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Catalyst-free microwave-assisted arylglyoxal-based multicomponent reactions for the synthesis of fused pyrans

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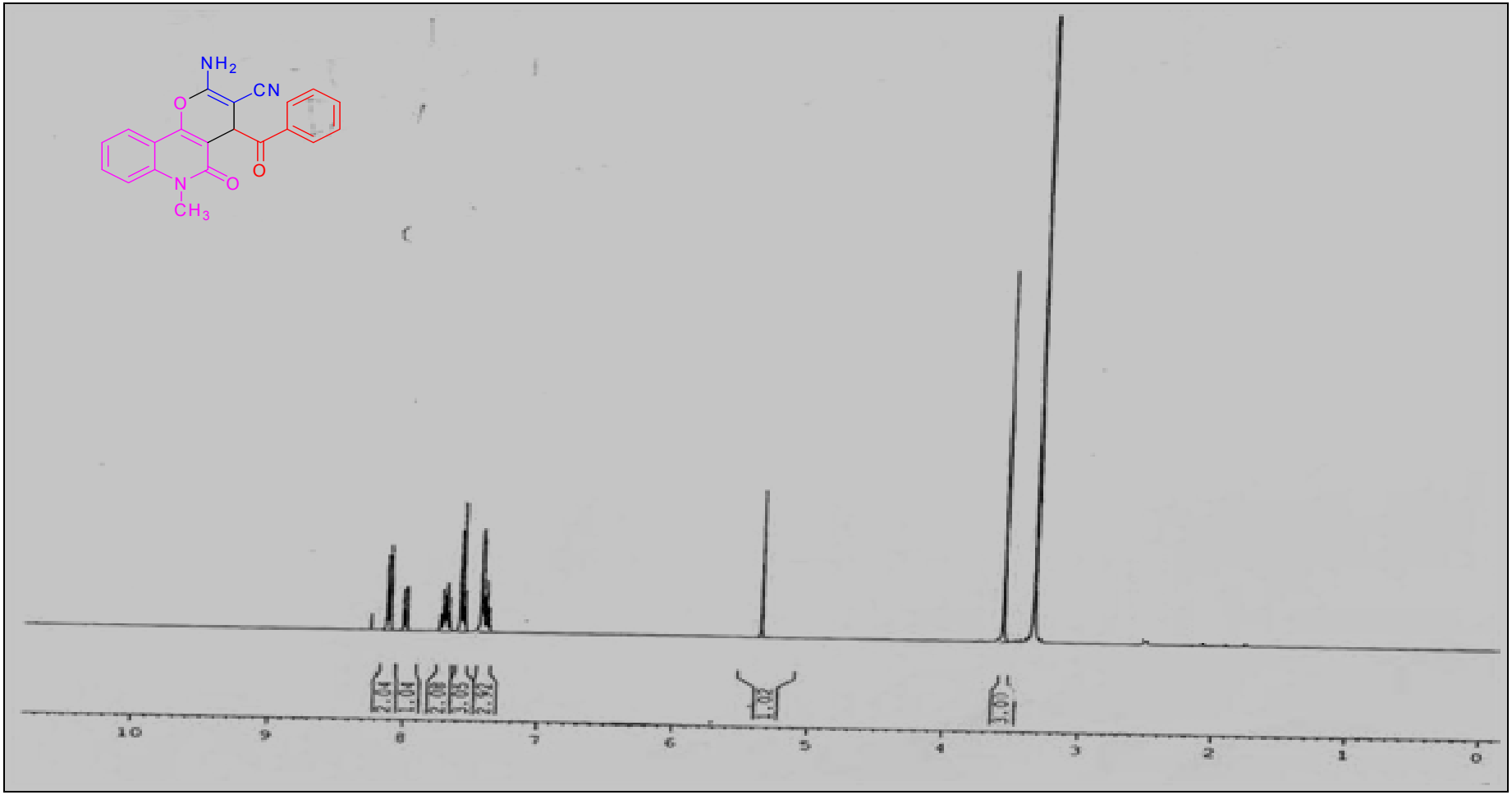
General Experimental Information

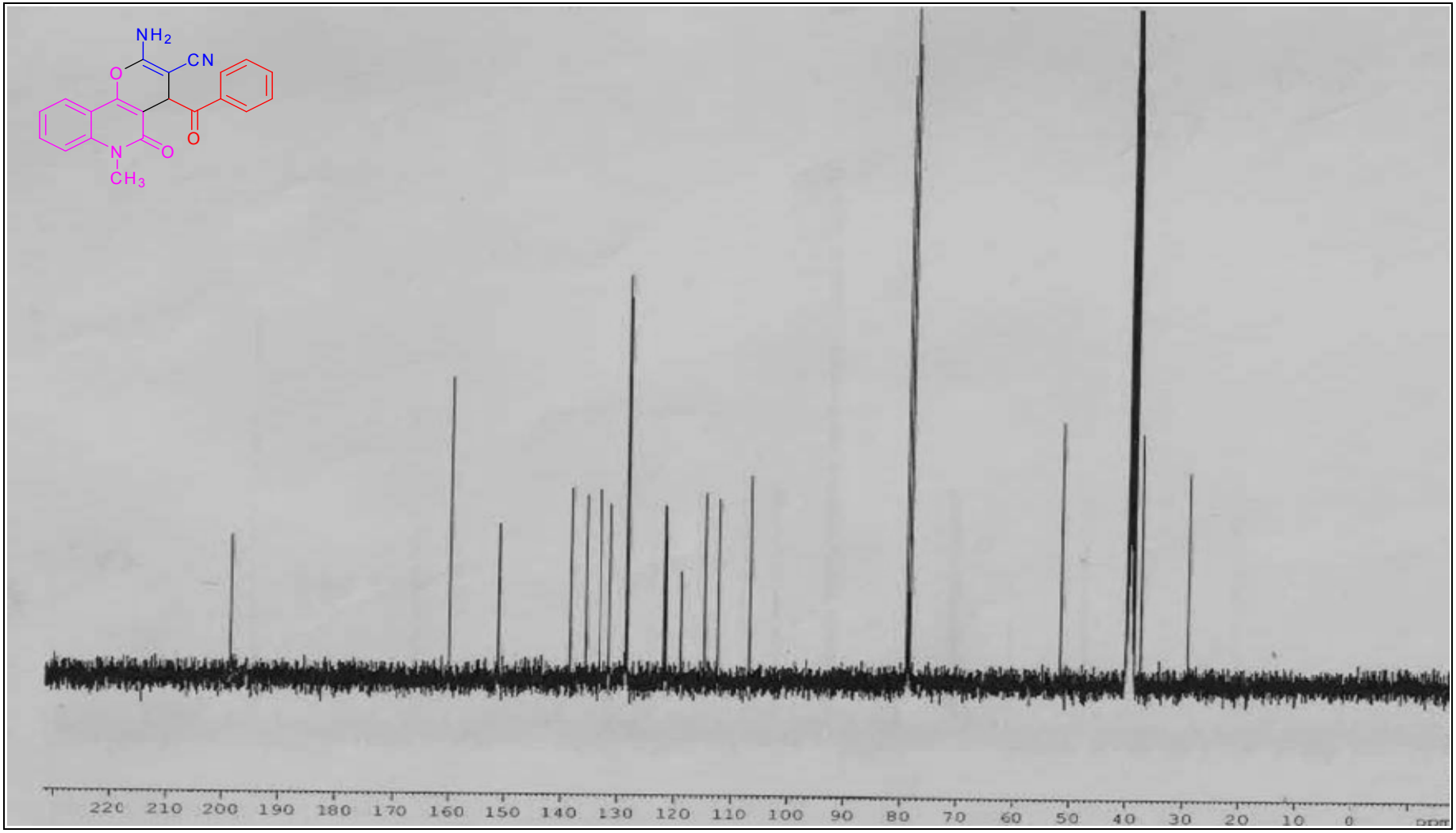
All starting materials were purchased from either Sigma Aldrich or Alfa Aesar and used without further purification. Microwave irradiation was carried out with Initiator 2.5 Microwave Synthesizers from Biotage, Uppsala, Sweden. NMR spectra were recorded on Bruker 400 or 500 MHz for ^{1}H and 100 or 125 MHz for ^{13}C in $CDCl_3$/DMSO-d6, chemical shift values were reported in δ values ppm downfield from tetra methyl silane. Infrared spectra were recorded on a Shimadzu FTIR spectrometer. CHN analyses were carried out using Elementar, Vario micro cube elemental analyzer and melting points were recorded using SRS EZ- Melt automated melting point apparatus by capillary methods and uncorrected.

Typical experimental procedure for the synthesis of 1(a). Typical experimental procedure for the preparation of 1a: A mixture of phenylglyoxal monohydrate (1.0 mmol) and malononitrile (1.0 mmol) in ethanol (2 ml) was stirred at room temperature for 5 minutes. To this mixture 4-hydroxy-1-methyl-2(1H)-quinolone (1 mmol) was added and the resultant mixture was kept under microwave irradiation with sealed and stirring conditions for 10 minutes, keeping the temperature at 110 ^{o}C . After completion of the reaction, the reaction mixture was cooled to room temperature and solid product was separated by just filtration and purified by recrystallization from mixture of ethanol and THF. Using similar procedure all other products were prepared.

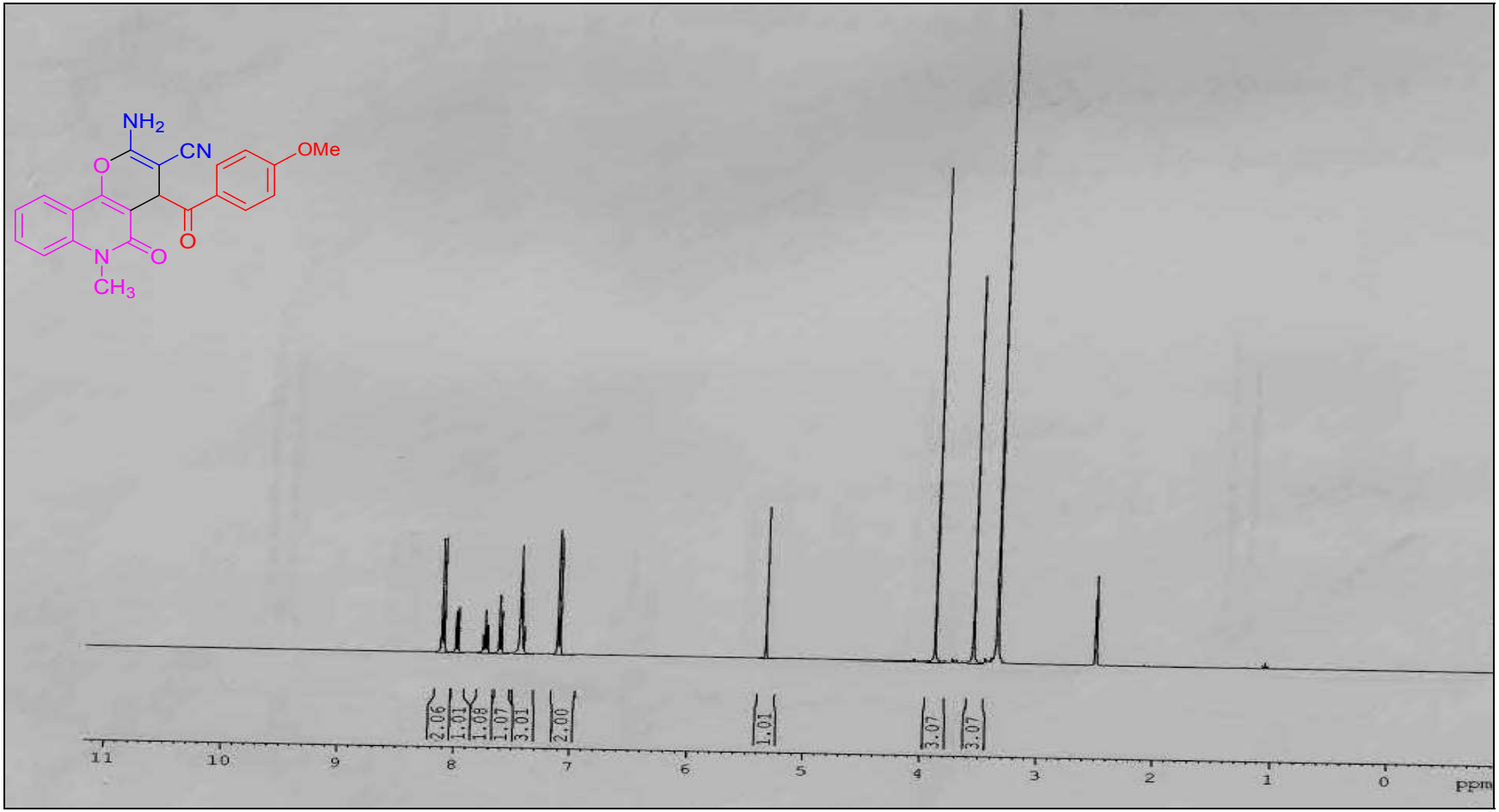
^{1}H NMR spectra of 1a



^{13}C NMR spectra of 1a



^{1}H NMR spectra of 1b



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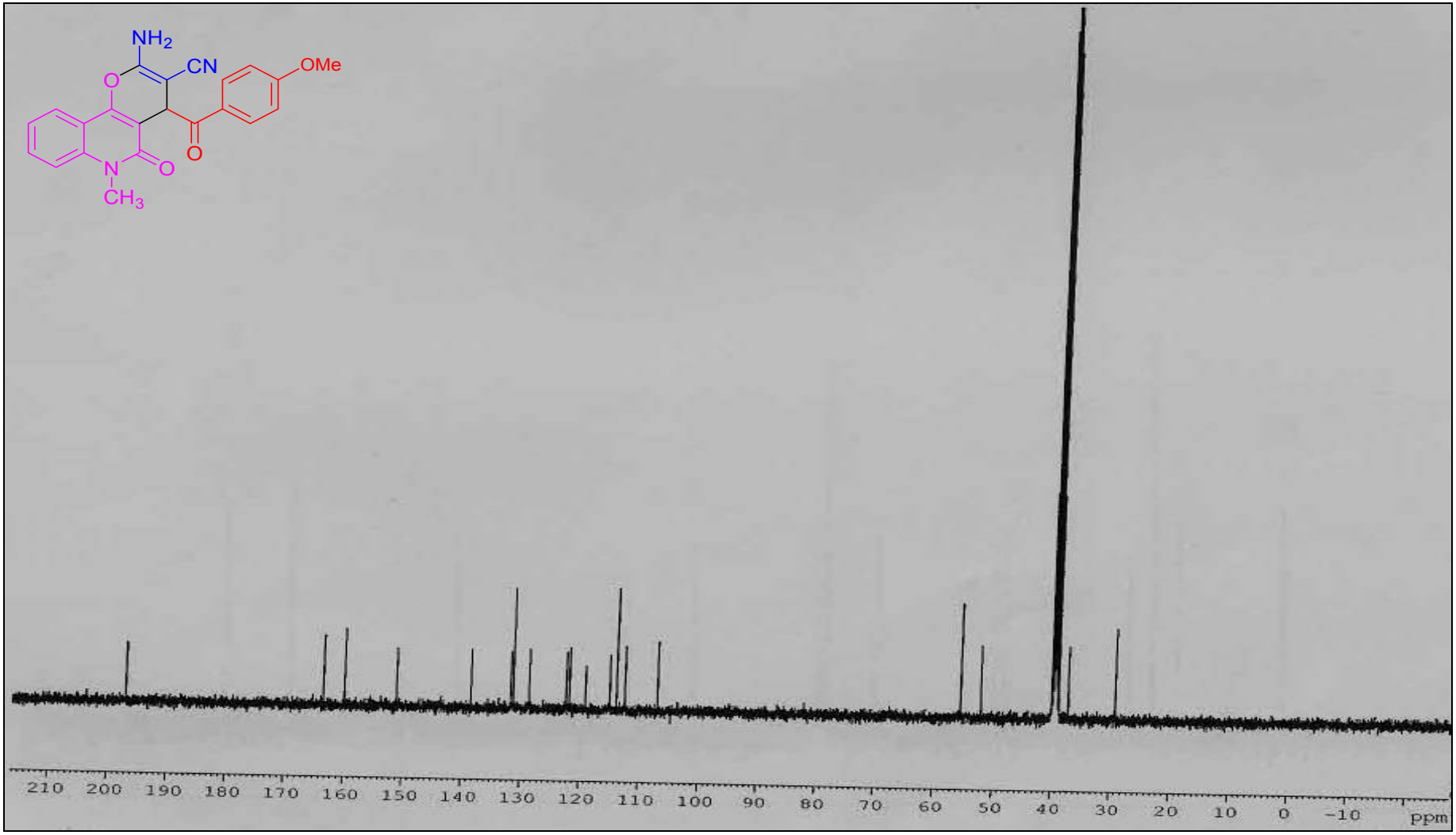

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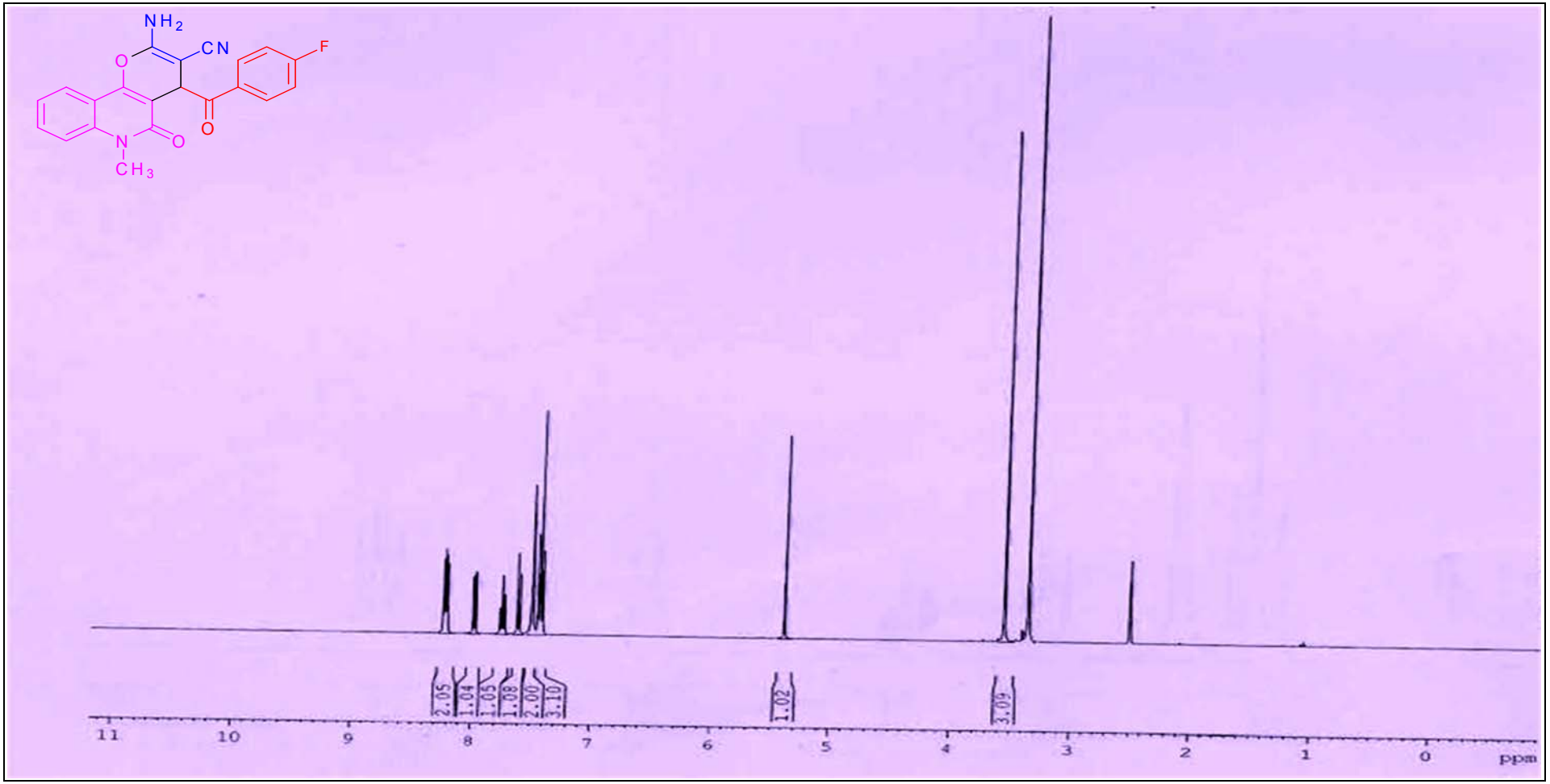

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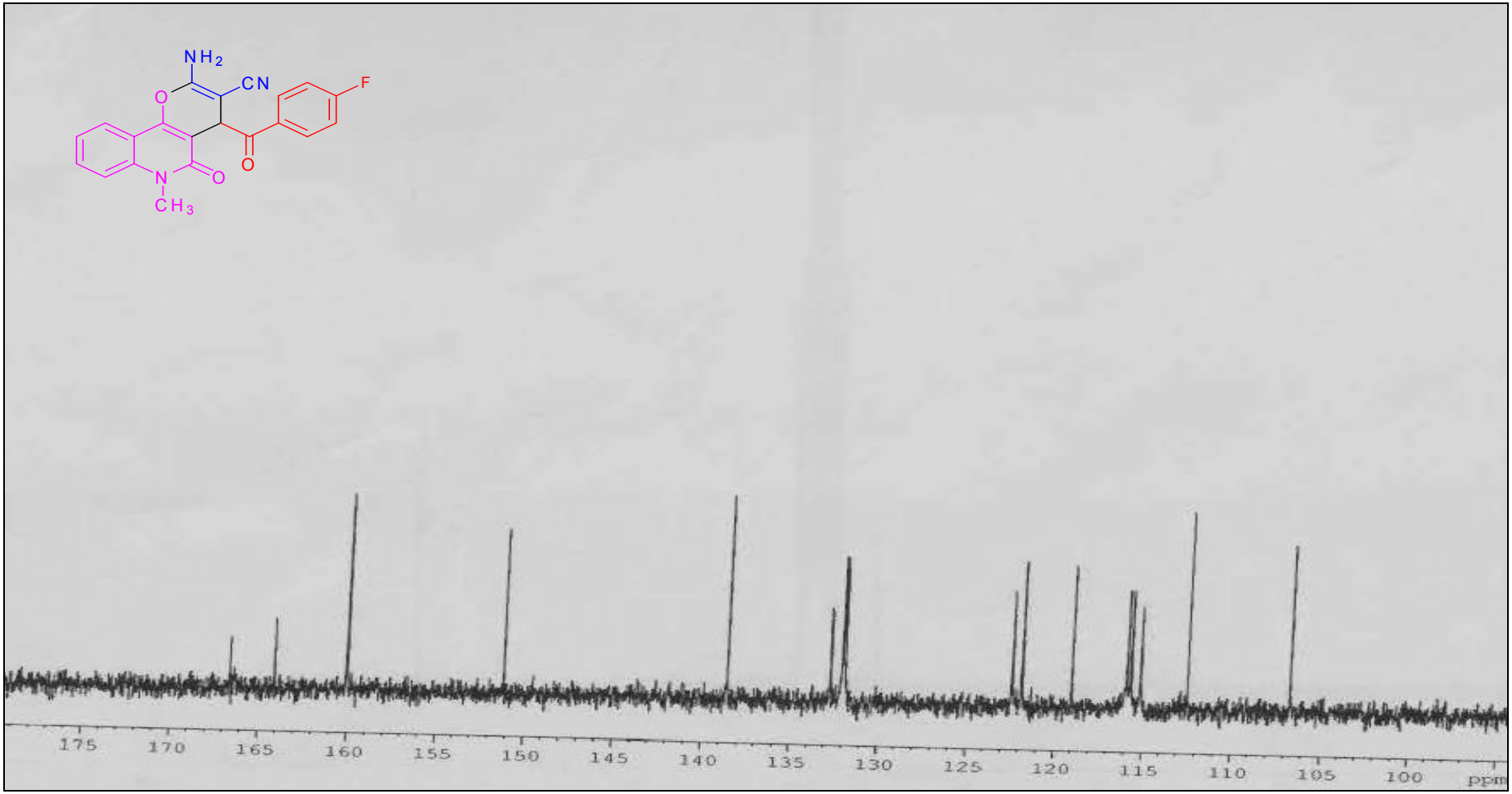

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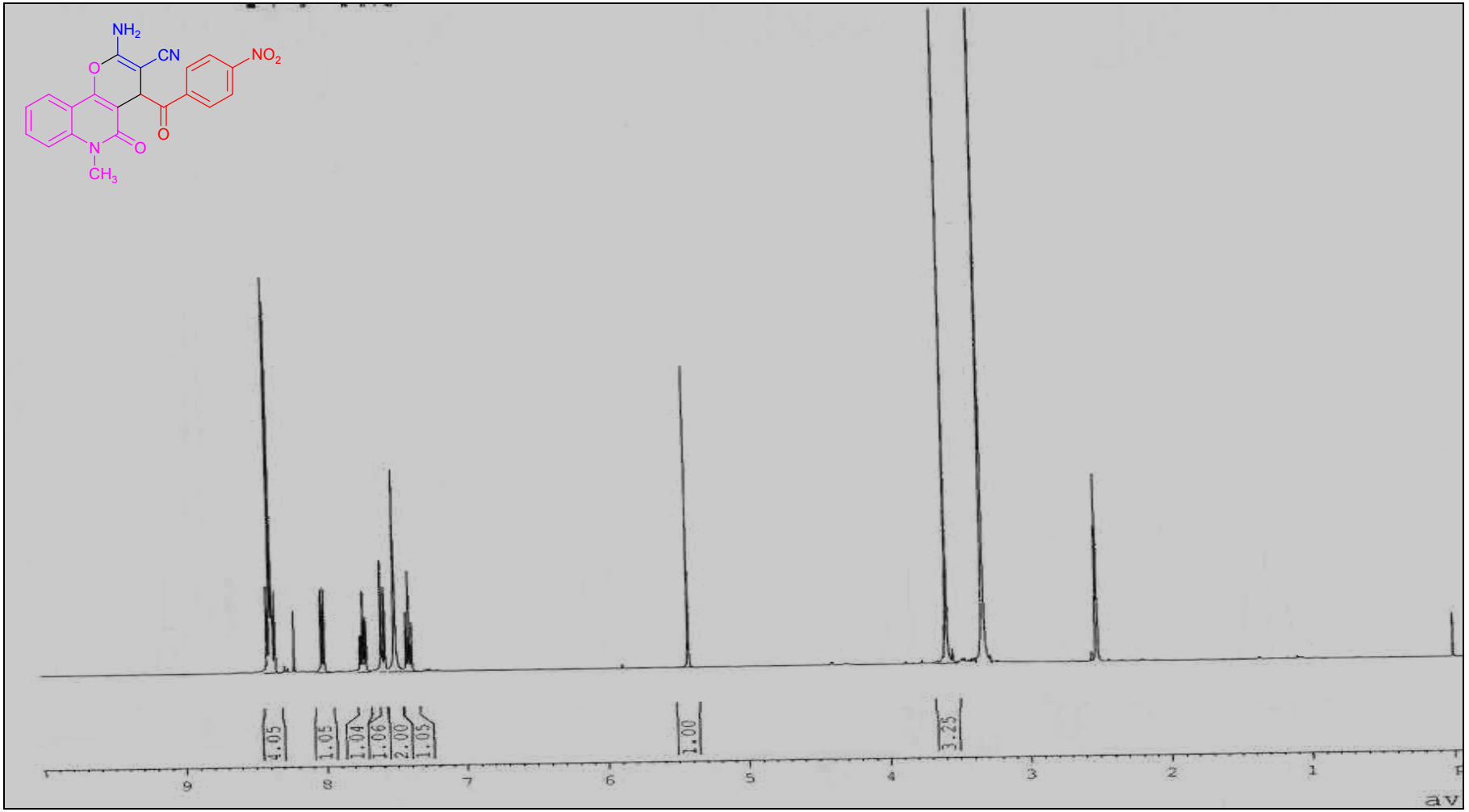

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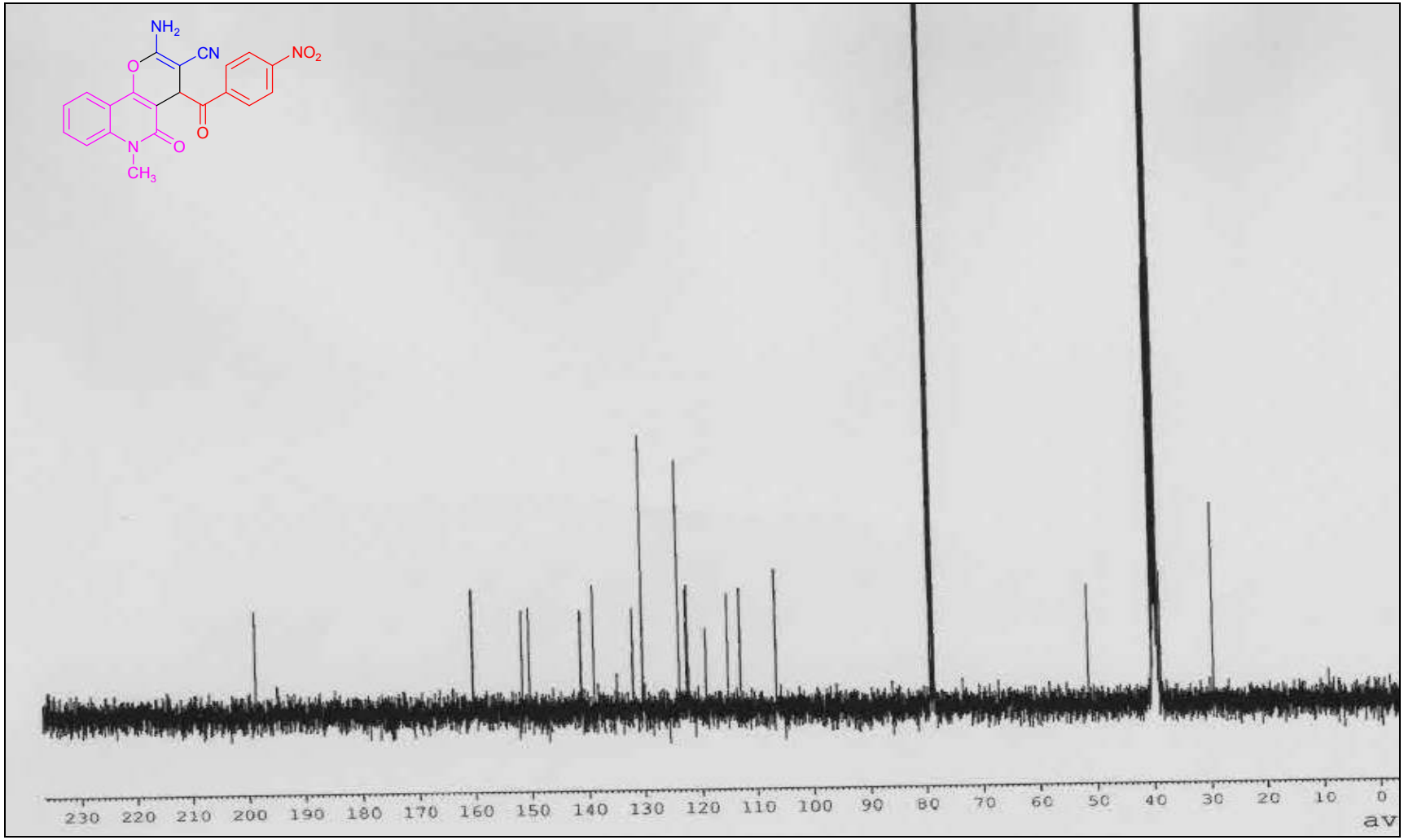

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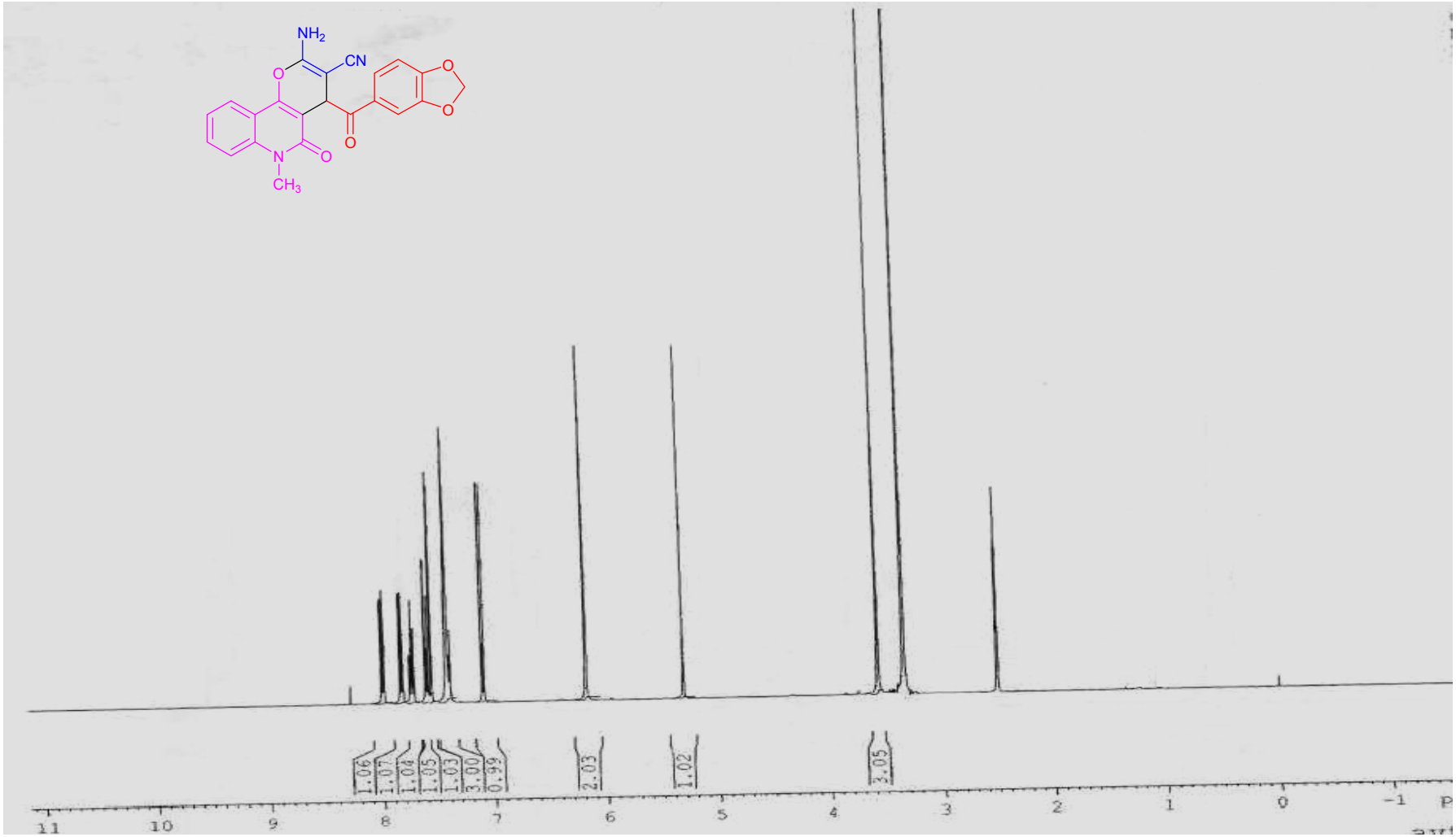

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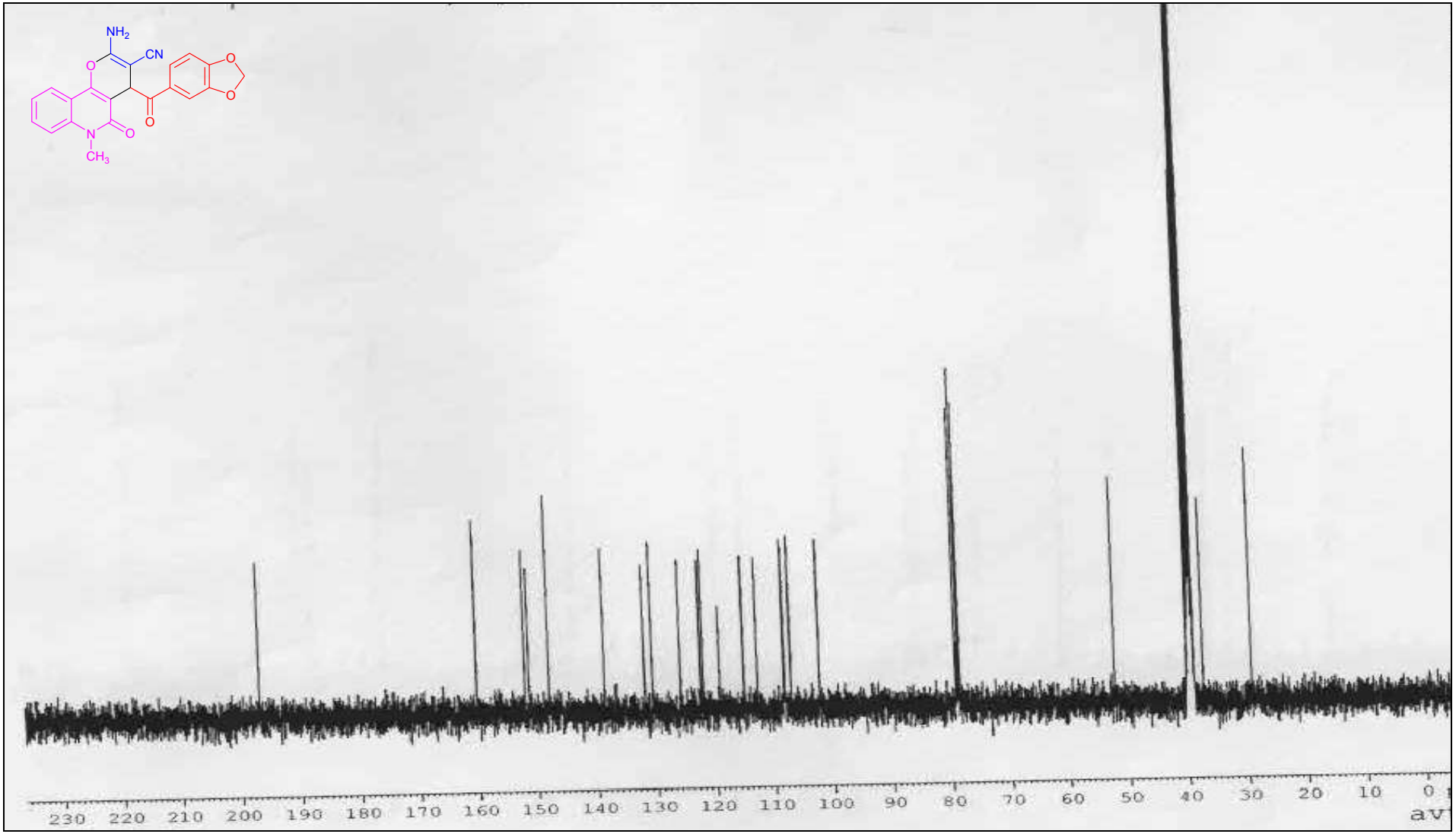

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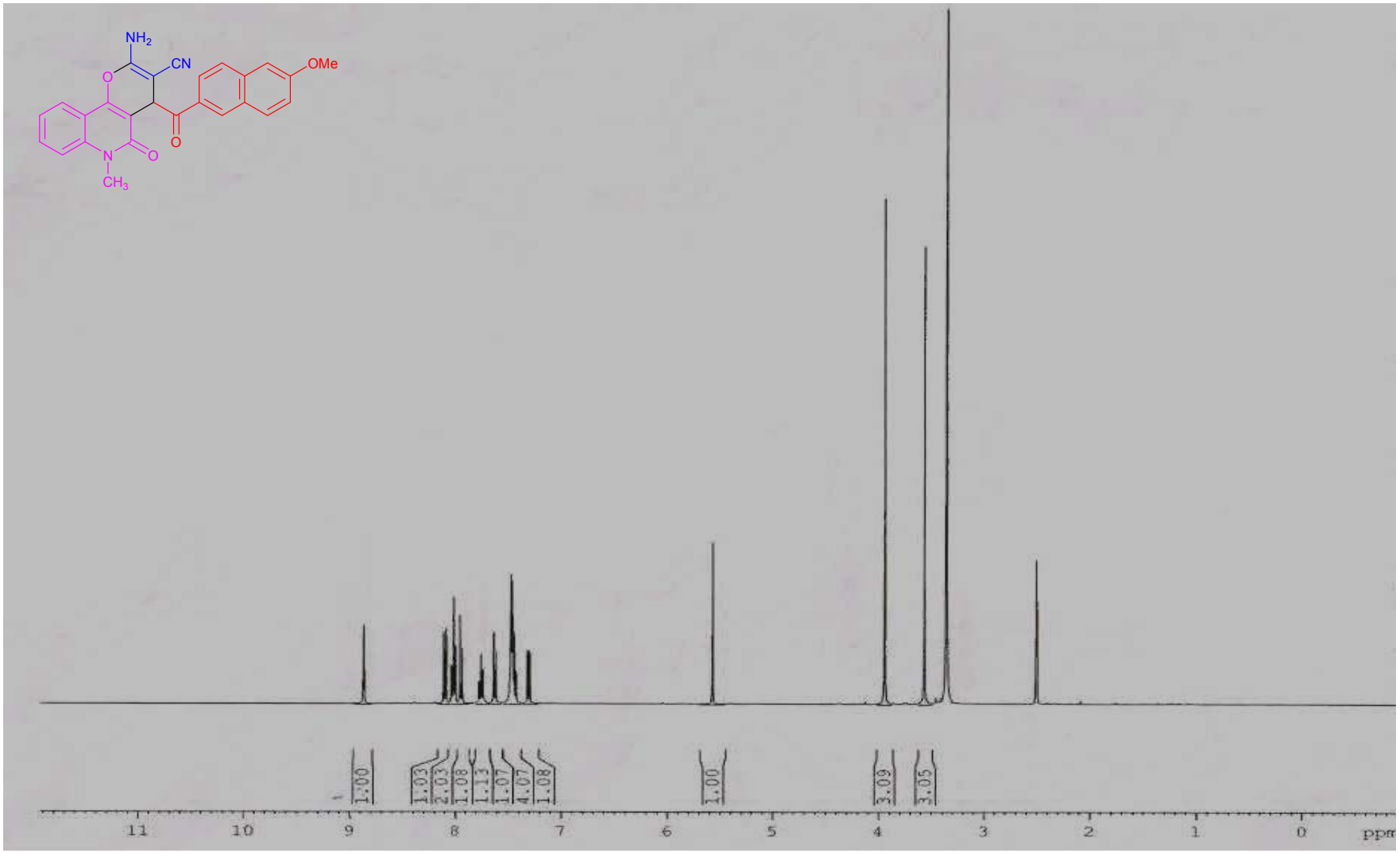

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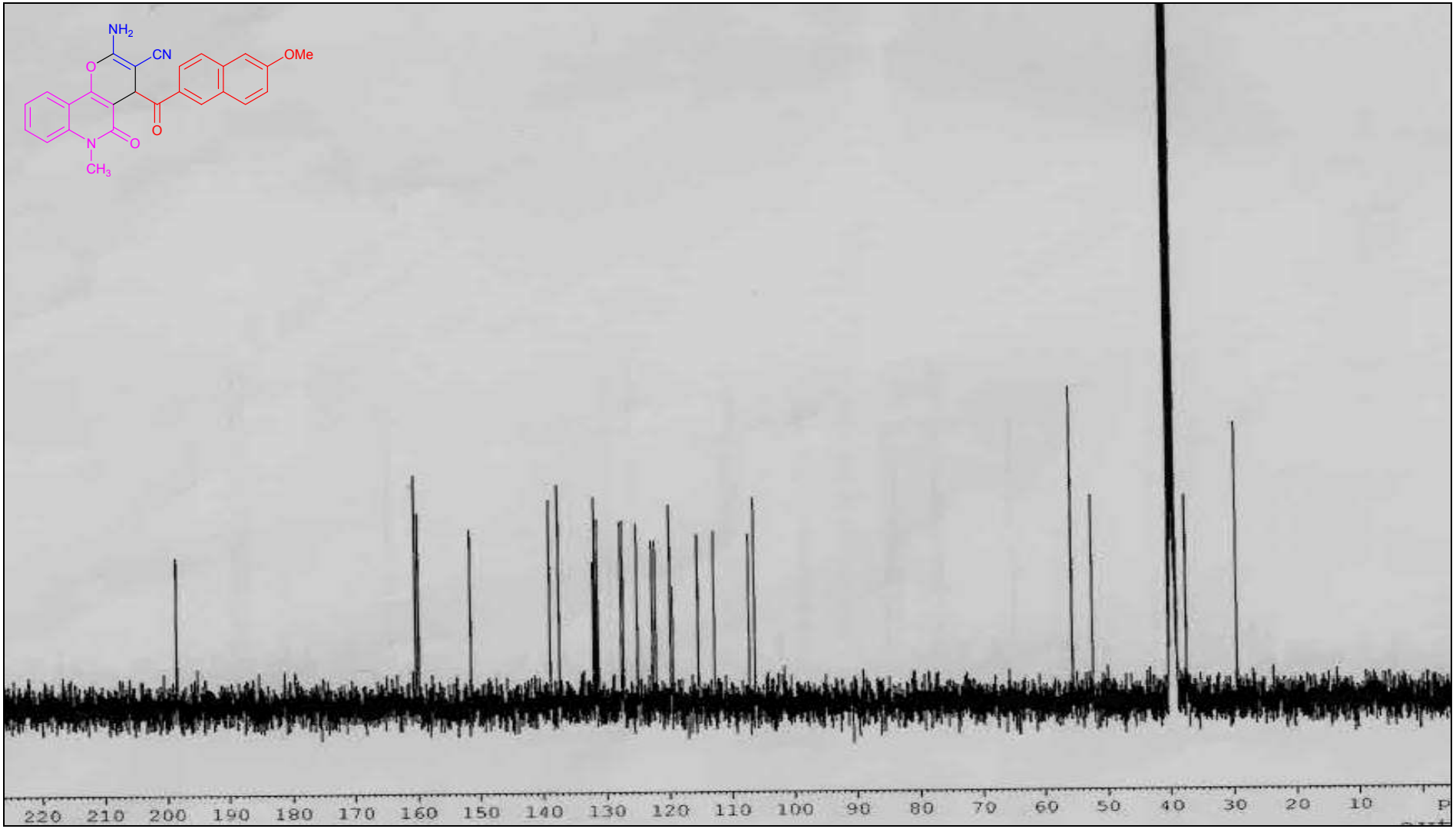

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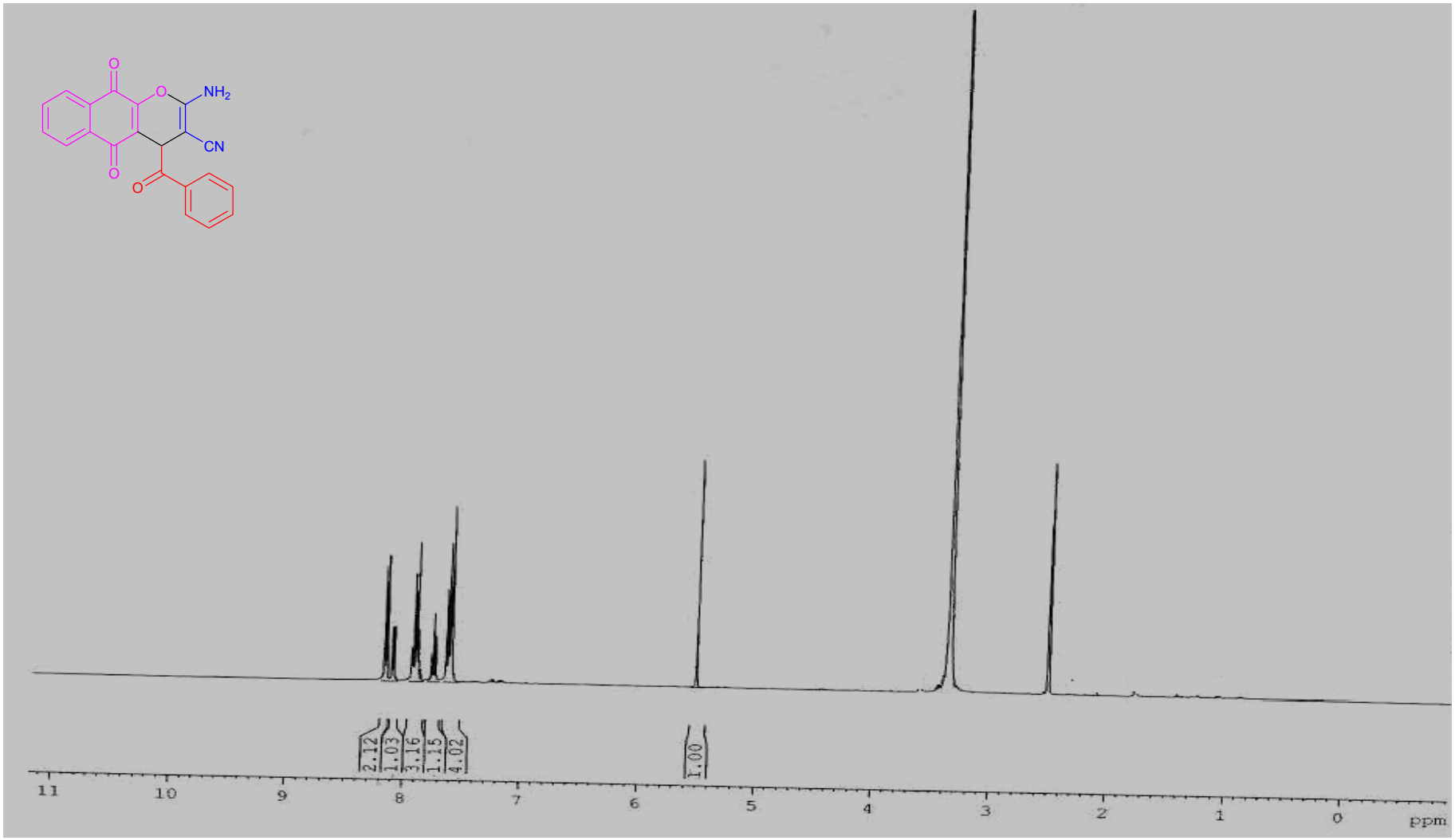

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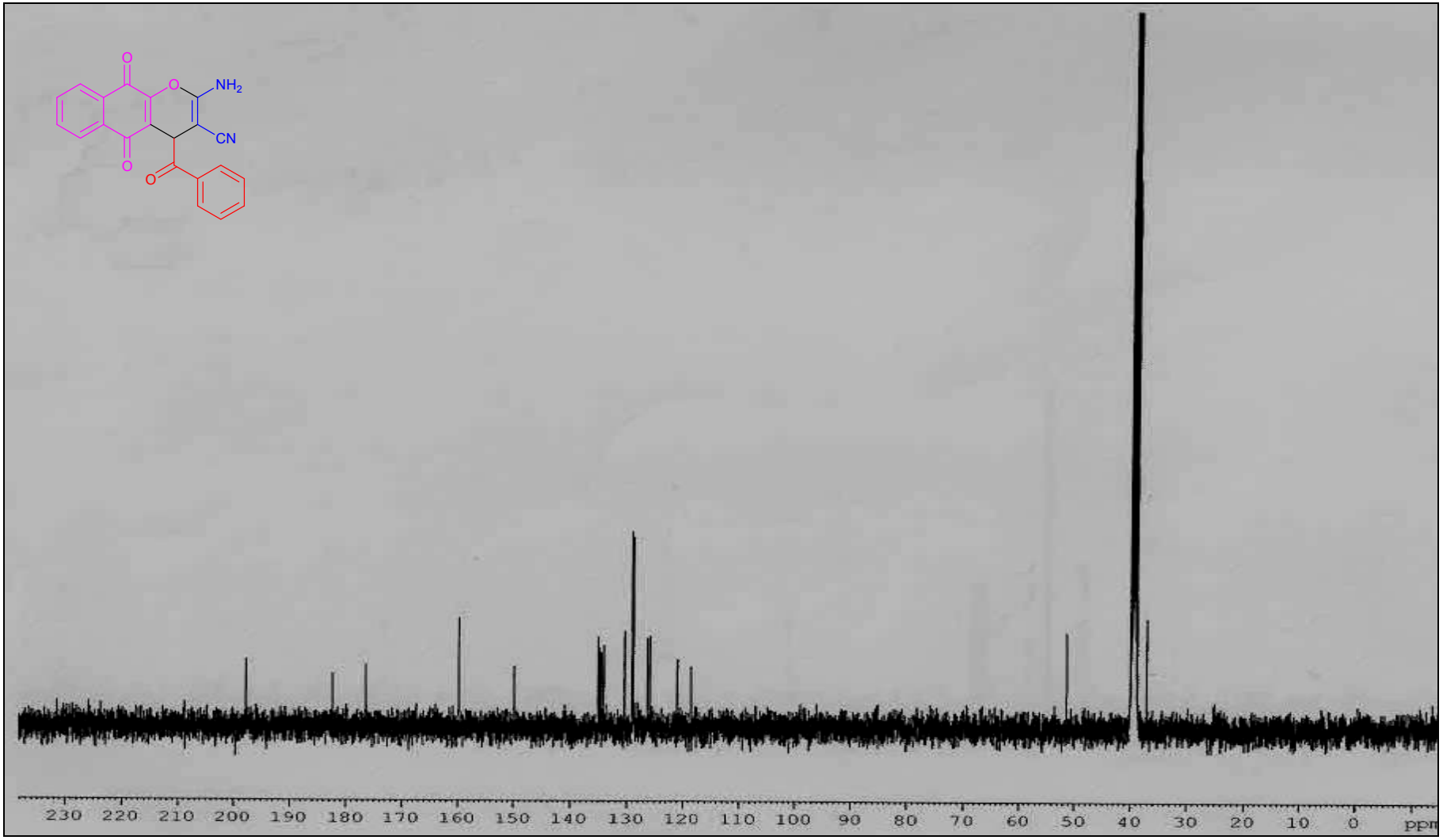

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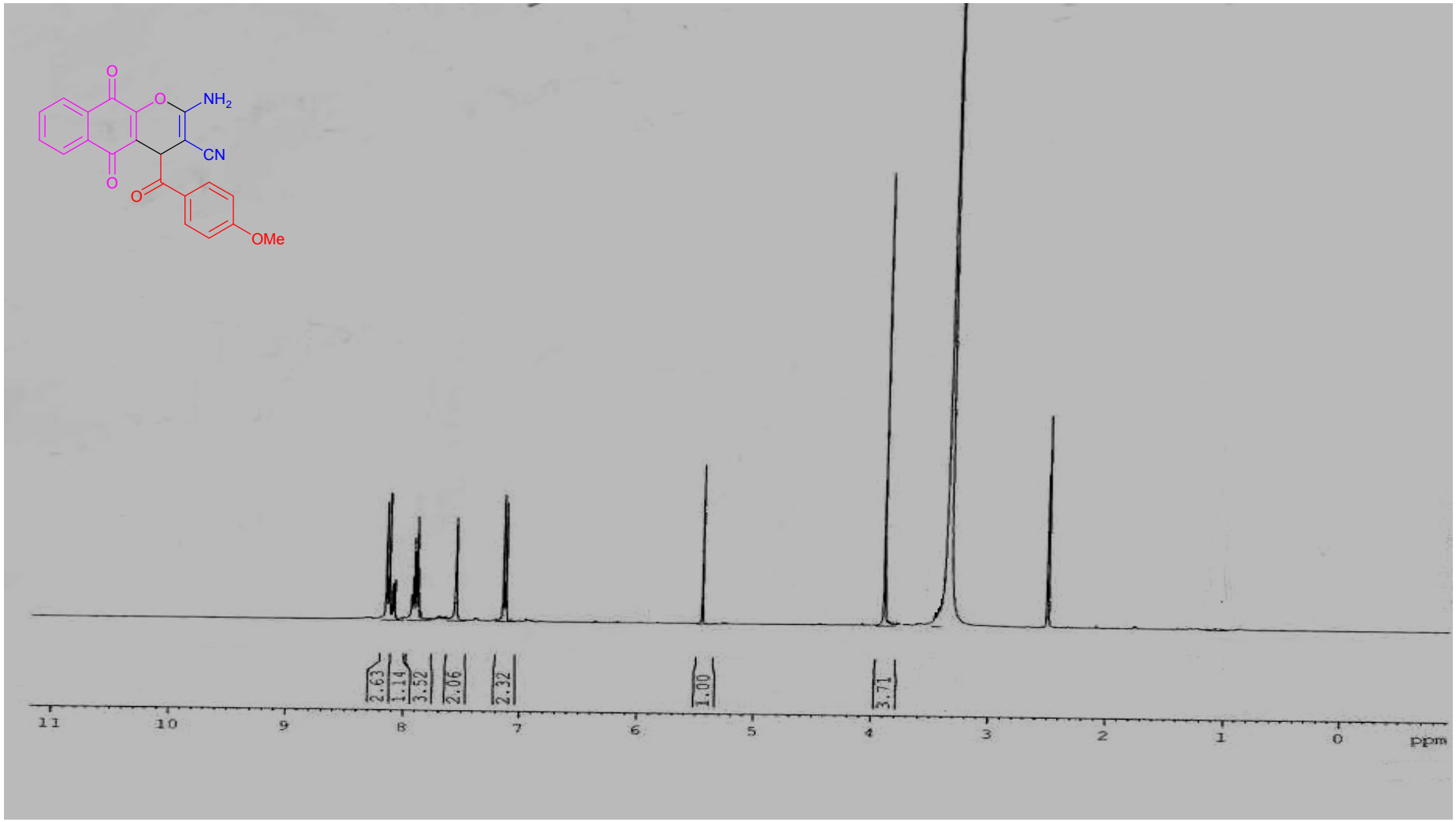

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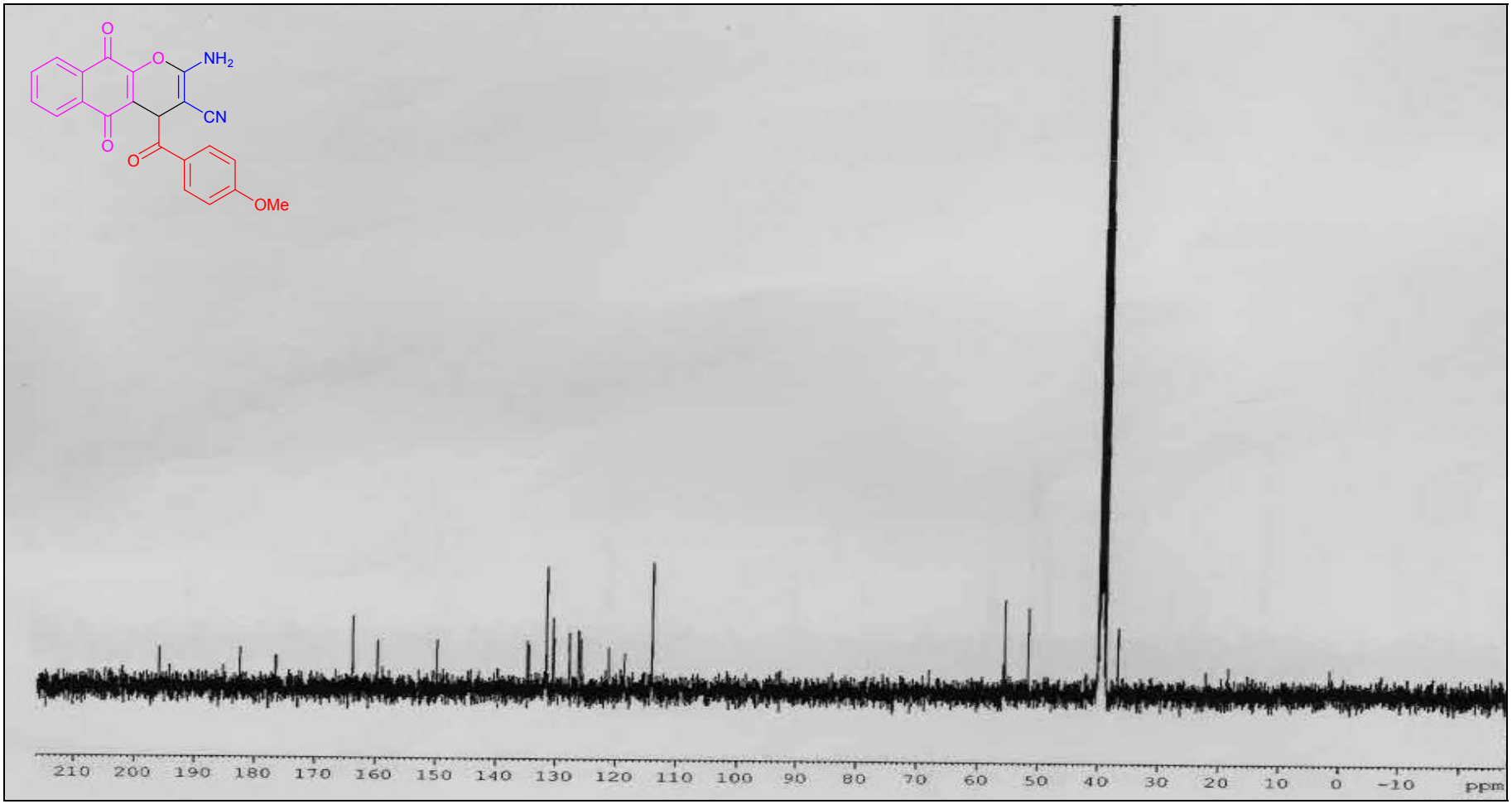

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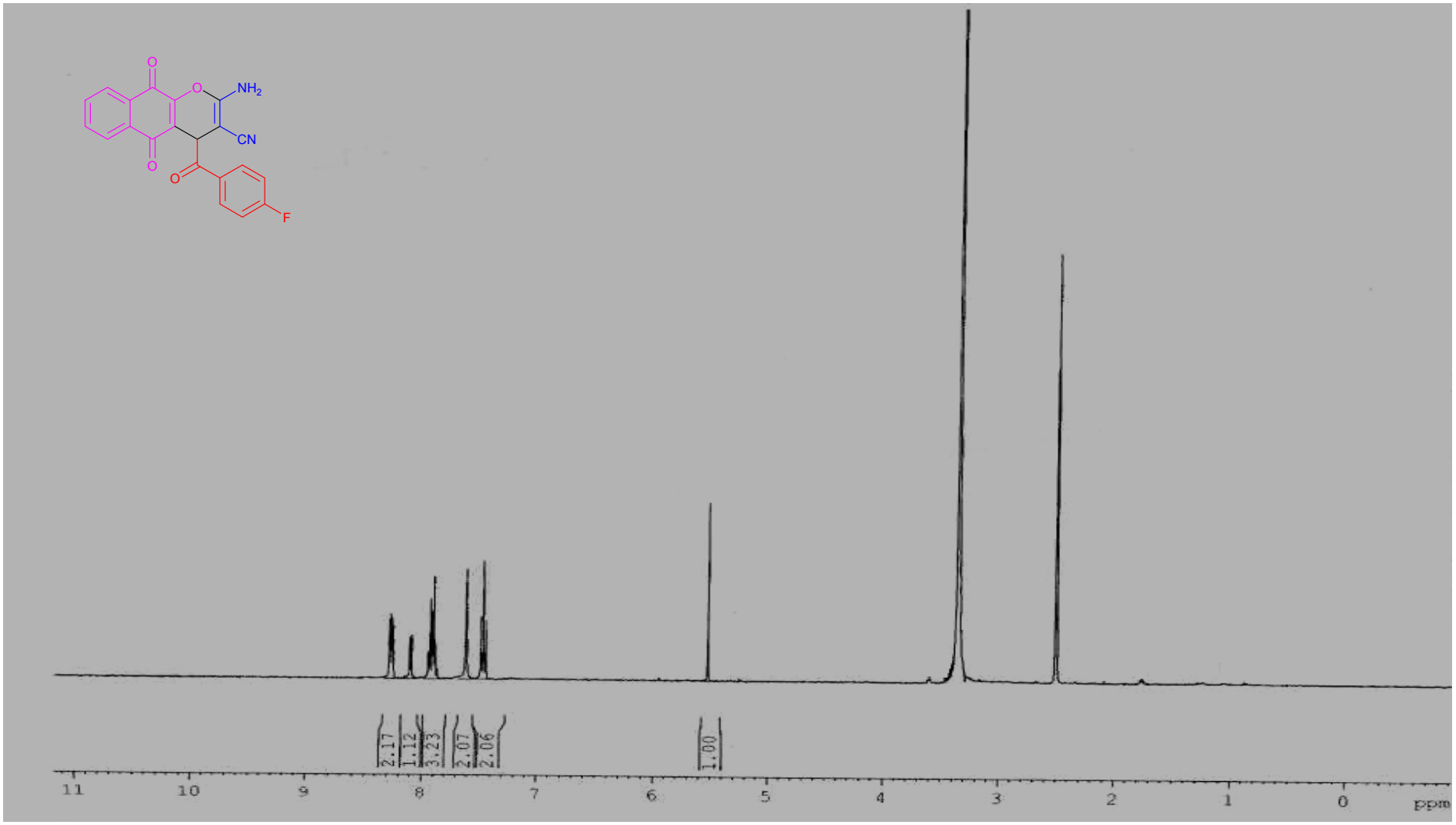

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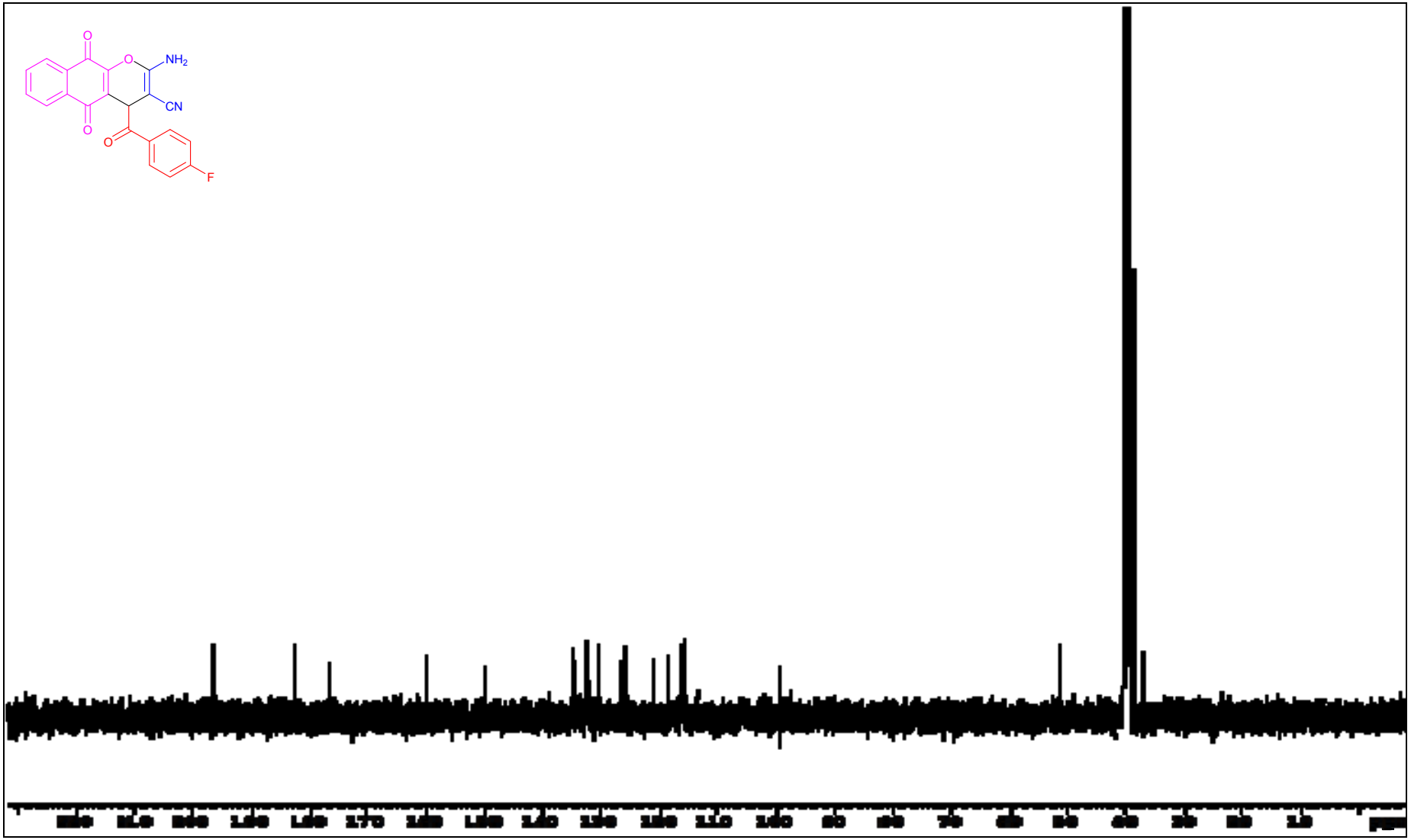

^{1}H NMR spectra of 2d

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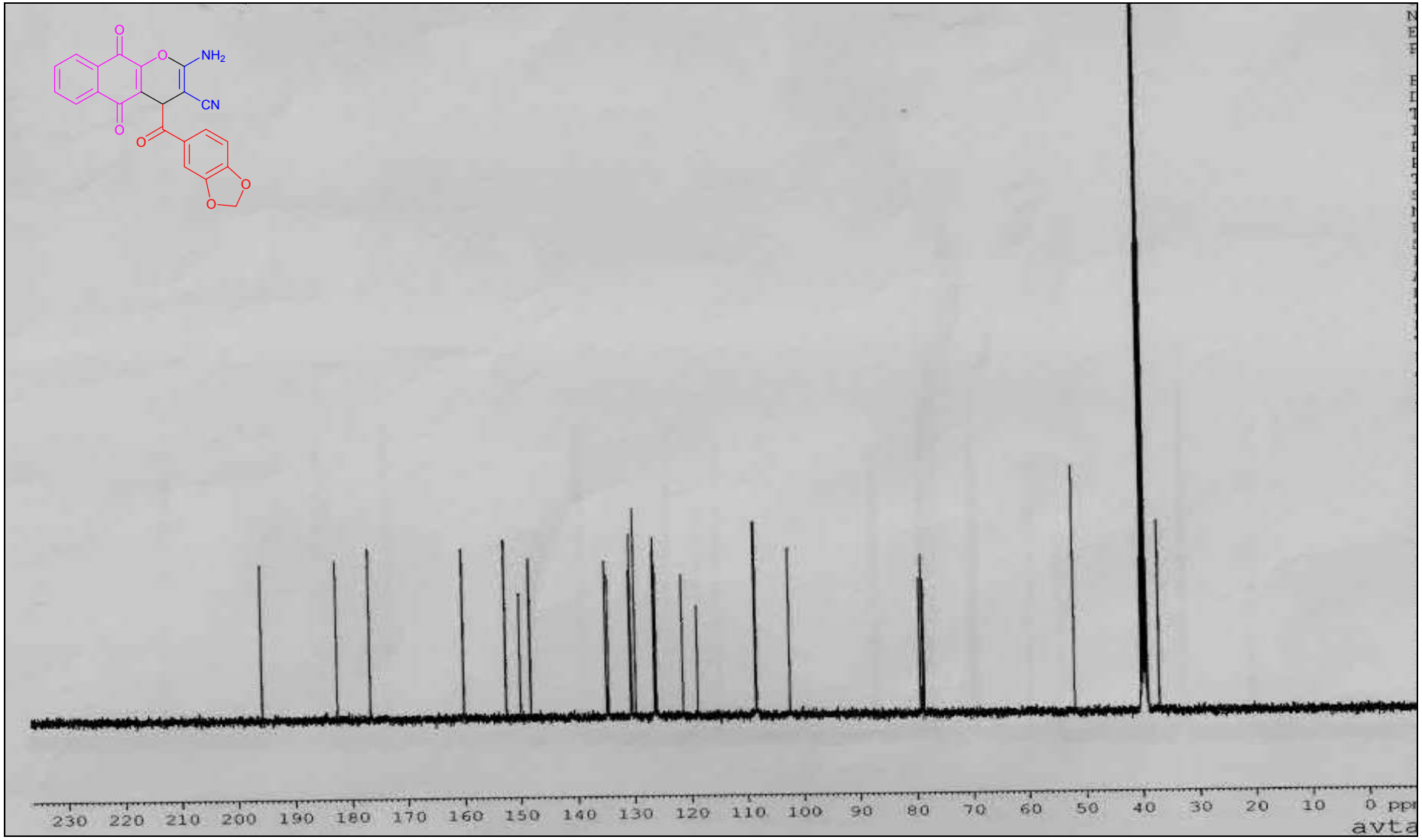

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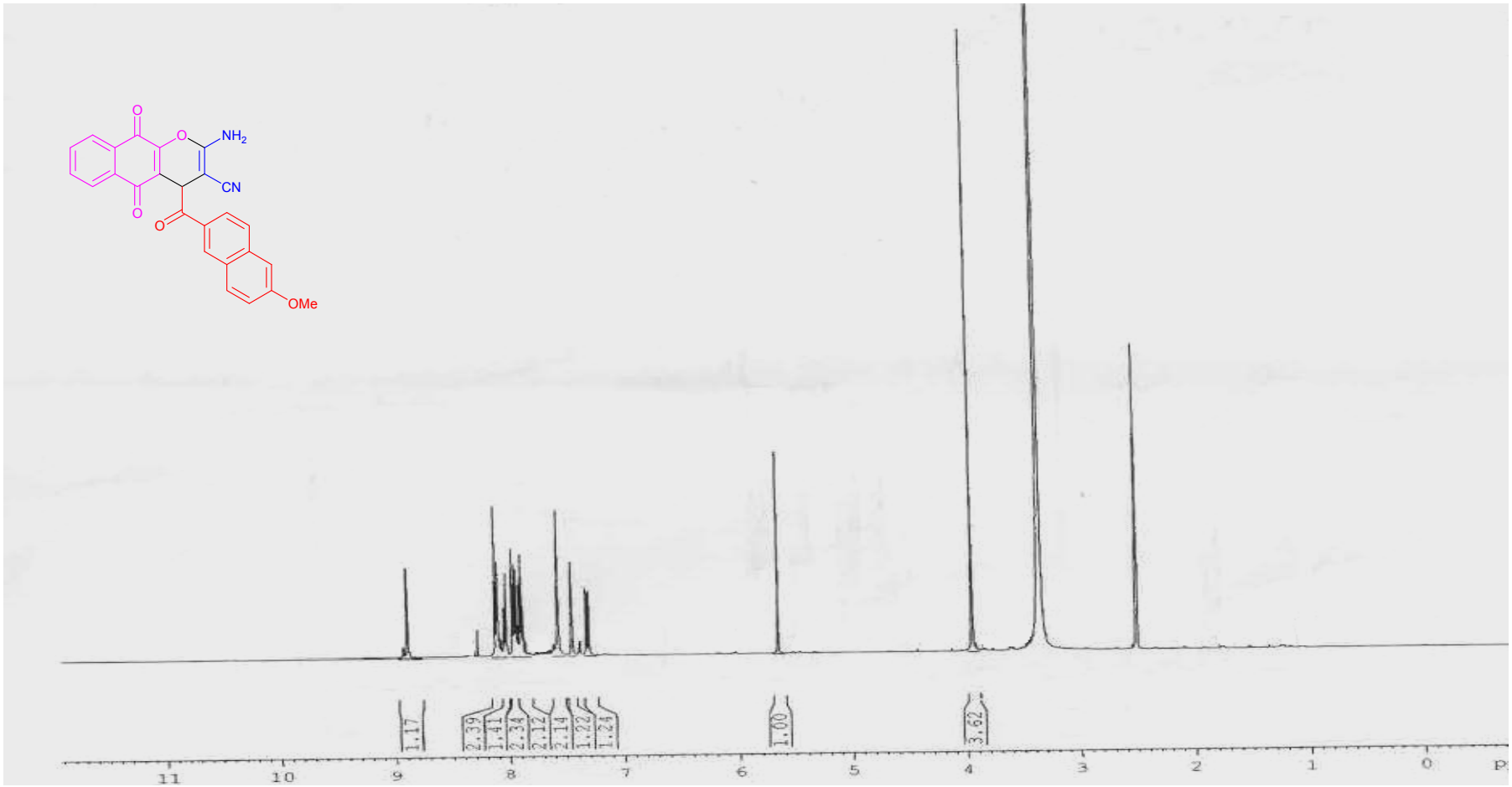

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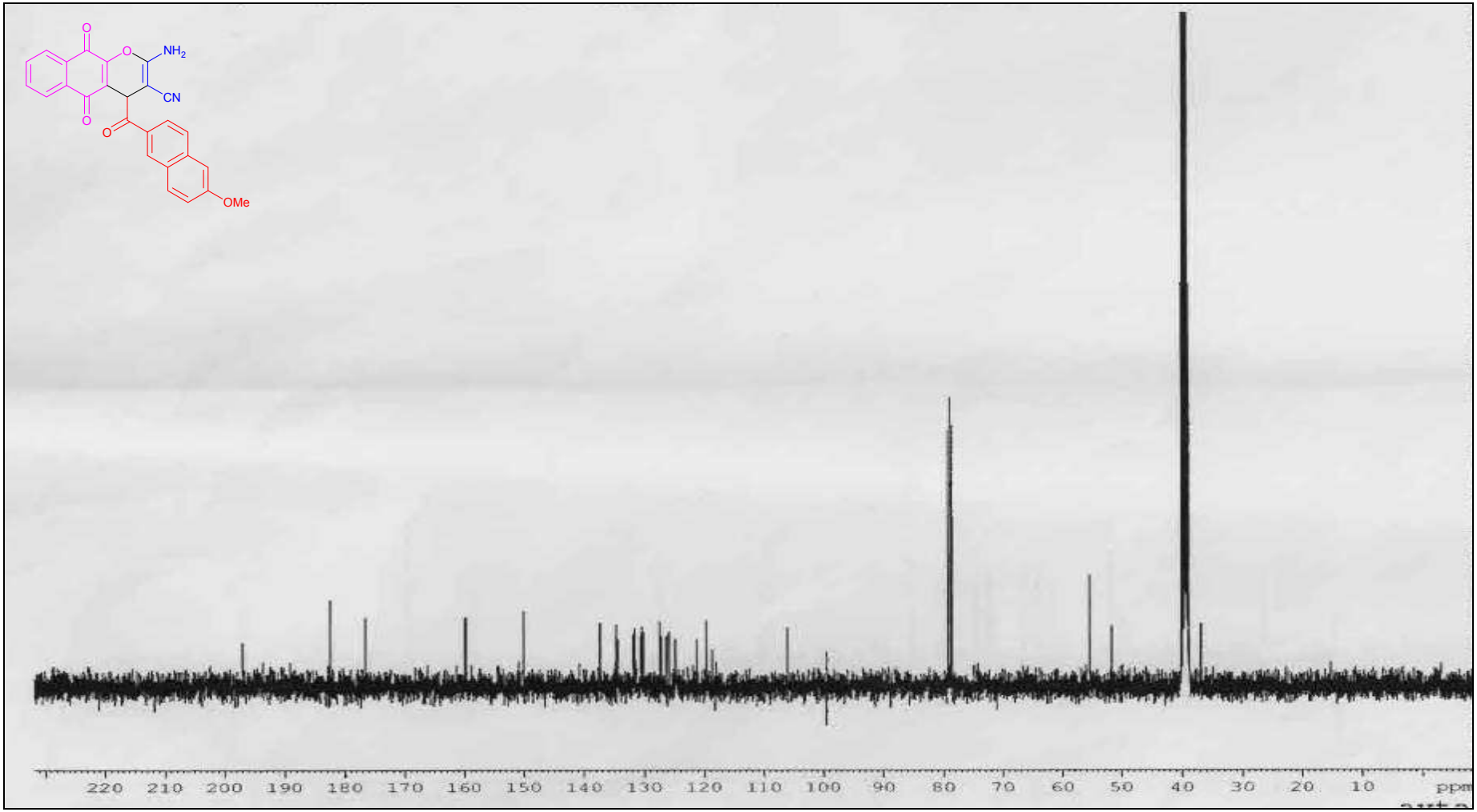

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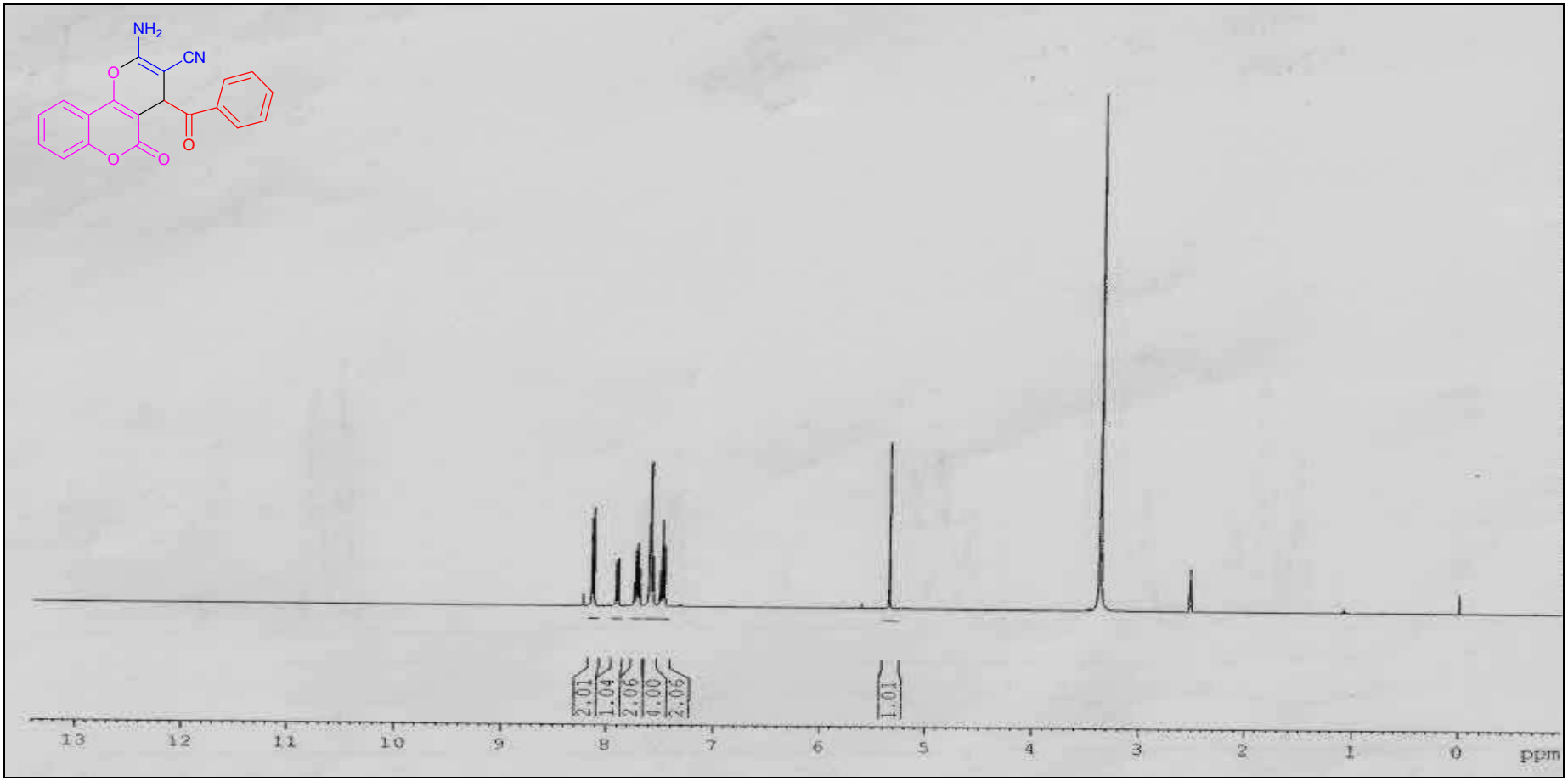

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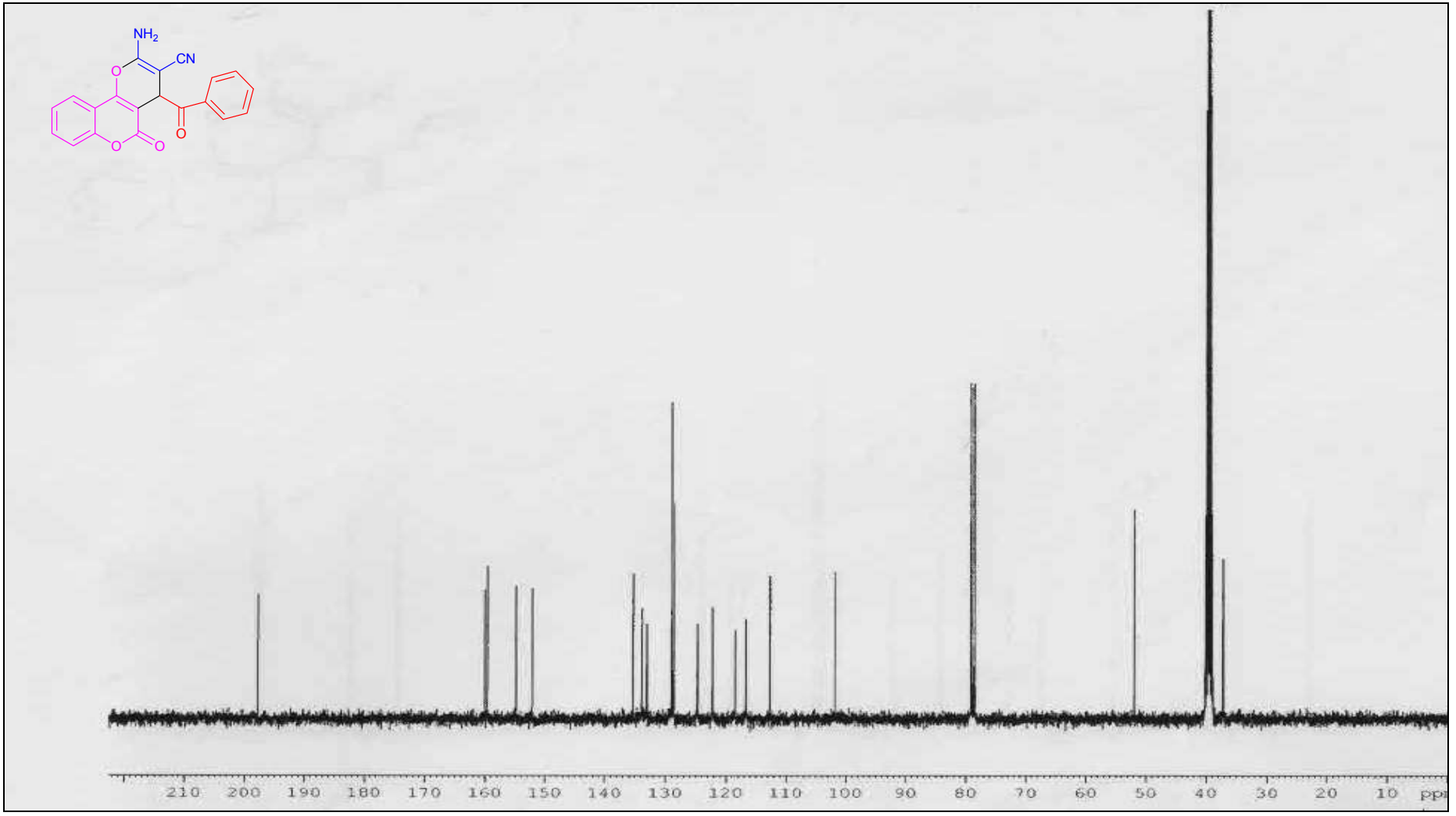

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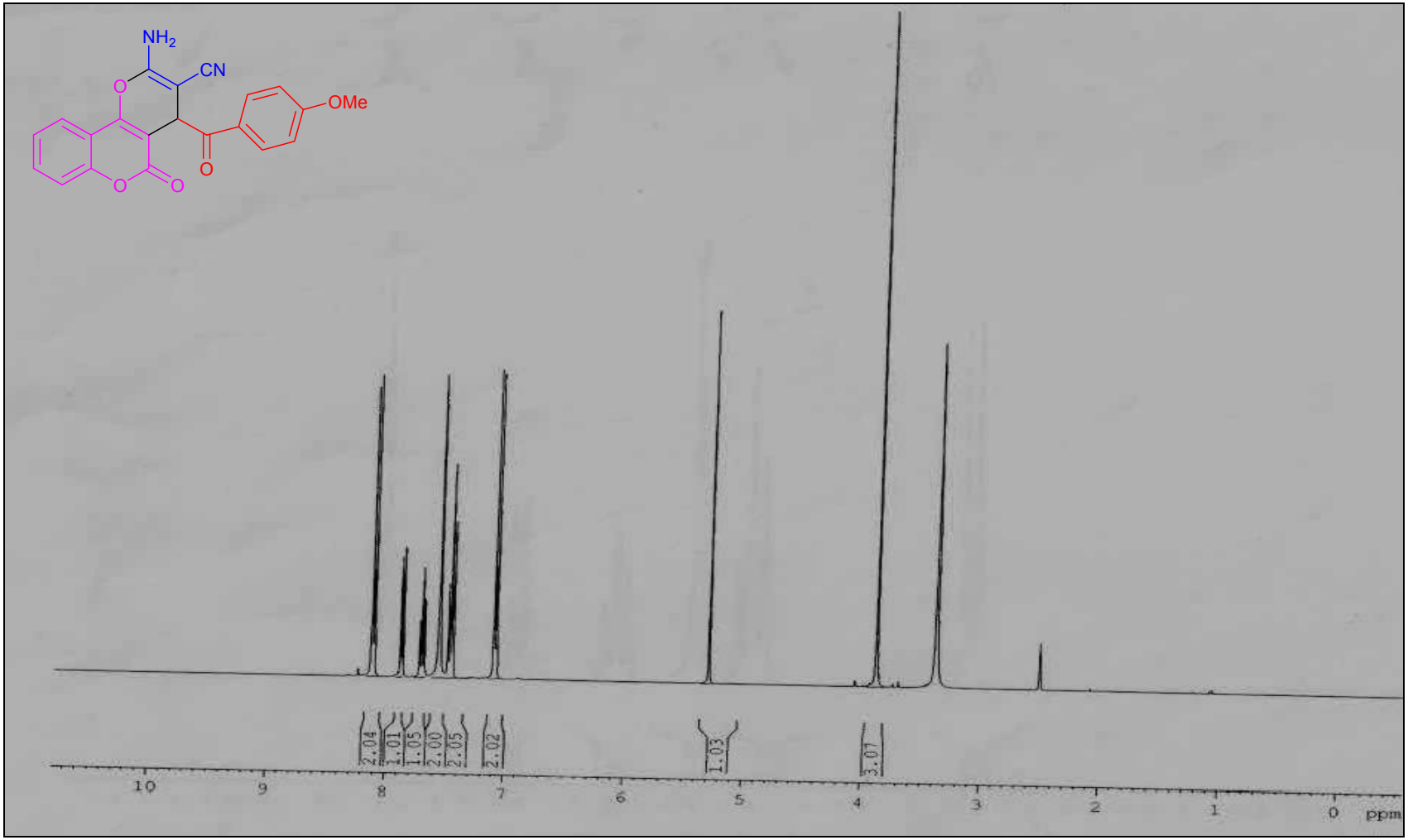

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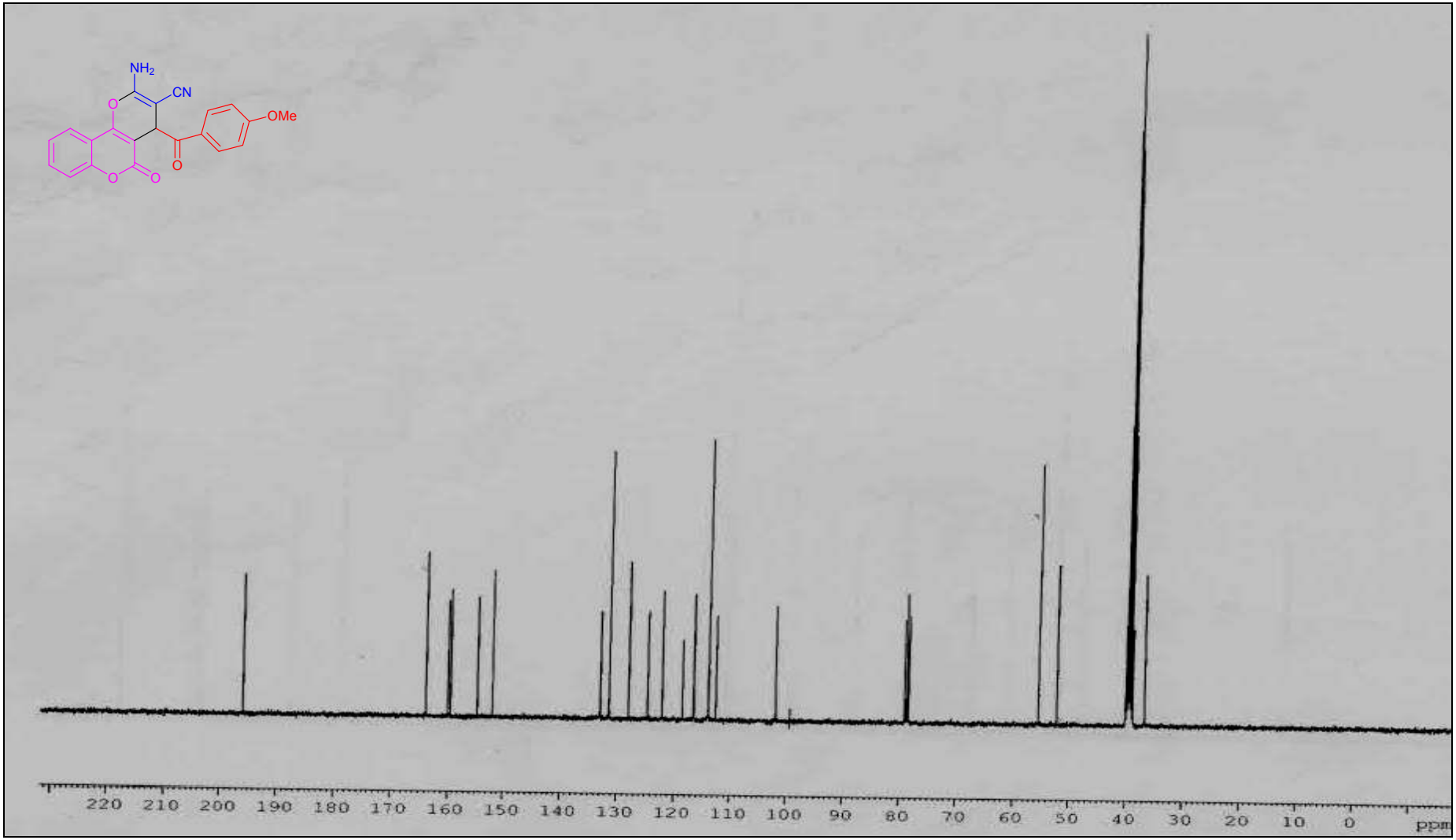

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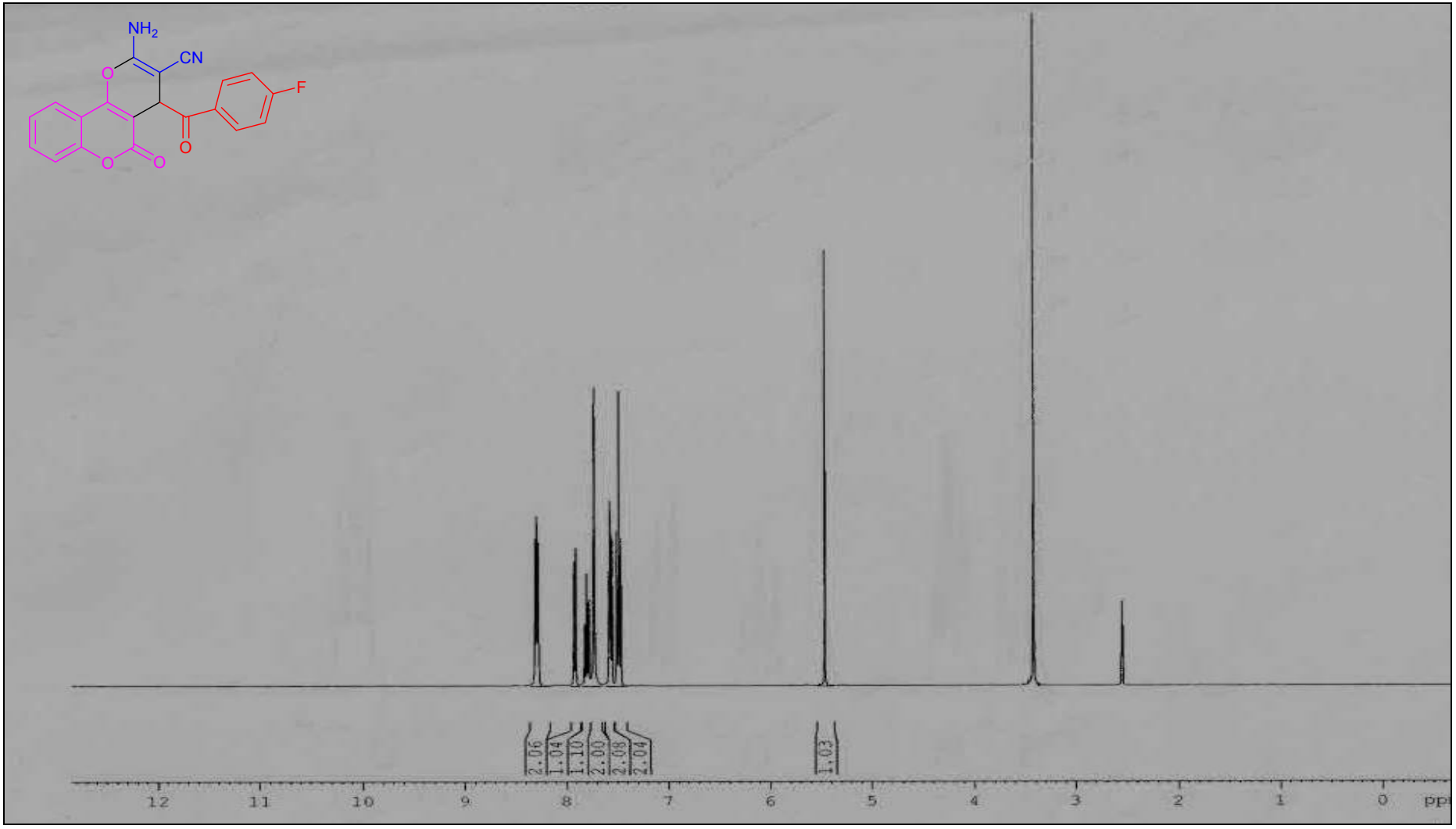

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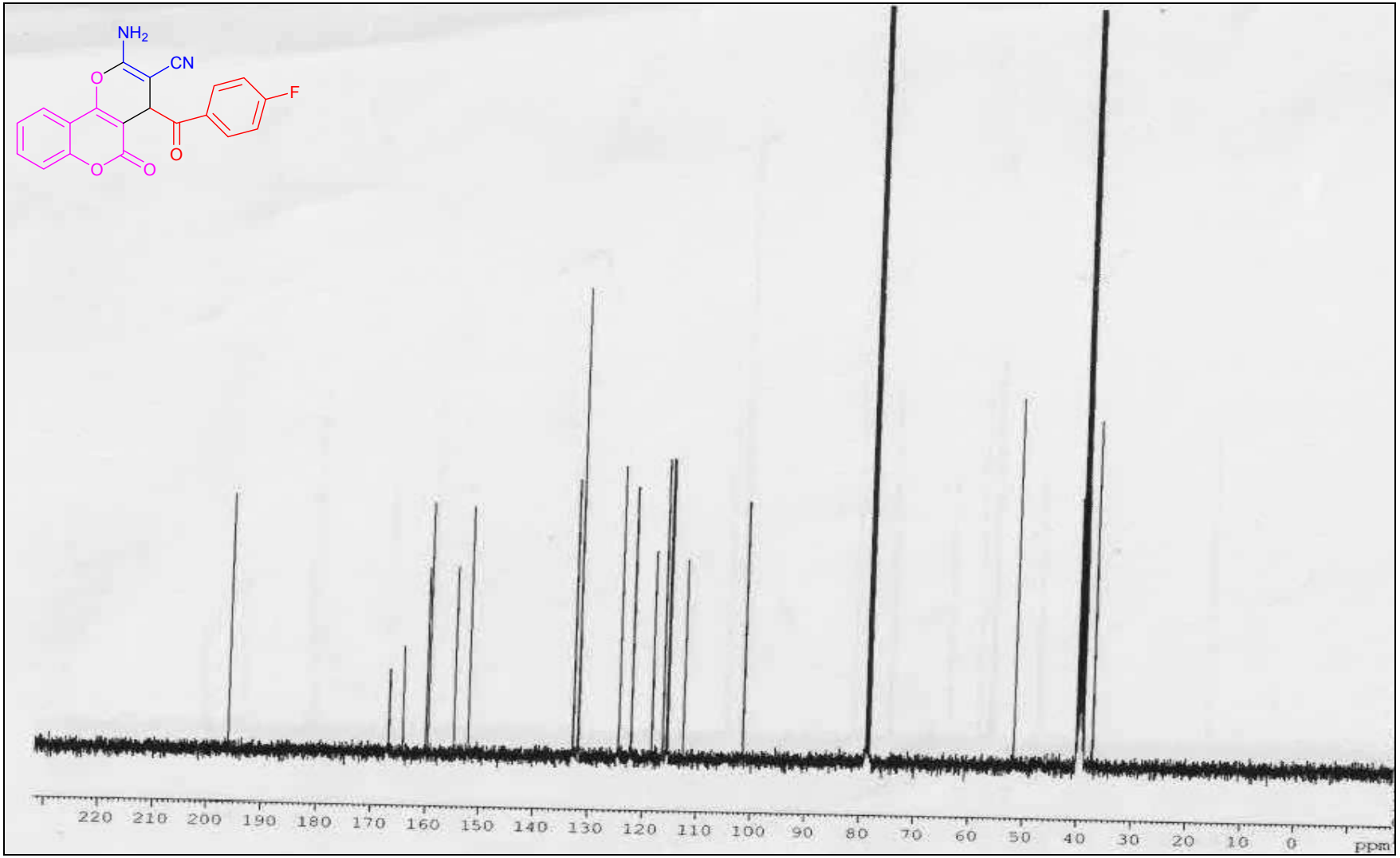

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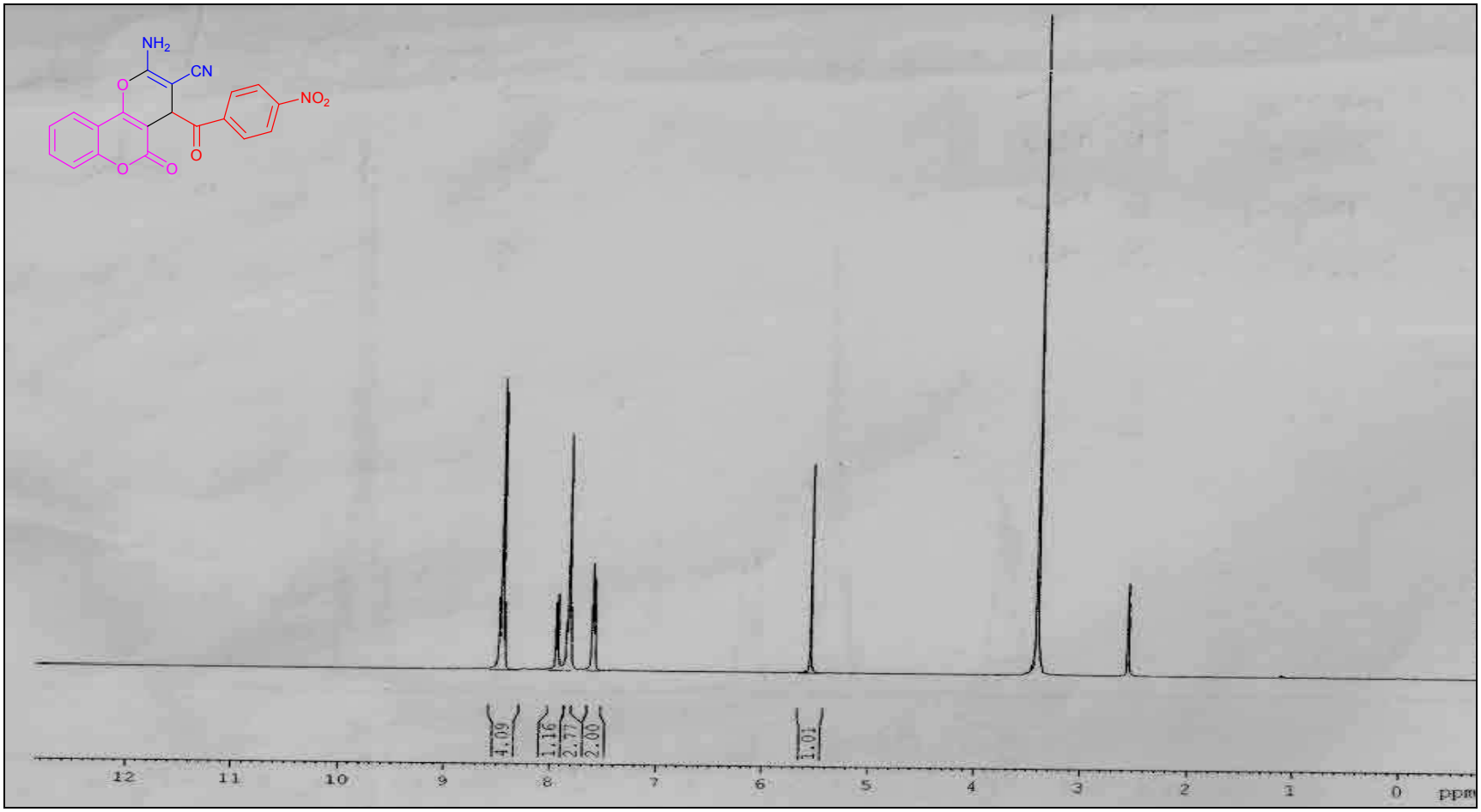

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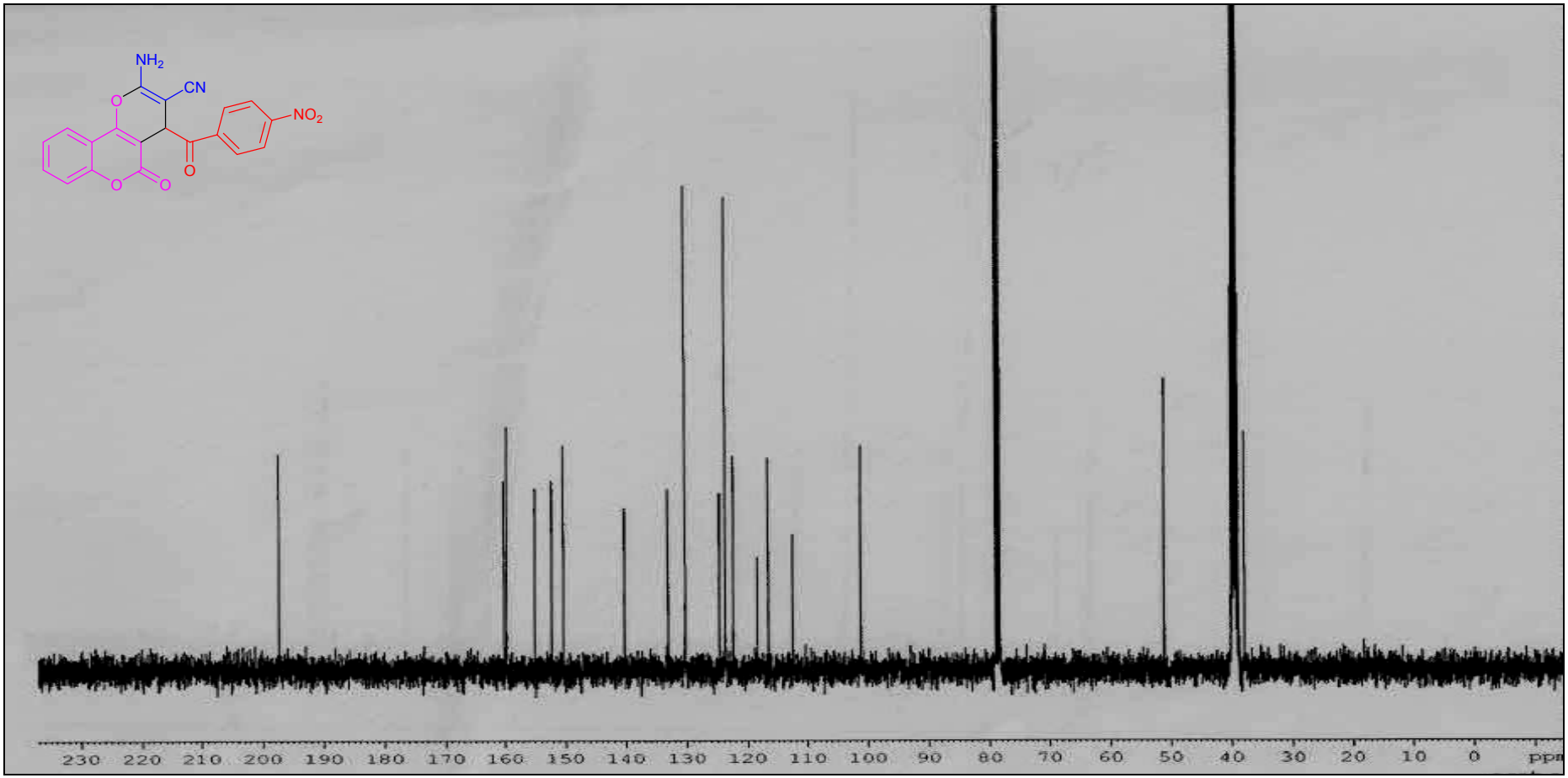

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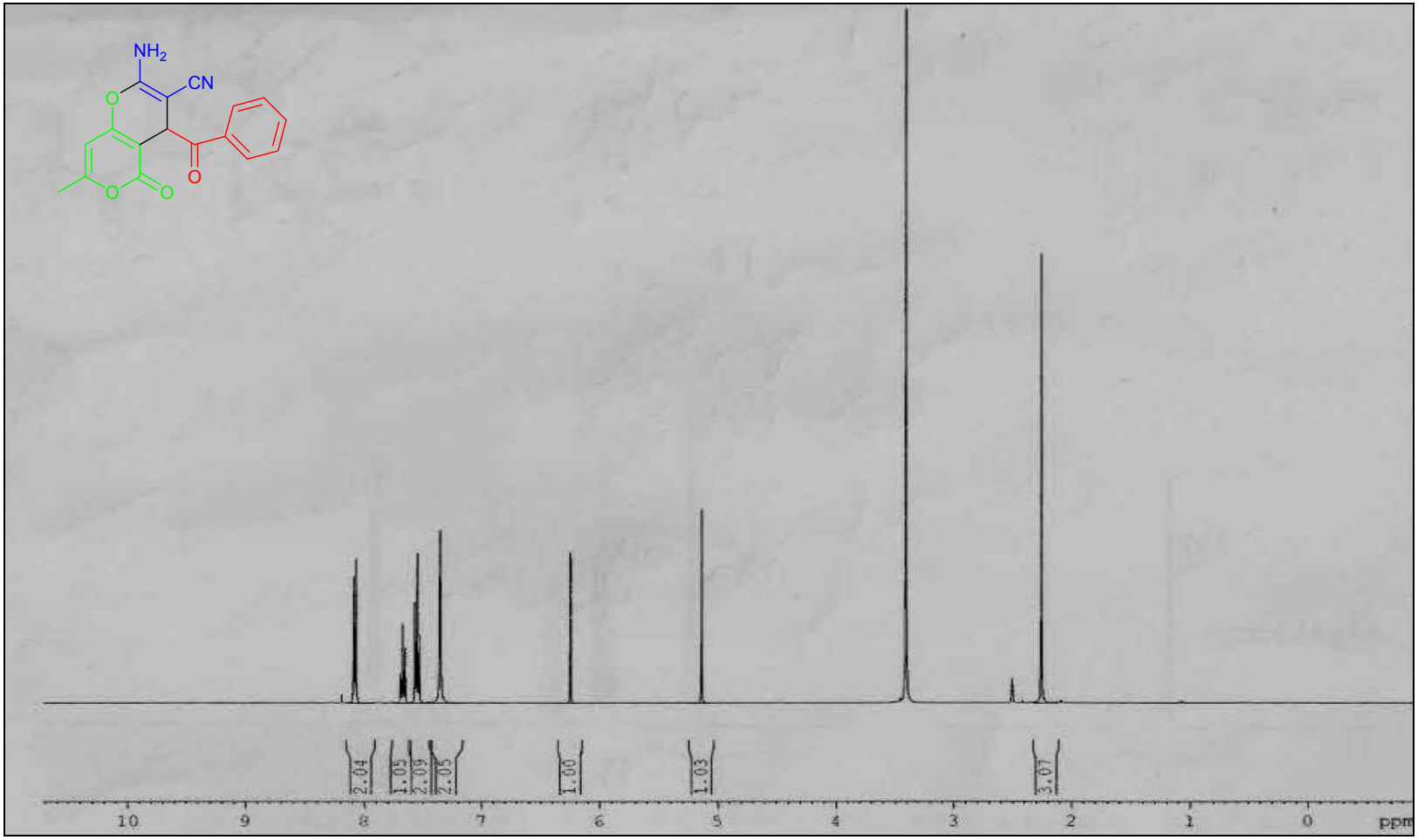

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