Synthesis of Thioamides via One-Pot A³-Coupling of Alkynyl Bromides, Amines, and Sodium Sulfide

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Supporting Information

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A. General method

Melting points were measured with a melting point instrument and were uncorrected. ^{1}H NMR and ^{13}C NMR spectra were recorded on Bruker Avance (400 and 100 MHz, respectively) instrument internally referenced to tetramethylsilane (TMS) or chloroform signals. IR spectra were obtained either as potassium bromide pellets or as liquid films between two potassium bromide pellets with a Bruker Vector 22 spectrometer. GC-MS was obtained using electron ionization (EI). High-resolution mass spectra were obtained with a LCMS-IT-TOF mass spectrometer. TLC was performed by using commercially prepared 100–400 mesh silica gel plates (GF$_{254}$) and visualization was effected at 254 nm. All reagents were obtained from commercial suppliers and used without further purification.

B. General procedure for the synthesis of products
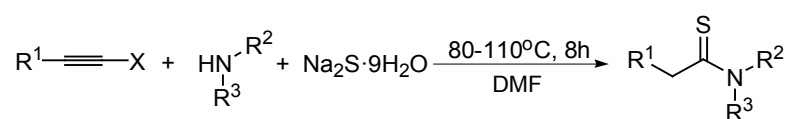
A mixture of alkynyl halide (1.0 mmol), amine (1.5 mmol), and Na$_2$S·9H$_2$O (1.5 mmol) in DMF (2.5 mL) was placed in a sealed tube (25 mL) equipped with a magnetic stirring bar. The mixture was stirred at 80 ^{o}C (or 110 ^{o}C) for 8h. After the reaction was completed, the mixture was washed with brine and extracted with ethyl acetate. The organic layer was dried with anhydrous MgSO$_4$, concentrated in vacuo and purified by flash silica gel chromatography using petroleum ether/ethyl acetate 15:1 to give the desired products.
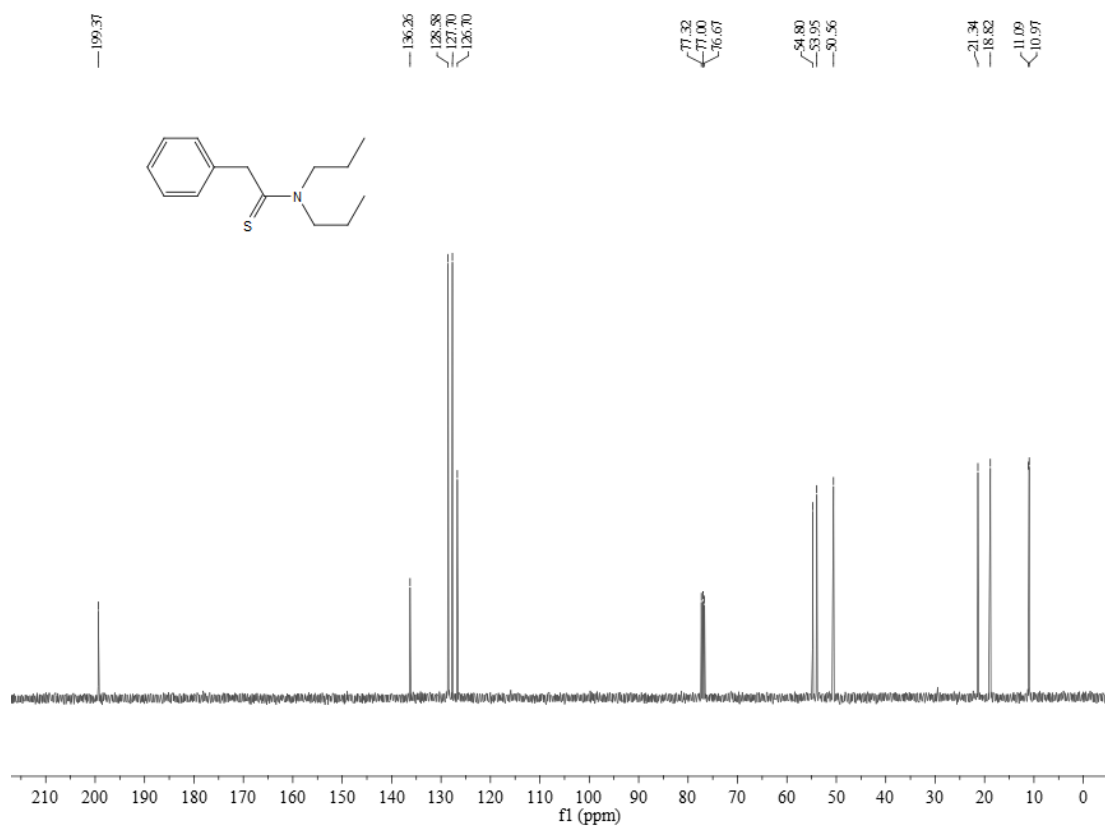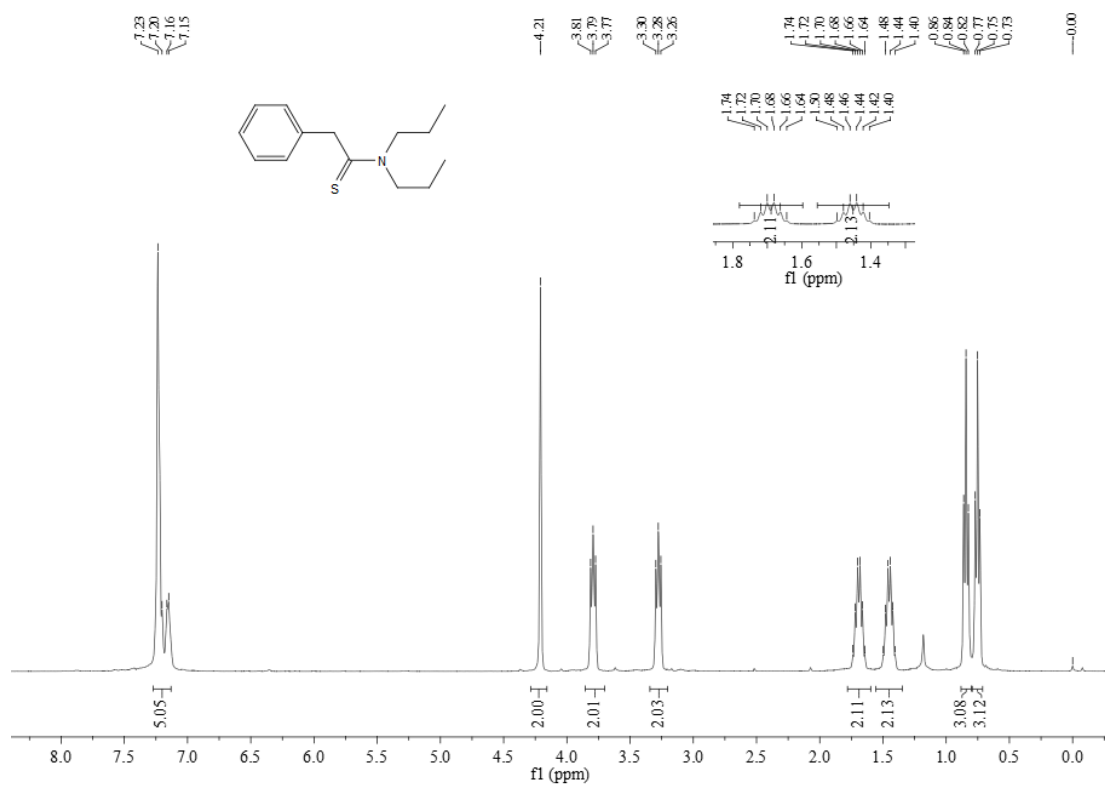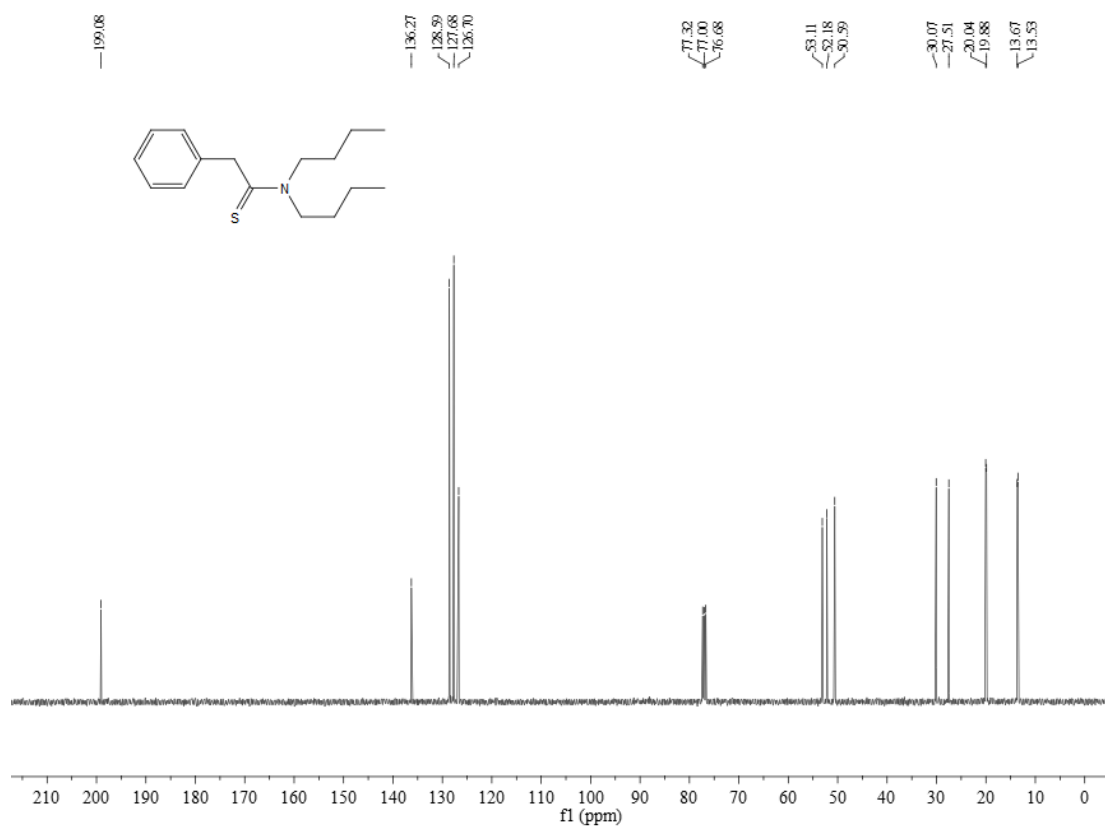
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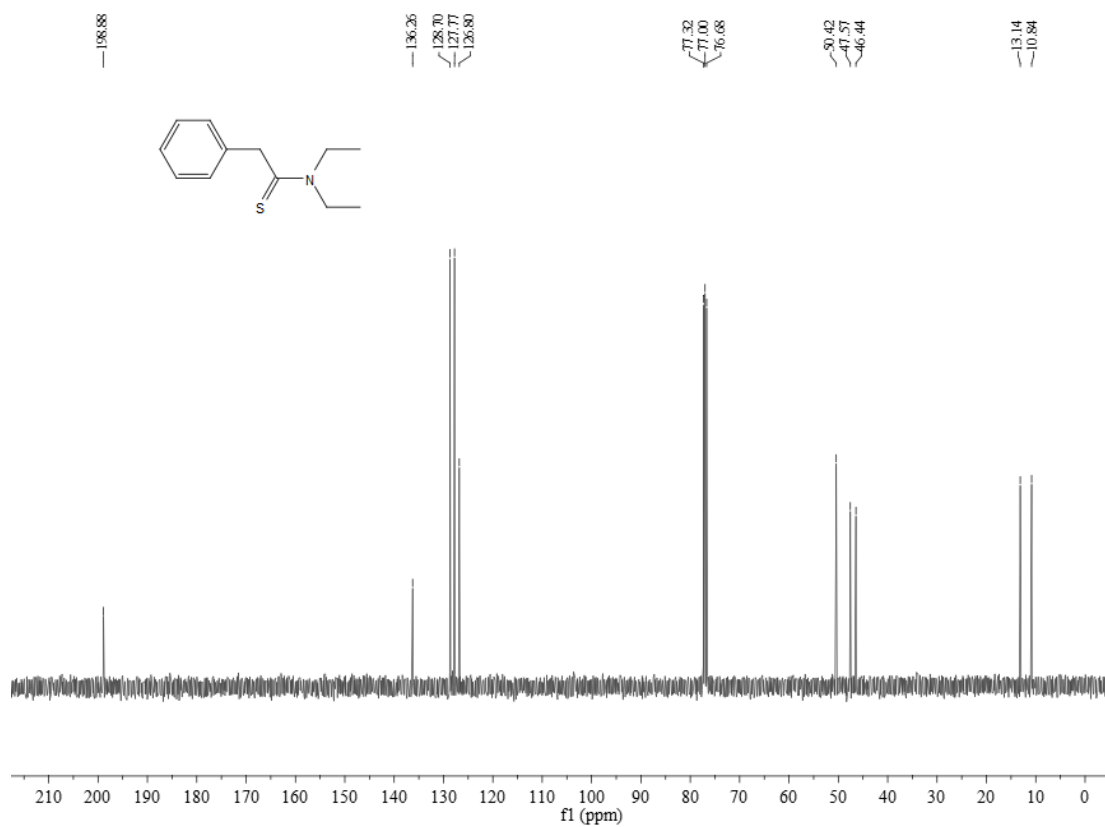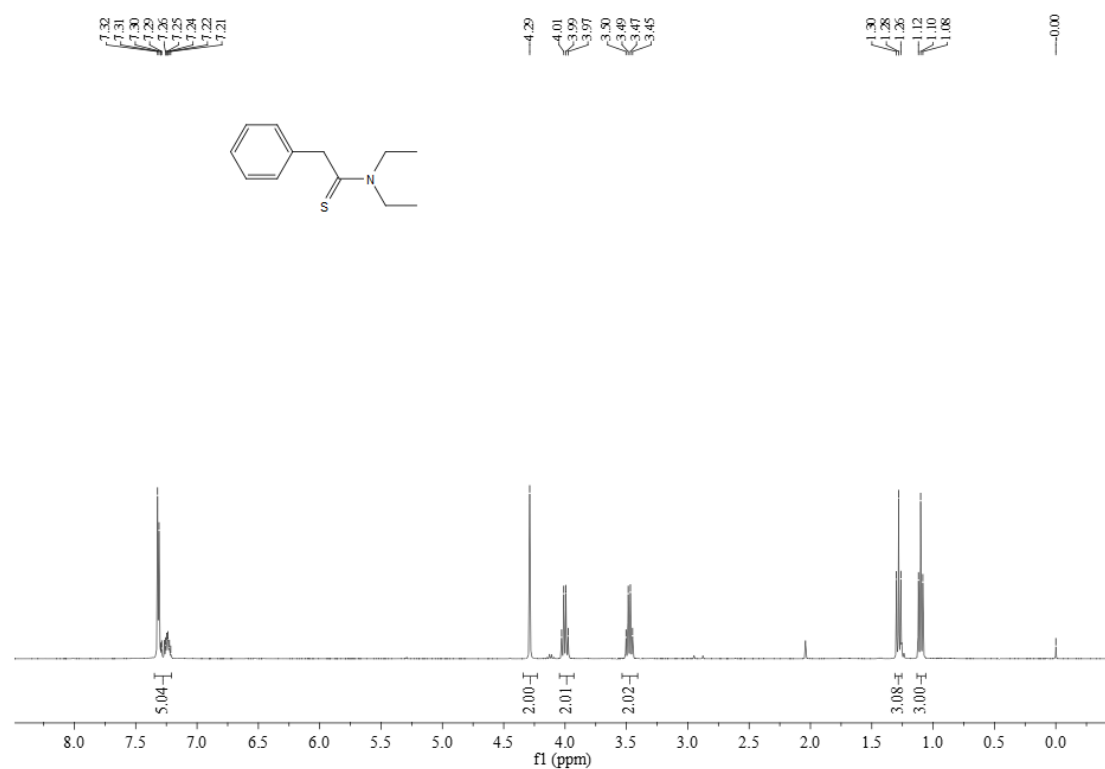
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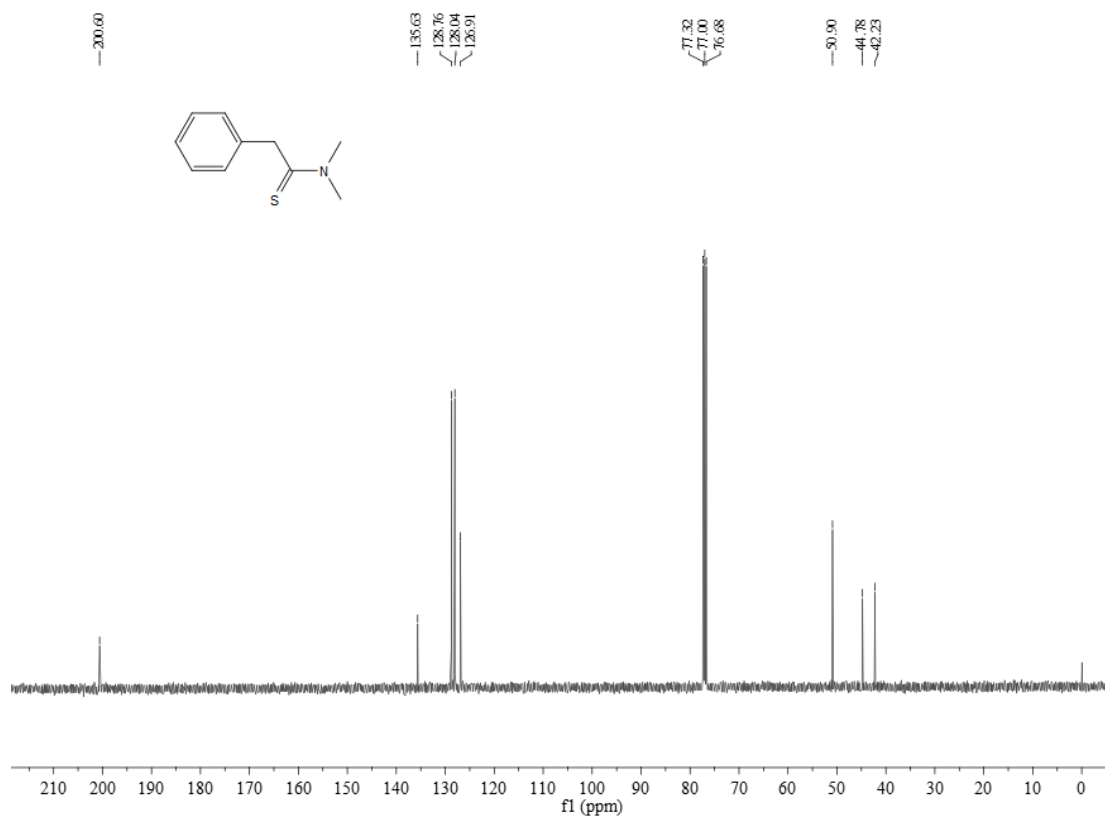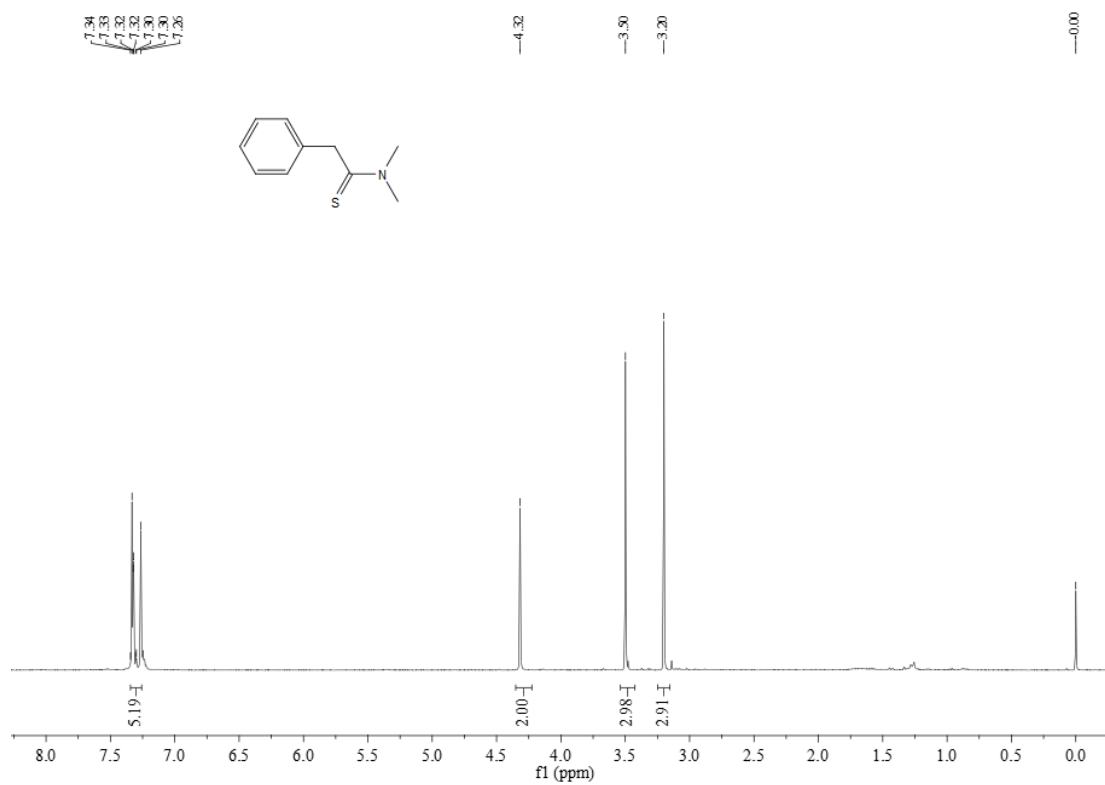
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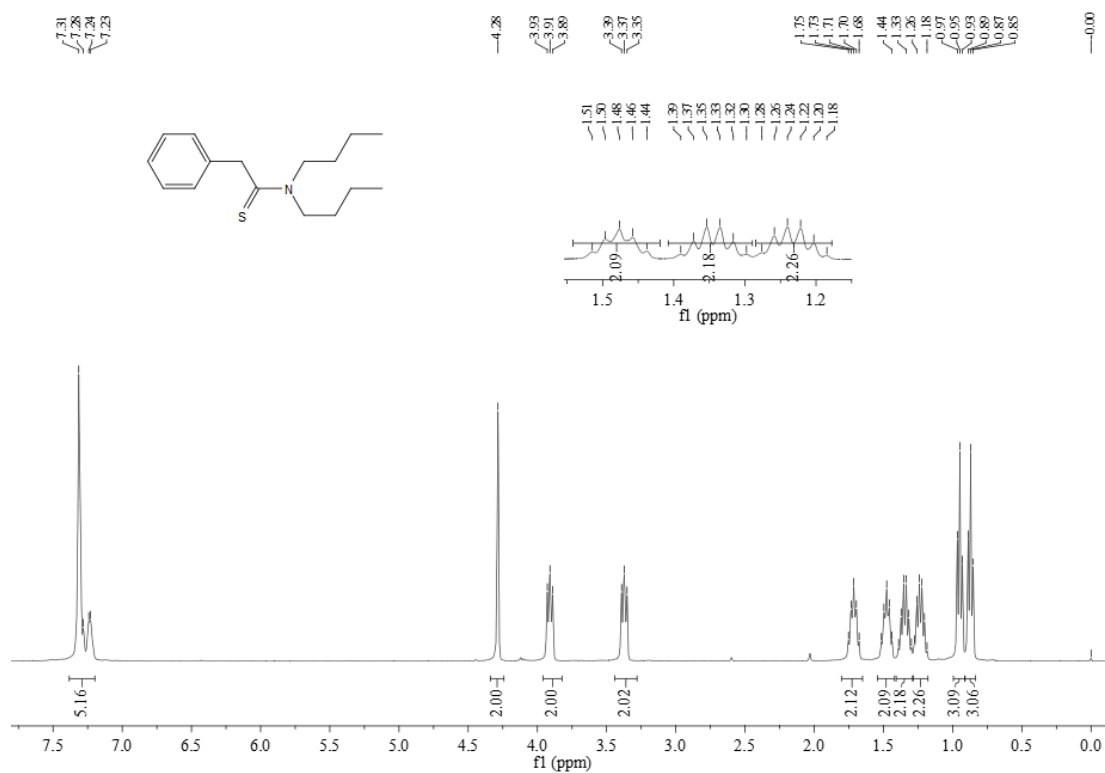
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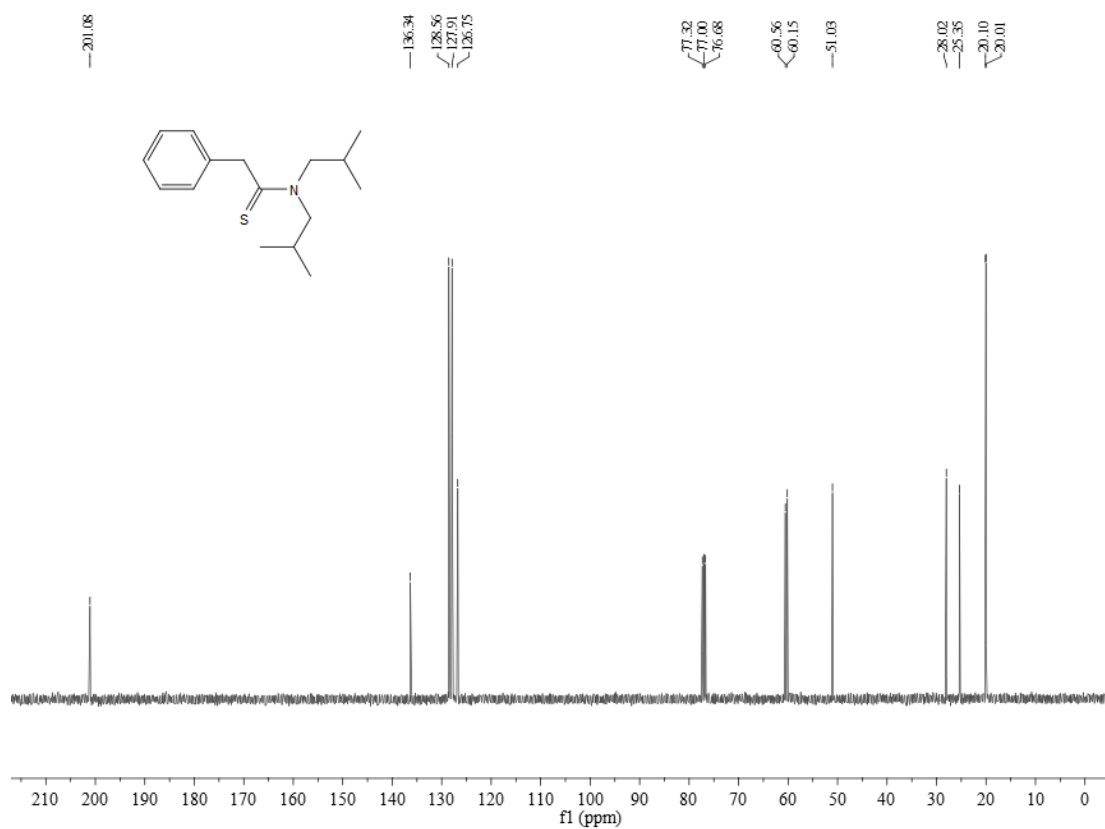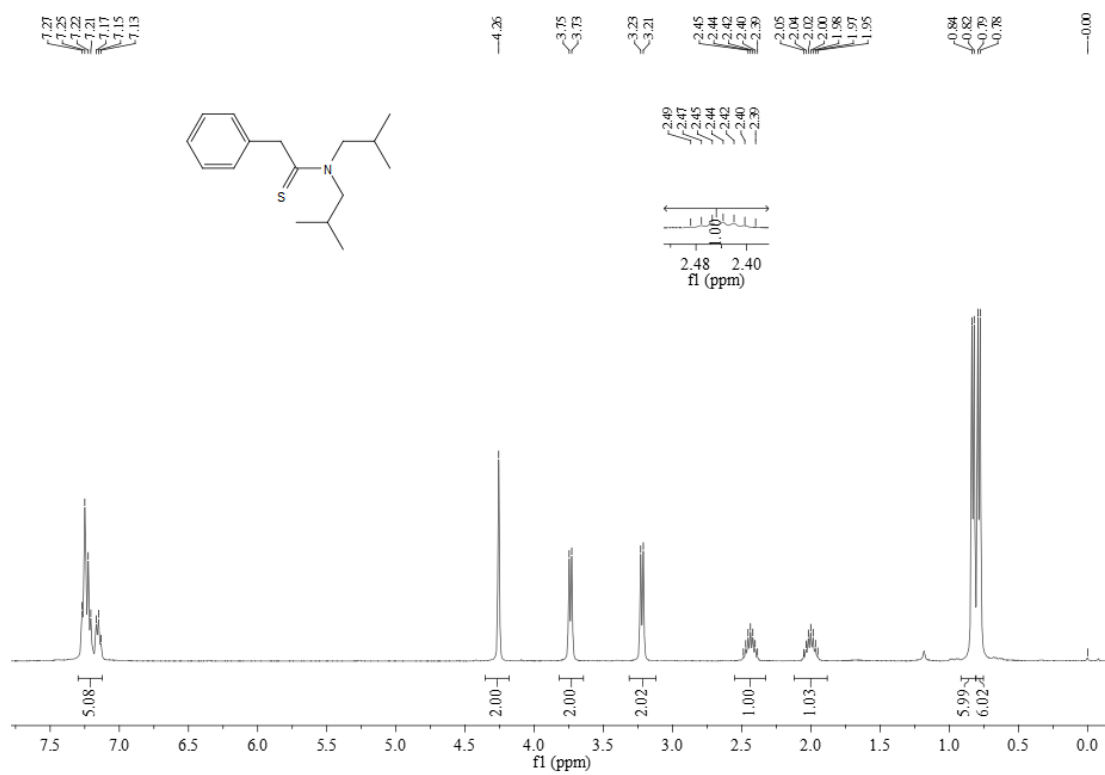
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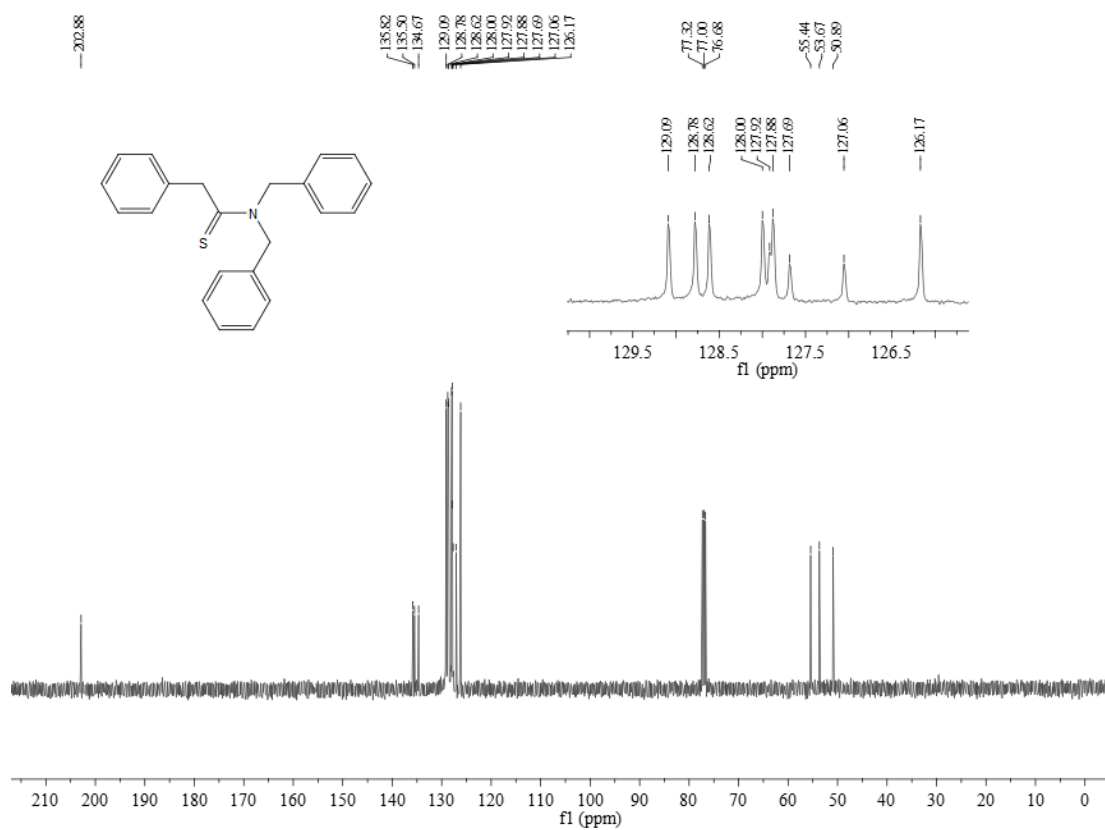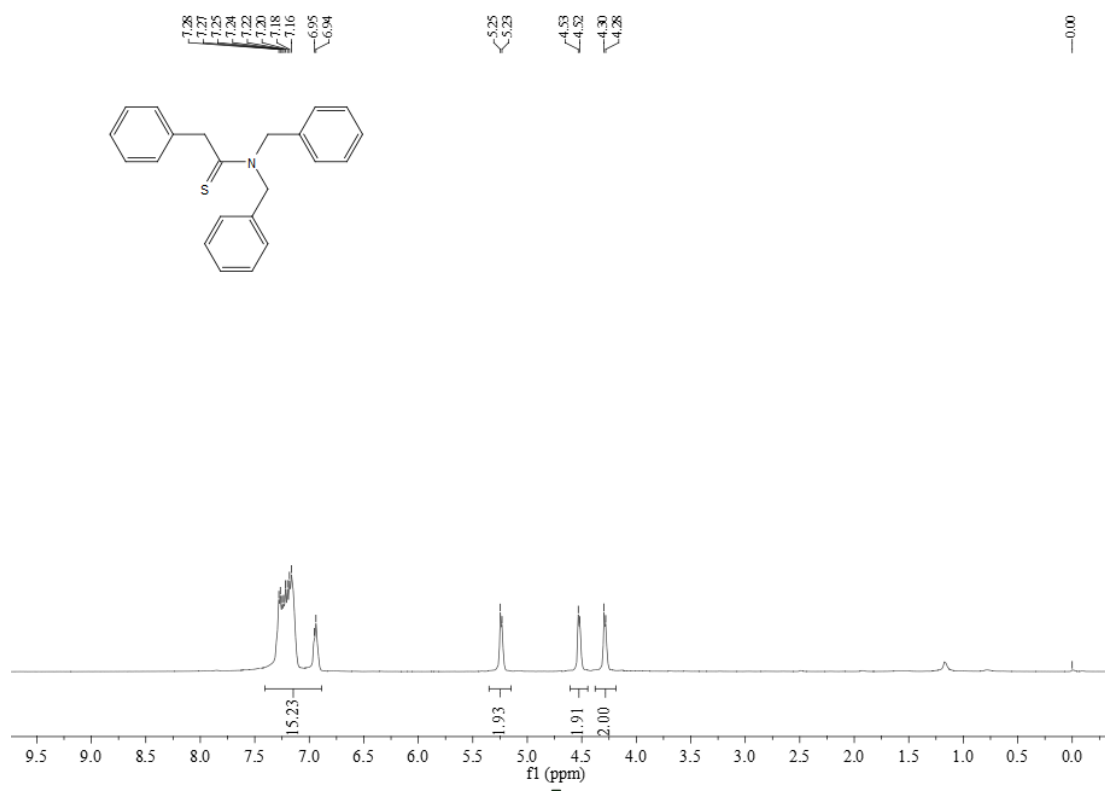
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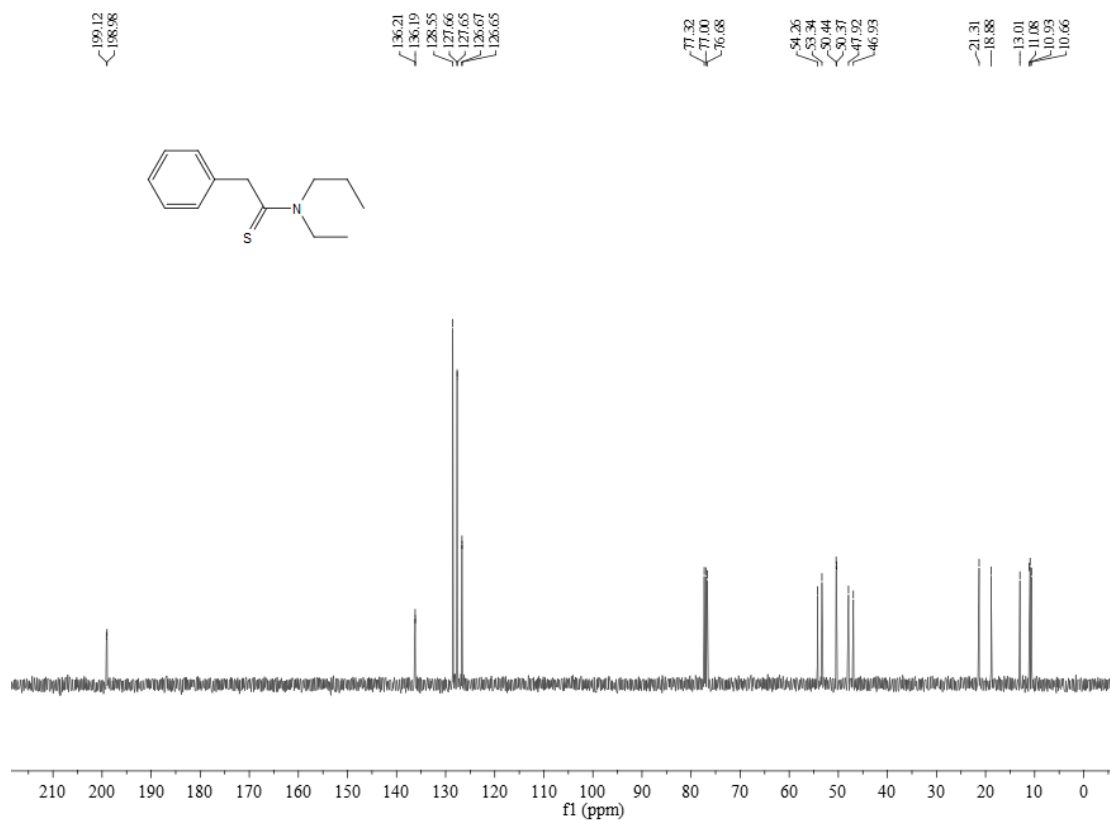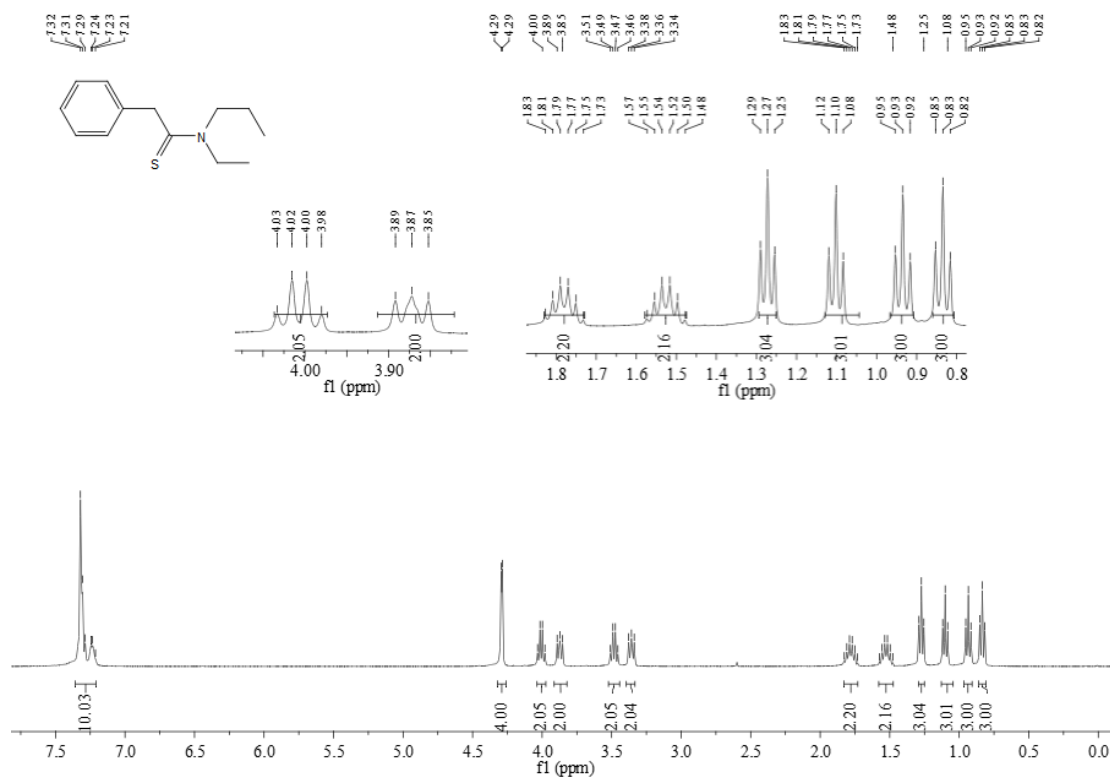
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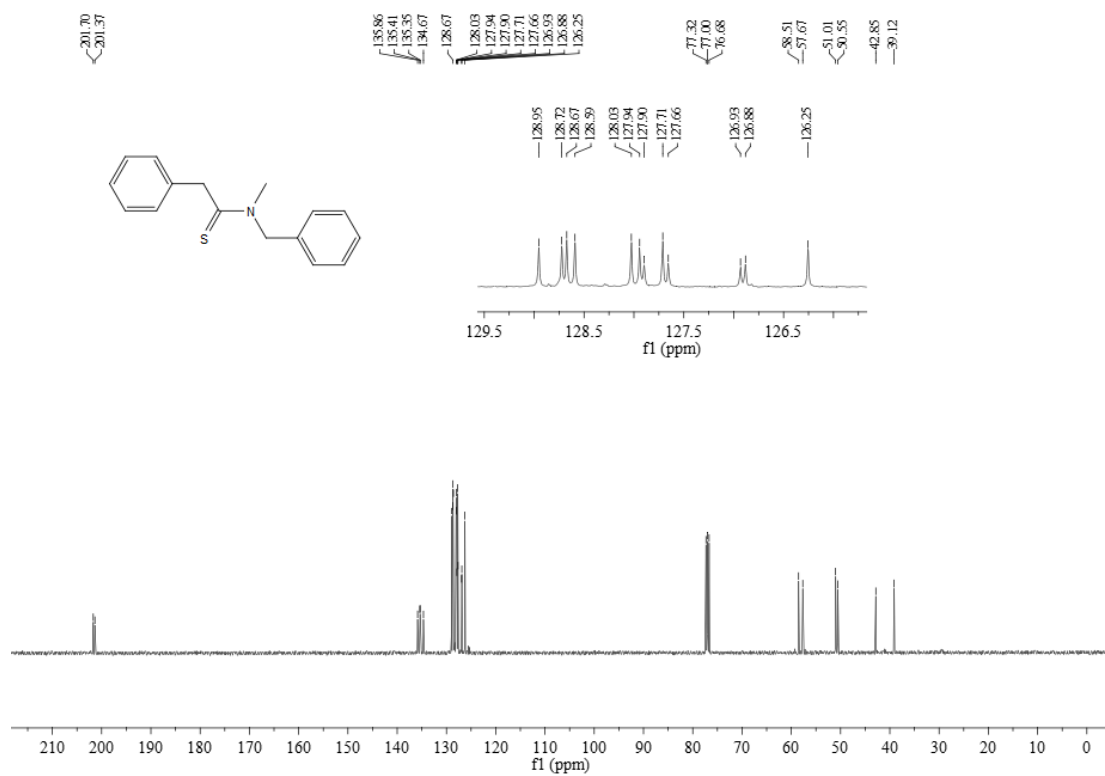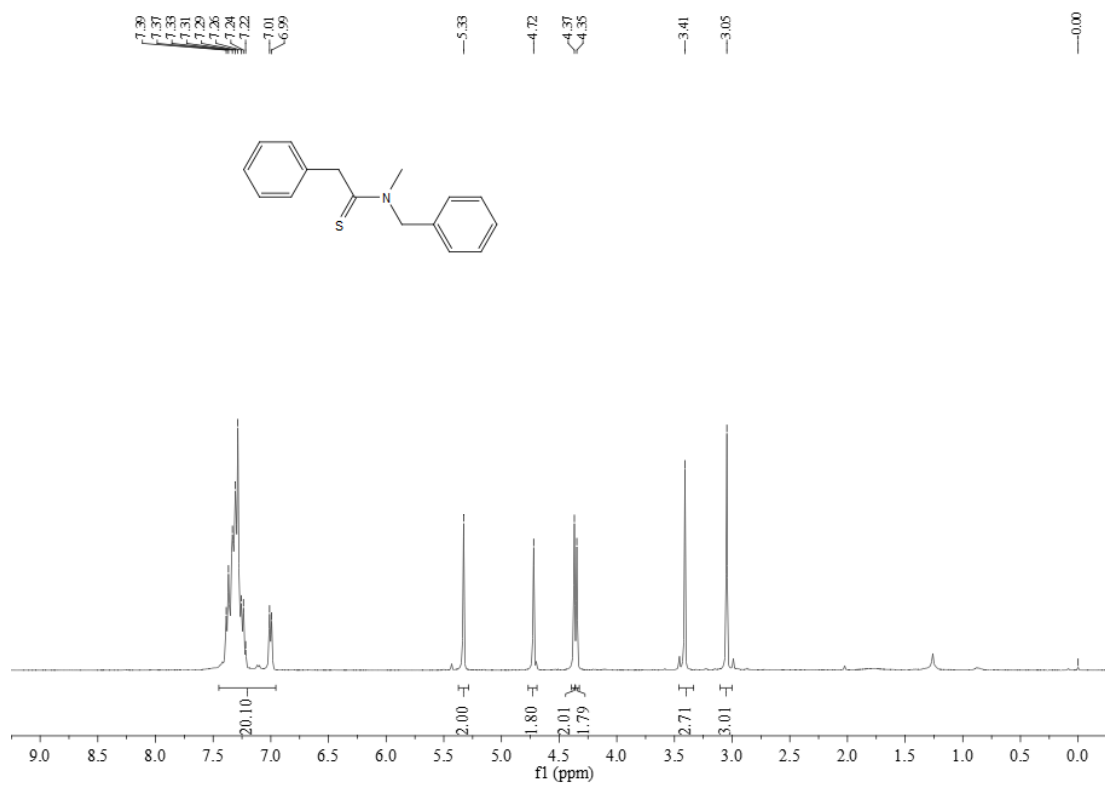
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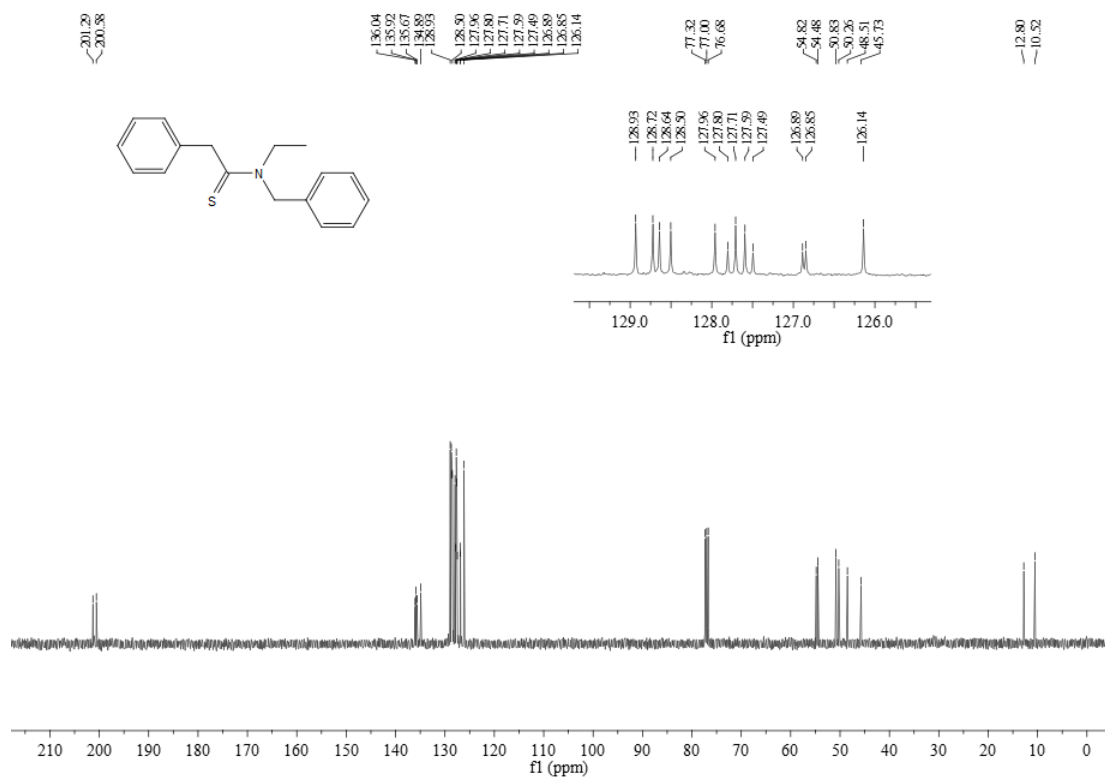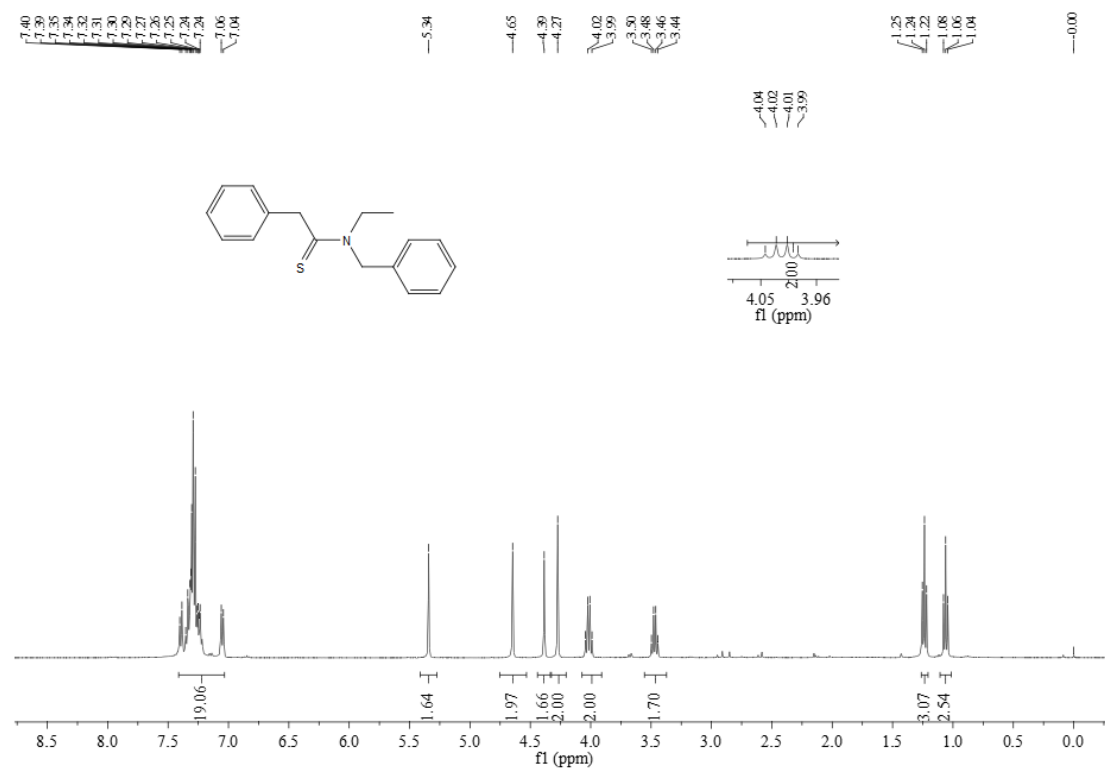
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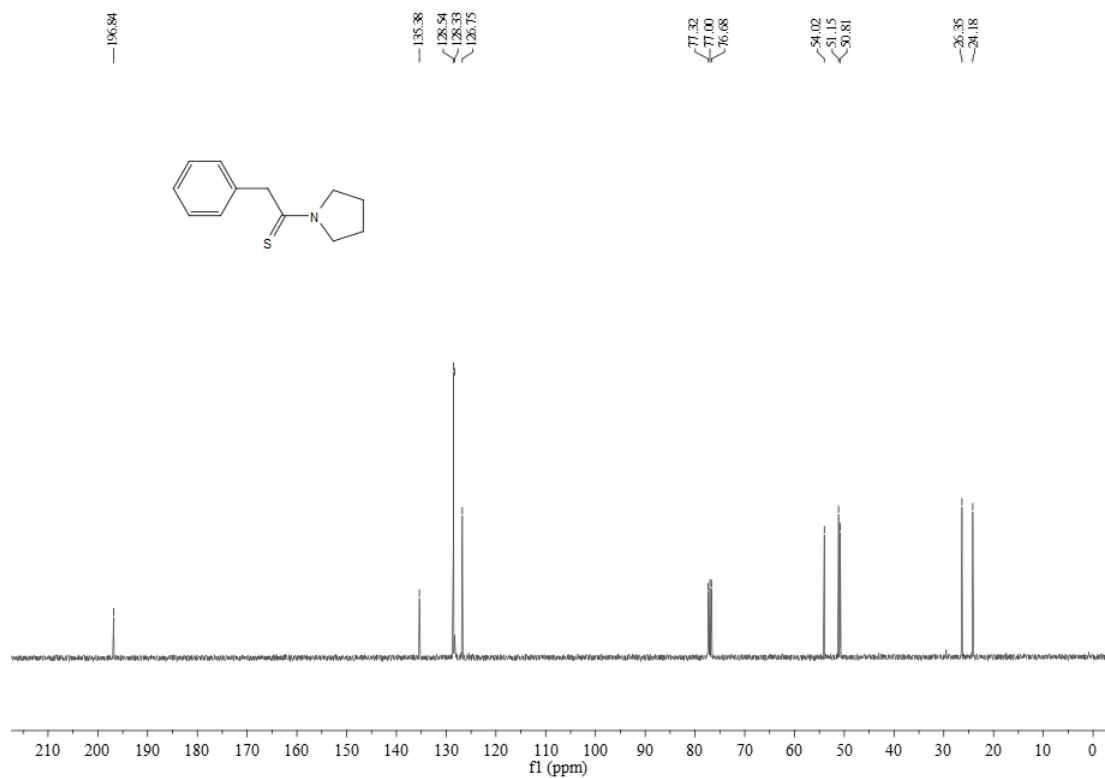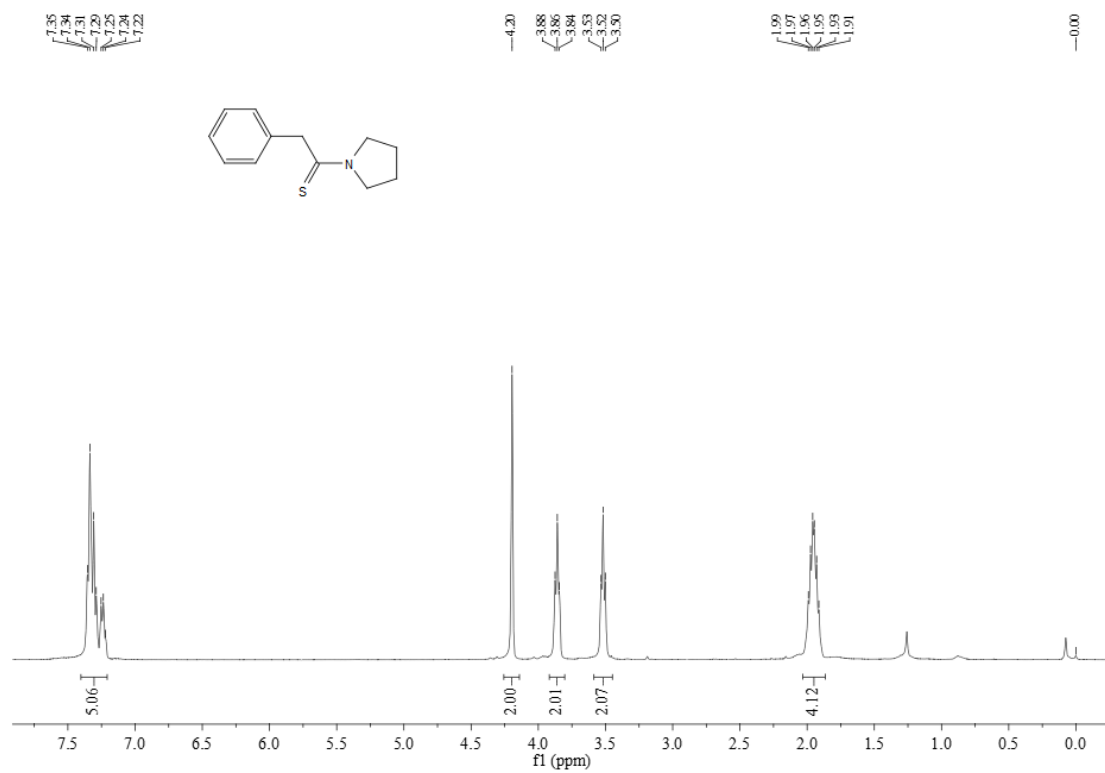
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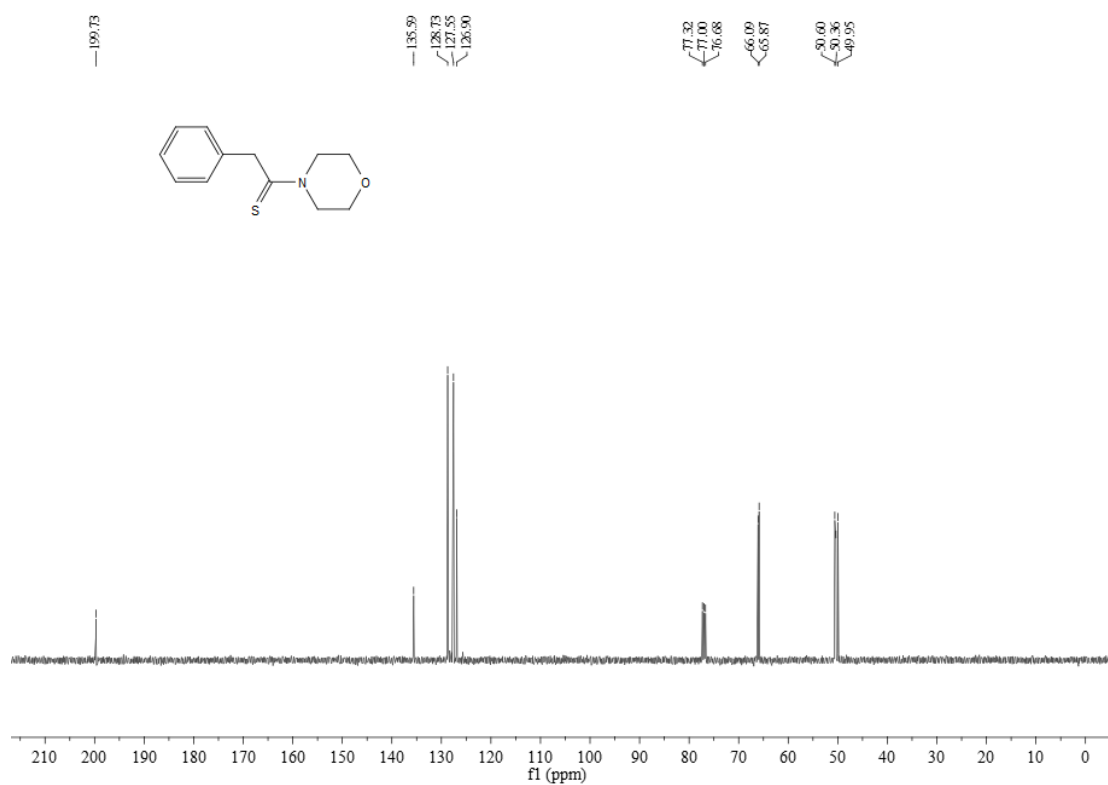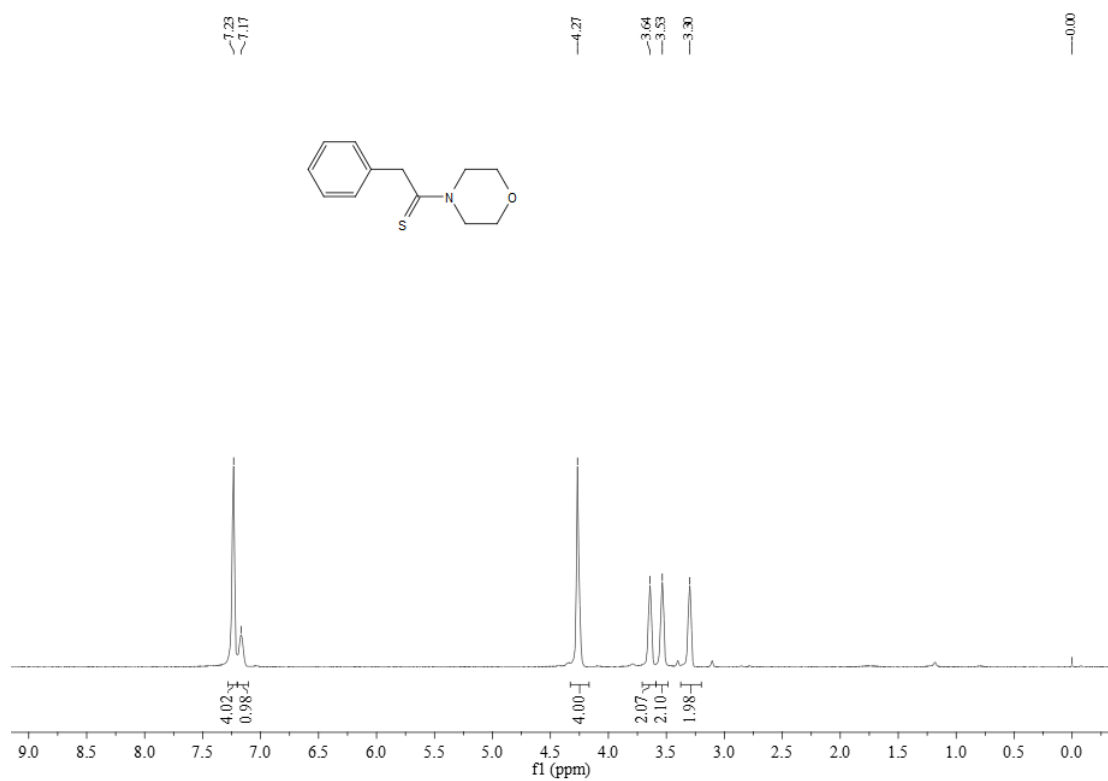
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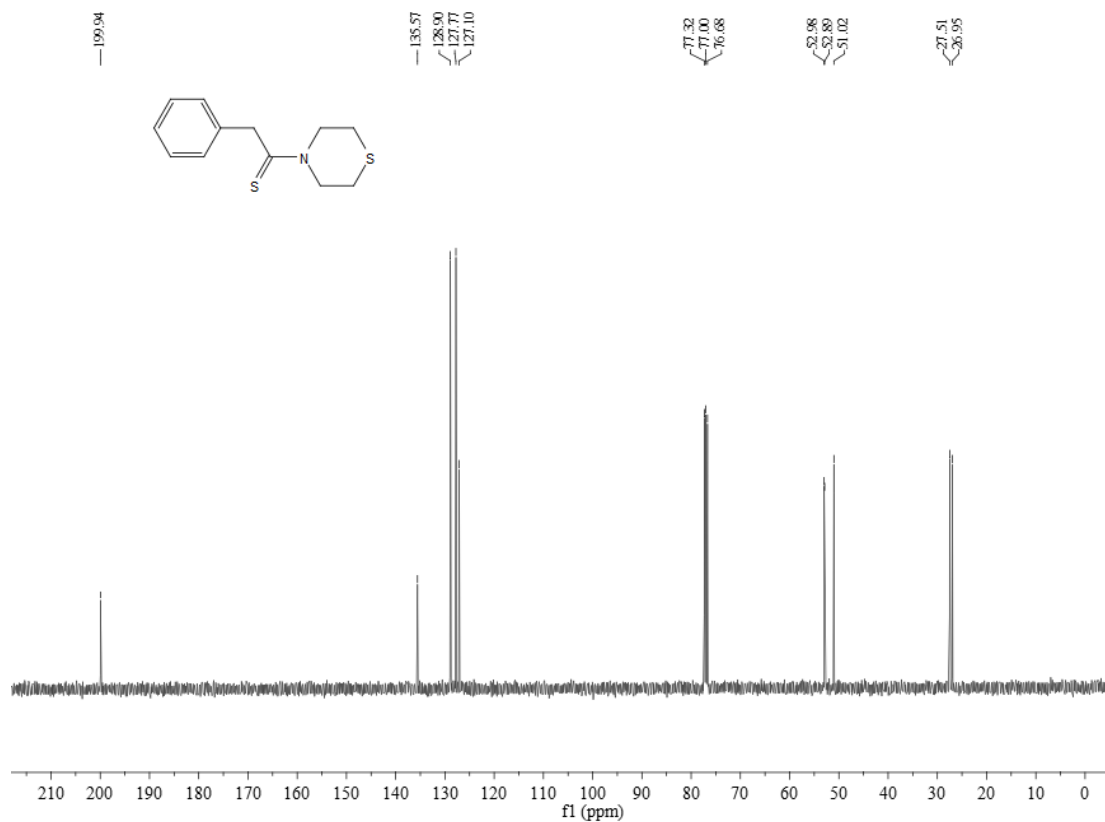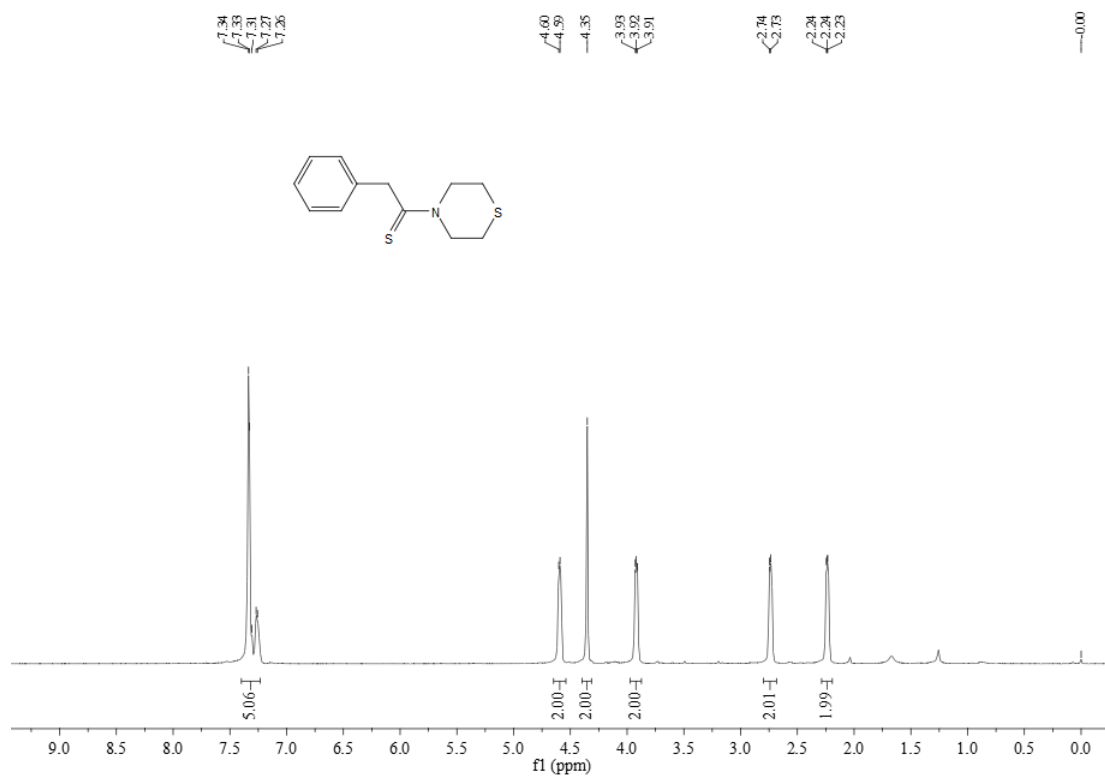
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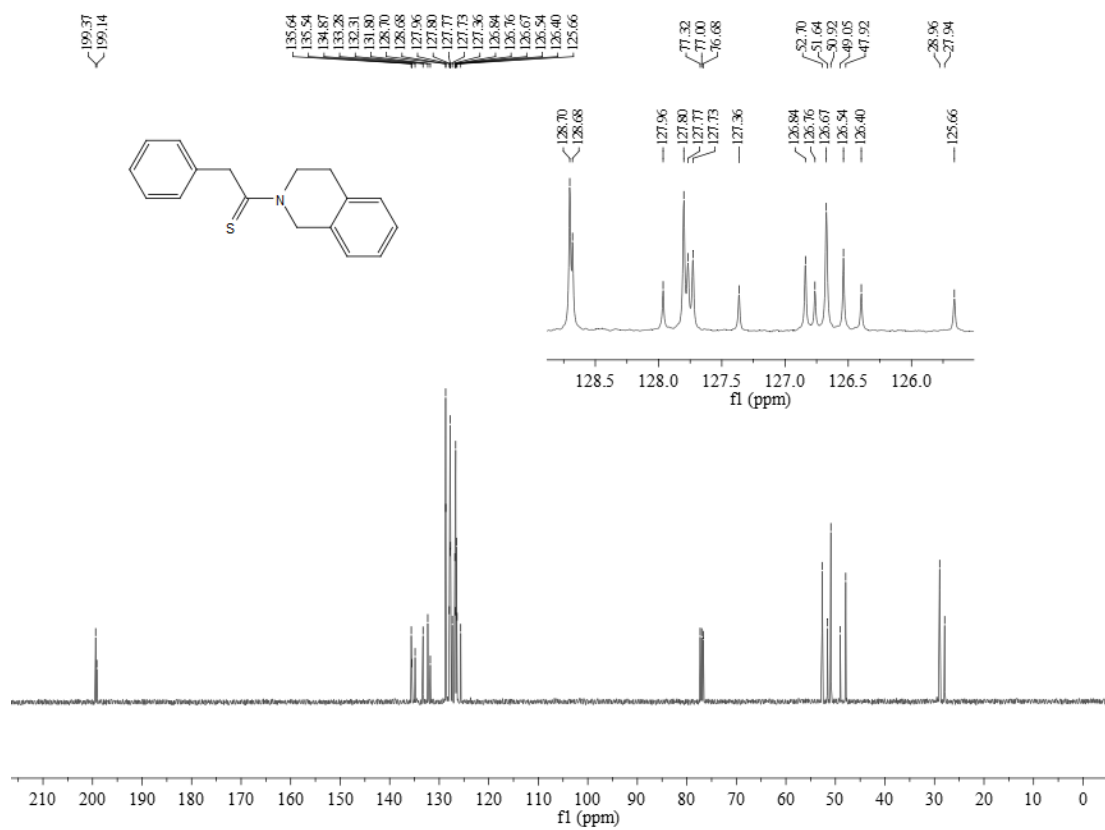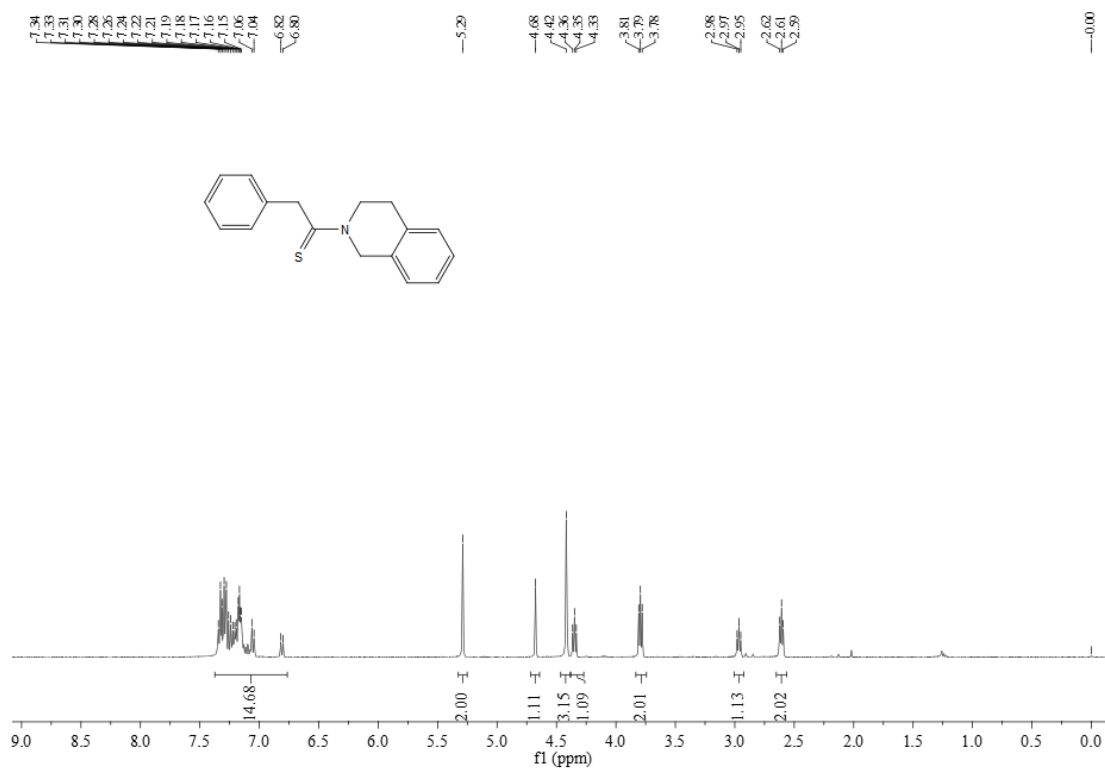
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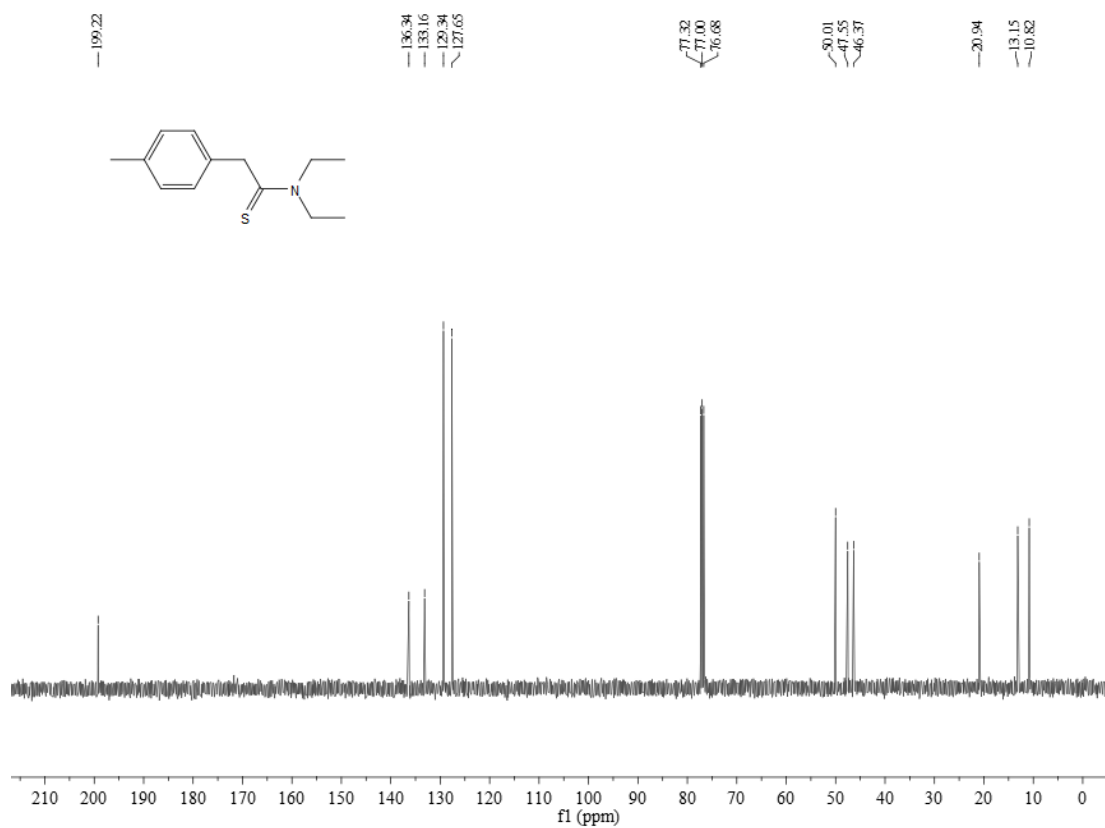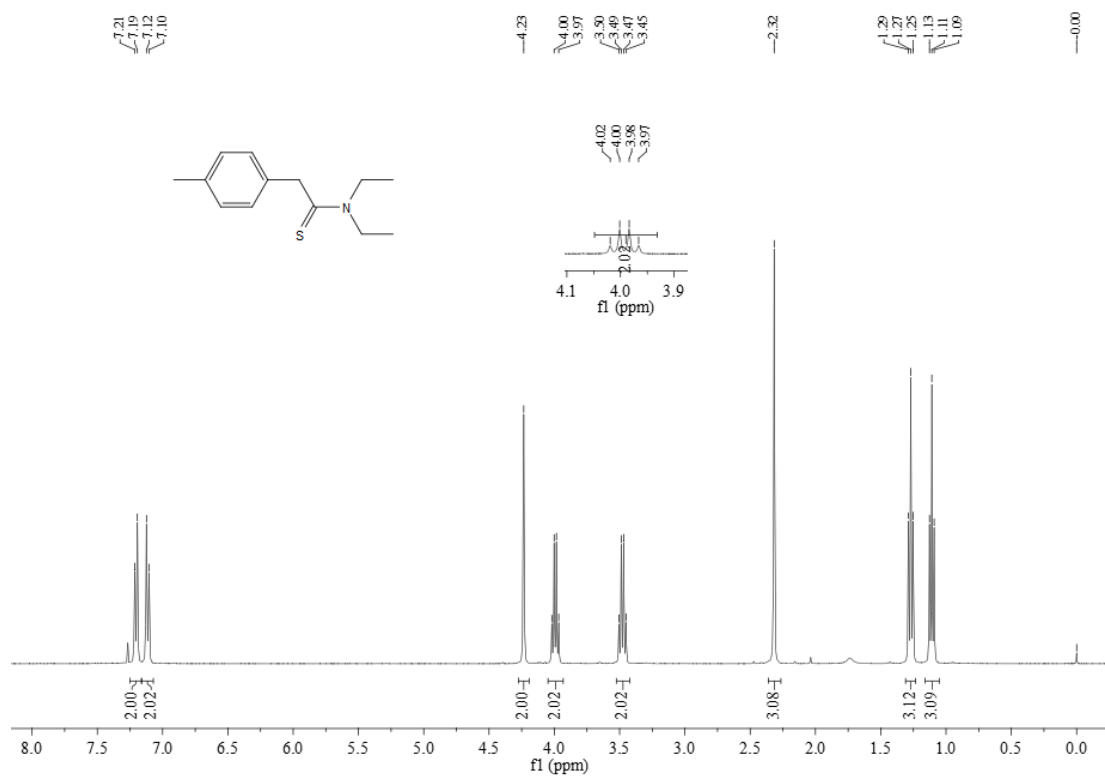
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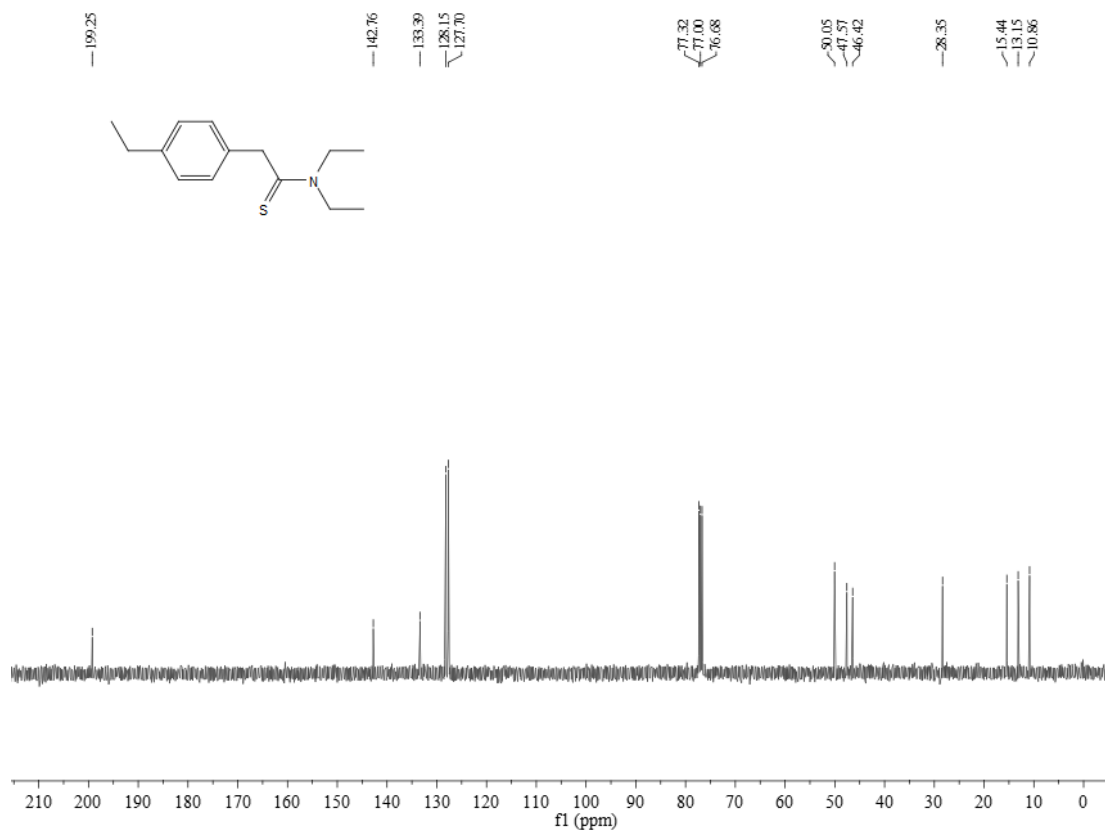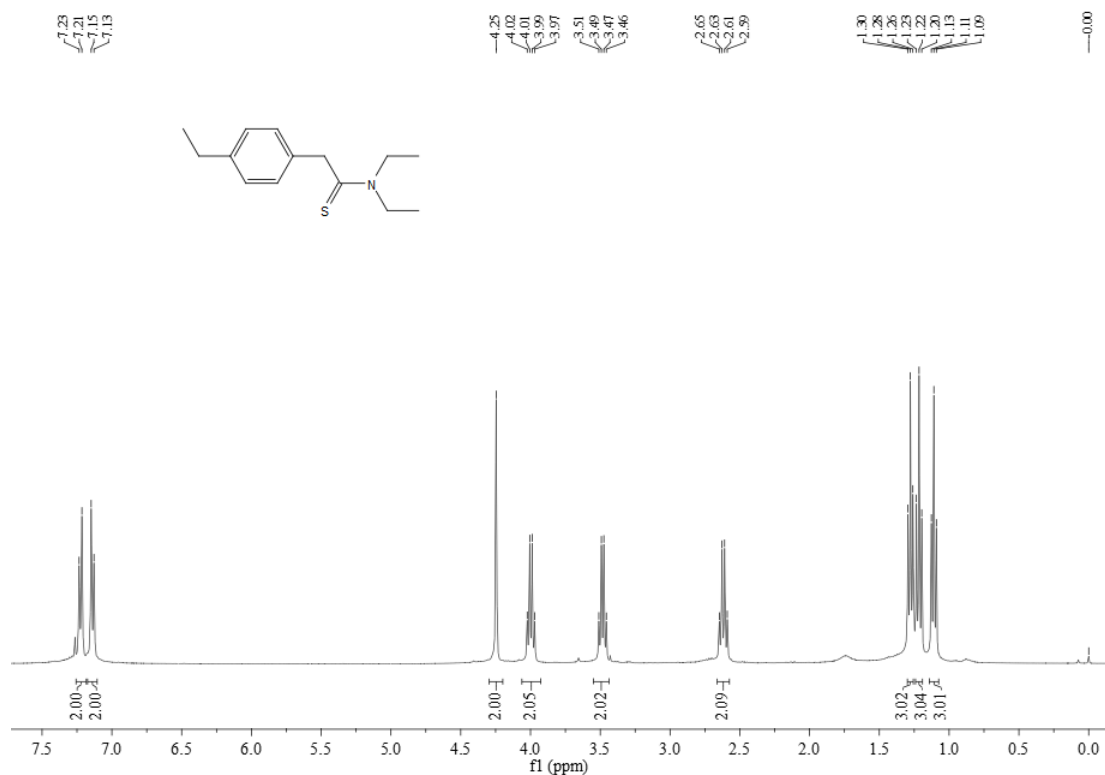
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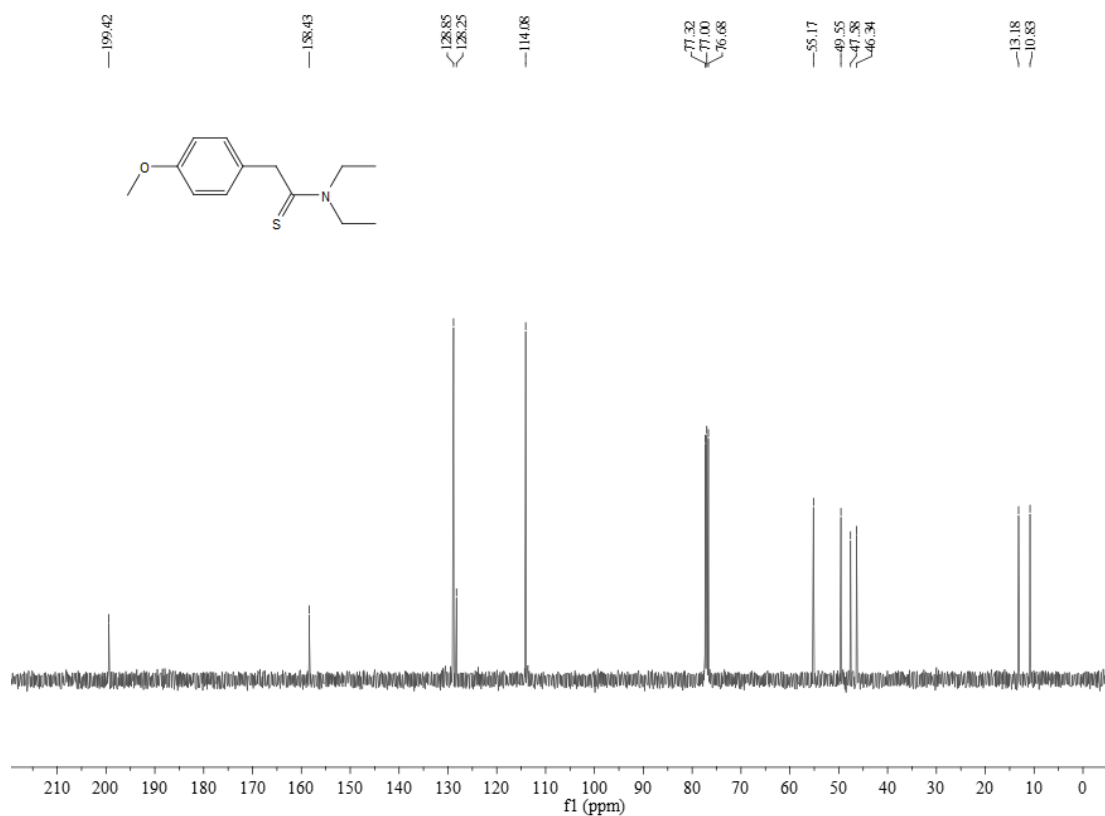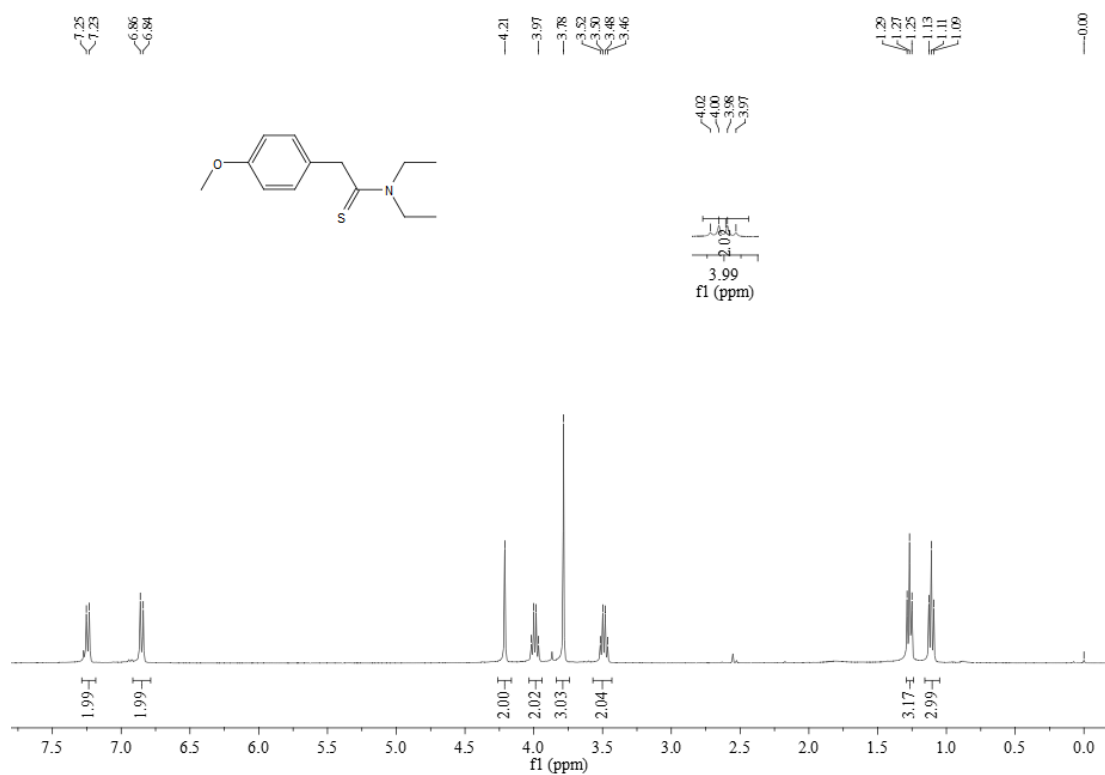
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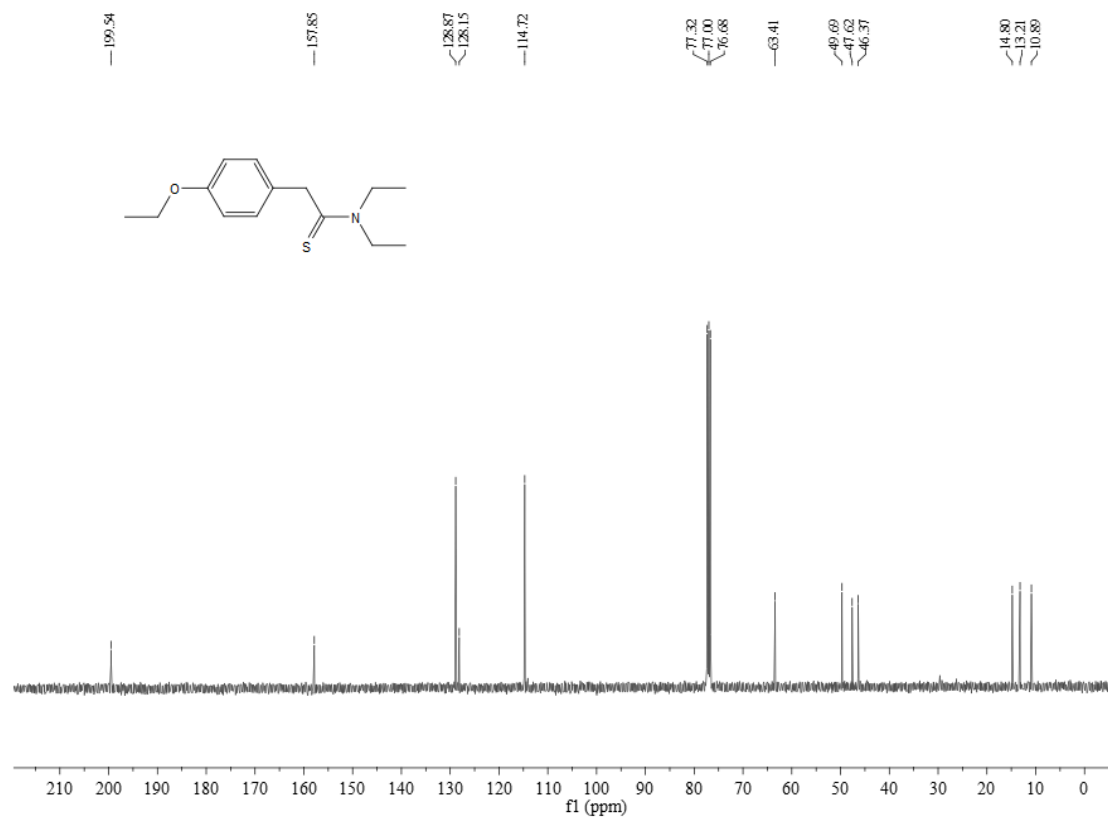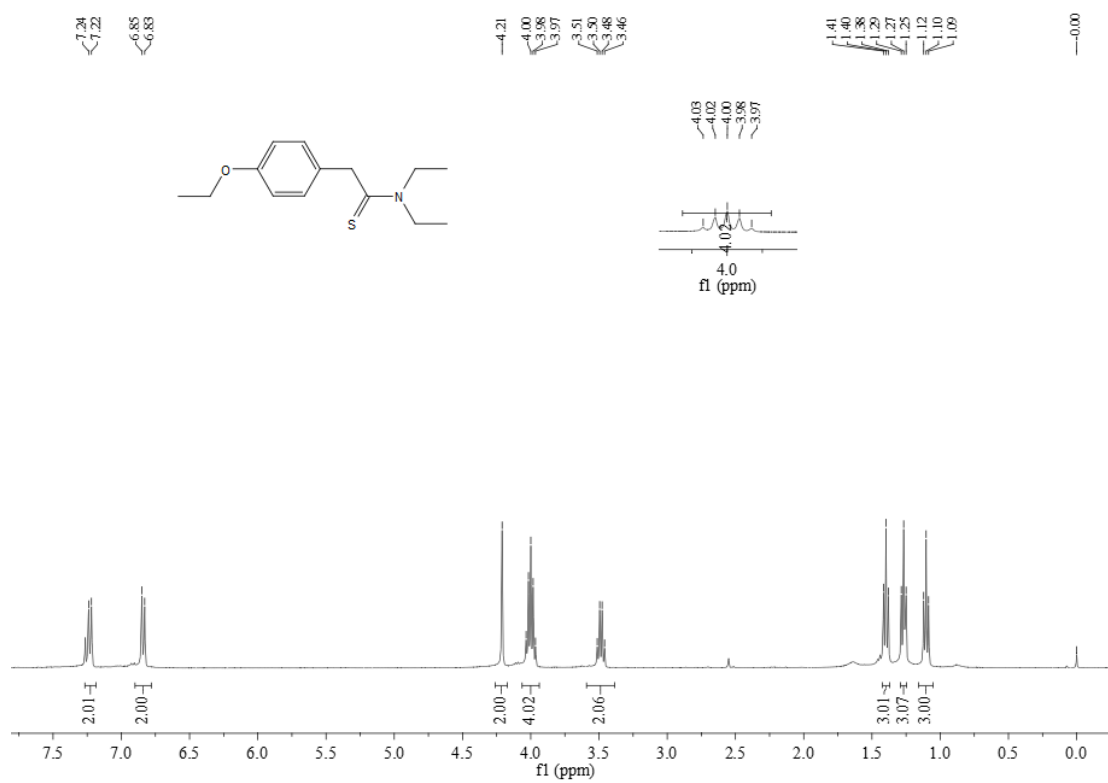
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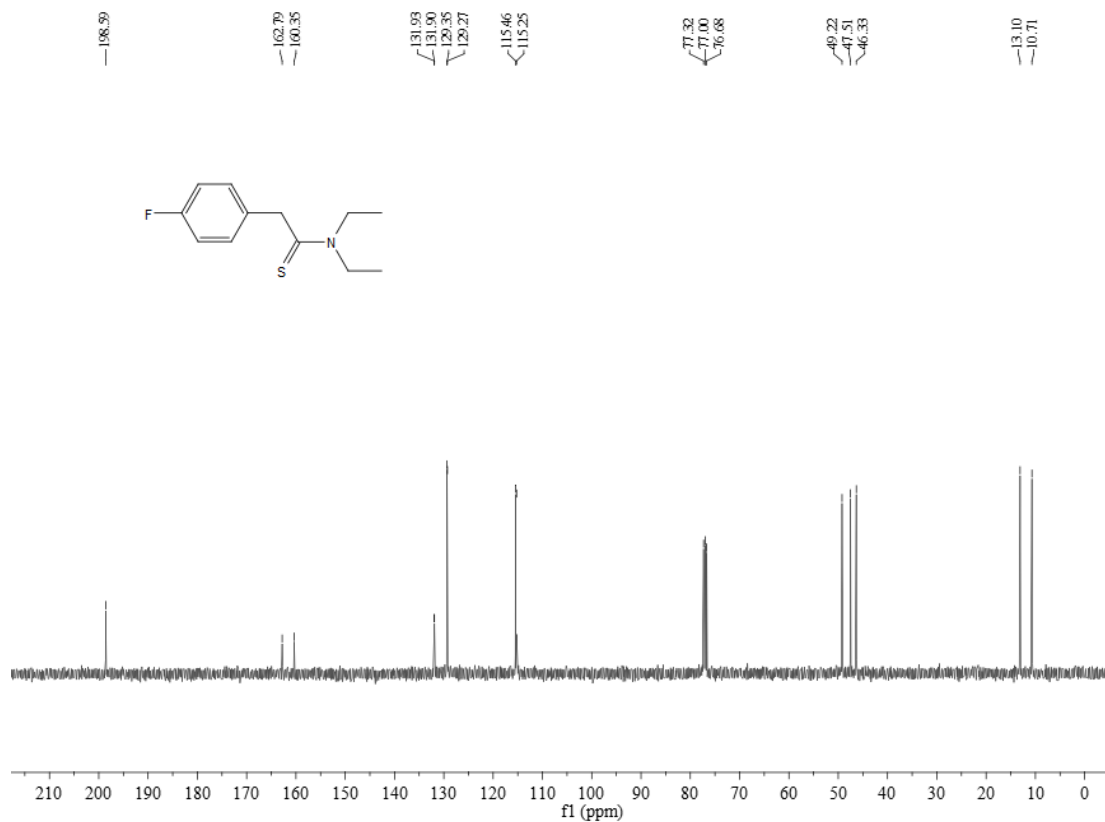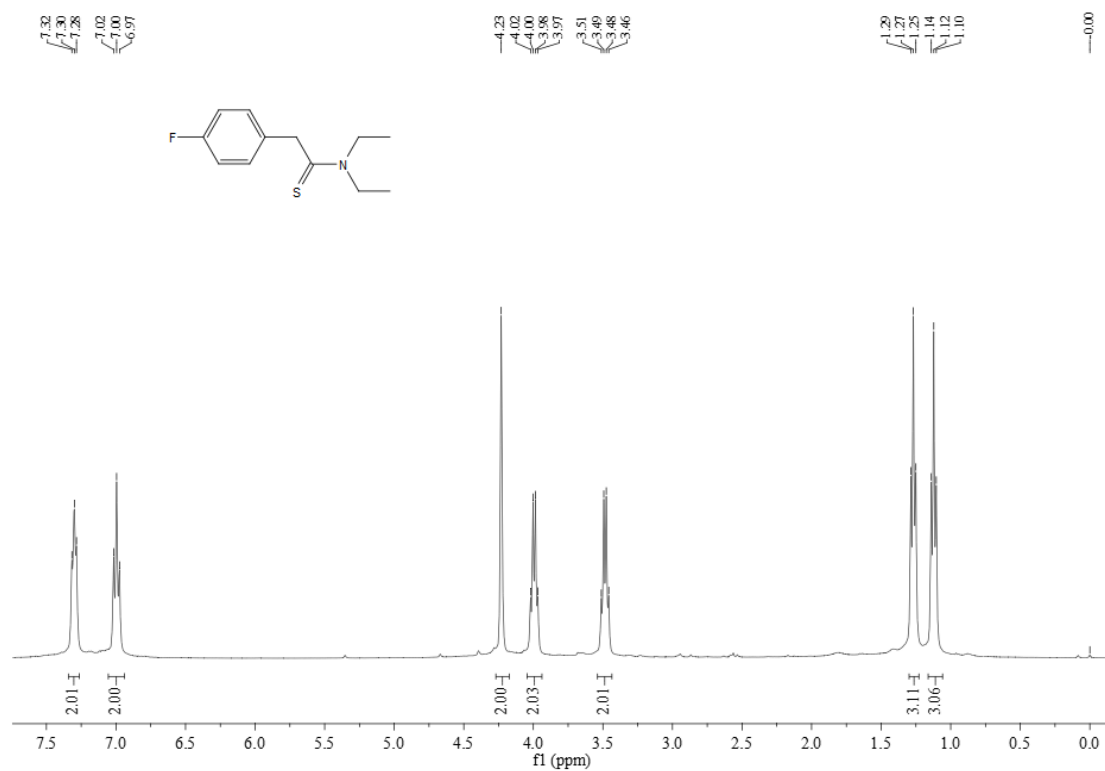
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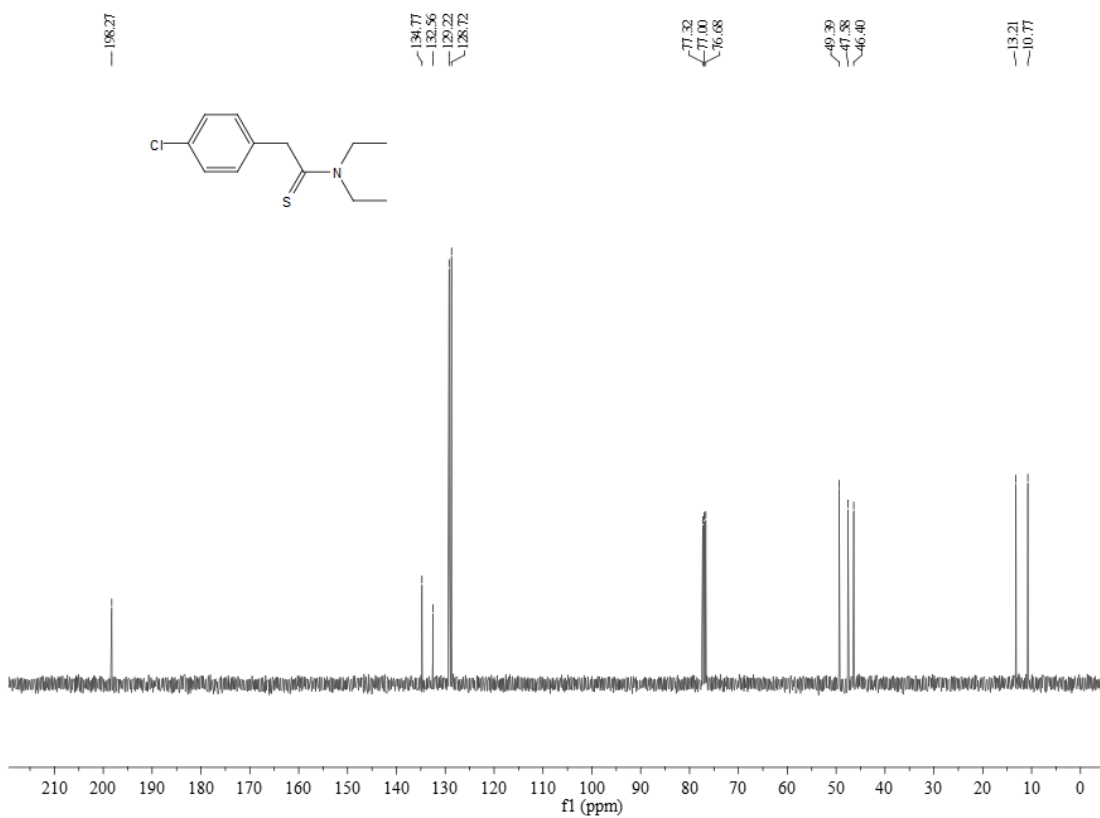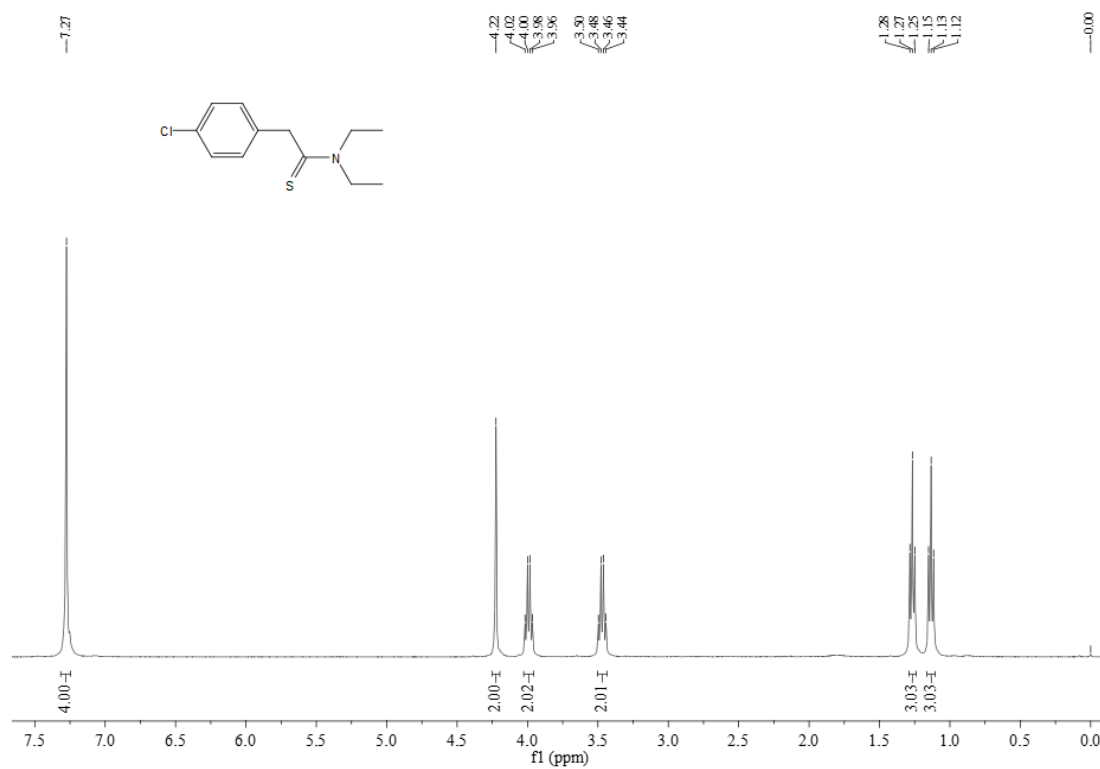
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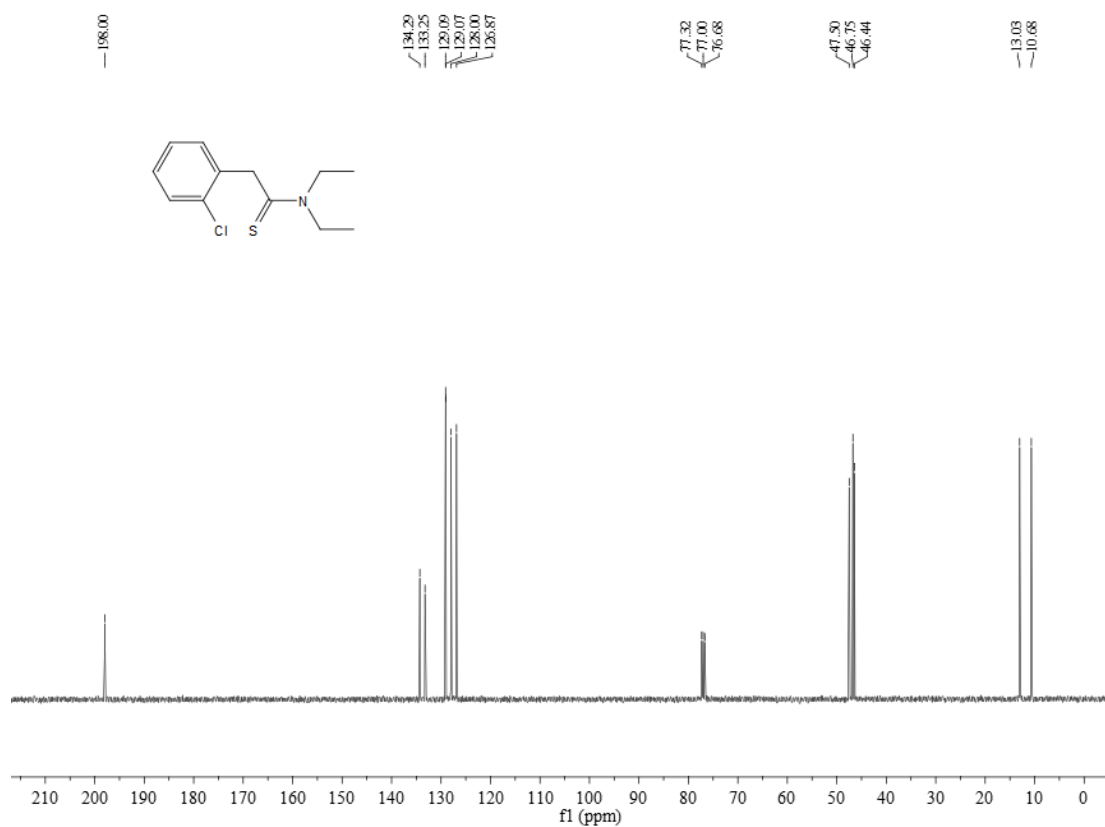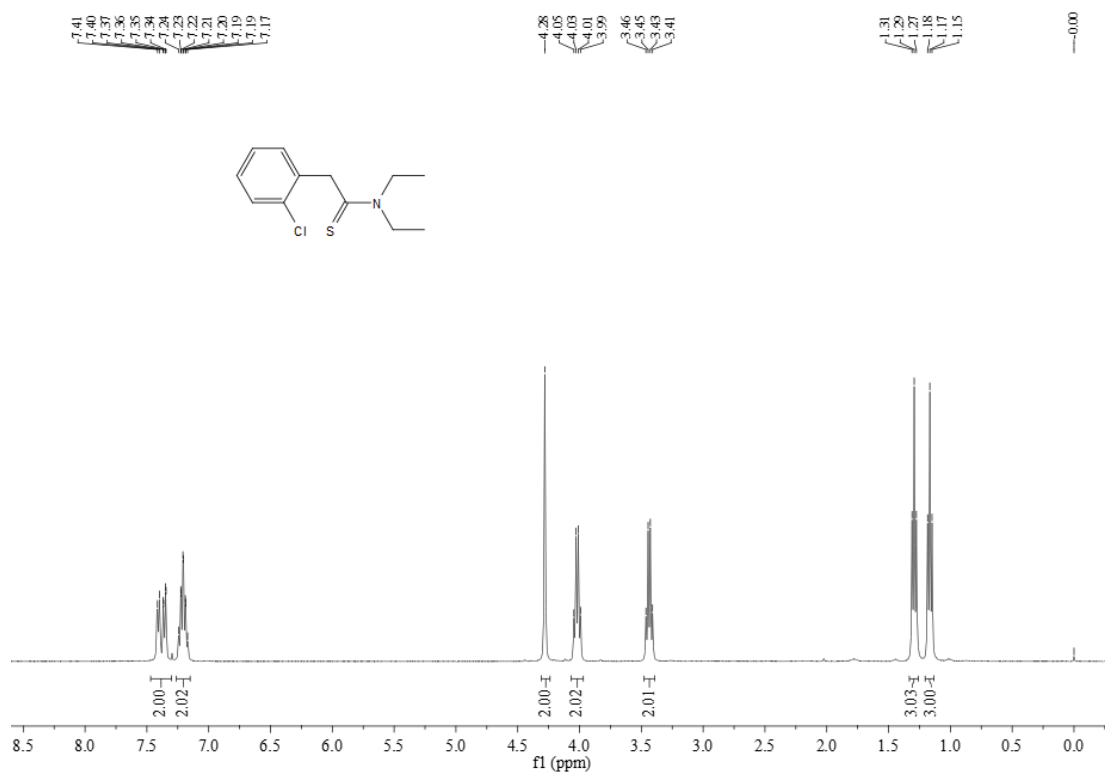
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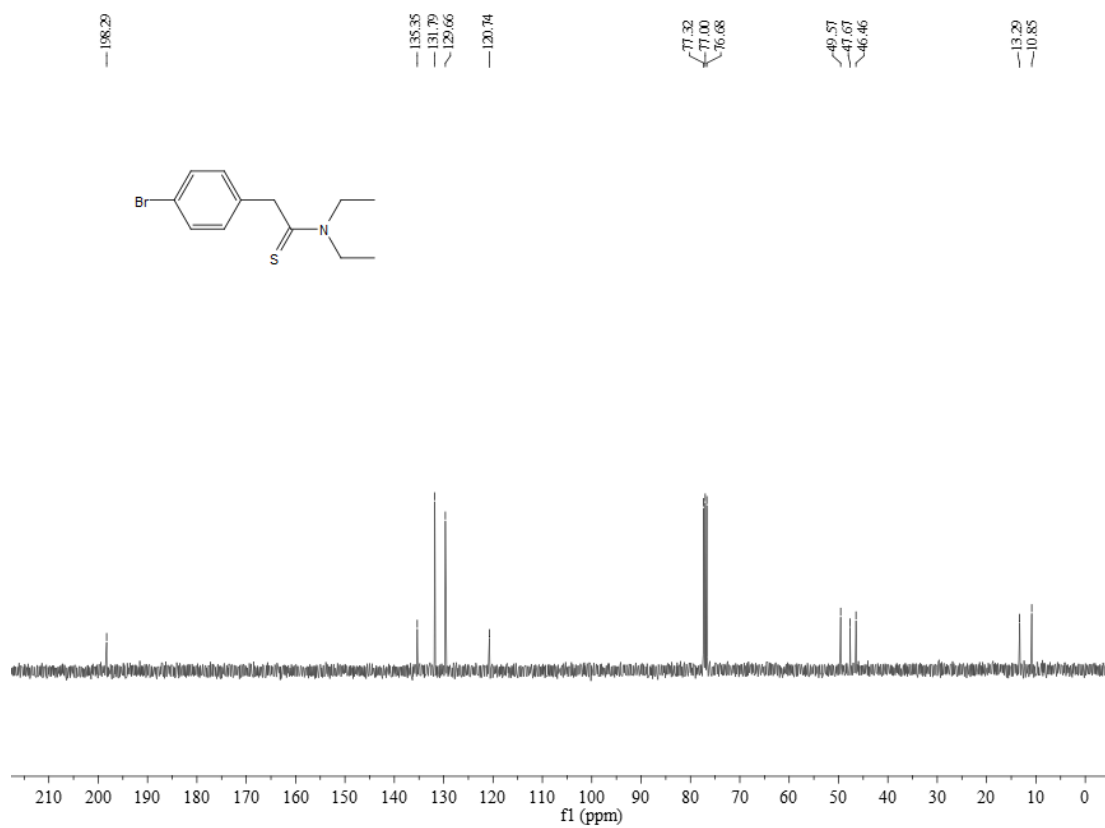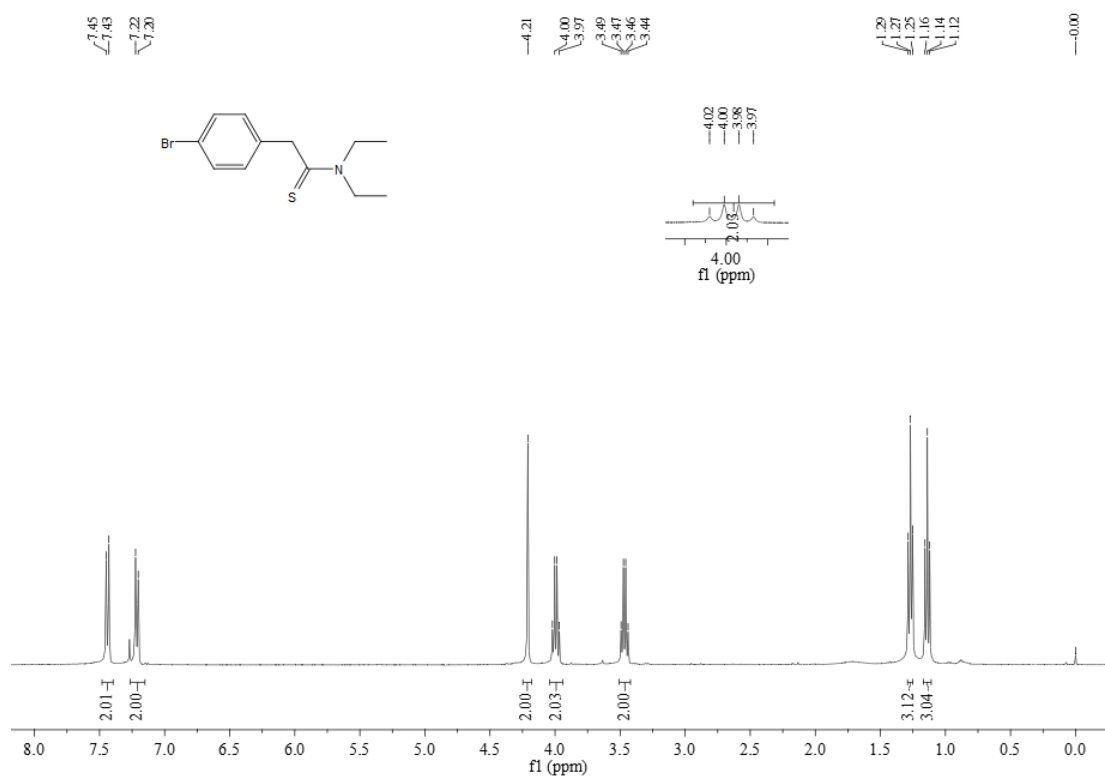
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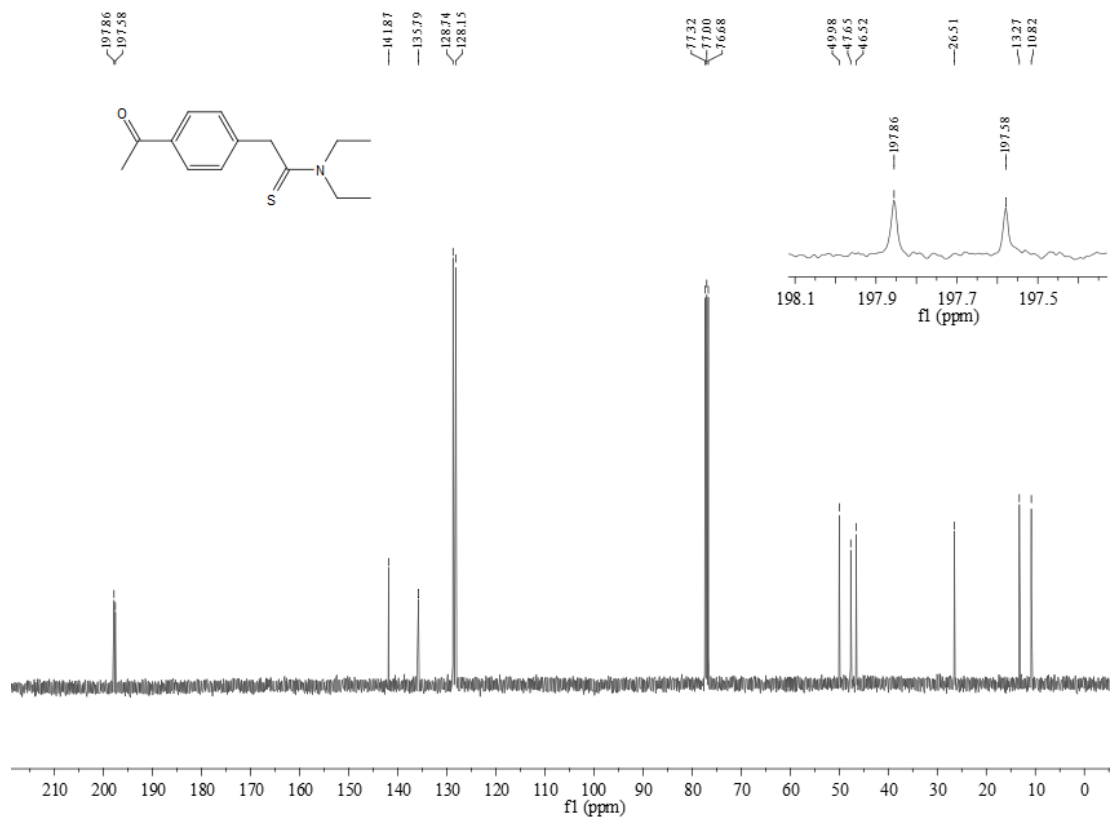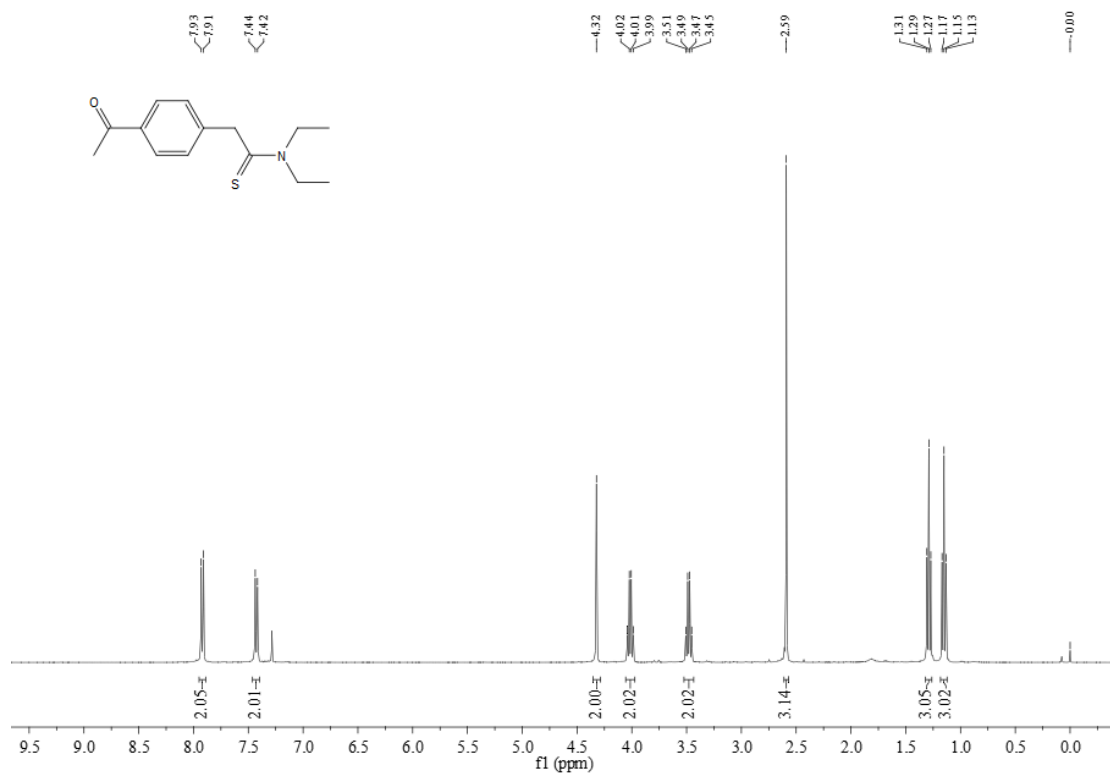
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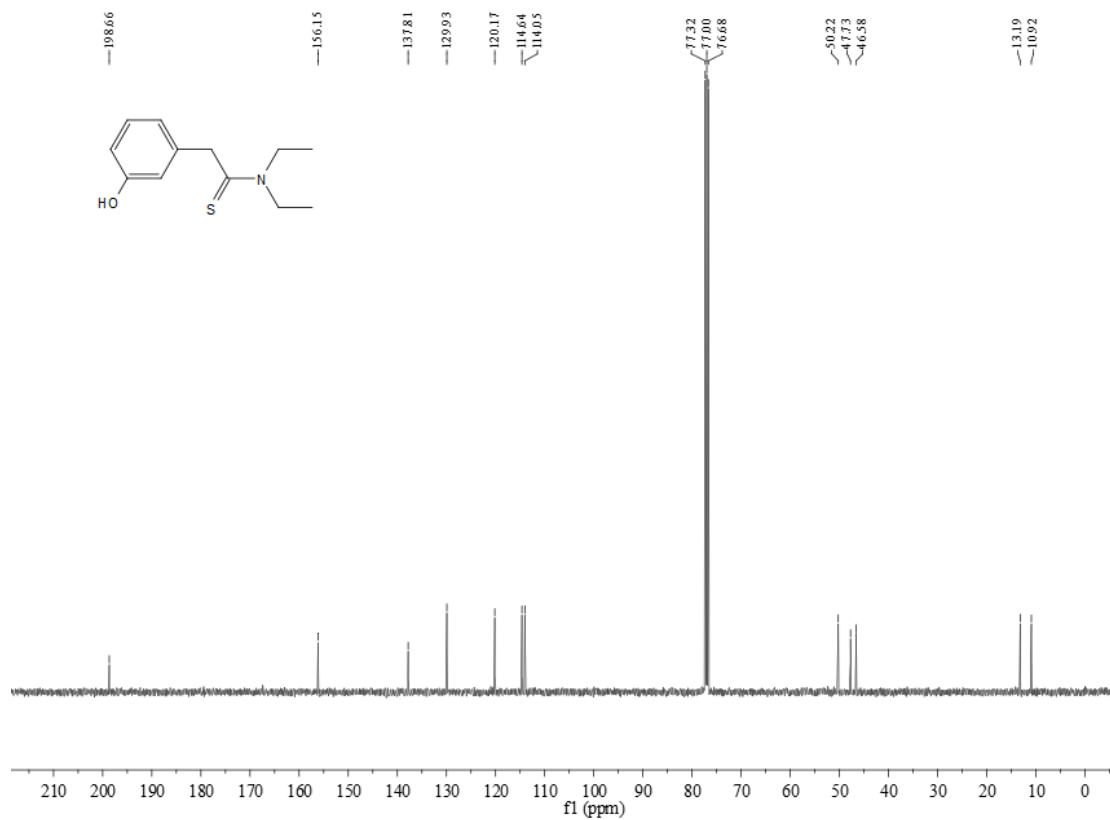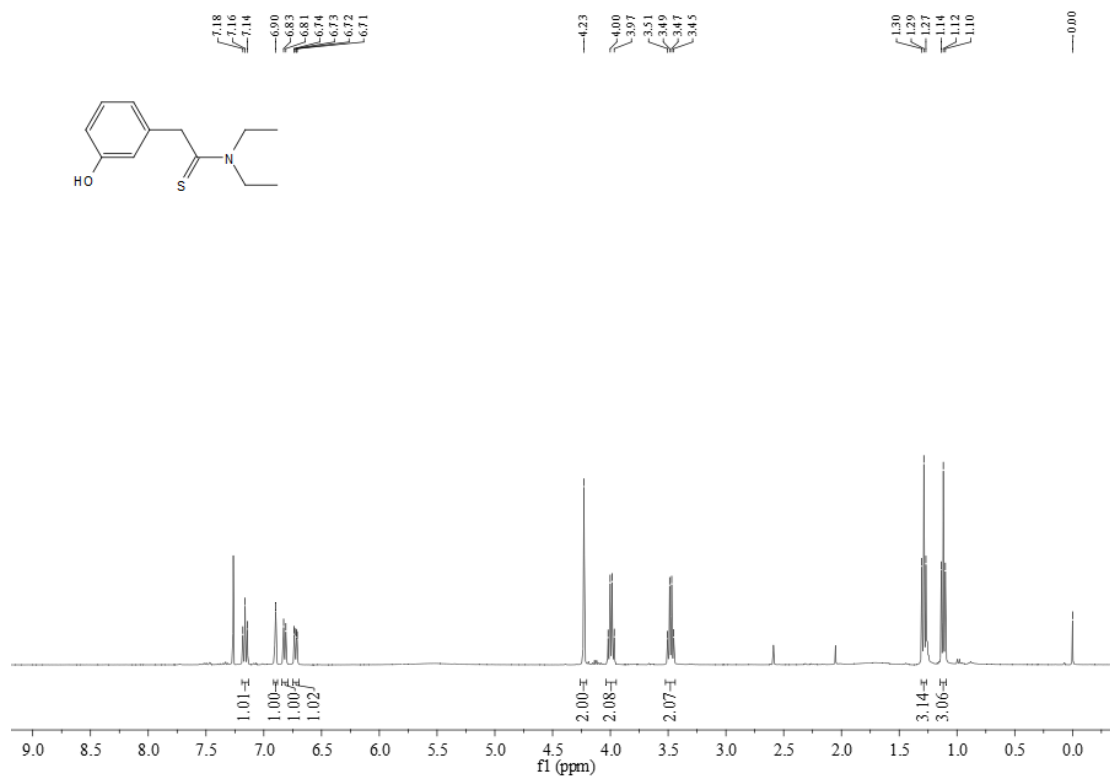
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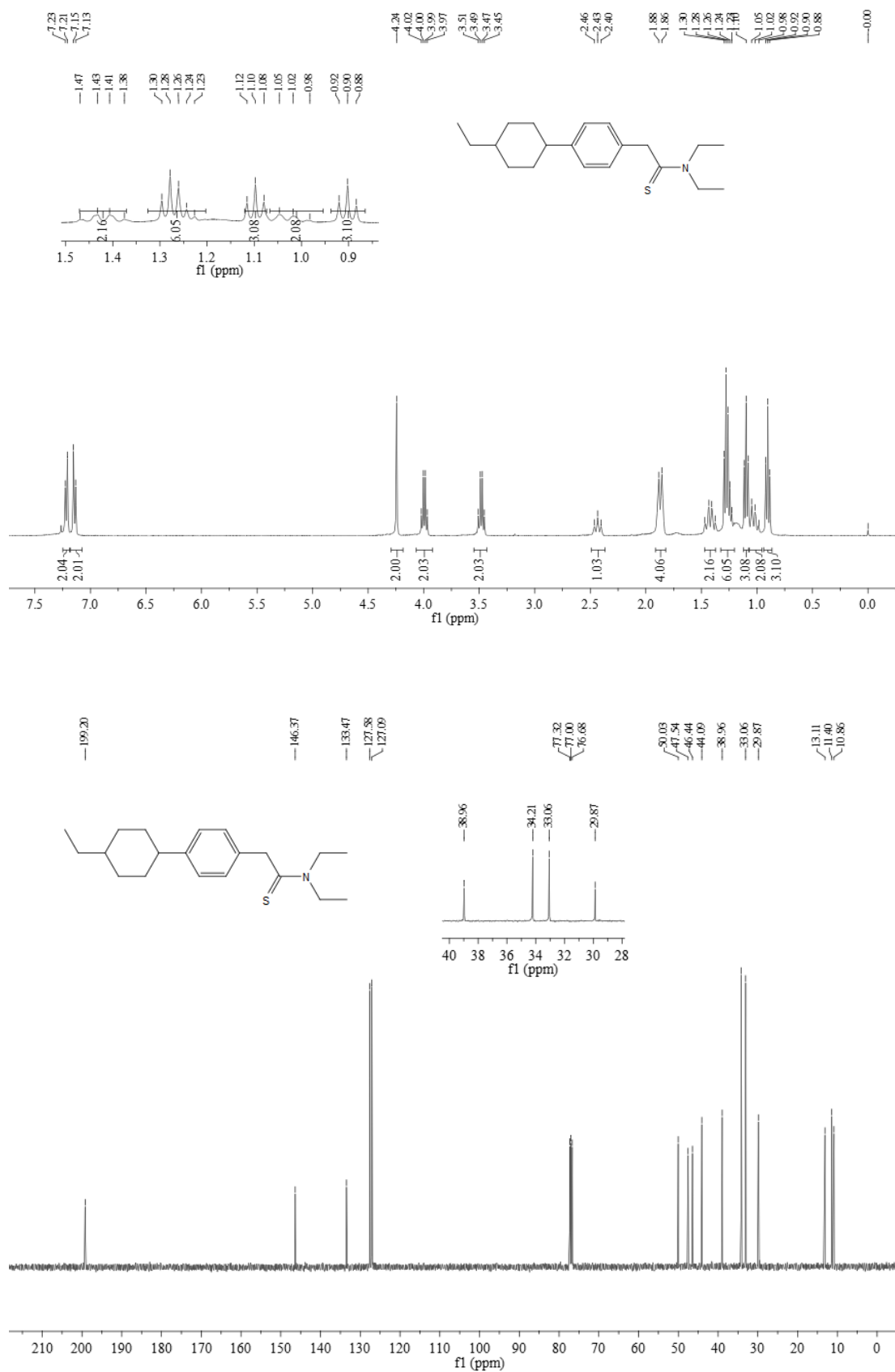
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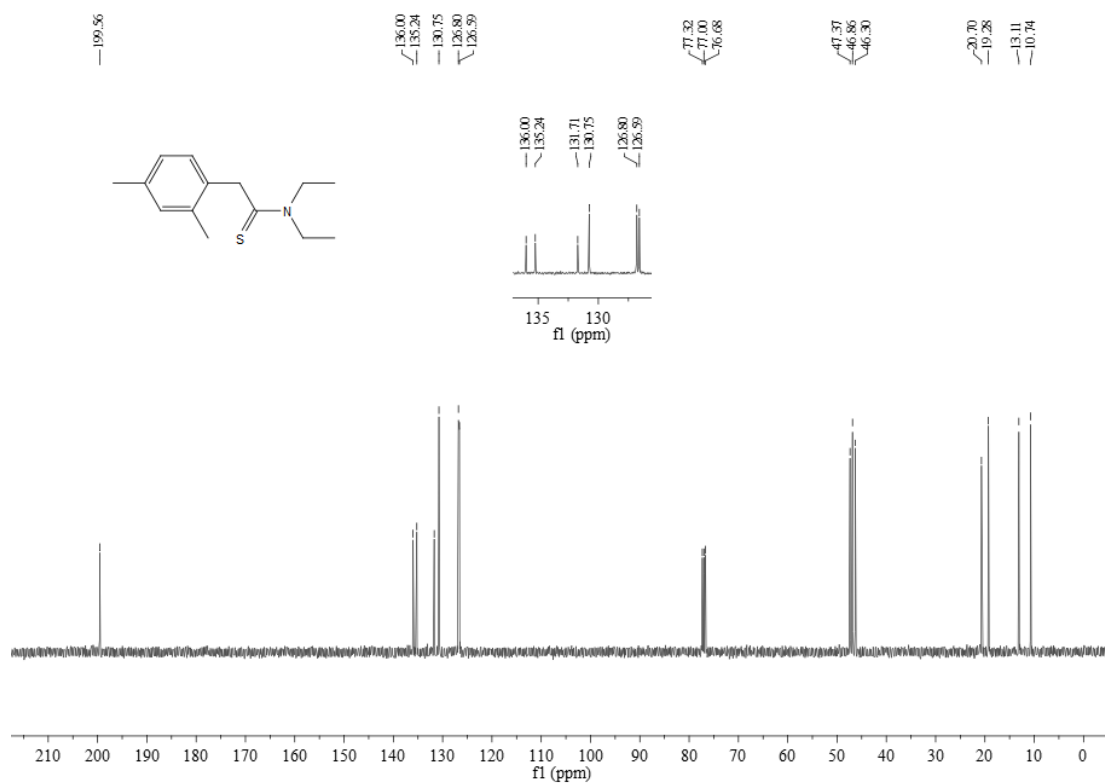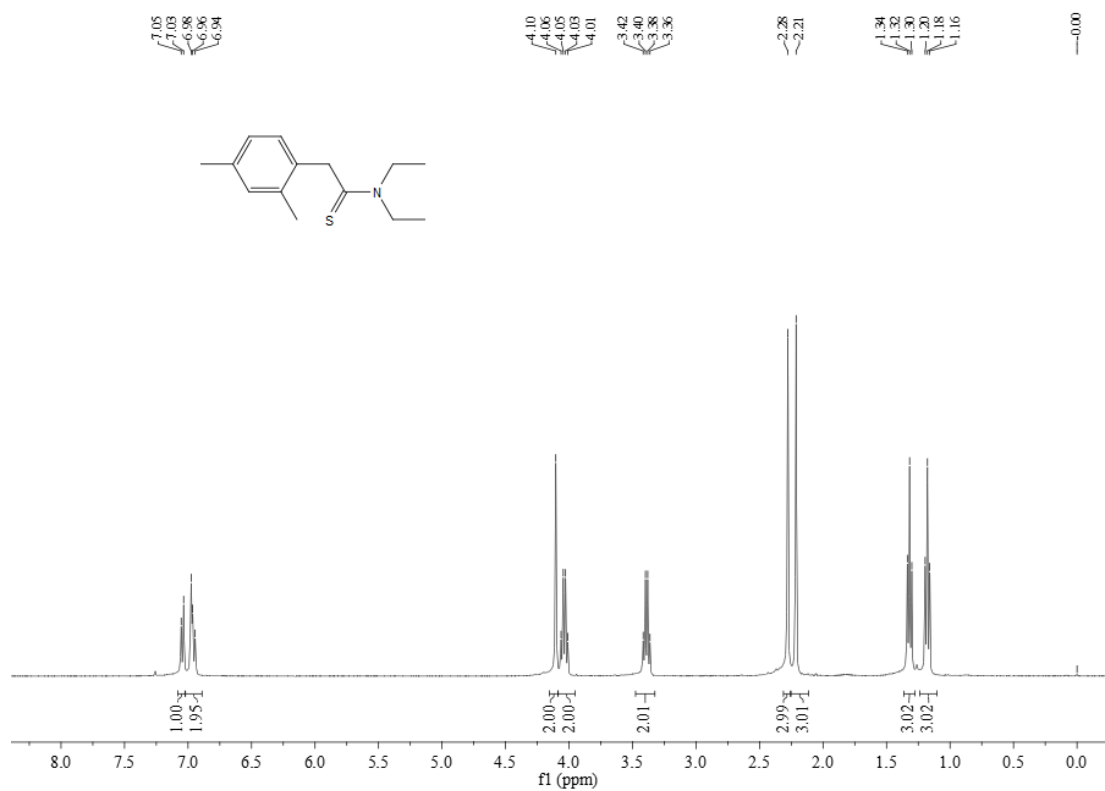
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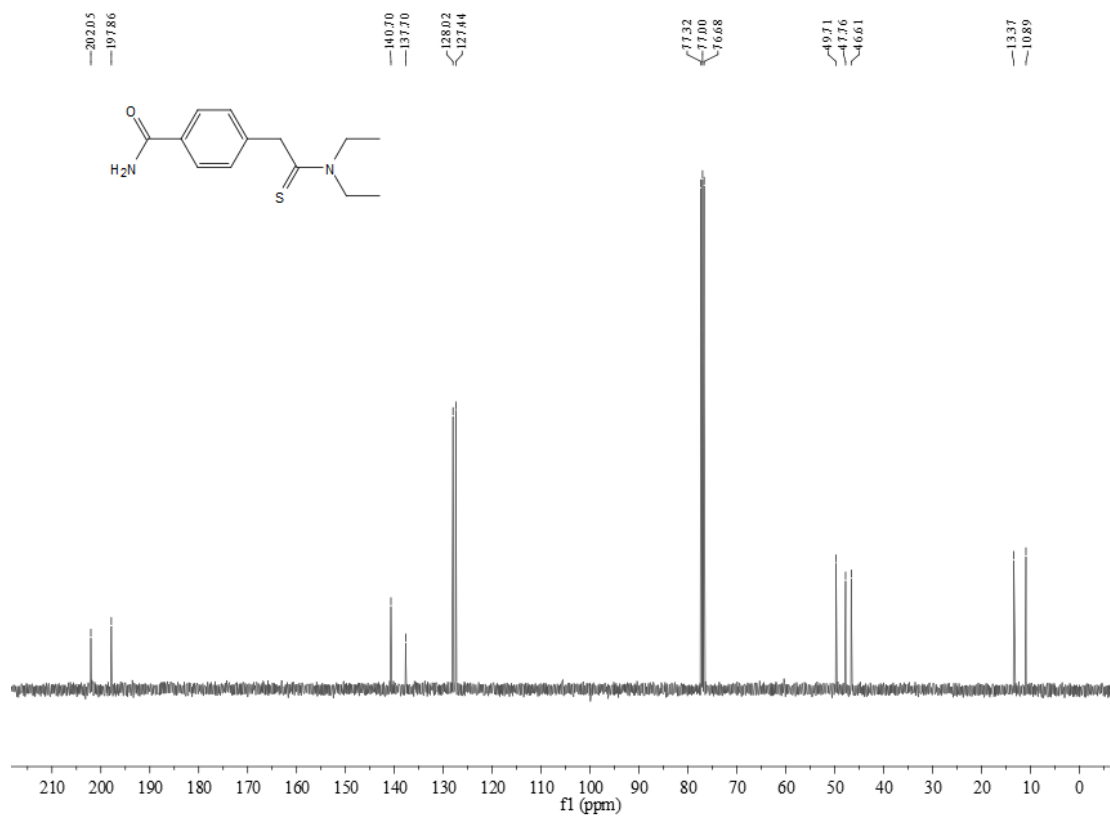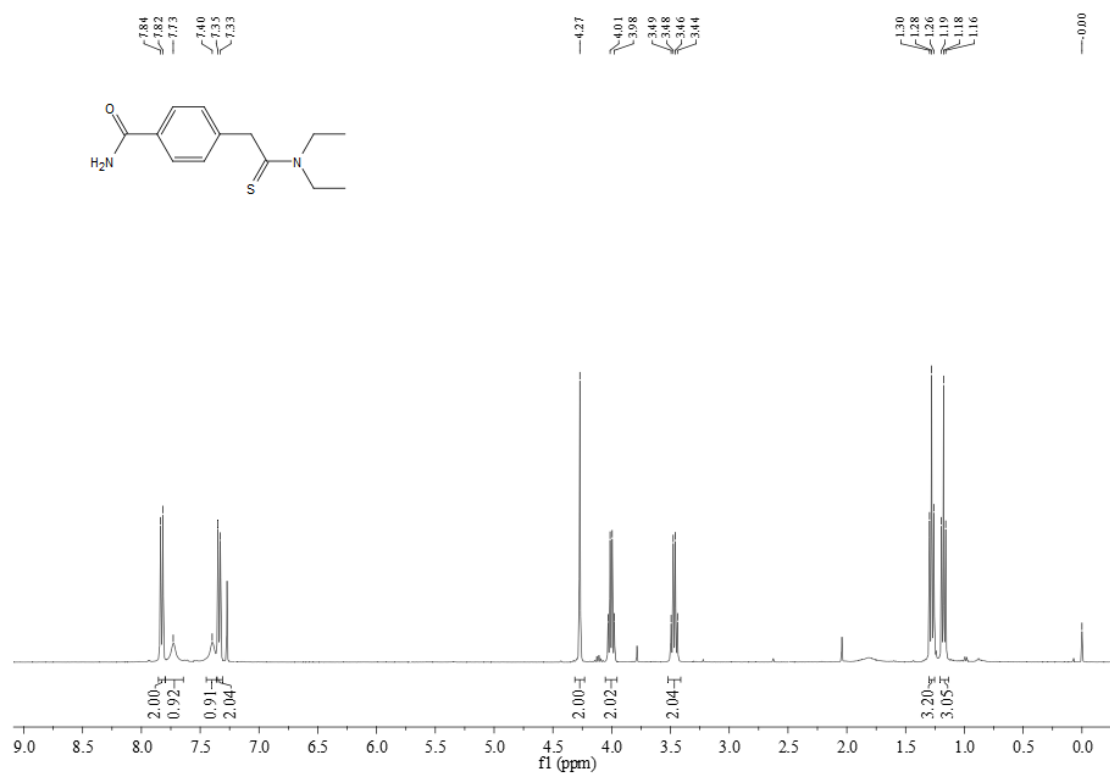
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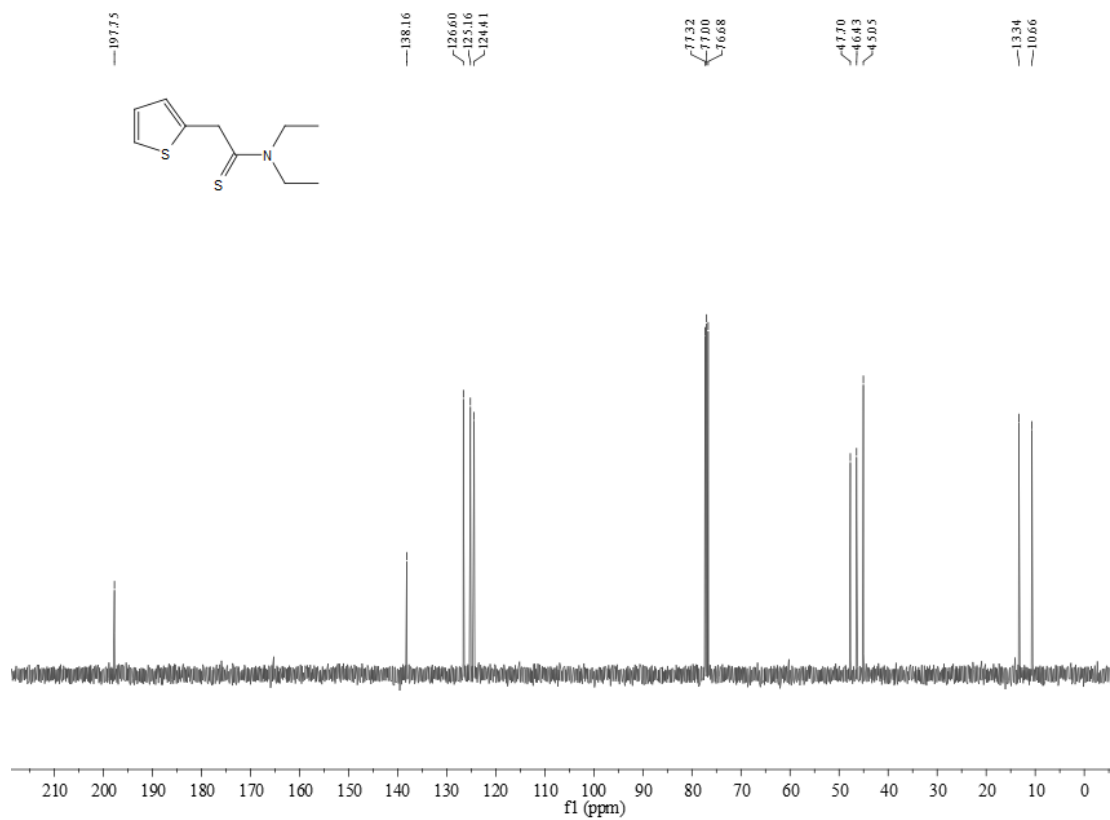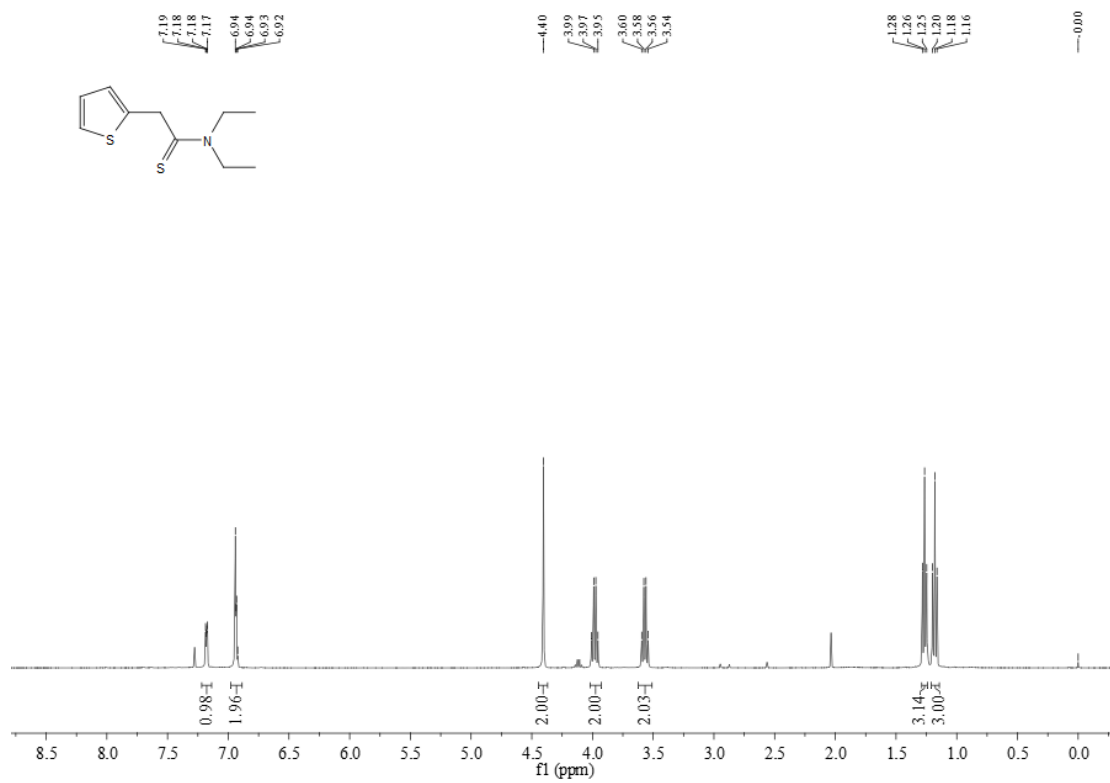
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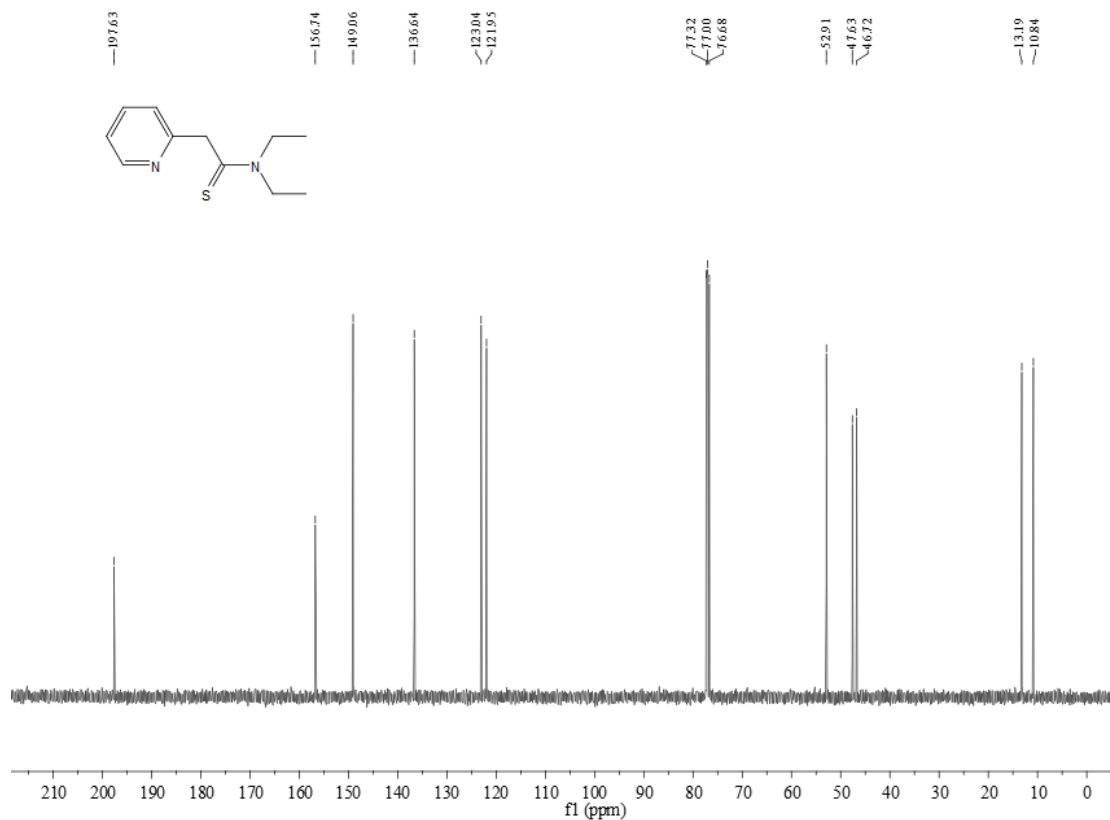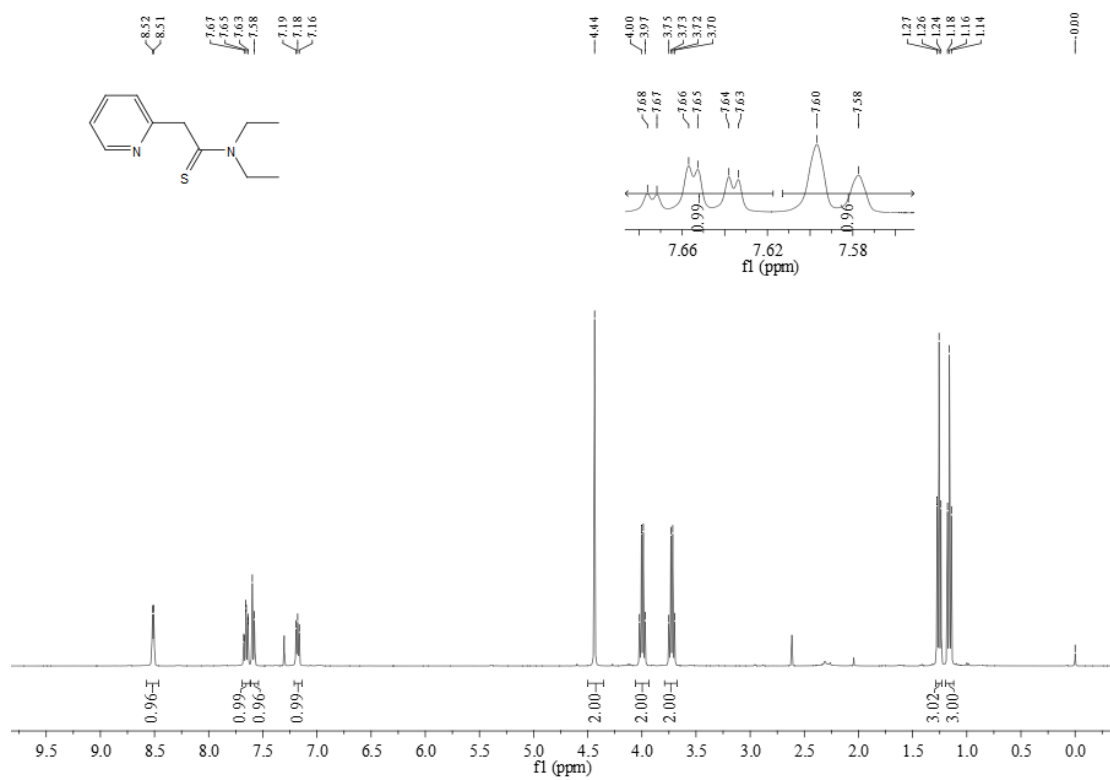
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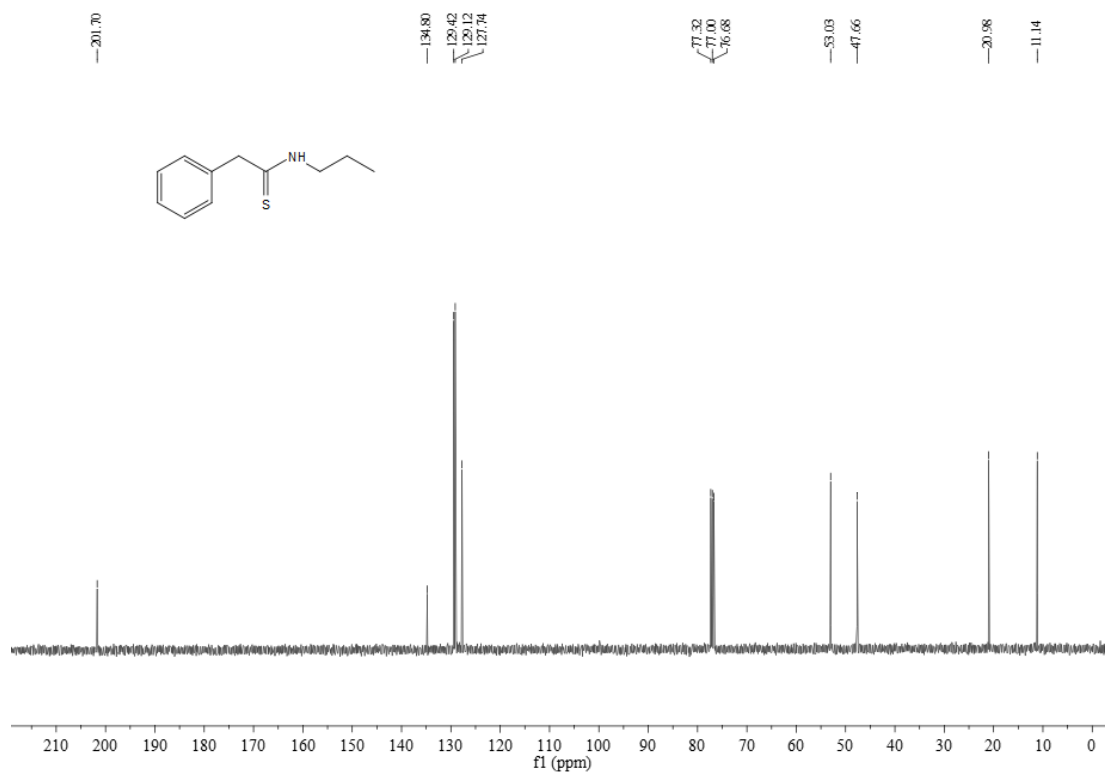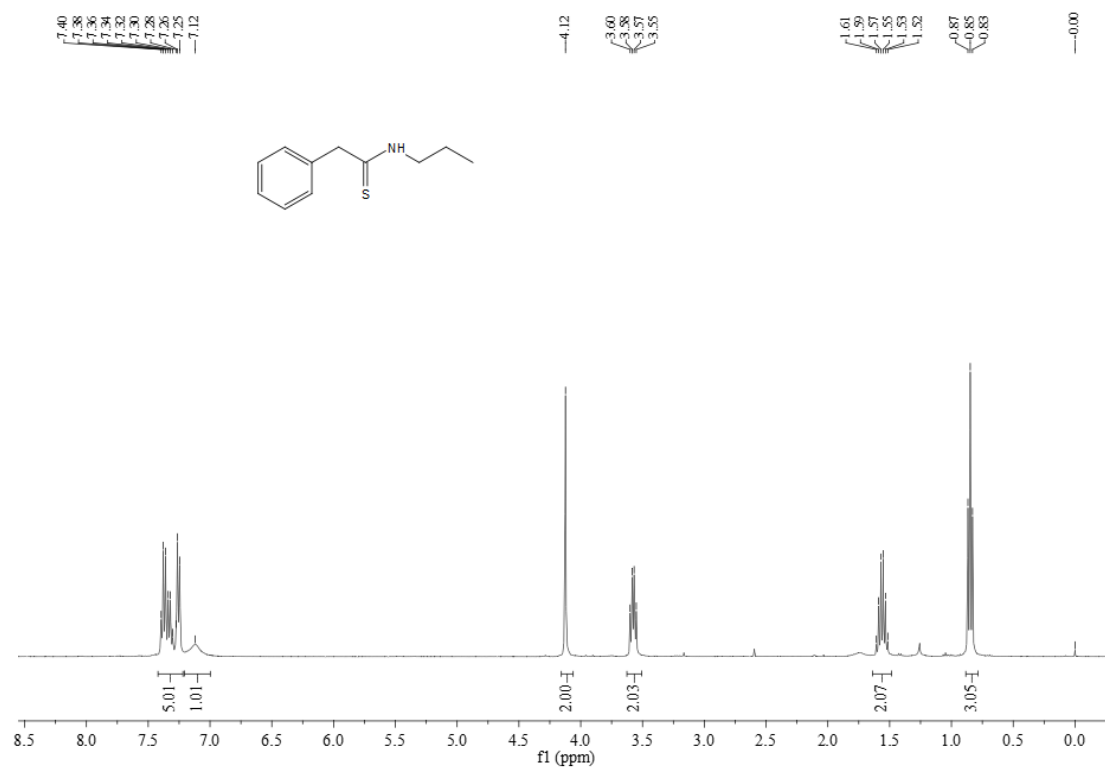
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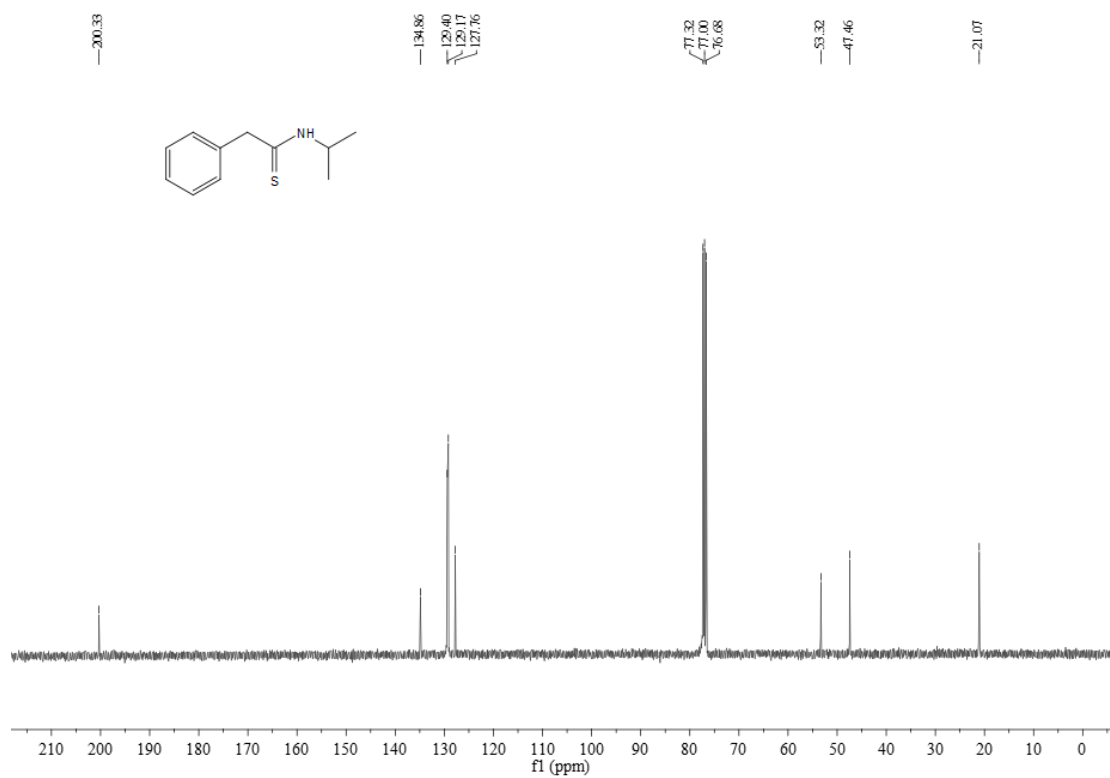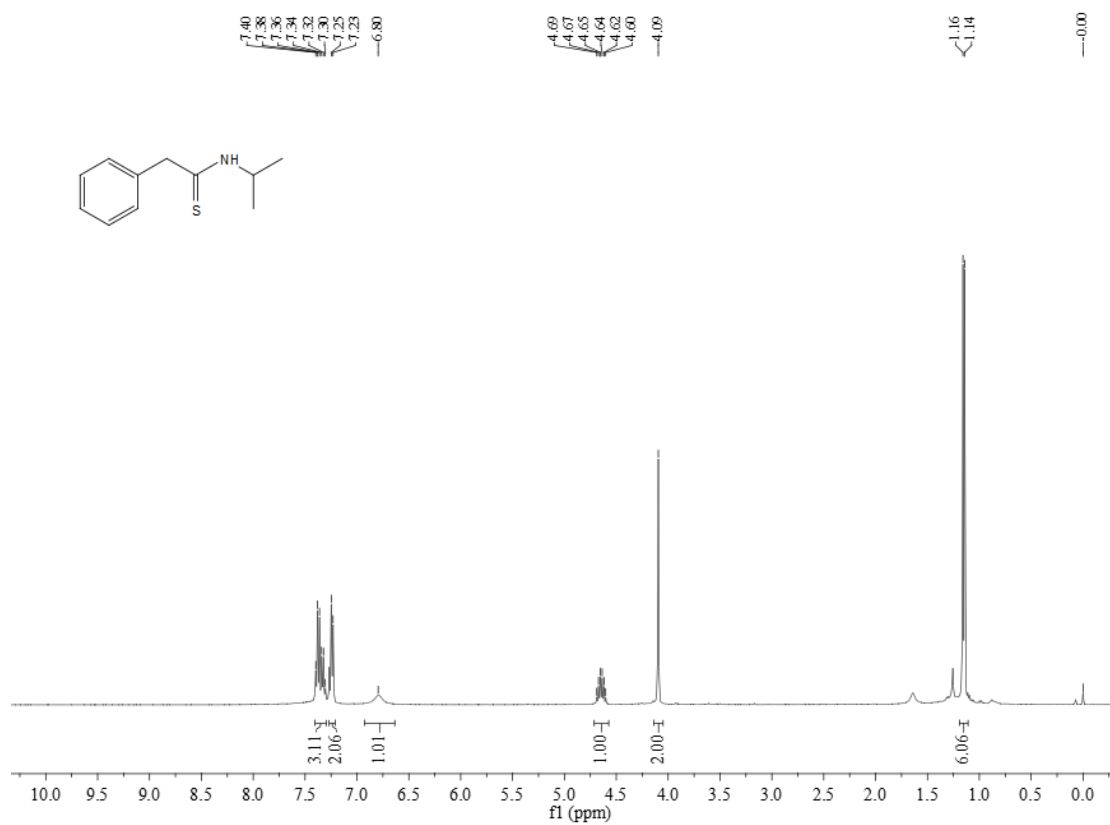
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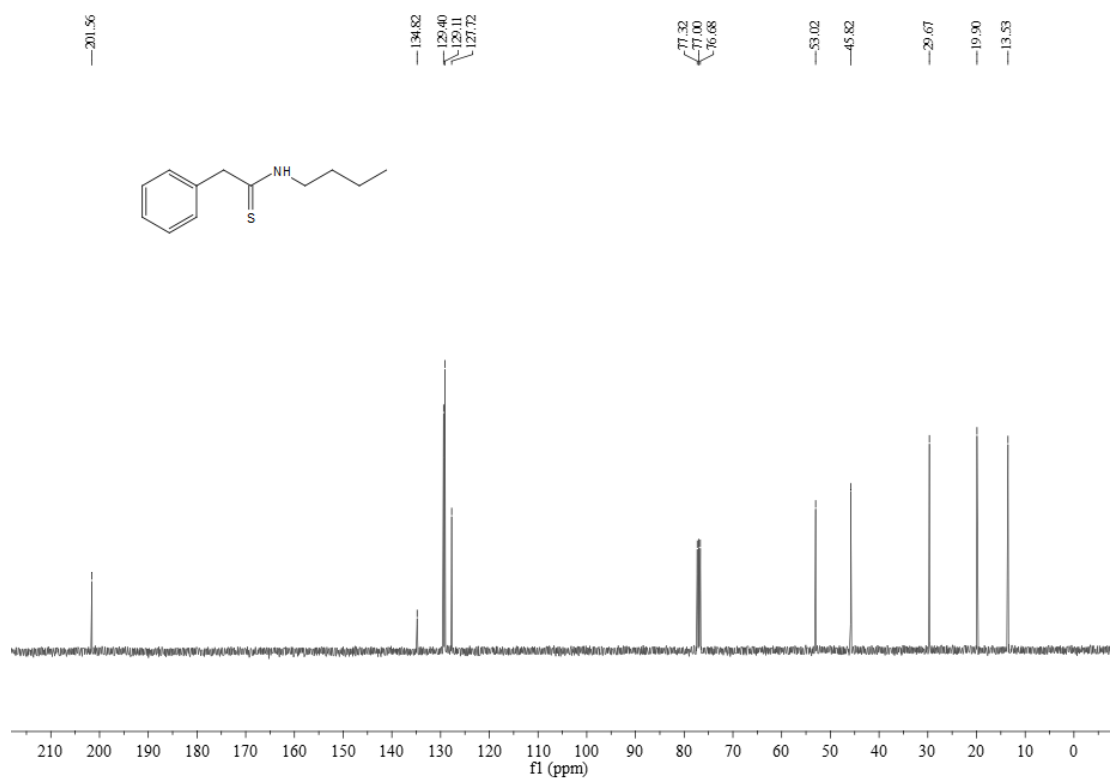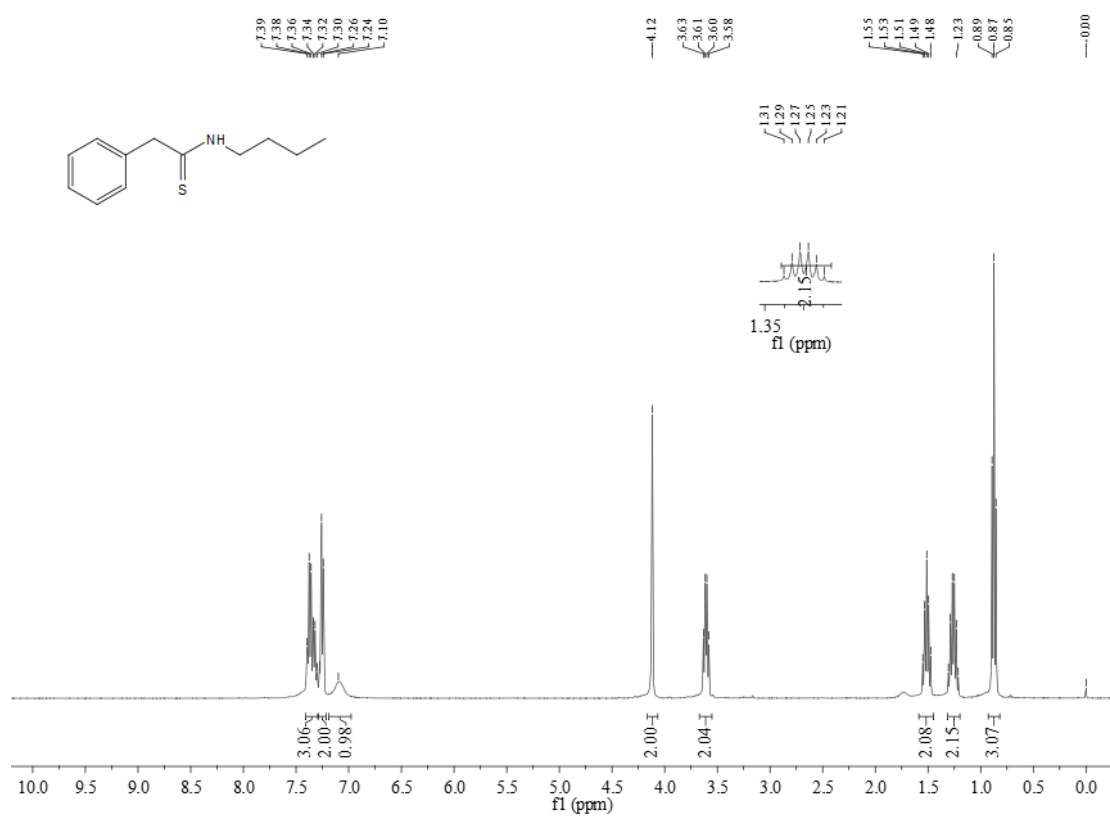
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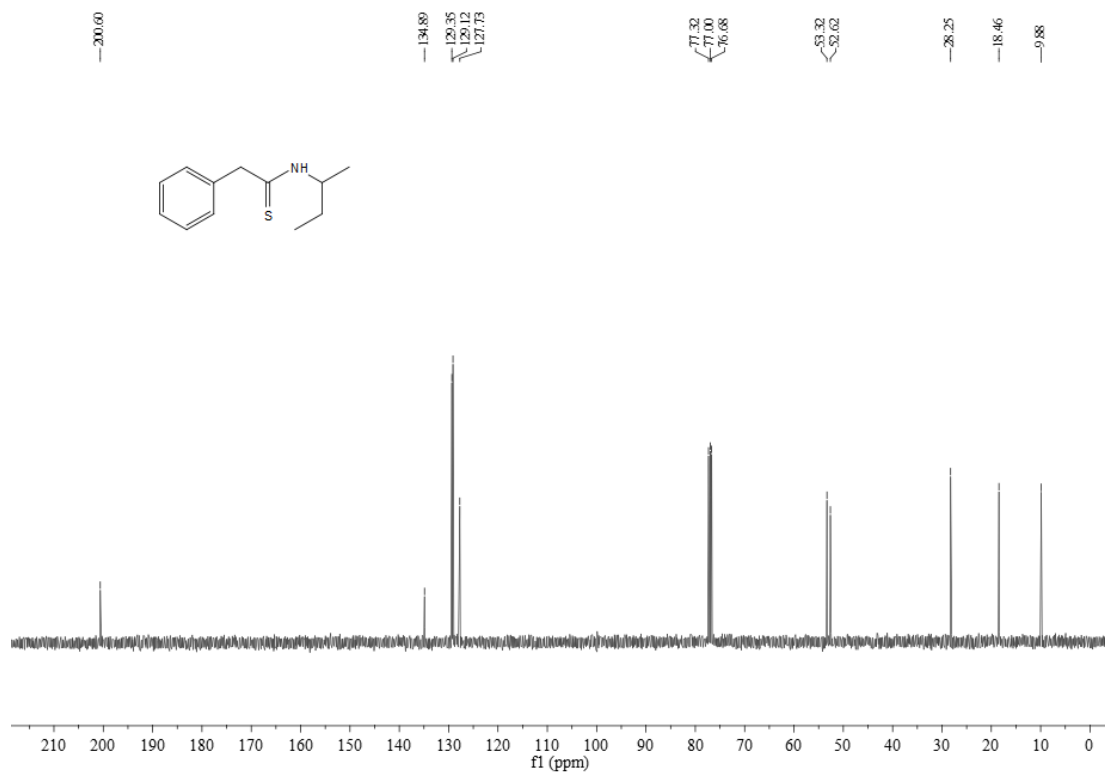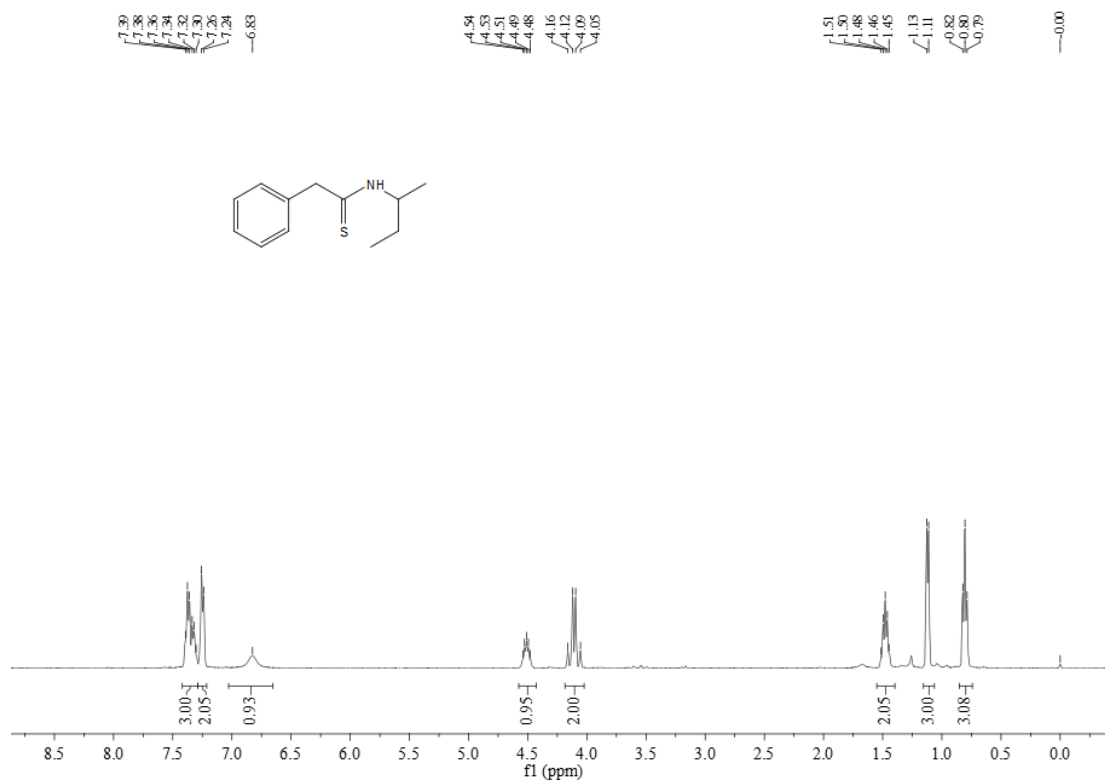
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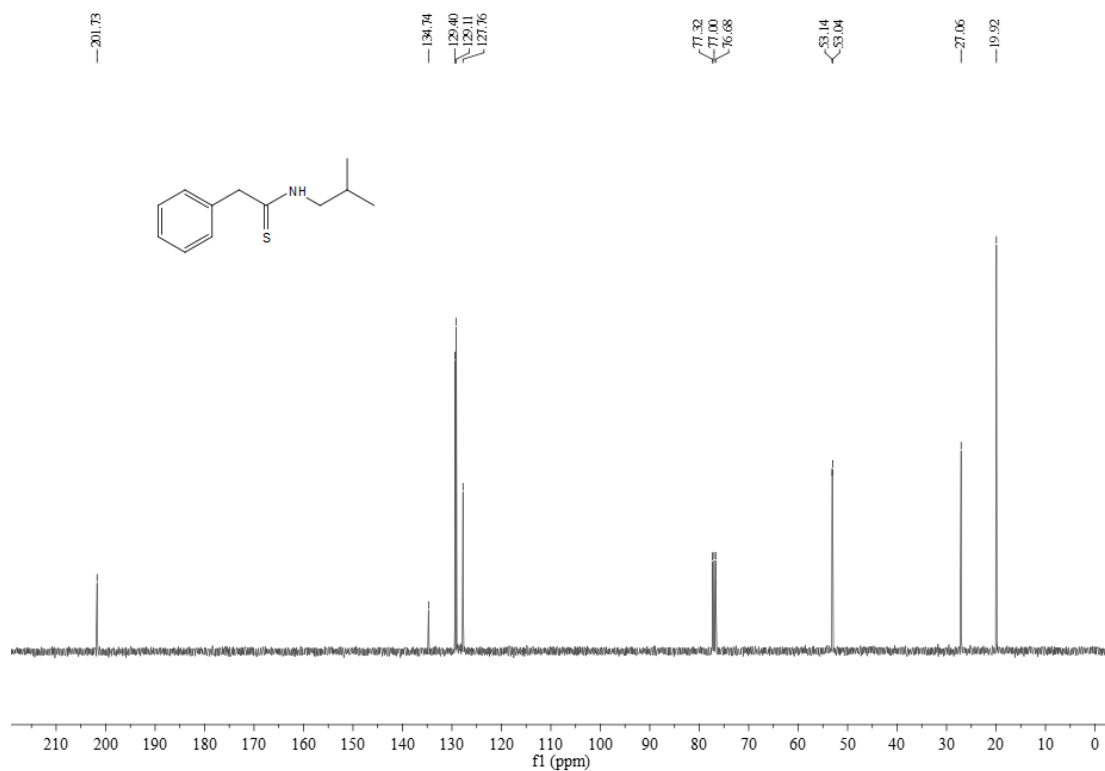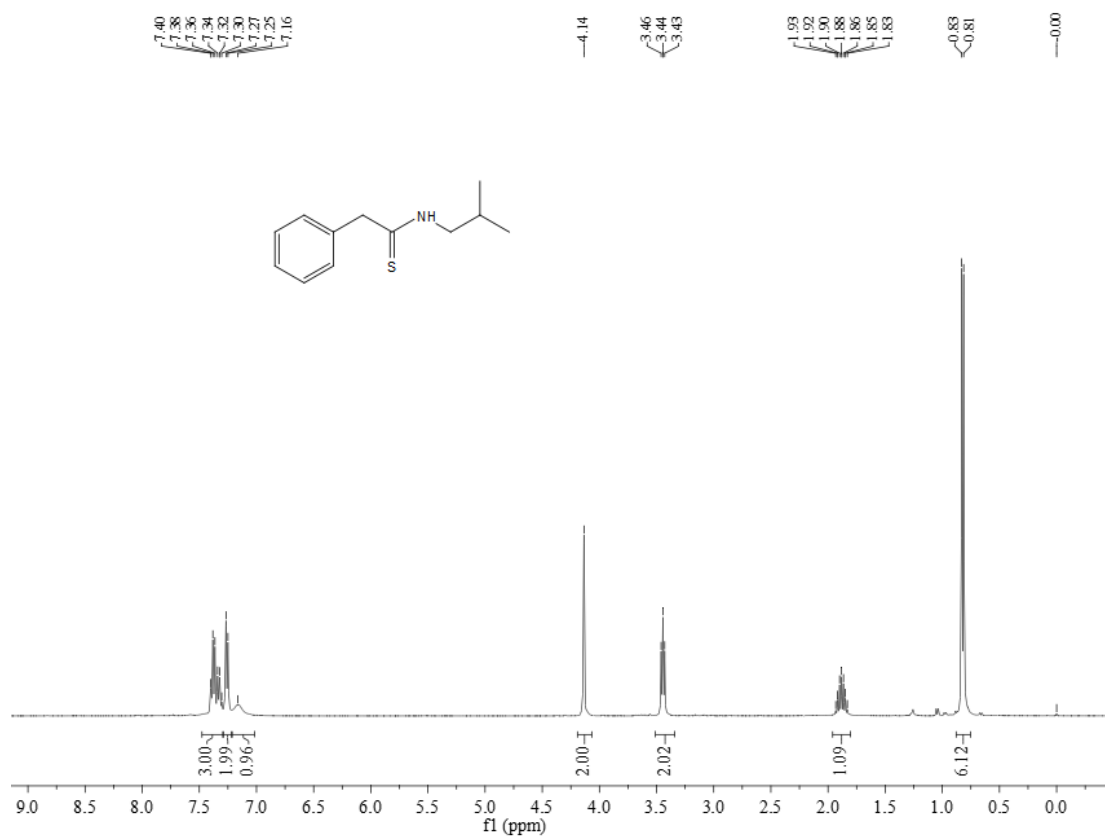
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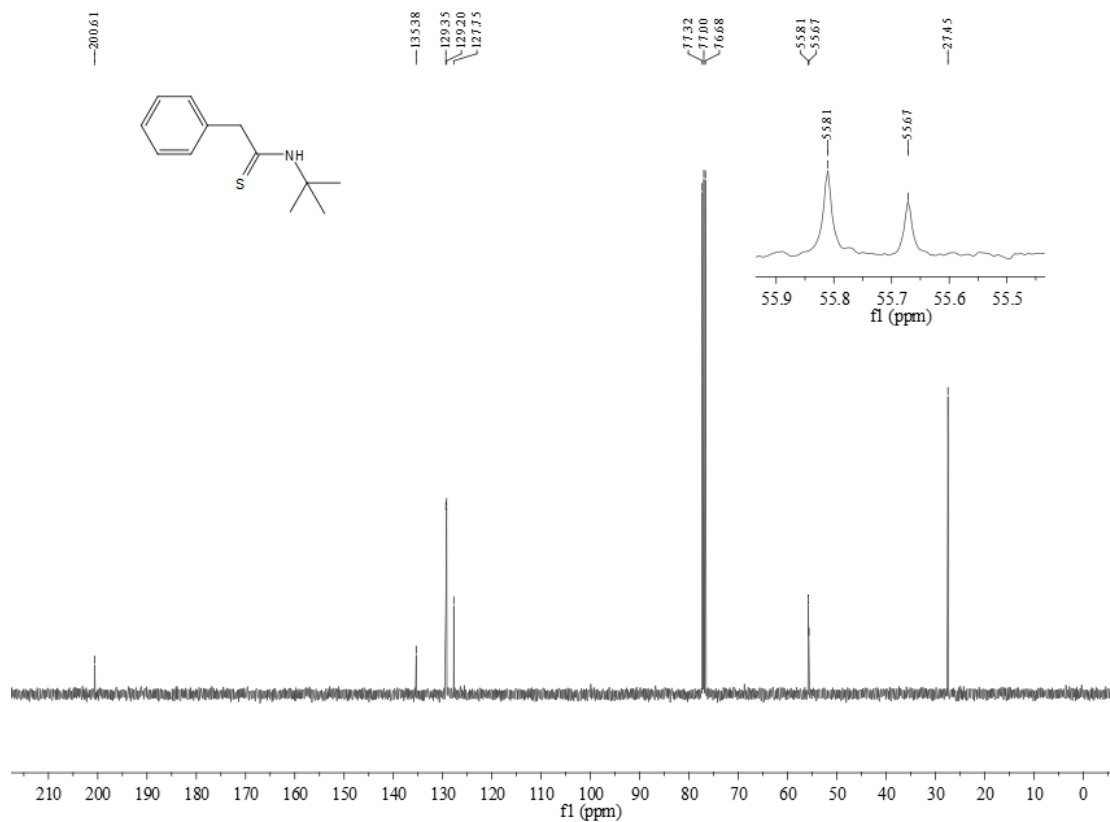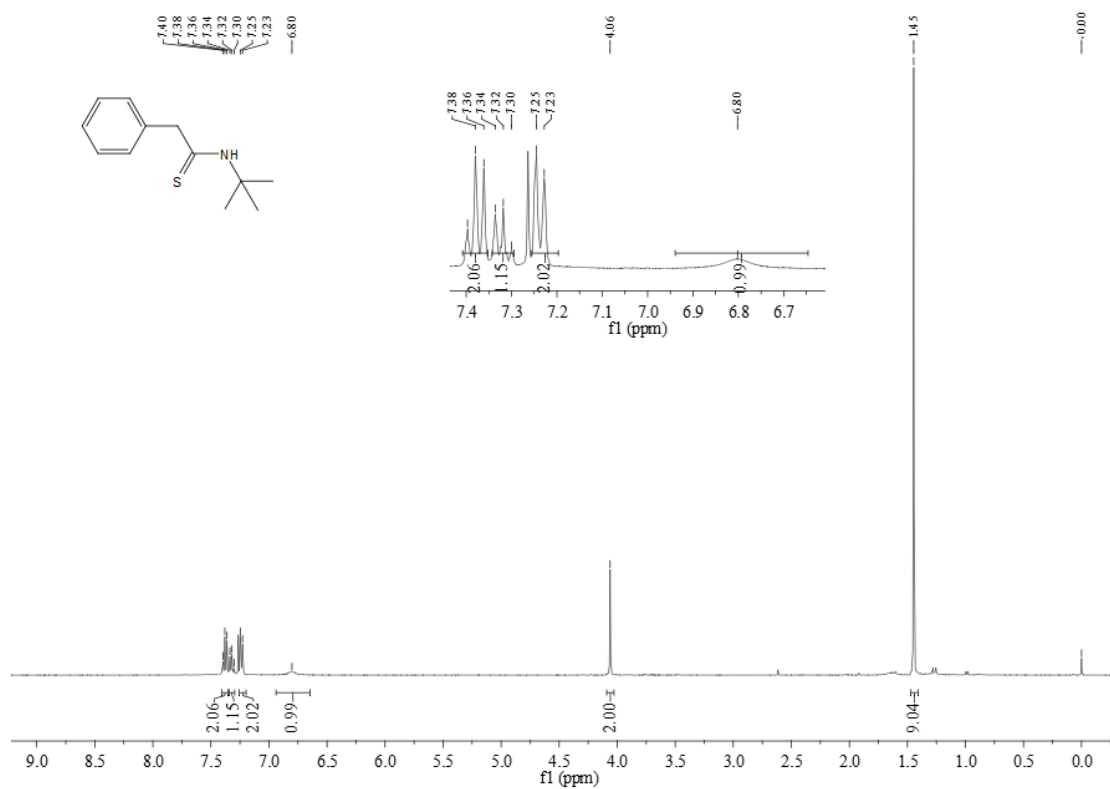
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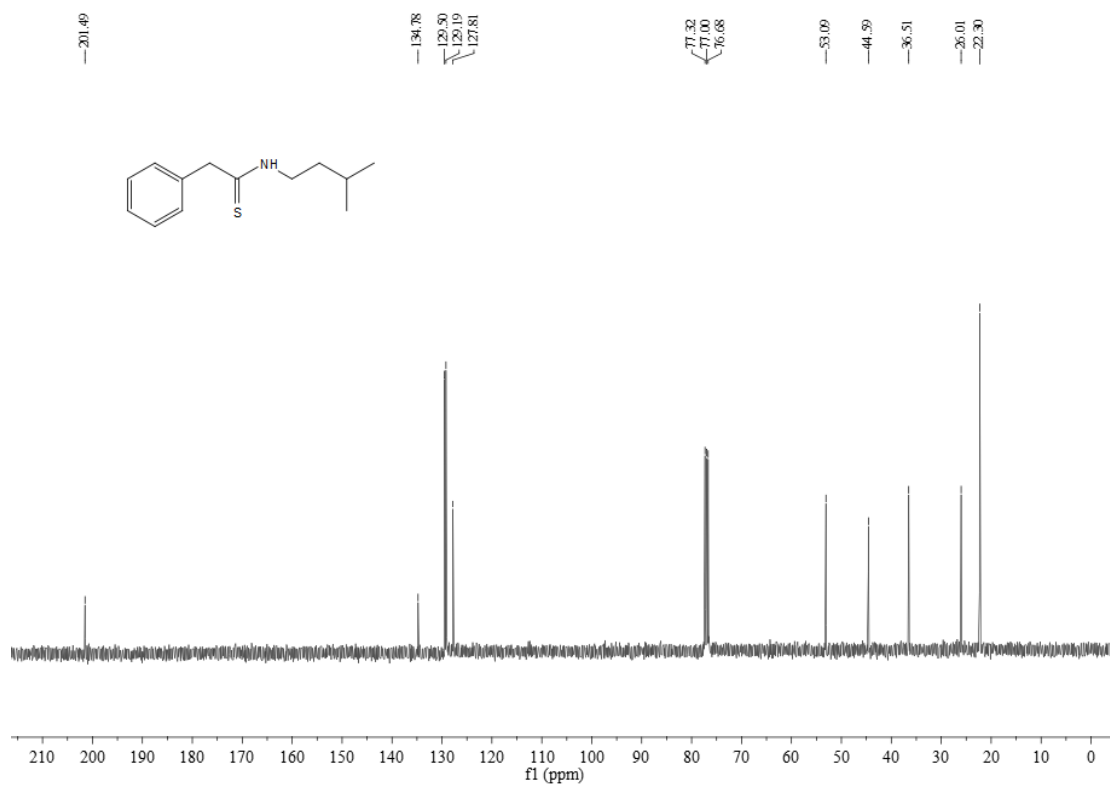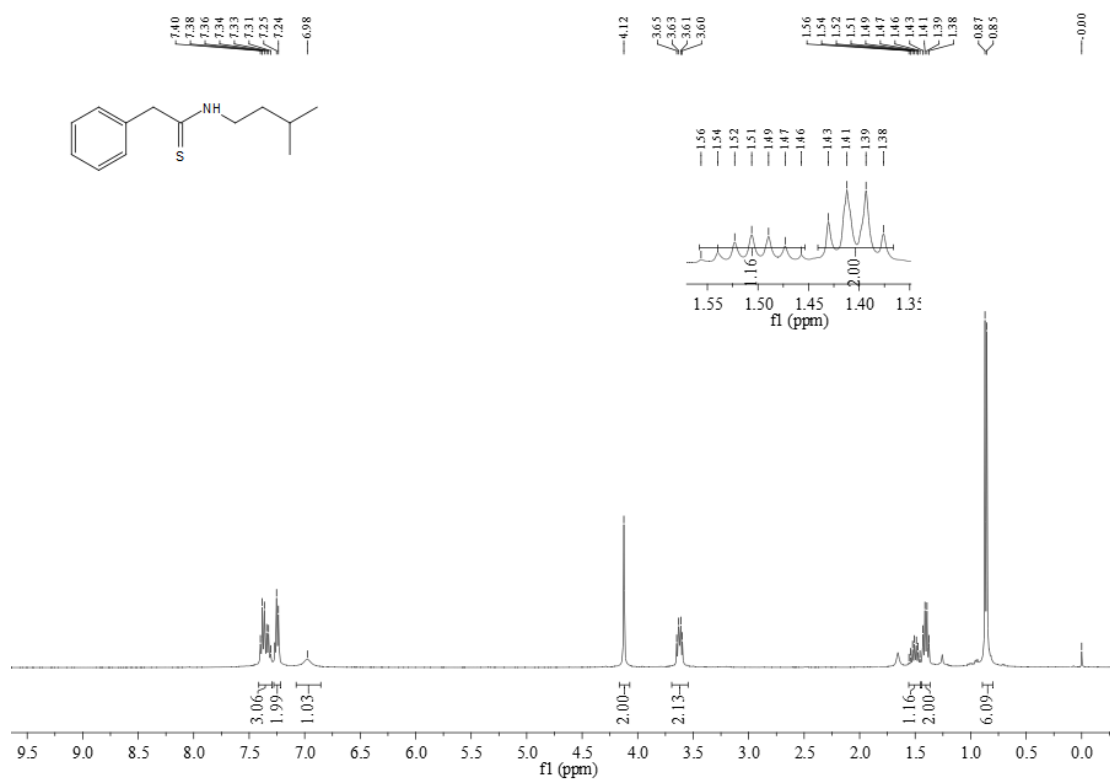
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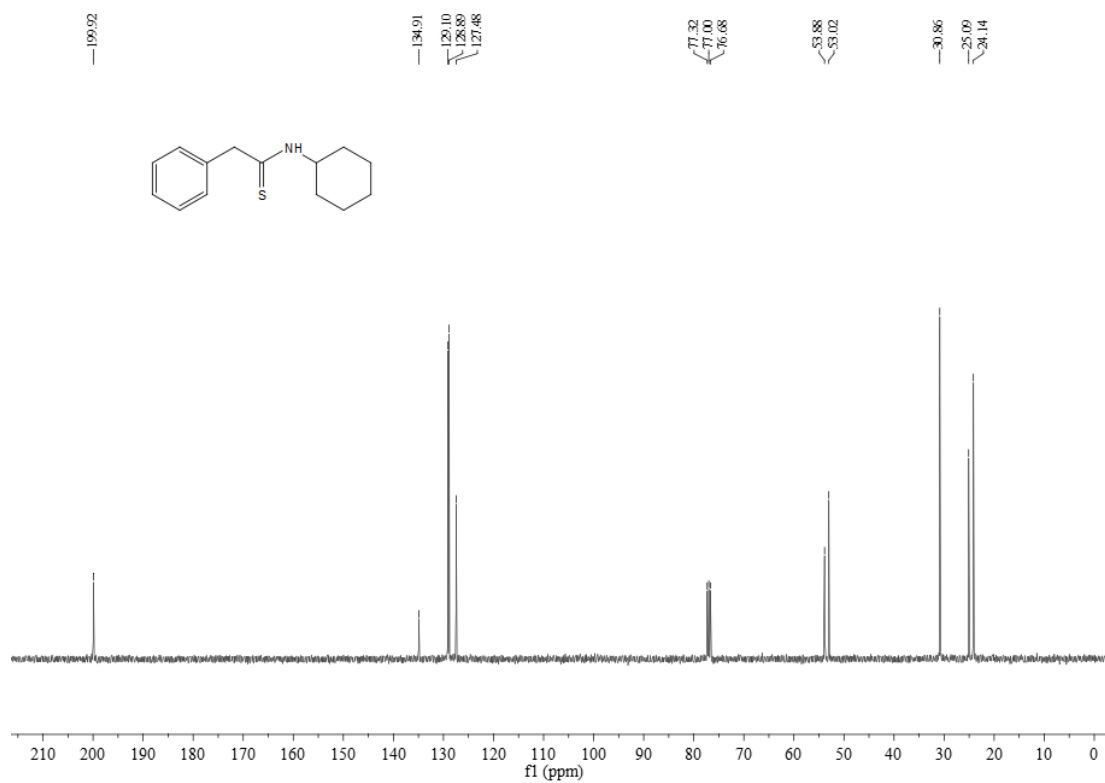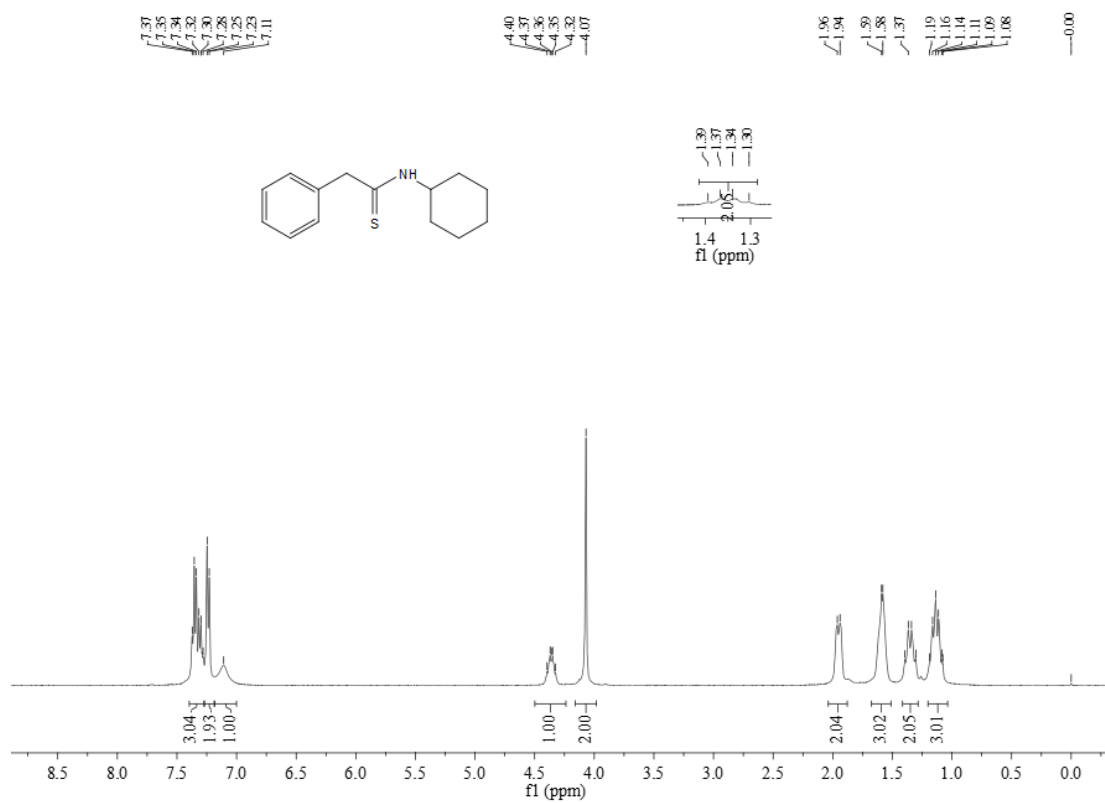
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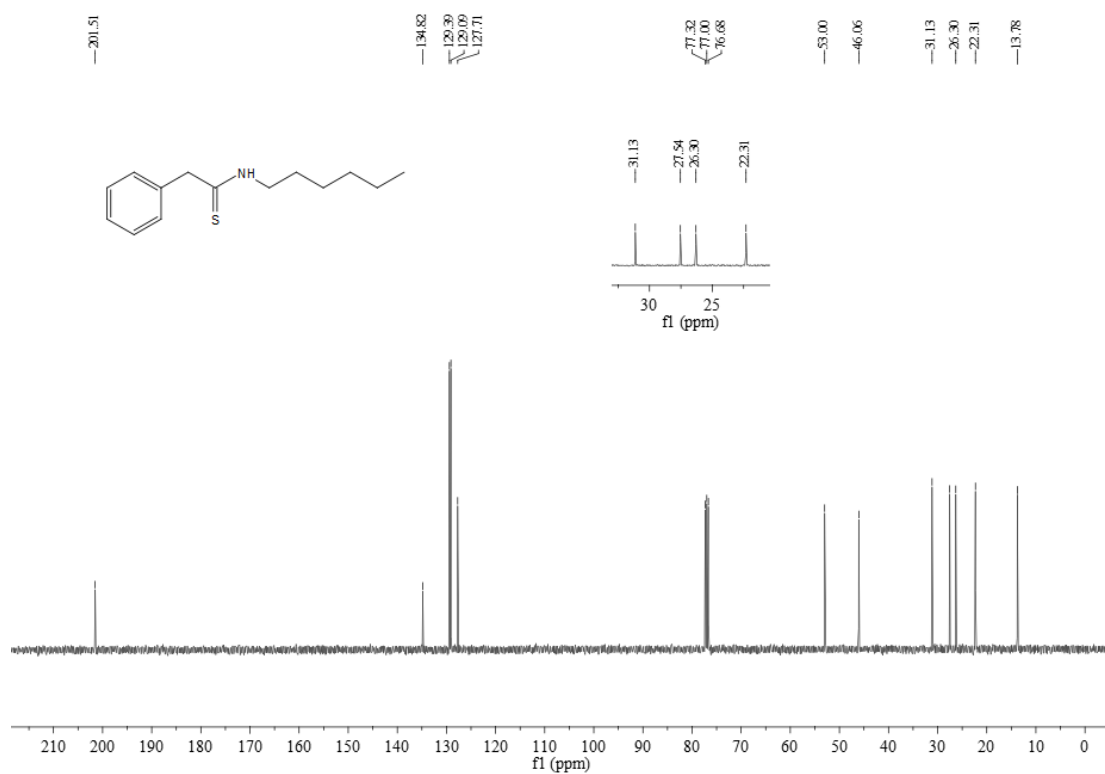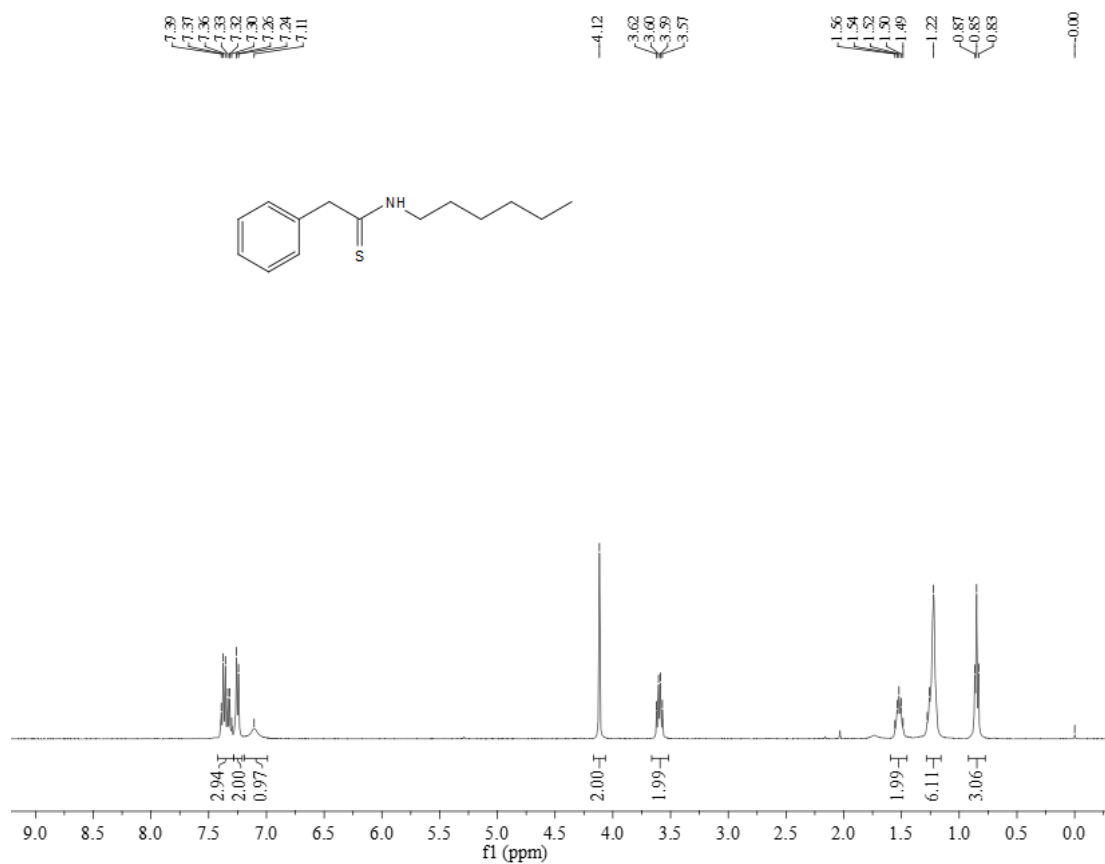
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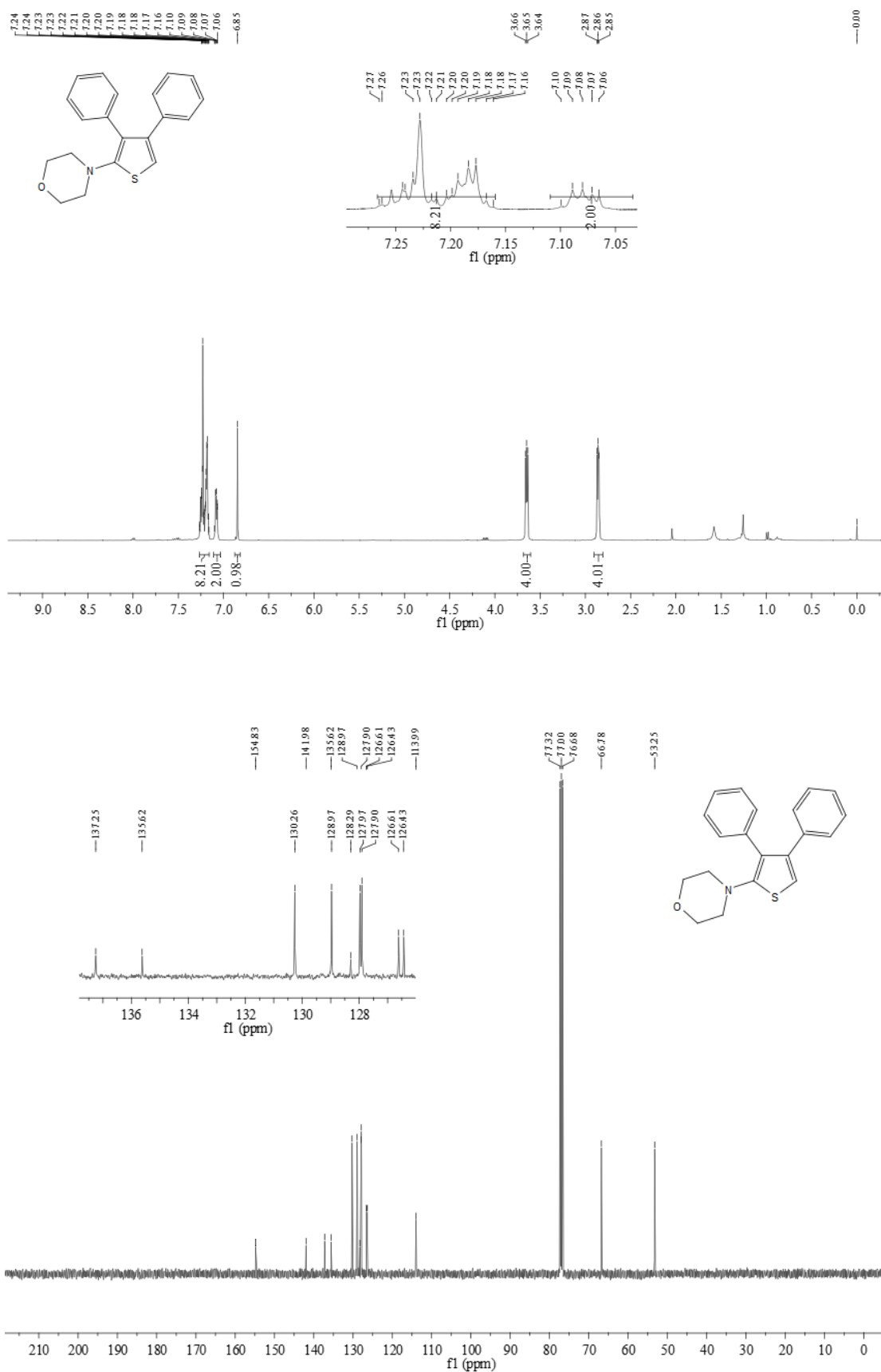
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