

# Ionic Liquids as a Convenient New Medium for the Catalytic Asymmetric Dihydroxylation of Olefins Using a Recoverable and Reusable Osmium/Ligand

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**General:** The following chemicals were purchased from commercial sources and used as supplied: styrene, -methylstyrene, 1-hexene, 1-methylcyclohexene, *trans*-stilbene, *trans*-5-decene, K<sub>2</sub>CO<sub>3</sub>, K<sub>2</sub>OsO<sub>2</sub>(OH)<sub>4</sub>, K<sub>3</sub>[Fe(CN)<sub>6</sub>], *N*-methyl-morpholine oxide (NMO), (DHQD)<sub>2</sub>PHAL, (DHQD)<sub>2</sub>PYR and *t*-butanol. All aqueous solutions were prepared using distilled water. The room temperature ionic liquids (RTIL) based on the methylimidazolium cation [mim]<sup>1</sup>, and on the tetra-alkyl-dimethylguanidinium cation [(be)<sub>2</sub>dmg]<sup>2</sup> were prepared following reported procedures. <sup>1</sup>H, and <sup>13</sup>C NMR spectra were recorded on a Bruker AMX 400 spectrometer. Chemical shifts are reported downfield in parts per million (ppm) from a tetramethylsilane reference for <sup>1</sup>H and <sup>13</sup>C NMR. IR spectra were recorded on a FTIR instrument as thinly dispersed films.

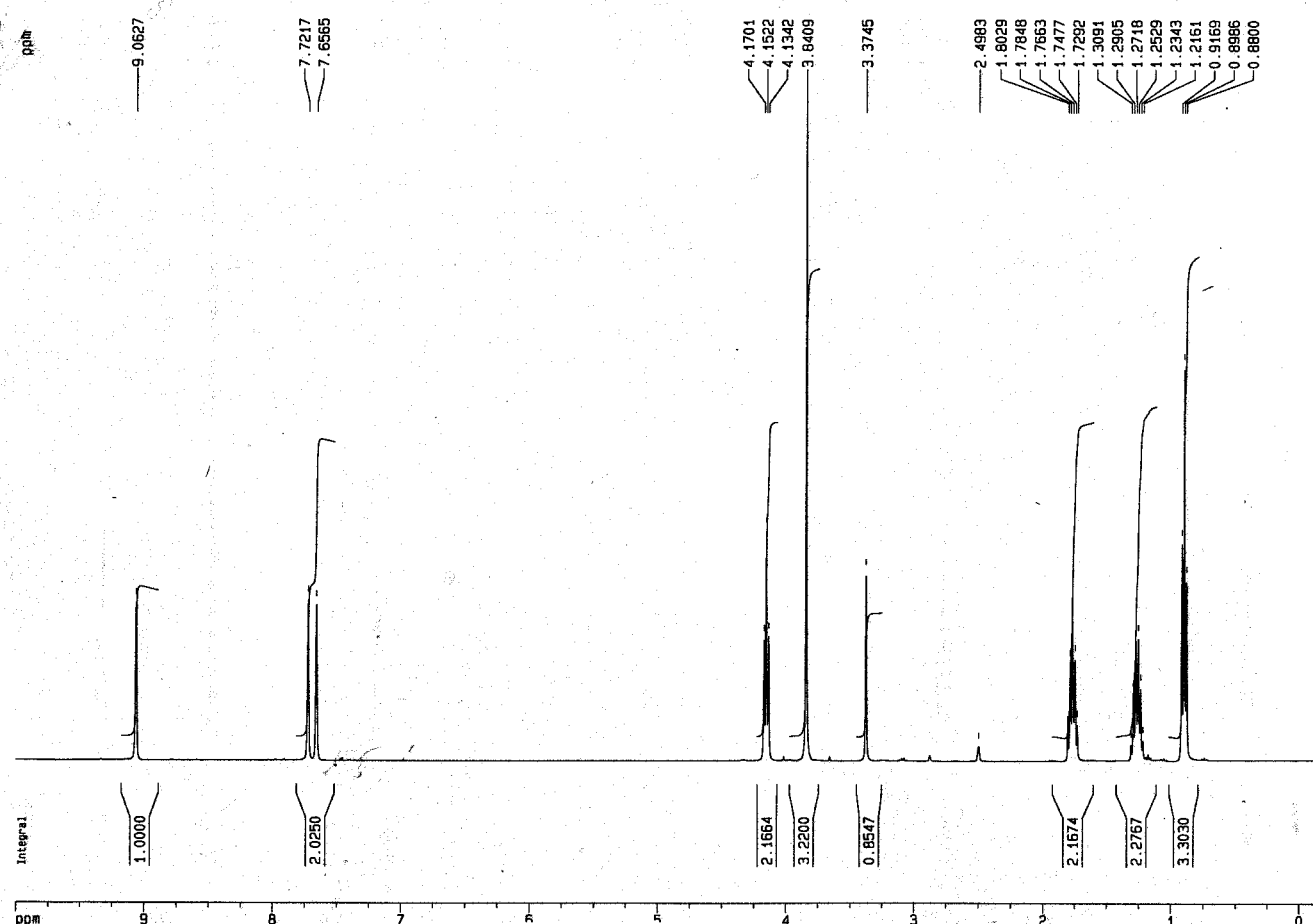
Gas liquid chromatography (GLC) was carried out using He as carrier gas and chiral capillary column Supelco  $\alpha$ -dex 120 (30m x 0.25 mm). HPLC analyses were performed using Chiralpak AD column at 25 °C. Flash chromatography was carried out using an MN-Kiesel-gel 60M gel (230-400 mesh ASTM, Art. 815381). All eluents were distilled prior to use. Preparative and analytical thin layer chromatography (TLC) was carried out using, respectively, MN Kieselgel G/UV254 (Art. 816320) glass-backed plates and MN Alugram, SIL G/UV<sub>254</sub> (Art. 818133). The plates were visualised using ultraviolet light (254 nm) or using phosphomolybdic acid.

## References

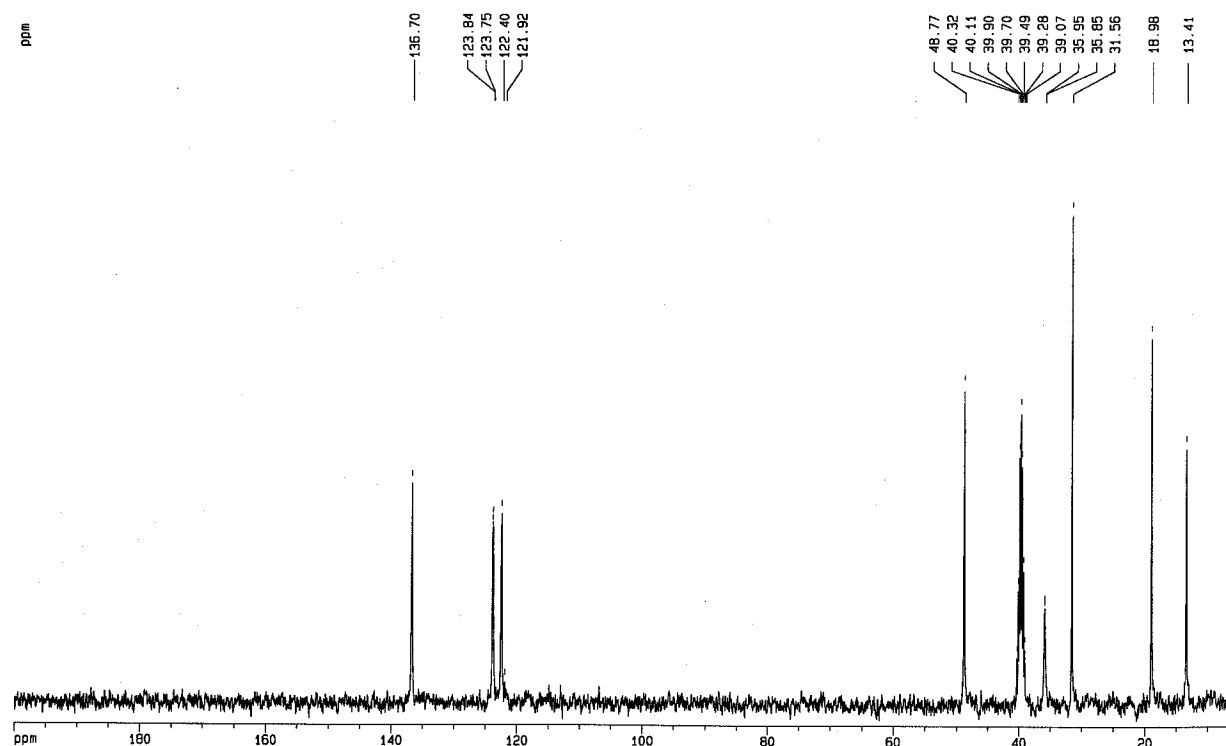
- [1] a) T. Kitazume, F. Zulfiqar, G. Tanaka, *Green Chemistry*, **2000**, 2, 133; b) A. E Visser, R. P. Swatloski, R. D. Rogers, *Green Chemistry*, **2000**, 2, 1. c) L. C. Branco, J. N. Rosa, J. J. M. Ramos, C. A. M. Afonso, *Chem. Eur. J.* **2002**, 8, 3671.  
 [2] N. M. M. Mateus, L. C. Branco, N. M. T. Lourenço, C. A. M. Afonso, *Green Chem.* **2003**, 5, 347.

## Comparison of Spectral Data (<sup>1</sup>H and <sup>13</sup>C NMR) of the Ionic Liquid Between the Initial and Final Recycled Samples

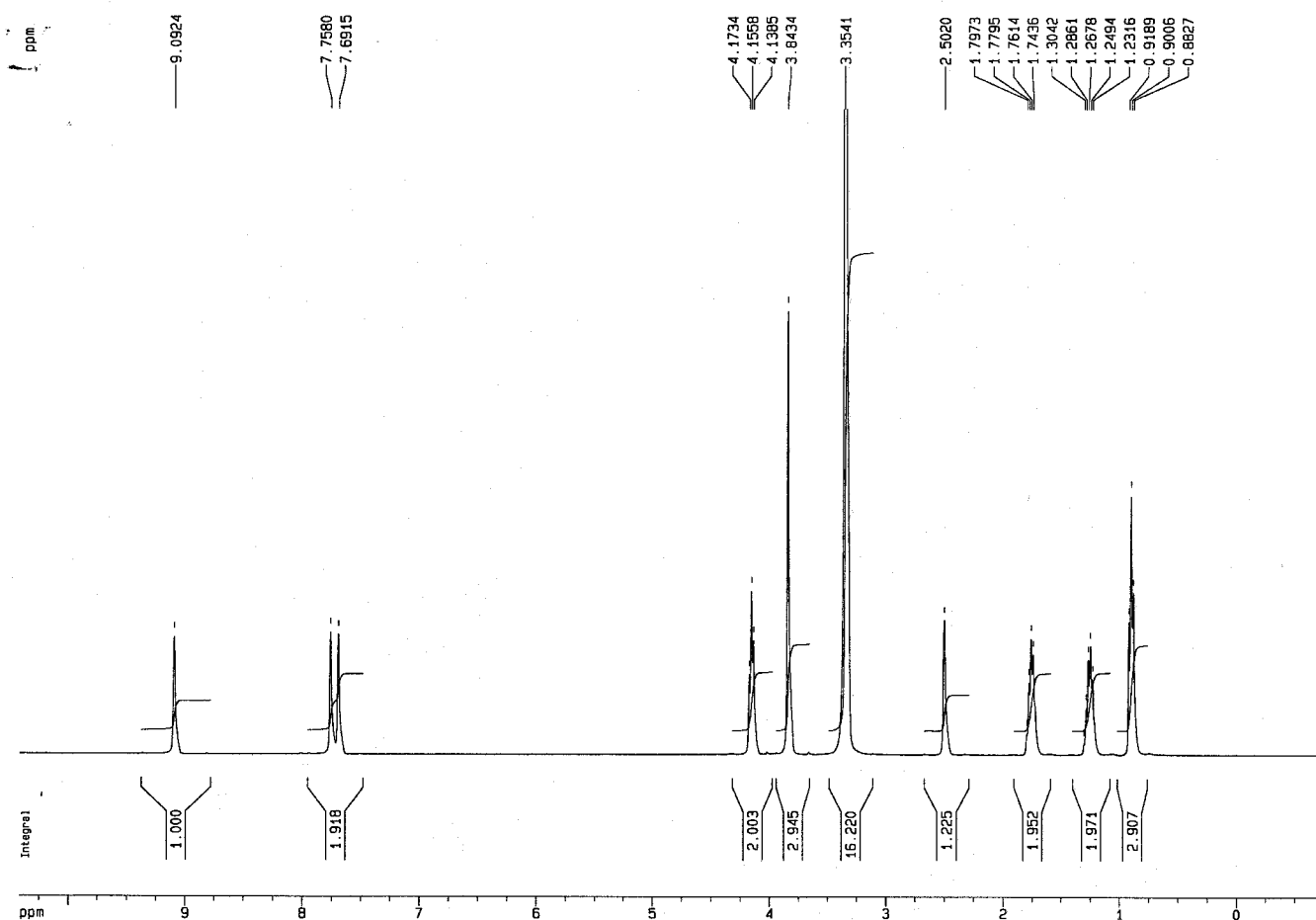
**Figure 1.** <sup>1</sup>H NMR spectra (in *d*<sub>6</sub>-DMSO) of initial [C<sub>4</sub>mim][PF<sub>6</sub>]



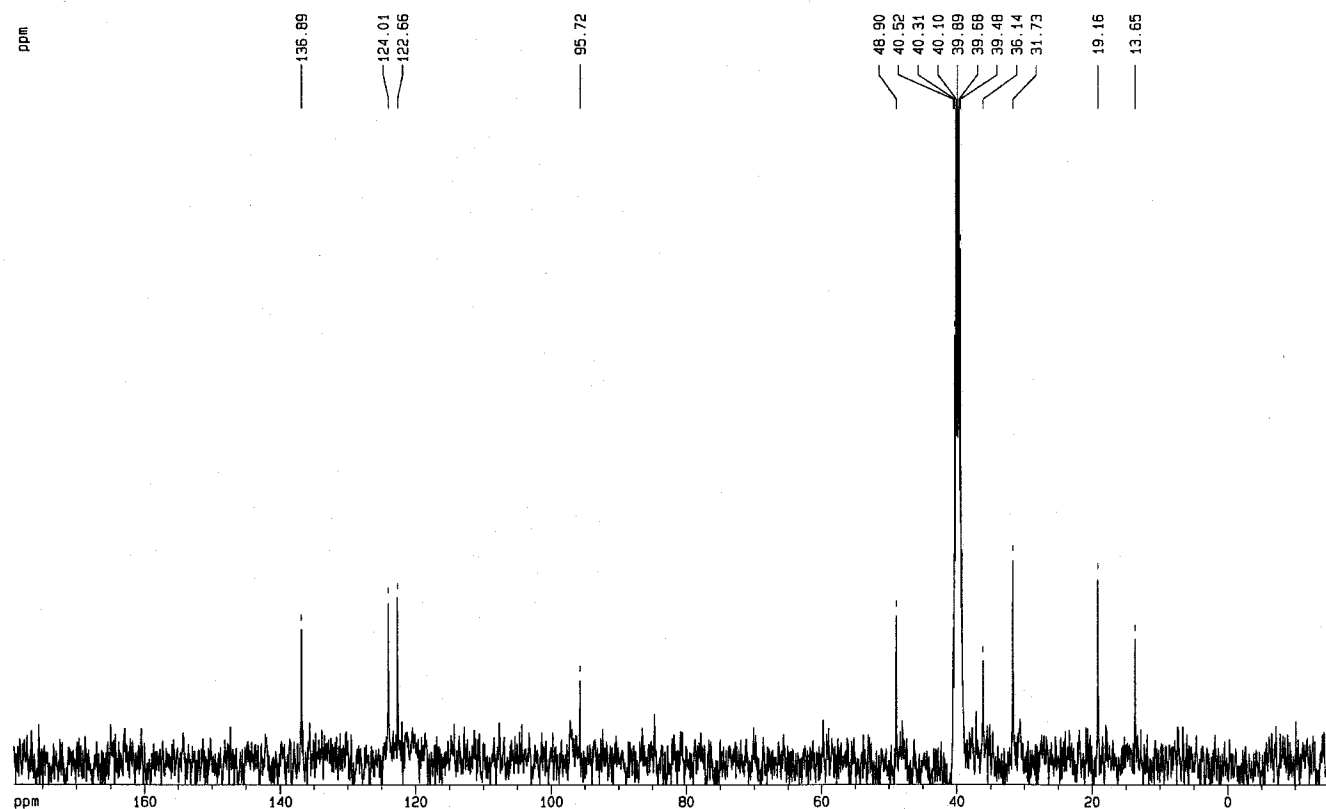
**Figure 2.**  $^{13}\text{C}$  NMR spectra (in  $d_6$ -DMSO) of initial  $[\text{C}_4\text{mim}][\text{PF}_6]$



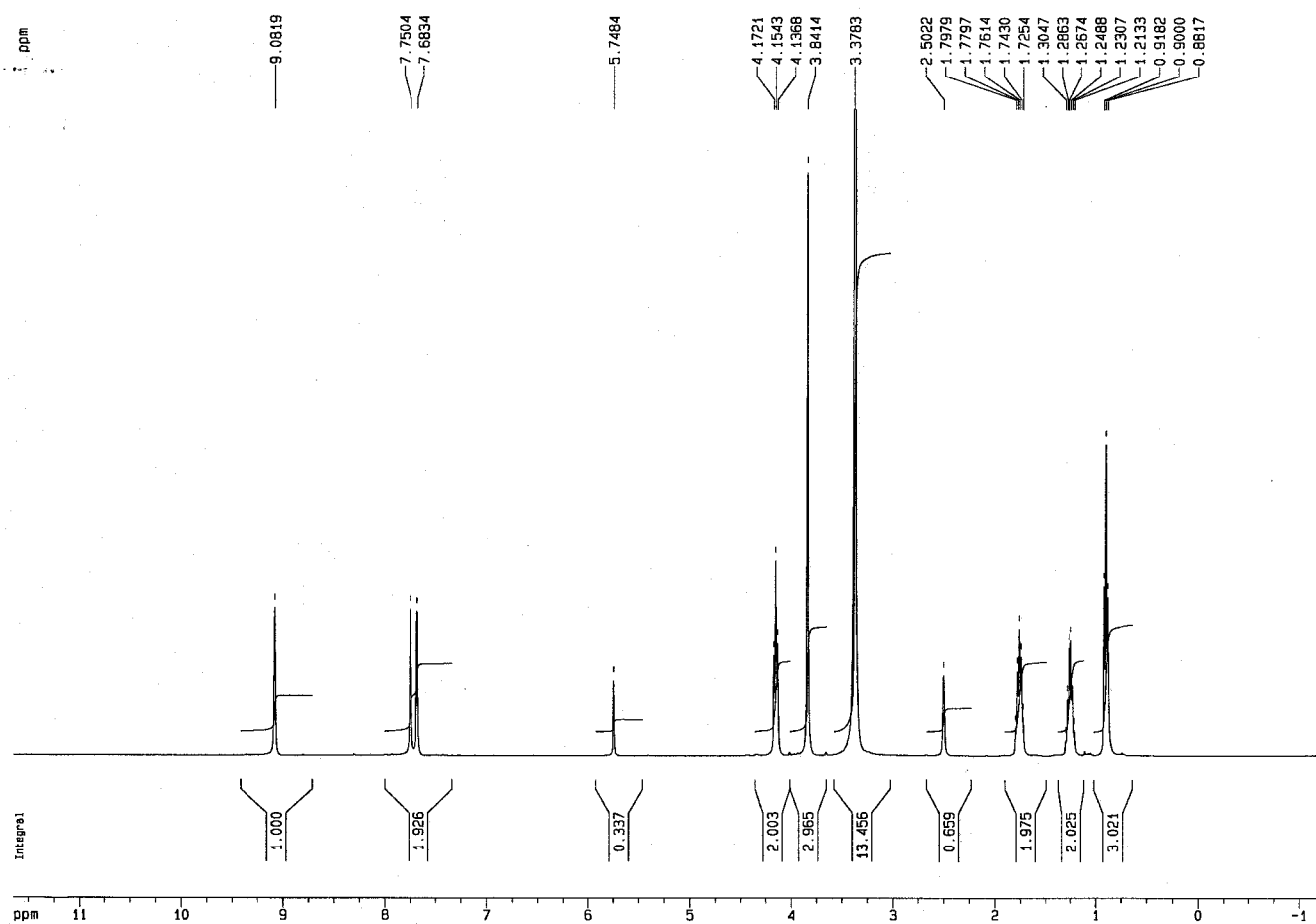
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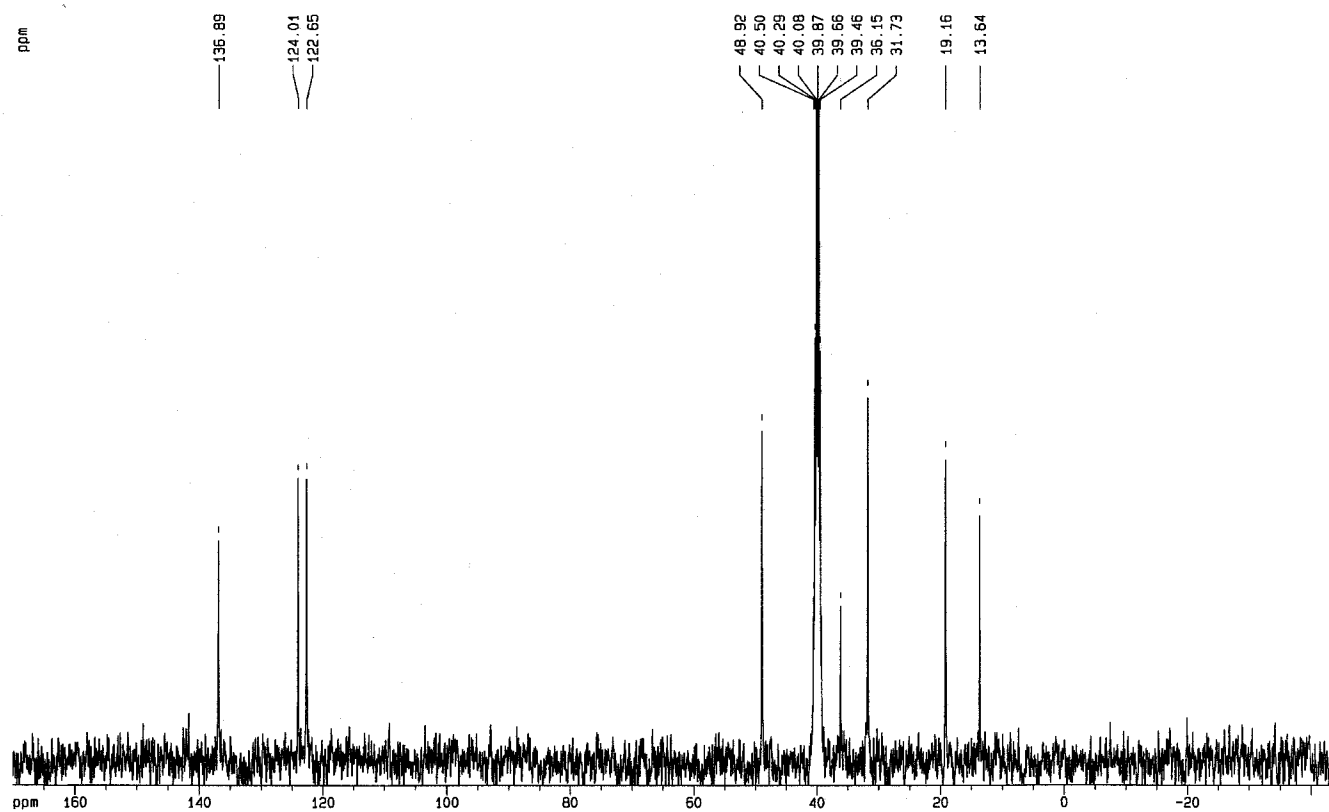
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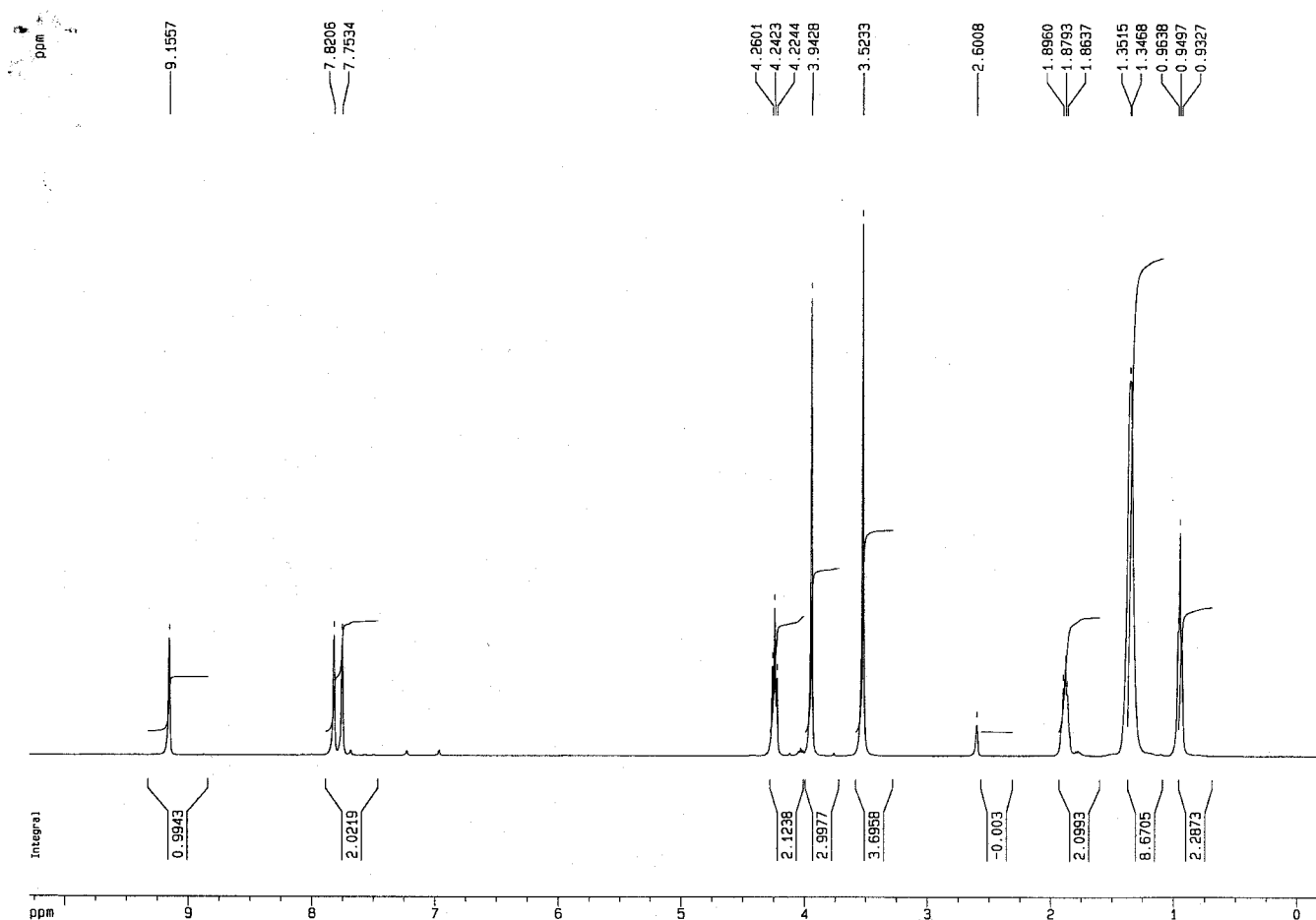
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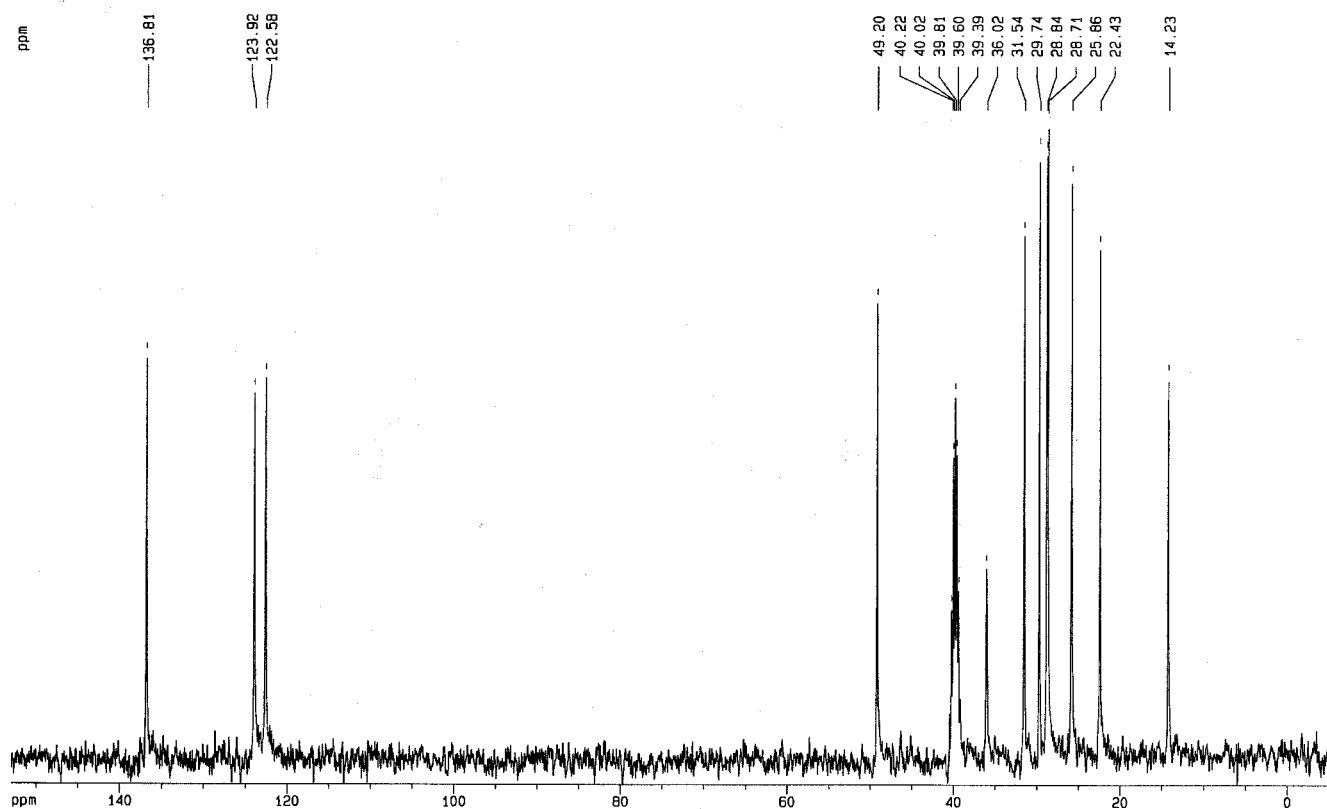
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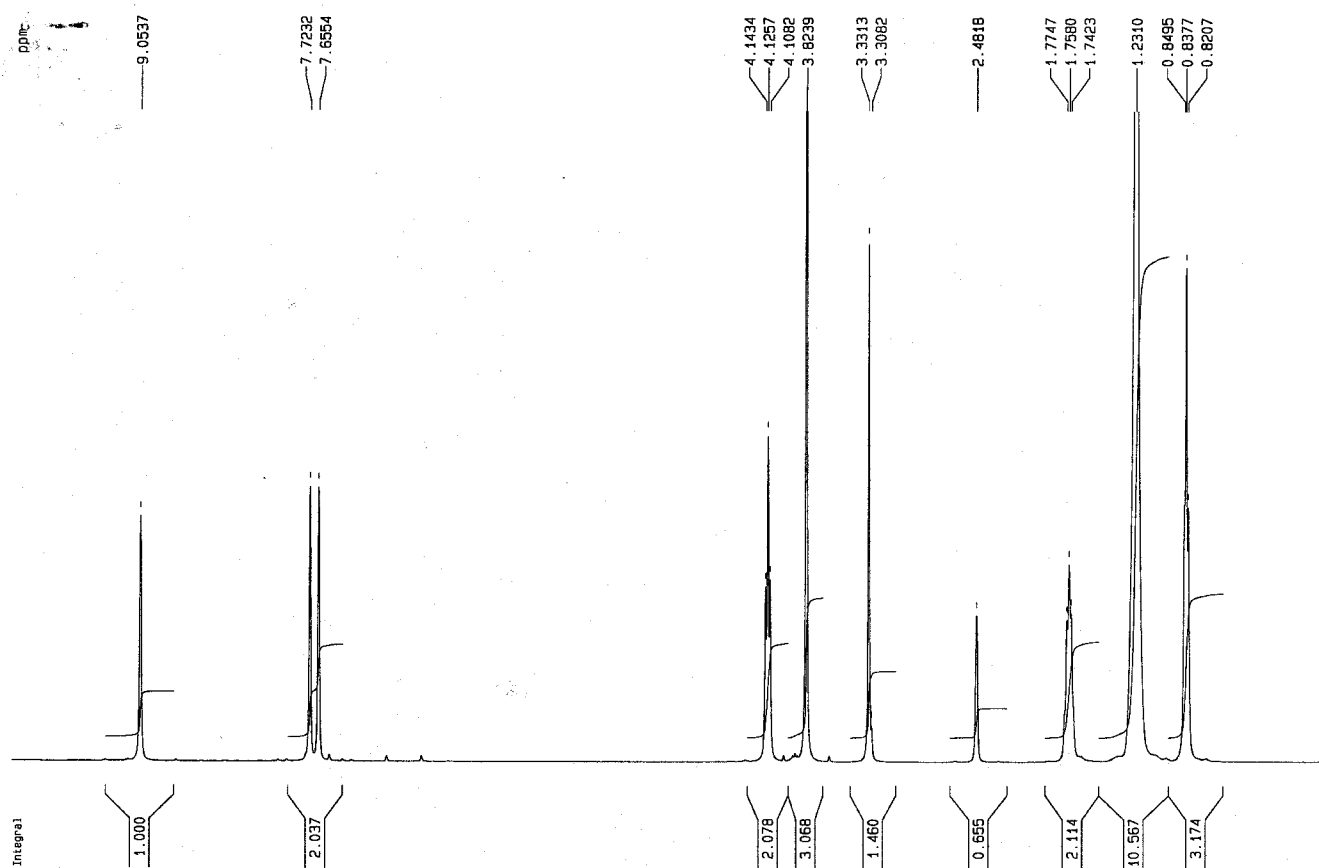
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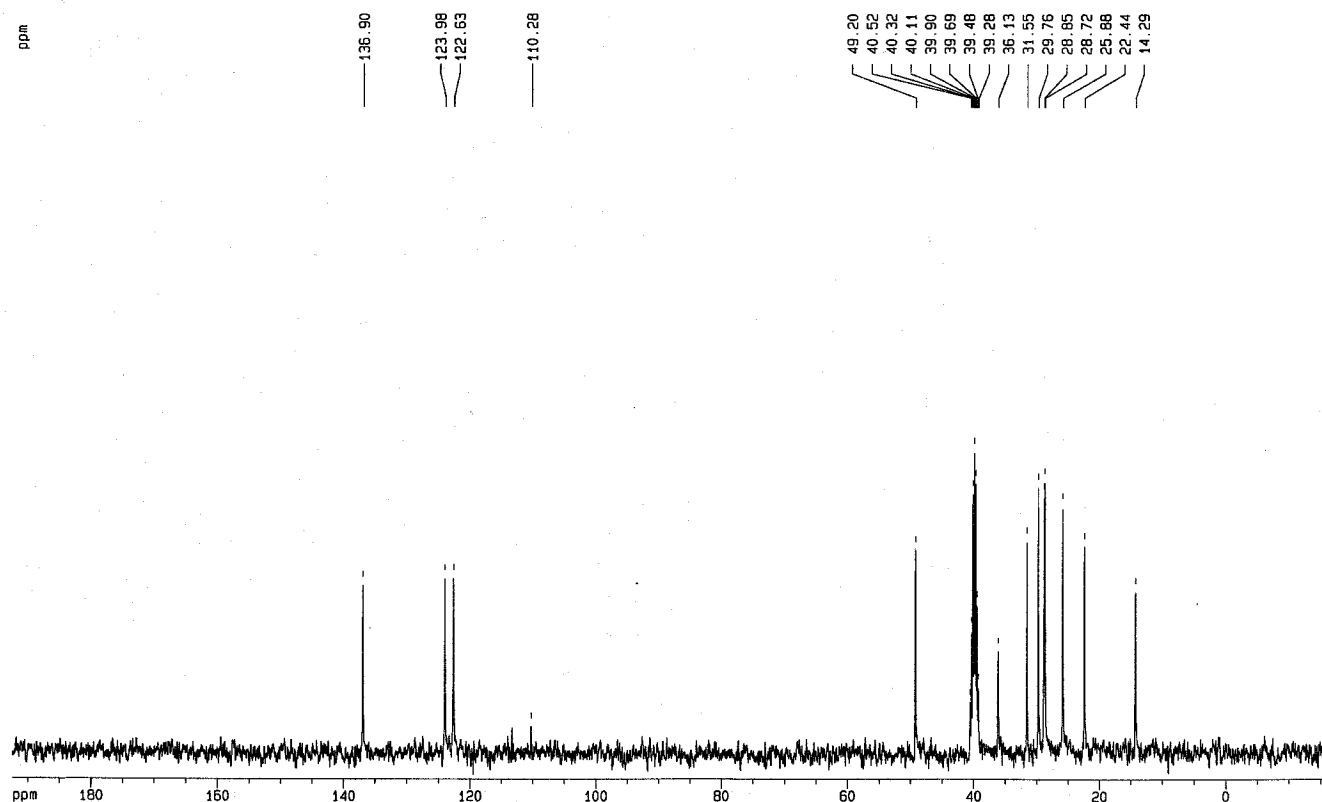
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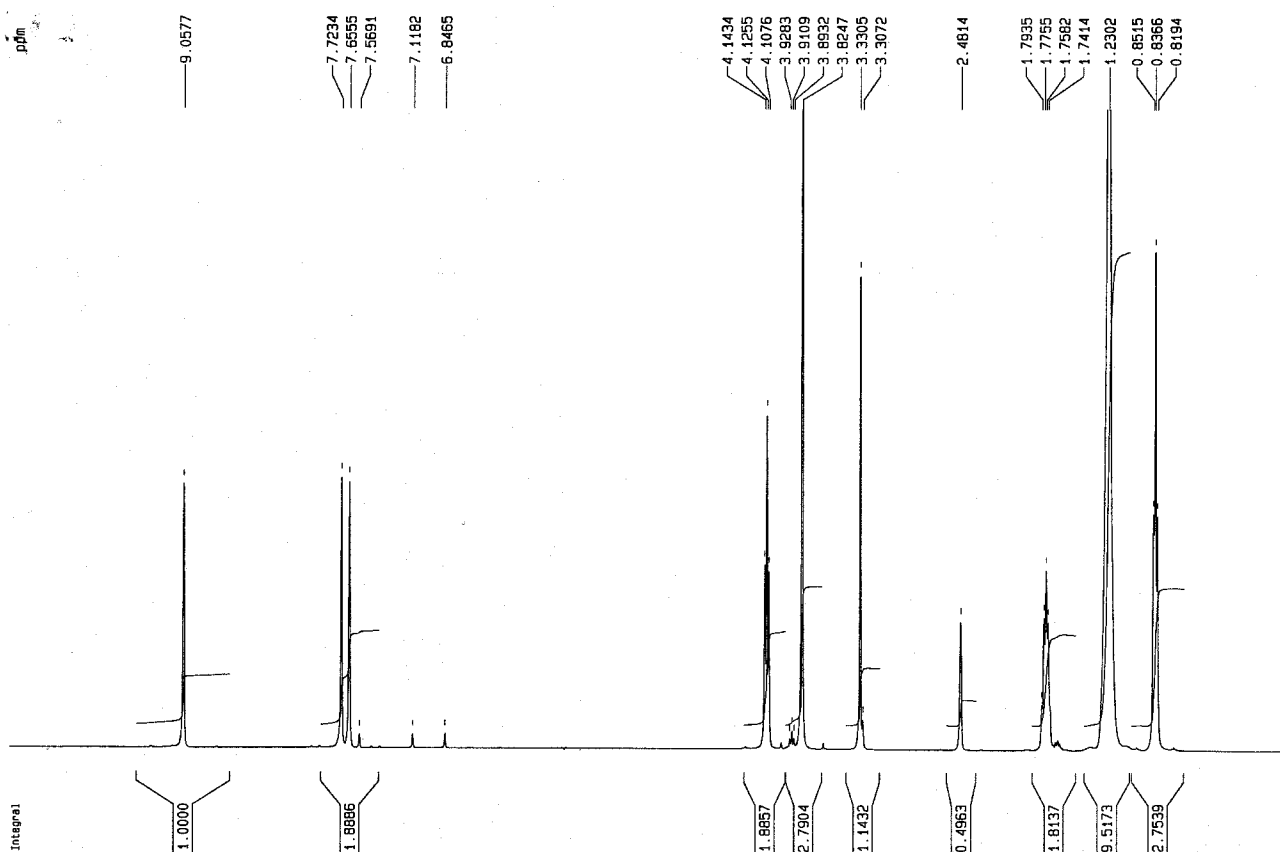
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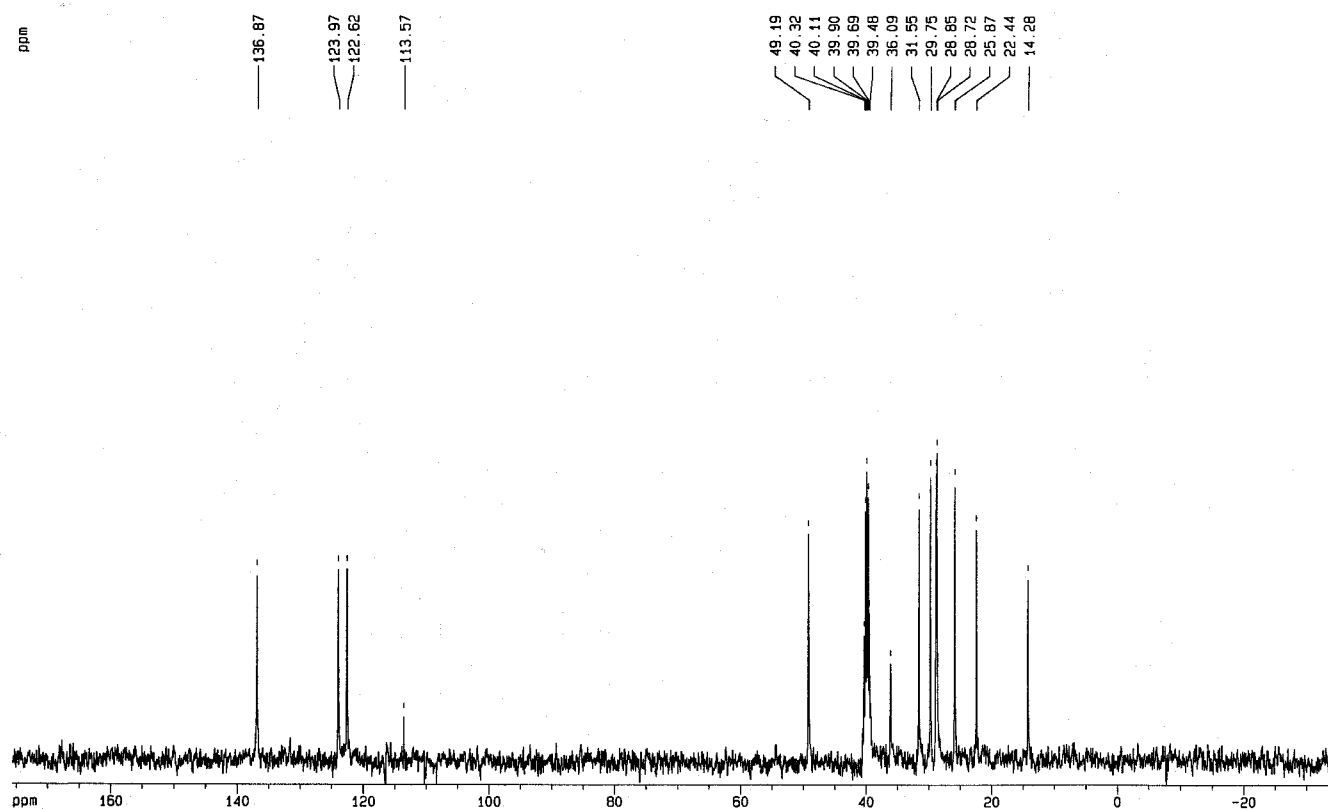
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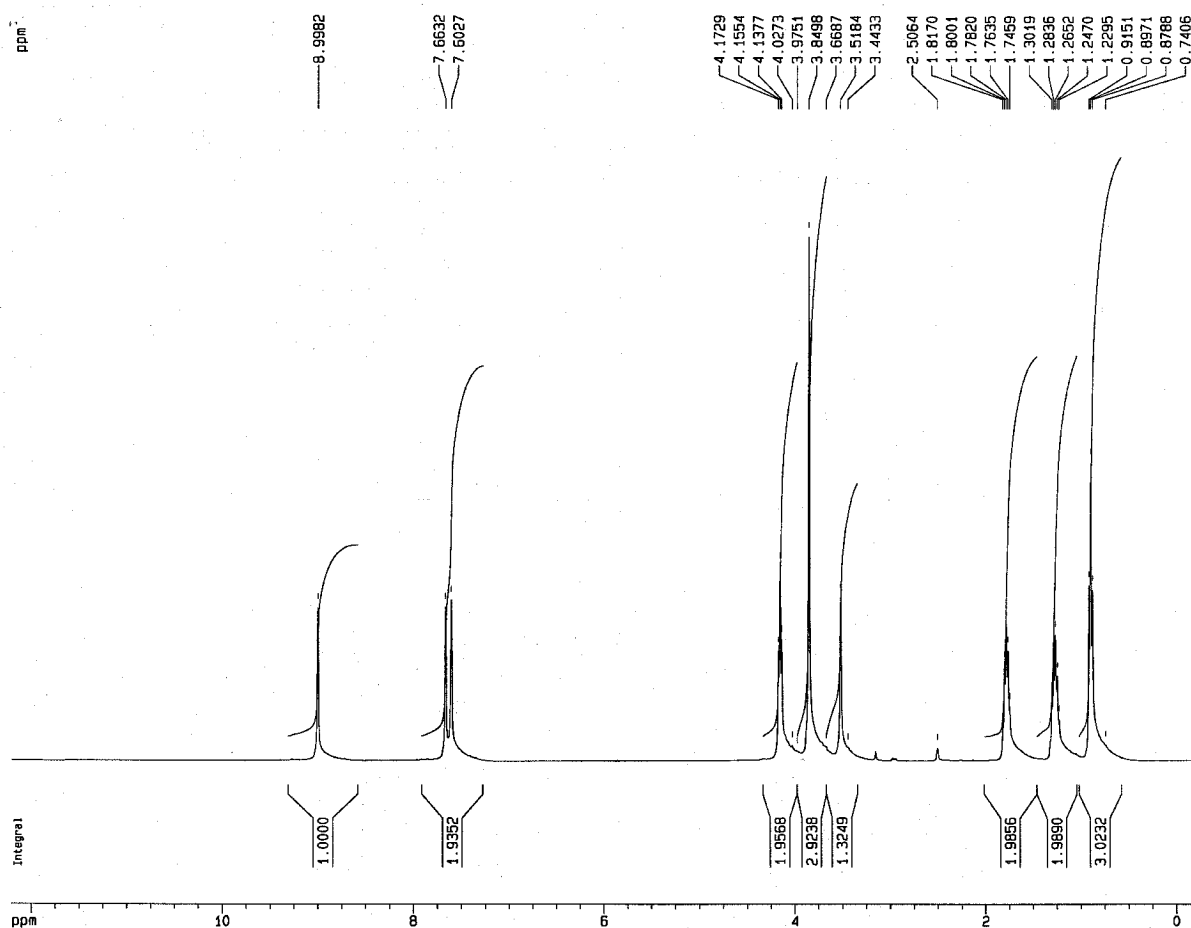
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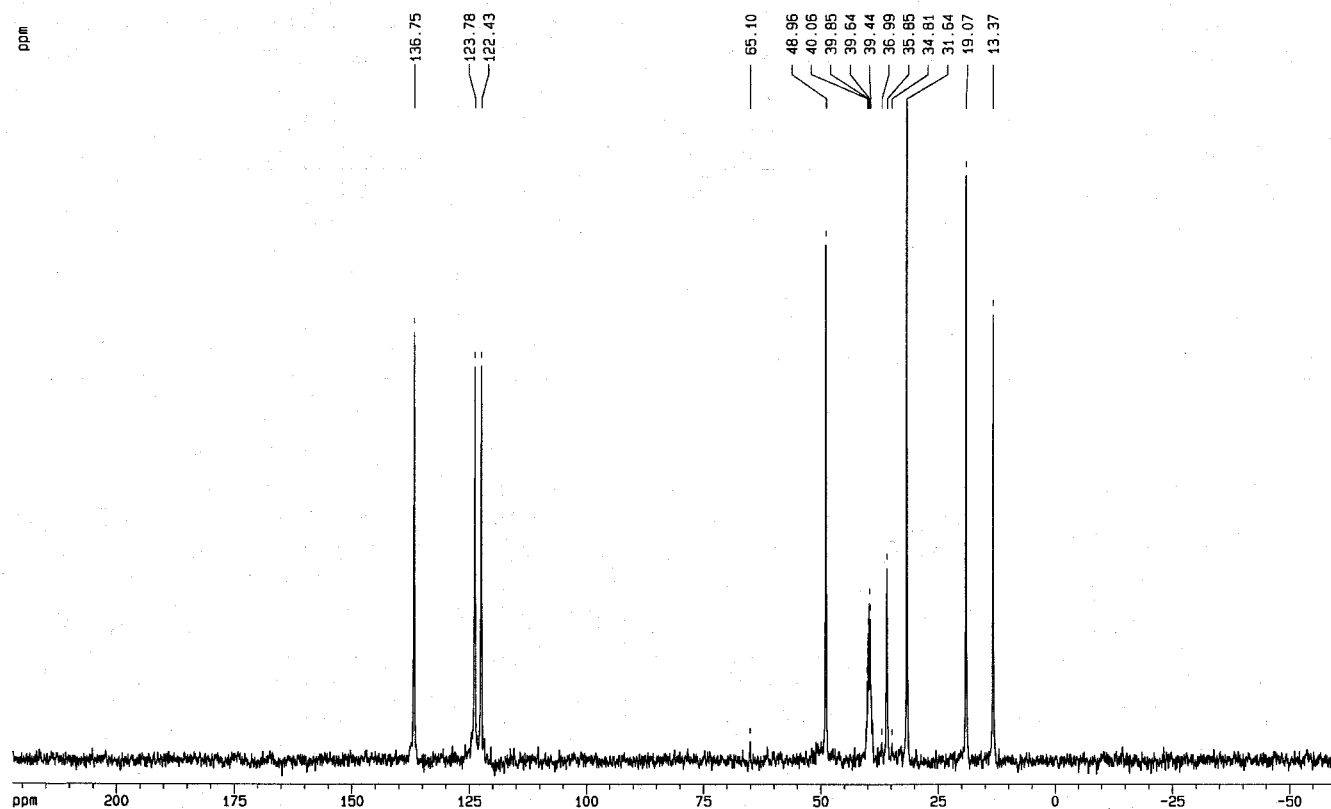


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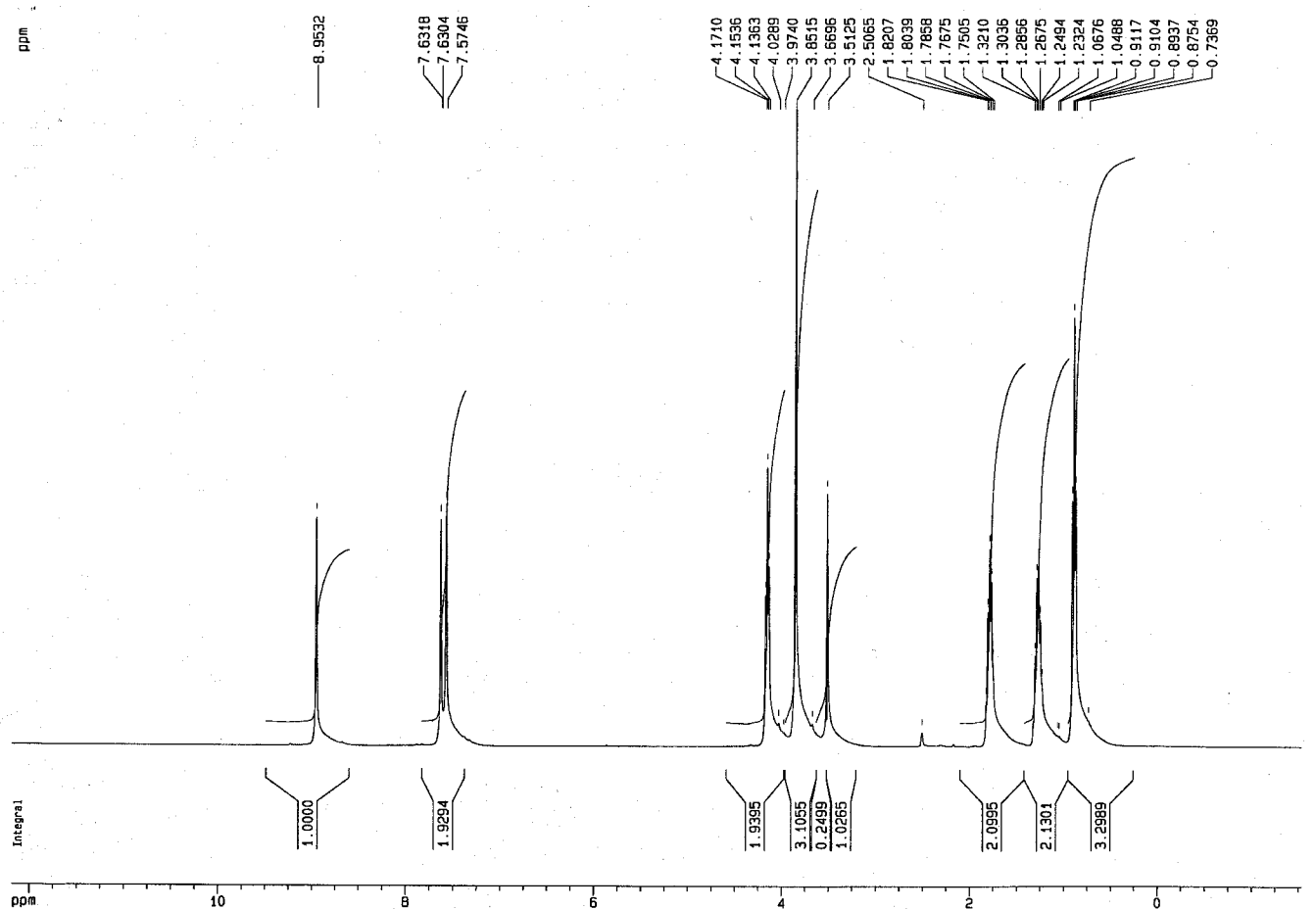




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