

One-pot two component tandem multiple transformations in the synthesis of *N*,4-diaryl-2,3-dihydropyrrolo[3,4-*c*]quinolin-1,3-diones and their 3-thioxo-analogues in neat phase

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Supporting information for Publication

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General Information: β-Aroyl-thioacetanilides (**5**, X = S) are prepared as reported in literature. Isatine derivatives (**1**), DMAP (4-dimethylaminopyridine) and solvents used are commercially available. IR spectra of all the compounds are recorded in KBr on FT-IR spectrometer and ν values are reported in reciprocal centimetres (cm⁻¹). ¹H NMR spectra are recorded at 300 MHz with TMS as internal standard ($\delta = 0$) and the values are reported as follows: chemical shift (δ ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), coupling constants (in Hz) and integration. For ¹³C NMR at 75 MHz, CDCl₃ ($\delta = 77.27$) and/or TFA ($\delta = 115$ q, 161 q) are used as internal standards and spectra are obtained with complete proton decoupling. Low-resolution MS and HRMS data are obtained using Electron Spray Ionization (ESI) and high-resolution spectra are recorded on QSTARXL hybrid MS/MS system under ESI.

All the reactions are monitored by thin layer chromatography (TLC) on precoated silica gel 60 F₂₅₄ (mesh) and spots are visualized under UV light.

X-ray Crystallography Data of compound 3a:

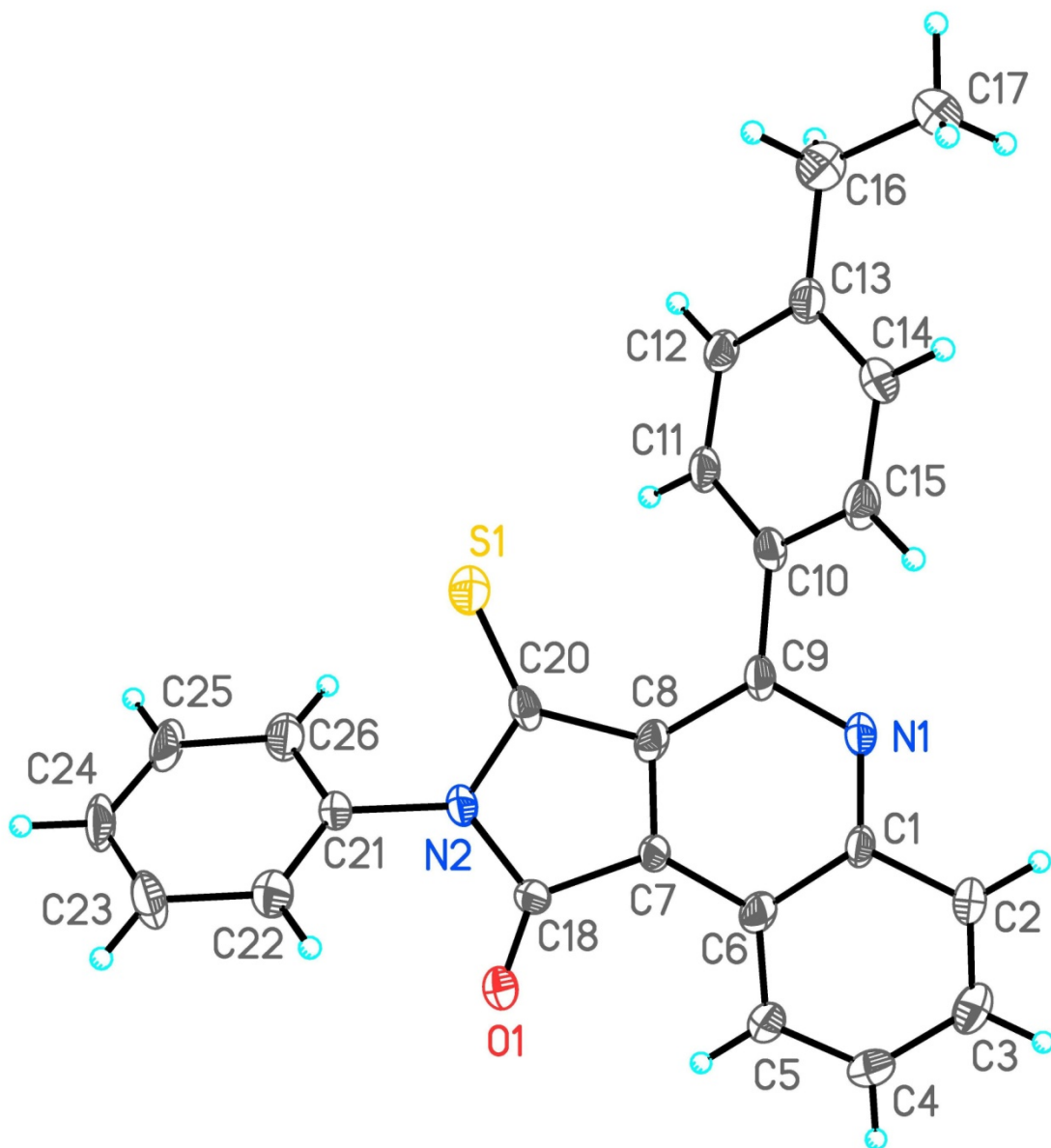


Figure caption: The molecular structure of **3a**, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radius.

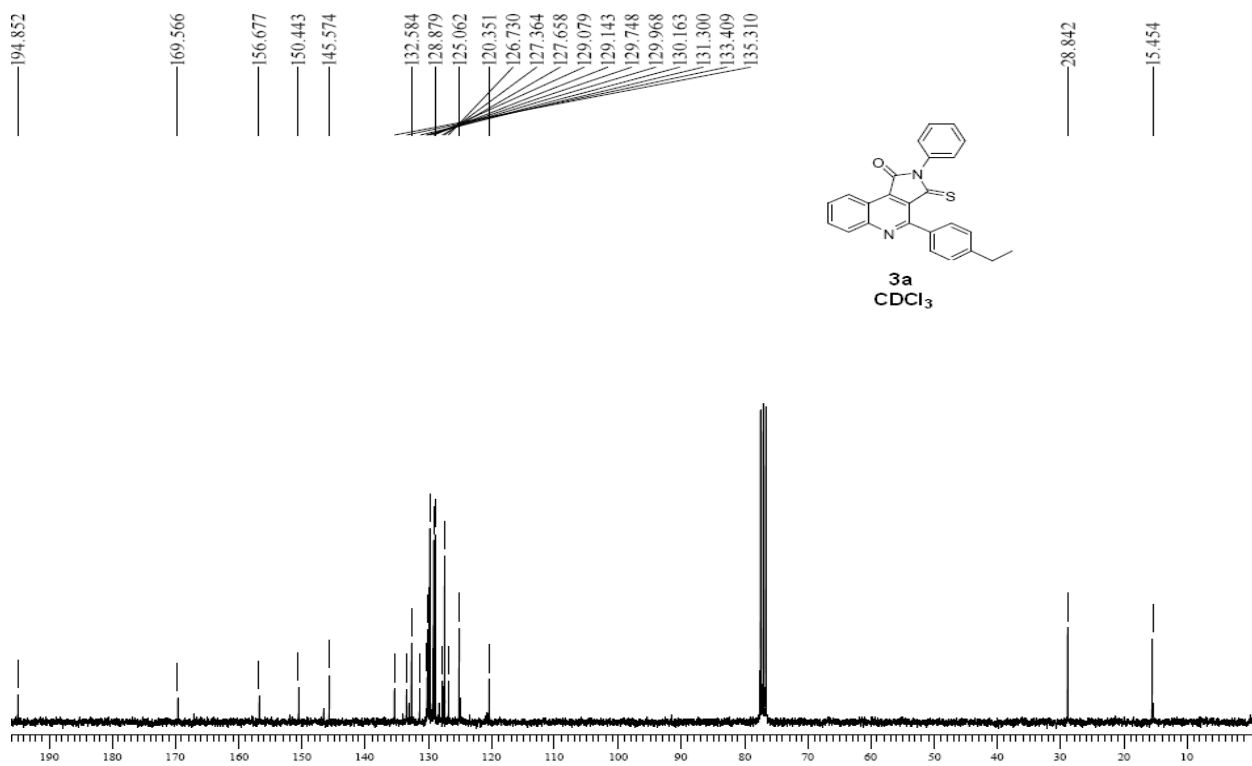
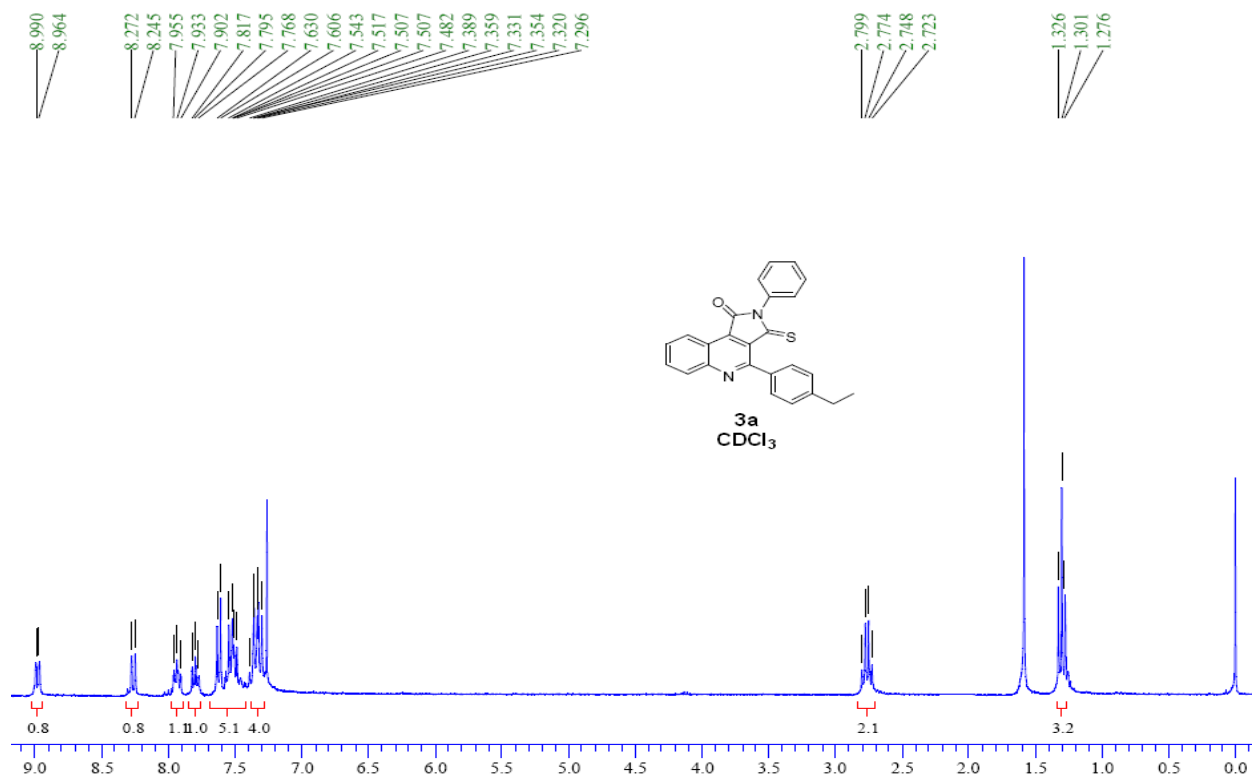
Crystallographic data for 3a

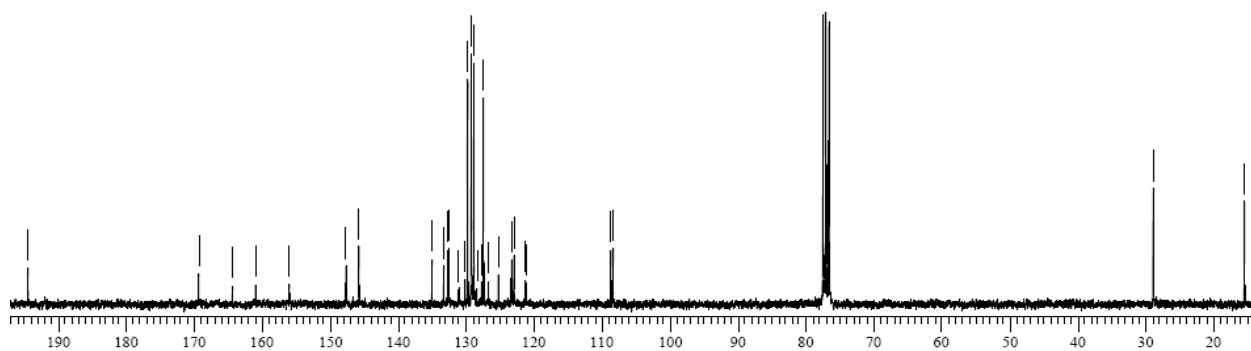
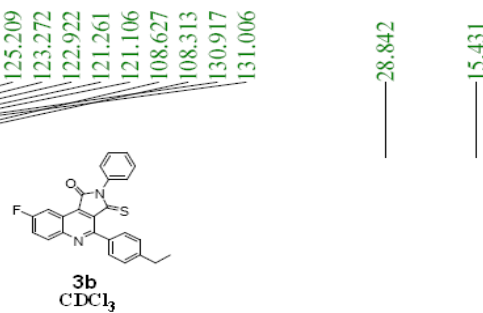
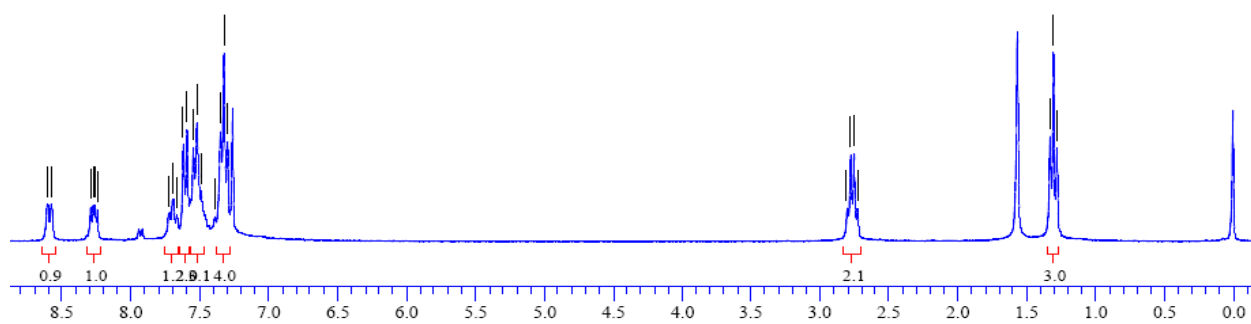
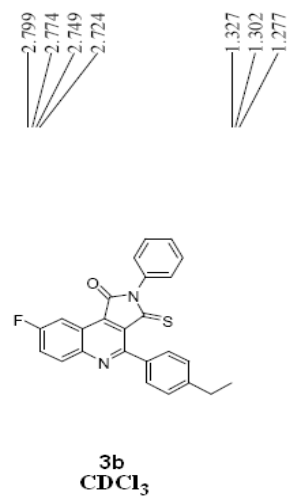
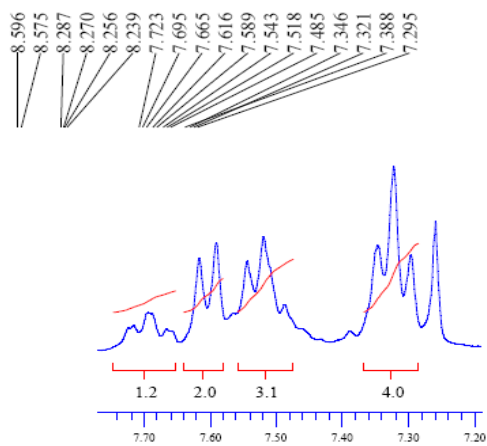
X-ray data for the compound were collected at room temperature with a graphite monochromated MoK α radiation ($\lambda=0.71073\text{\AA}$) with ω -scan method.¹ Preliminary lattice parameters and orientation matrices were obtained from four sets of frames. Unit cell dimensions were determined using 3740 reflections for **3a** data. Integration and scaling of intensity data were accomplished using SAINT program.¹ The structures were solved by Direct Methods using SHELXS97² 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 \AA , and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $1.5U_{\text{eq}}(\text{C})$ for methyl atoms.

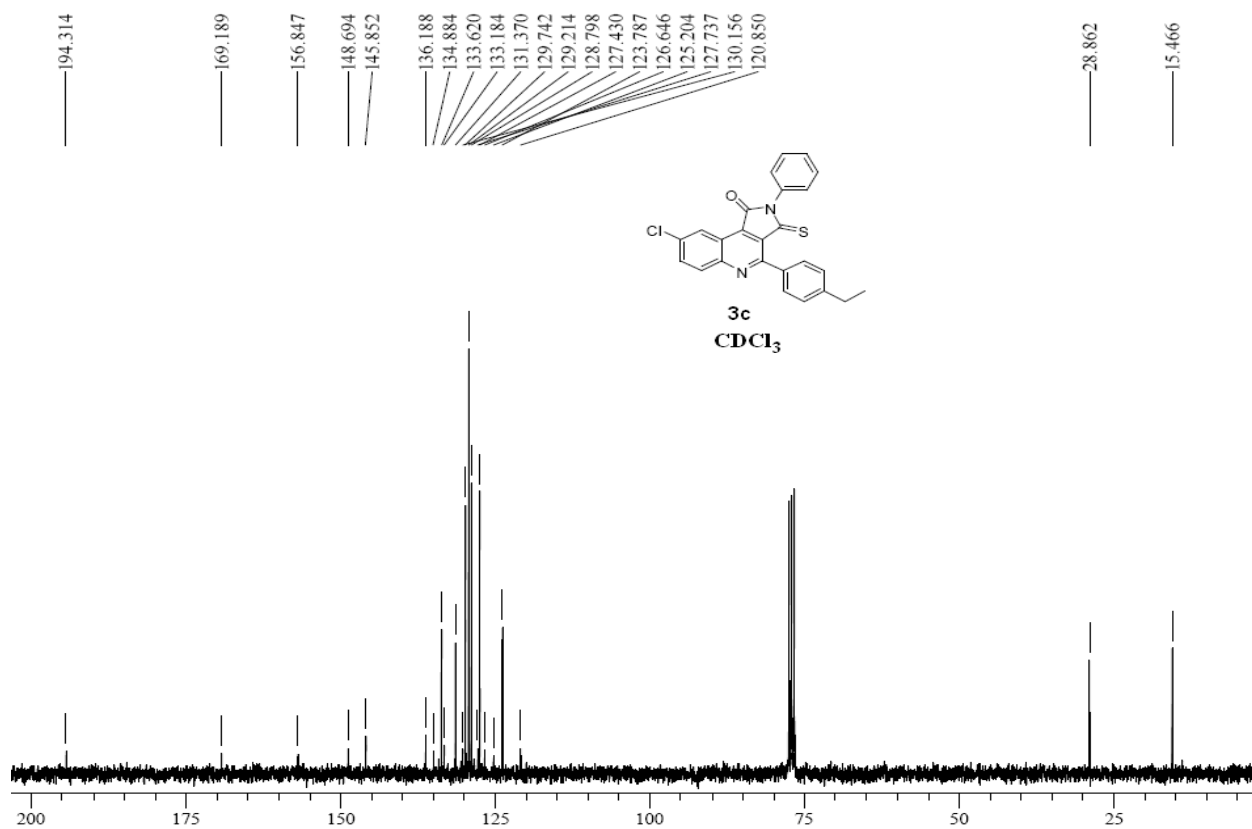
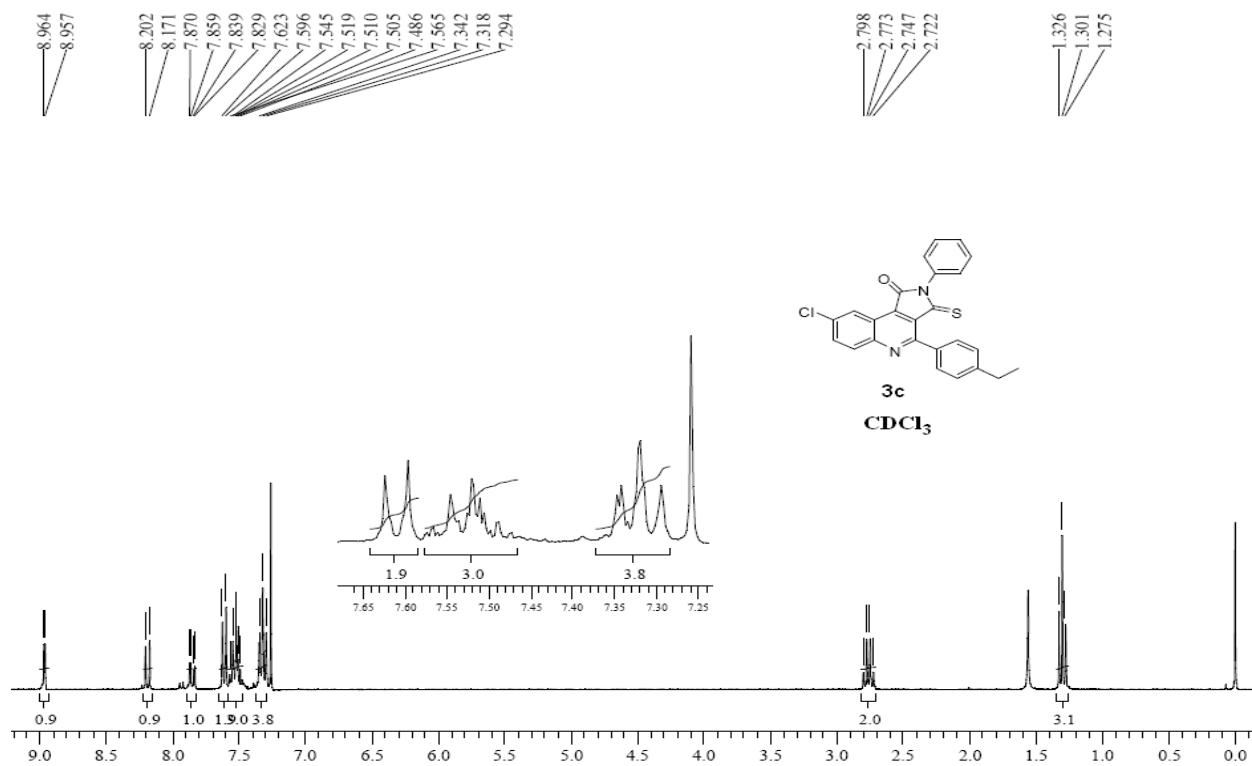
Crystal data for AO68: C₂₅H₁₈N₂OS, $M = 394.47$, colourless needle, $0.42 \times 0.23 \times 0.11$ mm³, orthorhombic, space group $P2_12_12_1$ (No. 19), $a = 4.2876(5)$, $b = 19.102(2)$, $c = 23.711(3)$ \AA , $V = 1942.0(4)$ \AA^3 , $Z = 4$, $D_c = 1.349$ g/cm³, $F_{000} = 824$, CCD area detector, Mo-K α radiation, $\lambda = 0.71073$ \AA , $T = 293(2)\text{K}$, $2\theta_{\text{max}} = 50.0^\circ$, 18599 reflections collected, 3411 unique ($R_{\text{int}} = 0.0629$). Final $Goof = 1.315$, $RI = 0.0867$, $wR2 = 0.1938$, R indices based on 3223 reflections with $I > 2\sigma(I)$ (refinement on F^2), 263 parameters, $\mu = 0.186$ mm⁻¹. Absolute structure parameter (Flack parameter)³ = 0.16(19). CCDC 922749 contains the supplementary crystallographic data for this paper. 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].

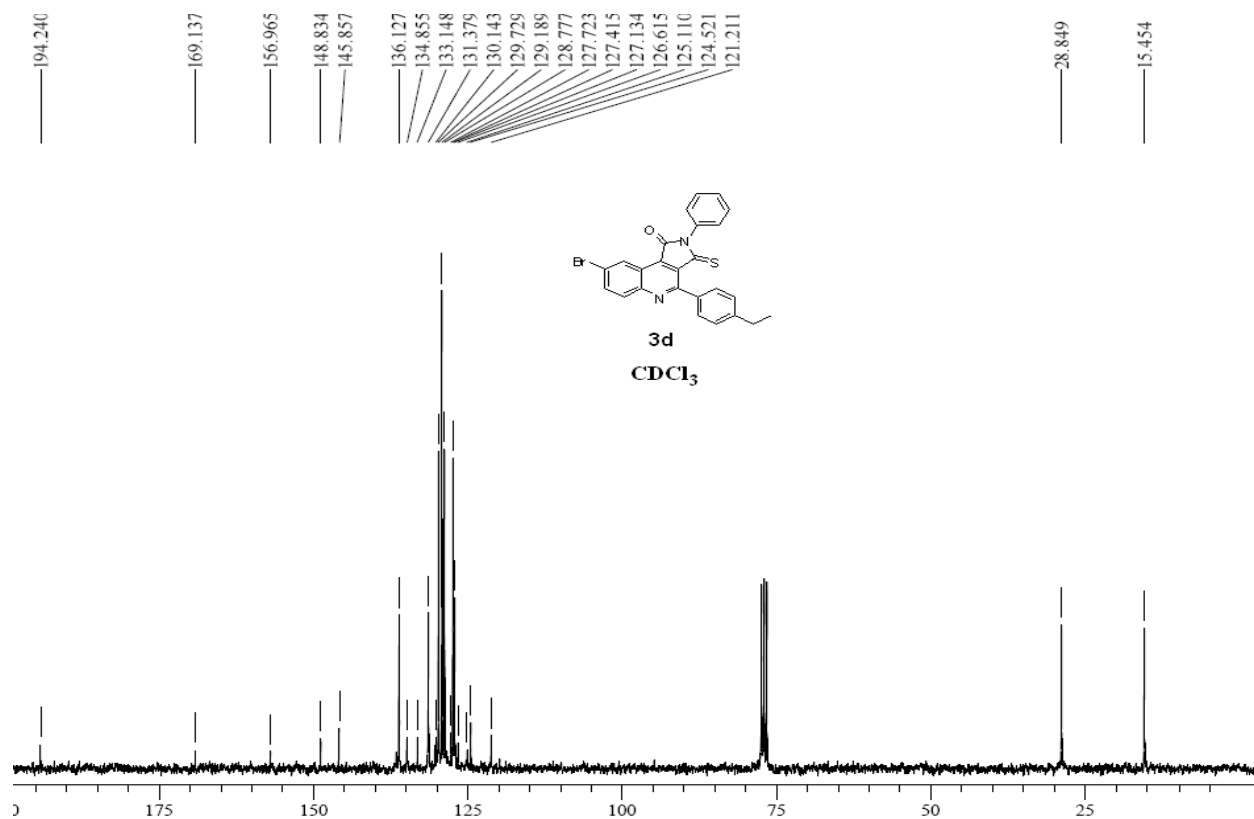
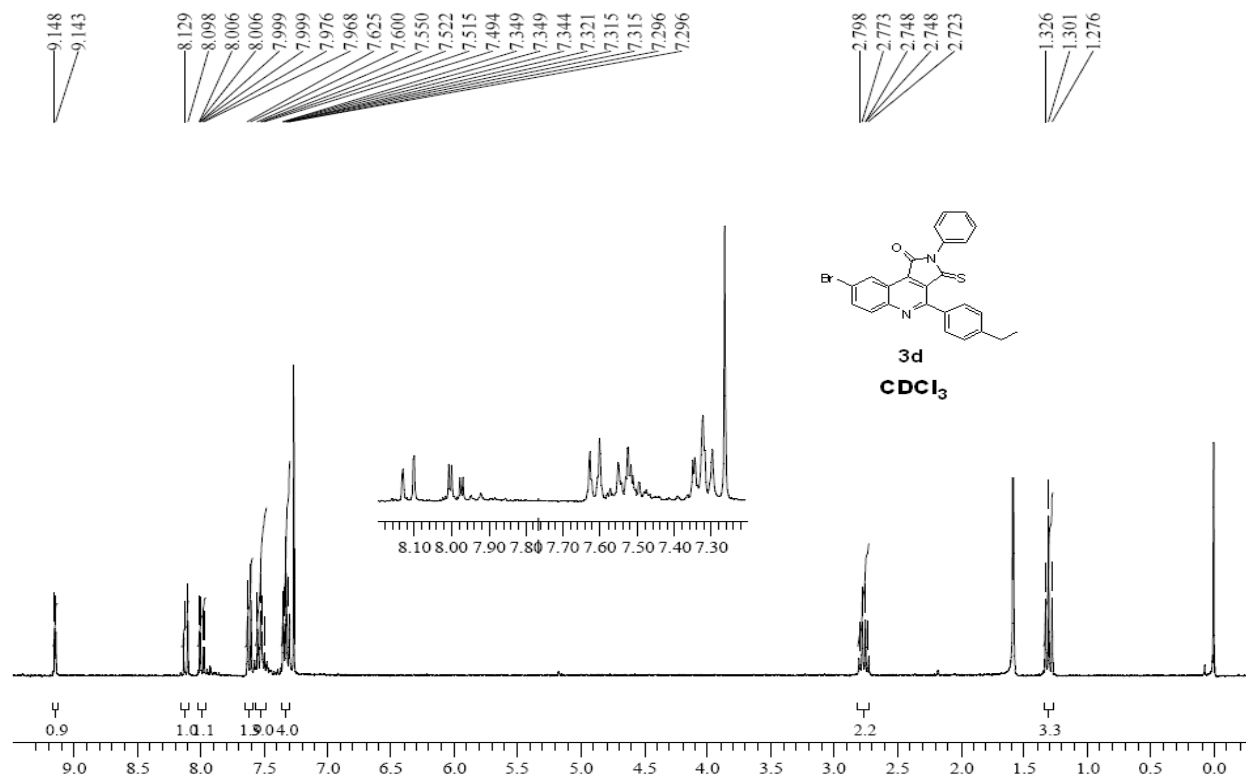
1. SMART & SAINT. Software Reference manuals. Versions 6.28a & 5.625, Bruker Analytical X-ray Systems Inc., Madison, Wisconsin, U.S.A., 2001.
2. Sheldrick, G. M. SHELXS97 and SHELXL97, Programs for crystal structure solution and refinement; University of Gottingen: Germany, 1997.
3. Flack, H. D. *Acta Cryst.* **1983**, *A39*, 876-881.

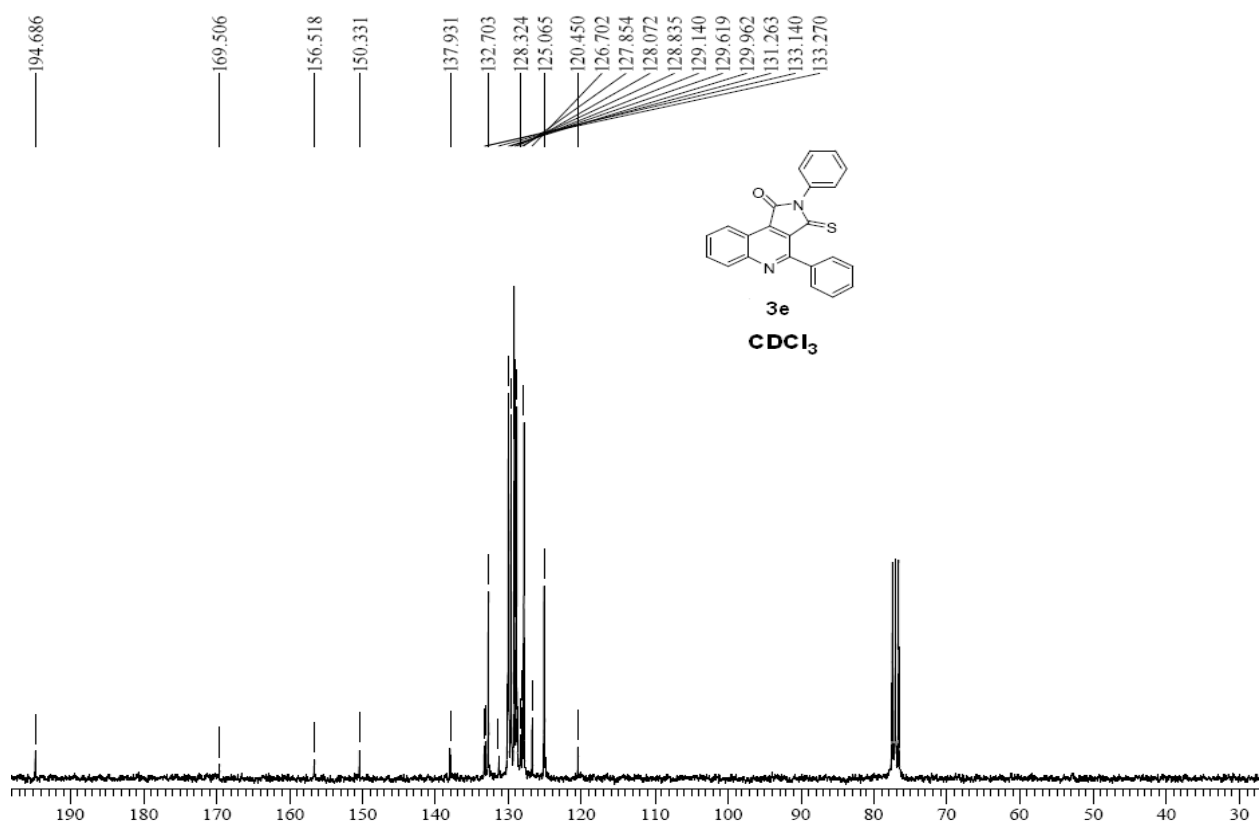
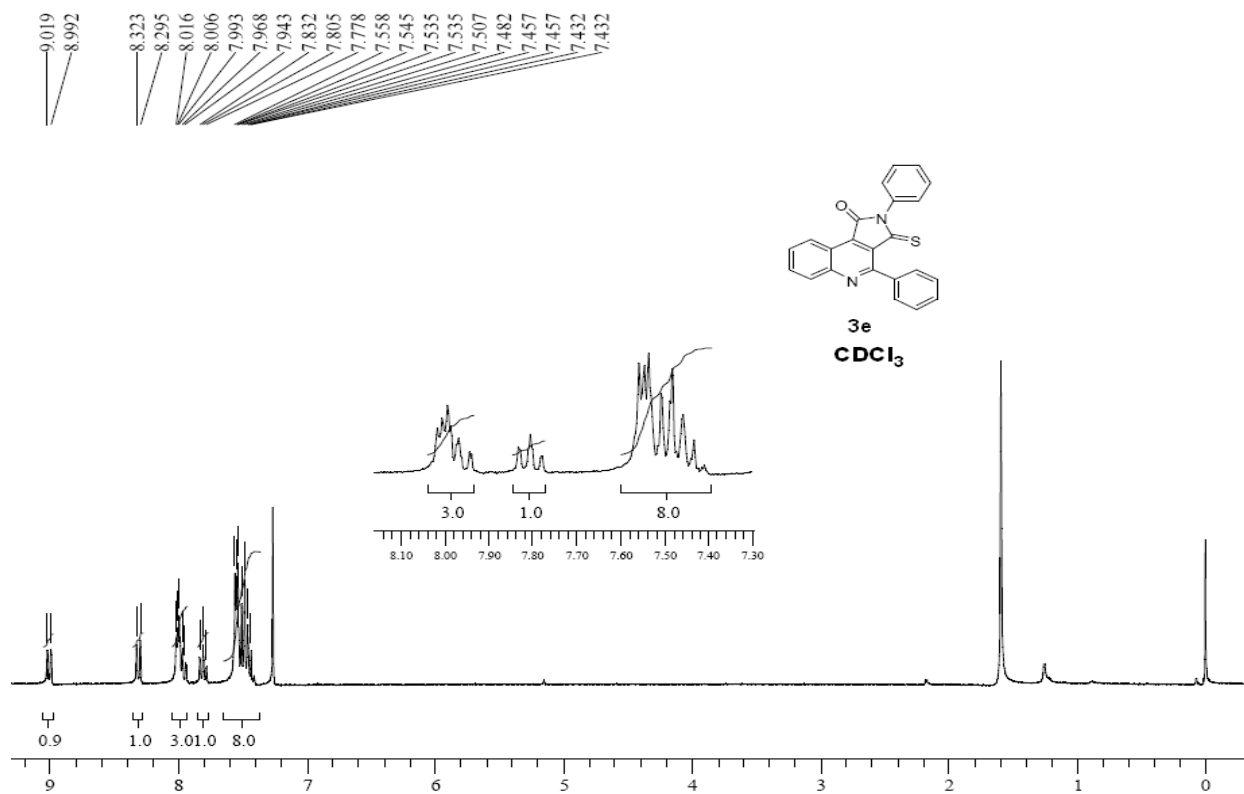
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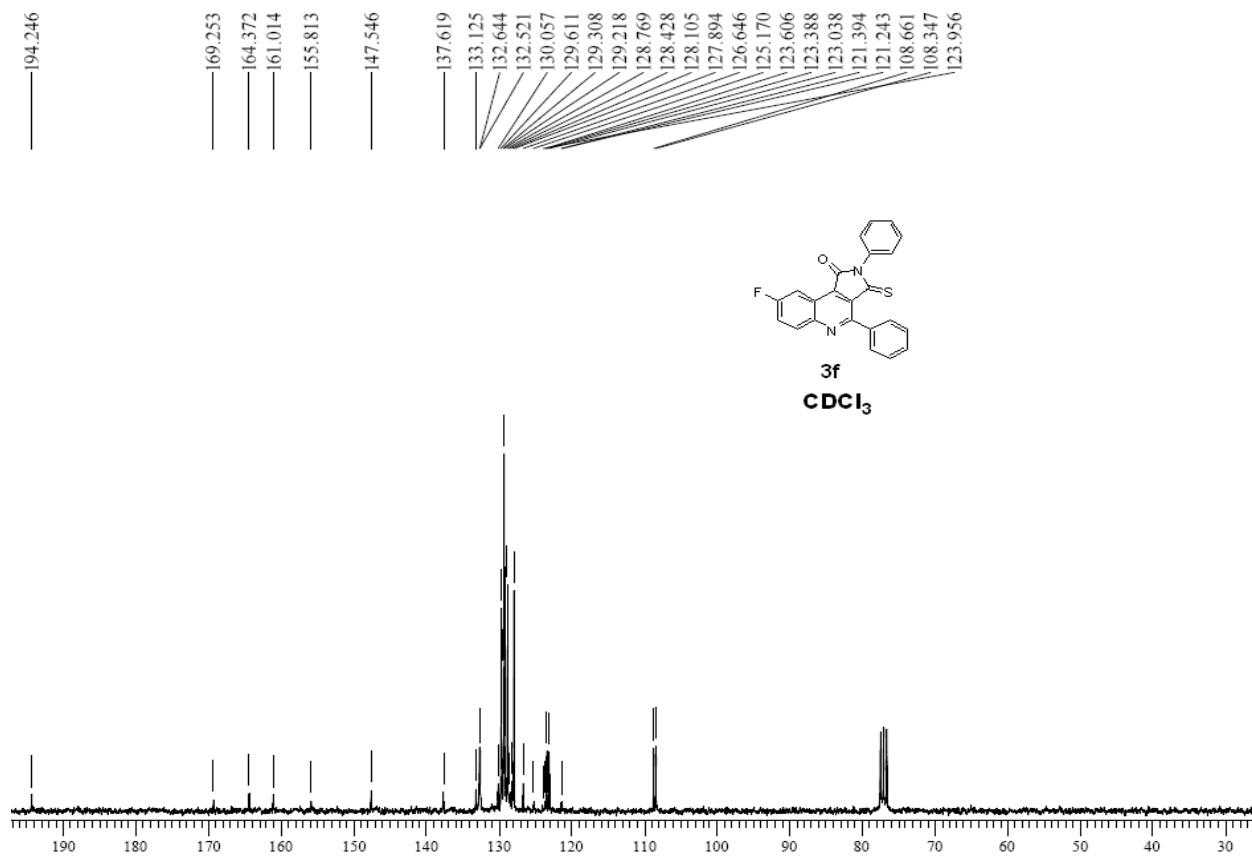
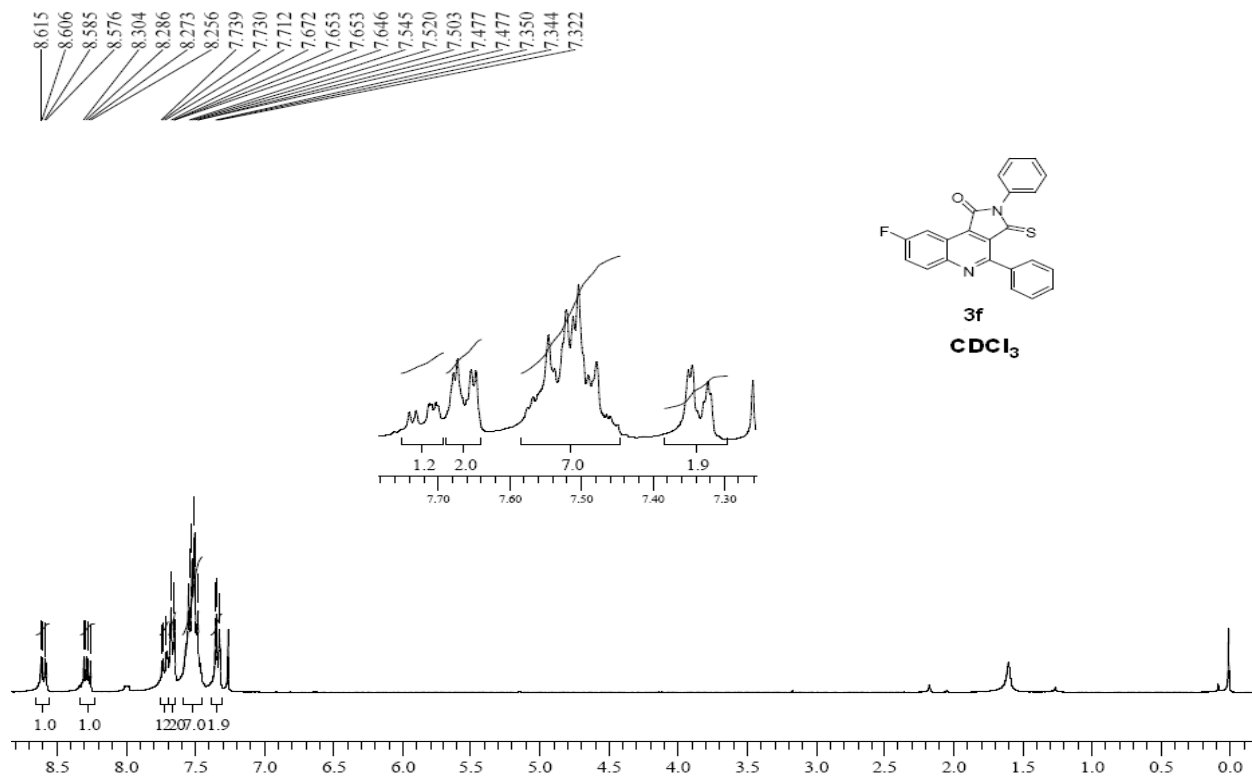


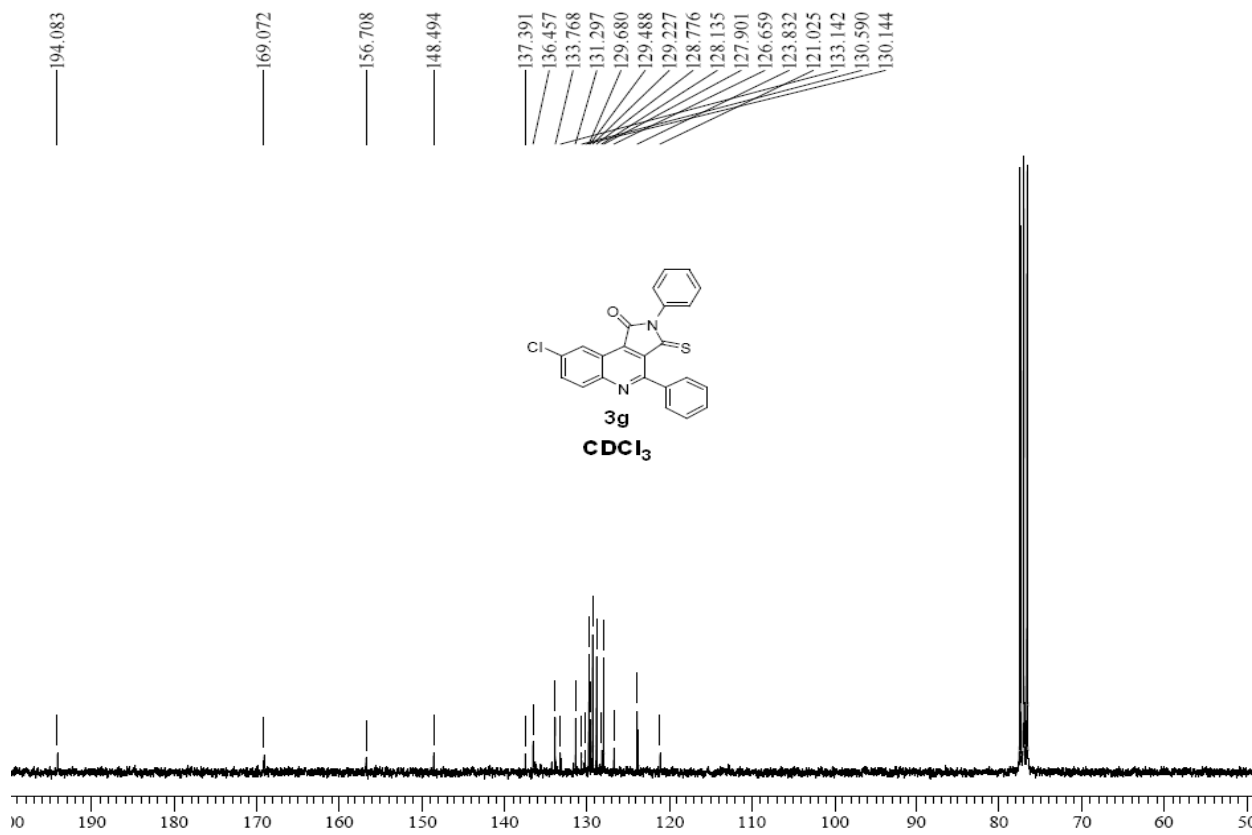
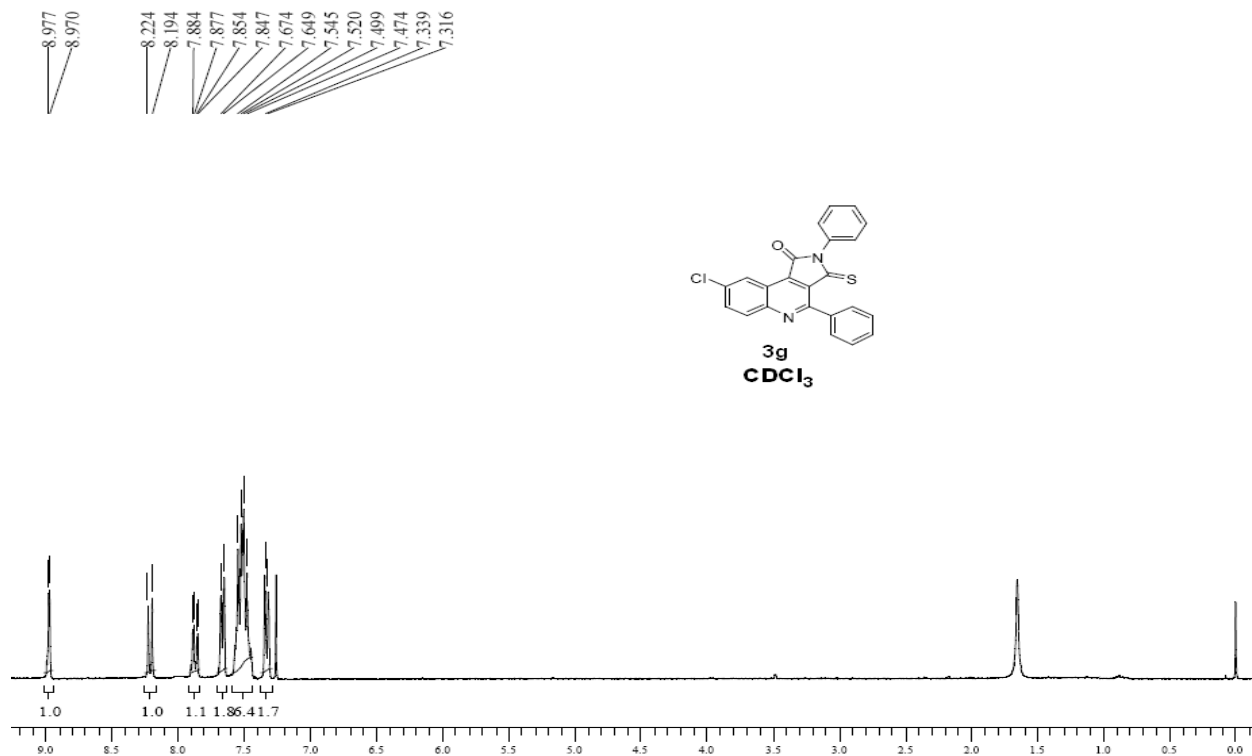


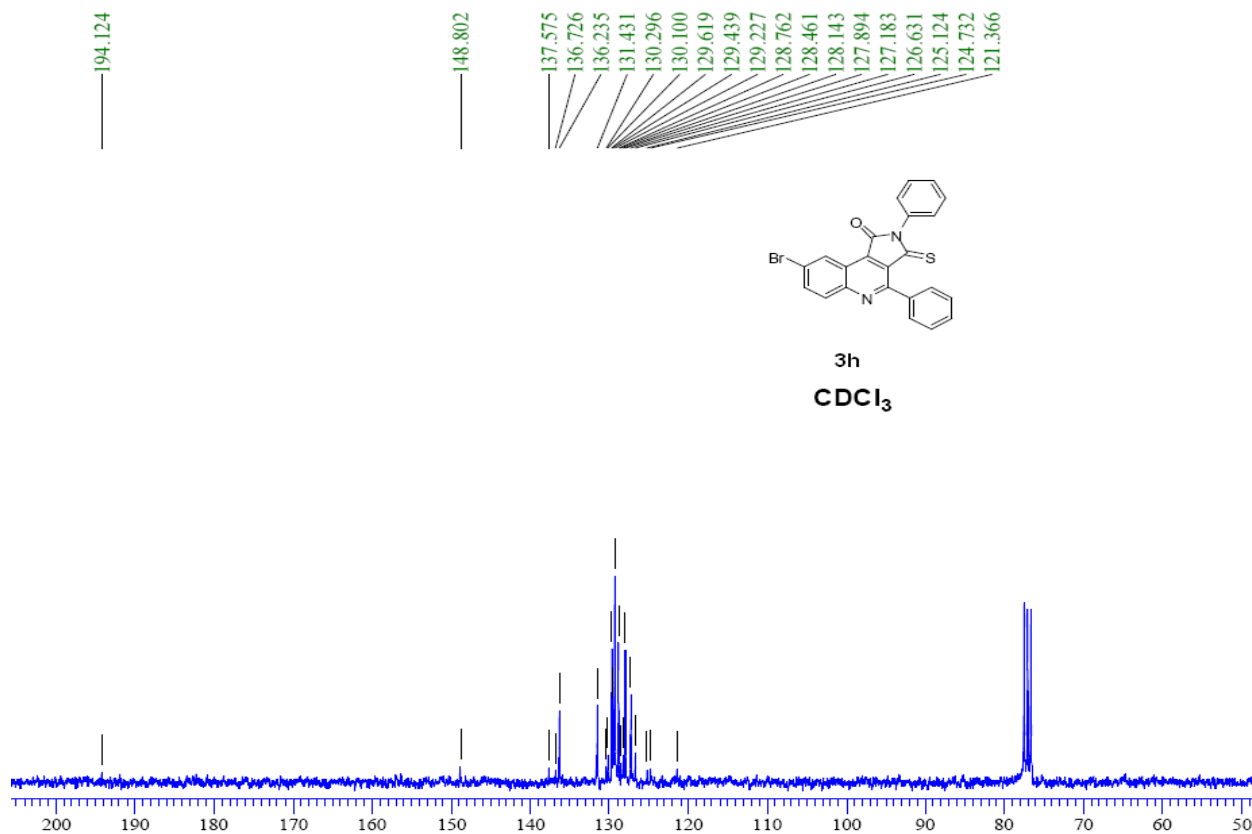
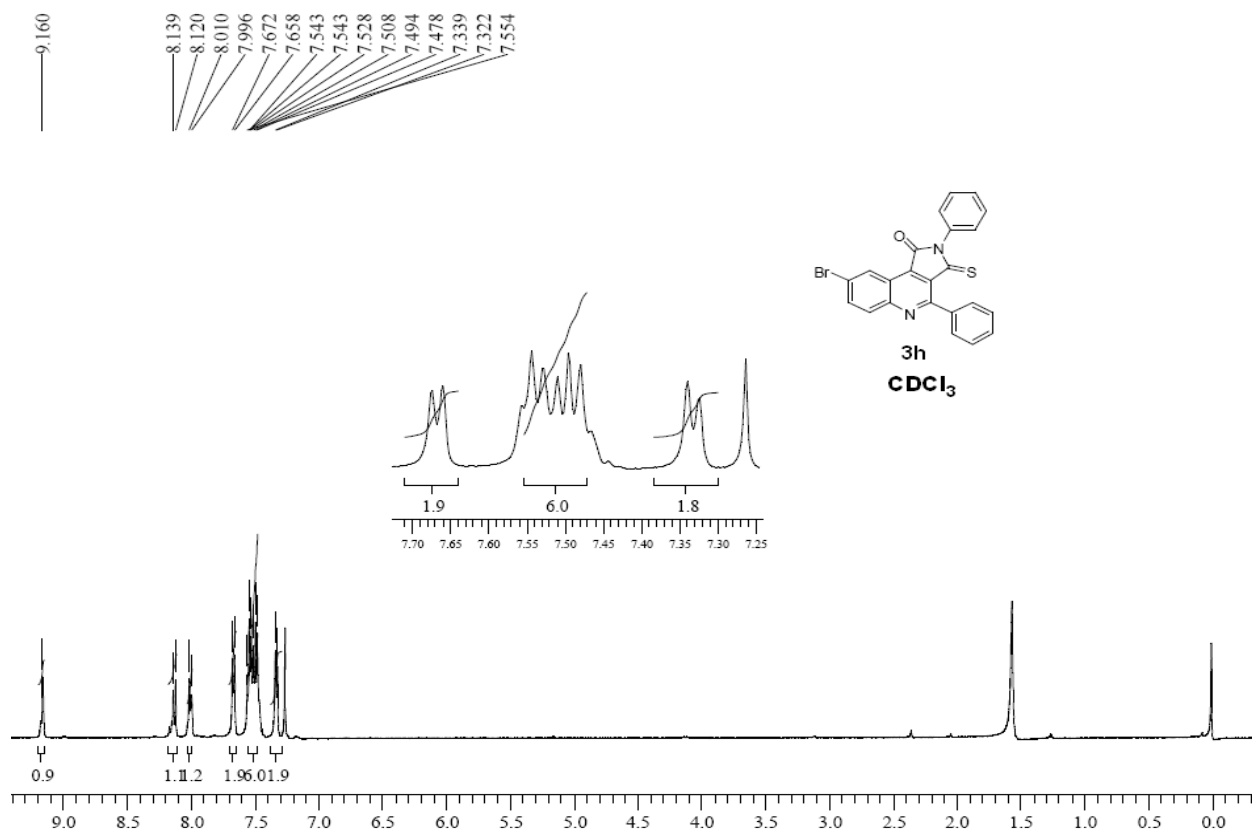


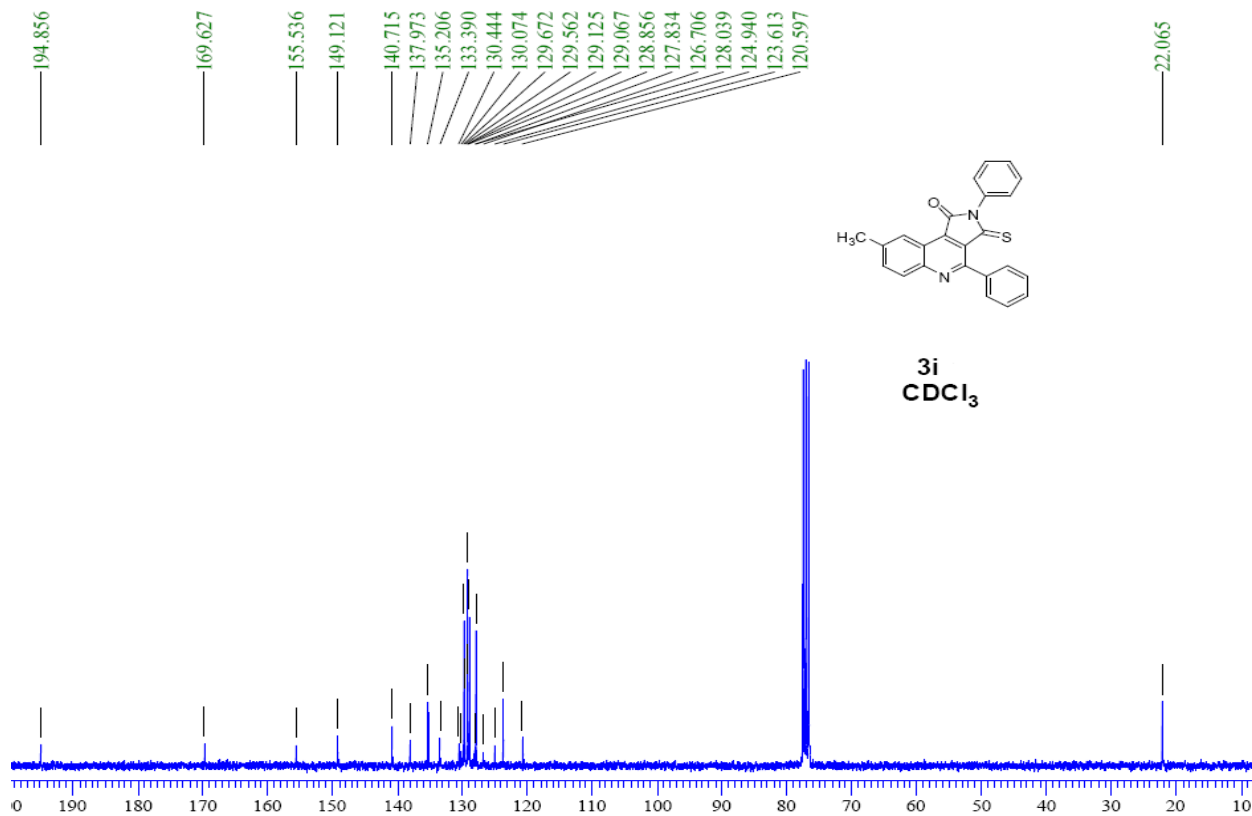
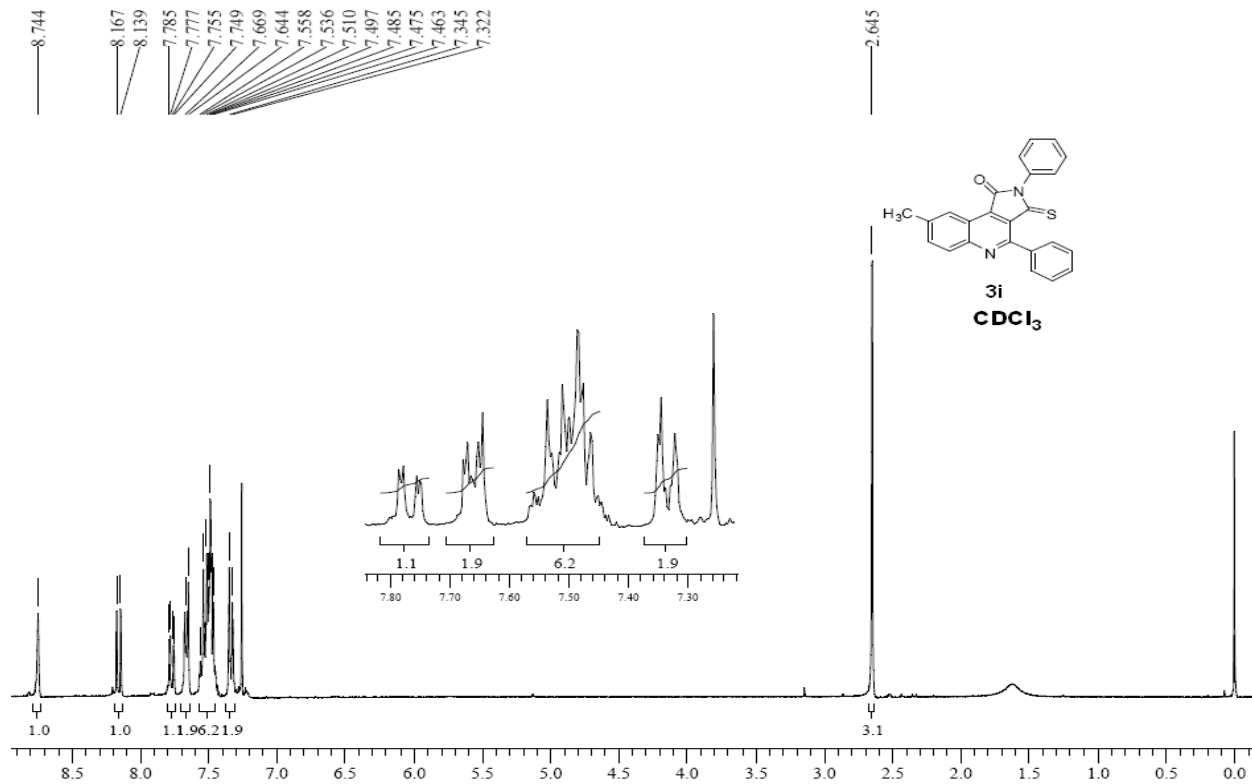


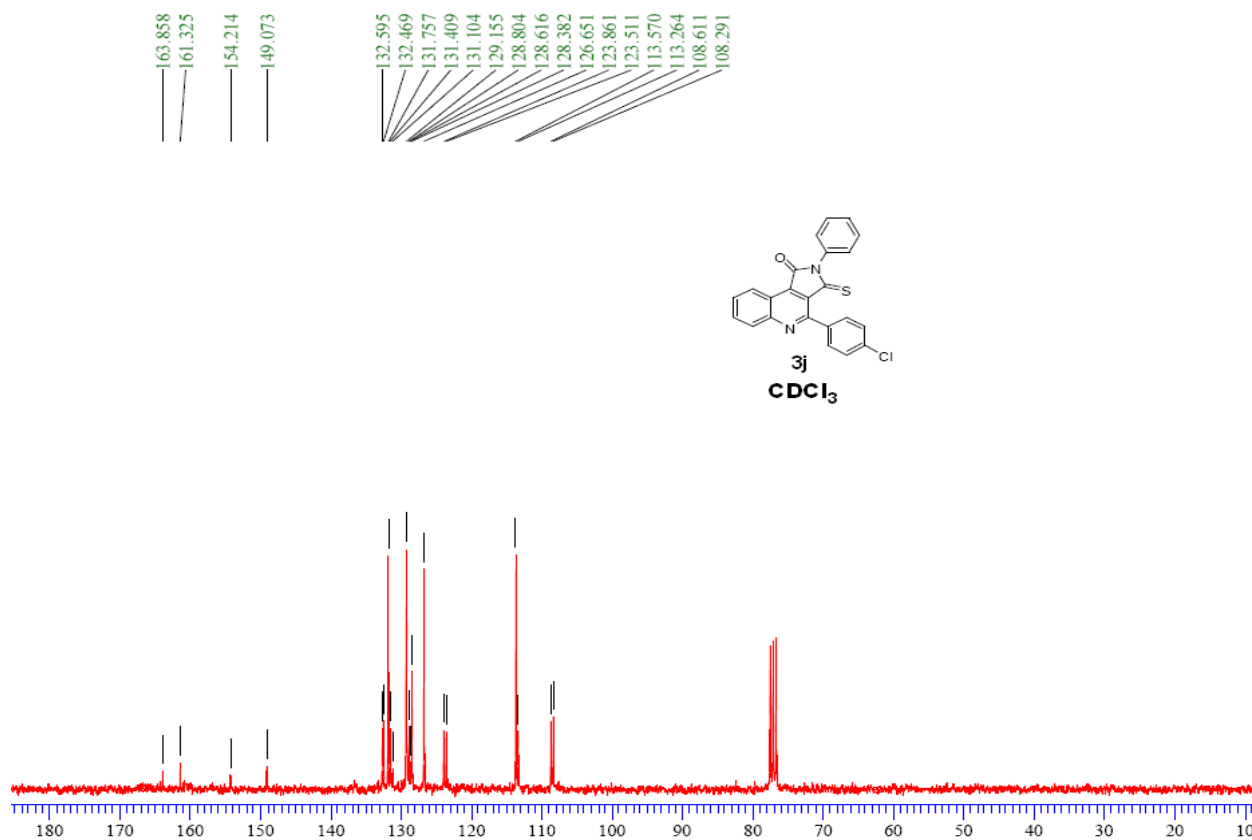
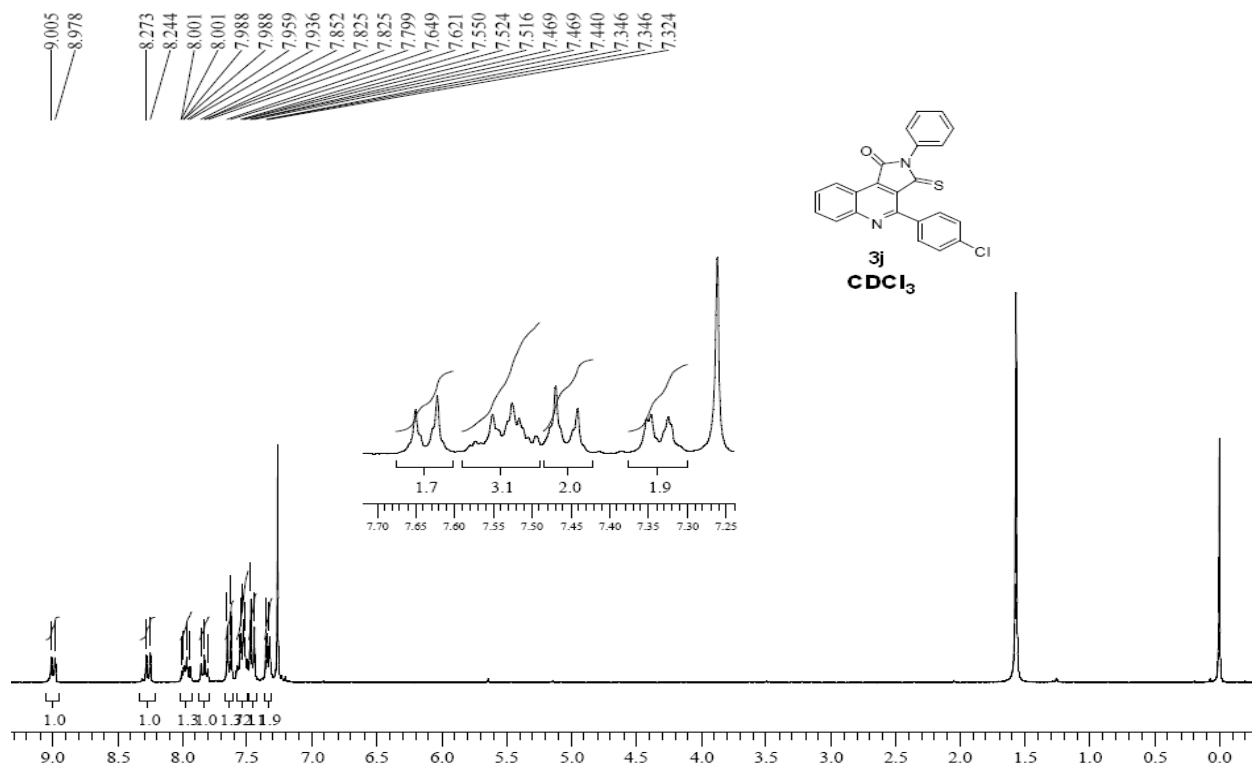


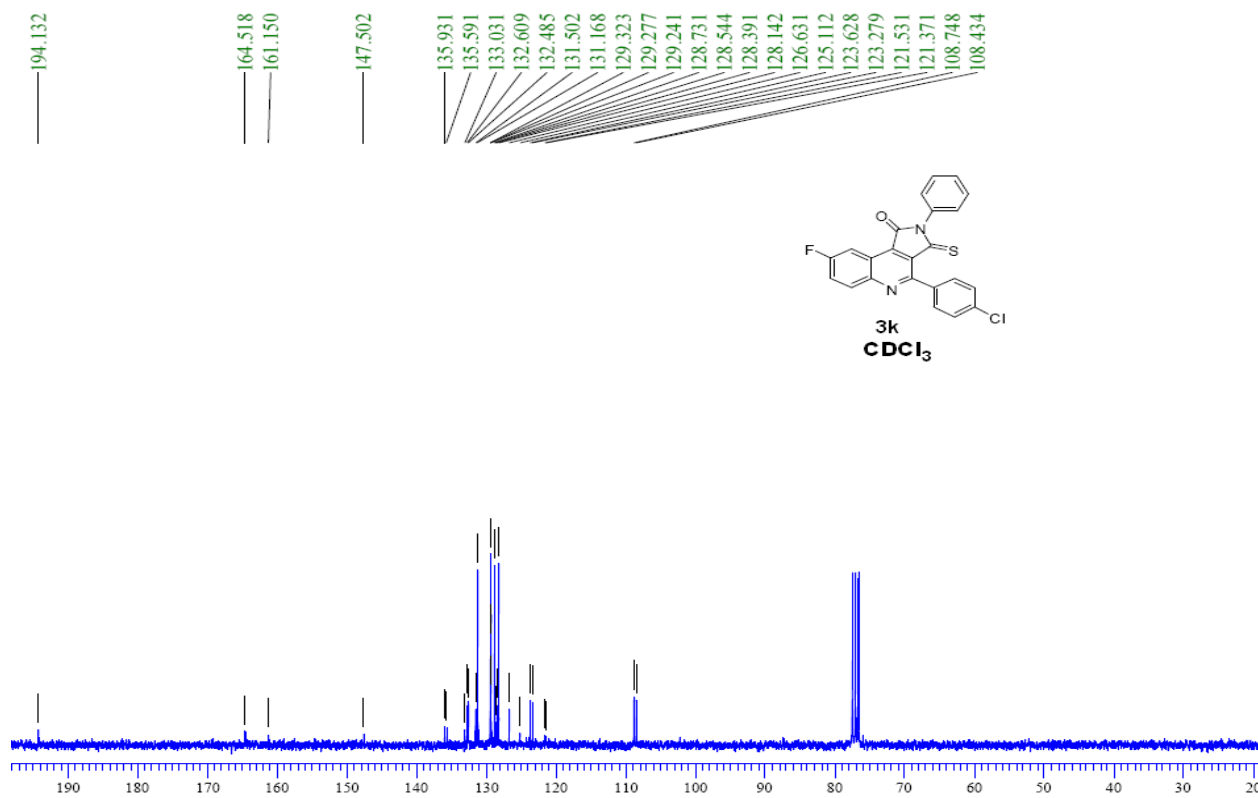
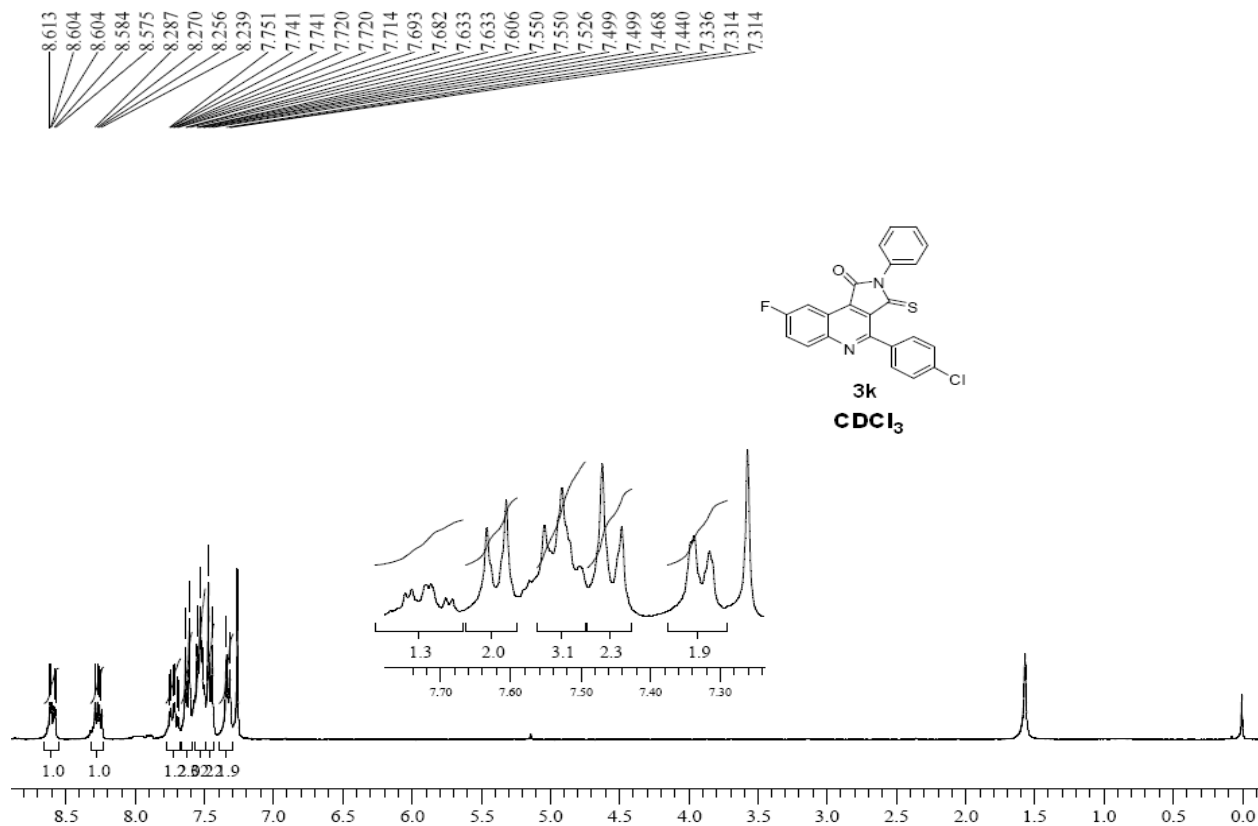


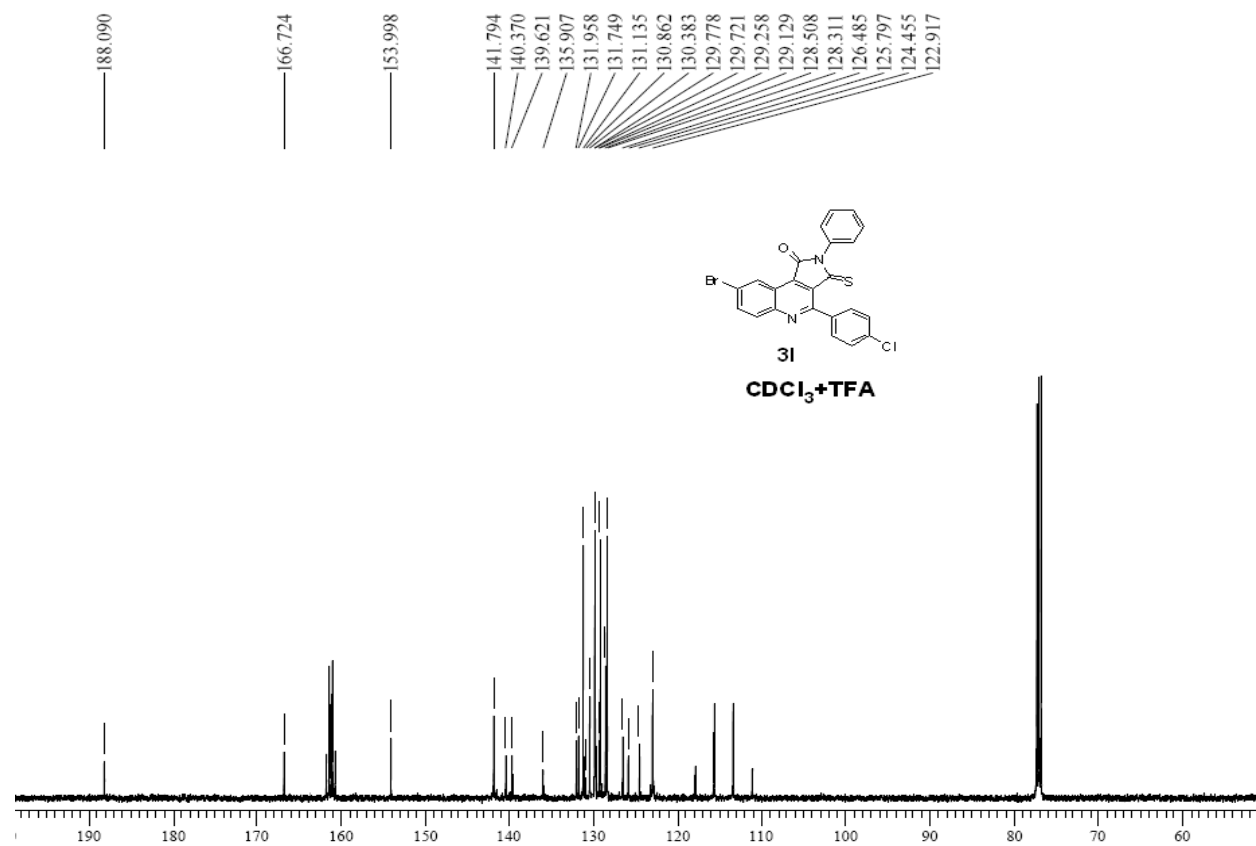
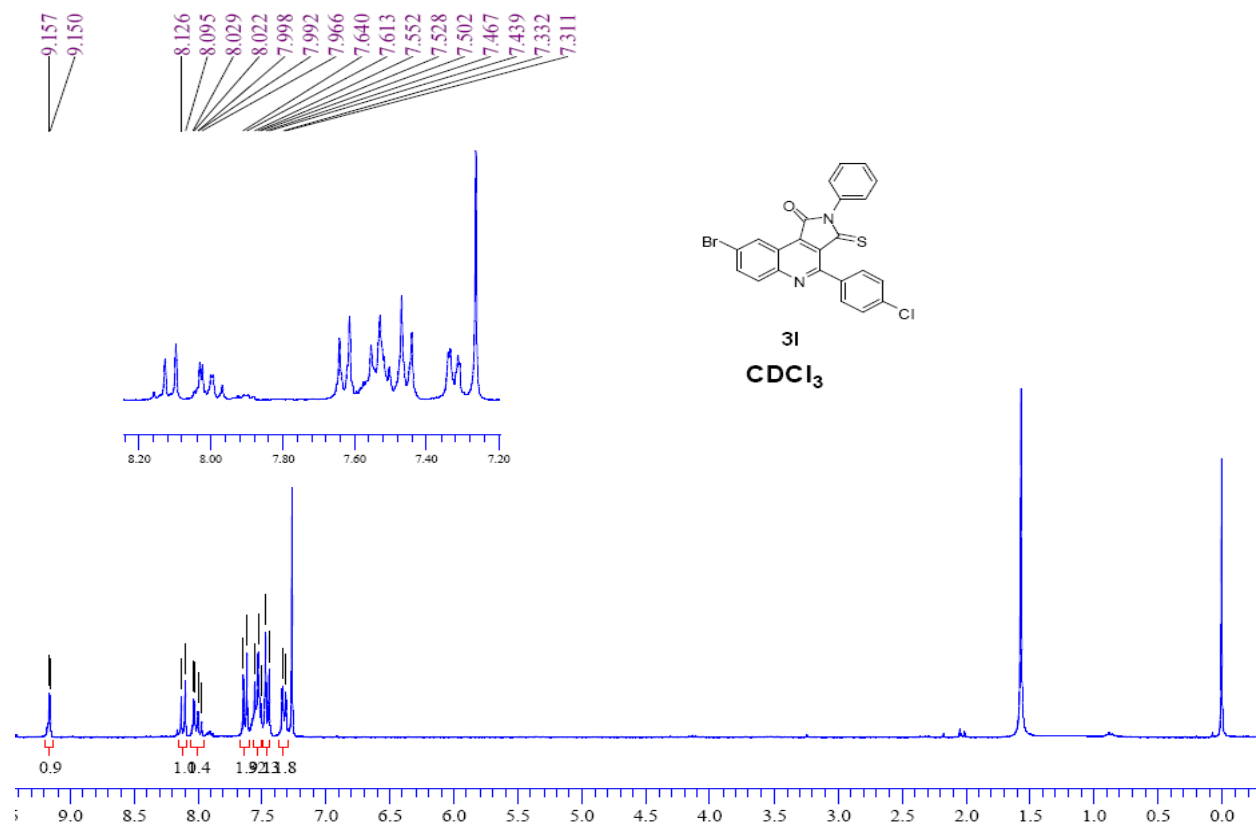


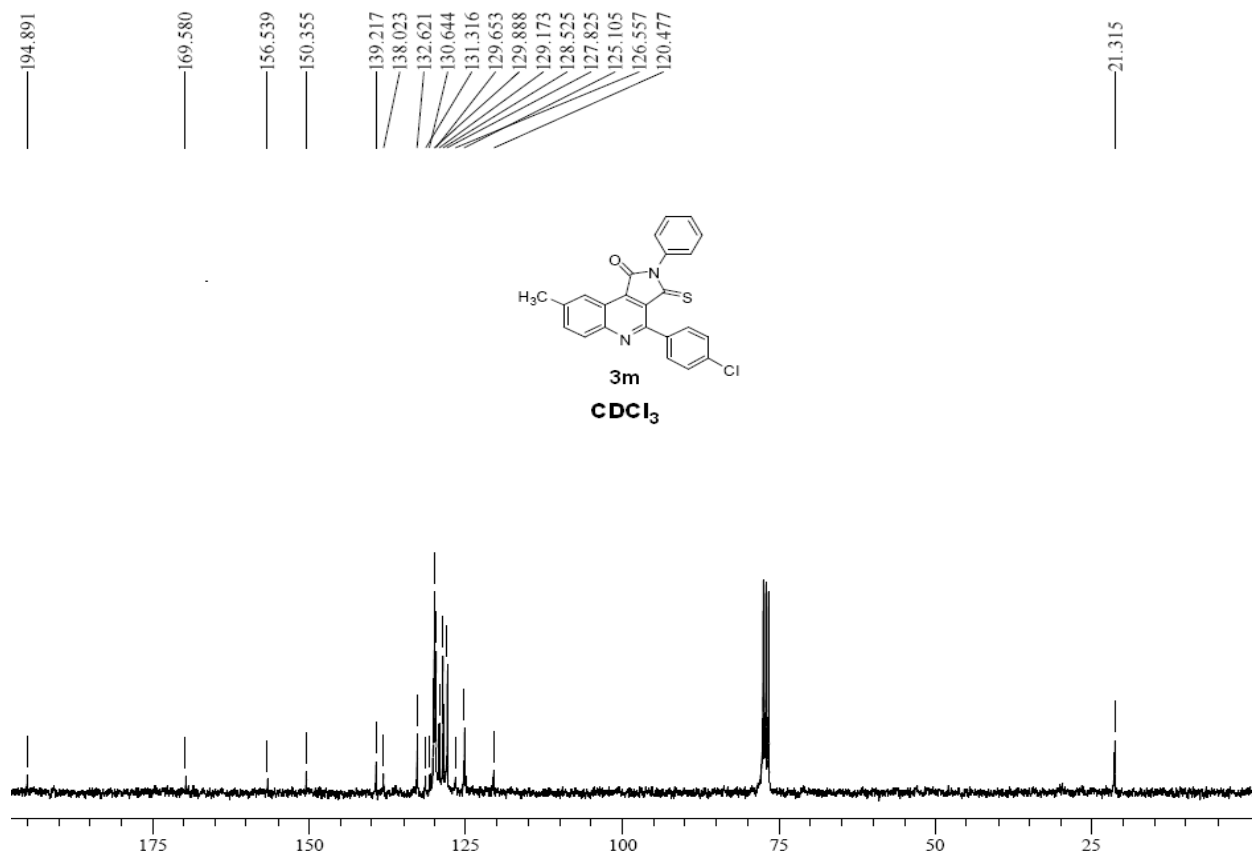
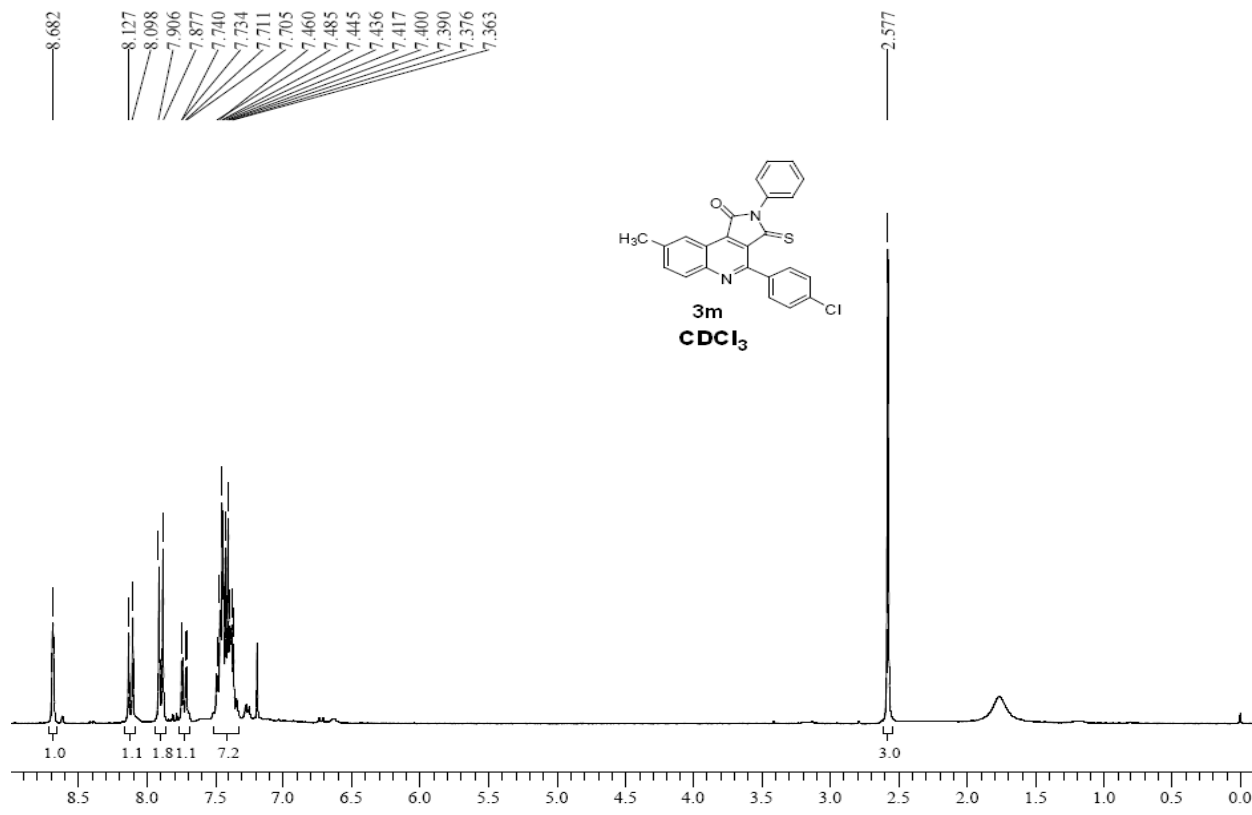


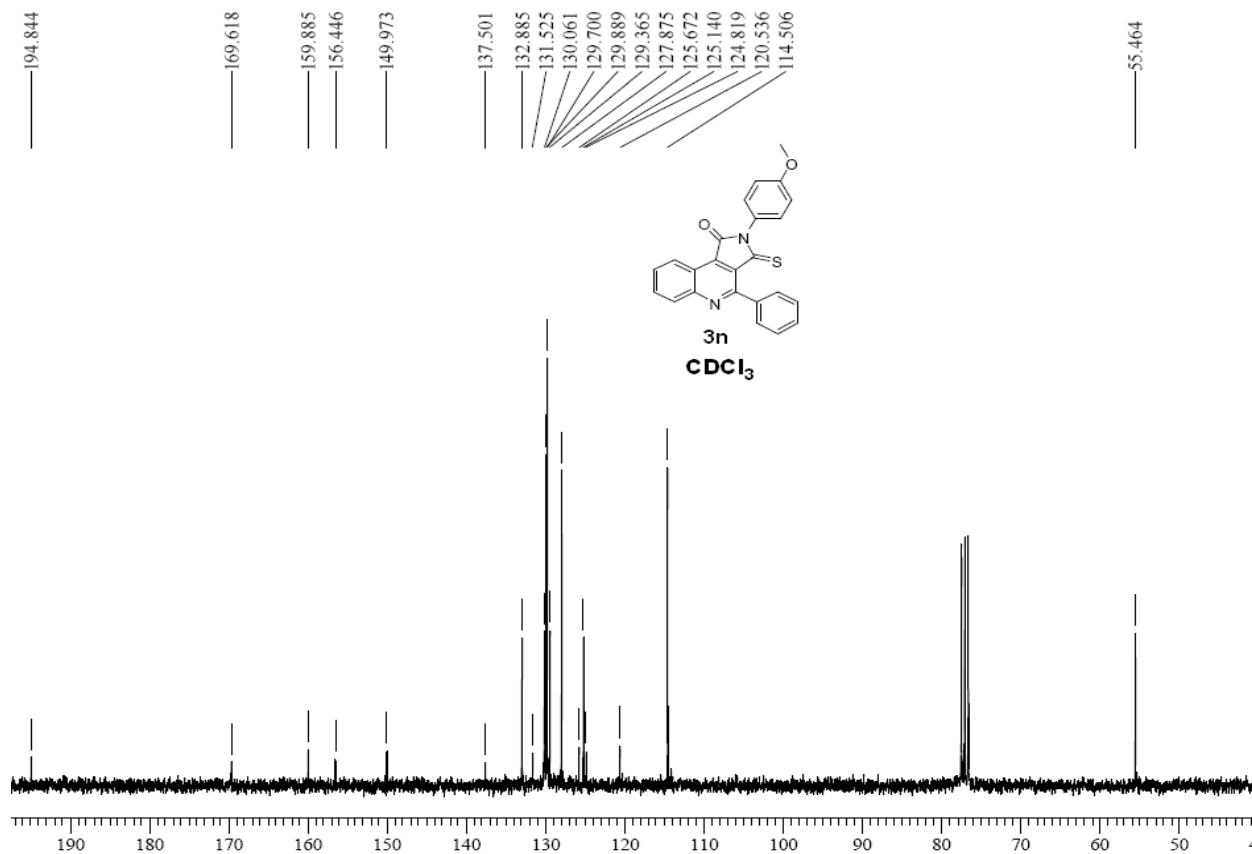
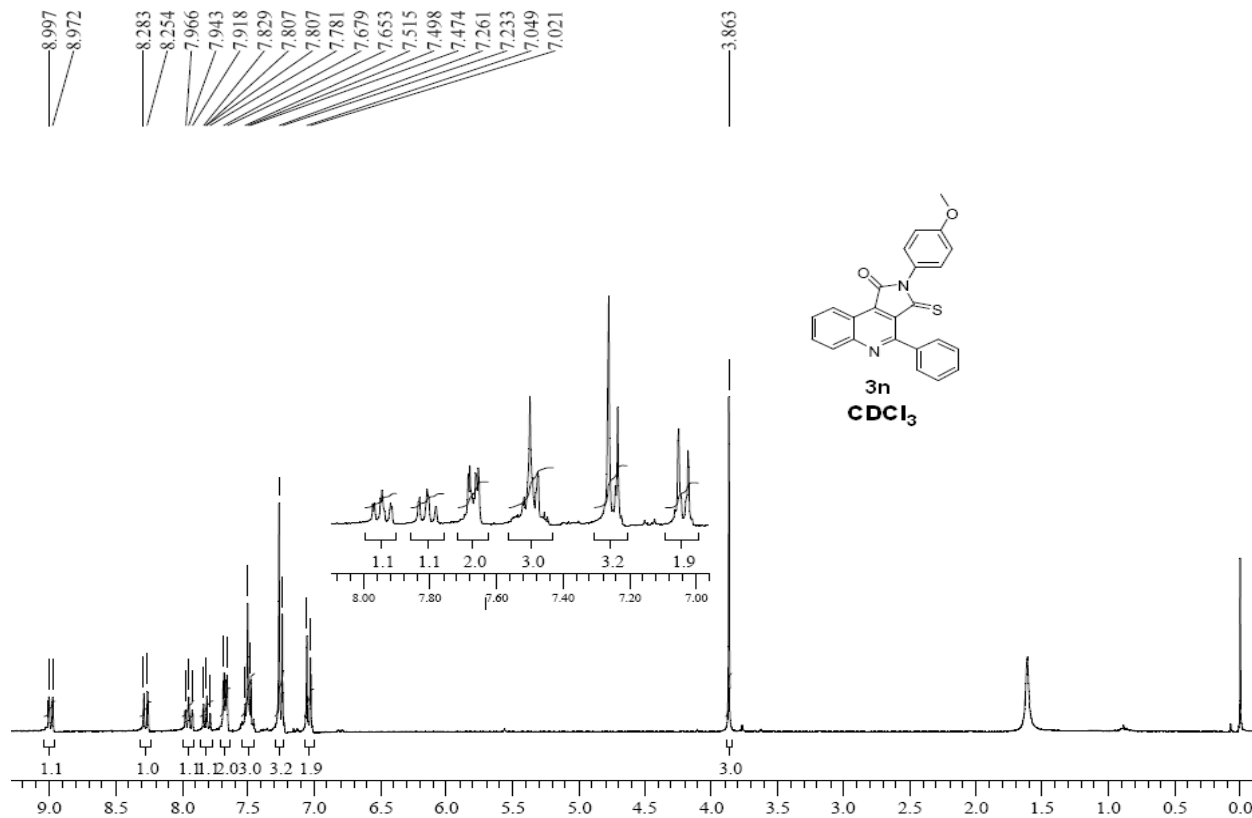


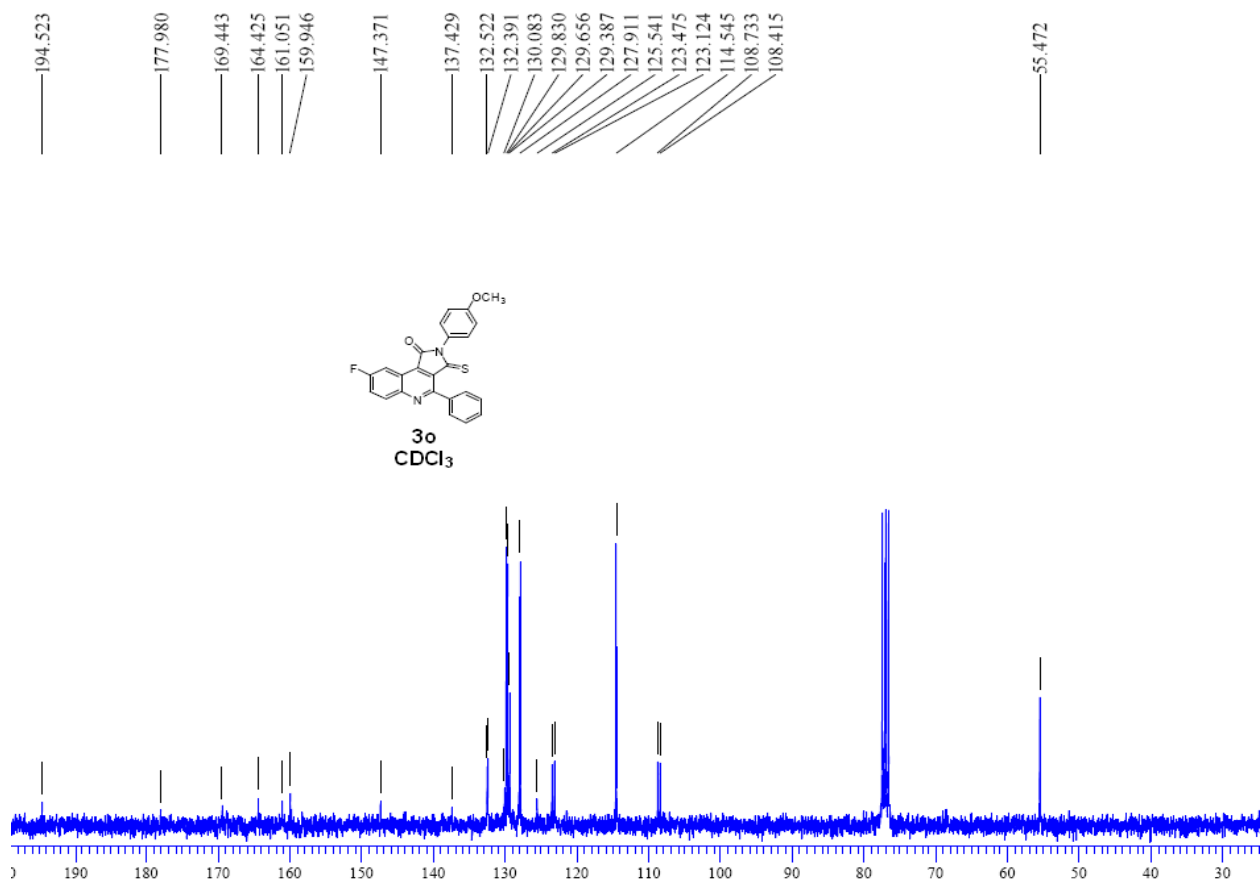
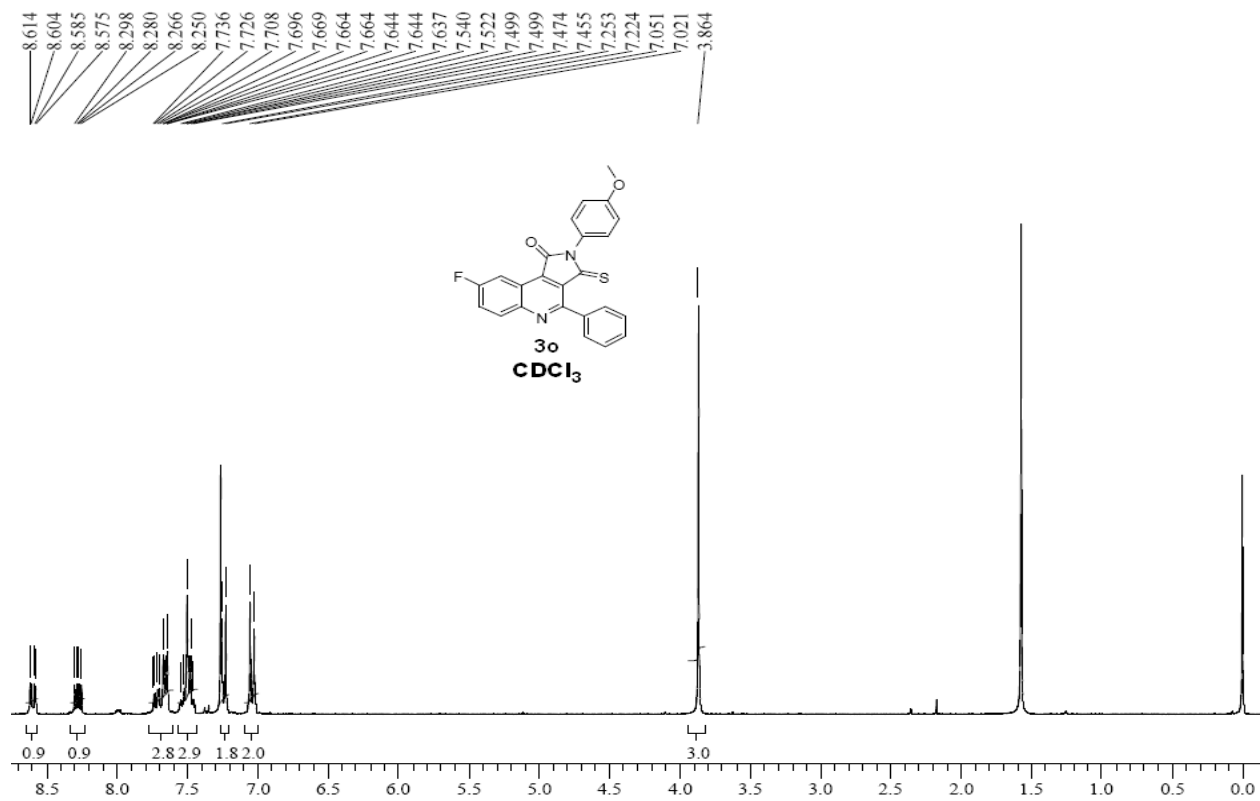


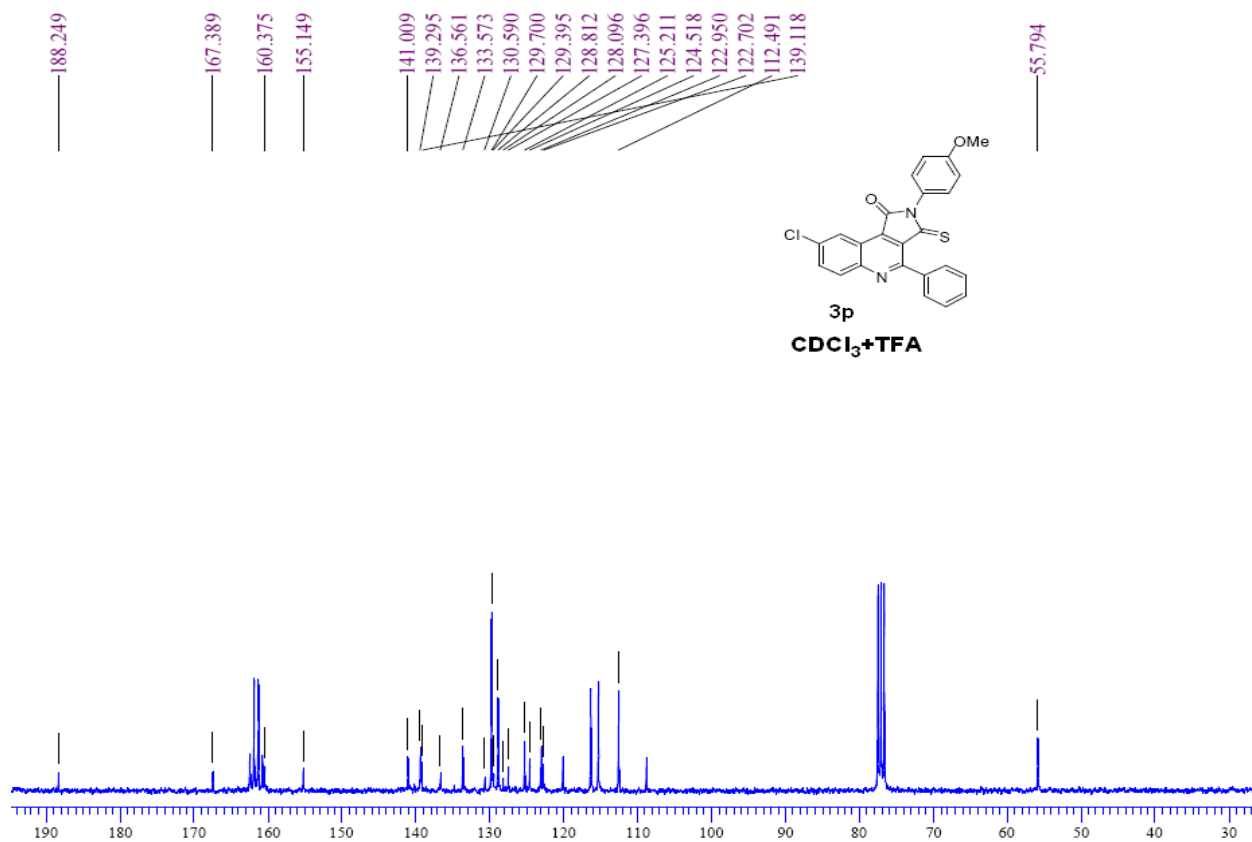
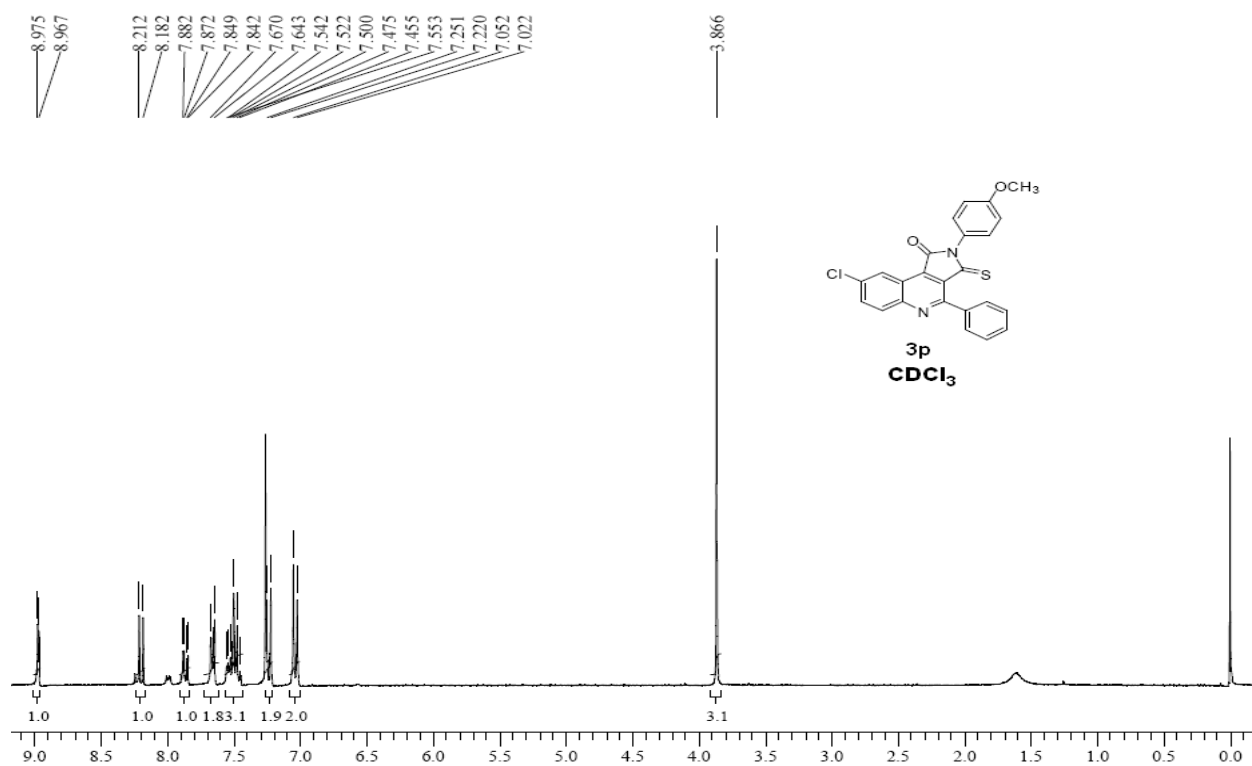


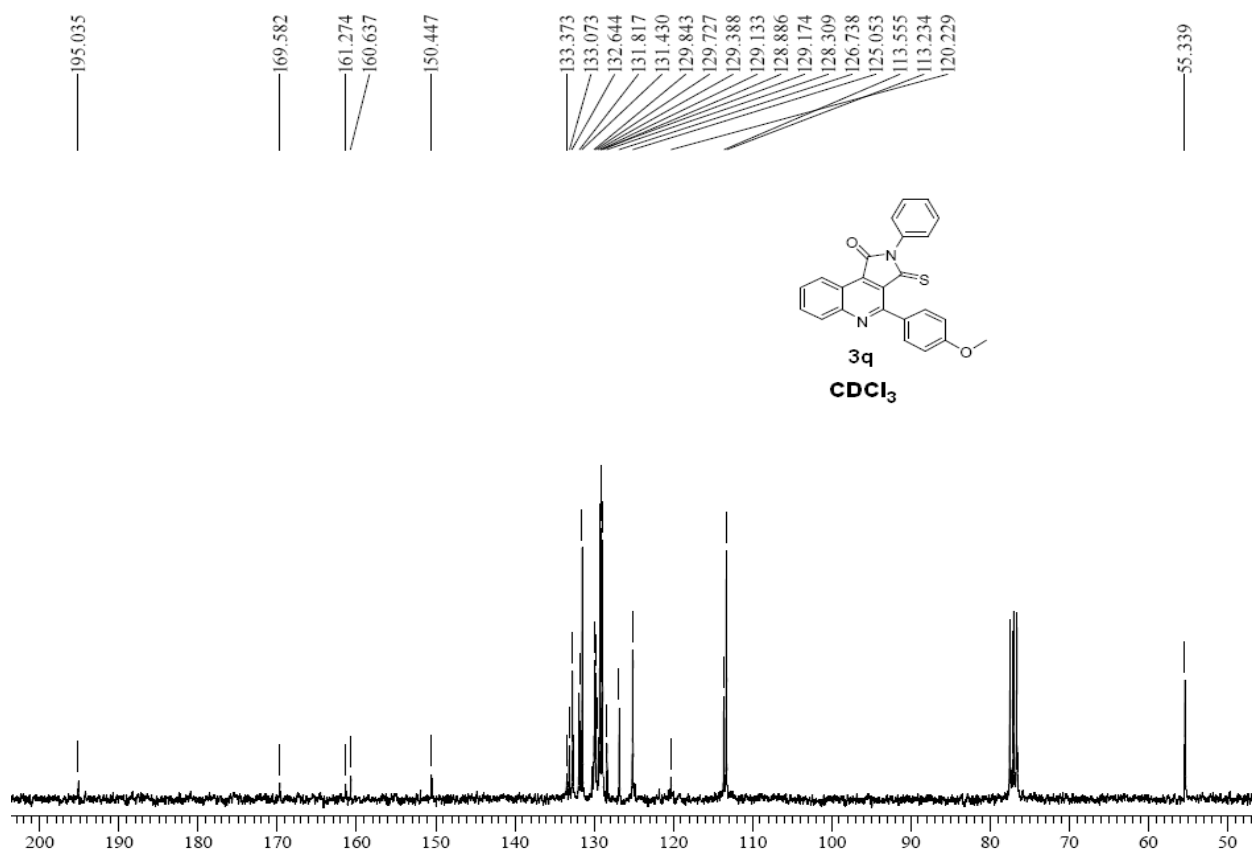
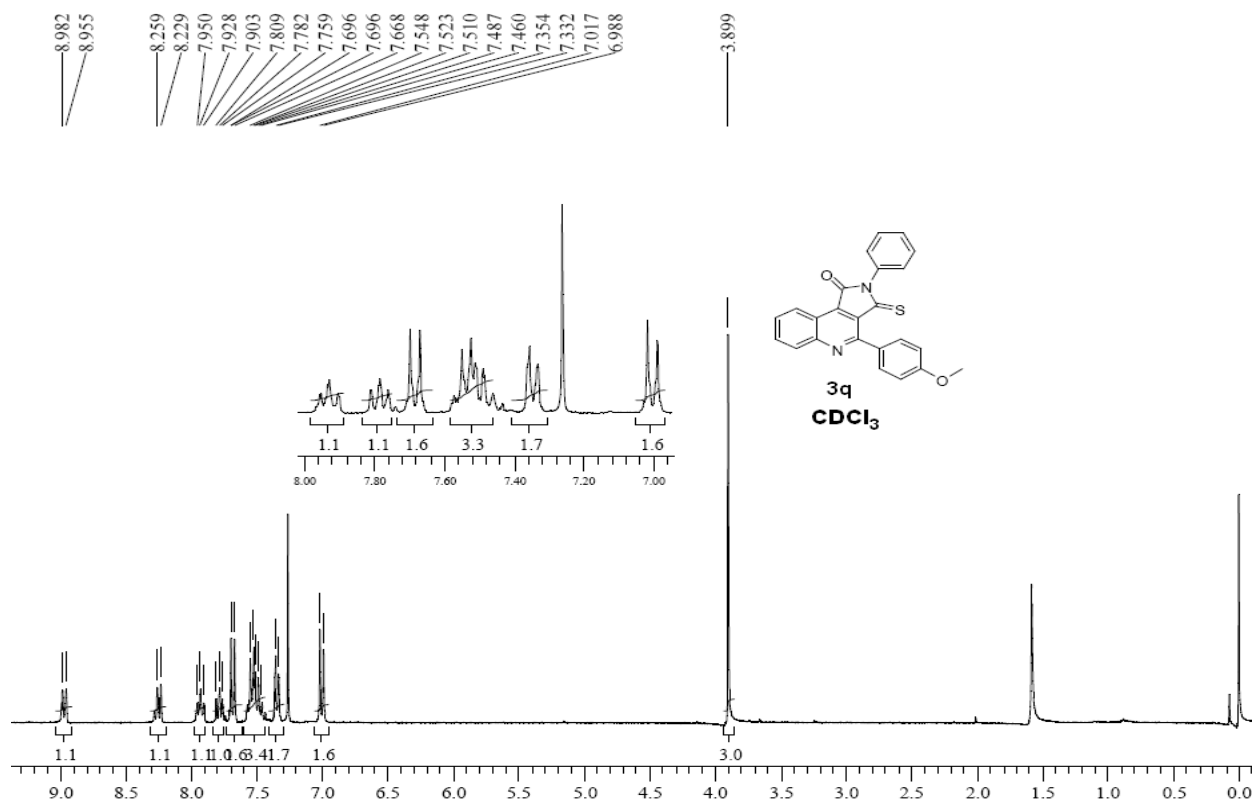


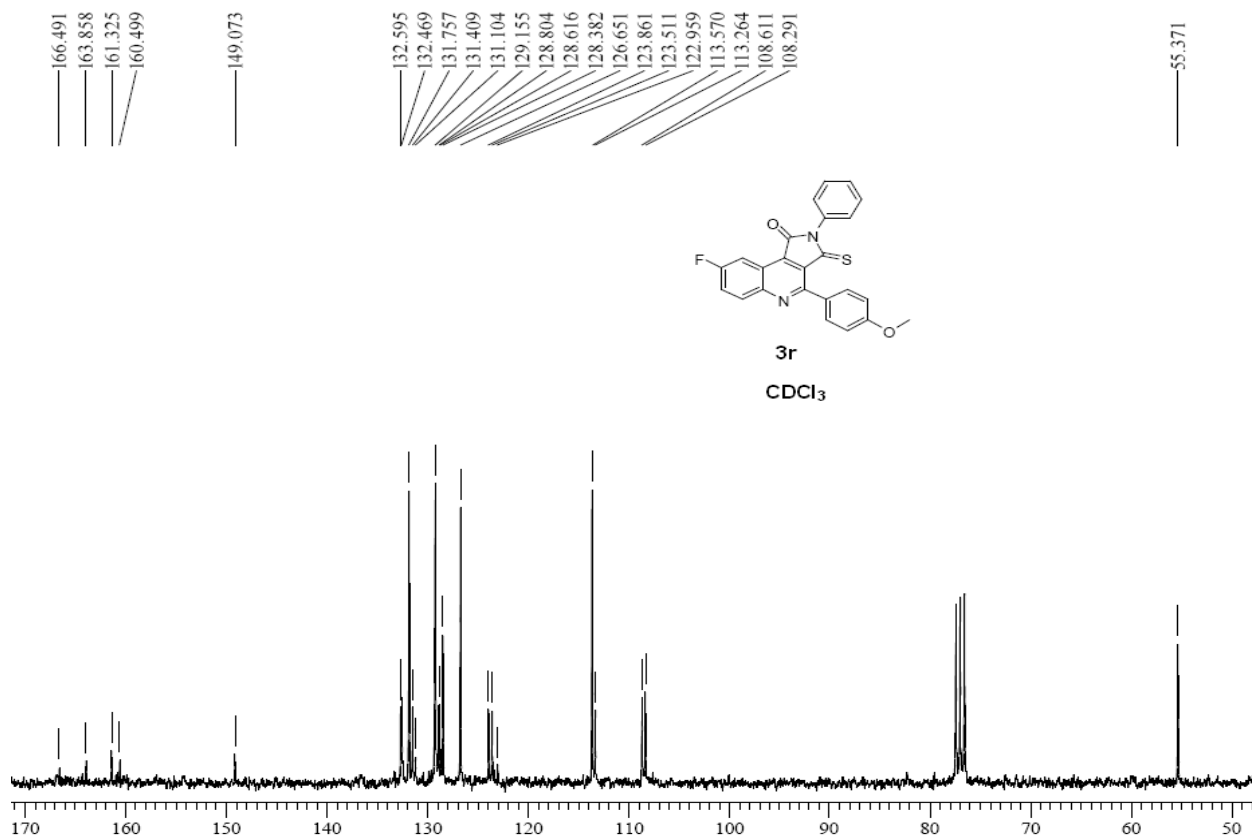
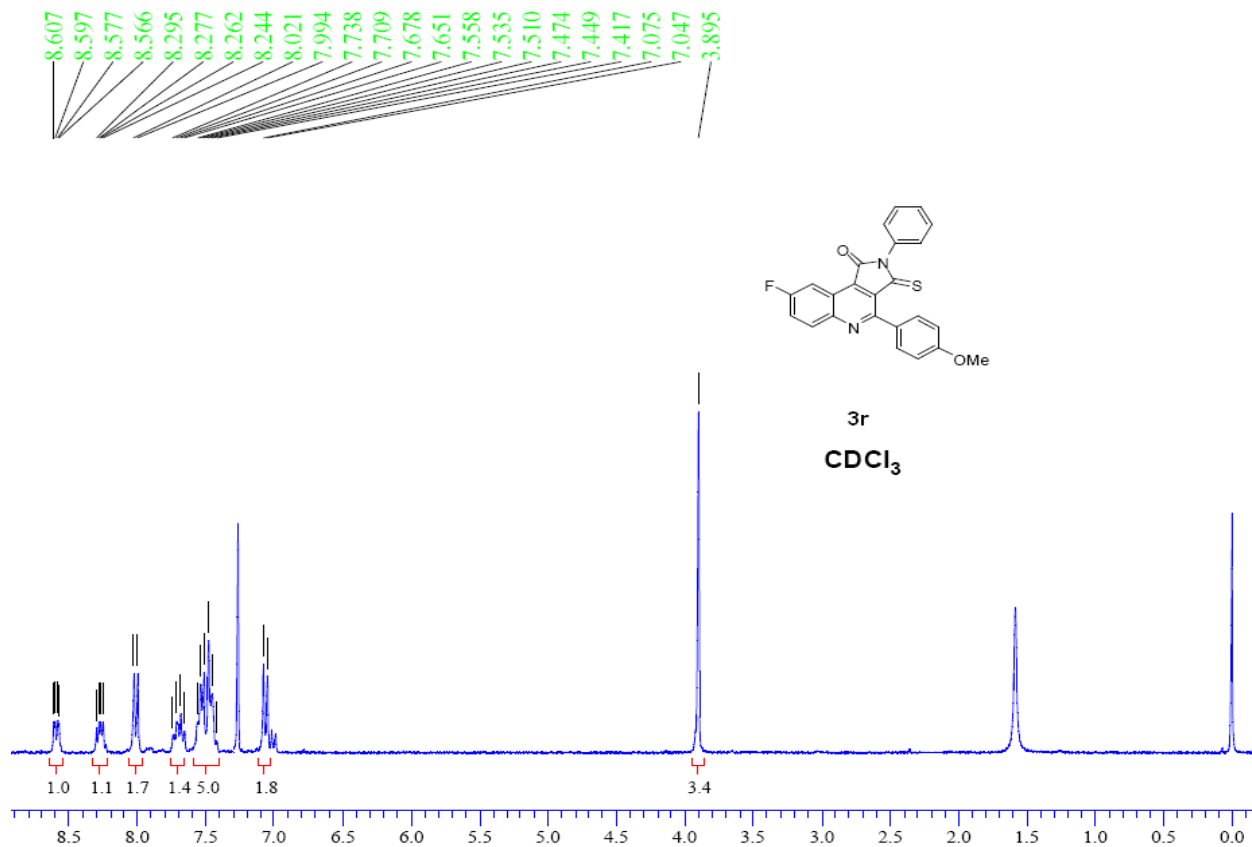


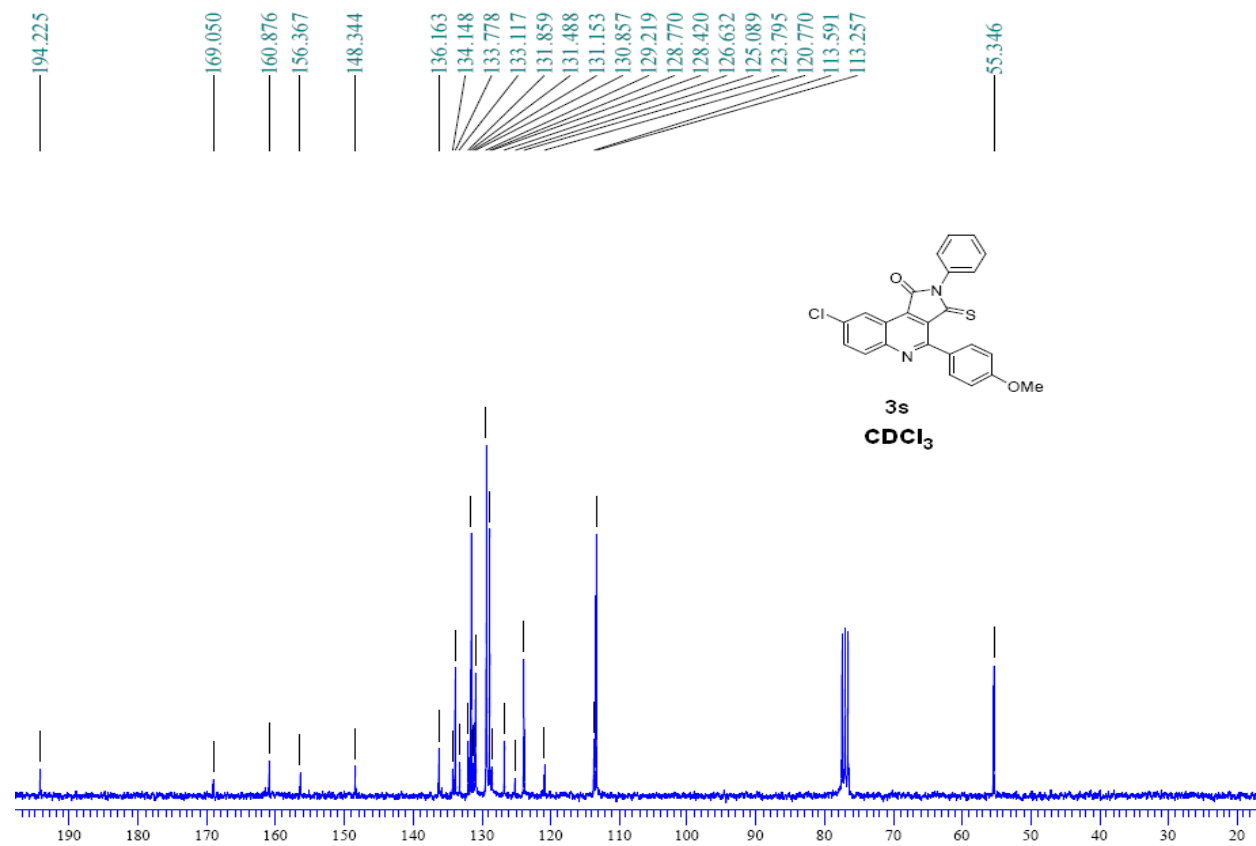
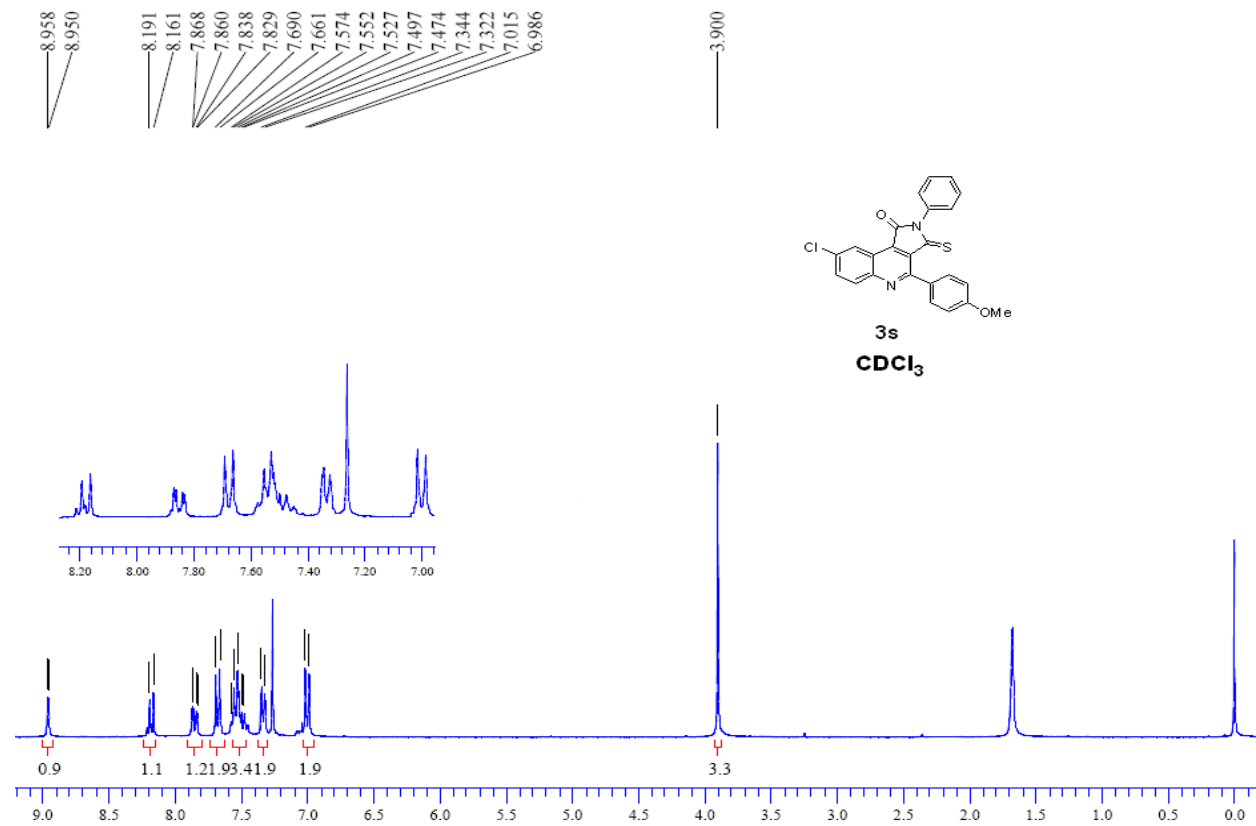


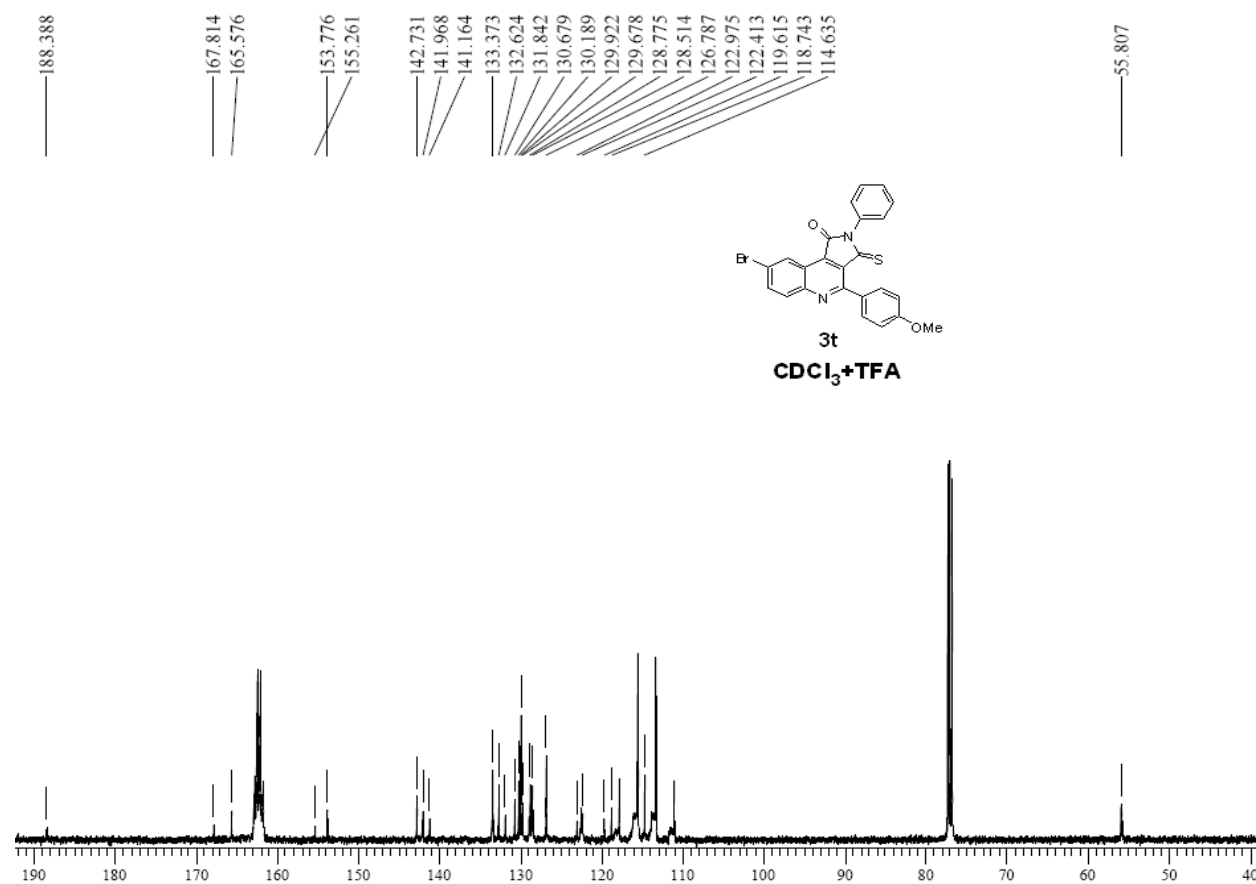
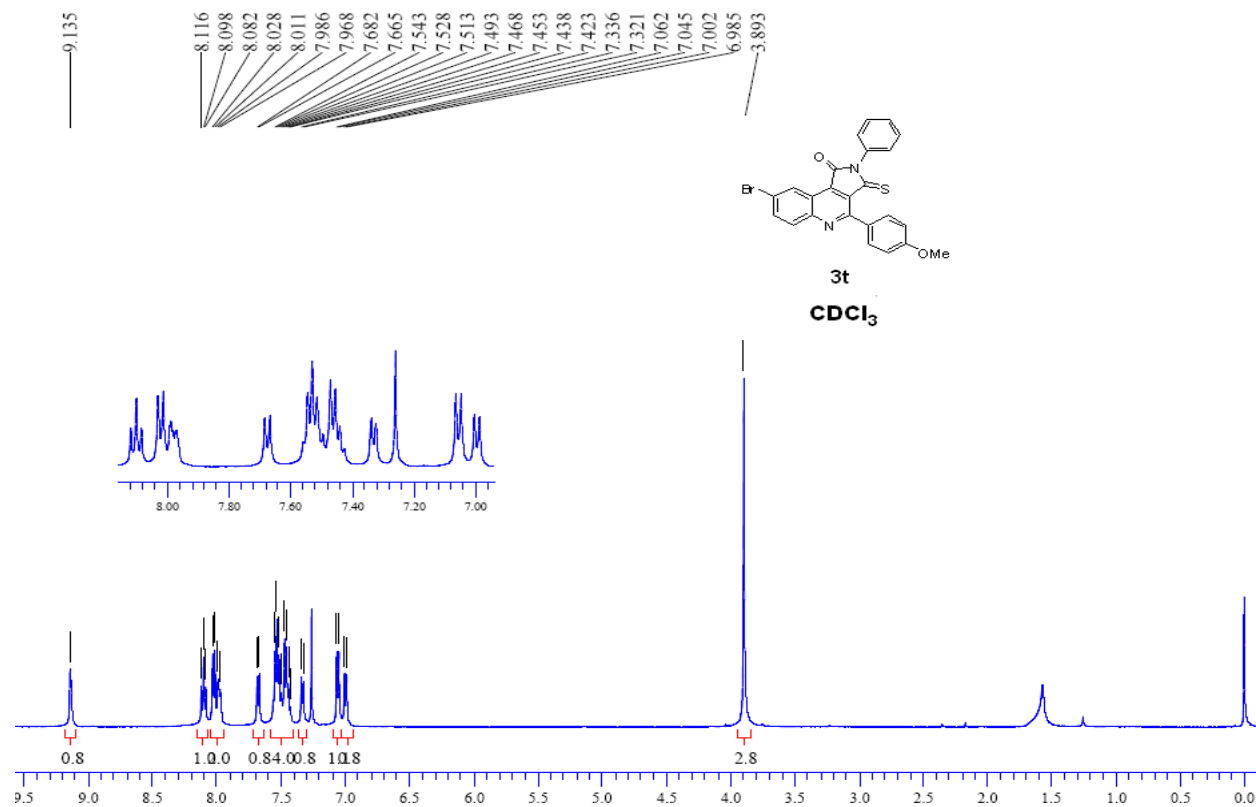


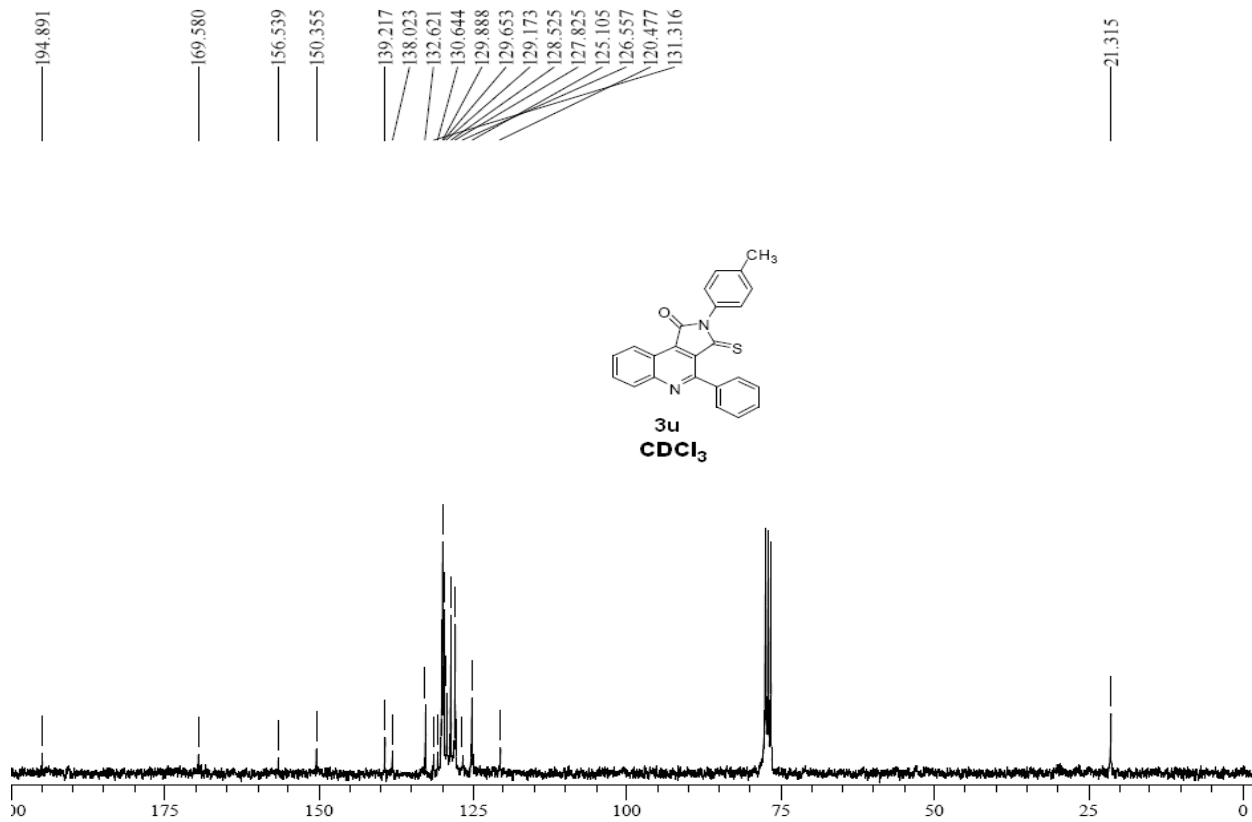
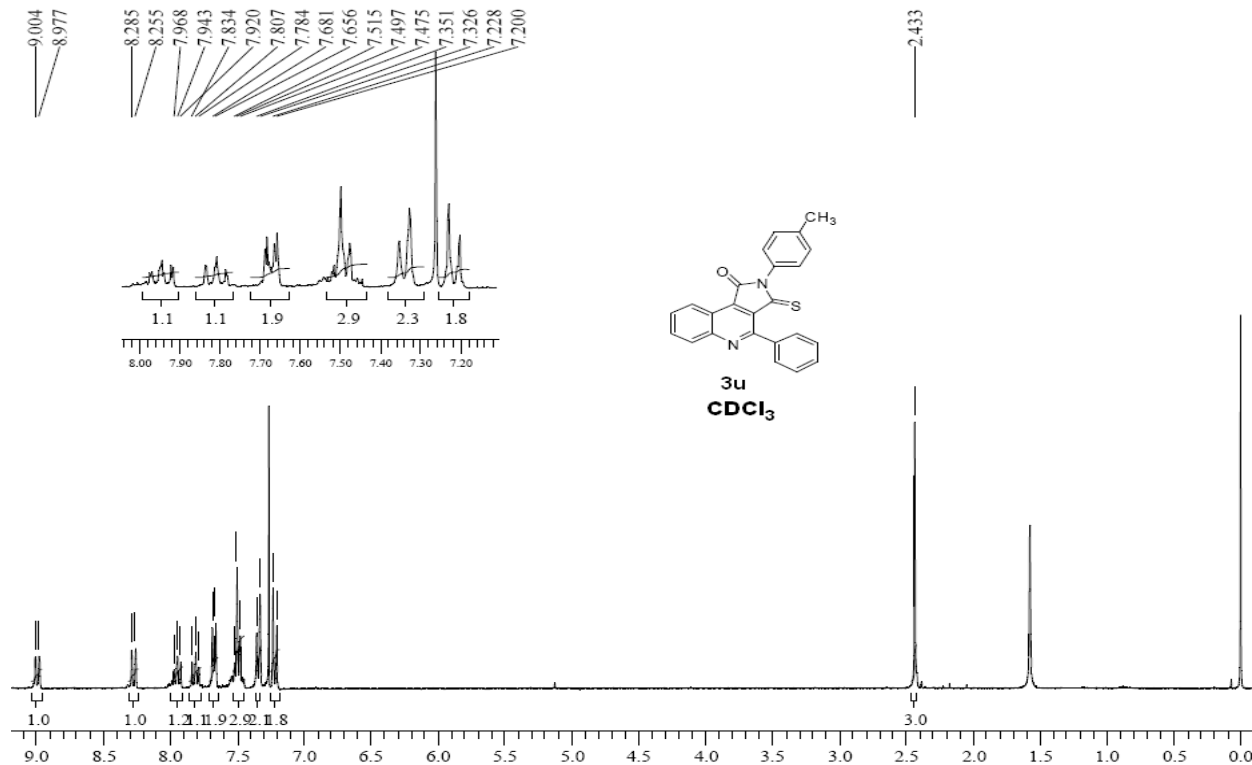


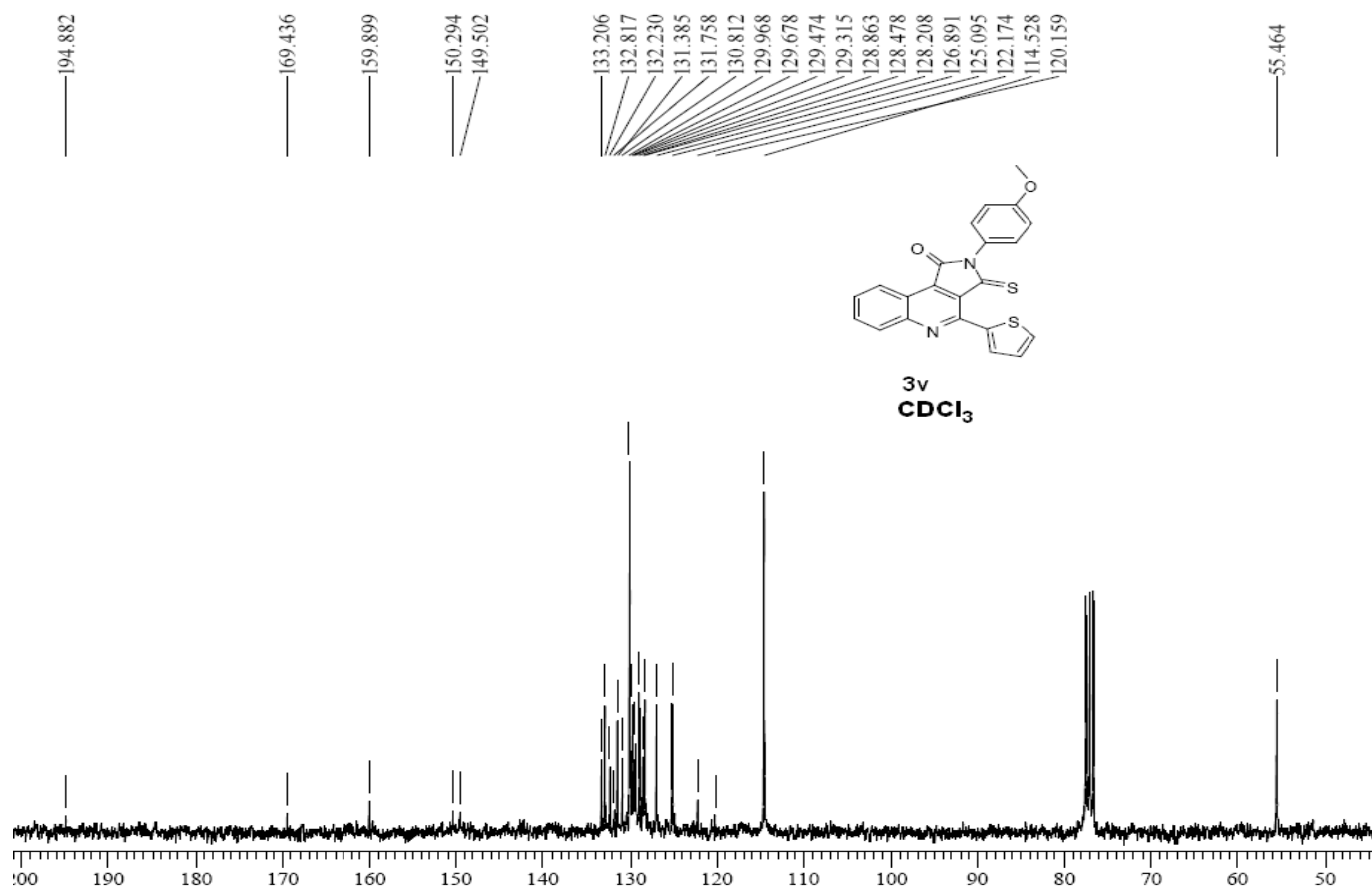
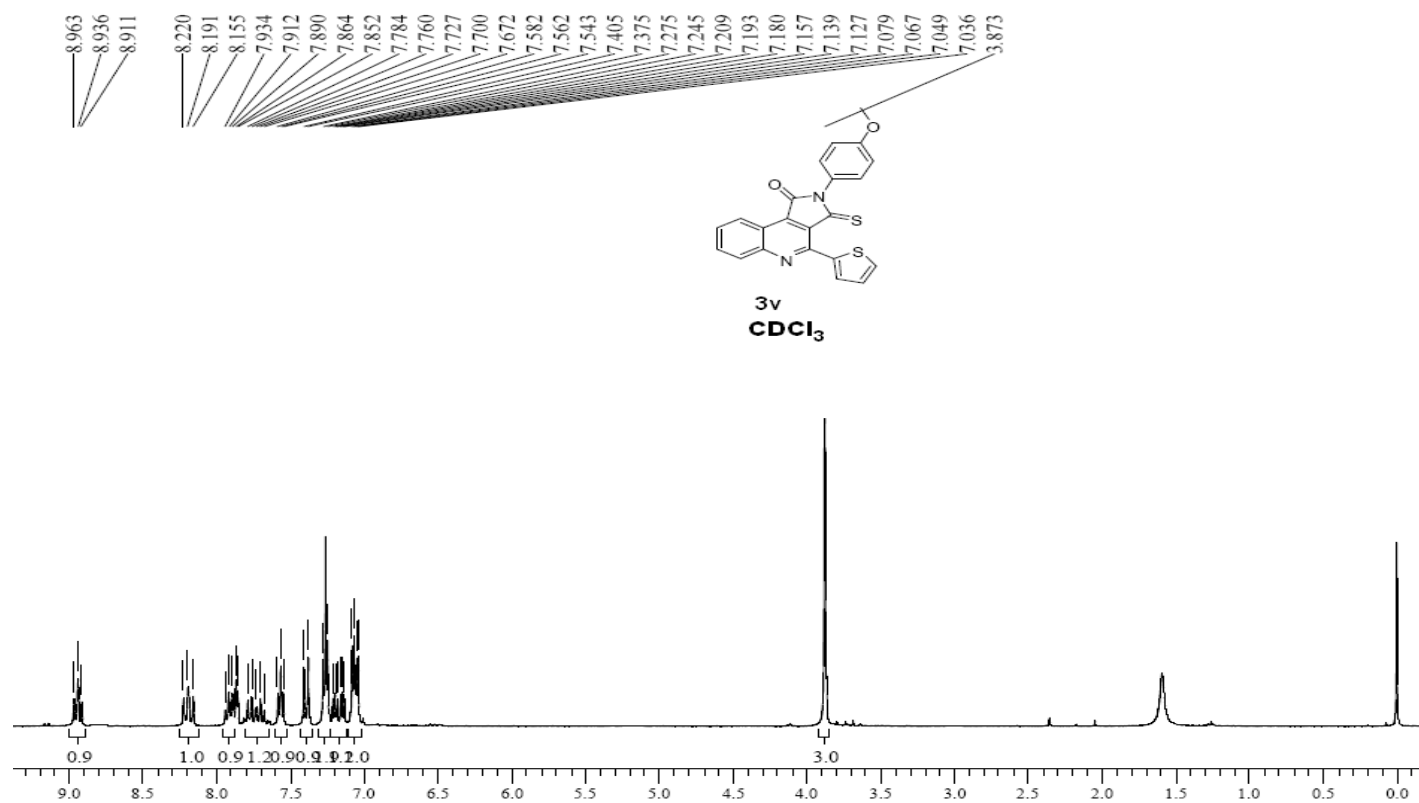


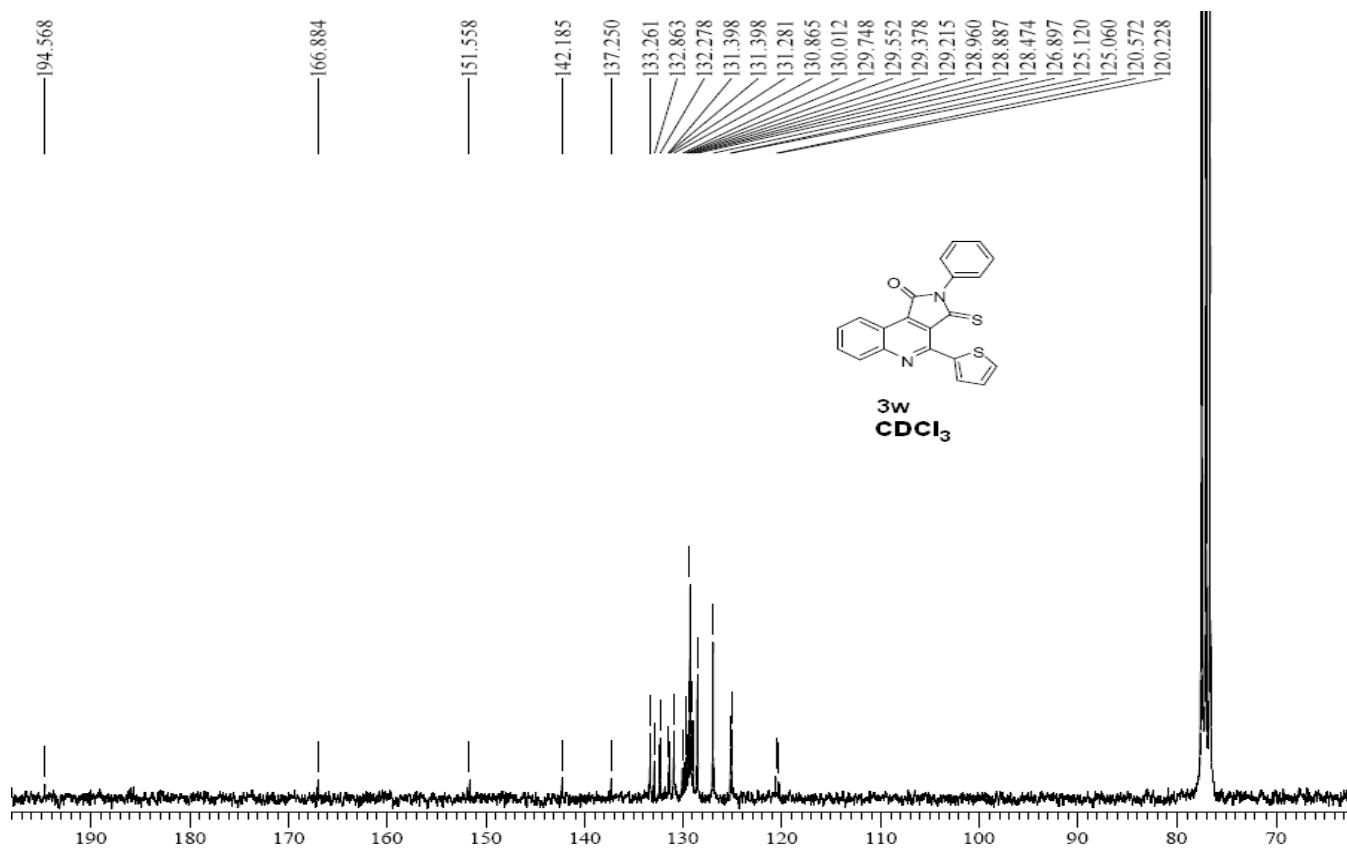
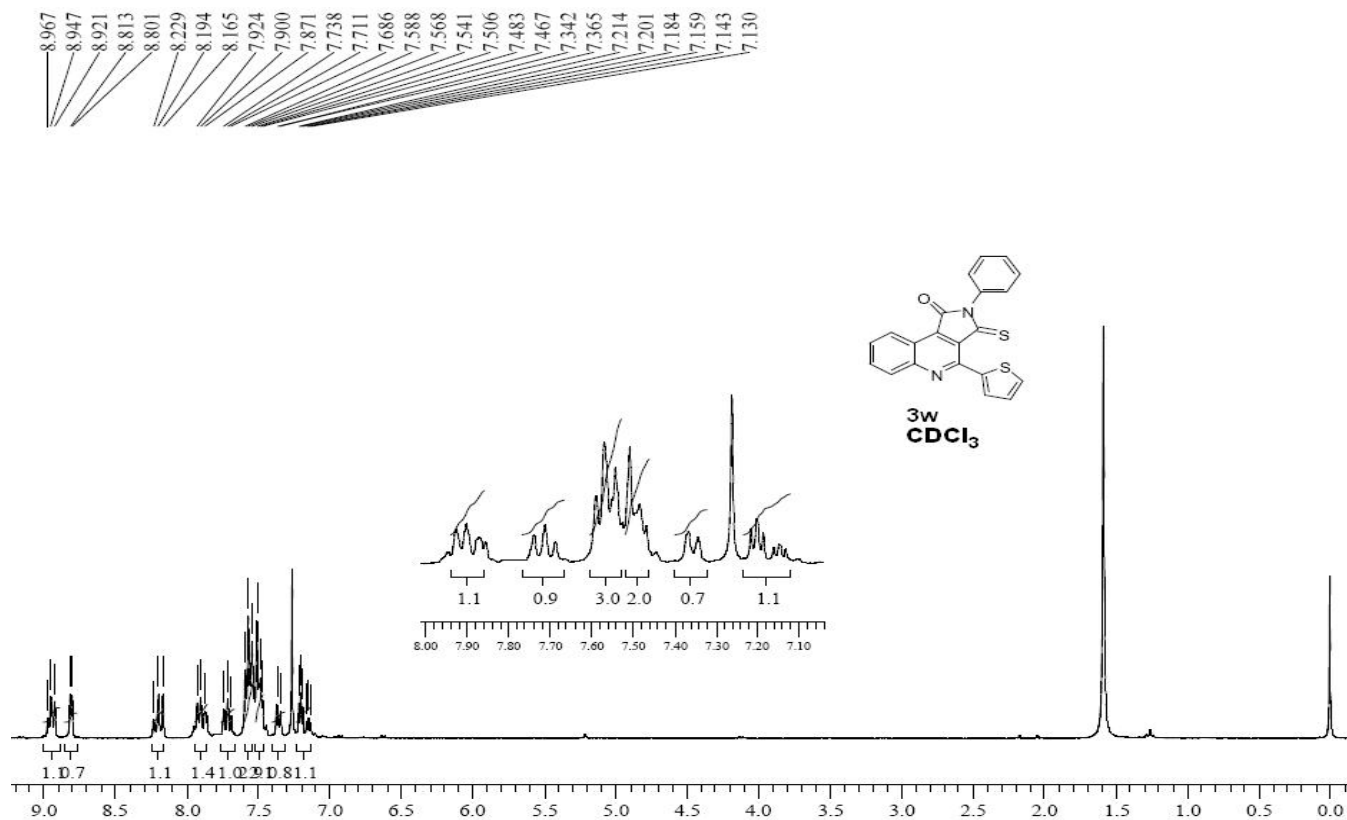


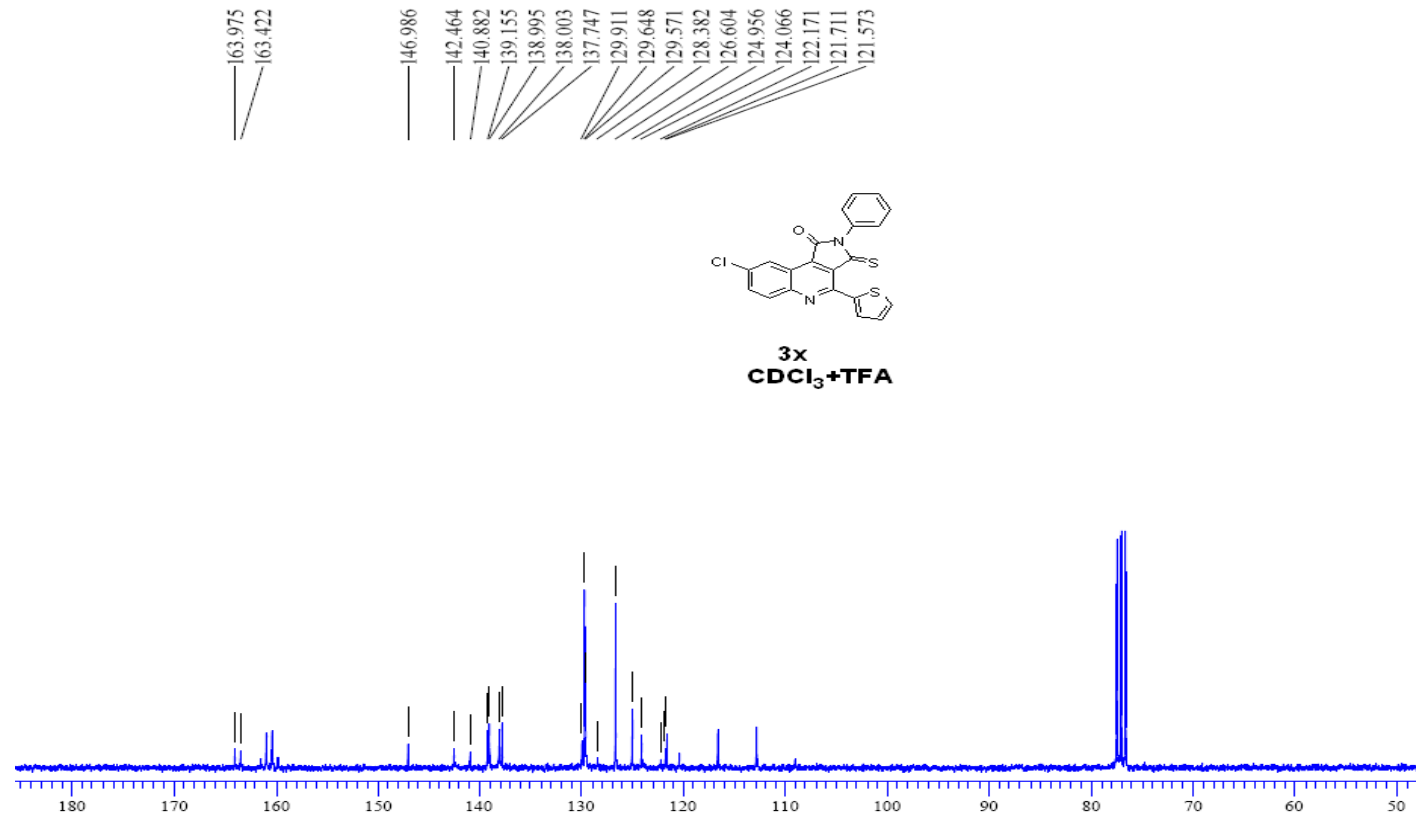
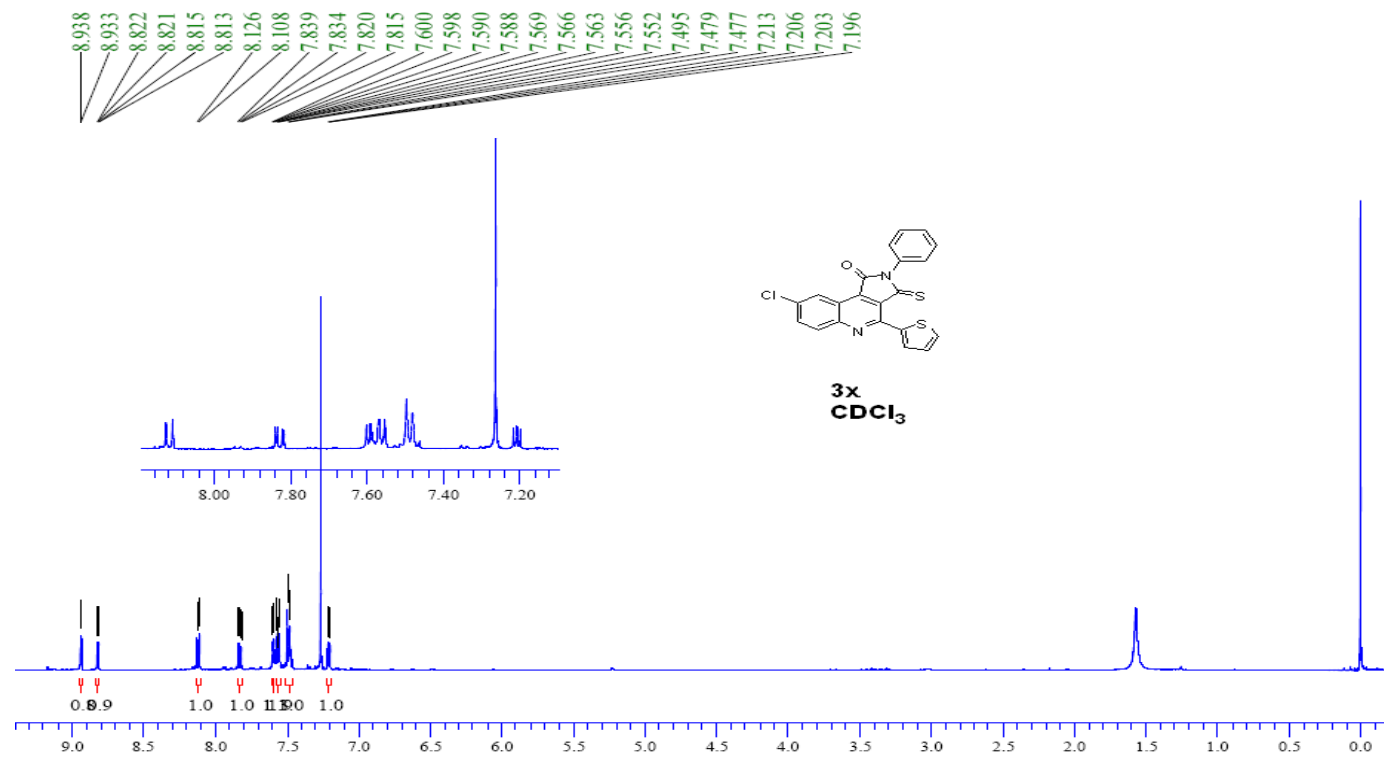


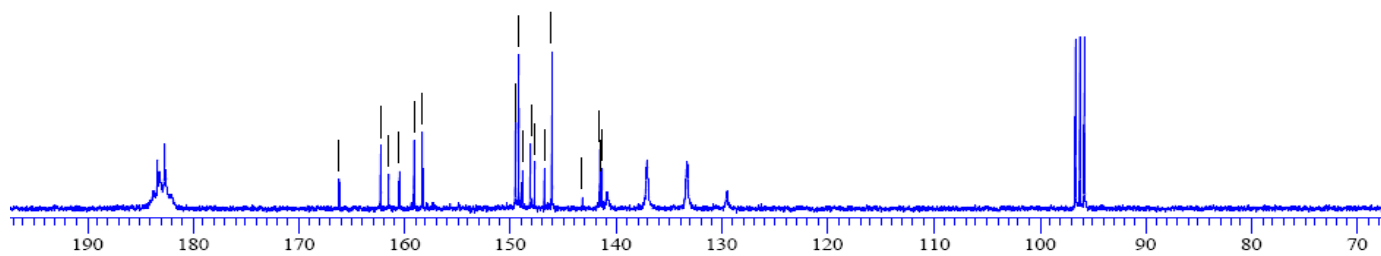
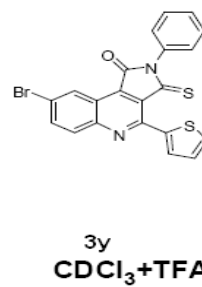
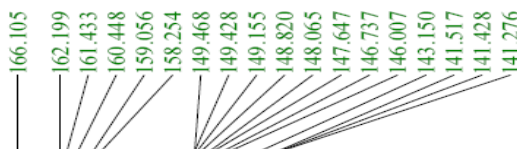
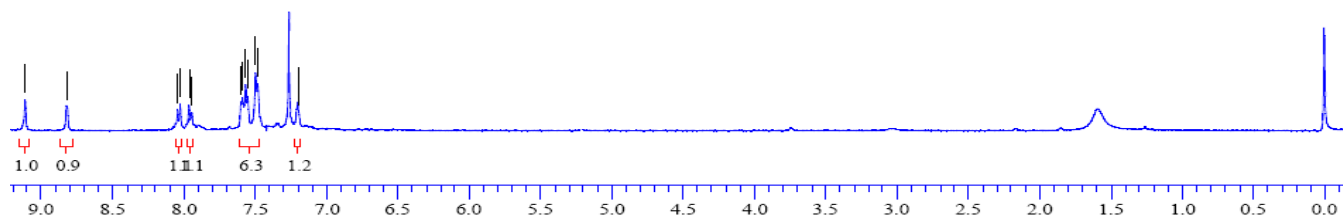
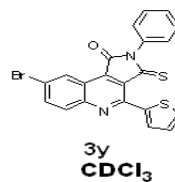


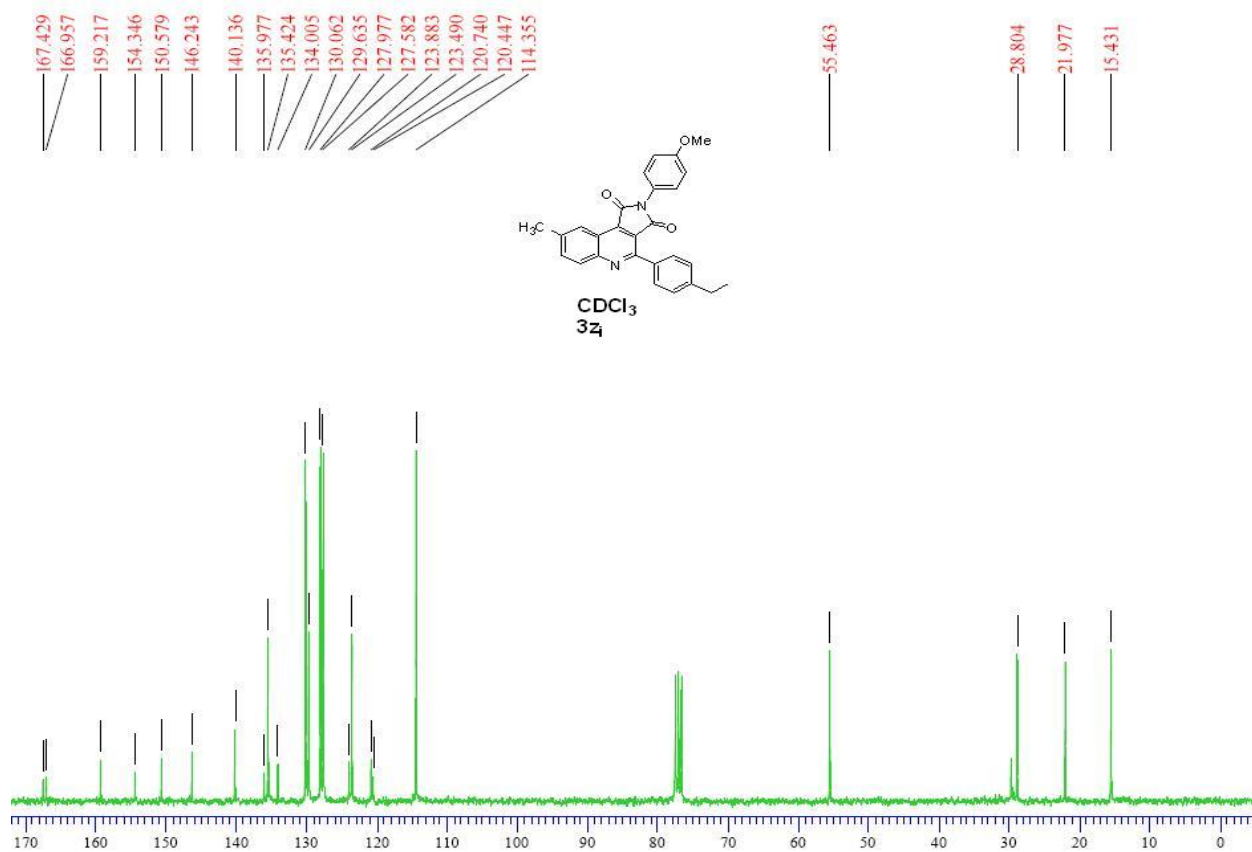
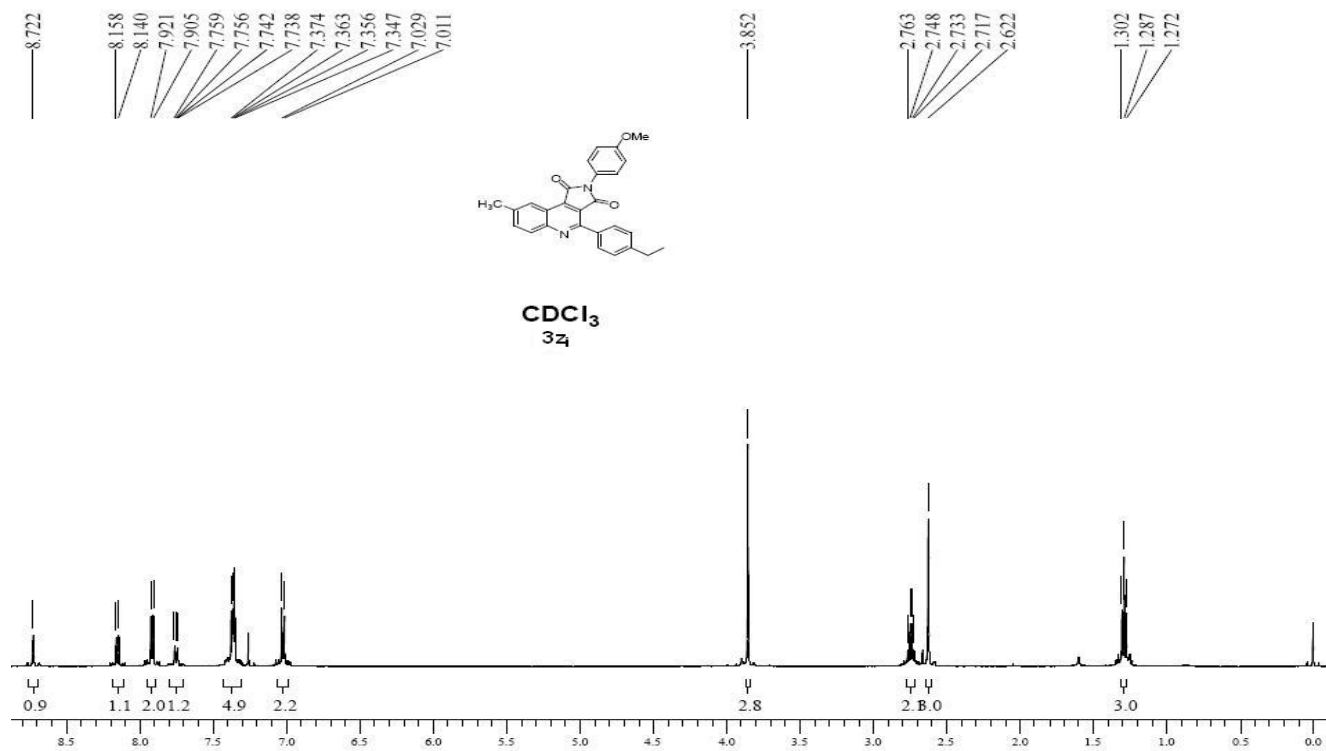


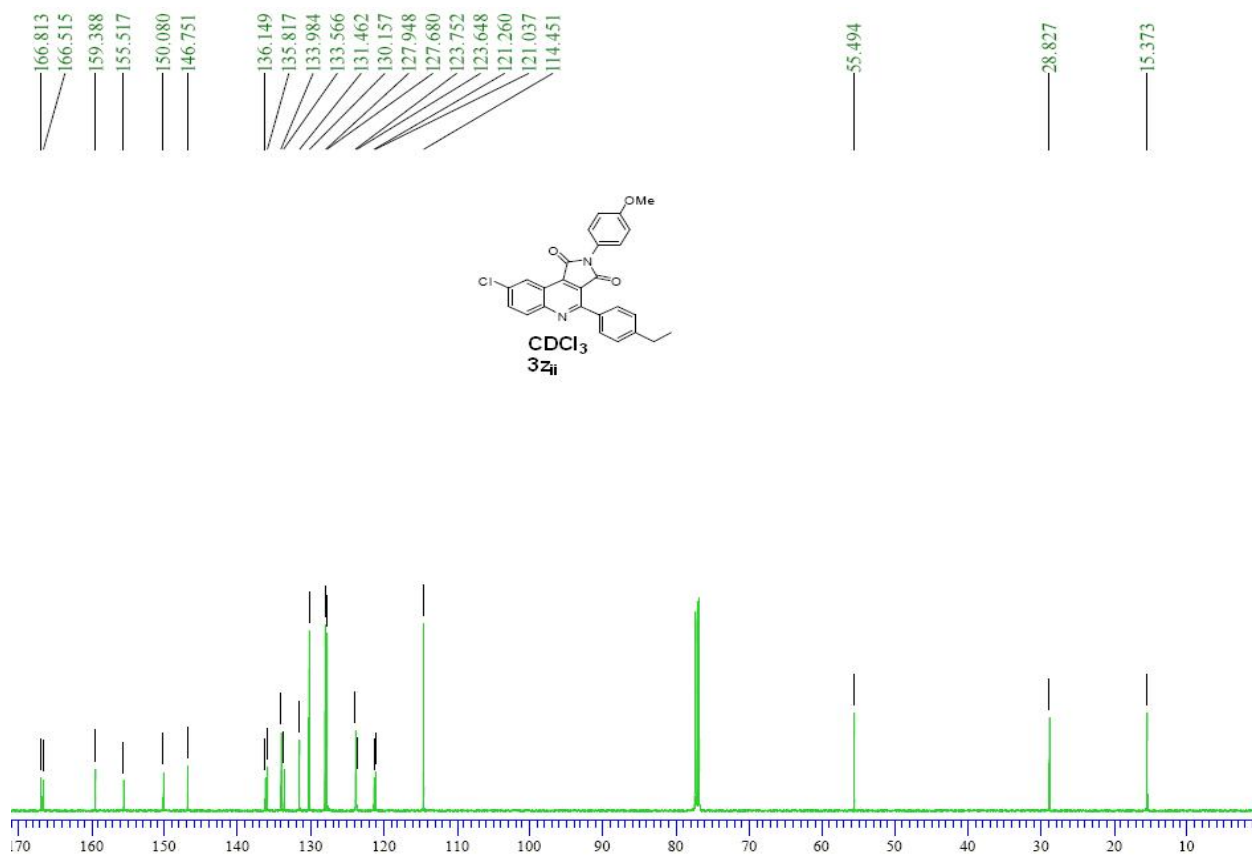
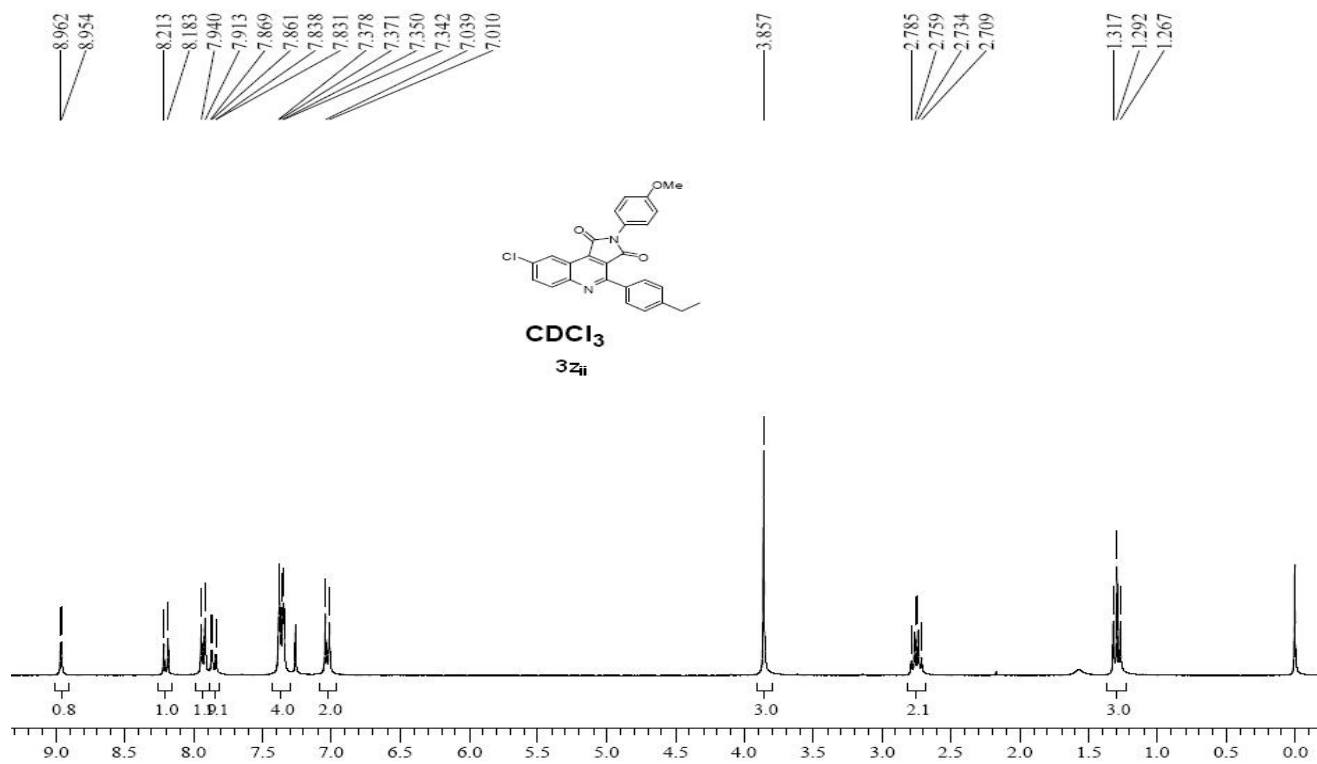


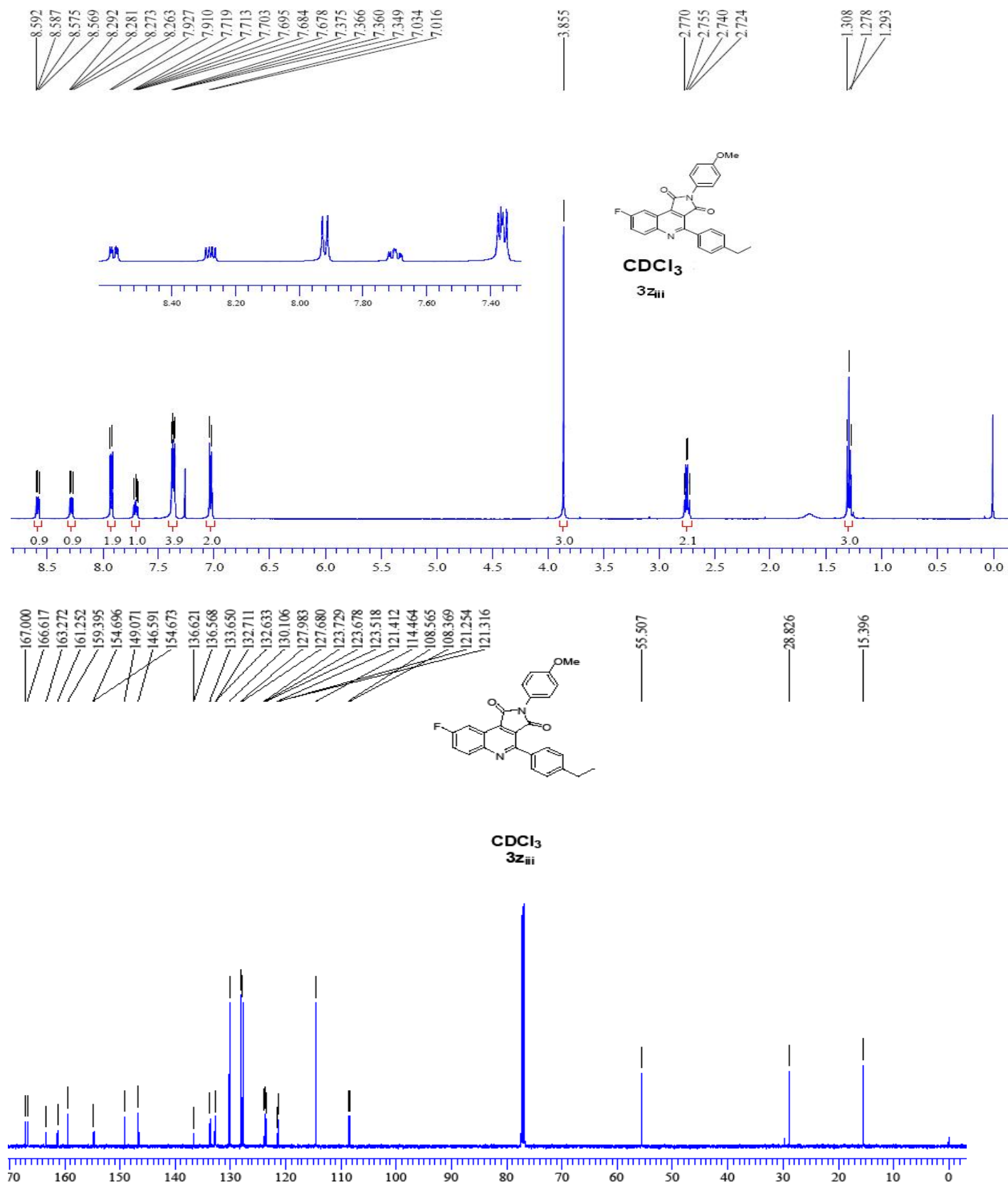


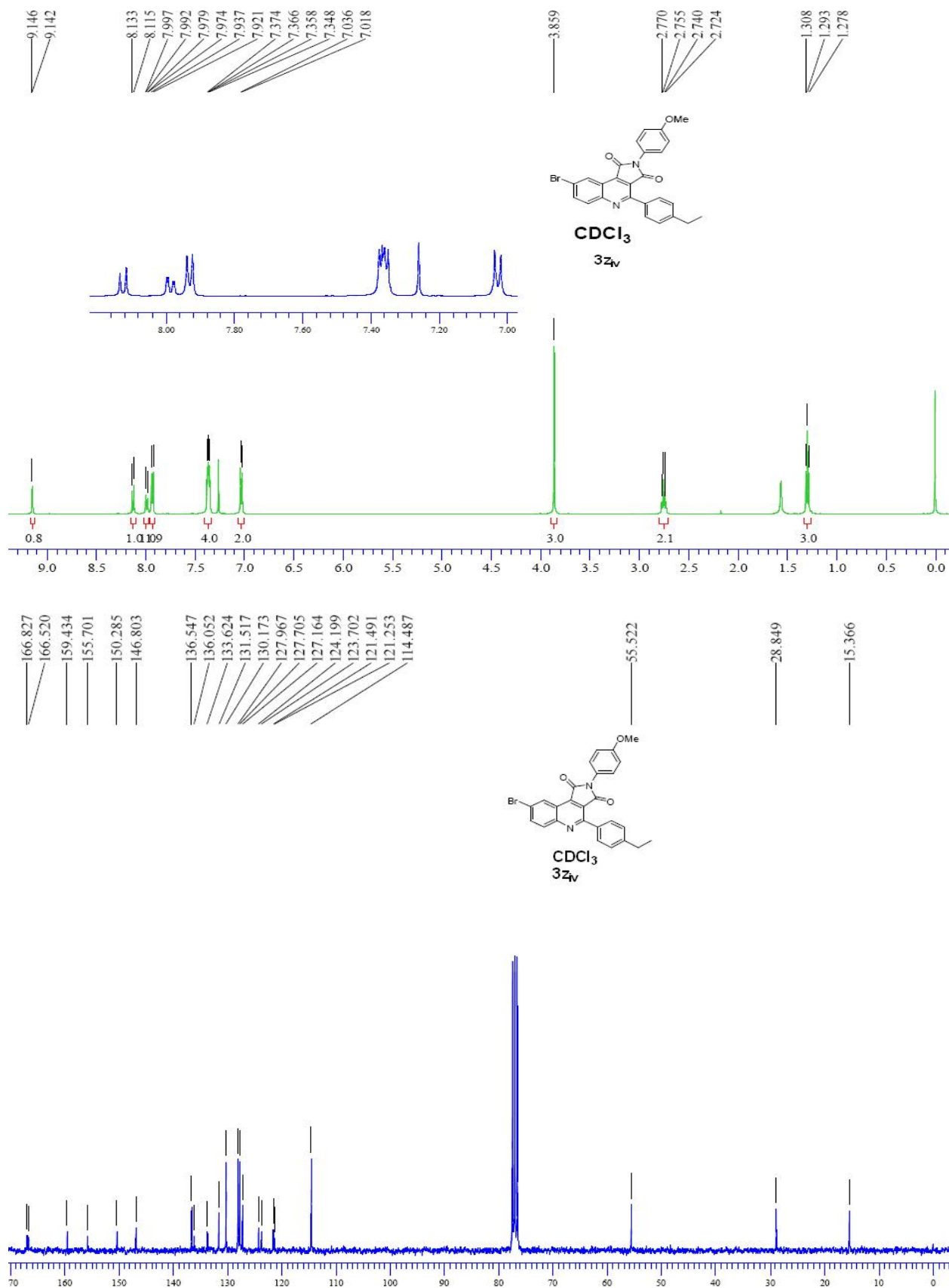


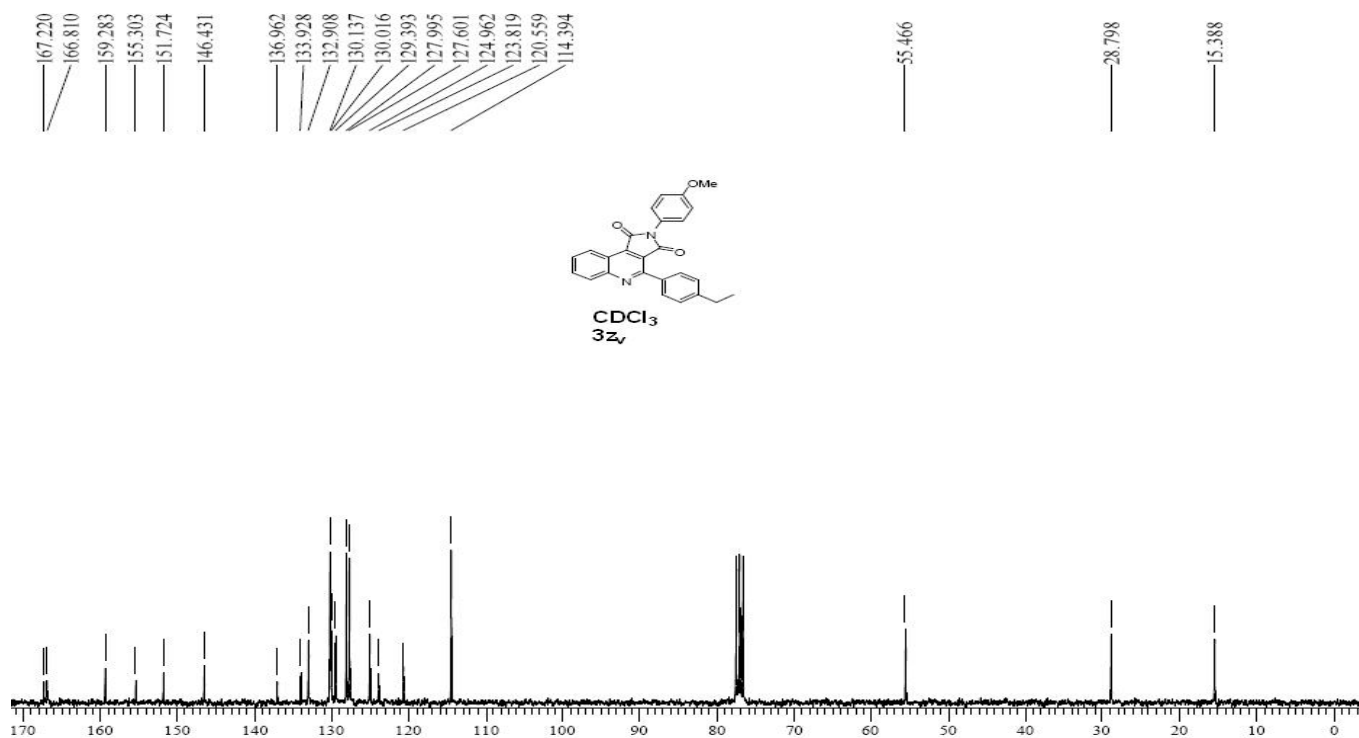
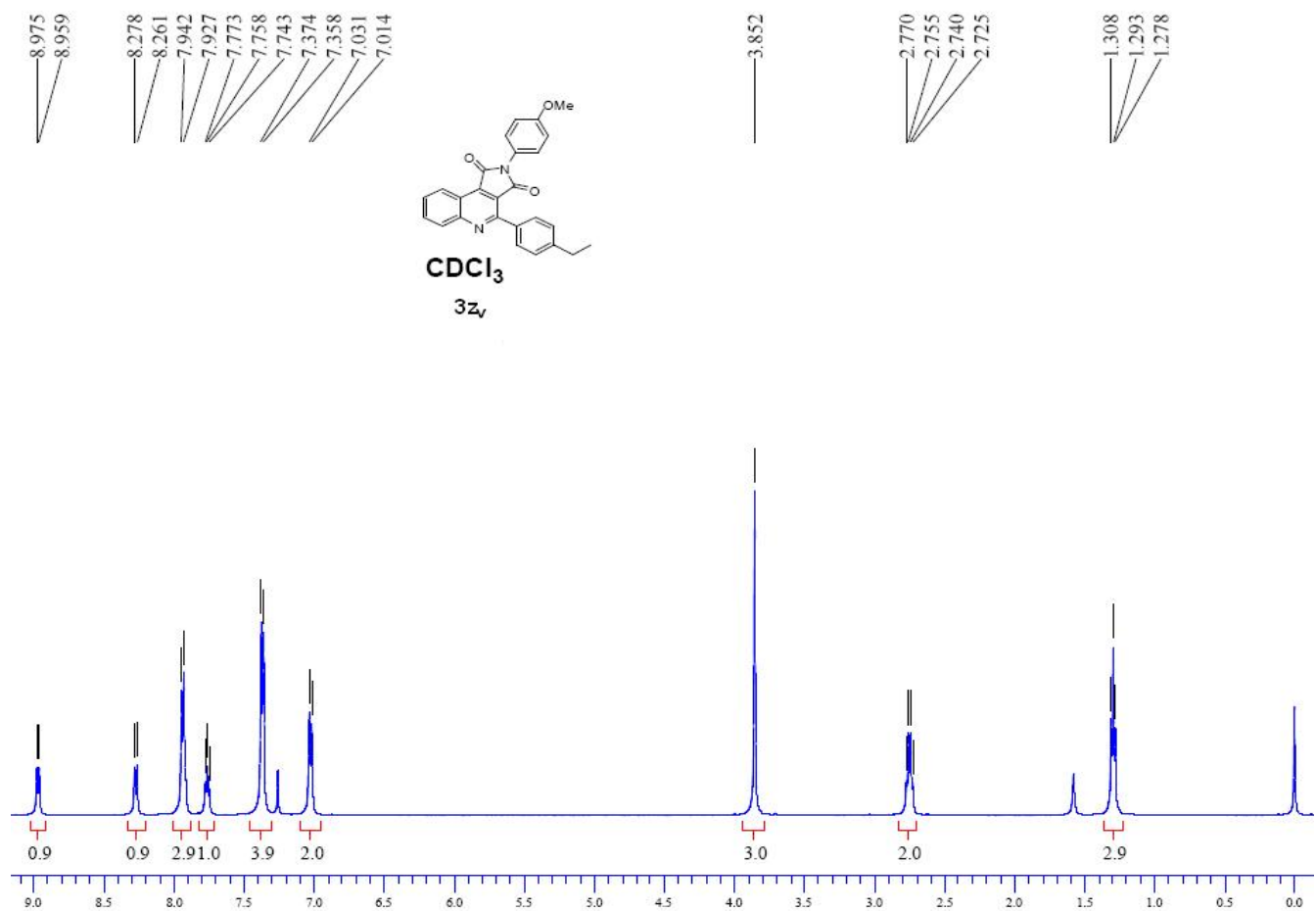


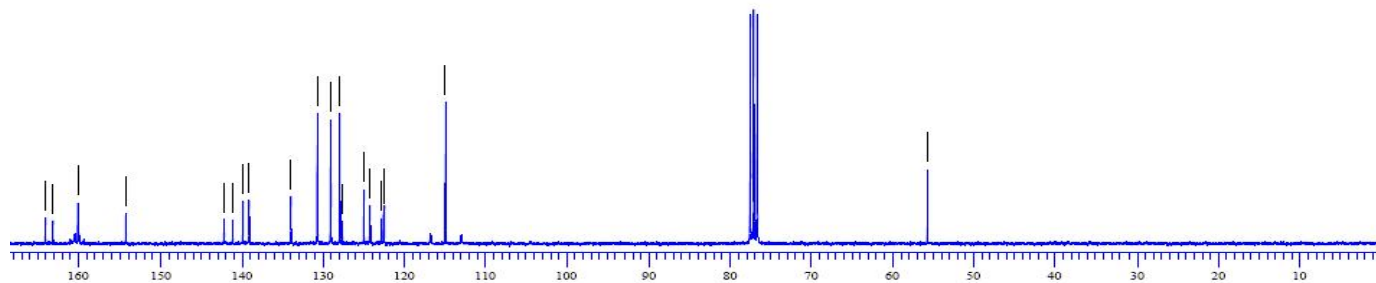
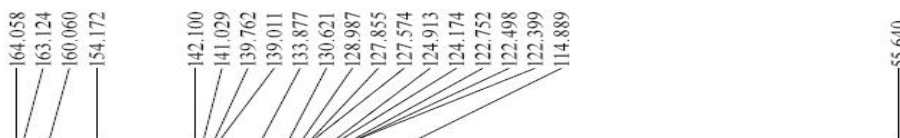
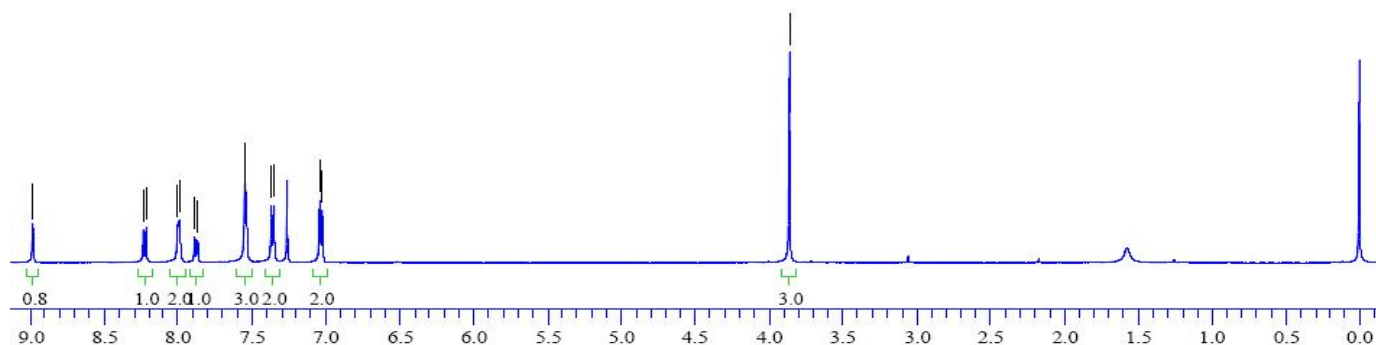
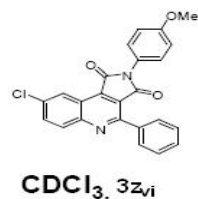
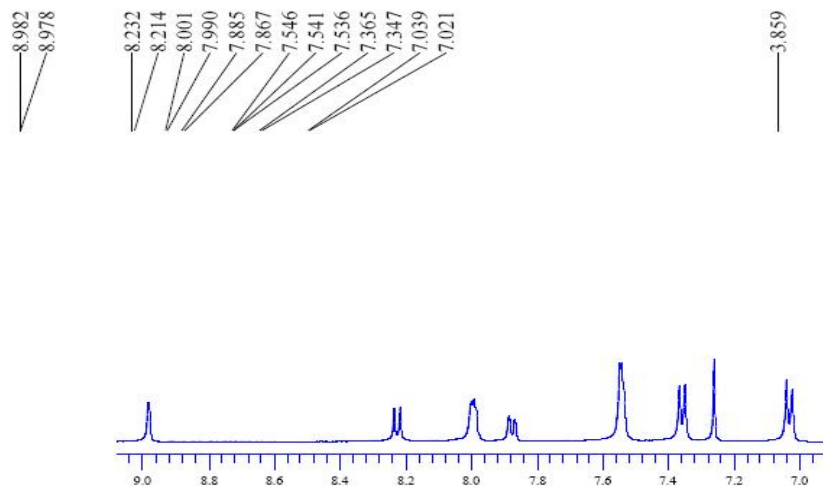










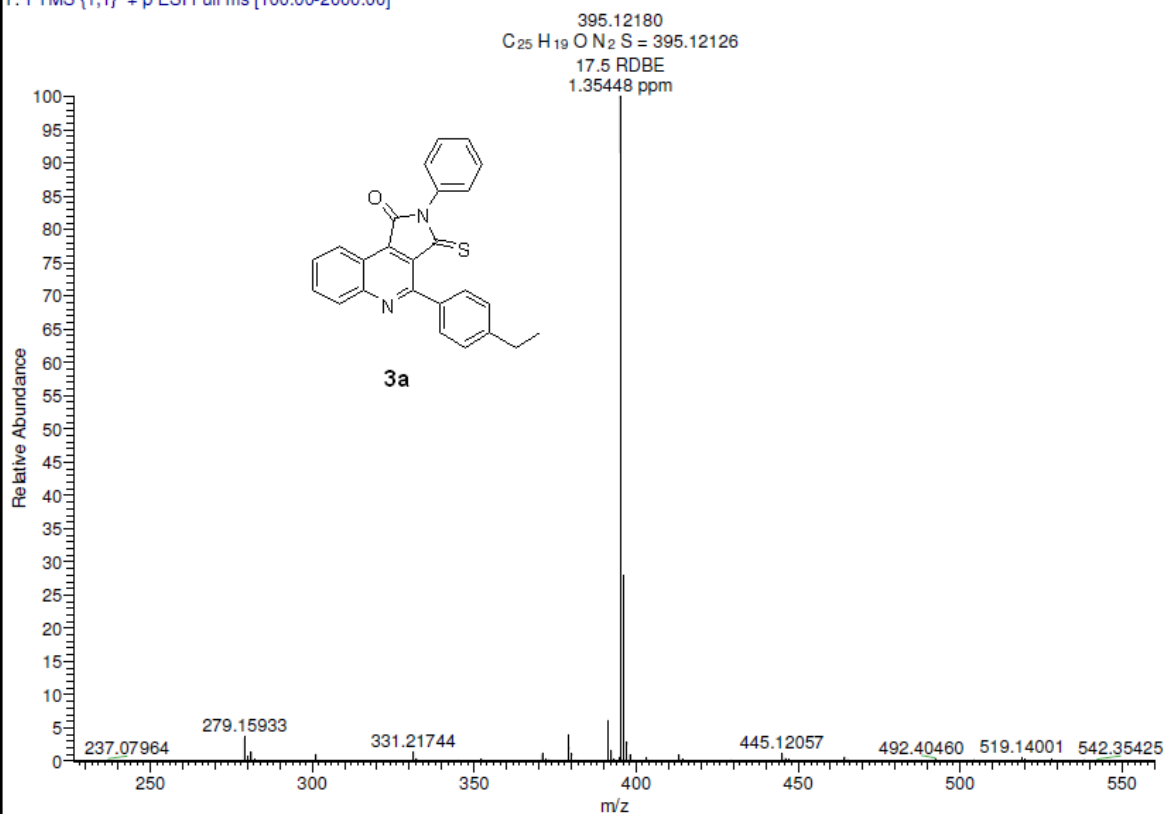


HRMS-DATA (3a-z_{vi}):

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NATIONAL CENTRE FOR MASS SPECTROMETRY

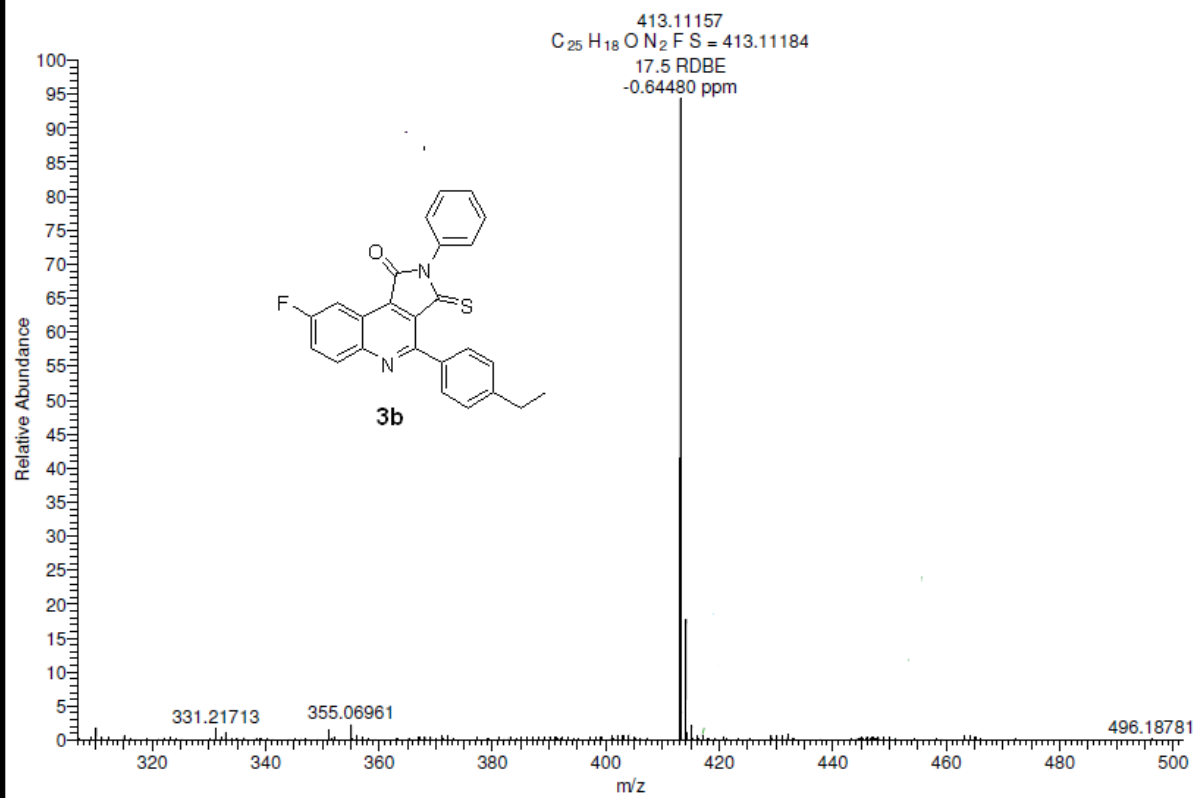
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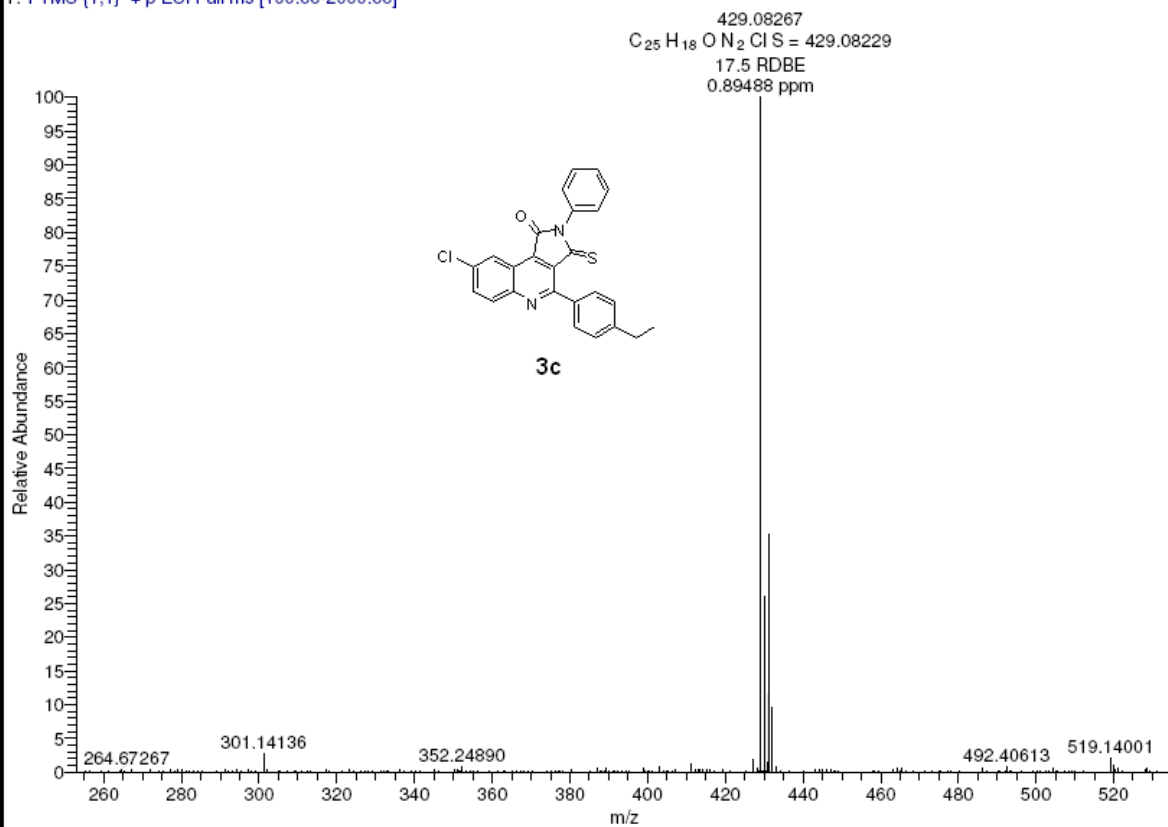
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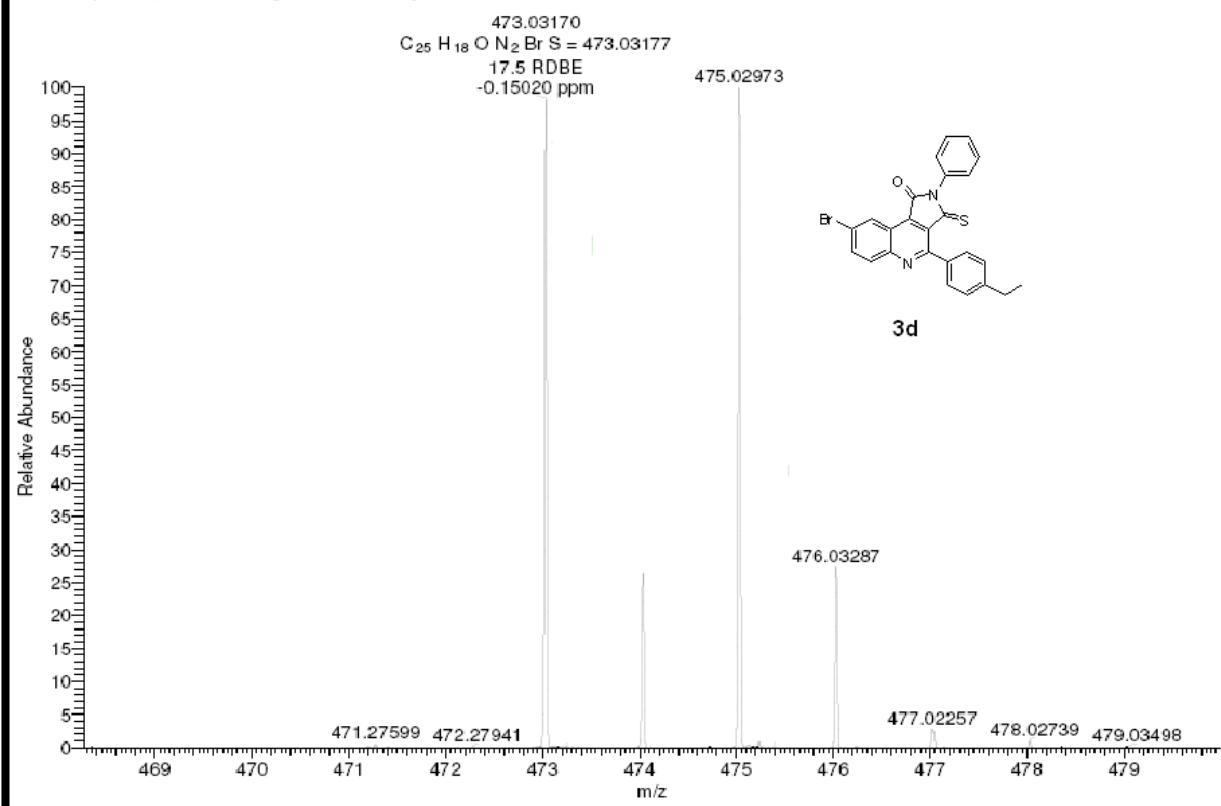
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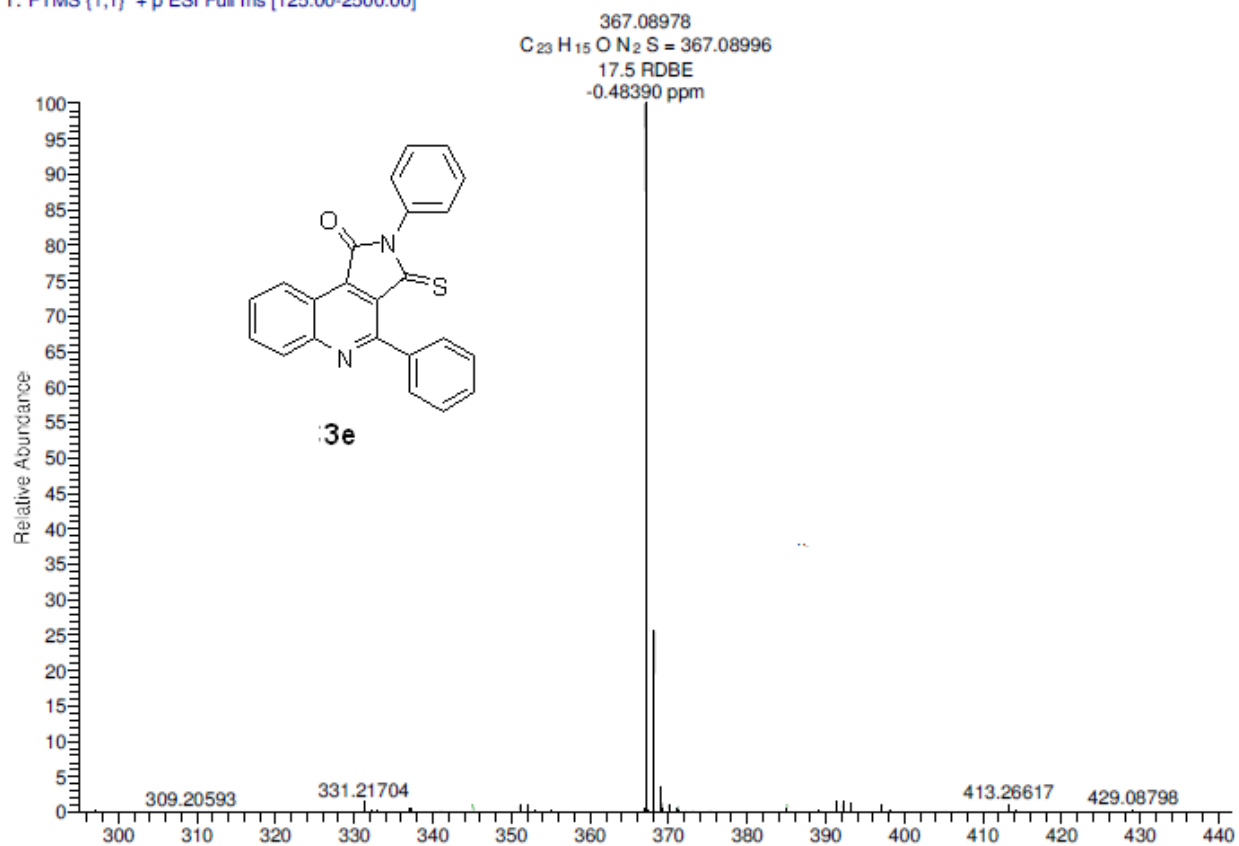
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T: FTMS (1,1) + p ESI Full ms [100.00-2000.00]



A SRINIVAS
11/29/2012 6:19:28 PM

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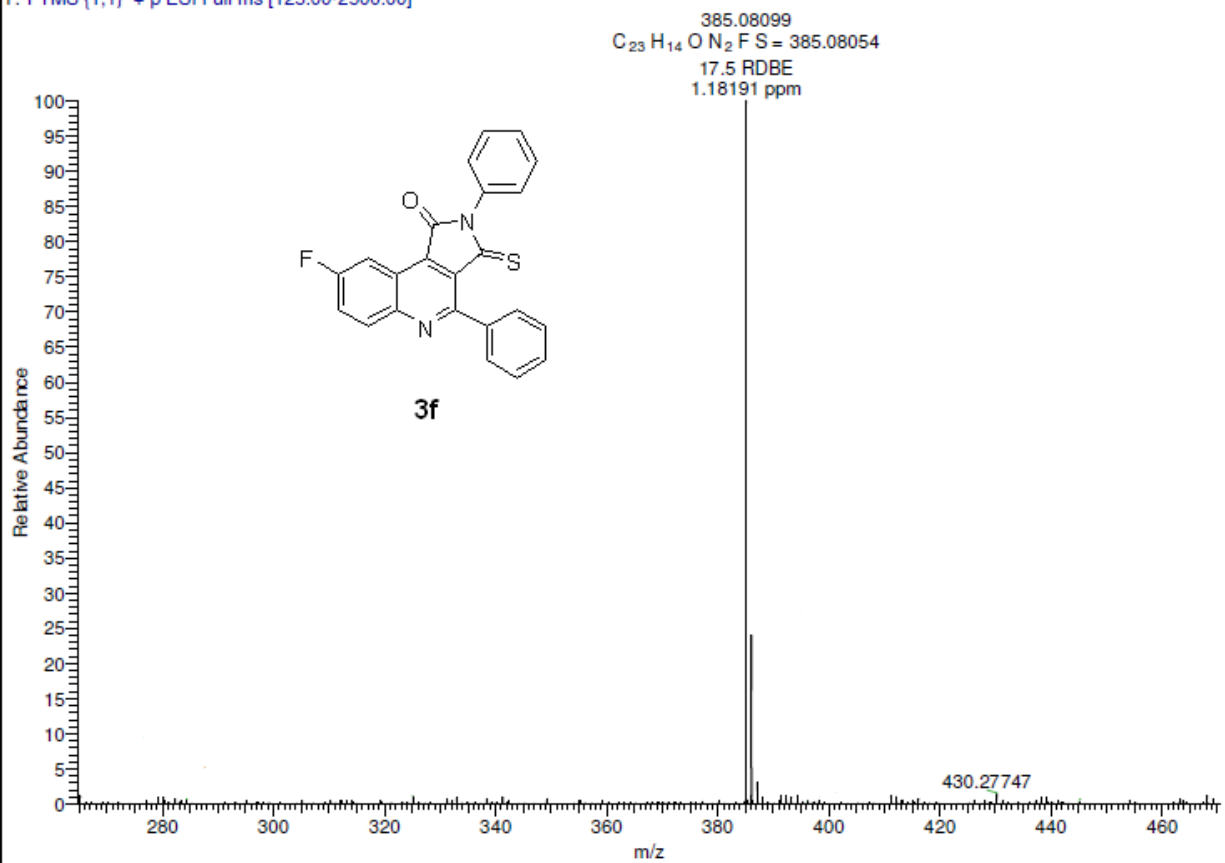
YR-QUI-4 #12 RT: 0.12 AV: 1 NL: 2.06E7
T: FTMS (1,1) +p ESI Full ms [125.00-2500.00]



A SRINIVAS
11/29/2012 6:16:42 PM

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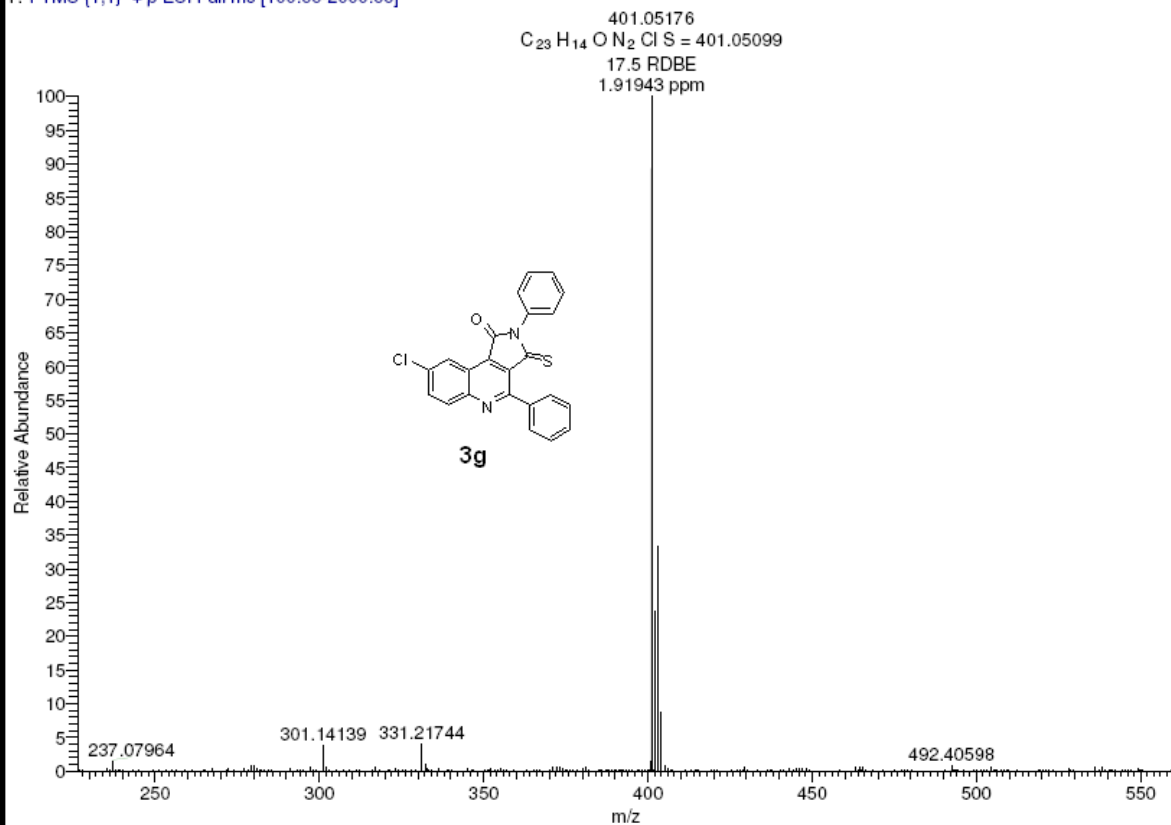
YR-QUI-5 #12 RT: 0.12 AV: 1 NL: 7.94E6
T: FTMS (1,1) + p ESI Full ms [125.00-2500.00]



A SRINIVAS
2/6/2013 6:27:37 PM

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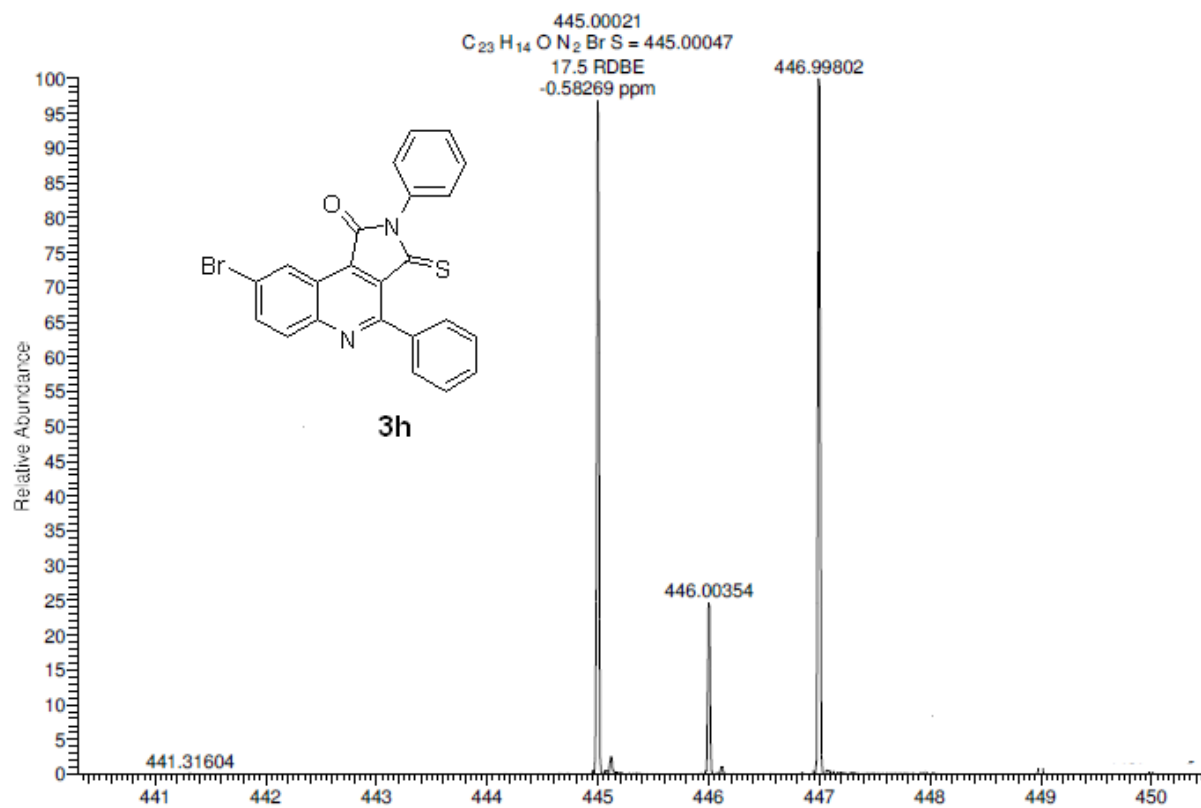
YR-QUI-24 #11 RT: 0.11 AV: 1 NL: 3.56E7
T: FTMS (1,1) +p ESI Full ms [100.00-2000.00]



A SRINIVAS
11/29/2012 6:14:08 PM

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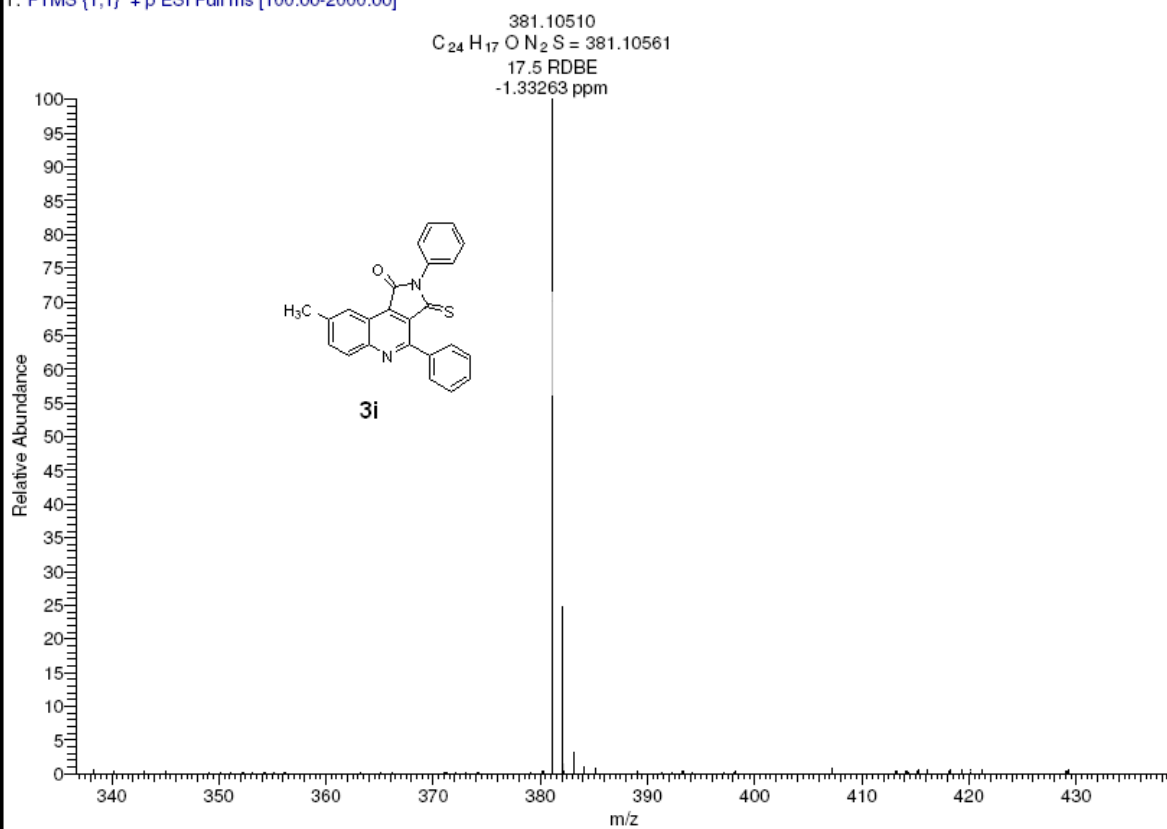
YR-QUI-6 #12 RT: 0.12 AV: 1 NL: 6.21E6
T: FTMS (1,1) + p ESI Full ms [125.00-2500.00]



A SRINIVAS
2/19/2013 4:47:00 PM

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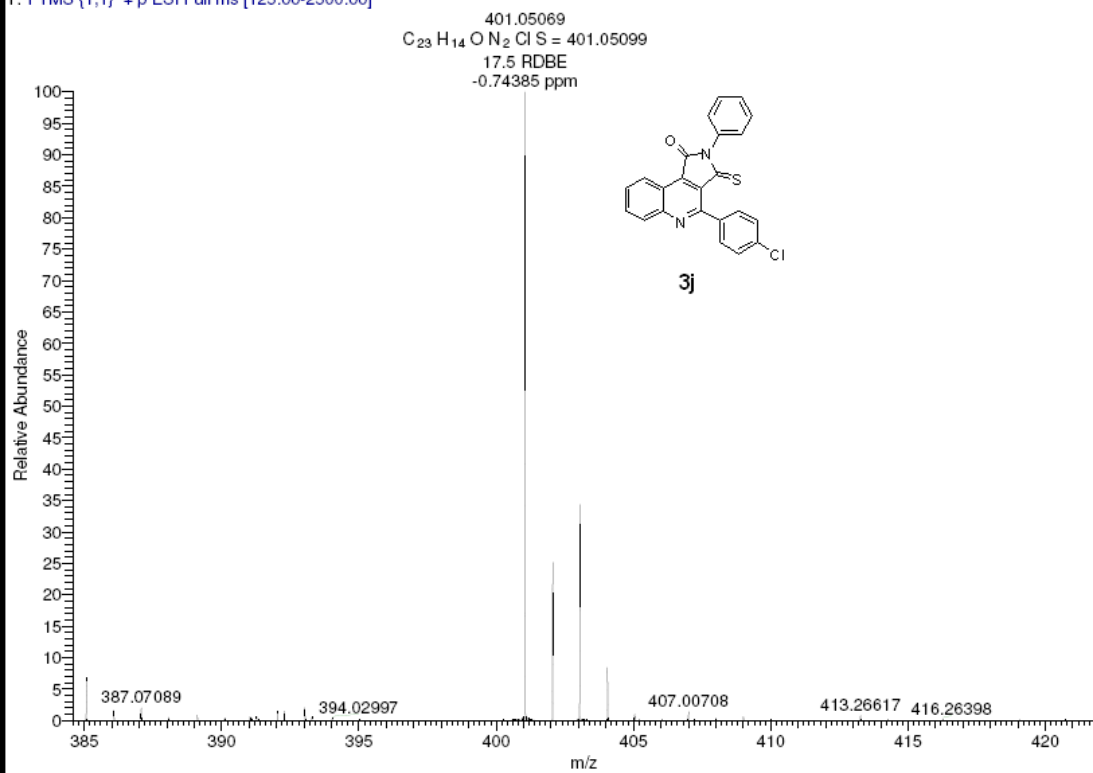
YR-QUI-27 #63 RT: 0.22 AV: 1 NL: 6.86E6
T: FTMS (1,1) + p ESI Full ms [100.00-2000.00]



A SRINIVAS
11/29/2012 6:11:36 PM

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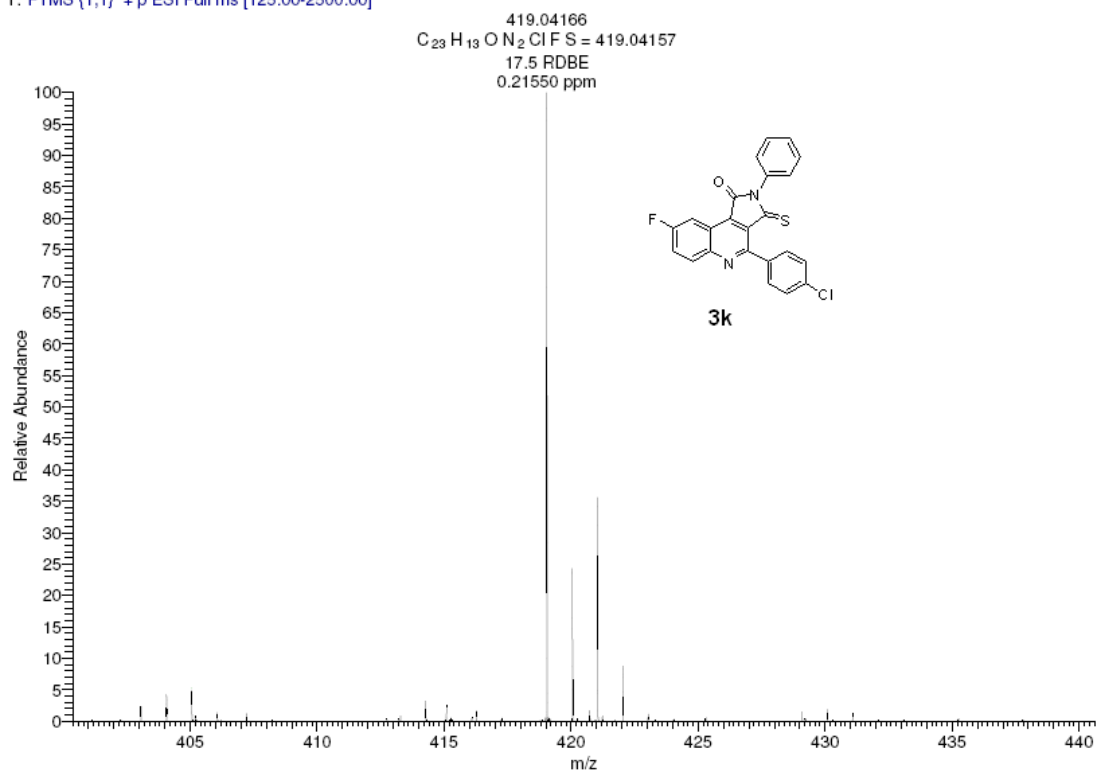
YR-QUI-7 #12 RT: 0.12 AV: 1 NL: 8.57E6
T: FTMS (1,1) + p ESI Full ms [125.00-2500.00]



A SRINIVAS
11/29/2012 6:09:02 PM

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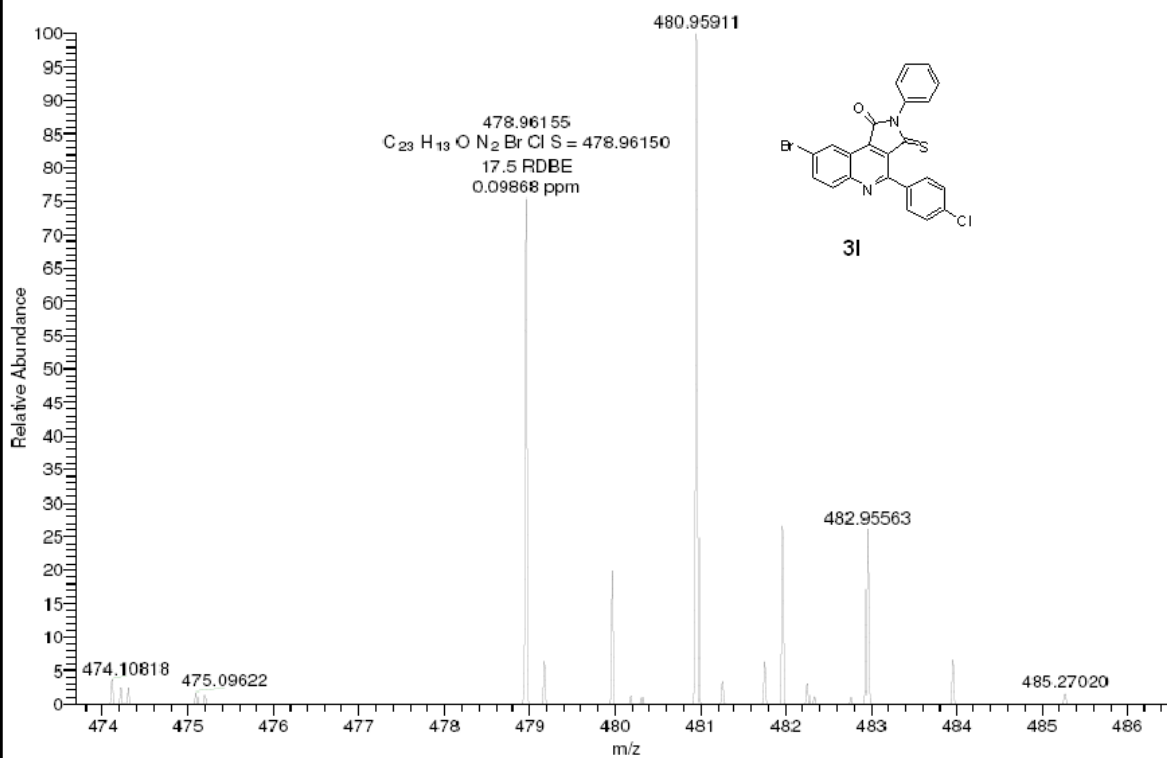
YR-QUI-8 #13 RT: 0.13 AV: 1 NL: 1.30E6
T: FTMS (1,1) + p ESI Full ms [125.00-2500.00]



A SRINIVAS
11/29/2012 6:06:28 PM

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NATIONAL CENTRE FOR MASS SPECTROMETRY

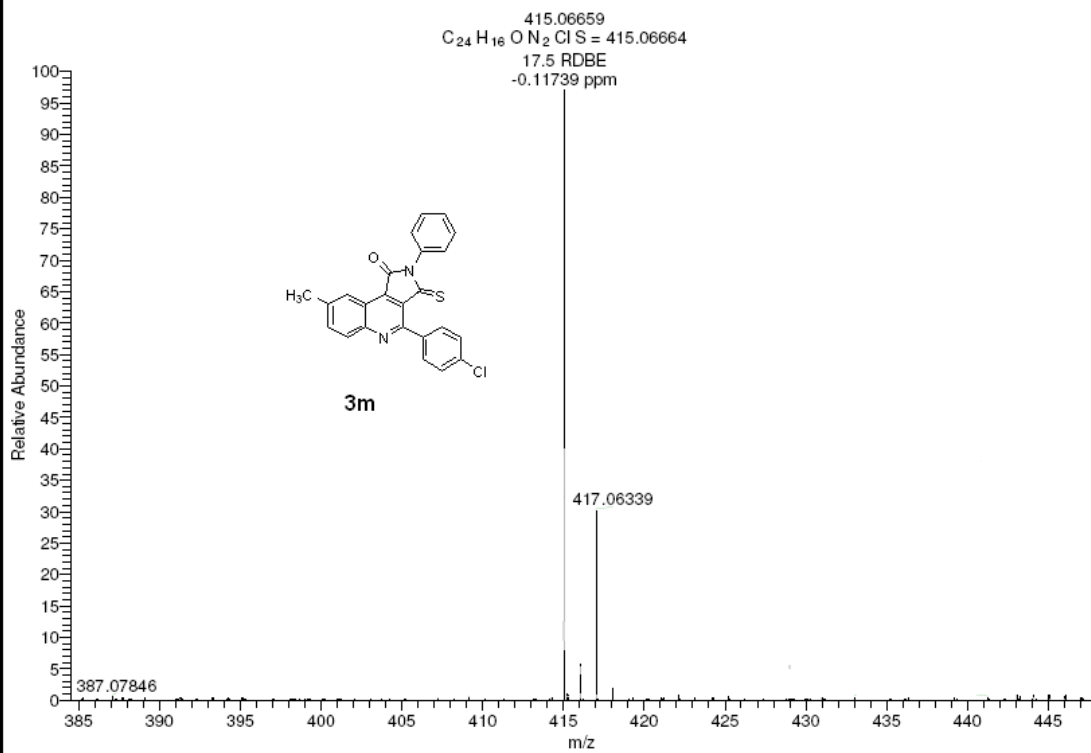
YR-QUI-9 #12 RT: 0.12 AV: 1 NL: 6.11E4
T: FTMS [1,1] + p ESI Full ms [125.00-2500.00]



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3/7/2013 10:22:44 PM

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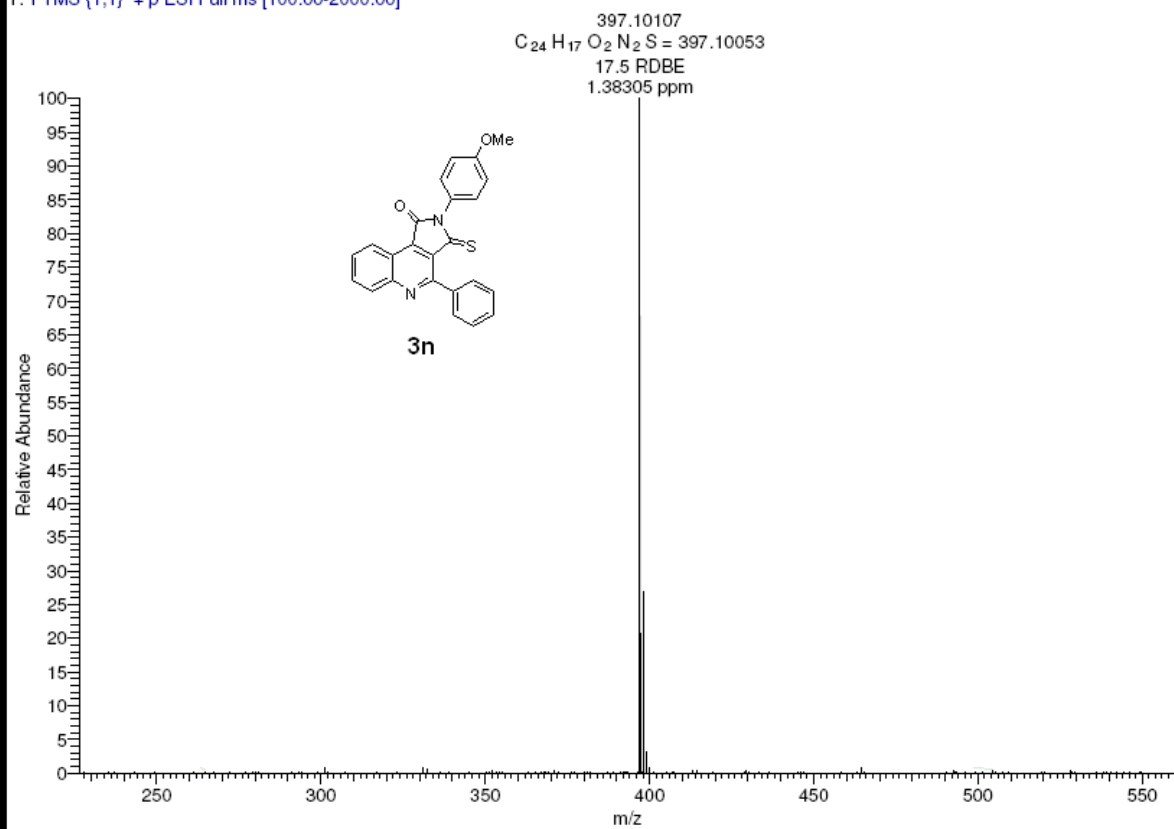
YR-QUI-28 #11 RT: 0.11 AV: 1 NL: 9.24E6
T: FTMS (1,1) + p ESI Full ms [100.00-2000.00]

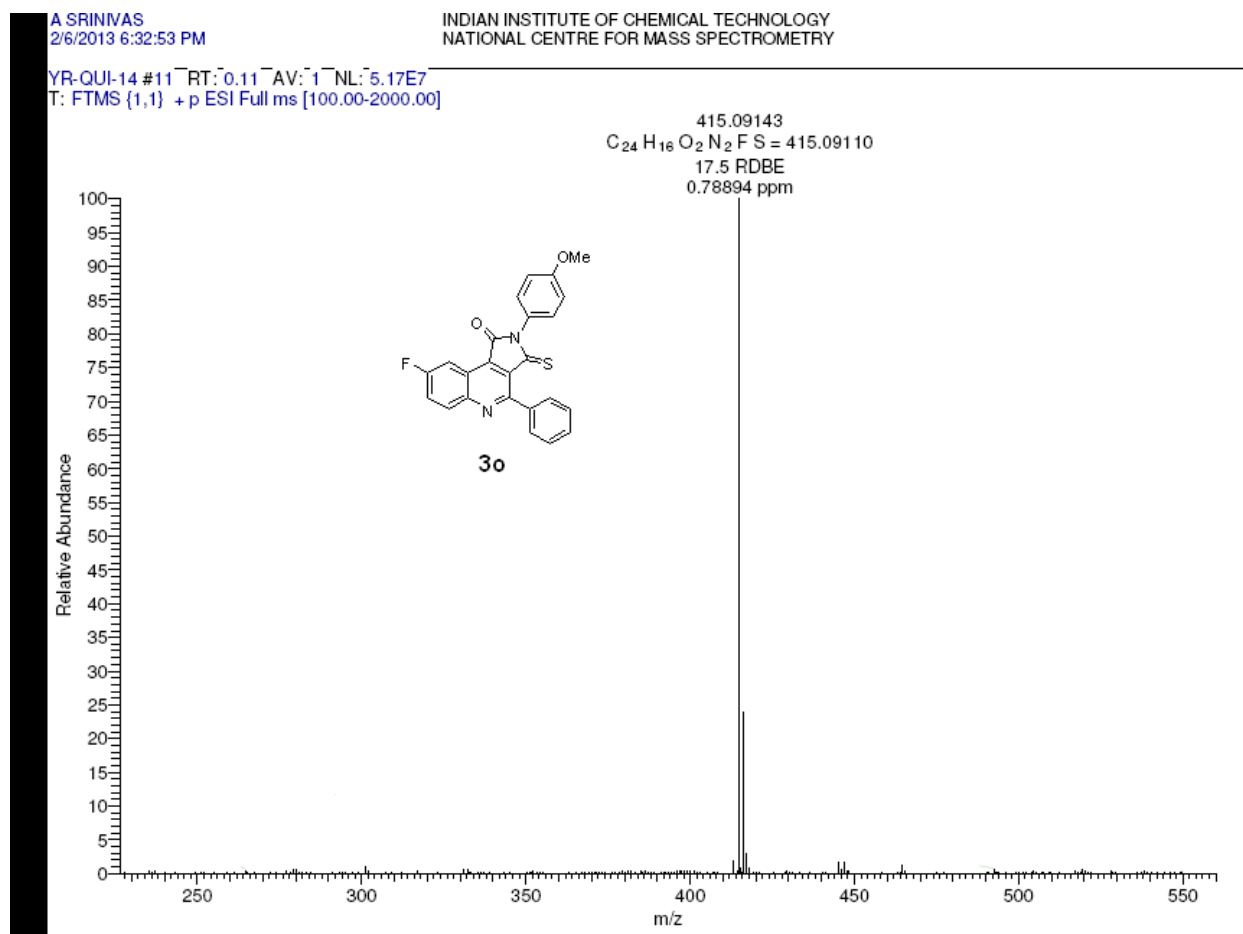


A SRINIVAS
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YR-QUI-12 #11 RT: 0.11 AV: 1 NL: 7.86E7
T: FTMS (1,1) + p ESI Full ms [100.00-2000.00]

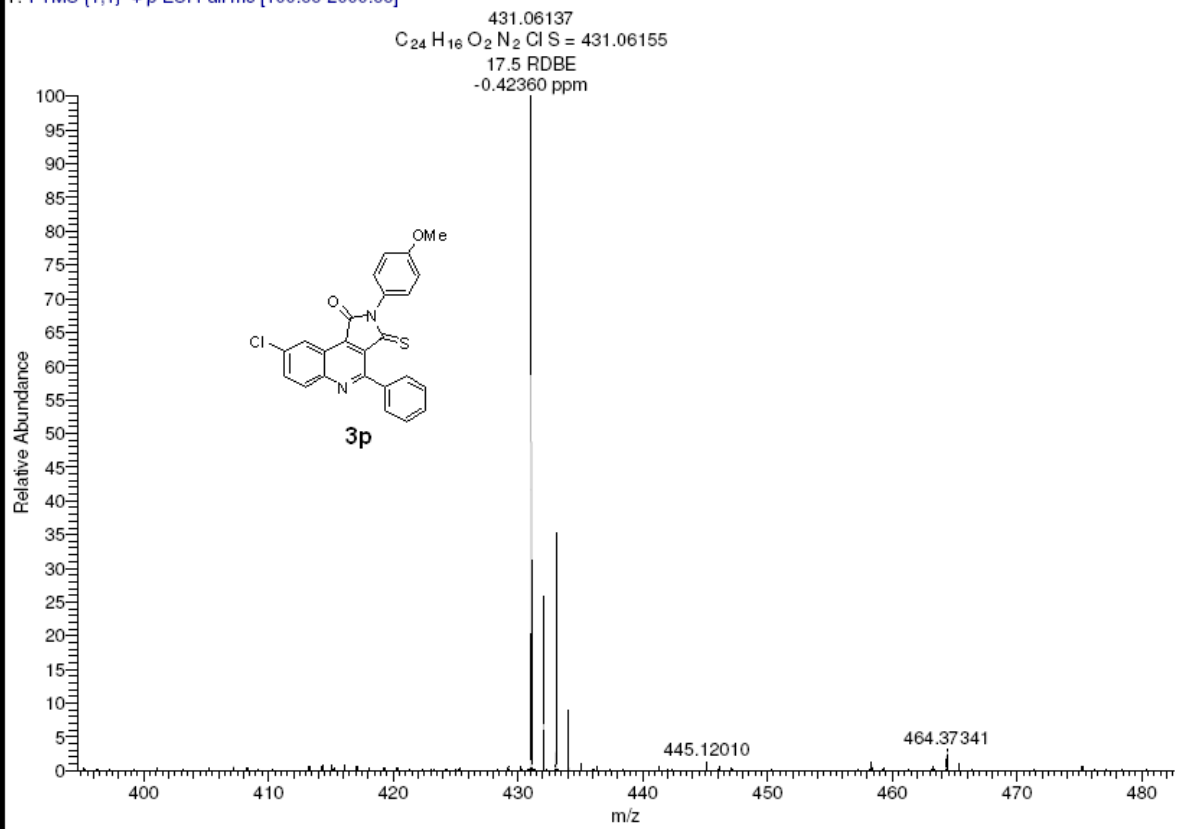




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NATIONAL CENTRE FOR MASS SPECTROMETRY

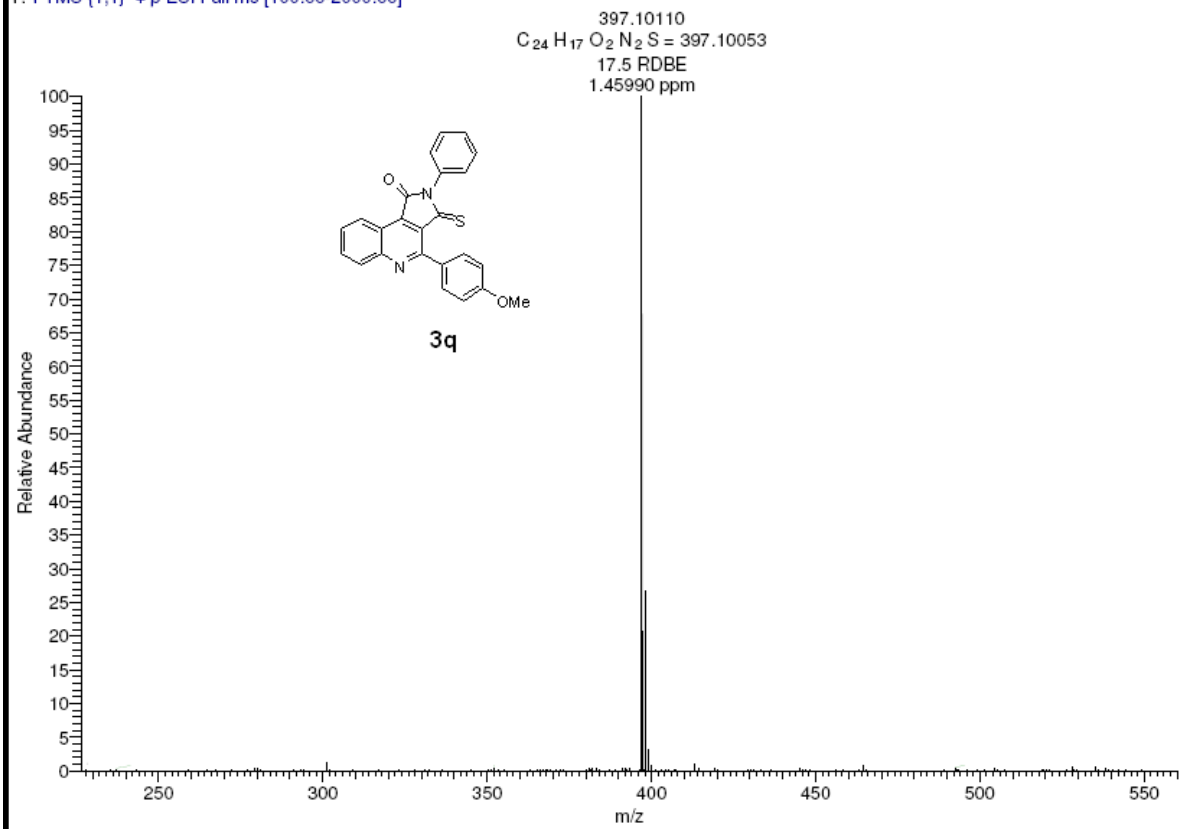
YR-QUI-25 #3-17 RT: 0.03-0.17 AV: 15 NL: 4.27E6
T: FTMS (1,1) + p ESI Full ms [100.00-2000.00]



A SRINIVAS
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NATIONAL CENTRE FOR MASS SPECTROMETRY

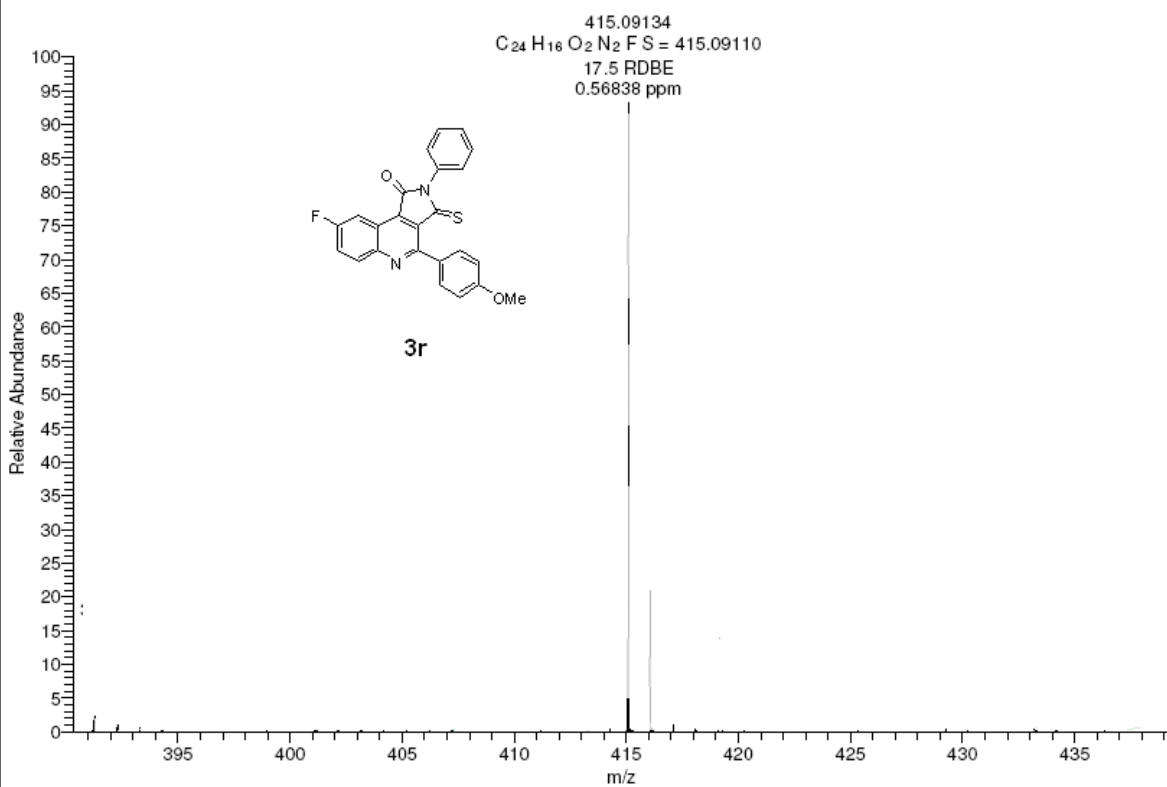
YR-QUI-16 #11 RT: 0.11 AV: 1 NL: 7.98E7
T: FTMS (1,1) + p ESI Full ms [100.00-2000.00]



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NATIONAL CENTRE FOR MASS SPECTROMETRY

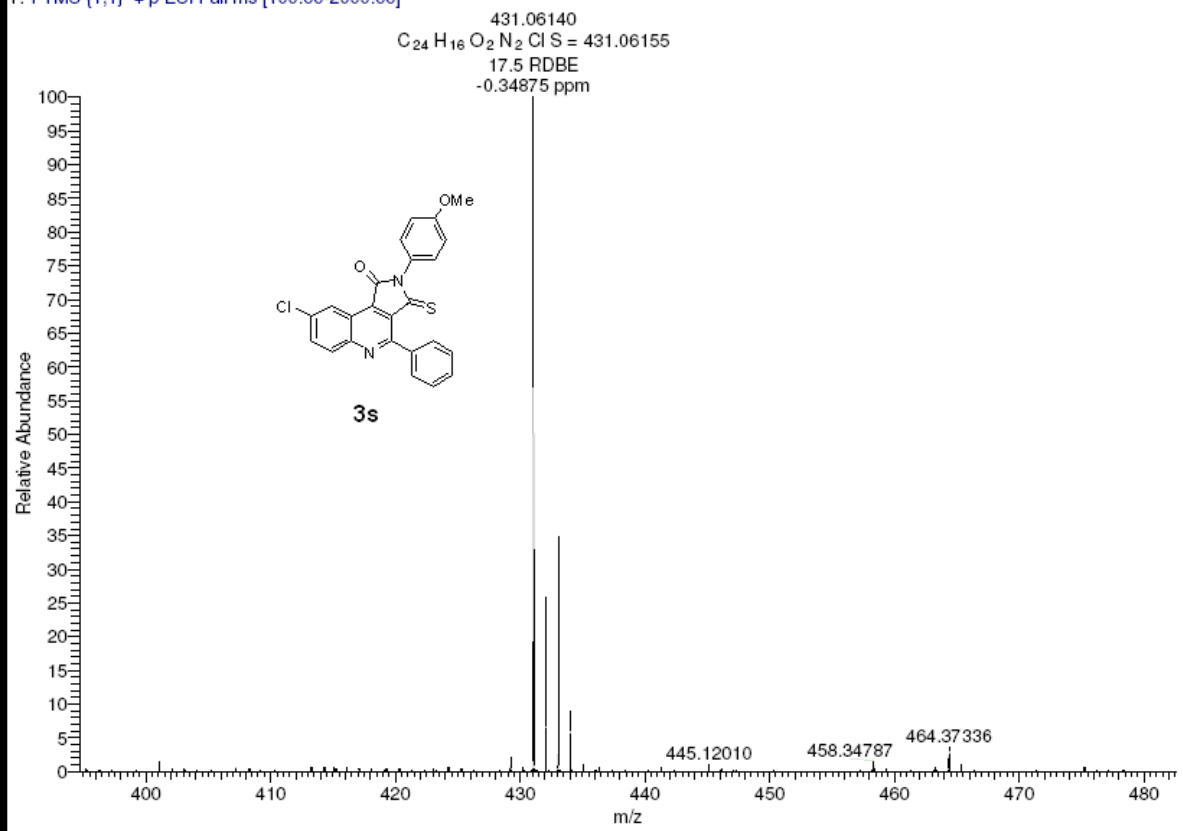
YR-QUI-17 #11 RT: 0.11 AV: 1 NL: 4.65E7
T: FTMS (1,1) + p ESI Full ms [100.00-2000.00]



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NATIONAL CENTRE FOR MASS SPECTROMETRY

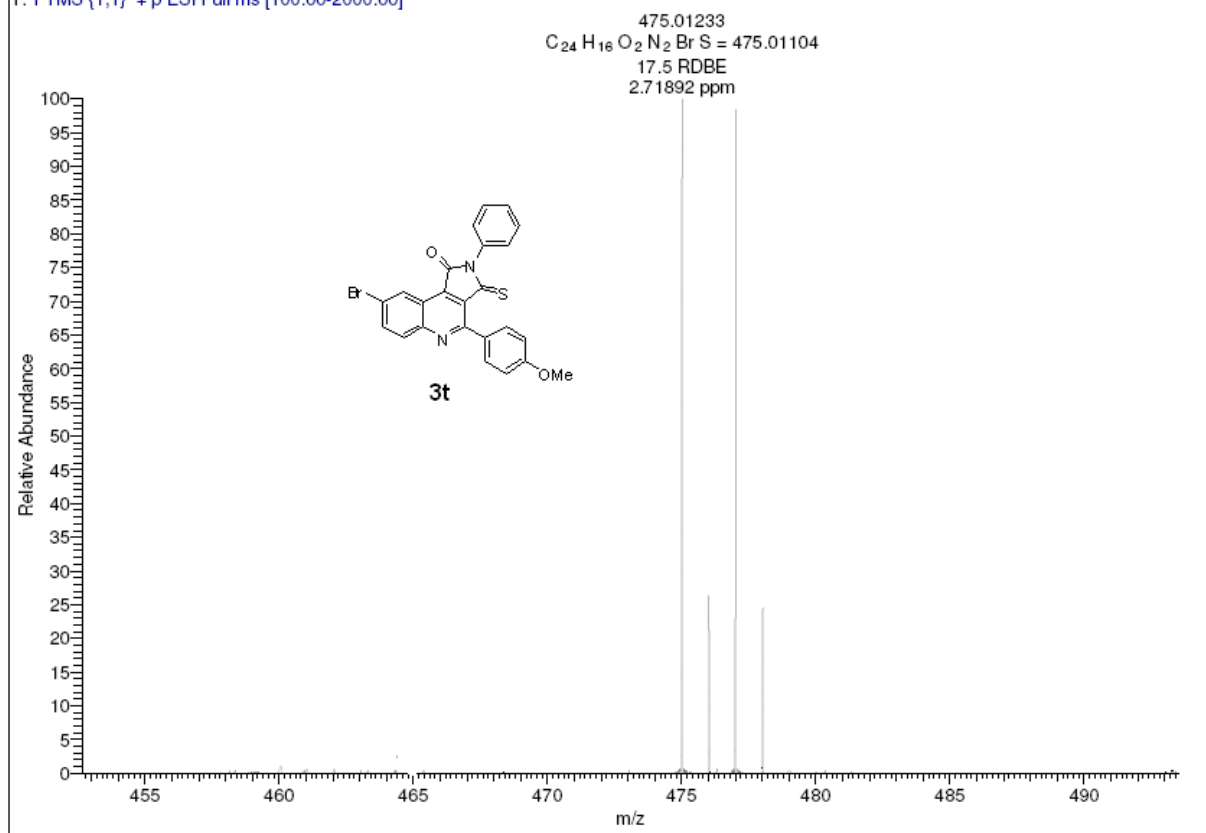
YR-QUI-29 #3-17 RT: 0.03-0.17 AV: 15 NL: 4.05E6
T: FTMS (1,1) +p ESI Full ms [100.00-2000.00]



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NATIONAL CENTRE FOR MASS SPECTROMETRY

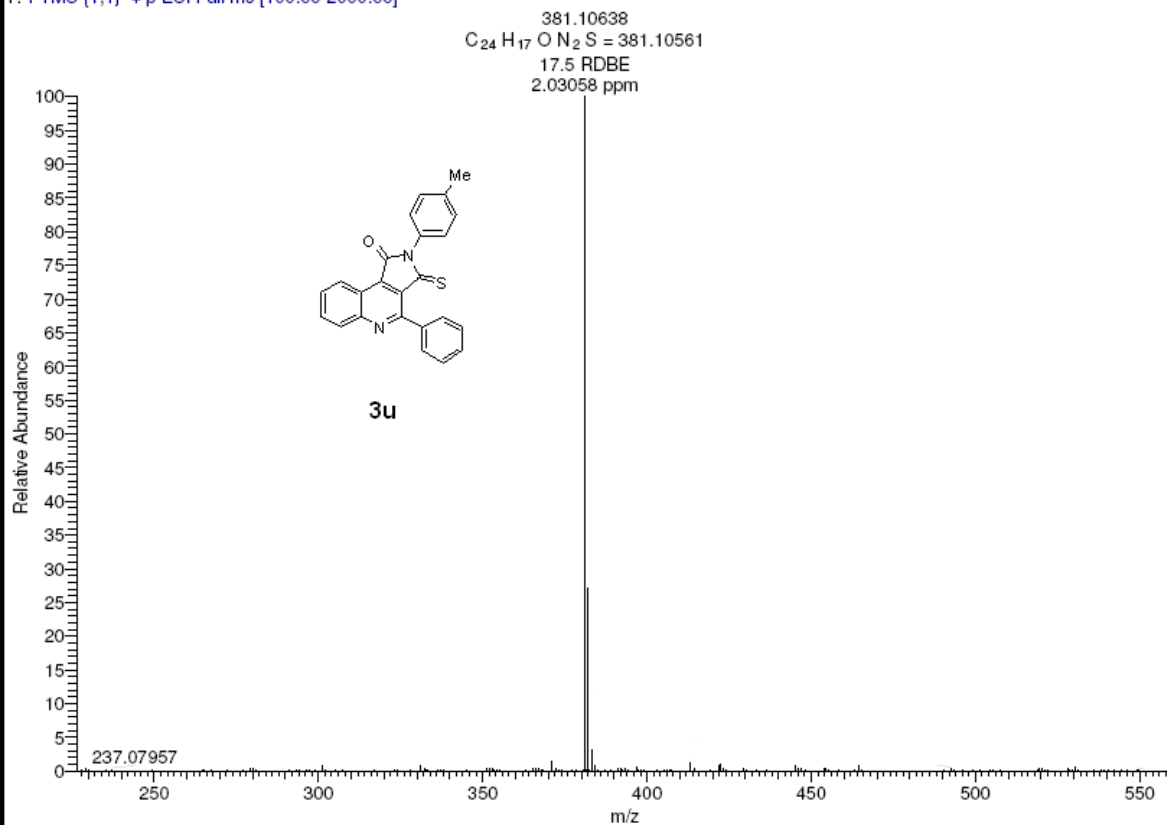
YR-QUI-18 #11 RT: 0.11 AV: 1 NL: 1.09E7
T: FTMS (1,1) + p ESI Full ms [100.00-2000.00]

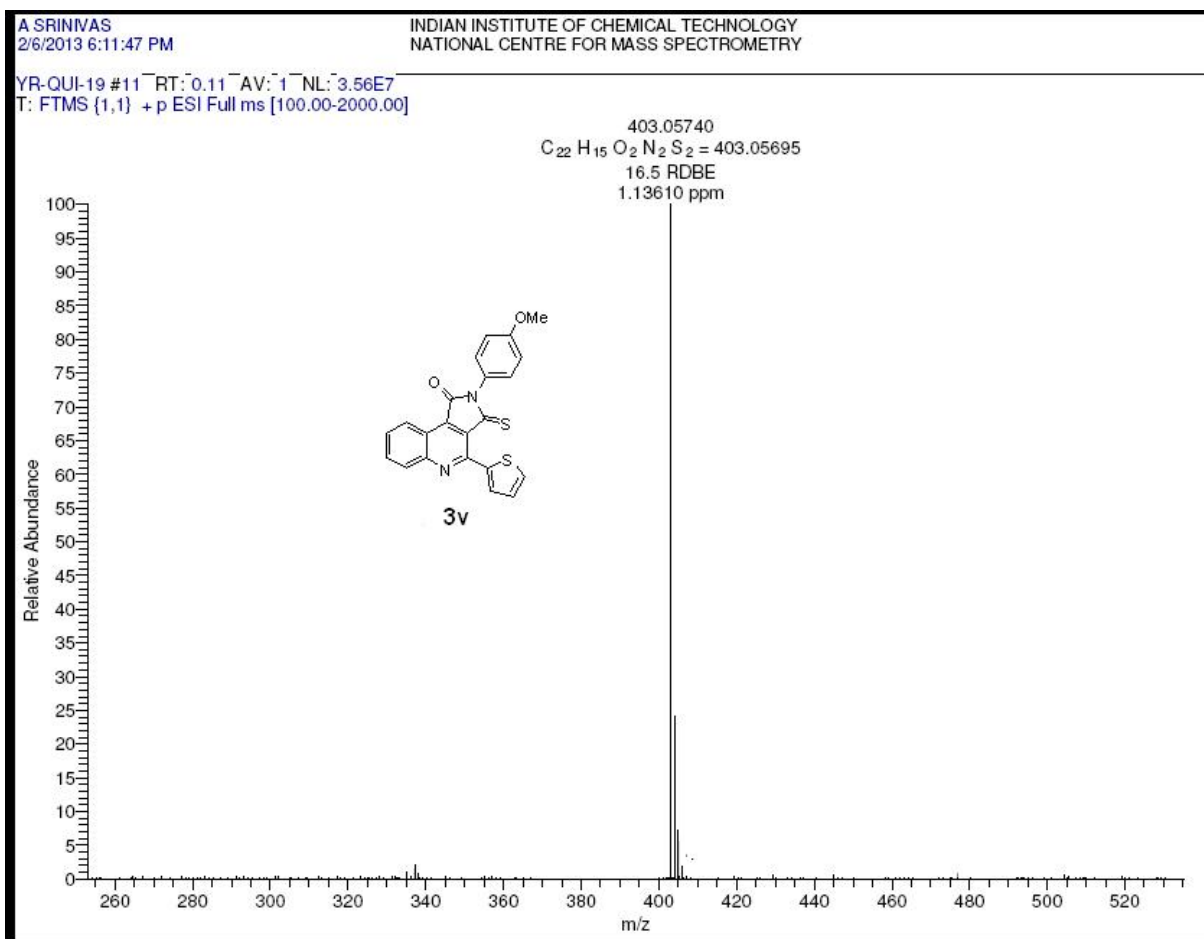


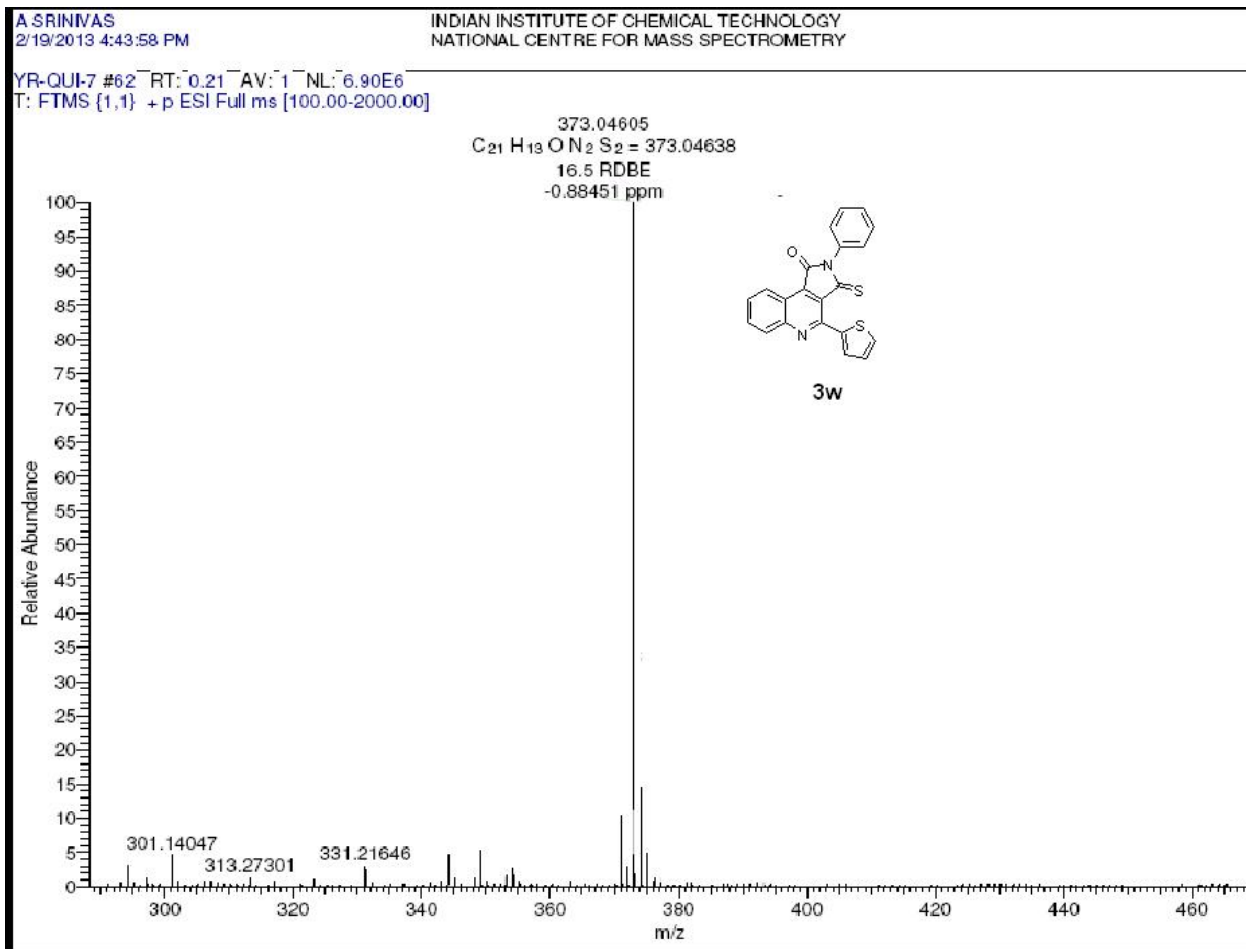
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INDIAN INSTITUTE OF CHEMICAL TECHNOLOGY
NATIONAL CENTRE FOR MASS SPECTROMETRY

YR-QUI-21 #11 RT: 0.11 AV: 1 NL: 7.20E7
T: FTMS (1,1) + p ESI Full ms [100.00-2000.00]



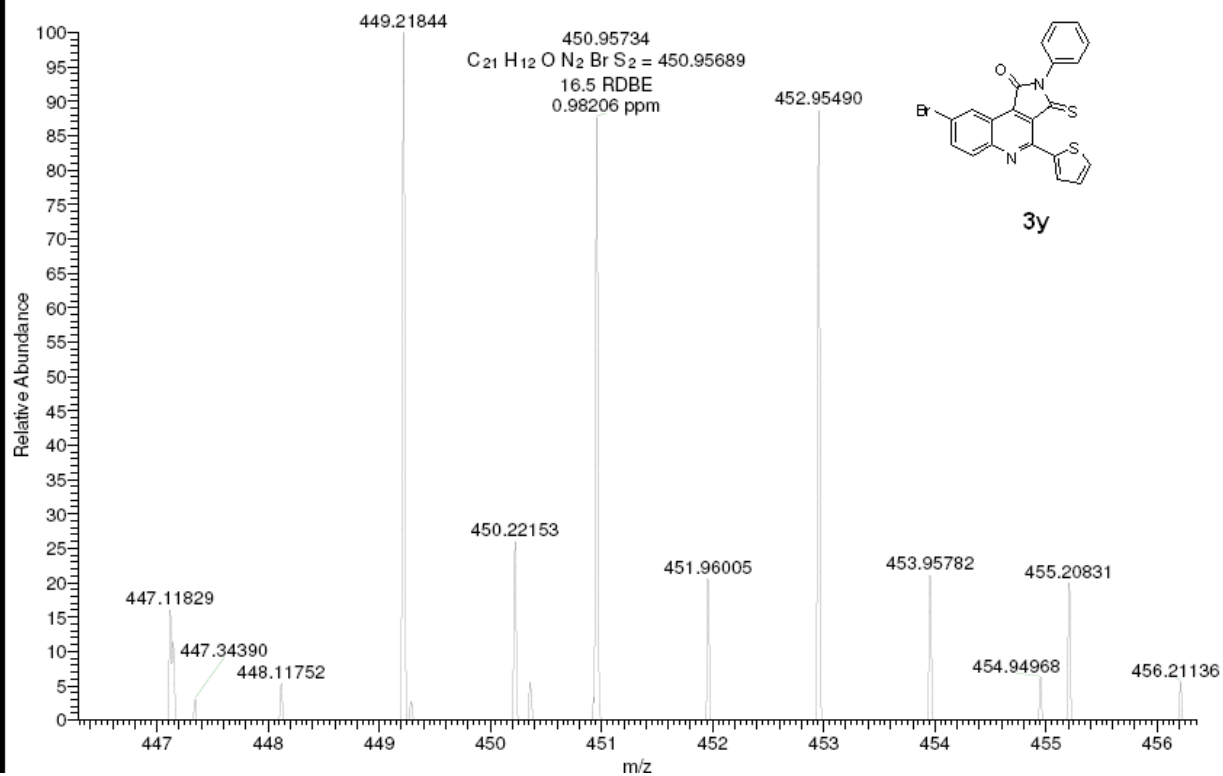


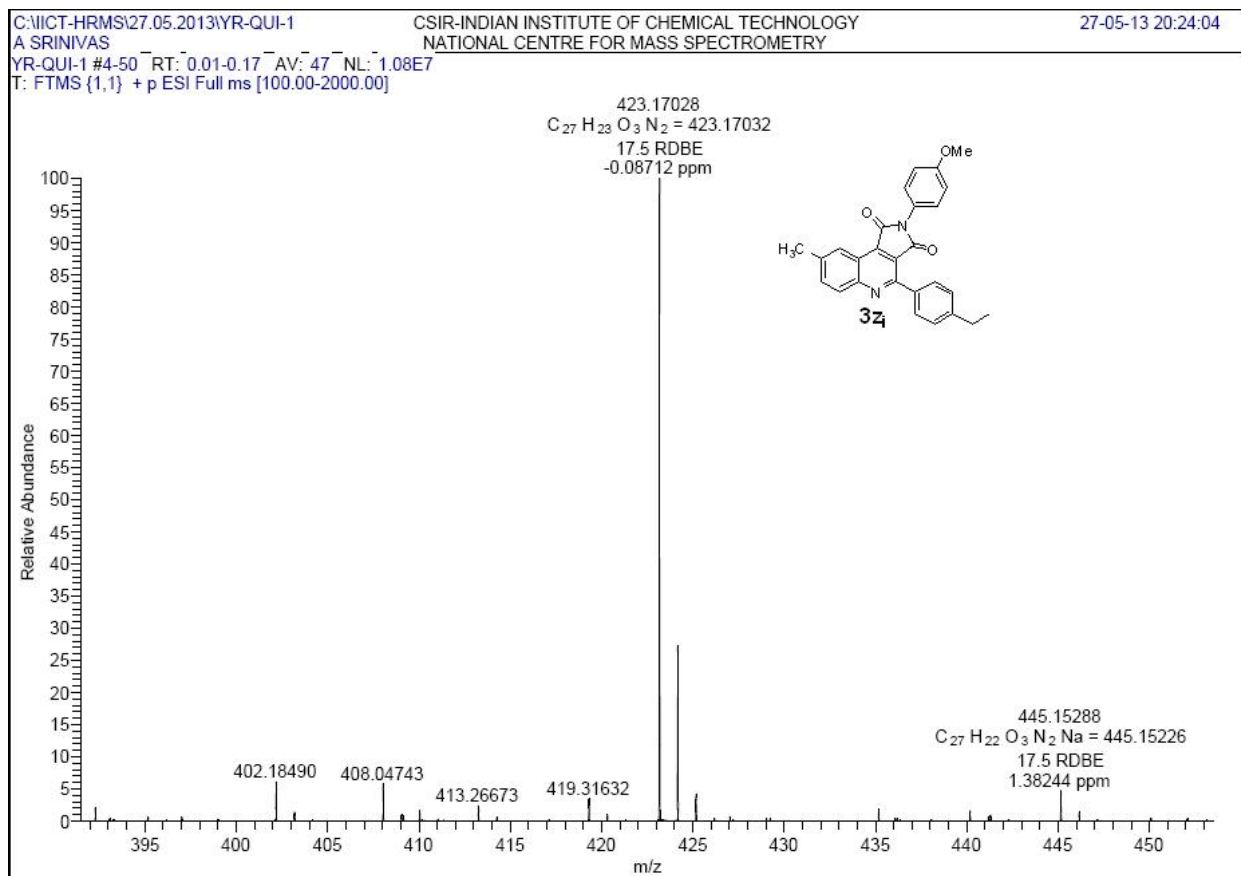


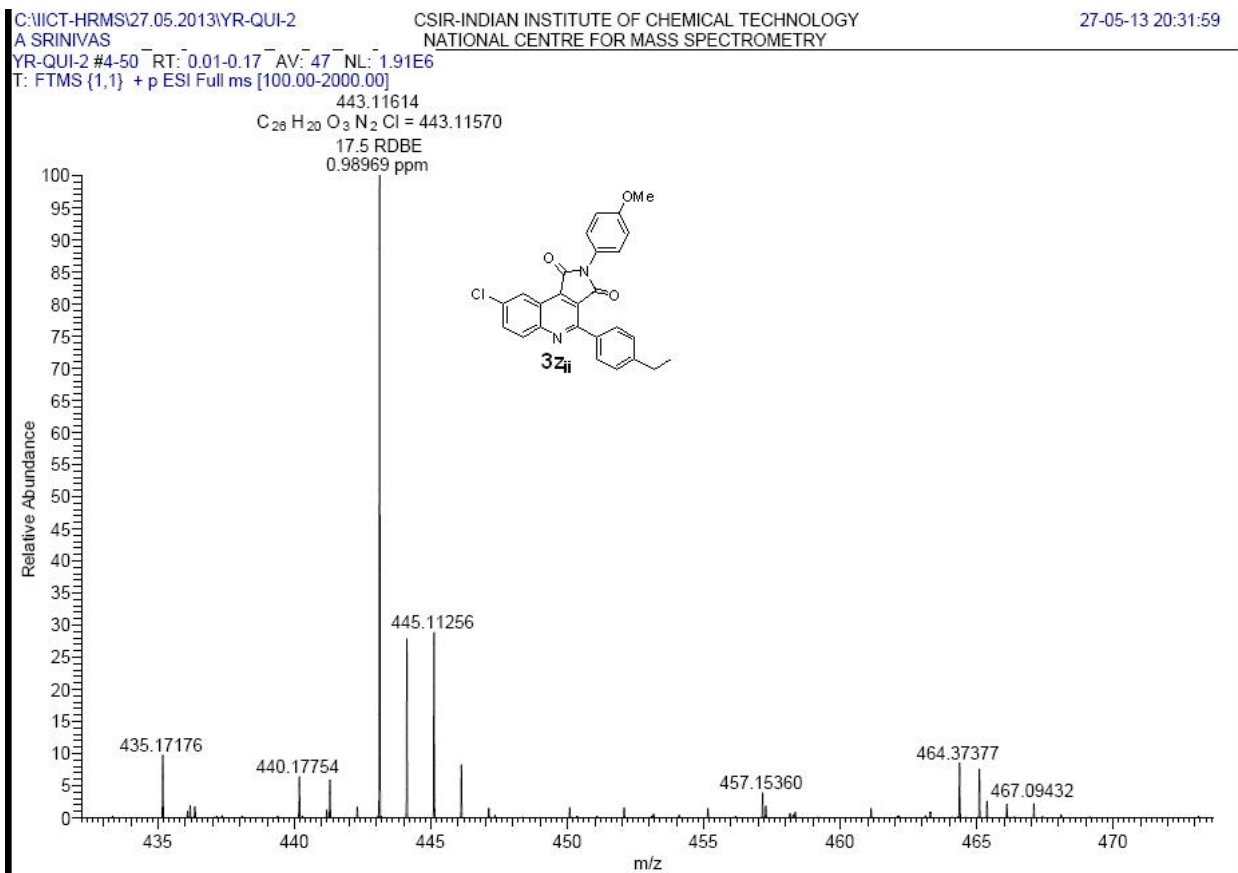
SRINIVAS
3/7/2013 10:17:38 PM

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NATIONAL CENTRE FOR MASS SPECTROMETRY

YR-QUI-12 #11 RT: 0.11 AV: 1 NL: 2.96E5
T: FTMS (1,1) + p ESI Full ms [100.00-2000.00]







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CSIR-INDIAN INSTITUTE OF CHEMICAL TECHNOLOGY

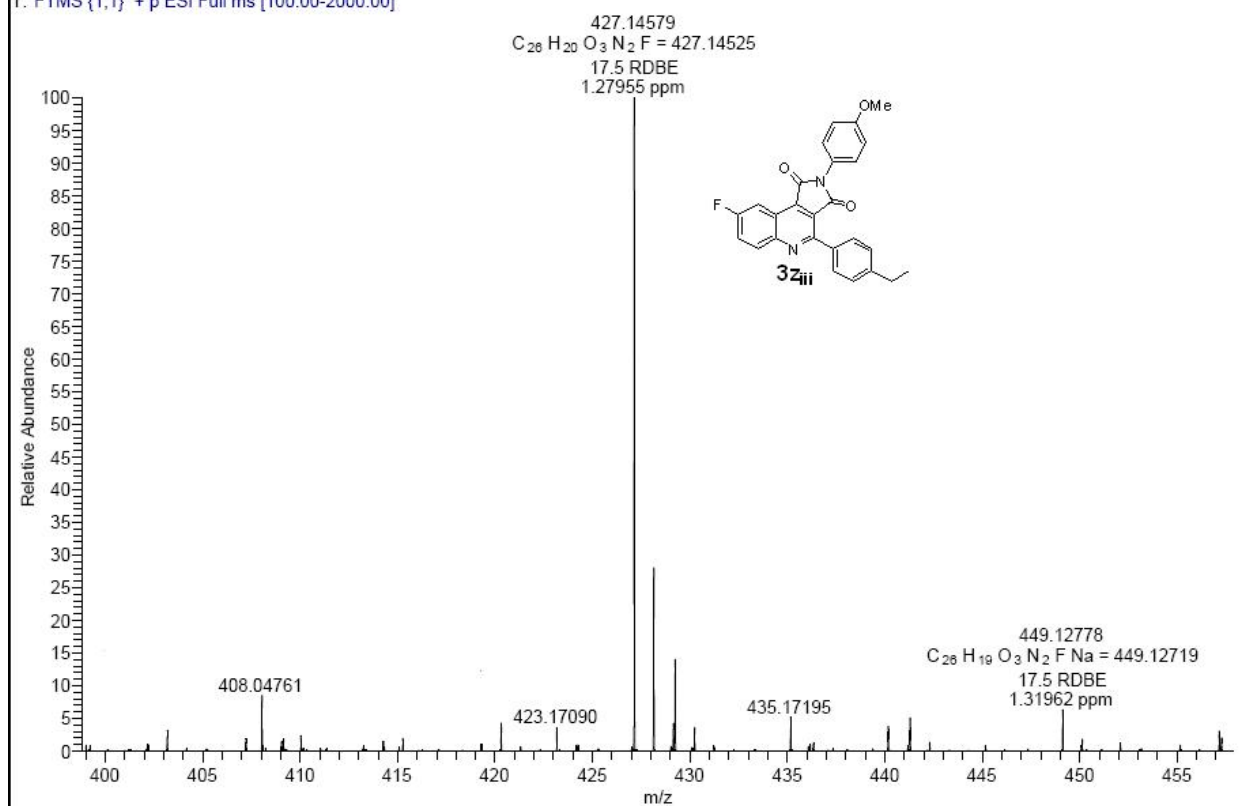
27-05-13 20:29:18

A SRINIVAS

NATIONAL CENTRE FOR MASS SPECTROMETRY

YR-QUI-3 #4-50 RT: 0.01-0.17 AV: 47 NL: 3.30E6

T: FTMS {1,1} + p ESI Full ms [100.00-2000.00]



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CSIR-INDIAN INSTITUTE OF CHEMICAL TECHNOLOGY

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A SRINIVAS

NATIONAL CENTRE FOR MASS SPECTROMETRY

YR-QUI-4 #4-50 RT: 0.01-0.17 AV: 47 NL: 9.05E5

T: FTMS (1,1) + p ESI Full ms [100.00-2000.00]

