Supporting Information:

A simple and catalyst free one pot access to the pyrazolone fused 2,8-dioxabicyclo[3,3,1]nonanes

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Crystallographic data for X-ray Code: 3a

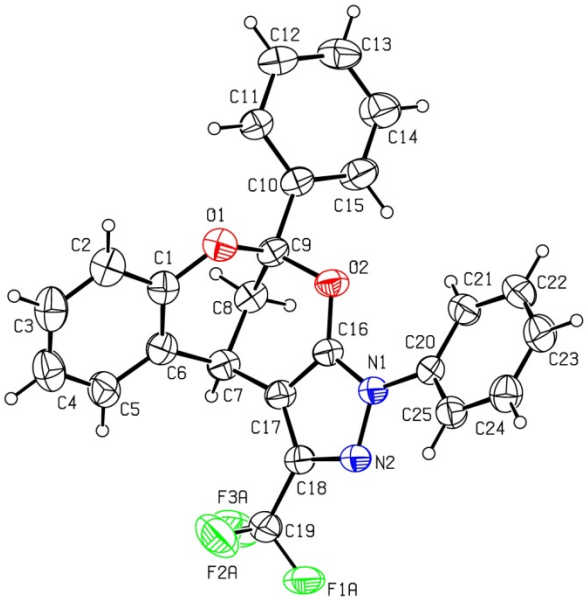


Figure caption: The molecular structure of **3a** with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radius. Only major component of the disordered $-CF_3$ group is shown for clarity.

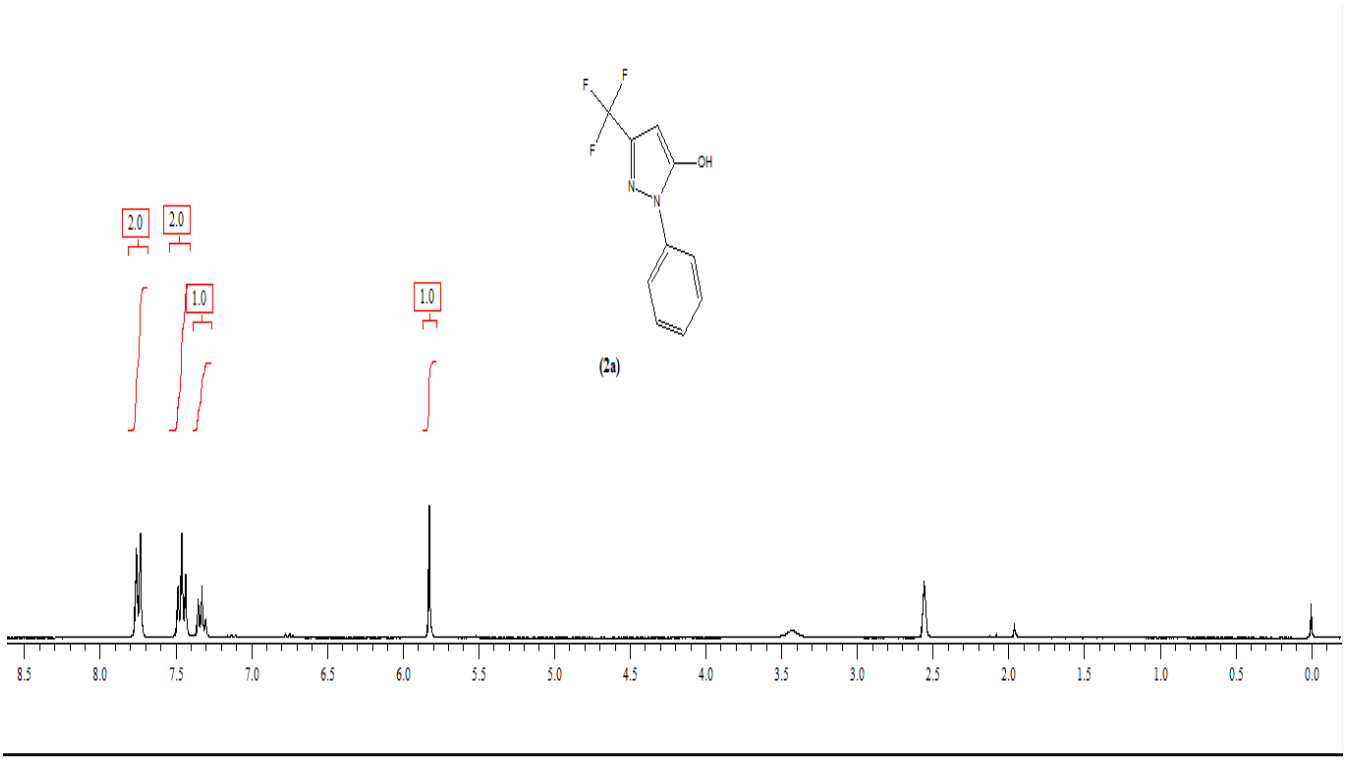
Crystal data for 3a: $C_{25}H_{17}F_3N_2O_2$, M = 434.41, colourless plate, 0.39 × 0.31 × 0.12 mm^3, monoclinic, space group $P2_1/n$ (No. 14), a = 12.4985(13), b = 9.3025(9), c = 18.0441(18) Å, β = 90.754(2)°, V = 2097.8(4) Å^3, Z = 4, D_c = 1.375 g/cm^3, F_{000} = 896, CCD area detector, Mo-Kα radiation, λ = 0.71073 Å, T = 293(2)K, $2\theta_{max}$ = 50.0°, 19513 reflections collected, 3691 unique (R_{int} = 0.0214), Final *GooF* = 1.021, *R1* = 0.0435, *wR2* = 0.1149, *R* indices based on 2984 reflections with I >2σ(I) (refinement on F^2), 316 parameters, μ = 0.106 mm^{-1}. CCDC 994173 contains supplementary crystallographic data for the structure. These data can be obtained free of charge at www.ccdc.cam.ac.uk/conts/retrieving.html [or from the Cambridge Crystallographic Data Centre (CCDC), 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44(0) 1223 336 033; email: deposit@ccdc.cam.ac.uk].

Supporting Information

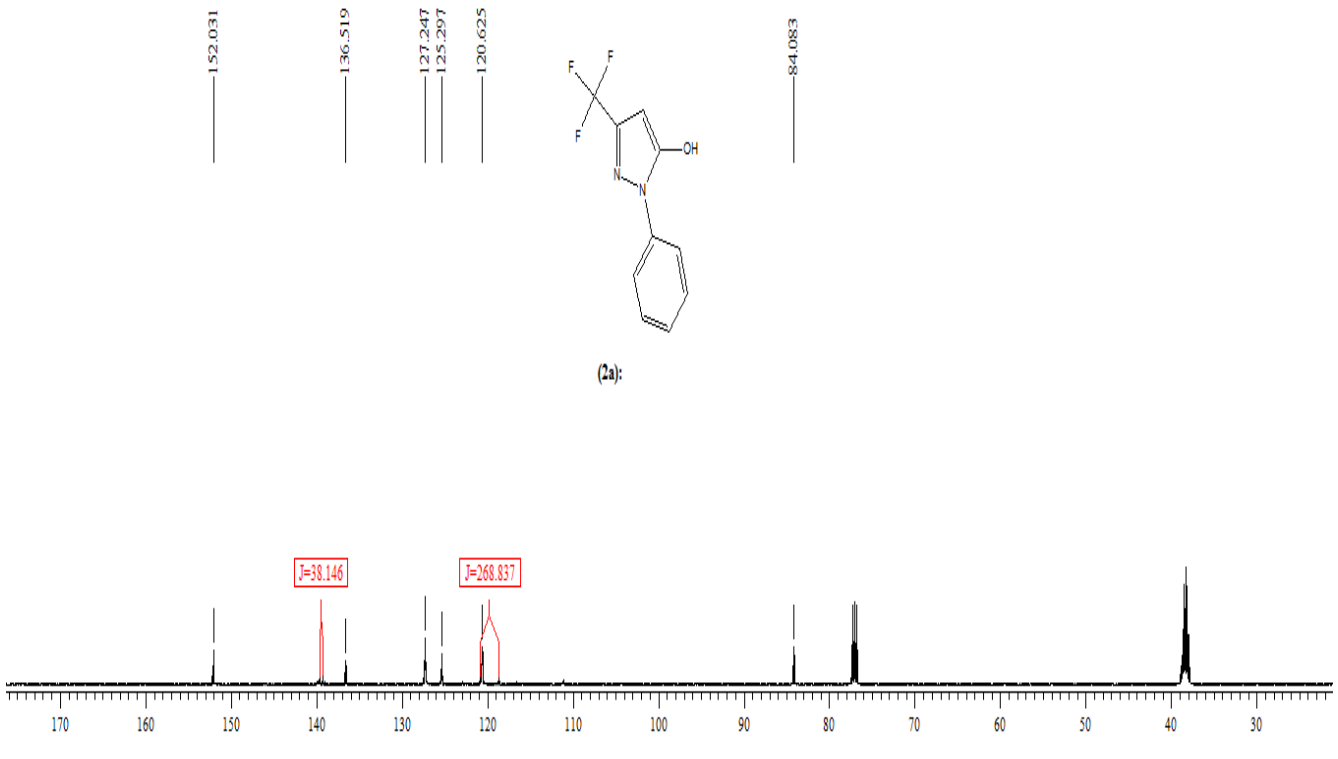
X-ray data for the compound were collected at room temperature using a Bruker Smart Apex CCD diffractometer with graphite monochromated MoKα radiation (λ=0.71073Å) with ω-scan method.[1] Preliminary lattice parameters and orientation matrices were obtained from four sets of frames. Unit cell dimensions were determined using 6829 reflections for 3a data. Integration and scaling of intensity data were accomplished using SAINT program.[1] The structures were solved by Direct Methods using SHELXS97[2] and refinement was carried out by full-matrix least-squares technique using SHELXL97.[2] Anisotropic displacement parameters were included for all non-hydrogen atoms. All H atoms were positioned geometrically and treated as riding on their parent C atoms, with C-H distances of 0.93--0.97 Å, and with U_{iso}(H) = 1.2U_{eq} (C) or 1.5U_{eq} for methyl atoms. N bound H atoms have been located from the difference Fourier map. Three fluorine atoms of $-CF_3$ group were disordered over two sites

[F1A, F2A and F3A with site occupancy of 0.55 & F1B, F2B and F3B with site occupancy of 0.45]. PART and ISOR instructions were used for disorder modeling of the fluorine atoms and DFIX was used for restraining all the C–F bond distances to 1.300 Å with e.s.d 0.002 in the crystal structure refinement.

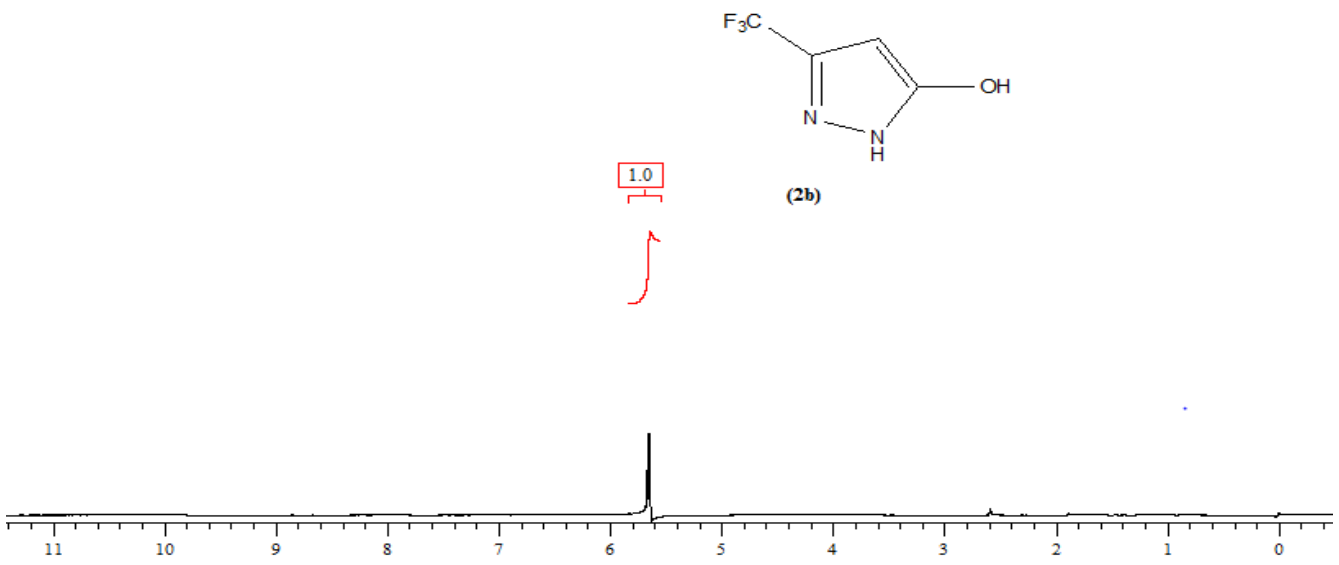
1. SMART & SAINT. Software Reference manuals. Versions 6.28a & 5.625, Bruker Analytical X-ray Systems Inc., Madison, Wisconsin, U.S.A., 2001.
2. Sheldrick, G. M. SHELXS97 and SHELXL97, Programs for crystal structure solution and refinement; University of Gottingen: Germany, 1997



^{1}H NMR (500 MHz) spectrum of **2a** in $CDCl_3$ + DMSO-d_6



^{13}C NMR (125 MHz) spectrum of **2a** in $CDCl_3$ + DMSO-d_6



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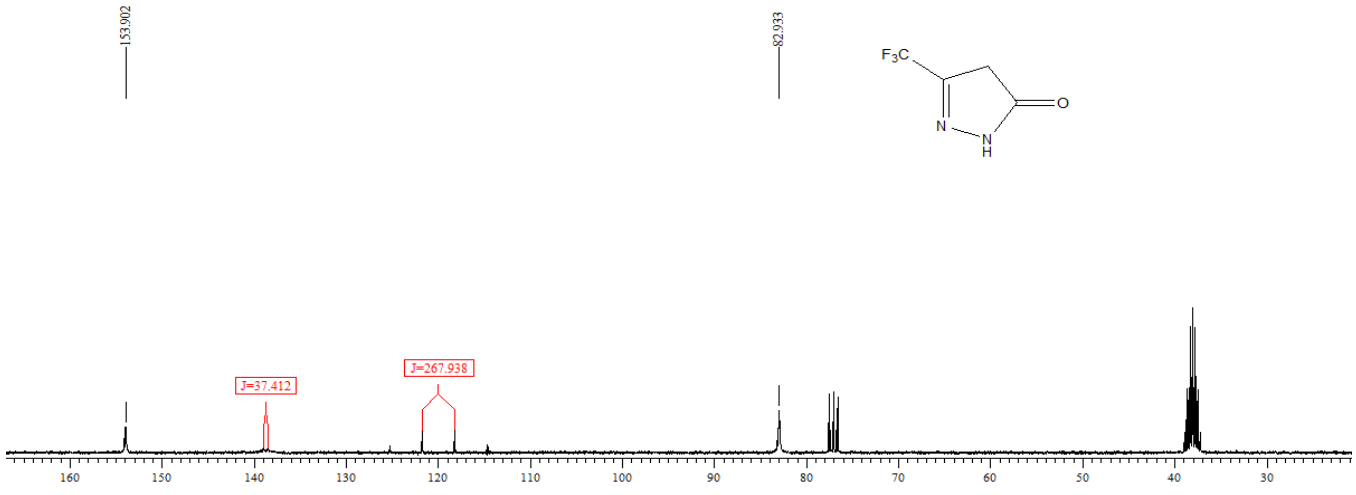

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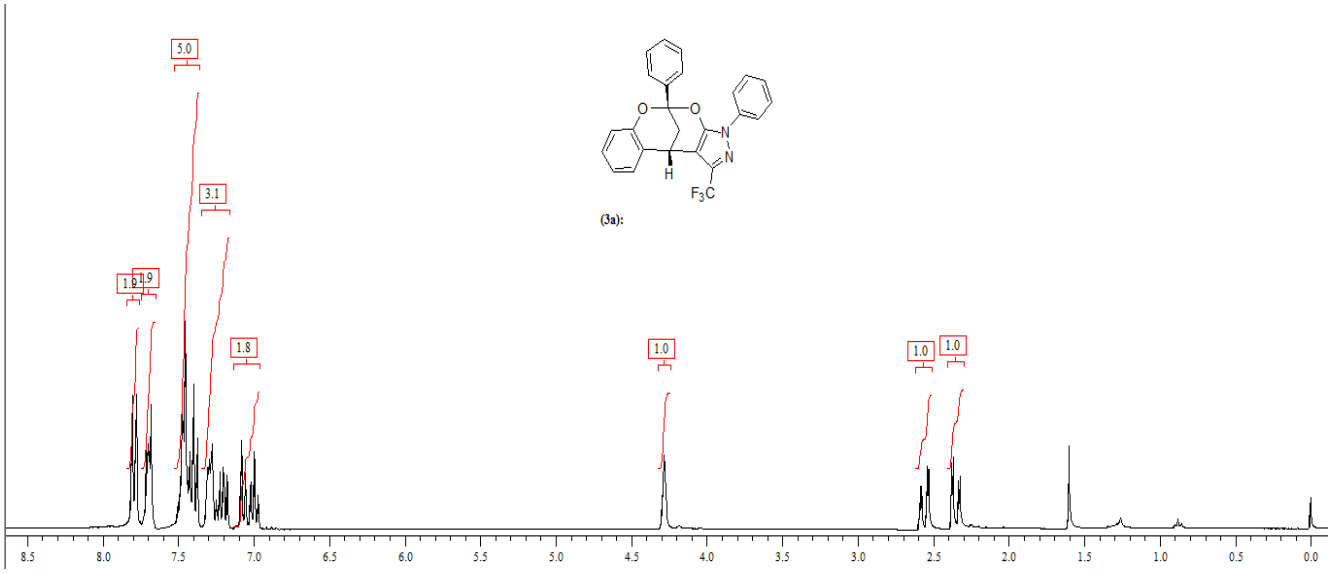

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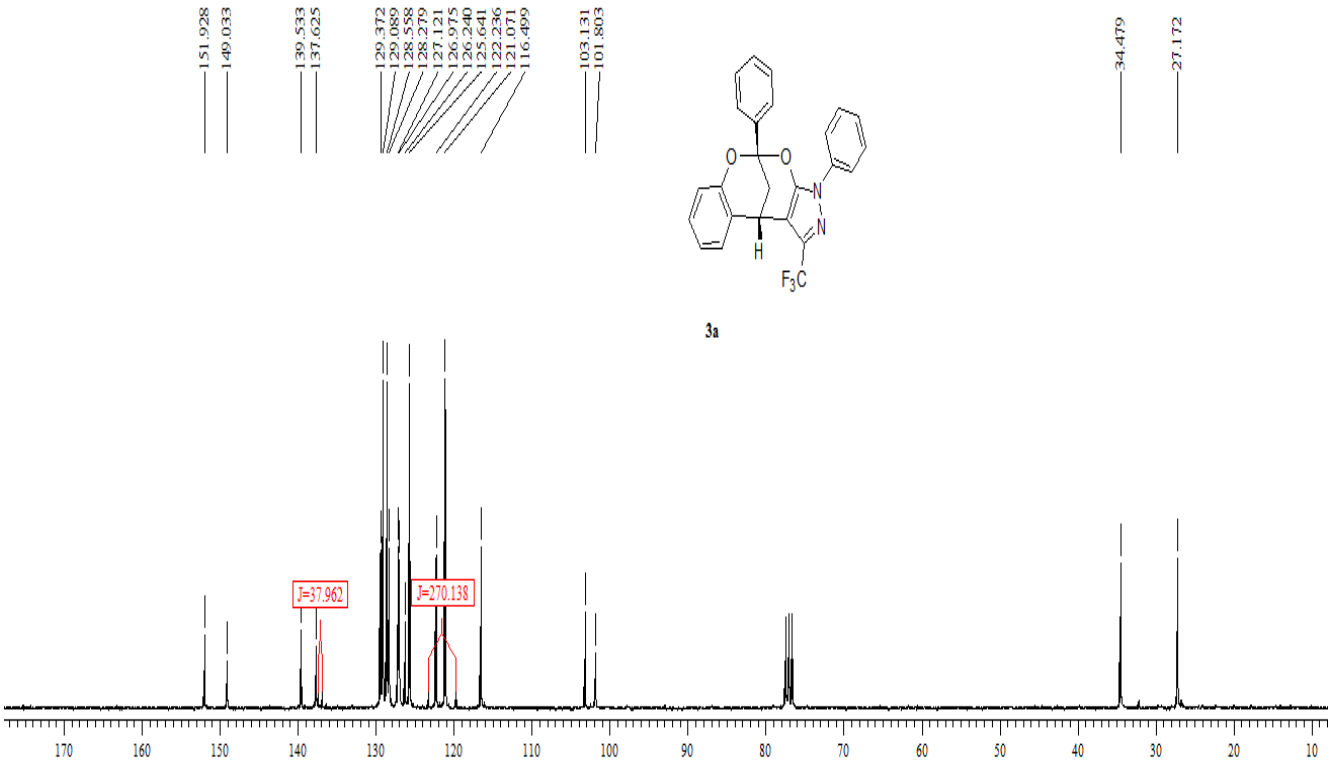

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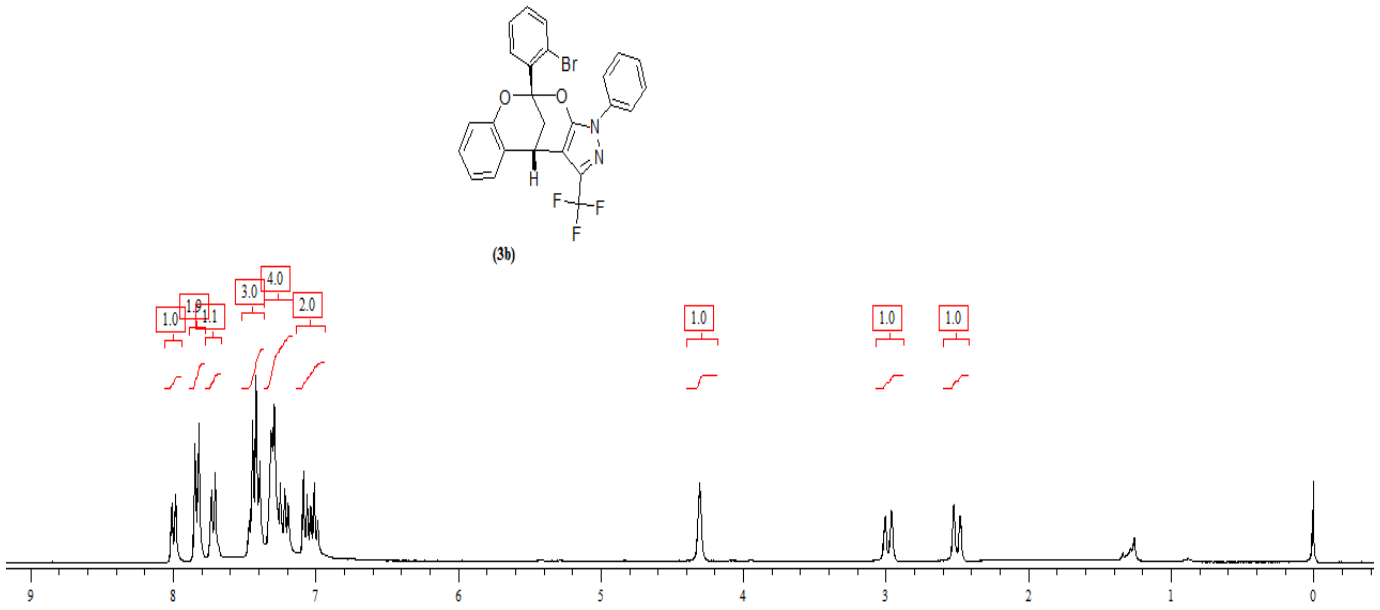

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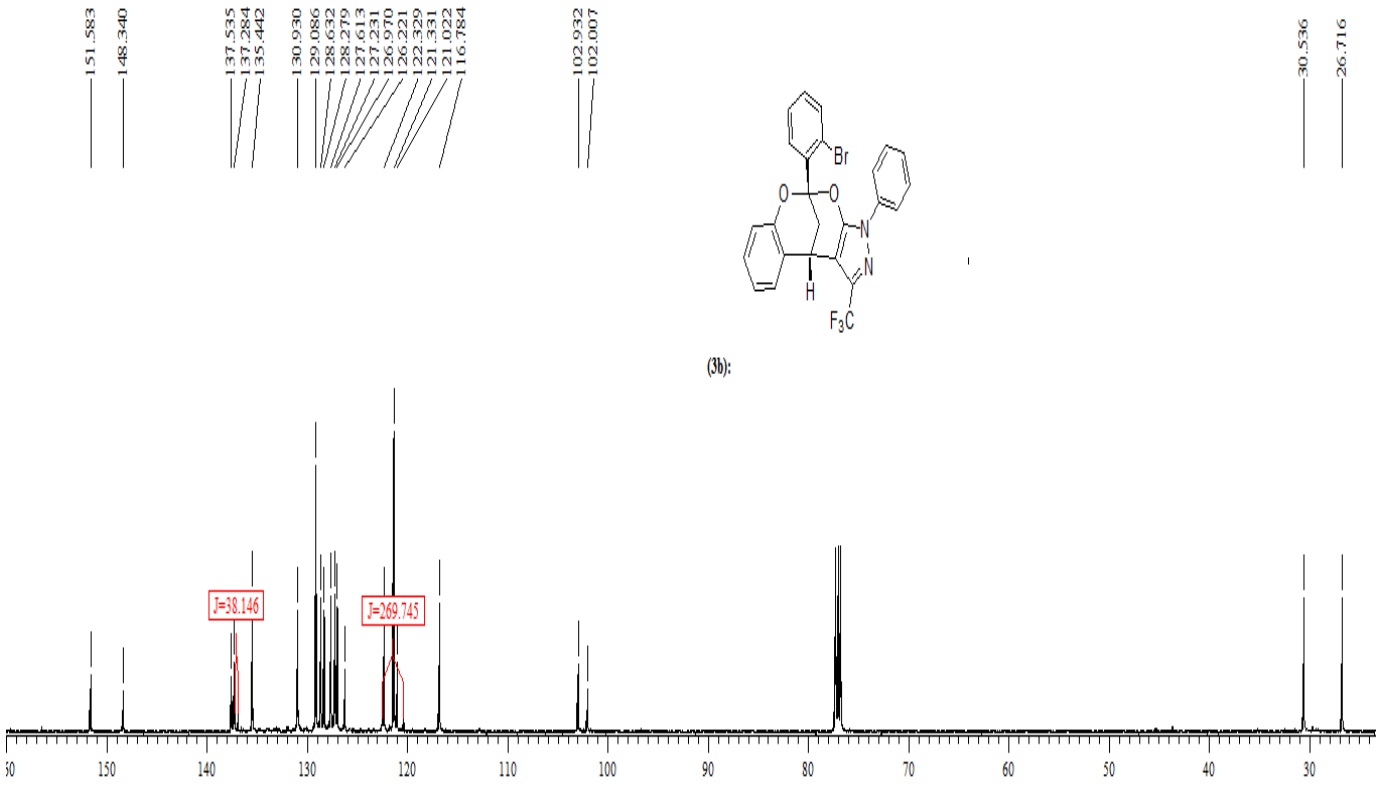

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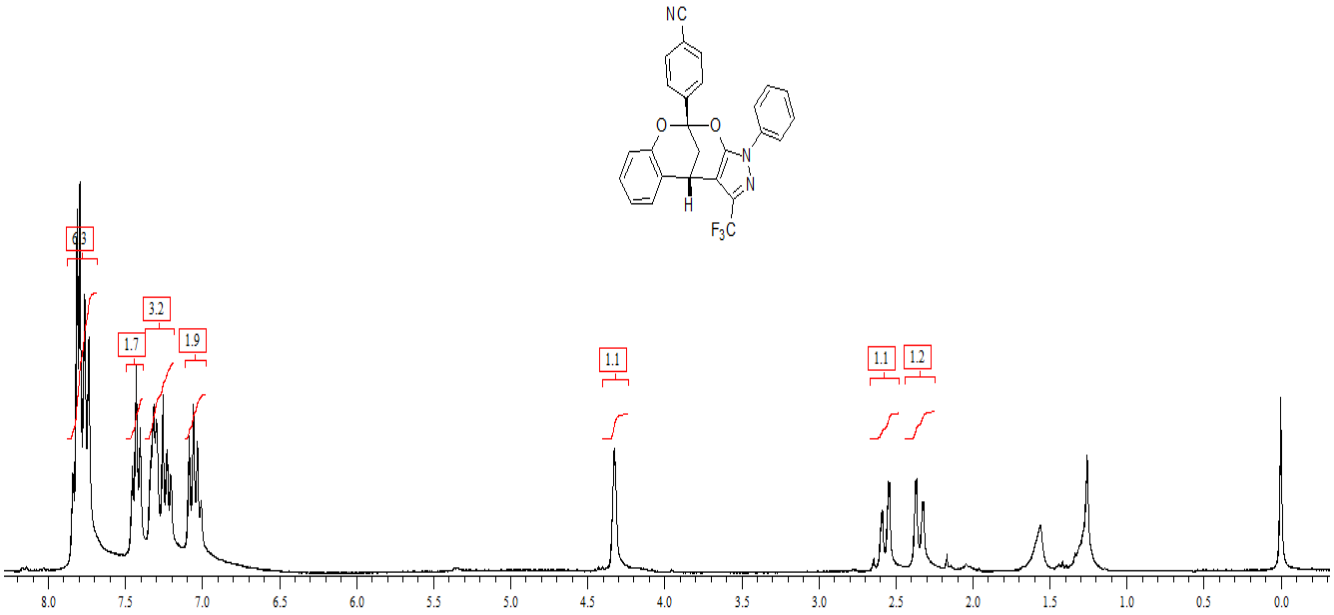

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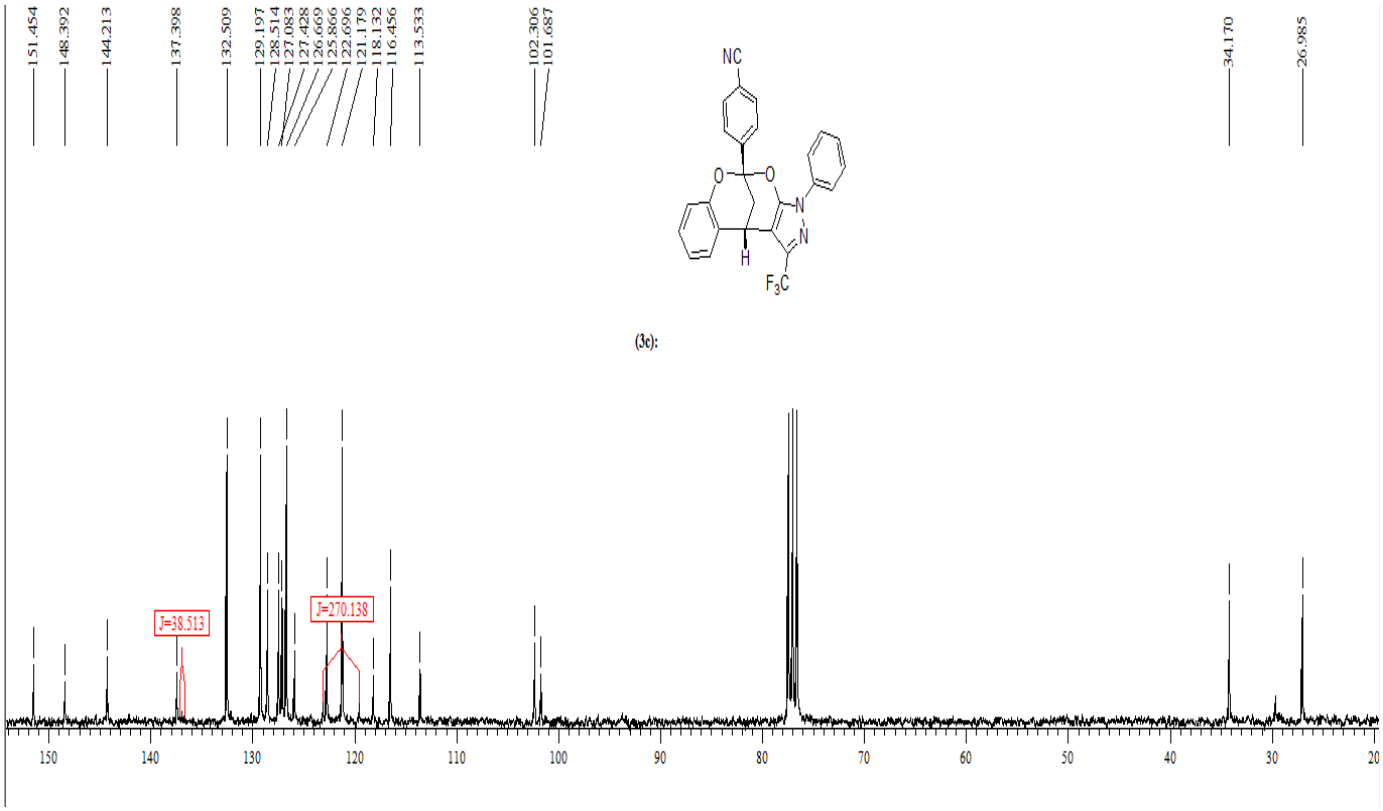

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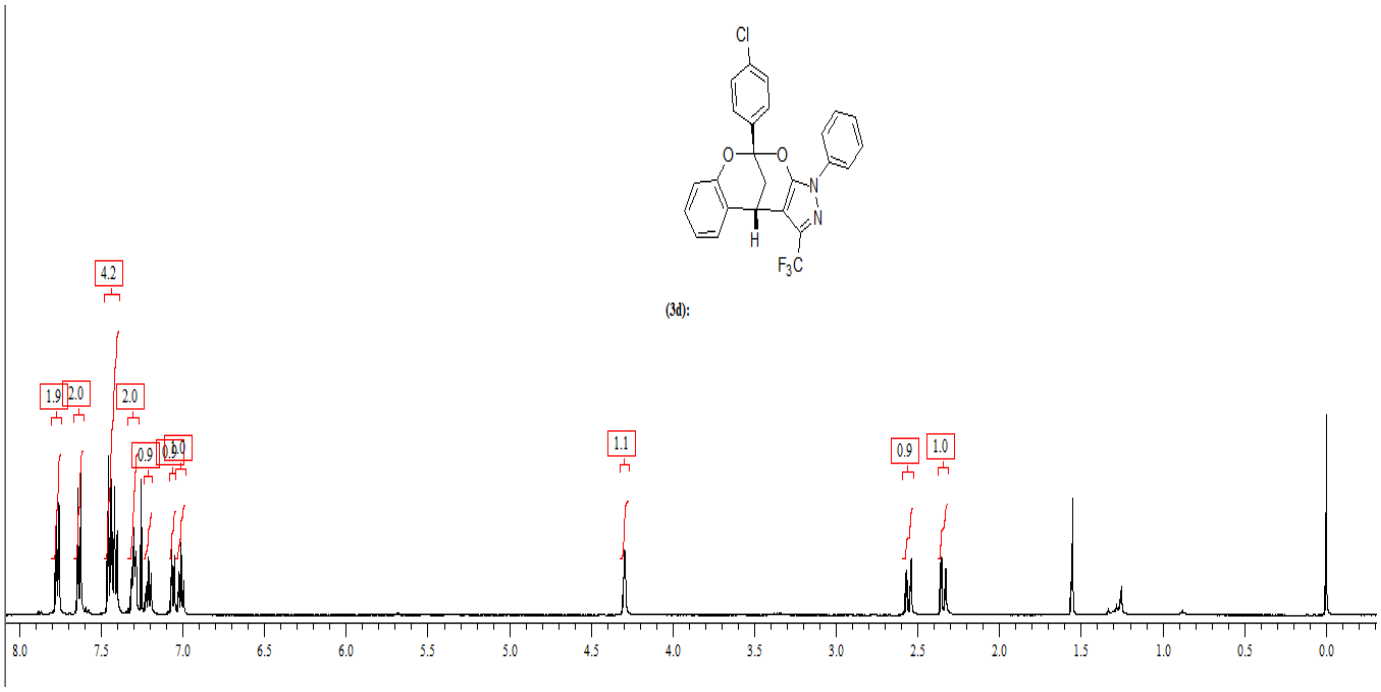

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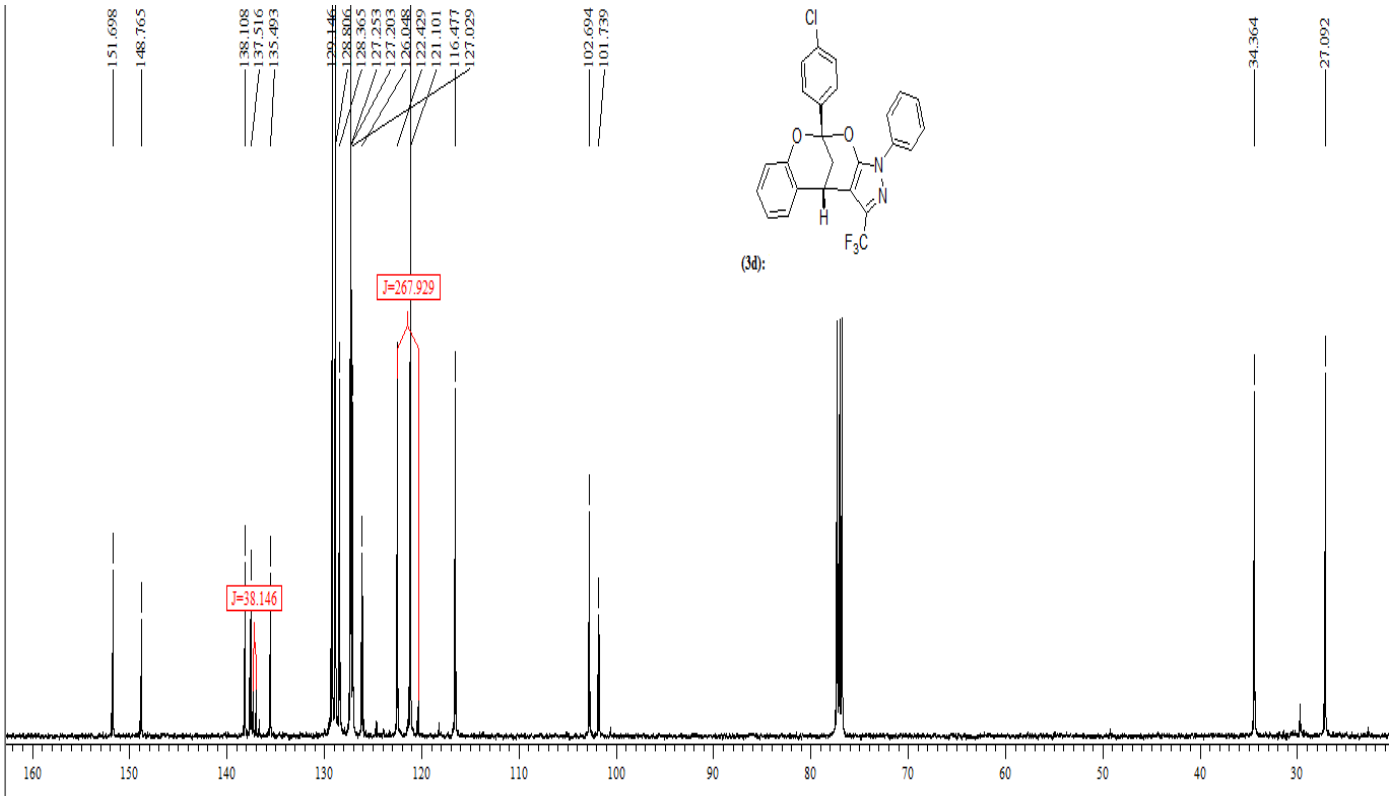

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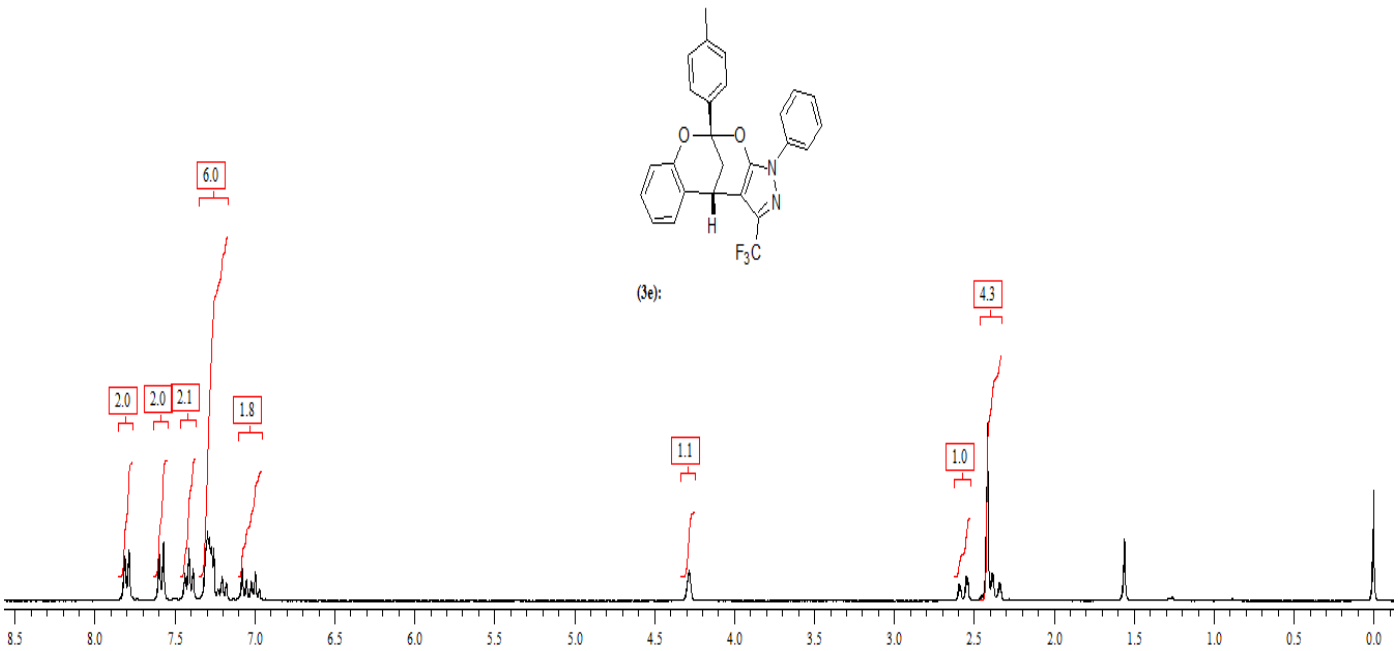

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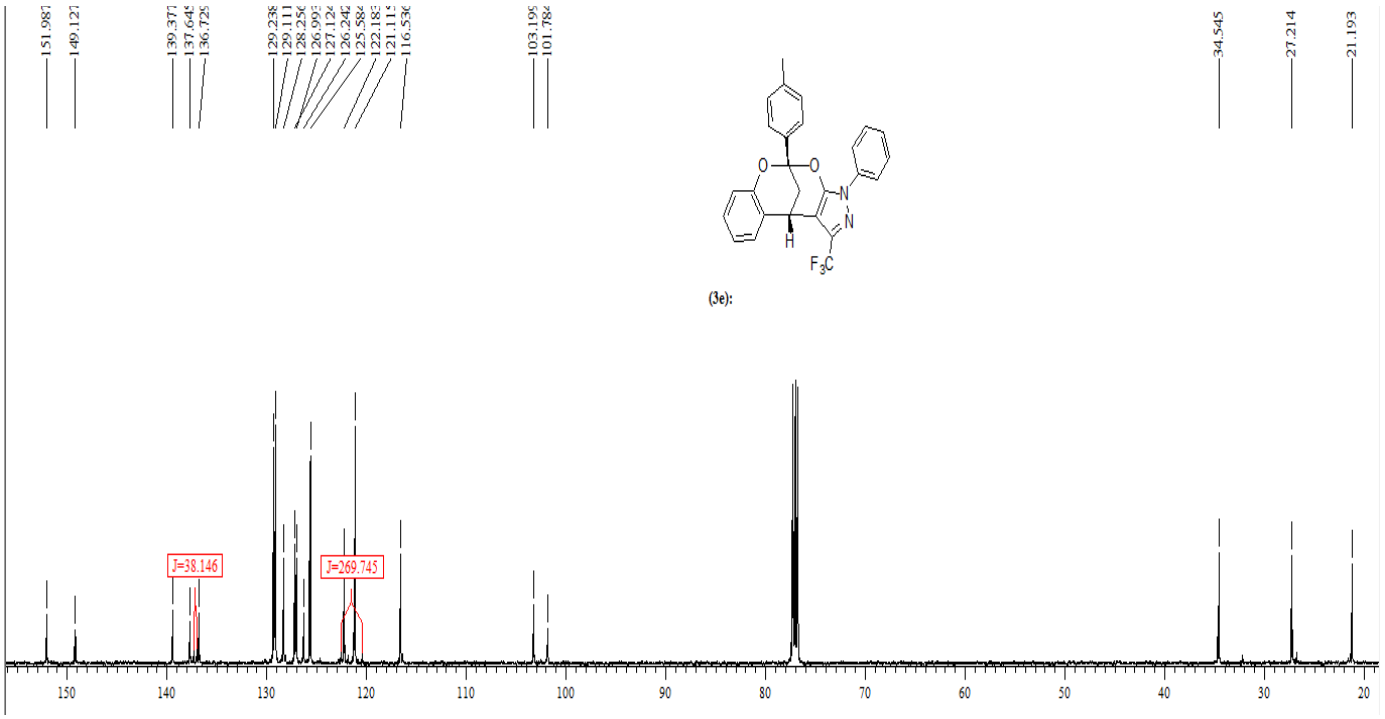

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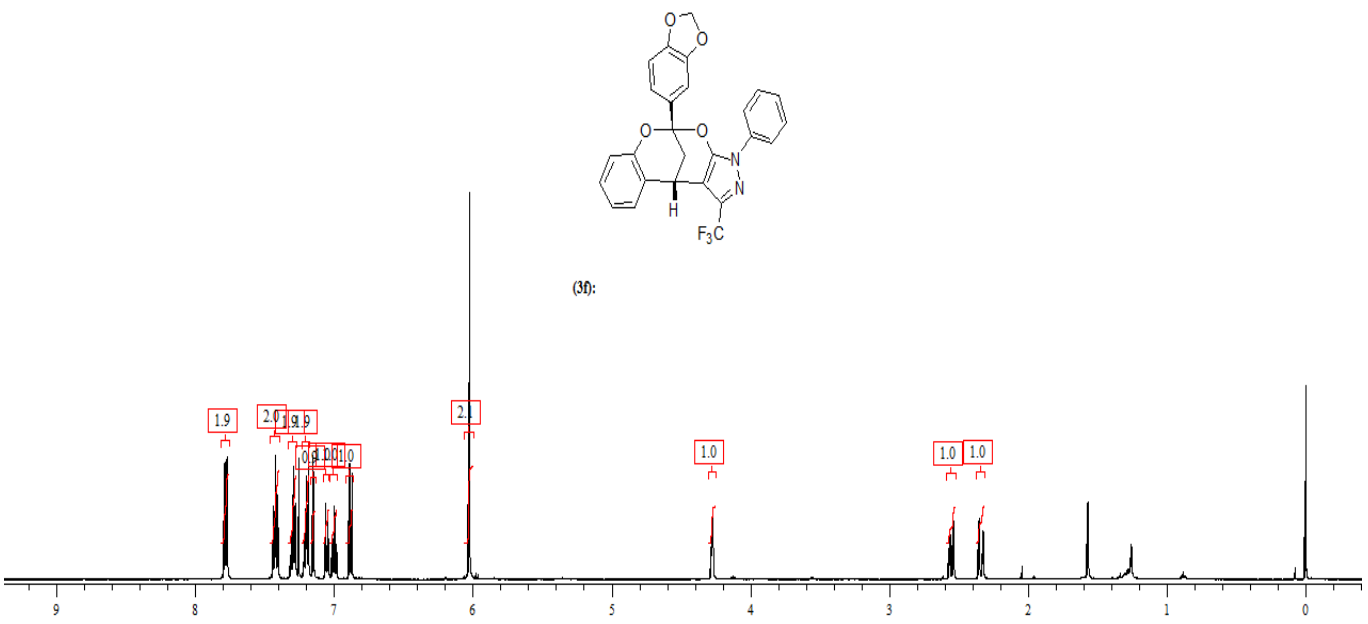

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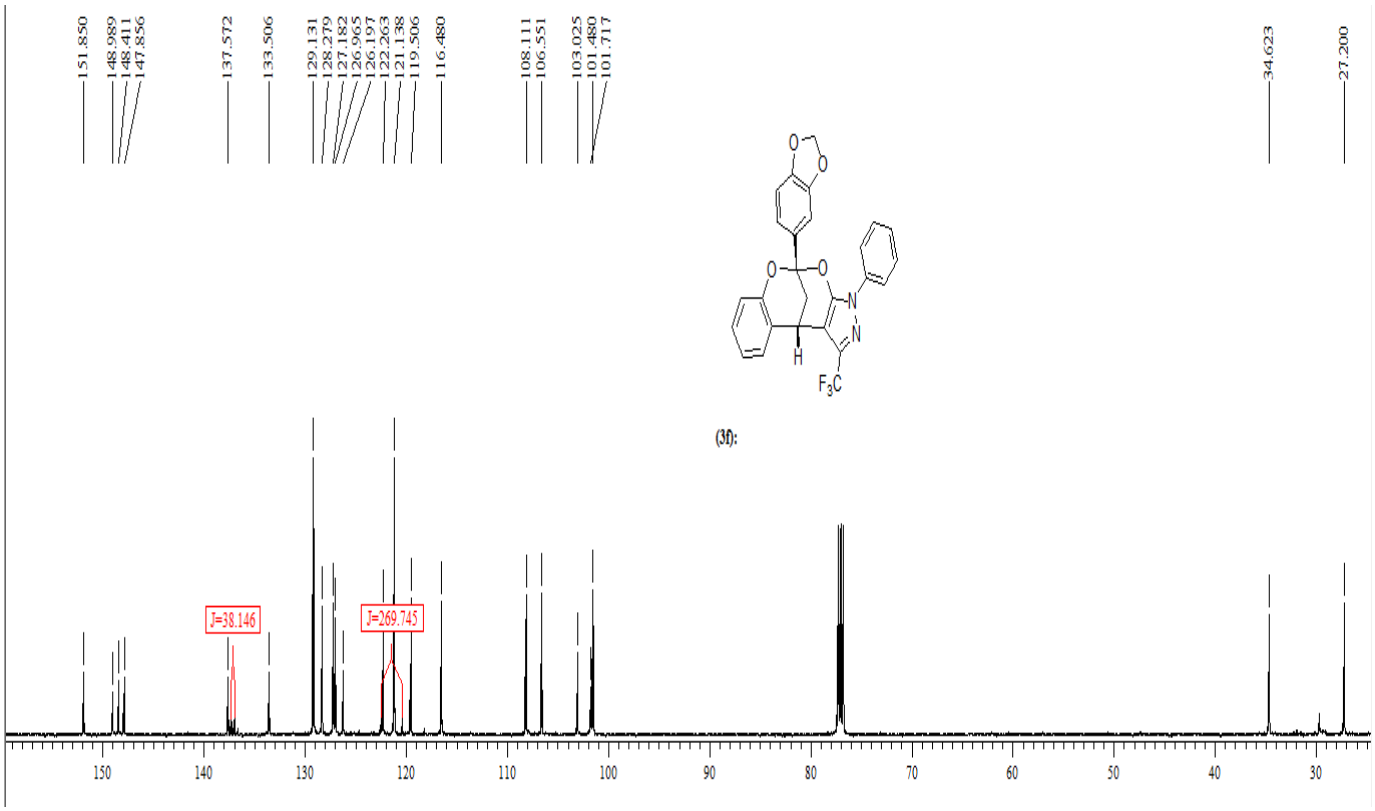

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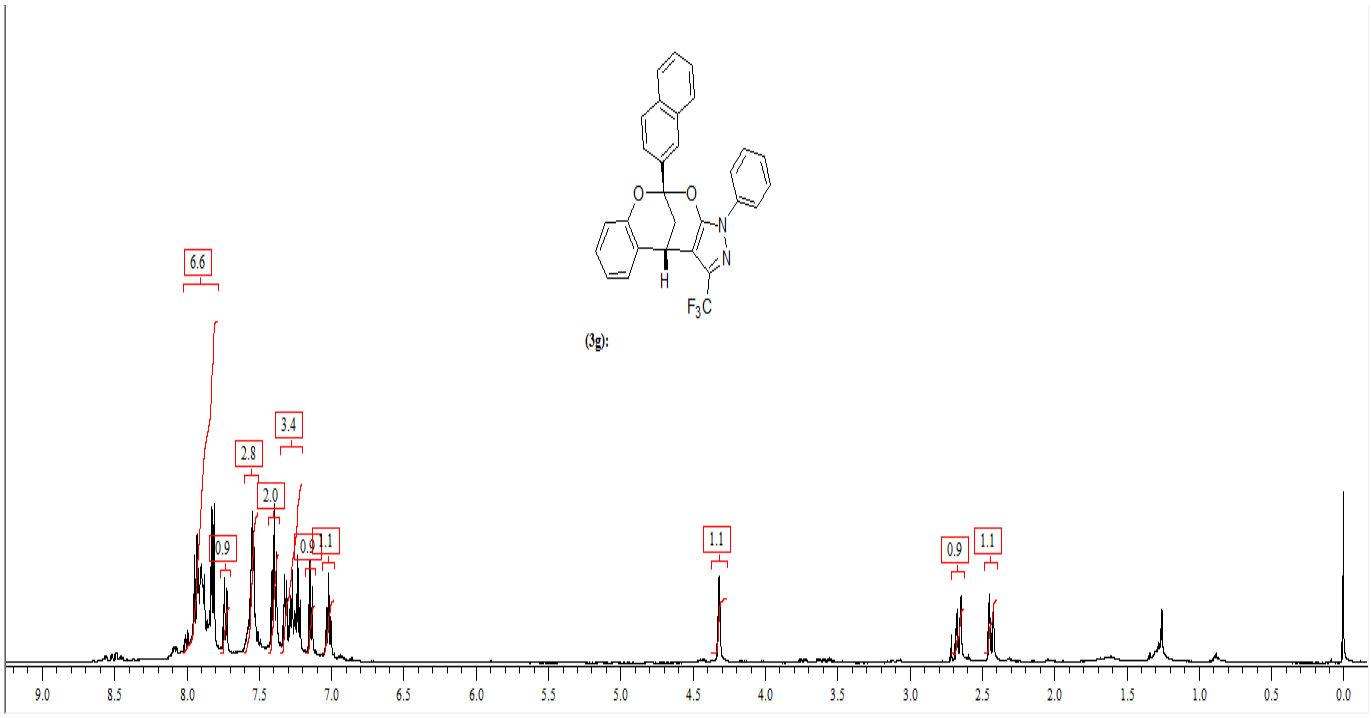

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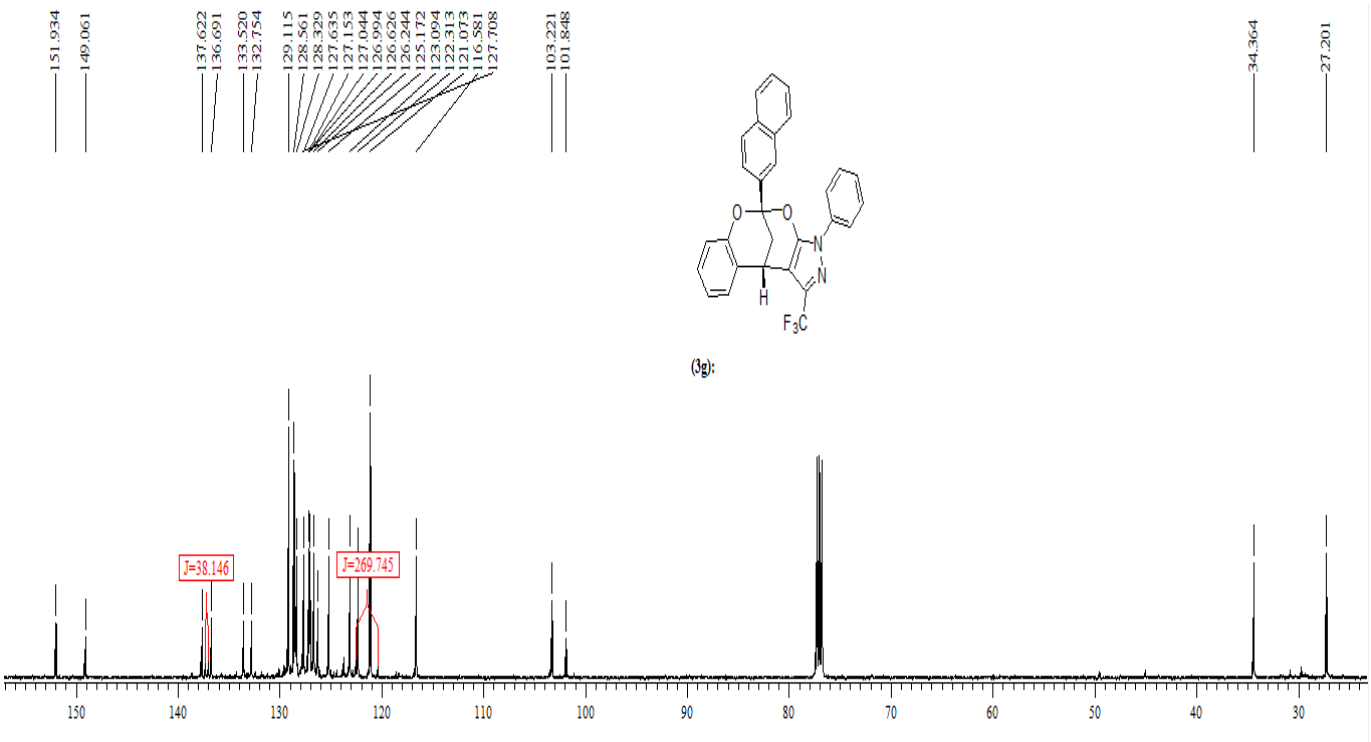

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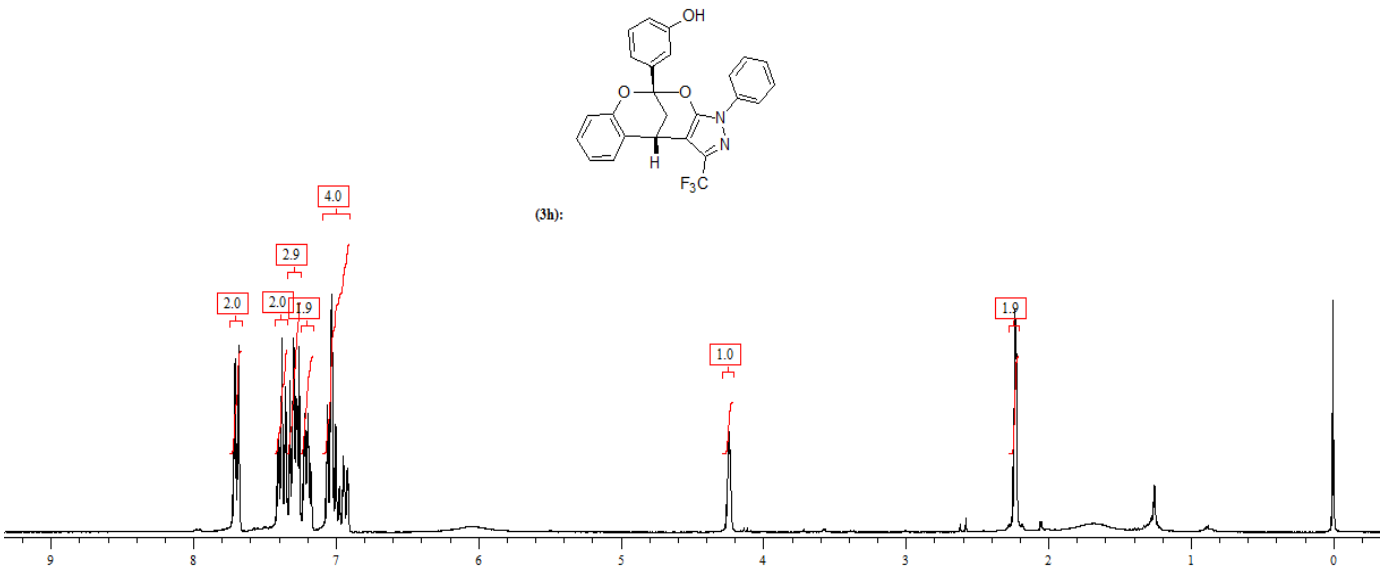

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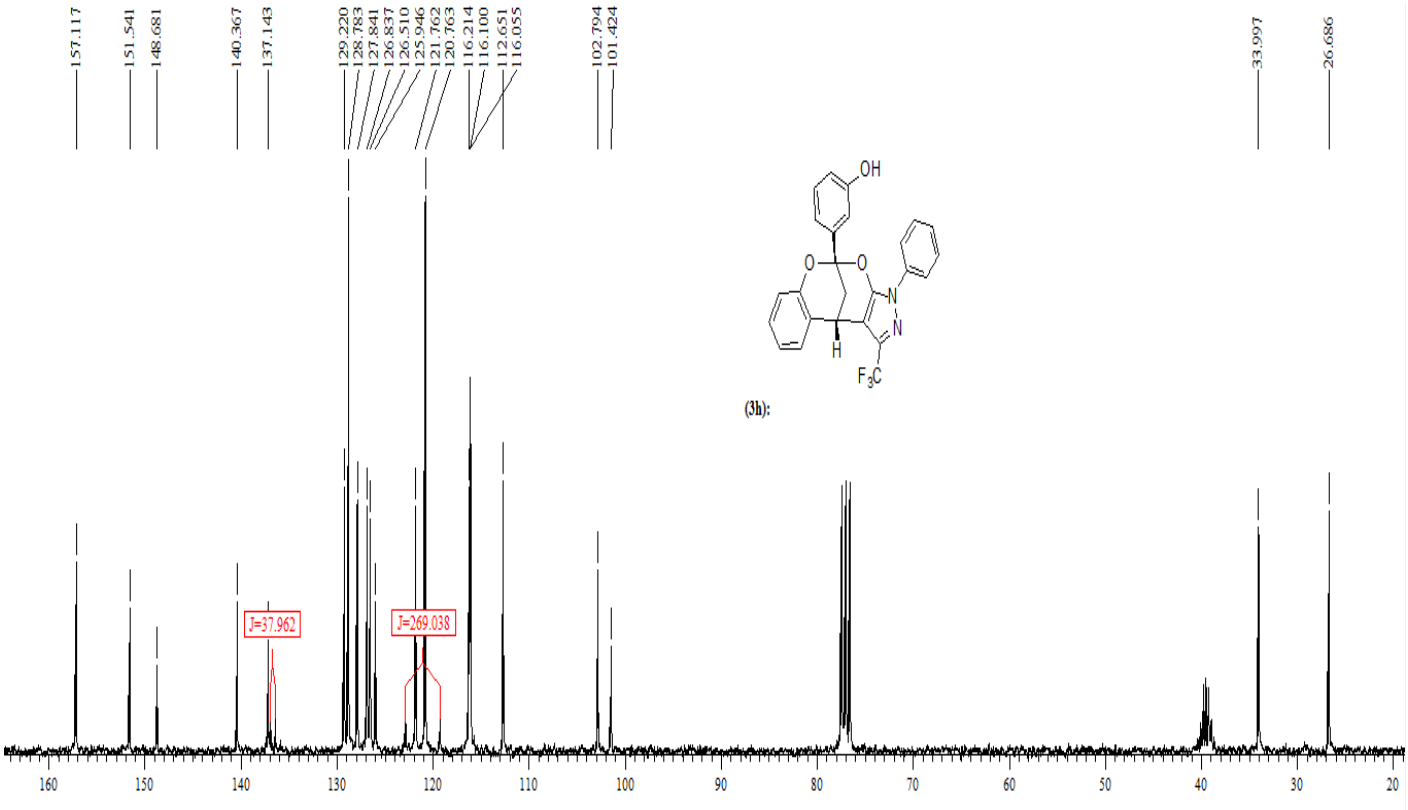
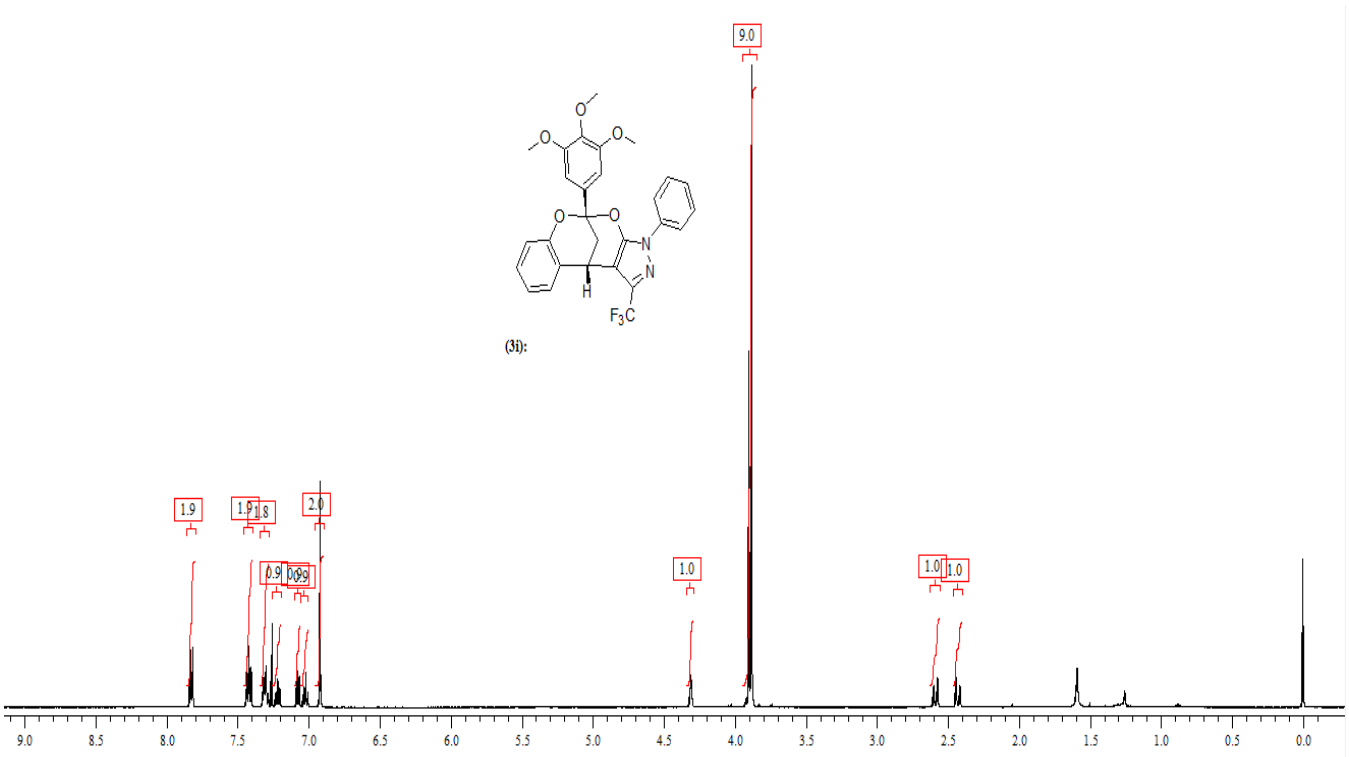


^{13}C NMR (125 MHz) spectrum of **3h** in $CDCl_3$+DMSO-d_6



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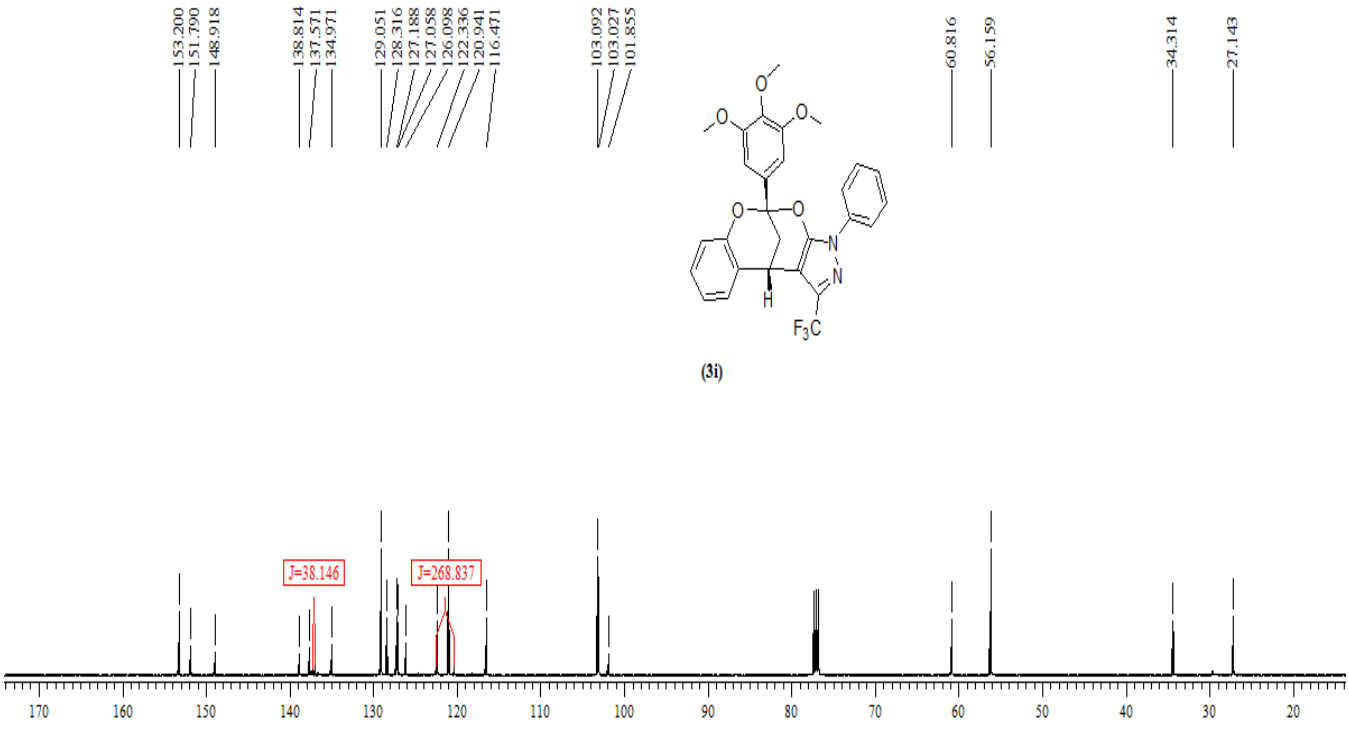
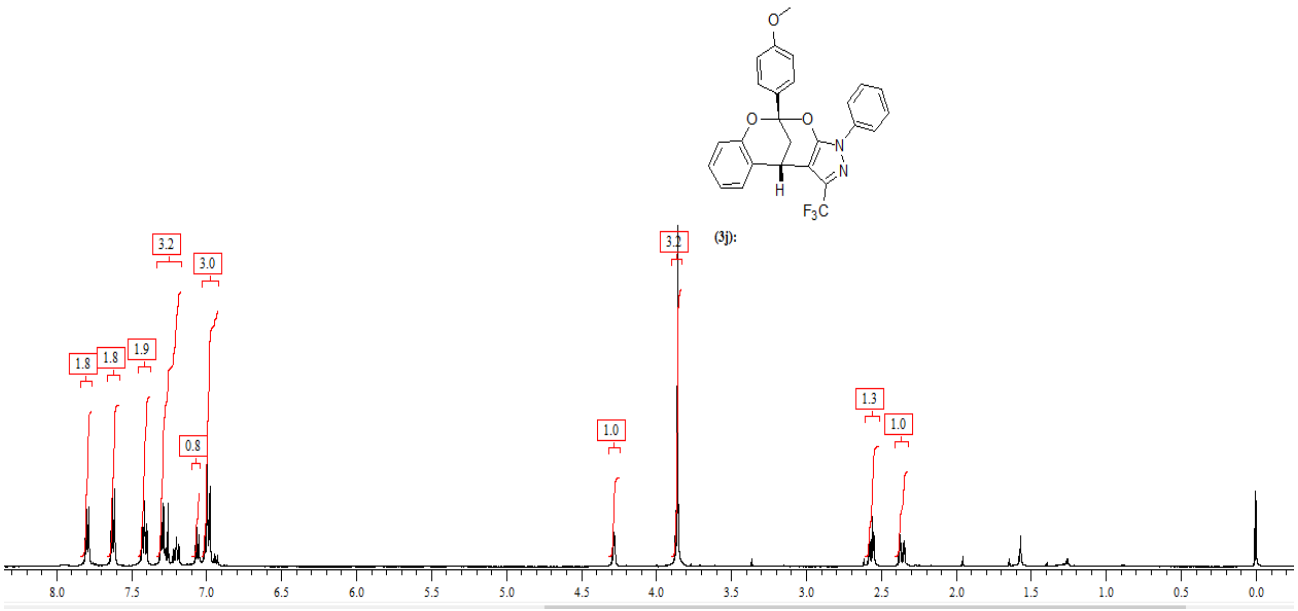


^{13}C NMR (125 MHz) spectrum of **3i** in $CDCl_3$



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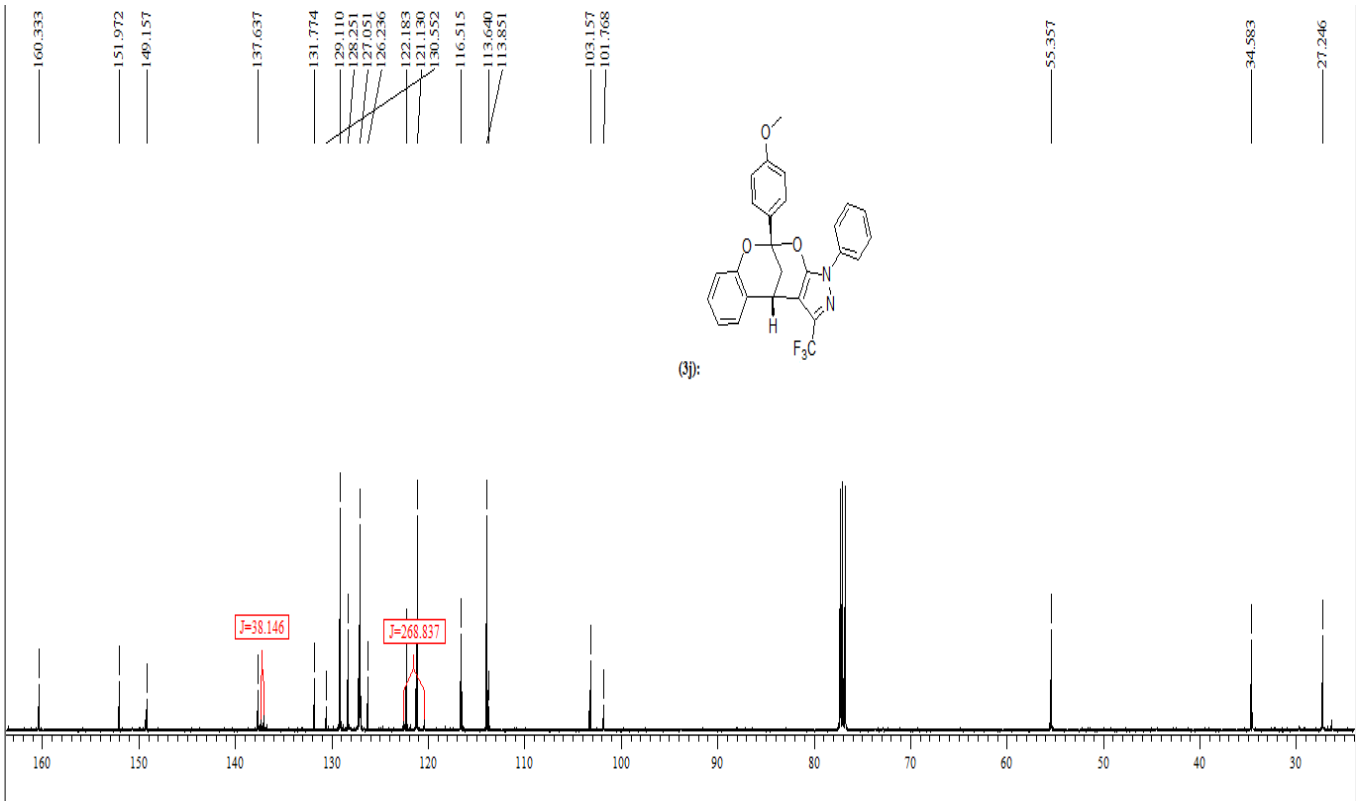

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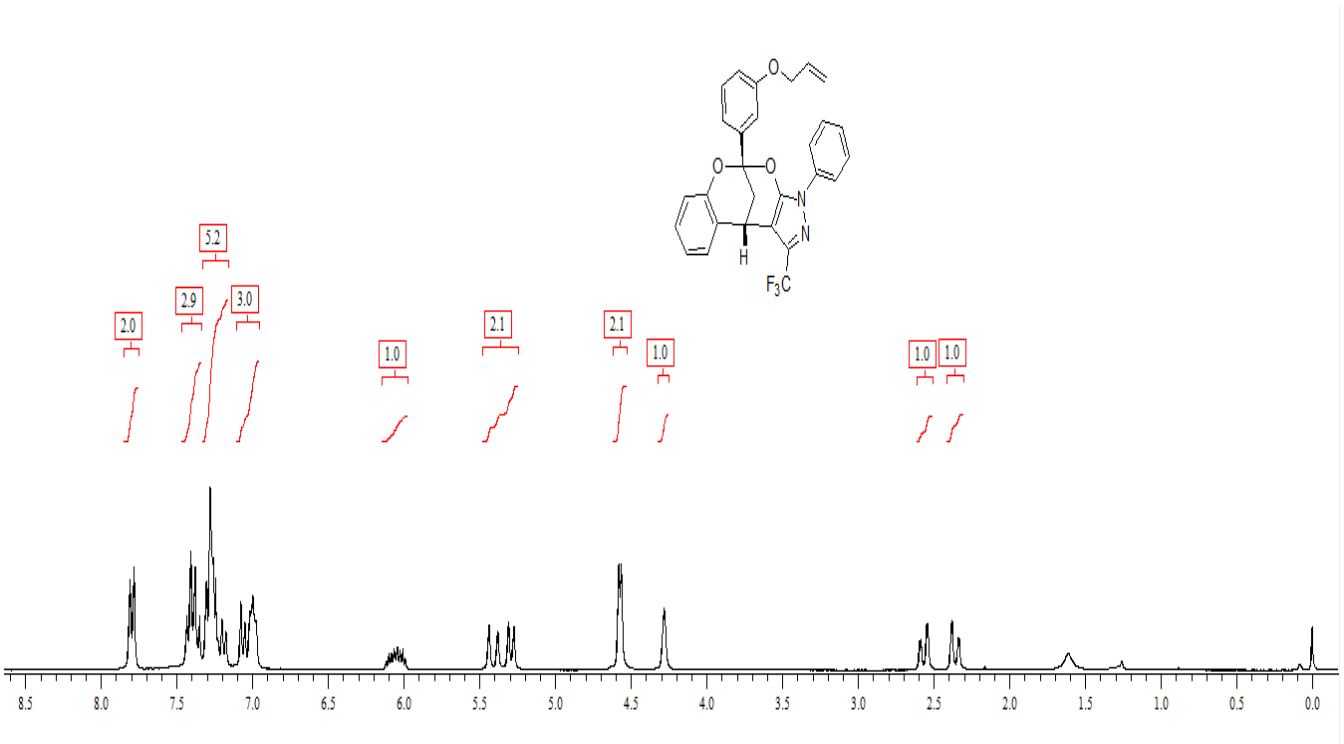

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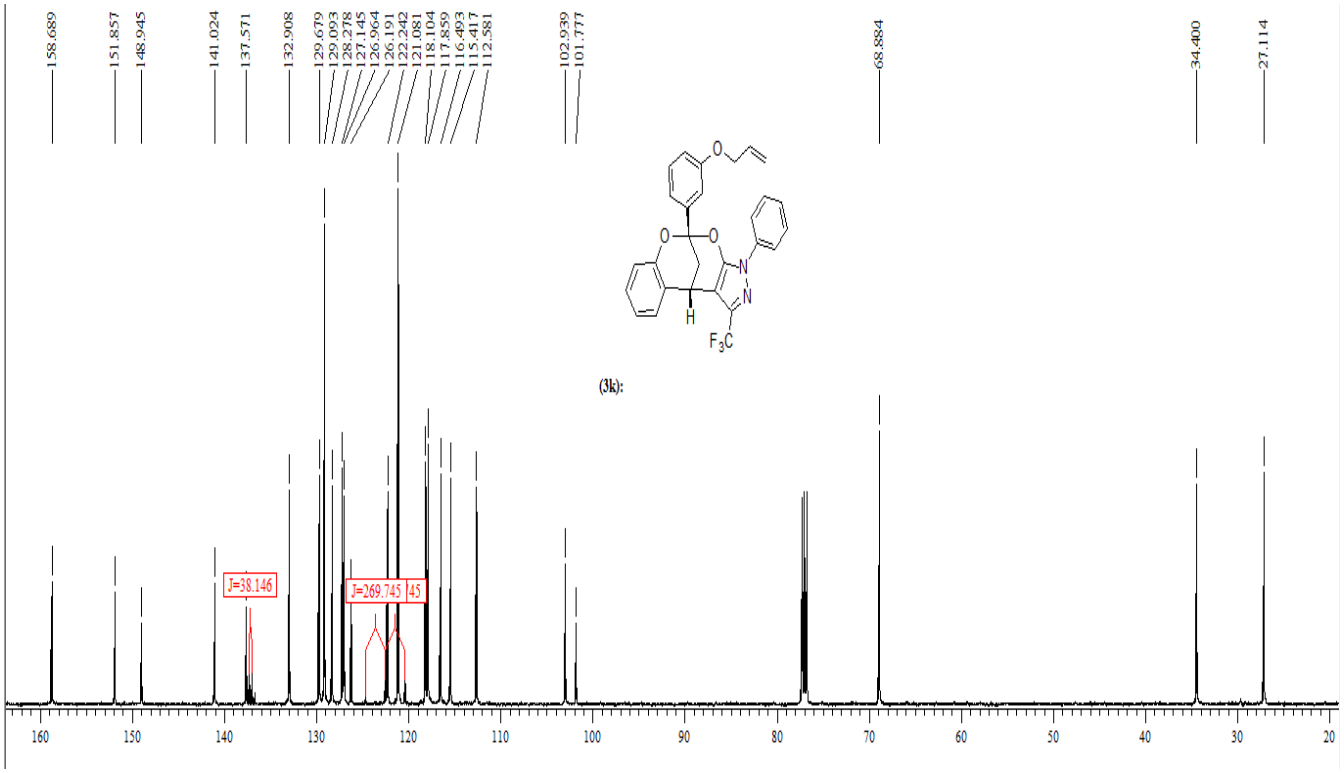

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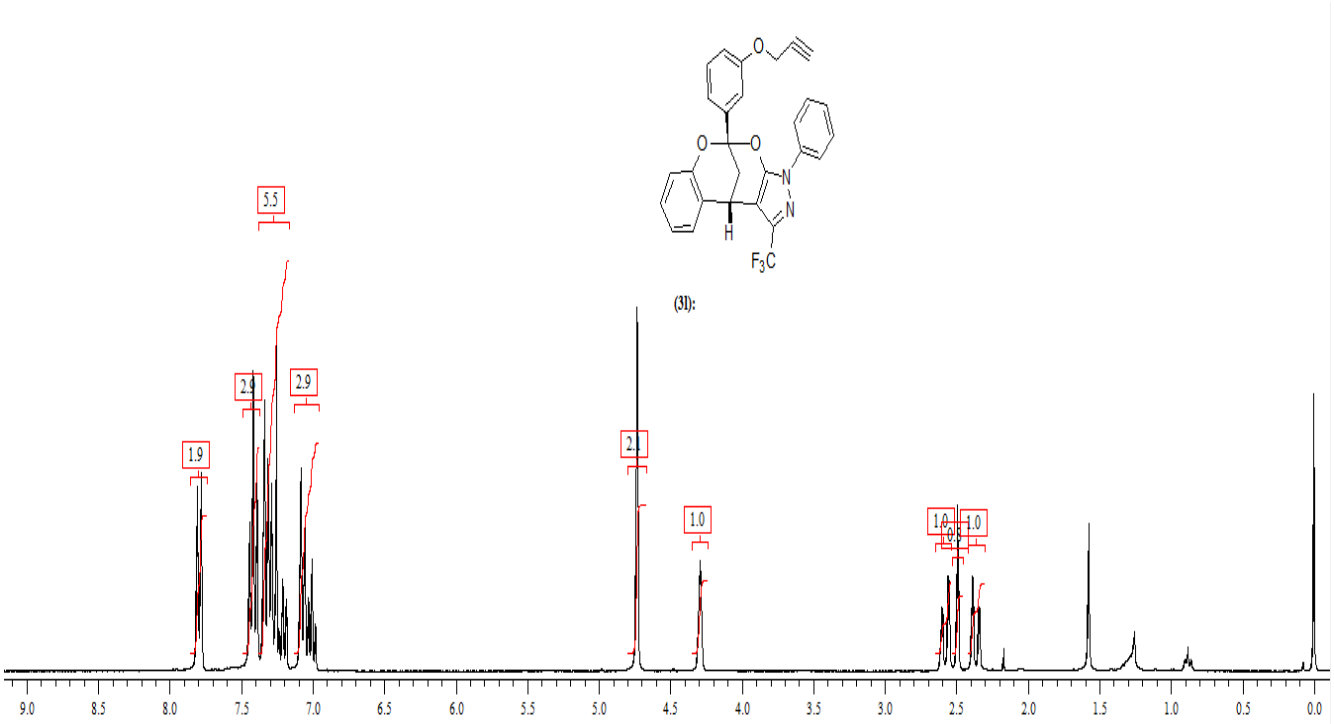

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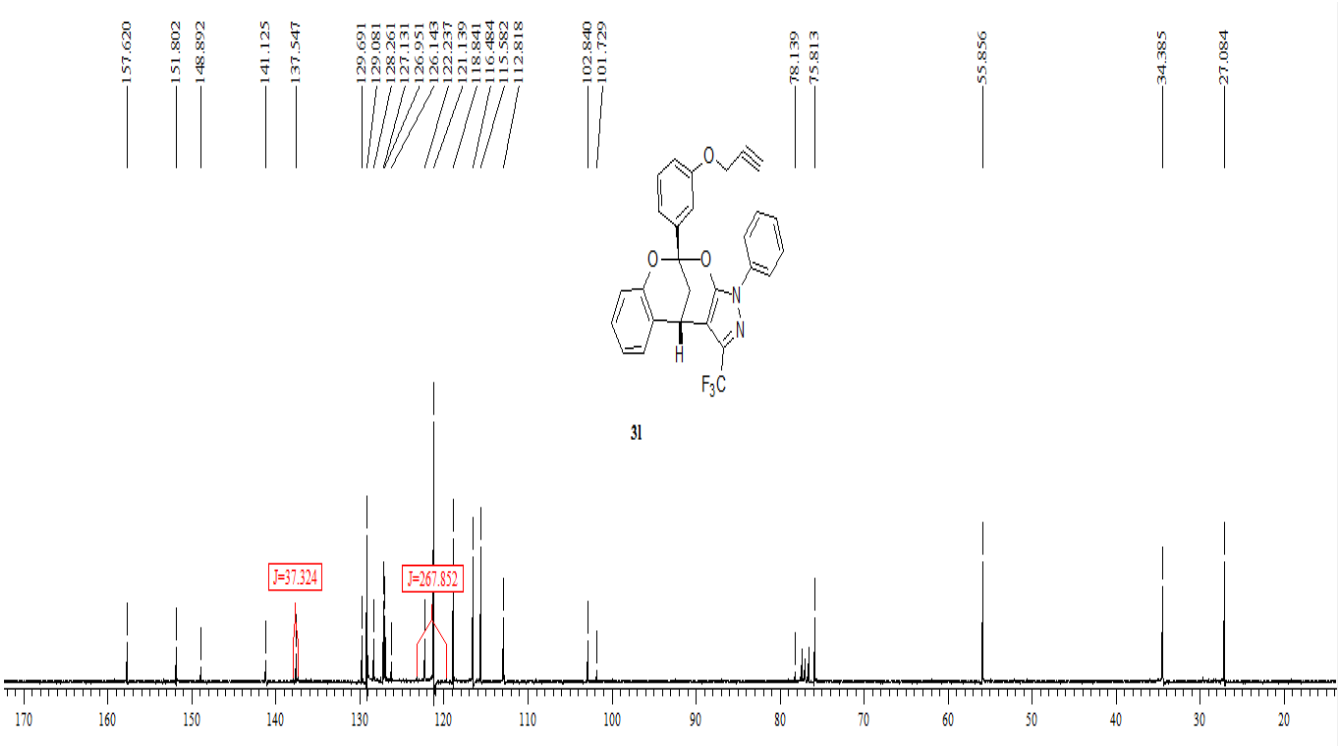

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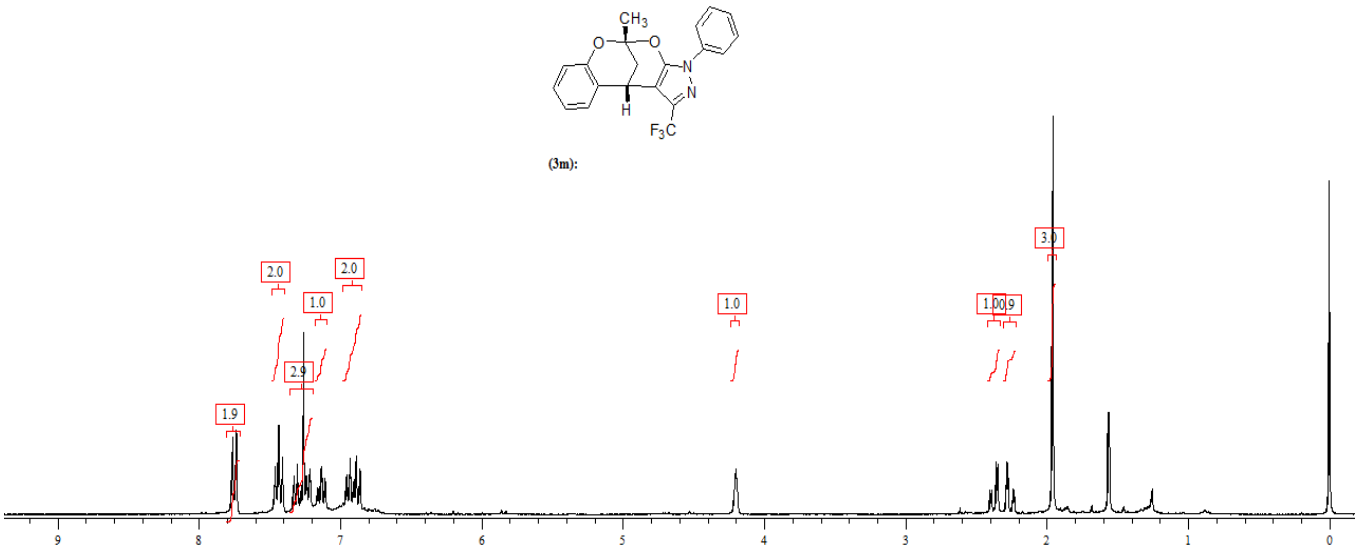

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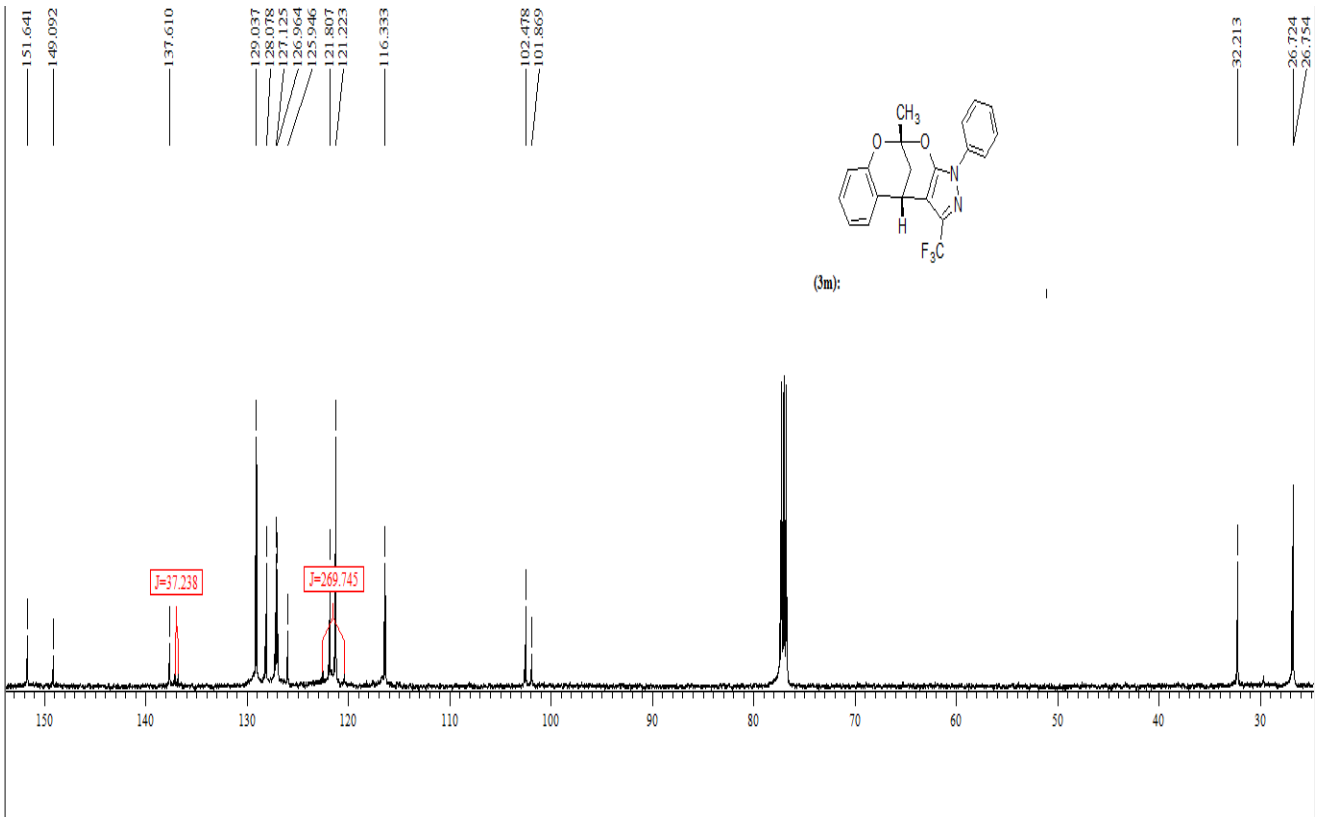

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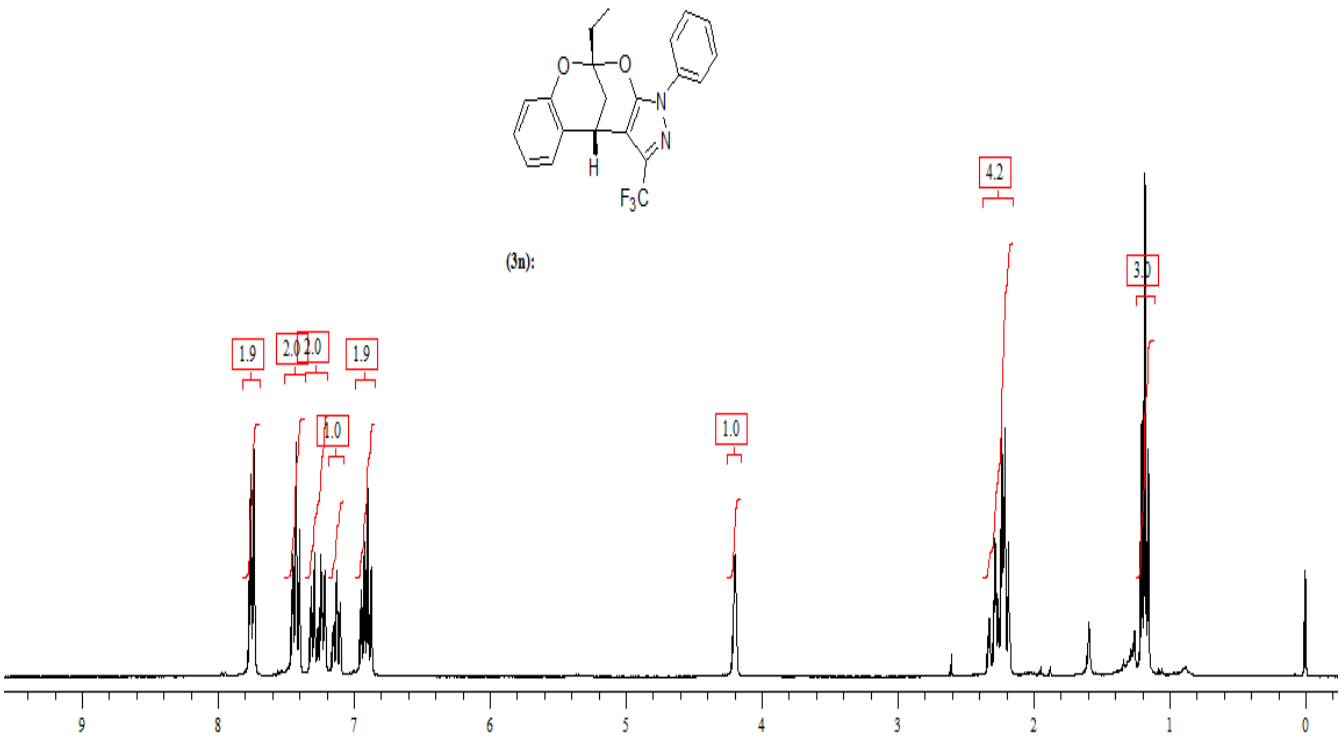

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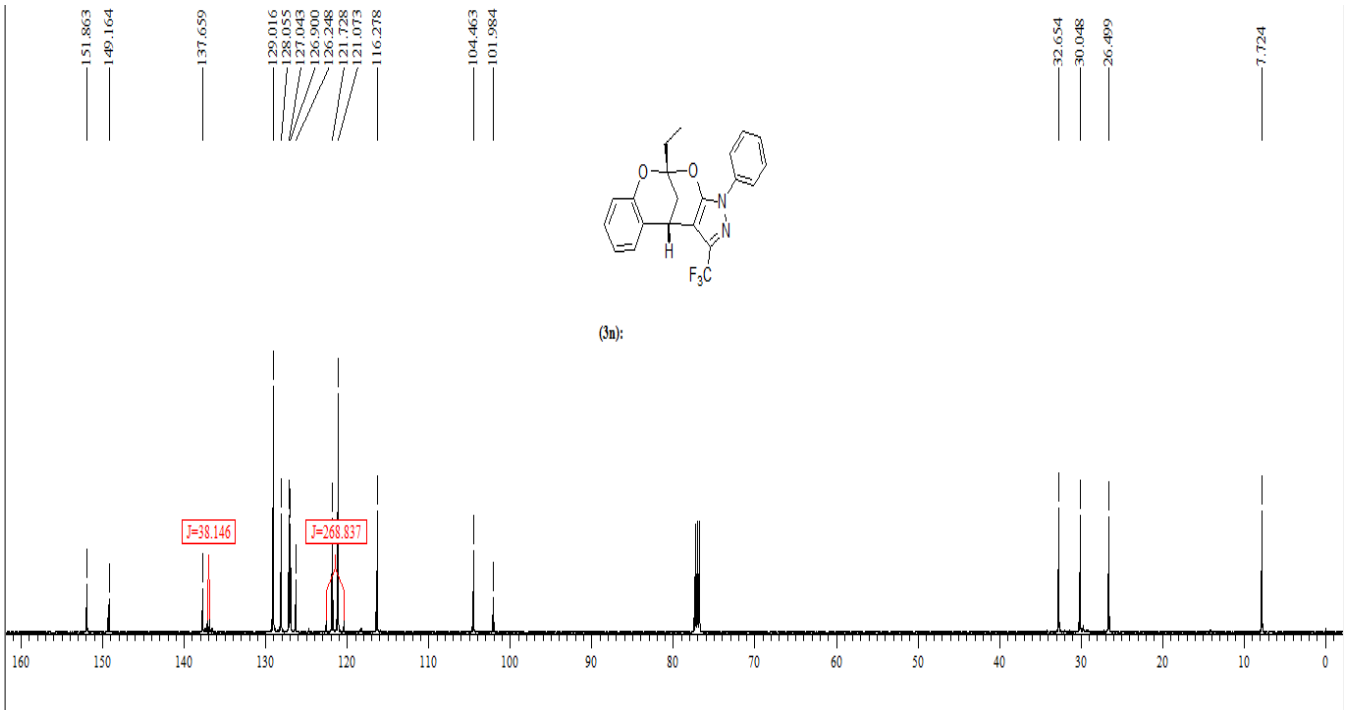

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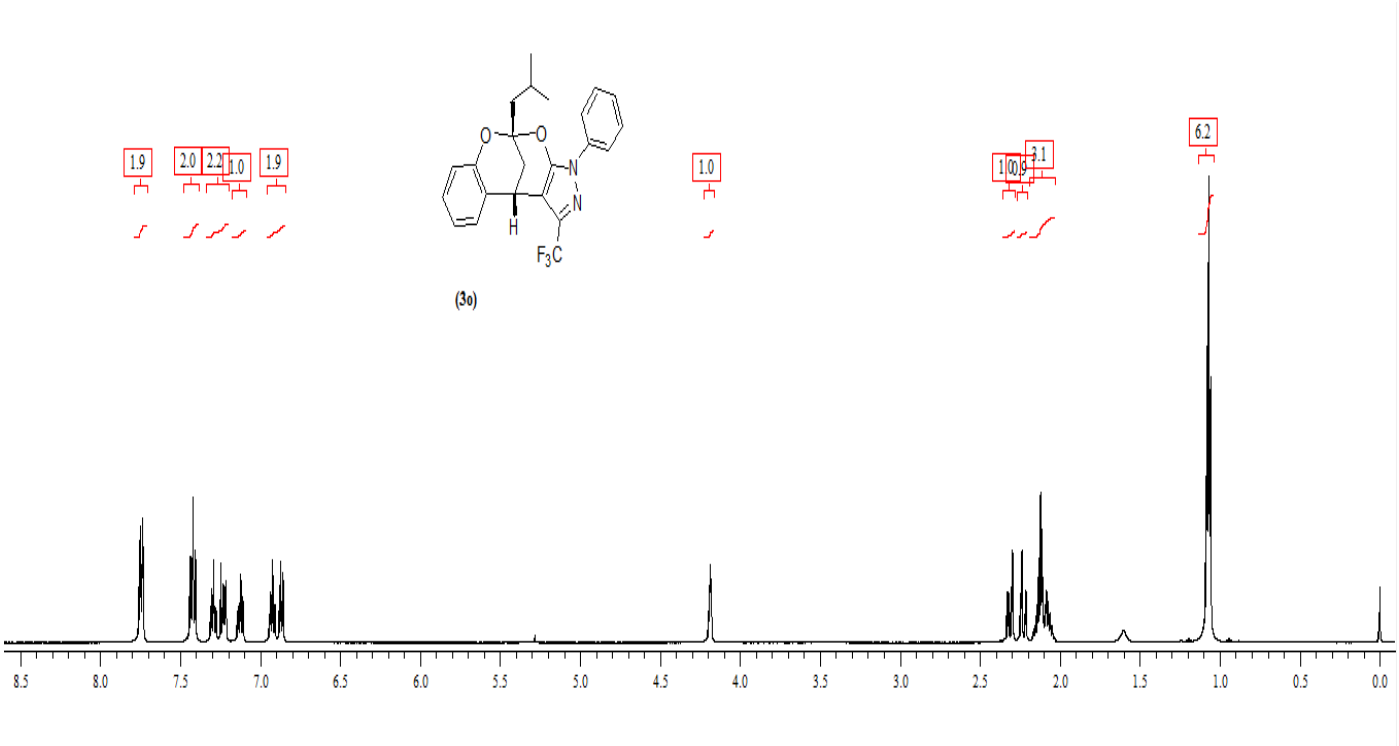

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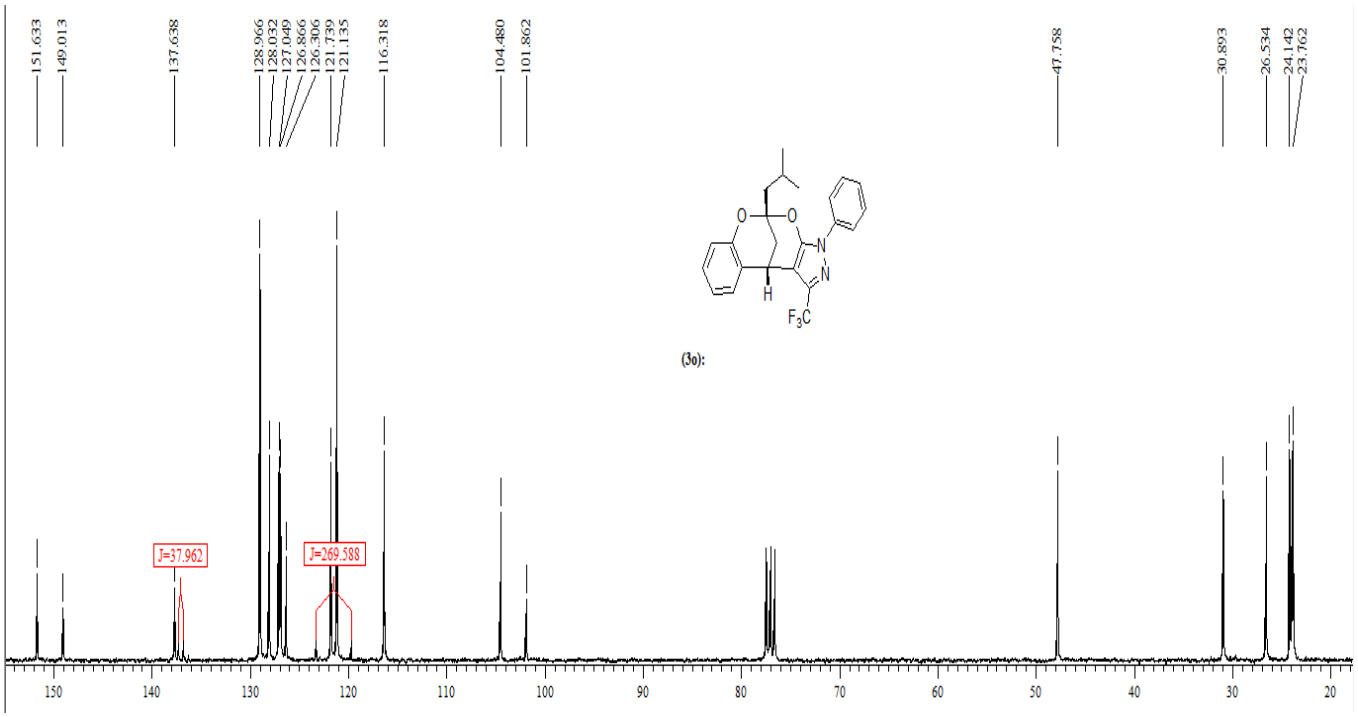

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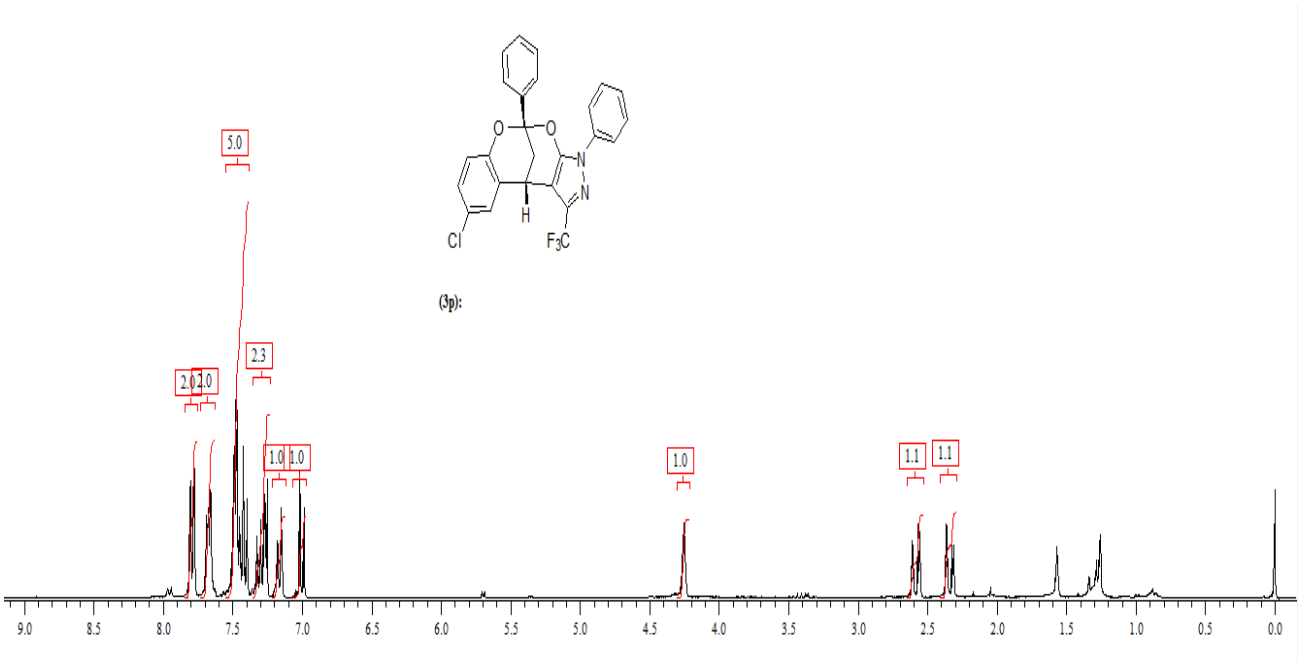

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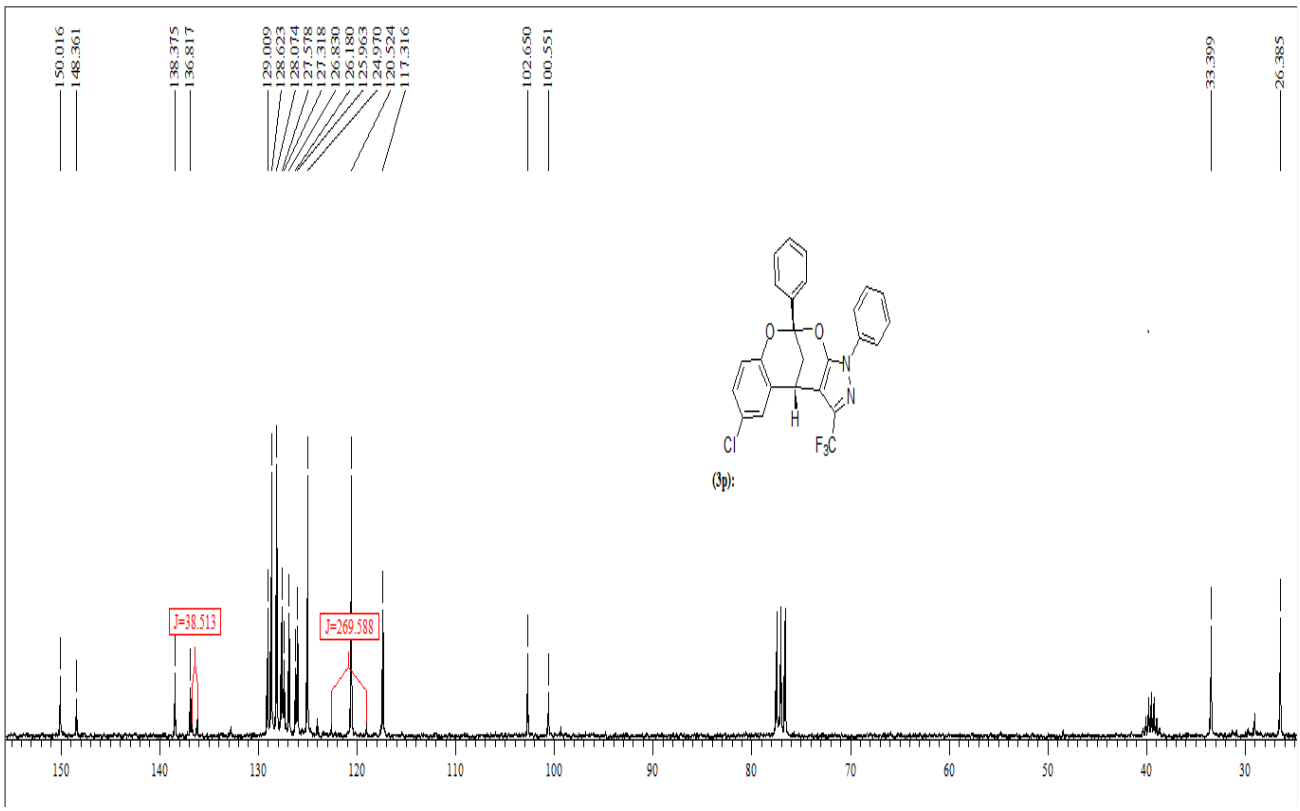

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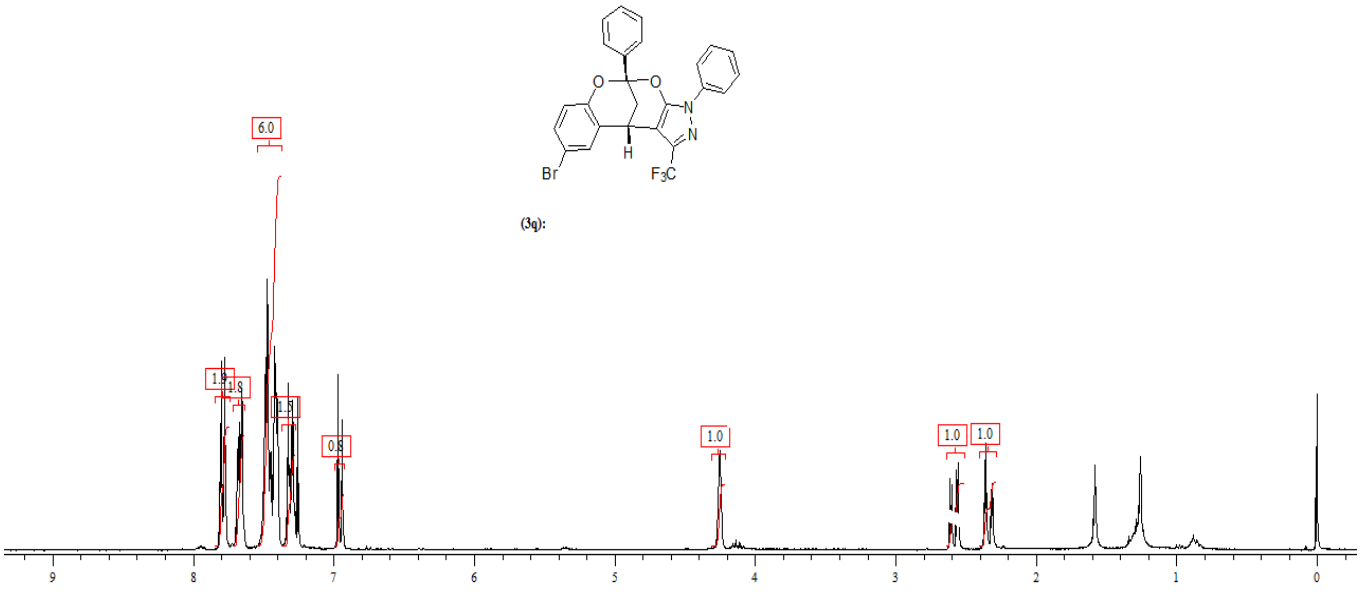

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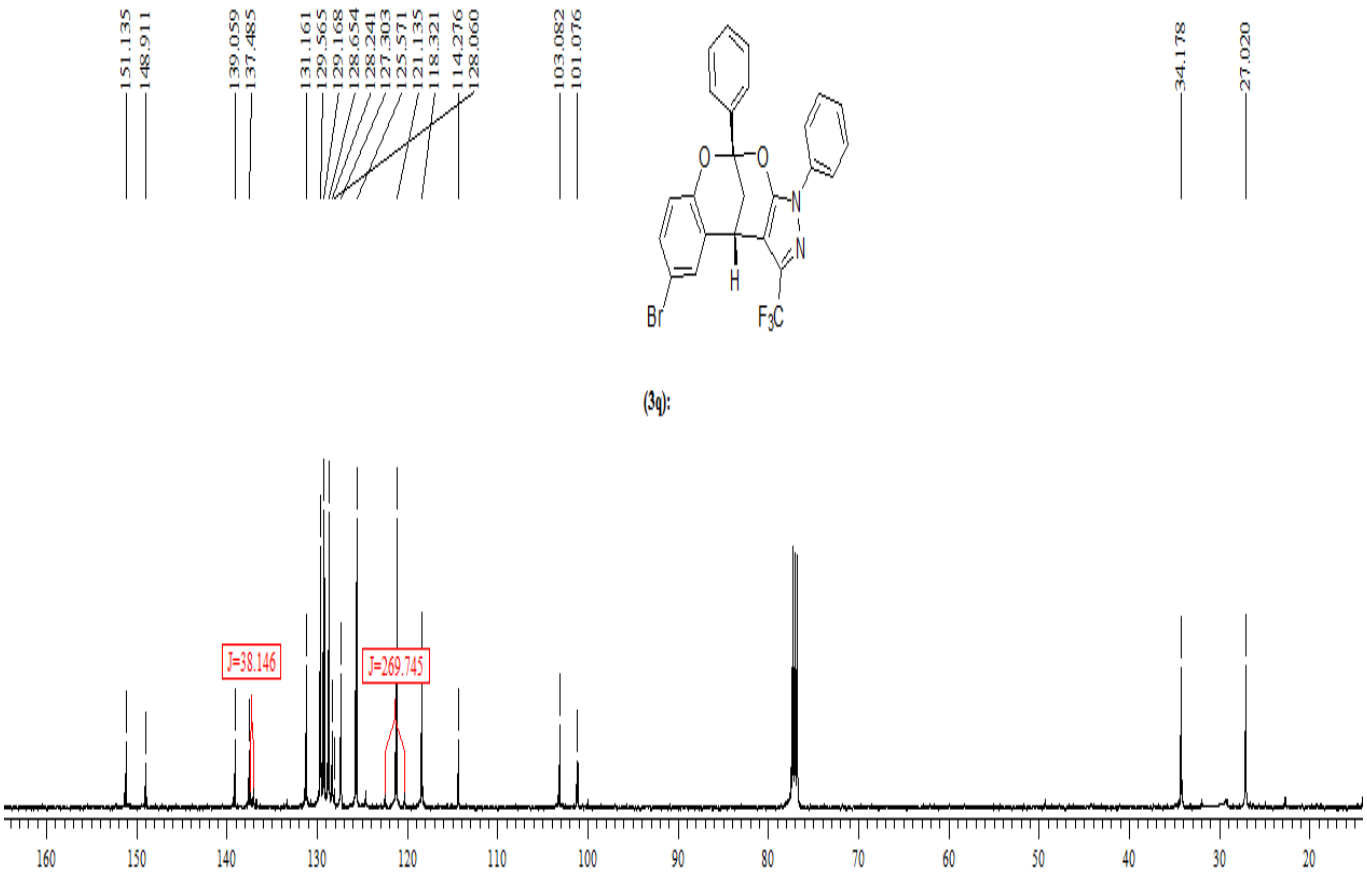

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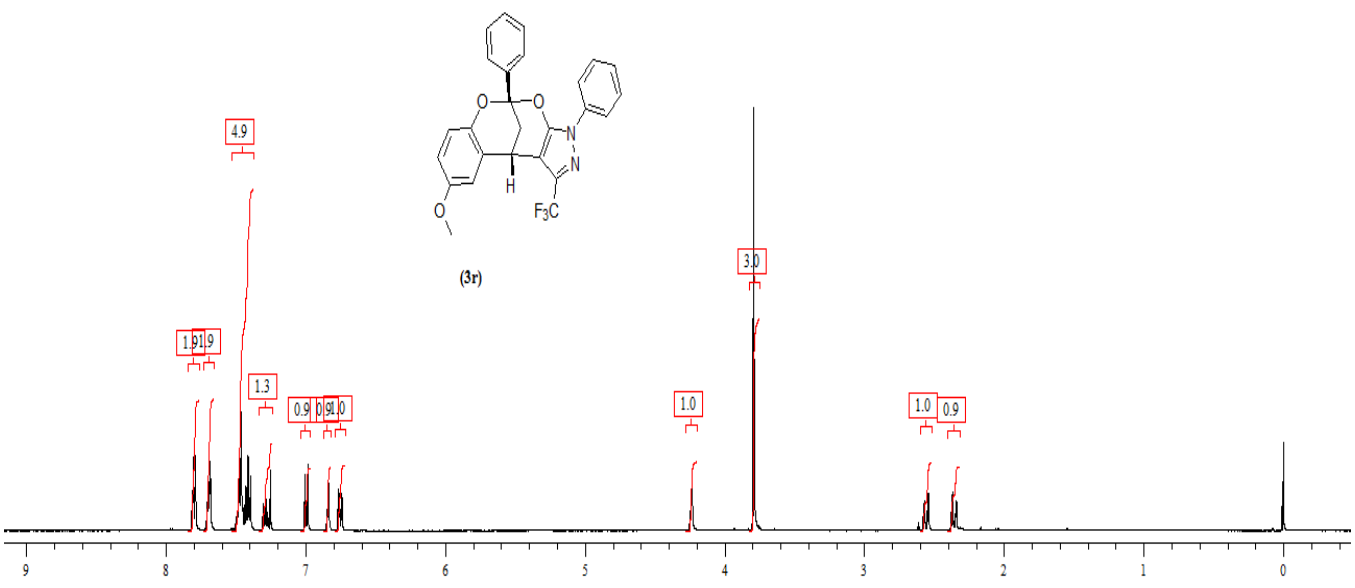

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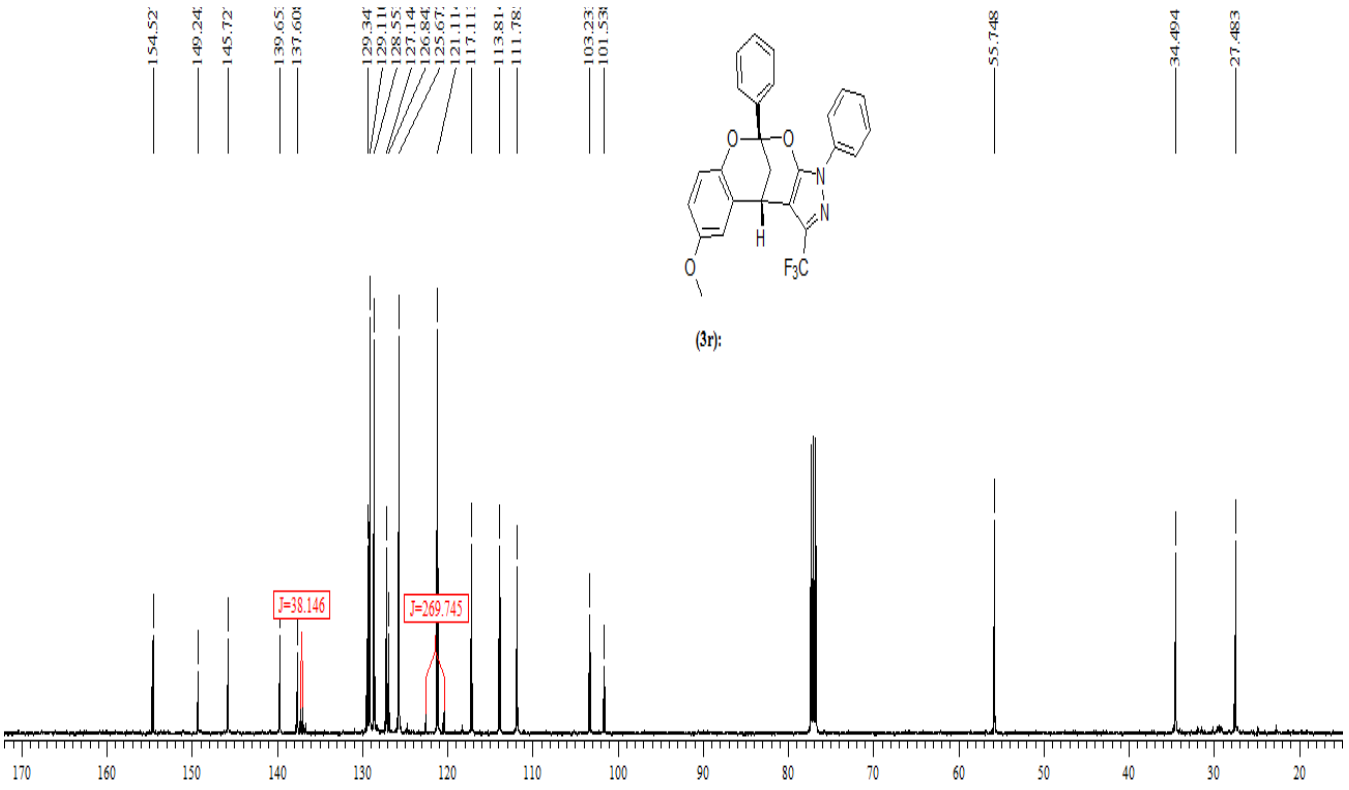

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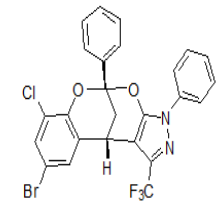

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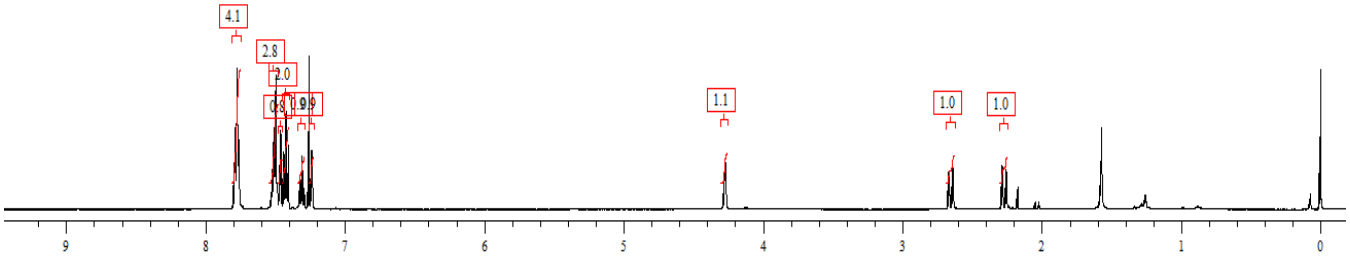

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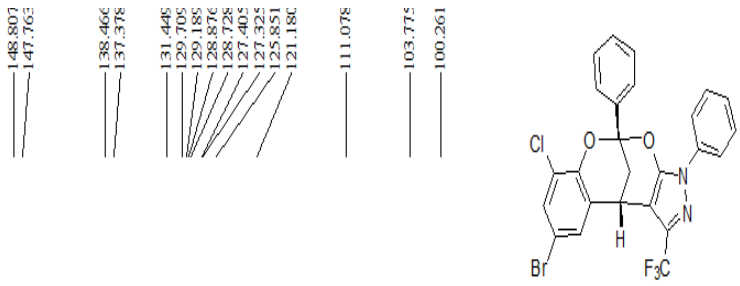
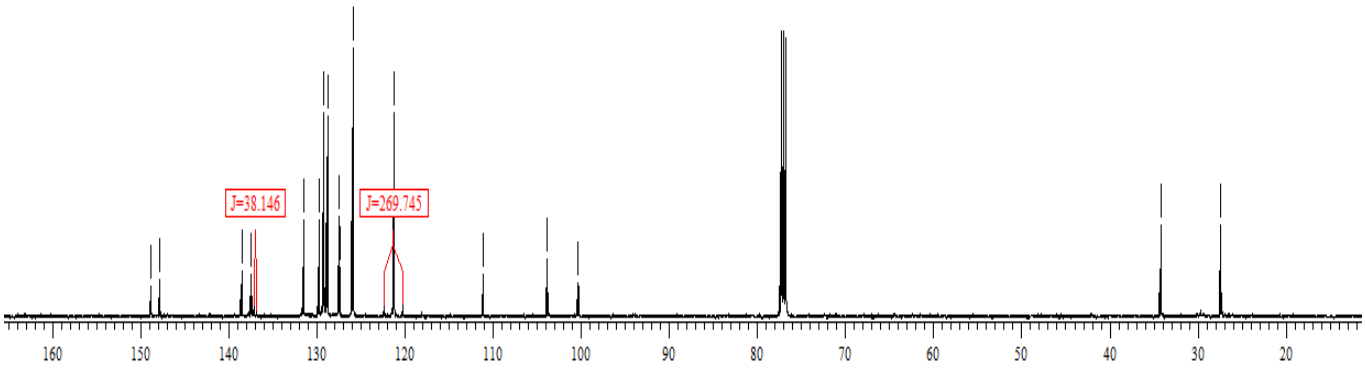
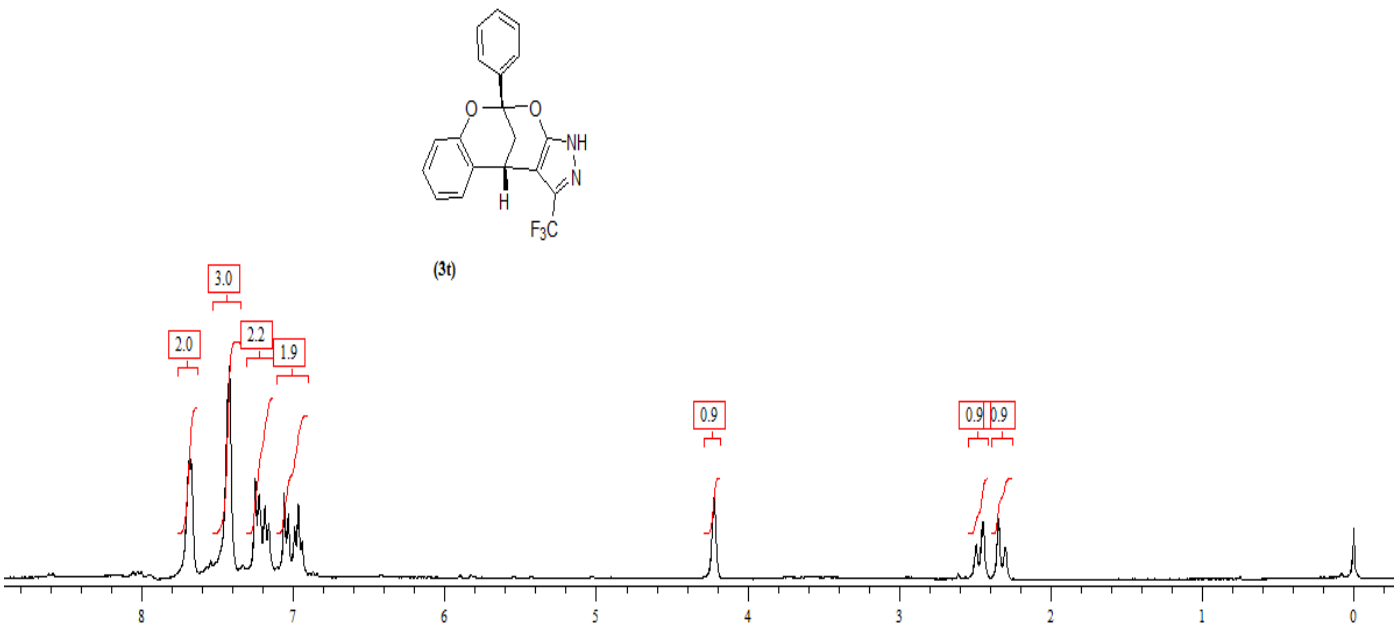


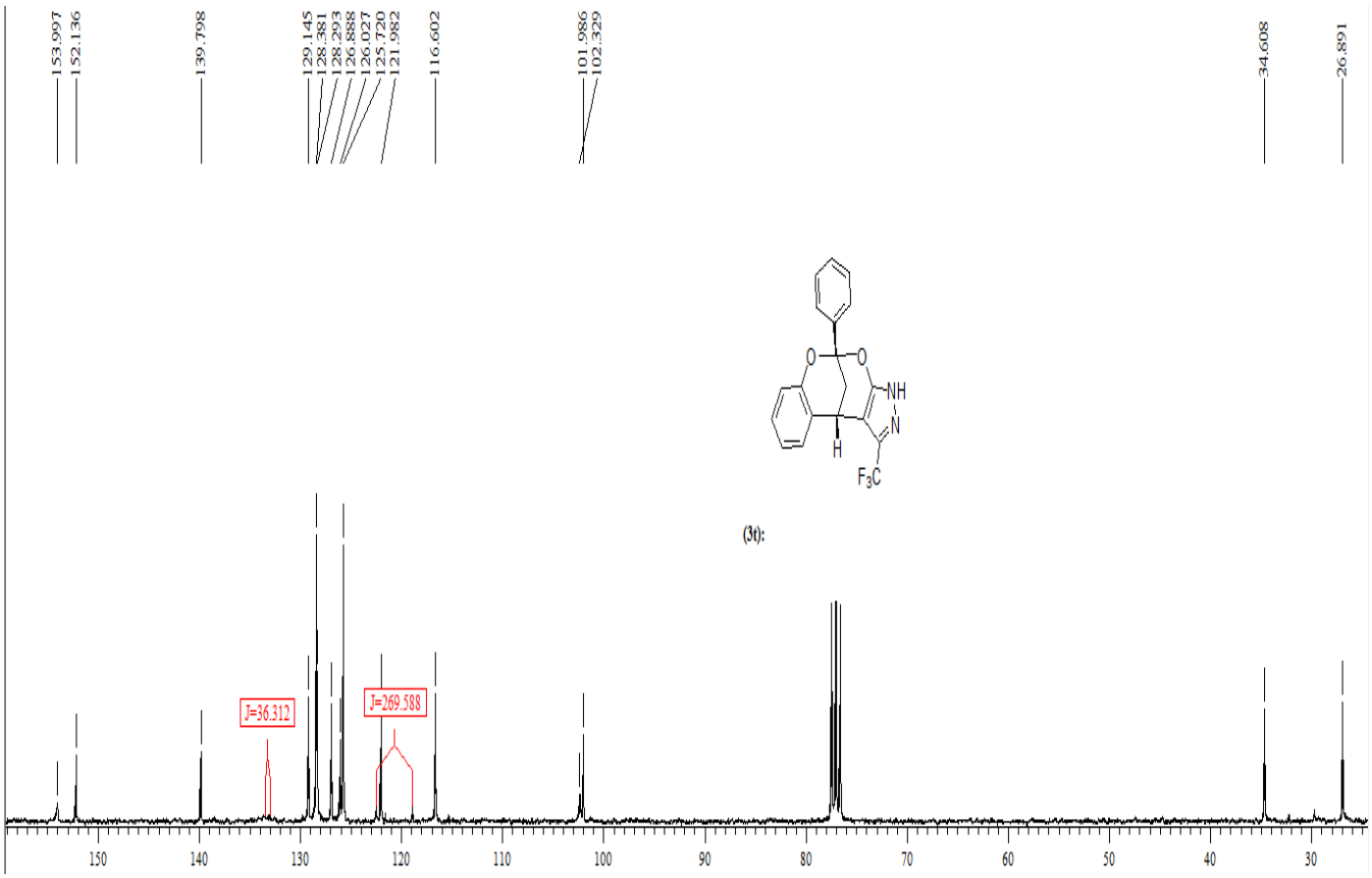
(3s):



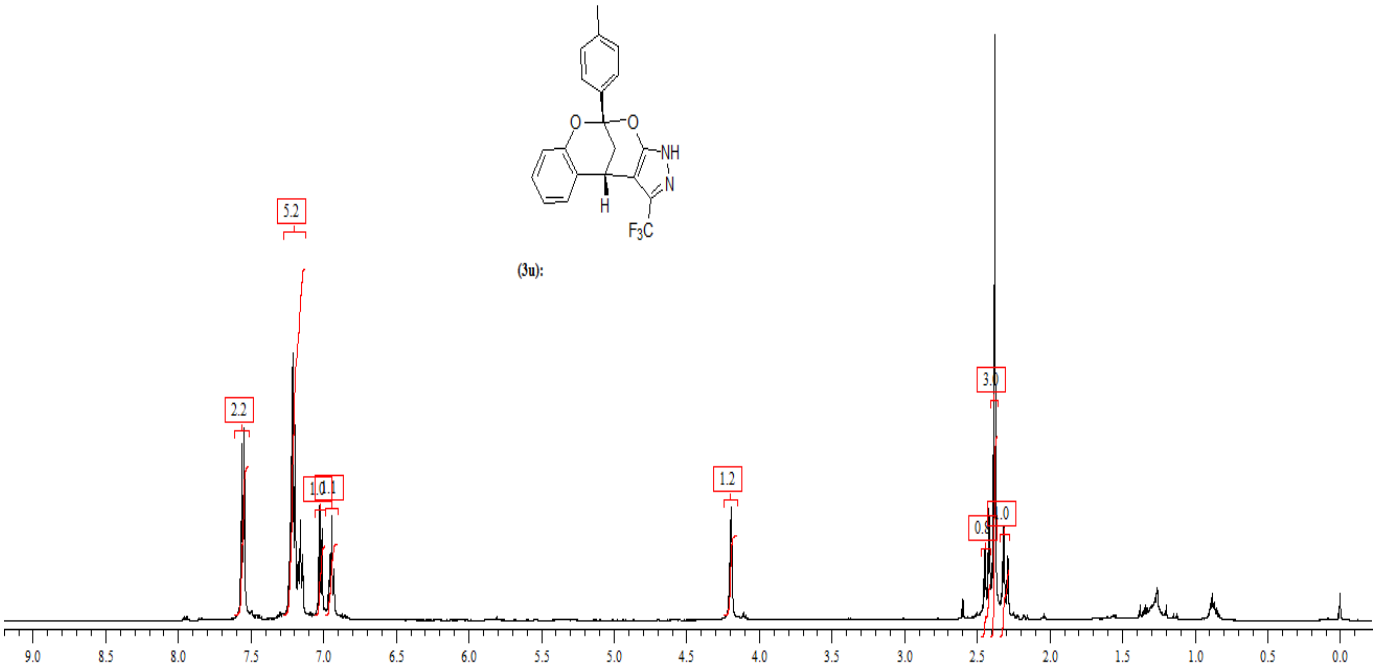
^{13}C NMR (125 MHz) spectrum of **3s** in $CDCl_3$



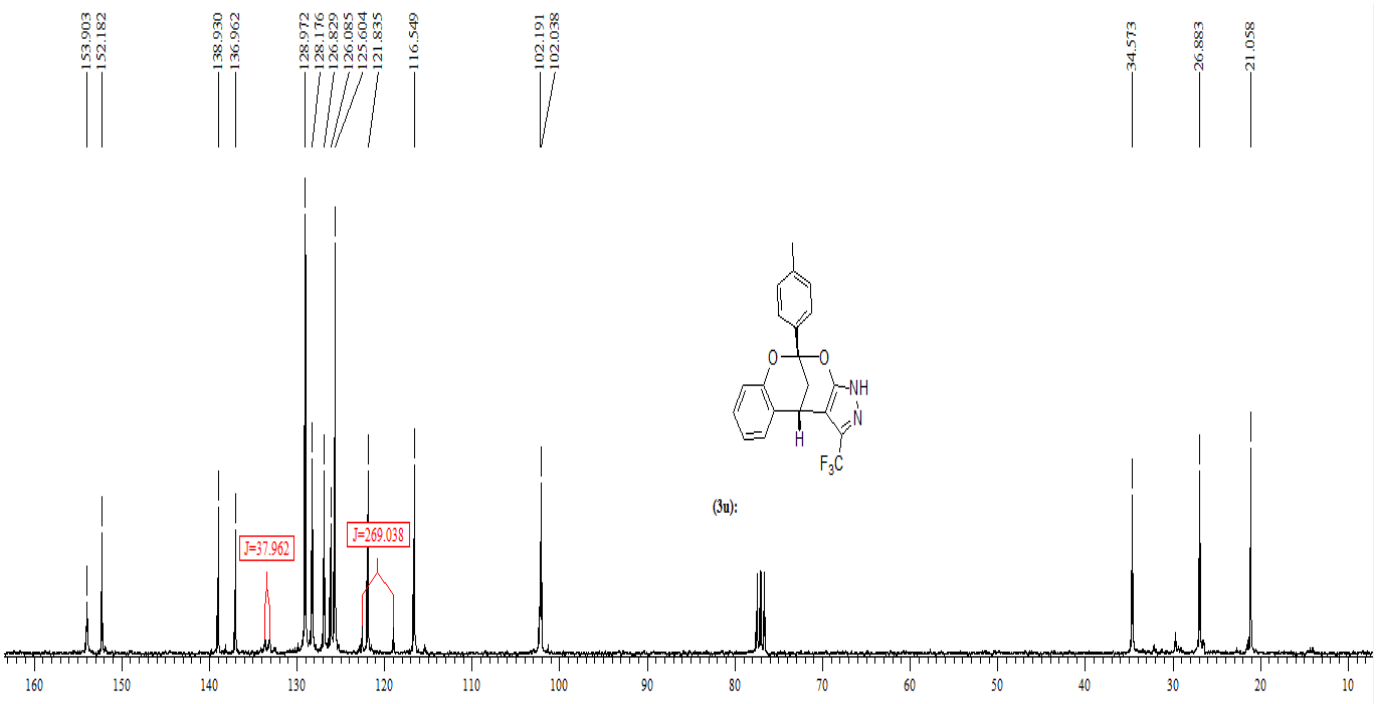
^{1}H NMR (300 MHz) spectrum of **3t** in $CDCl_3$



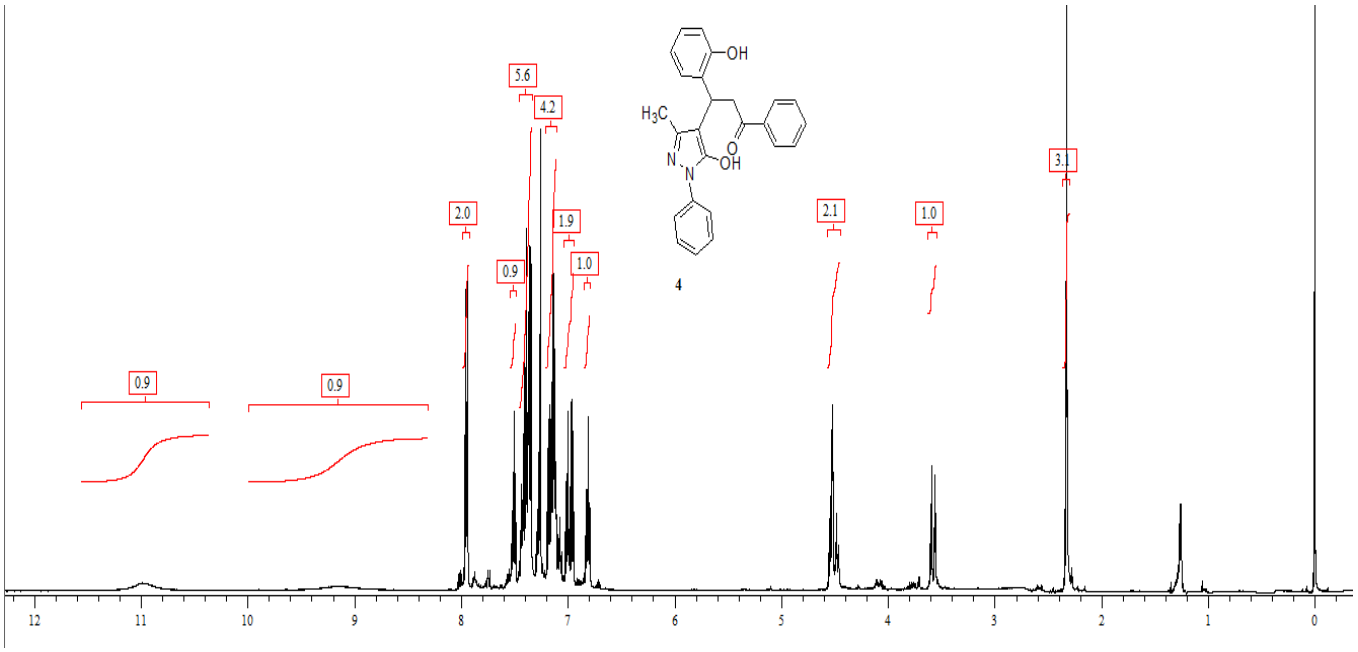
^{13}C NMR (125 MHz) spectrum of **3t** in $CDCl_3$



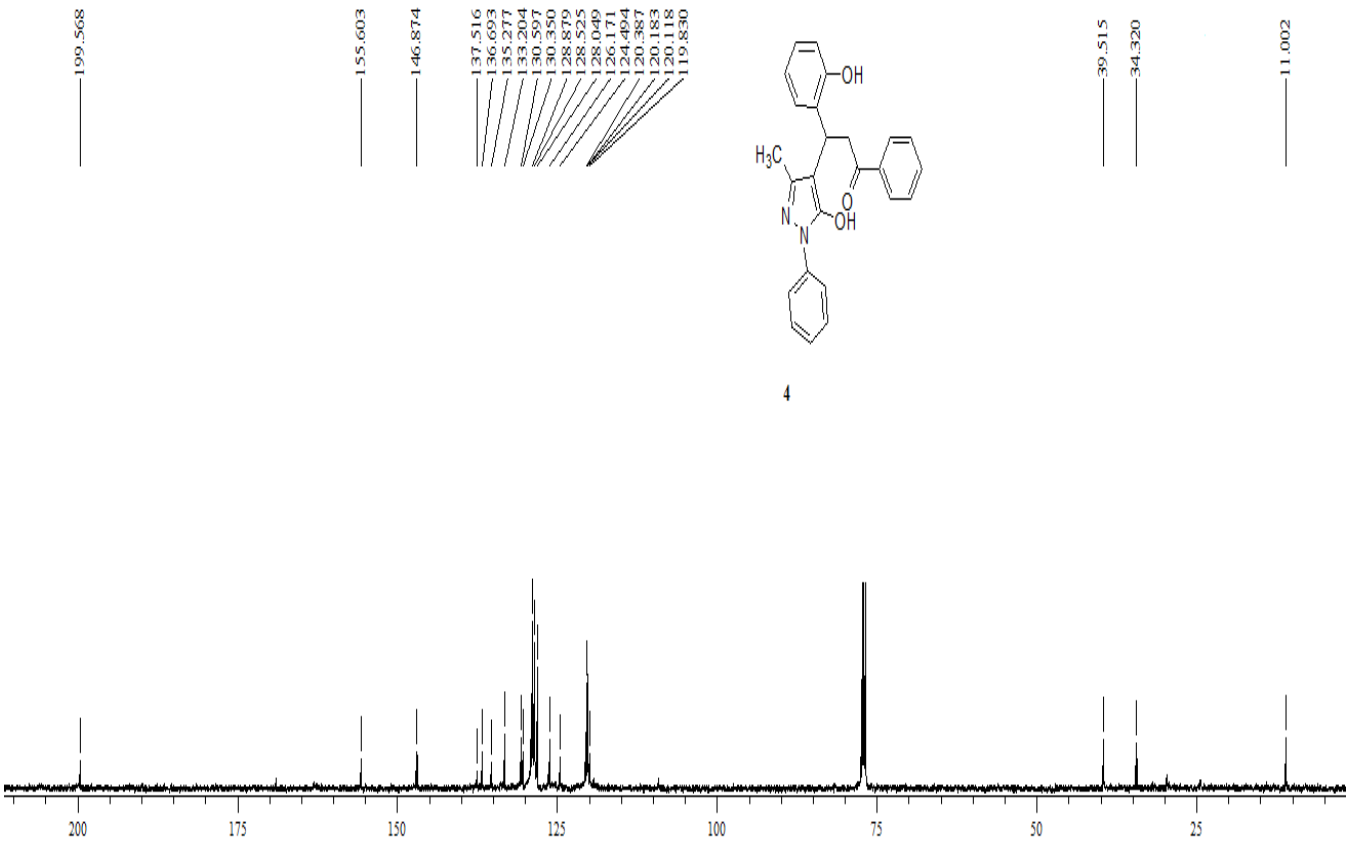
^{1}H NMR (500 MHz) spectrum of **3u** in $CDCl_3$



^{13}C NMR (125 MHz) spectrum of **3u** in $CDCl_3$



^{1}H NMR (500 MHz) spectrum of **4** in $CDCl_3$



^{13}C NMR (125 MHz) spectrum of **4** in $CDCl_3$