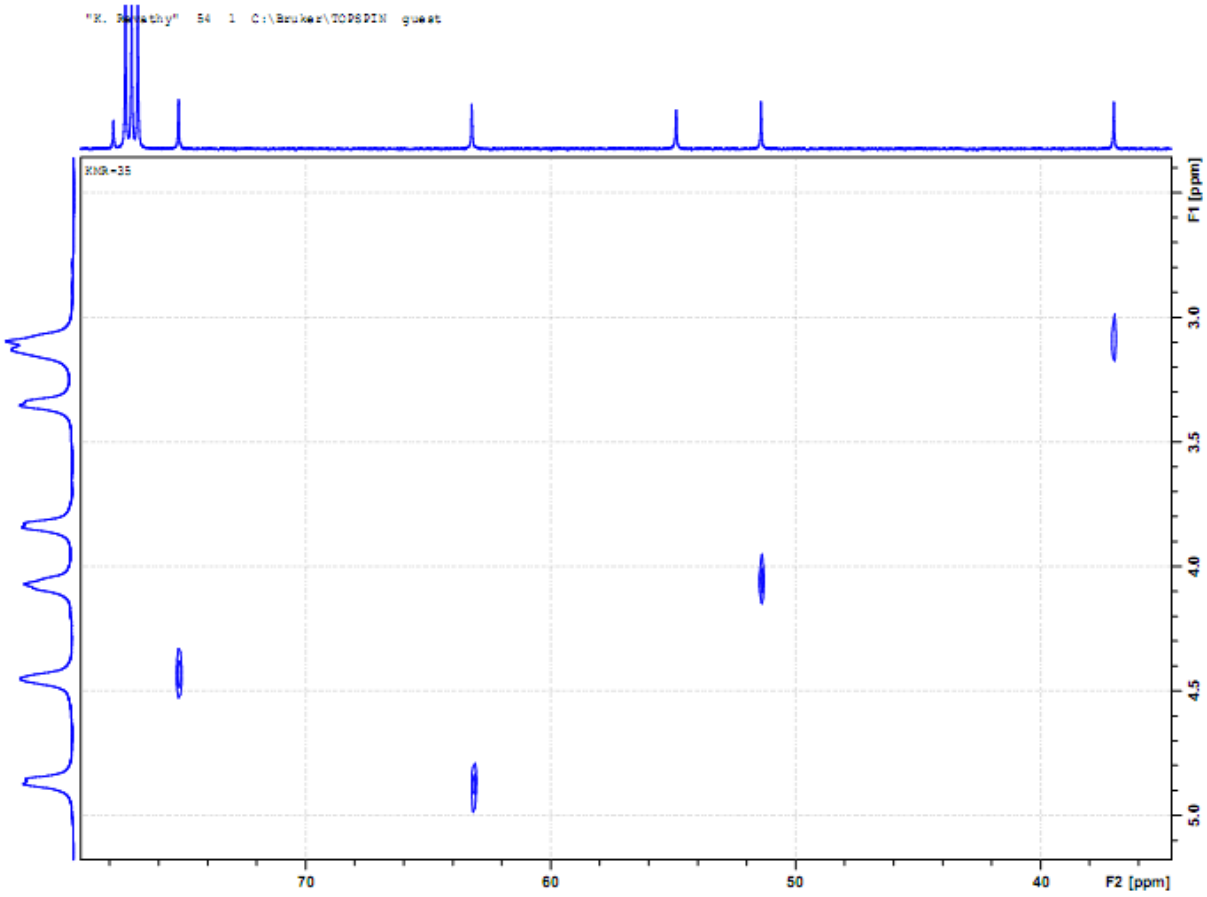
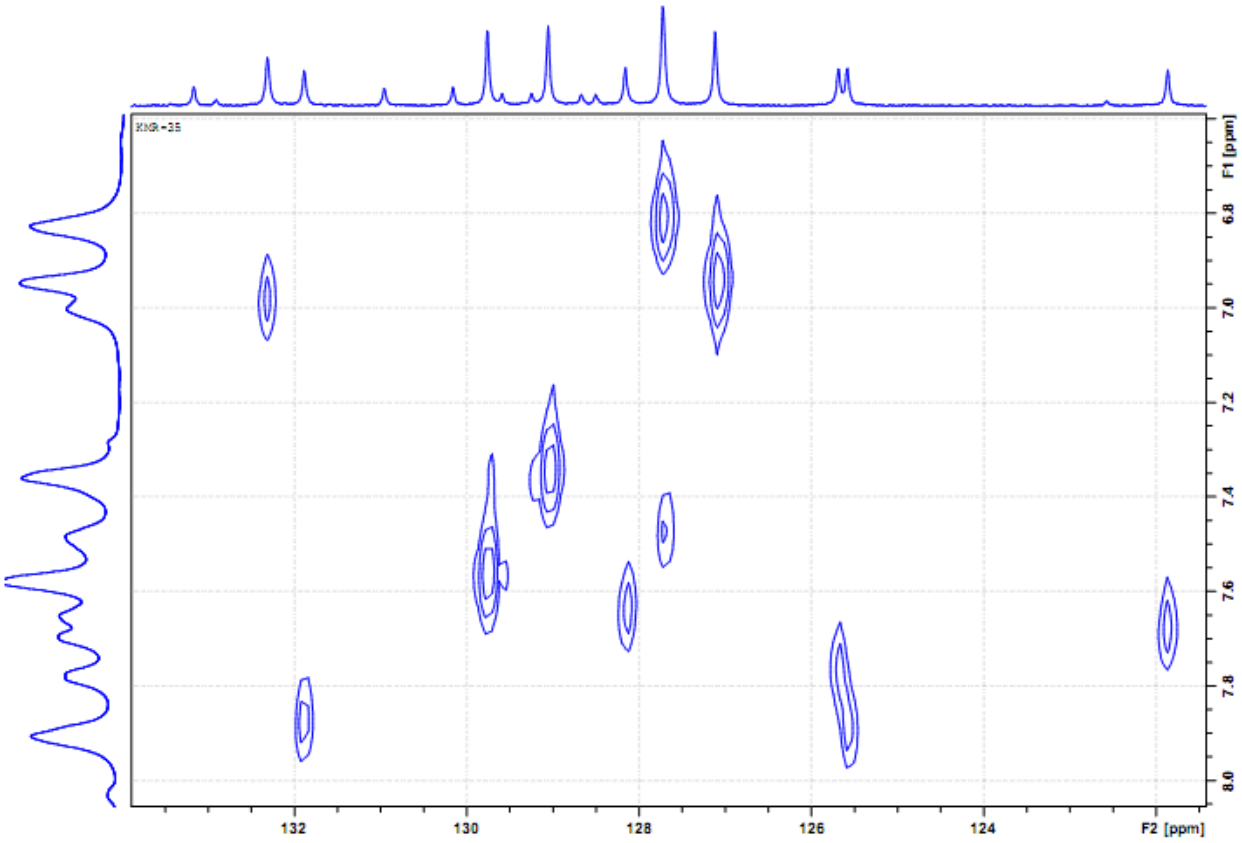
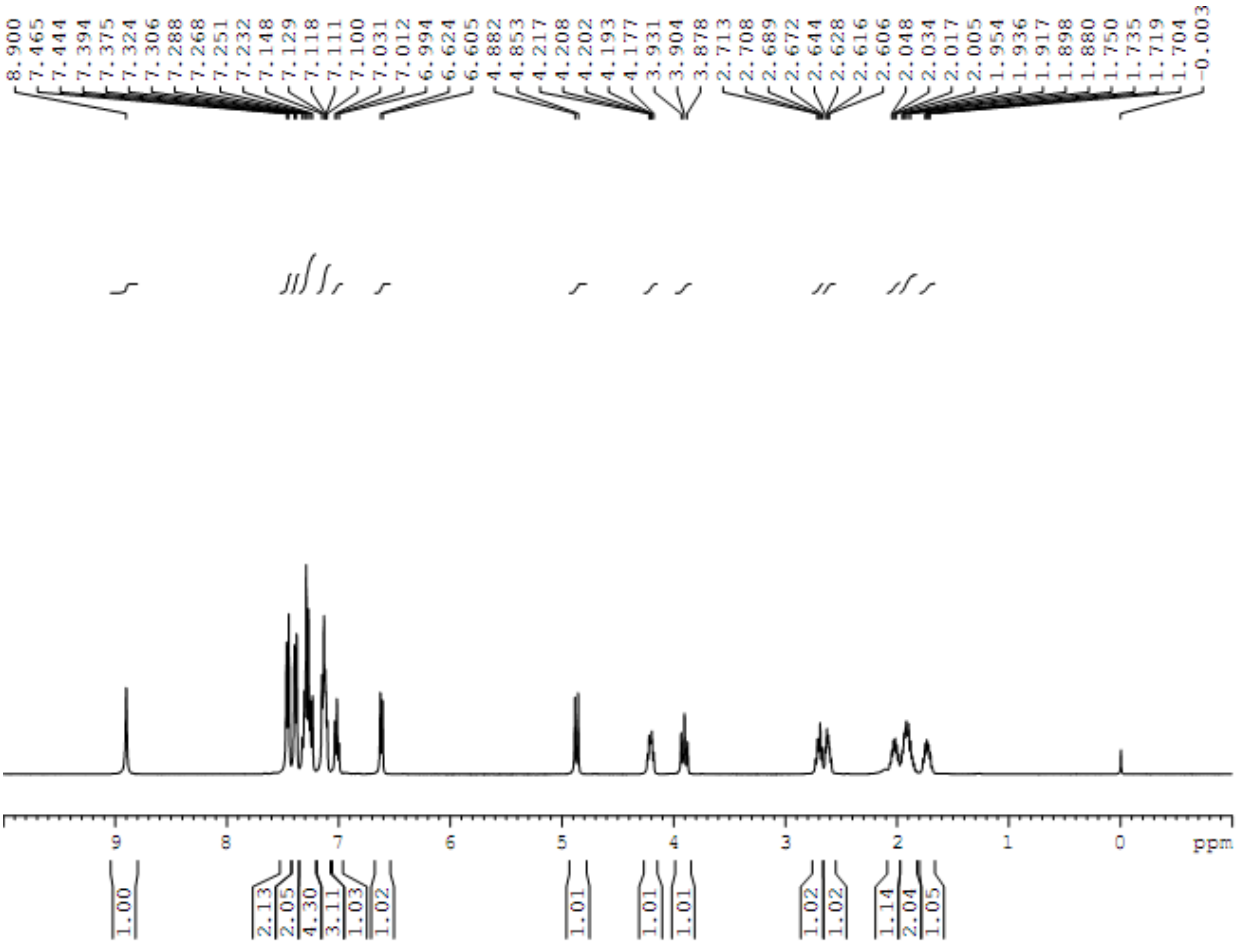


Expanded ^{1}H-^{13}C COSY spectrum of compound 10d

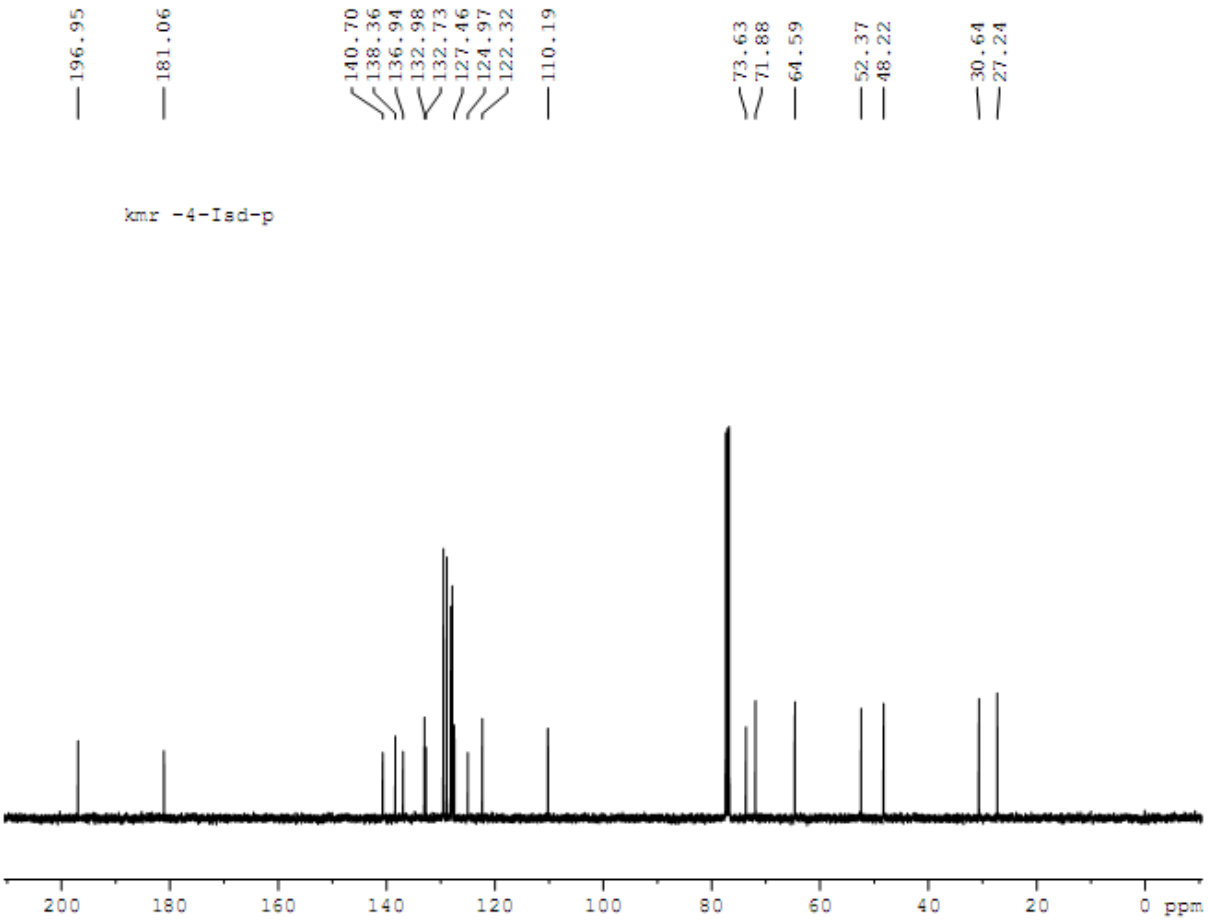


Expanded ^{1}H-^{13}C COSY spectrum of compound 10d



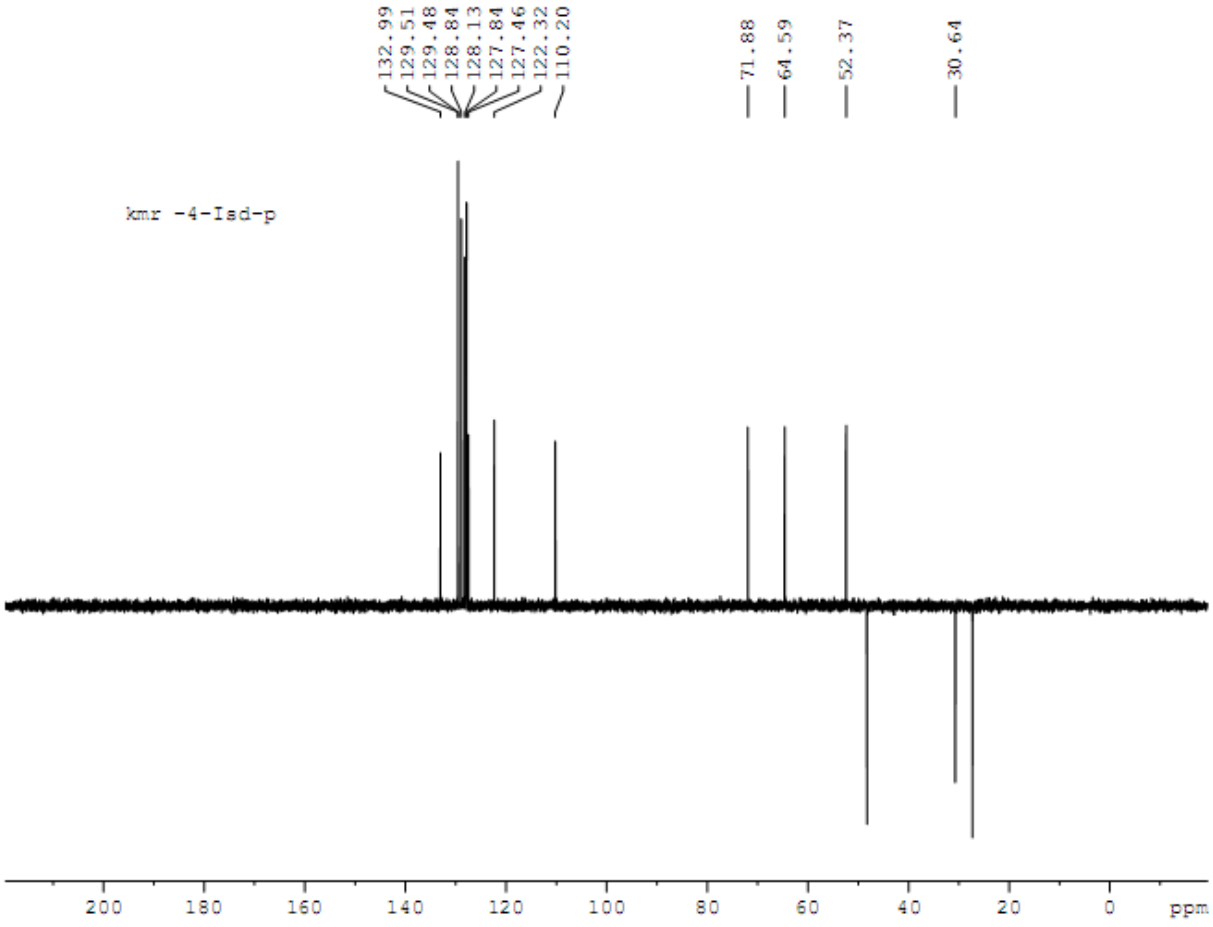
^{1}H NMR Spectrum of **6B**

(Spiropyrrolizidine from Isatin, D-Proline and 4-chlorobenzylidine acetophenone)



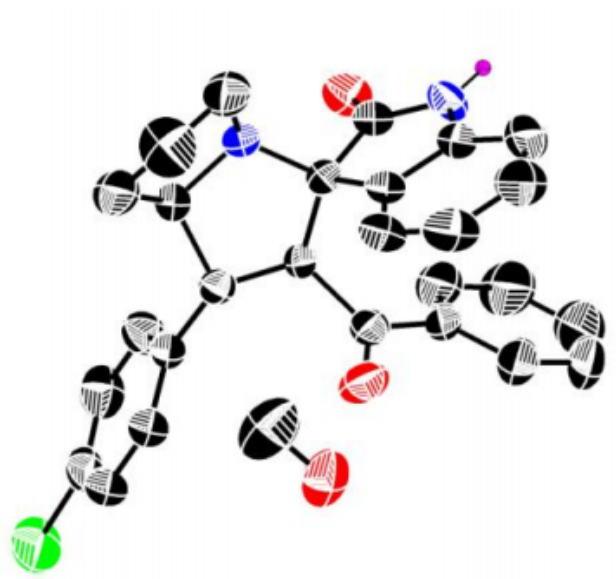
^{13}C NMR Spectrum of **6B**

(Spiropyrrolizidine from Isatin, D-Proline and 4-chlorobenzylidine acetophenone)

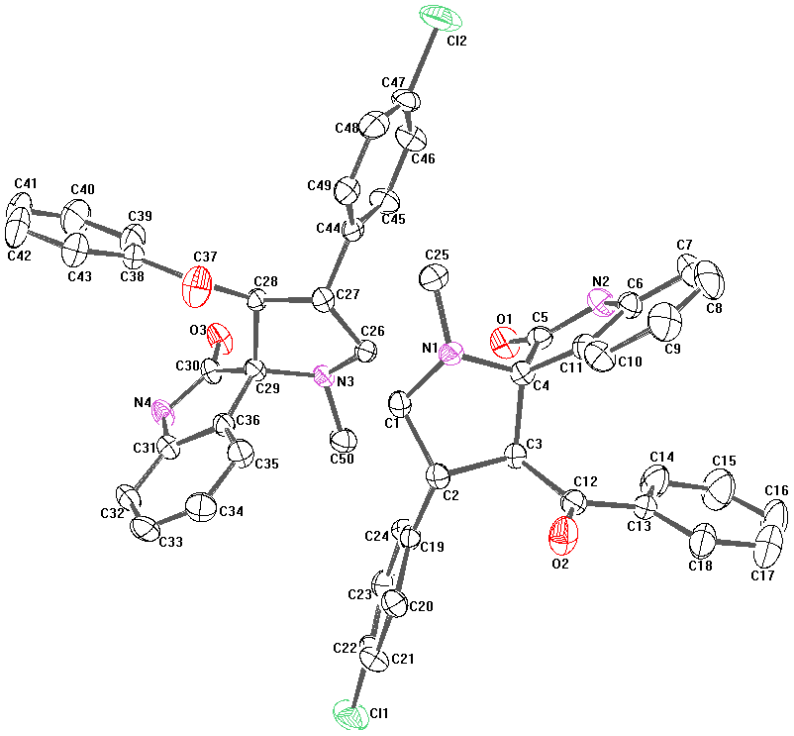


DEPT ^{13}C NMR Spectrum of **6B**

(Spiropyrrolizidine from Isatin, D-Proline and 4-chlorobenzylidine acetophenone)



ORTEP representation of the crystal structure of compound **6b**. Thermal ellipsoids are drawn at 50%probability and all H atoms are removed for clarity



ORTEP representation of the crystal structure of compound **6n**. Thermal ellipsoids are drawn at 30%probability and all H atoms are removed for clarity