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SUPPLEMENTARY MATERIAL

A Green Approach for the Synthesis of Novel 7,11-Dihydro-6*H*-chromeno[3,4-*e*]isoxazolo[5,4-*b*]pyridin-6-one Derivatives Using Acidic Ionic Liquid [C_4mim][HSO_4]

*Sudesh Kumari, Rajeswari M. and J. M. Khurana**

Department of Chemistry, University of Delhi, Delhi-110007
*Email: jmkhurana1@yahoo.co.in, jmkhurana@chemistry.du.ac.in

NMR Interpretation

The structures of all the novel compounds were identified by NMR (^{1}H and ^{13}C), IR and mass spectral analysis. NMR spectra of compound **4a** showed one singlet at δ 1.88 for three protons of methyl and one singlet at δ 5.16 for one methine proton. The eight aromatic protons appeared in the range of δ 7.30-8.18 and 1 N-H proton appeared as singlet at δ 11.50. One methyl carbon in ^{13}C NMR appeared at δ 9.8 and one methine carbon appeared at δ 36.8. The 15 aromatic carbons and 2 olefinic carbons appeared in the range of δ 94.8-159.2. The carbonyl carbon appeared at δ 160.2. IR spectra showed peaks at 3223 cm^{-1} (N-H stretch) and 1679 cm^{-1} (Carbonyl stretch). Mass spectrum of **4a** showed a molecular ion peak at (*m/z*) 365.0694 $(M+H)^+$. The position of peaks methyl, methylene, methine and quaternary carbons were assigned by DEPT spectra of compound **4a**.

Spectral data

7-(4-Chlorophenyl)-8-methyl-7,11-dihydro-6*H*-chromeno[3,4-*e*]isoxazolo[5,4-*b*]pyridin-6-one (4a)

White solid; Obs. M.p.: 253-255°C; Yield: Method A = 92% , Method B = 94%; ^{1}H NMR (400 MHz, DMSO-d_6) δ_H: 11.50 (s, 1H, N-H), 8.18 (d, 1H, J = 7.63 Hz, Ar-H), 7.65-7.61 (t, 1H, Ar-H), 7.43-7.39 (t, 1H, Ar-H), 7.36 (d, 1H, J = 8.39 Hz, Ar-H) 7.32-7.27 (m, 4H, Ar-H), 5.16 (s, 1H, -CH), 1.88 (s, 3H, -CH_3); ^{13}C NMR (100 MHz, DMSO-d_6) δ_C: 160.2, 159.2,

158.4, 152.2, 144.3, 143.2, 132.3, 131.2, 129.8, 128.2, 124.2, 123.1, 116.8, 113.3, 100.1, 94.8, 36.8, 9.8; IR (ν_{max}, cm^{-1}) (KBr): 3223, 1679, 1509, 1264; HRMS (ESI) *m/z* calcd. for $C_{20}H_{13}ClN_2O_3$: 364.0615, HRMS (ESI) found: 365.0694 $[M+H]^+$.

7-(4-Methoxyphenyl)-8-methyl-7,11-dihydro-6*H*-chromeno[3,4-*e*]isoxazolo[5,4-*b*]pyridin-6-one (4b)

White solid; Obs. M.p.: 248-249ºC; Yield: Method A = 93% , Method B = 92%; ^1H NMR (400 MHz, DMSO-d_6) δ_H: 11.40 (s, 1H, N-H), 8.14 (d, 1H, *J* = 6.87 Hz, Ar-H), 7.61-7.57 (t, 1H, Ar-H), 7.40-7.36 (t, 1H, Ar-H), 7.32 (d, 1H, *J* = 7.63 Hz, Ar-H), 7.12 (d, 2H, *J* = 7.63 Hz, Ar-H), 6.78 (d, 2H, *J* = 7.63 Hz, Ar-H), 5.03 (s, 1H, -CH), 3.64 (s, 3H, $-OCH_3$), 1.86 (s, 3H, $-CH_3$); ^{13}C NMR (100 MHz, DMSO-d_6) δ_C: 160.2, 159.0, 158.5, 157.9, 152.1, 142.7, 137.6, 132.2, 128.9, 124.2, 123.0, 116.8, 113.6, 113.4, 100.8, 95.3, 55.0, 36.4, 9.8; IR (ν_{max}, cm^{-1}) (KBr): 3233, 1680, 1517, 1258; HRMS (ESI) *m/z* calcd. for $C_{21}H_{16}N_2O_4$: 360.1104, HRMS (ESI) found: 361.1177 $[M+H]^+$.

8-Methyl-7-(4-nitrophenyl)-7,11-dihydro-6*H*-chromeno[3,4-e]isoxazolo[5,4-b]pyridin-6-one (4c)

Pale yellow solid; Obs. M.p.: 247-248ºC; Yield: Method A = 90% , Method B = 90%; ^1H NMR (400 MHz, DMSO-d_6) δ_H: 11.55 (s, 1H, N-H), 8.14-8.08 (m, 3H, Ar-H), 7.58-7.53 (m, 3H, Ar-H), 7.37-7.29 (m, 2H, Ar-H), 5.26 (s, 1H, -CH), 1.84 (s, 1H, $-CH_3$); ^{13}C NMR (100 MHz, DMSO-d_6) δ_C: 160.3, 159.3, 158.4, 152.5, 152.3, 146.2, 143.7, 132.5, 129.3, 124.3, 123.6, 123.2, 116.8, 113.2, 99.4, 94.2, 37.5, 9.9; IR (ν_{max}, cm^{-1}) (KBr): 3190, 1678, 1509, 1262, 1070; HRMS (ESI) *m/z* calcd. for $C_{20}H_{13}N_3O_5$: 375.0863, HRMS (ESI) found: 376.0936 $[M+H]^+$.

7-(4-Bromophenyl)-8-methyl-7,11-dihydro-6*H*-chromeno[3,4-e]isoxazolo[5,4-b]pyridin-6-one (4d)

White solid; Obs. M.p.: 250-252°C; Yield: Method A = 92% , Method B = 94%; ^{1}H NMR (400 MHz, DMSO-*d*$_6$) δ_H: 11.51 (s, 1H, N-H), 8.18 (d, 1H, *J* = 7.63 Hz, Ar-H), 7.65-7.61 (t, 1H, Ar-H), 7.43-7.39 (t, 1H, Ar-H), 7.36 (d, 1H, *J* = 8.39 Hz, Ar-H) 7.30 (s, 4H, Ar-H), 5.15 (s, 1H, -CH), 1.88 (s, 3H, -CH_3); ^{13}C NMR (100 MHz, DMSO-*d*$_6$) δ_C: 160.3, 159.2, 158.5, 152.2, 144.3, 143.2, 132.4, 131.2, 129.8, 128.2, 124.2, 123.1, 116.8, 113.3, 100.1, 94.8, 36.8, 9.8; IR (ν_{max}, cm^{-1}) (KBr): 3137, 1673, 1557, 1258; HRMS (ESI) *m/z* calcd. for $C_{20}H_{13}BrN_2O_3$: 408.0110, HRMS (ESI) found: 409.0194 $[M+H]^+$.

8-Methyl-7-(4-methylphenyl)--7,11-dihydro-6*H*-chromeno[3,4-*e*]isoxazolo[5,4-*b*]pyridin-6-one (4e)

White solid; Obs. M.p.: 240-242°C; Yield: Method A = 92% , Method B = 90%; ^{1}H NMR (400 MHz, DMSO-*d*$_6$) δ_H: 11.42 (s, 1H, N-H), 8.16 (d, 1H, *J* = 7.63 Hz, Ar-H), 7.63-7.59 (t, 1H, Ar-H), 7.41-7.38 (t, 1H, Ar-H), 7.34 (d, 1H, *J* = 8.39 Hz, Ar-H), 7.12-7-03 (q, 4H, Ar-H), 5.06 (s, 1H, -CH), 2.20 (s, 3H, -CH_3), 1.87 (s, 3H, -CH_3); ^{13}C NMR (100 MHz, DMSO-*d*$_6$) δ_C: 160.2, 159.0, 158.4, 152.1, 142.9, 142.5, 135.7, 132.1, 128.8, 127.7, 124.1, 123.0, 116.7, 113.3, 100.6, 95.2, 36.9, 20.6, 9.8; IR (ν_{max}, cm^{-1}) (KBr): 3193, 1678, 1510, 1062; HRMS (ESI) *m/z* calcd. for $C_{21}H_{16}N_2O_3$: 344.1173, HRMS (ESI) found: 345.1246 $[M+H]^+$.

7-(4-Fluorophenyl)-8-methyl-7,11-dihydro-6*H*-chromeno[3,4-*e*]isoxazolo[5,4-*b*]pyridin-6-one (4f)

White solid; Obs. M.p.: 250-252°C; Yield: Method A = 92% , Method B = 93%; ^{1}H NMR (400 MHz, DMSO-*d*$_6$) δ_H: 11.43 (s, 1H, N-H), 8.13 (d, 1H, *J* = 7.63 Hz, Ar-H), 7.58-7.54 (t, 1H, Ar-H), 7.37-7.33 (t, 1H, Ar-H), 7.30-7.25 (m, 3H, Ar-H), 7.05-7-01 (t, 2H, Ar-H), 5.08 (s, 1H, -CH), 1.83 (s, 3H, -CH_3); ^{13}C NMR (100 MHz, DMSO-*d*$_6$) δ_C: 160.2, 159.1, 158.4, 152.2, 143.0, 141.6, 132.2, 129.8, 124.2, 123.1, 116.8, 114.9, 113.3, 100.3, 95.0, 36.6, 9.8 ; IR (ν_{max}, cm^{-1}) (KBr): 3153, 1681, 1510, 1385, 1225; HRMS (ESI) *m/z* calcd. for $C_{20}H_{13}FN_2O_3$: 348.0916, HRMS (ESI) found: 349.0988 $[M+H]^+$.

8-Methyl-7-(naphthalen-2-yl)-7,11-dihydro-6*H*-chromeno[3,4-*e*]isoxazolo[5,4-*b*]pyridin-6-one (4g)

White solid; Obs. M.p.: 247-249ºC; Yield: Method A = 90% , Method B = 92%; ^{1}H NMR (400 MHz, DMSO-d_6) δ_H: 11.52 (s, 1H, N-H), 8.22 (d, 1H, J = 7.63 Hz, Ar-H), 7.86-7.78 (m, 4H, Ar-H), 7.64-7.60 (t, 1H, Ar-H), 7.43-7.34 (m, 5H, Ar-H), 5.31 (s, 1H, -CH), 1.87 (s, 3H, -CH_3); ^{13}C NMR (100 MHz, DMSO-d_6) δ_C: 160.3, 159.2, 158.6, 152.3, 143.3, 142.8, 132.8, 132.3, 132.0, 128.0, 127.8, 127.5, 126.5, 126.2, 125.8, 124.3, 123.2, 116.8, 113.4, 100.4, 95.1, 37.6, 21.1, 9.9; IR (ν_{max}, cm^{-1}) (KBr): 3223, 1681, 1518, 1260; HRMS (ESI) *m/z* calcd. for $C_{24}H_{16}N_2O_3$: 380.1158, HRMS (ESI) found: 381.1230 $[M+H]^+$.

8-Methyl-7-(3-nitrophenyl)-7,11-dihydro-6*H*-chromeno[3,4-*e*]isoxazolo[5,4-*b*]pyridin-6-one (4h)

Pale yellow solid; Obs. M.p.: 248-250ºC; Yield: Method A = 93% , Method B = 91%; ^{1}H NMR (400 MHz, DMSO-d_6) δ_H: 11.50 (s, 1H, N-H), 8.10-8.05 (m, 2H, Ar-H), 7.91 (s, 1H, Ar-H), 7.65- 7.54 (m, 3H, Ar-H), 7.41-7.37 (m, 2H, Ar-H), 5.67 (s, 1H, -CH), 2.05 (s, 1H, -CH_3) ; ^{13}C NMR (100 MHz, DMSO-d_6) δ_C: 167.1, 163.2, 162.3, 160.6, 152.2, 147.9, 142.5, 134.3, 132.5, 129.6, 124.2, 123.7, 121.7, 121.1, 116.5, 116.1, 105.9, 89.0, 33.6, 10.5; IR (ν_{max}, cm^{-1}) (KBr): 3412, 1649, 1519, 1352, 1212; HRMS (ESI) *m/z* calcd. for $C_{20}H_{13}N_3O_5$: 375.0863, HRMS (ESI) found: 376.0951 $[M+H]^+$.

8-methyl-7-phenyl-7,11-dihydro-6*H*-chromeno[3,4-*e*]isoxazolo[5,4-*b*]pyridin-6-one (4i)

White solid; Obs. M.p.: 255-257ºC; Yield: Method A = 90% , Method B = 93%; ^{1}H NMR (400 MHz, DMSO-d_6) δ_H: 11.44 (s, 1H, N-H), 8.16 (d, 1H, J = 8.39 Hz, Ar-H), 7.63-7.59 (t, 1H, Ar-H), 7.42-7.38 (t, 1H, Ar-H), 7.34 (d, 1H, J = 8.39 Hz, Ar-H), 7.24-7.23 (m, 4H, Ar-H), 7.16-7.12 (m, 1H, Ar-H), 5.12 (s, 1H,-CH), 1.87 (s, 3H, Ar-H); ^{13}C NMR (100 MHz, DMSO-d_6) δ_C: 160.3, 159.1, 158.5, 152.2, 145.4, 143.1, 132.3, 128.3, 127.9, 126.7, 124.2,

123.1, 116.8, 113.3, 100.5, 95.2, 37.3, 9.8; IR (ν_{max}, cm^{-1}) (KBr): 3201, 1680, 1510, 1262, 1061; HRMS (ESI) *m/z* calcd. for $C_{20}H_{14}N_2O_3$: 330.1009, HRMS (ESI) found: 331.1081 $[M+H]^+$.

7-(3-Chlorophenyl)-8-methyl-7,11-dihydro-6*H*-chromeno[3,4-*e*]isoxazolo[5,4-*b*]pyridin-6-one (4j)

White solid; Obs. M.p.: 246-248°C; Yield: Method A = 92% , Method B = 90%; ^1H NMR (400 MHz, DMSO-d_6) δ_H: 11.47 (s, 1H, N-H), 8.14 (d, 1H, *J* = 7.63, Ar-H), 7.60-7.57 (t, 1H, Ar-H), 7.38-7.35 (t, 1H, Ar-H), 7.32-7.30 (m, 2H, Ar-H), 7.25 (d, 1H, *J* = 7.63, Ar-H), 7.20-7.18 (m, 2H, Ar-H), 5.12 (s, 1H, -CH), 1.85 (s, 3H, $-CH_3$); ^{13}C NMR (100 MHz, DMSO-d_6) δ_C: 160.3, 159.3, 158.5, 152.2, 147.7, 143.5, 132.9, 132.4, 130.3, 127.7, 126.8, 124.3, 123.2, 116.9, 99.9, 94.7, 37.2, 9.9; IR (ν_{max}, cm^{-1}) (KBr): 3251, 1682, 1520, 1260, 1049; HRMS (ESI) *m/z* calcd. for $C_{20}H_{13}ClN_2O_3$: 364.0617, HRMS (ESI) found: 365.0688 $[M+H]^+$.

8-Methyl-7-(3,4,5-trimethoxyphenyl)-7,11-dihydro-6*H*-chromeno[3,4-*e*]isoxazolo[5,4-*b*]pyridin-6-one (4k)

White solid; Obs. M.p.: 255-257°C; Yield: Method A = 95% , Method B = 93%; ^1H NMR (400 MHz, DMSO-d_6) δ_H: 11.40 (s, 1H, -NH), 8.14 (d, 1H, *J* = 6.87 Hz, Ar-H), 7.61 (s, 1H, Ar-H), 7.39-7.34 (m, 2H, Ar-H), 6.49 (s, 2H, Ar-H), 5.10 (s, 1H, -CH), 3.63 (s, 6H, 2-OMe), 3.56 (s, 3H, -OMe), 1.95 (s, 3H, $-CH_3$); ^{13}C NMR (100 MHz, DMSO-d_6) δ_C: 160.2, 159.5, 158.3, 153.3, 152.2, 150.6, 148.2, 147.5, 143.9, 134.7, 132.2, 124.2, 122.9, 116.8, 115.9, 113.4, 112.6, 111.5, 99.9, 94.7, 56.2, 55.2, 31.9, 9.8; IR (ν_{max}, cm^{-1}) (KBr): 3227, 1681, 1519, 1126; HRMS (ESI) *m/z* calcd. for $C_{23}H_{20}N_2O_6$: 420.1321, HRMS (ESI) found: 421.1402 $[M+H]^+$.

7-(3-Bromophenyl)-8-methyl-7,11-dihydro-6*H*-chromeno[3,4-*e*]isoxazolo[5,4-*b*]pyridin-6-one (4l)

White solid; Obs. M.p.: 239-241°C; Yield: Method A = 94% , Method B = 92%; ^{1}H NMR (400 MHz, DMSO-*d*$_6$) δ_H: 11.47 (s, 1H, -NH), 8.15 (d, *J* = 7.63 Hz, 1H, Ar-H), 7.62-7.58 (t, 1H, Ar-H), 7.41-7.37 (t, 1H, Ar-H), 7.33 (d, *J* = 8.39 Hz, 1H, Ar-H), 7.27 (s, 4H, Ar-H), 5.13 (s, 1H, -CH), 1.86 (s, 3H, -CH_3); ^{13}C NMR (100 MHz, DMSO-*d*$_6$) δ_C: 160.3, 159.2, 158.4, 152.2, 147.9, 143.5, 132.2, 130.5, 129.6, 127.2, 125.4, 124.2, 123.2, 121.6, 116.8, 113.3, 99.8, 94.6, 37.1, 9.9; IR (ν_{max}, cm^{-1}) (KBr): 3252, 1681, 1519, 1048; HRMS (ESI) *m/z* calcd. for $C_{20}H_{13}BrN_2O_3$: 408.0110, HRMS (ESI) found: 409.0167 $[M+H]^+$.

7-(2,5-Dimethoxyphenyl)-8-methyl-7,11-dihydro-6*H*-chromeno[3,4-*e*]isoxazolo[5,4-*b*]pyridin-6-one (4m)

White solid; Obs. M.p.: 246-248°C; Yield: Method A = 93% , Method B = 95%; ^{1}H NMR (400 MHz, DMSO-*d*$_6$) δ_H: 11.34 (s, 1H, N-H), 8.15 (d, 1H, *J* = 7.63 Hz, Ar-H), 7.61-7.59 (t, 1H, Ar-H), 7.41-7.38 (t, 1H, Ar-H), 7.34 (d, 1H, *J* = 7.63 Hz, Ar-H), 6.87-6.85 (m, 1H, Ar-H), 6.71-6.69 (m, 1H, Ar-H), 6.60 (s, 1H, Ar-H), 5.33 (s, 1H, -CH), 3.65 (s, 3H, -OMe), 3.56 (s, 3H, -OMe), 1.87 (s, 3H, -CH_3); ^{13}C NMR (100 MHz, DMSO-*d*$_6$) δ_C: 160.1, 159.4, 158.2, 153.2, 152.1, 150.6, 143.8, 134.6, 132.1, 124.1, 122.9, 116.7, 115.9, 113.3, 112.6, 111.5, 99.9, 94.7, 56.2, 55.2, 31.9, 9.7; IR (ν_{max}, cm^{-1}) (KBr): 3229, 1681, 1520, 1225, 1047; HRMS (ESI) *m/z* calcd. for $C_{22}H_{18}ClN_2O_5$: 390.1216, HRMS (ESI) found: 391.1313 $[M+H]^+$.

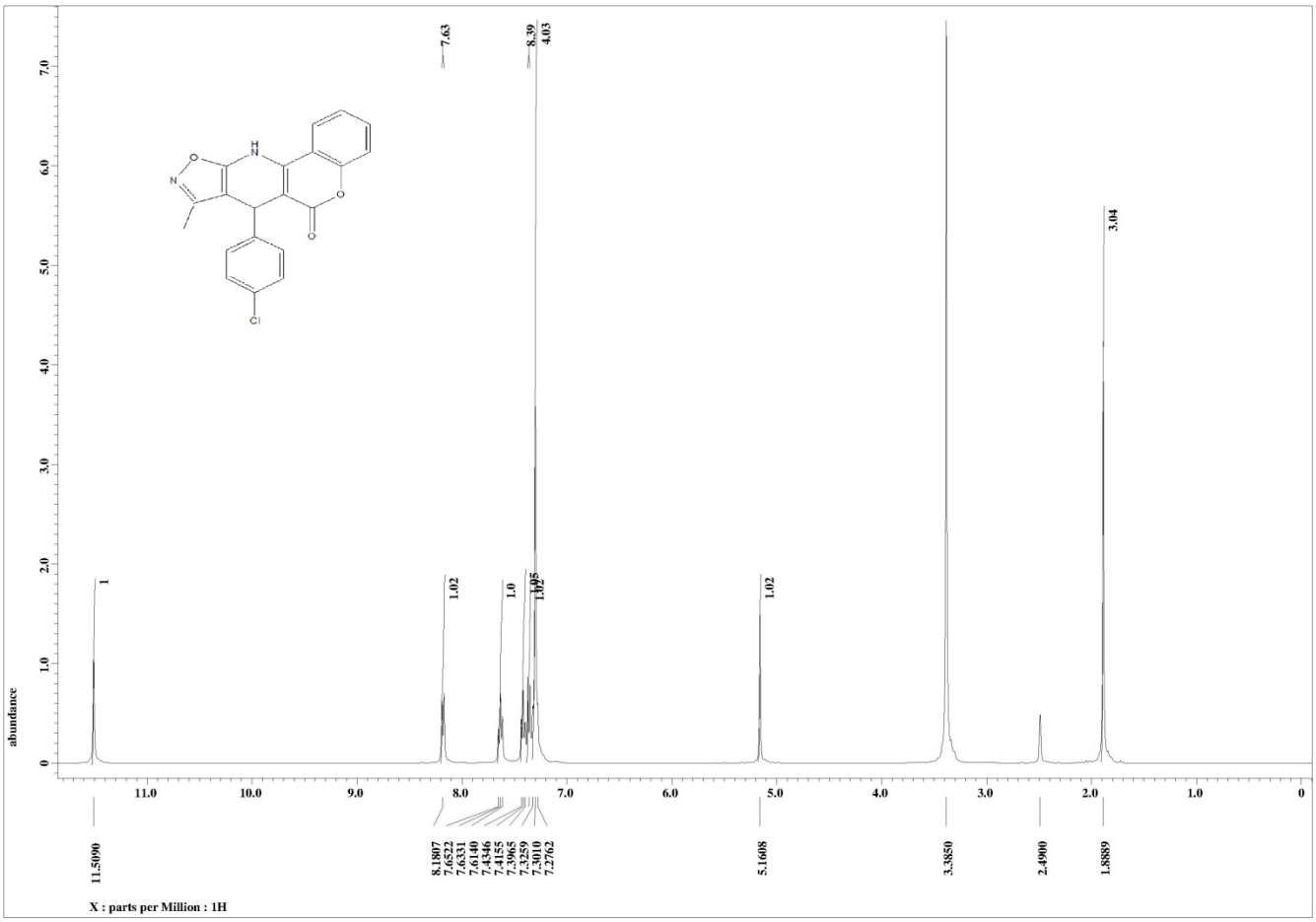
8-Methyl-7-(4-(trifluoromethyl)phenyl)-7,11-dihydro-6*H*-chromeno[3,4-*e*]isoxazolo[5,4-*b*]pyridin-6-one (4n)

White solid; Obs. M.p.: 255-257°C; Yield: Method A = 92% , Method B = 92%; ^{1}H NMR (400 MHz, DMSO-*d*$_6$) δ_H: 11.52 (s, 1H, N-H), 8.16 (d, 1H, *J* = 7.63 Hz, Ar-H), 7.60-7.46 (q, 5H, Ar-H), 7.39-7.36 (t, 1H, Ar-H), 7.31 (d, 1H, *J* = 8.39 Hz, Ar-H), 5.21 (s, 1H, -CH), 1.84 (s, 3H, -CH_3); ^{13}C NMR (100 MHz, DMSO-*d*$_6$) δ_C: 160.2, 159.3, 158.4, 152.2, 149.7, 143.5, 132.4, 128.8, (127.8-126.8 q for CF_3), 125.2, 124.2, 123.1, 116.8, 113.2, 99.8, 94.5, 37.4, 9.8; IR (ν_{max}, cm^{-1}) (KBr): 3195, 1681, 1510, 1328, 1111; HRMS (ESI) *m/z* calcd. for $C_{21}H_{13}F_3N_2O_3$: 398.0889, HRMS (ESI) found: 399.0964 $[M+H]^+$.

7-(2-Chlorophenyl)-8-methyl-7,11-dihydro-6*H*-chromeno[3,4-*e*]isoxazolo[5,4-*b*]pyridin-6-one (4o)

White solid; Obs. M.p.: 252-254ºC; Yield: Method A = 90% , Method B = 92%; ^{1}H NMR (400 MHz, DMSO-d_6) δ_H: 11.54 (s, 1H, N-H), 8.19 (s, 1H, Ar-H), 7.65 (s, 1H, Ar-H), 7.37-7.22 (m, 6H, Ar-H), 5.56 (s, 1H, -CH), 1.86 (s, 3H, -CH_3); ^{13}C NMR (100 MHz, DMSO-d_6) δ_C: 160.3, 159.4, 158.4, 152.5, 152.3, 146.3, 143.7, 132.5, 129.3, 124.3, 123.6, 123.2, 116.8, 113.2, 99.4, 94.2, 37.6, 37.5, 9.9; IR (ν_{max}, cm^{-1}) (KBr): 3240, 1682, 1510, 1262, 1061; HRMS (ESI) *m/z* calcd. for $C_{20}H_{13}ClN_2O_3$: 364.0633, HRMS (ESI) found: 365.0706 $[M+H]^+$.

^{1}H NMR of 4a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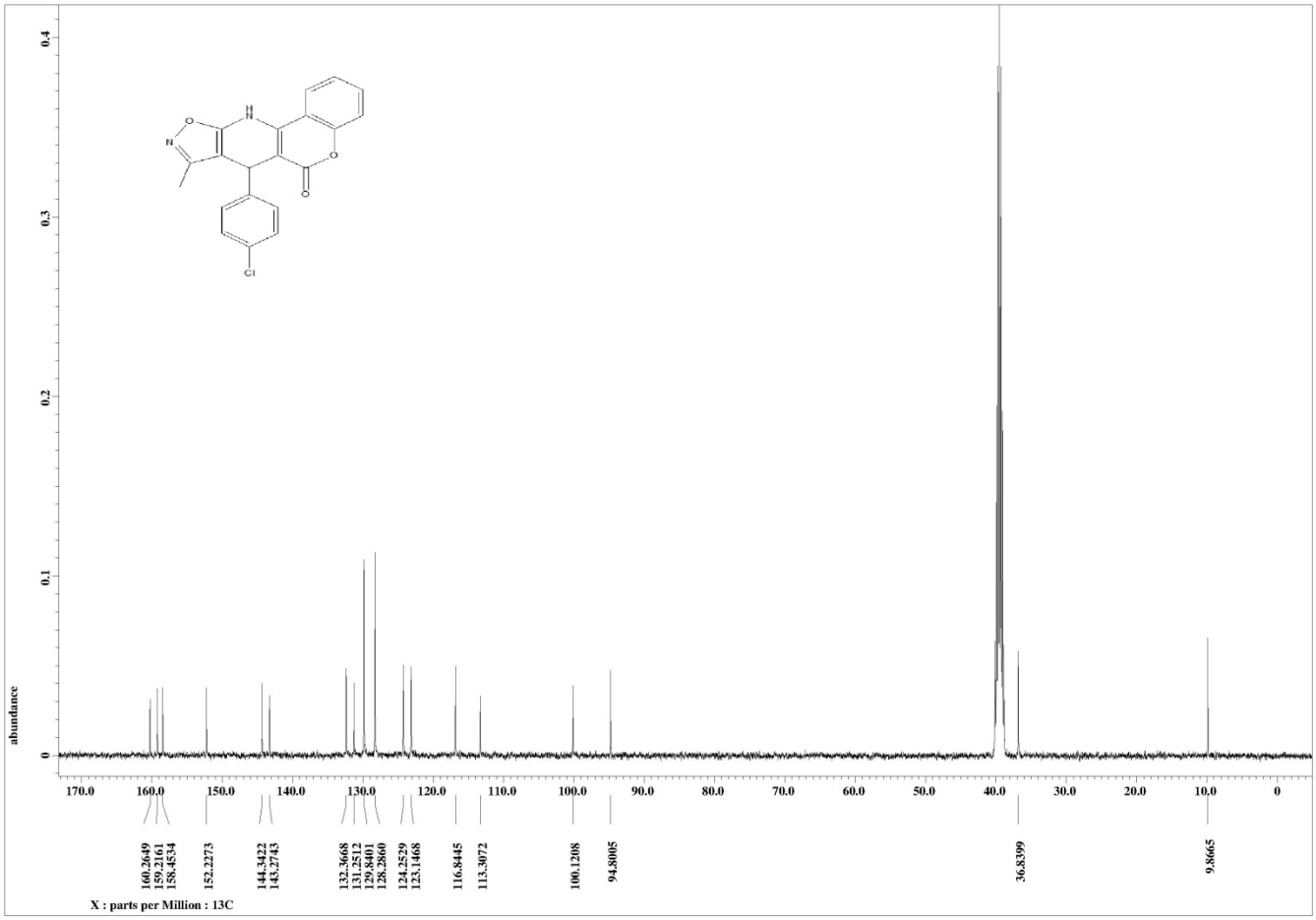
^{13}C NMR of 4a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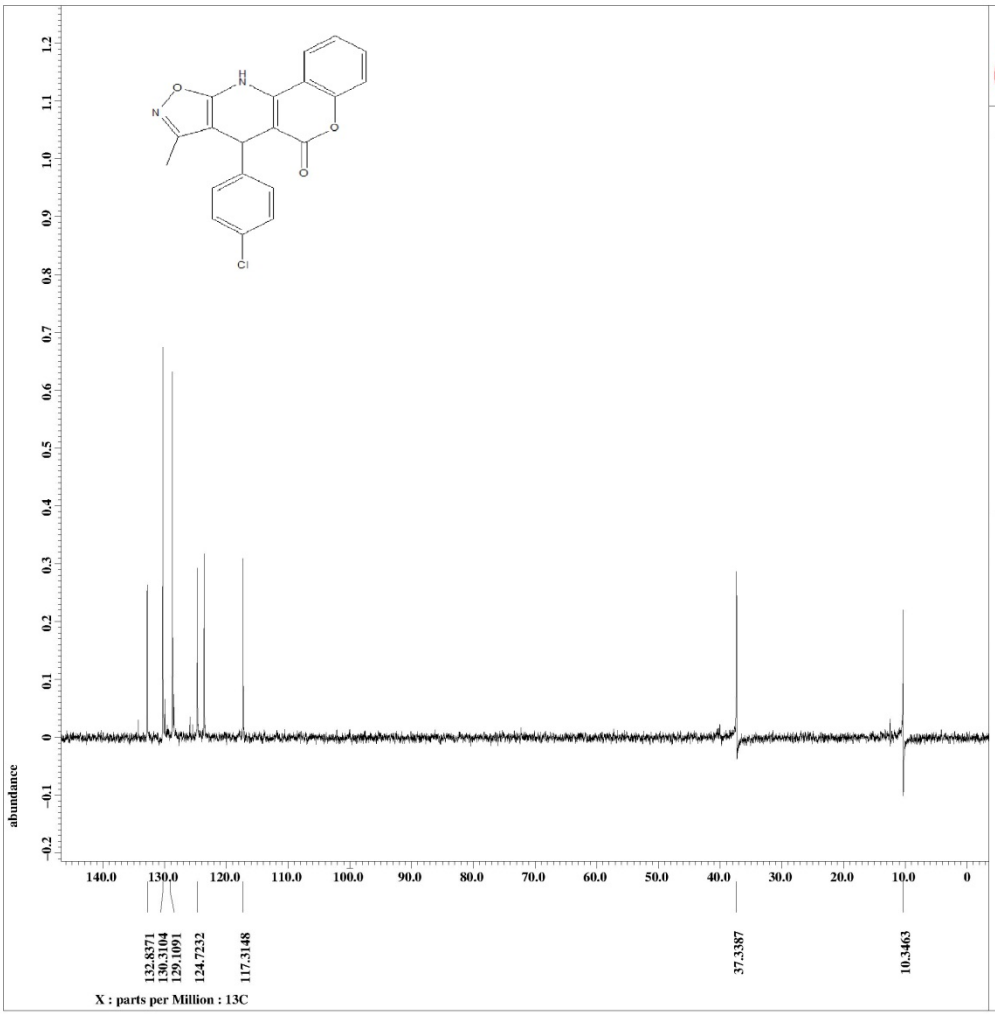


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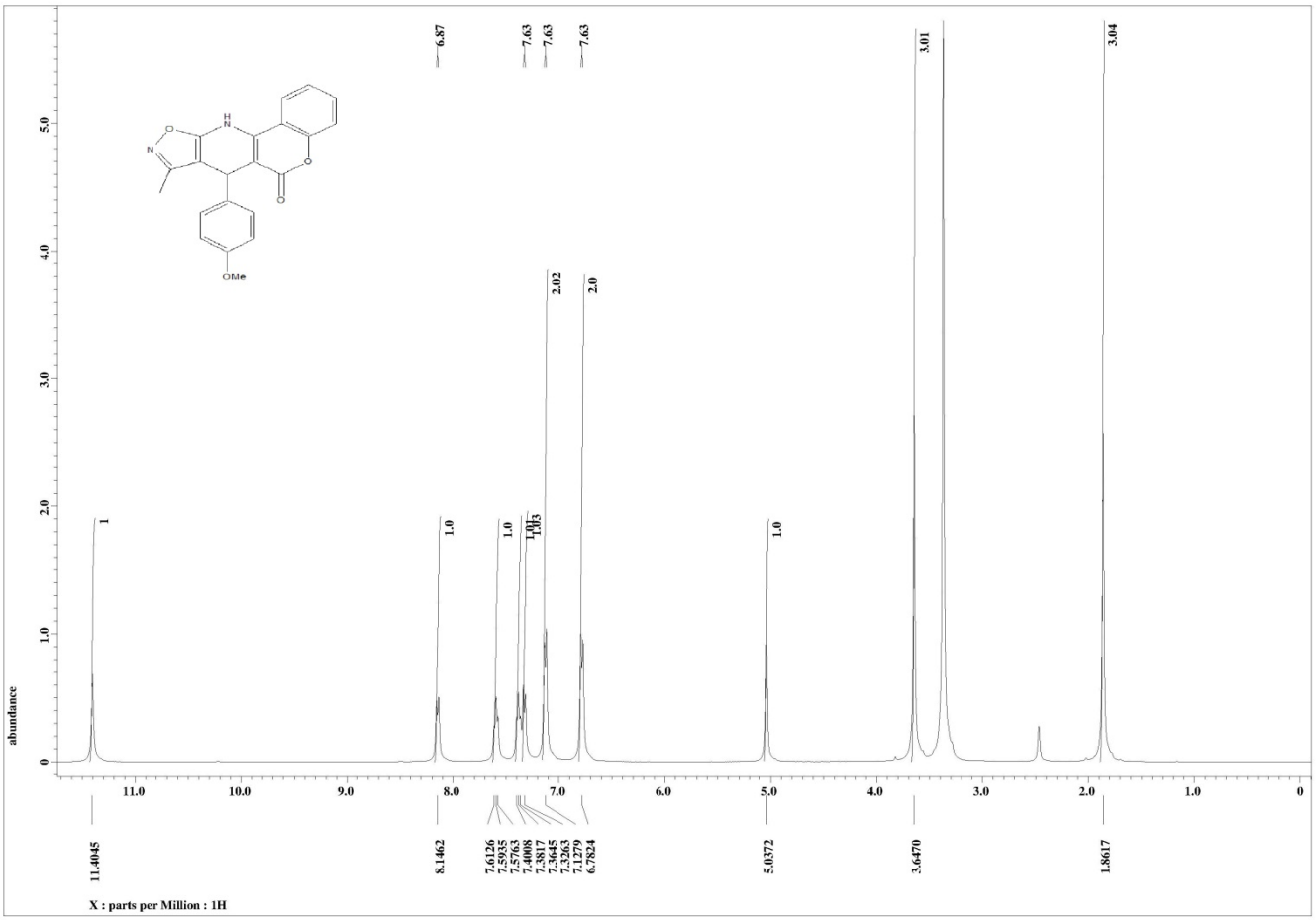
^{1}H NMR of 4b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7.63
7.63
3.01
3.04
2.02
2.0
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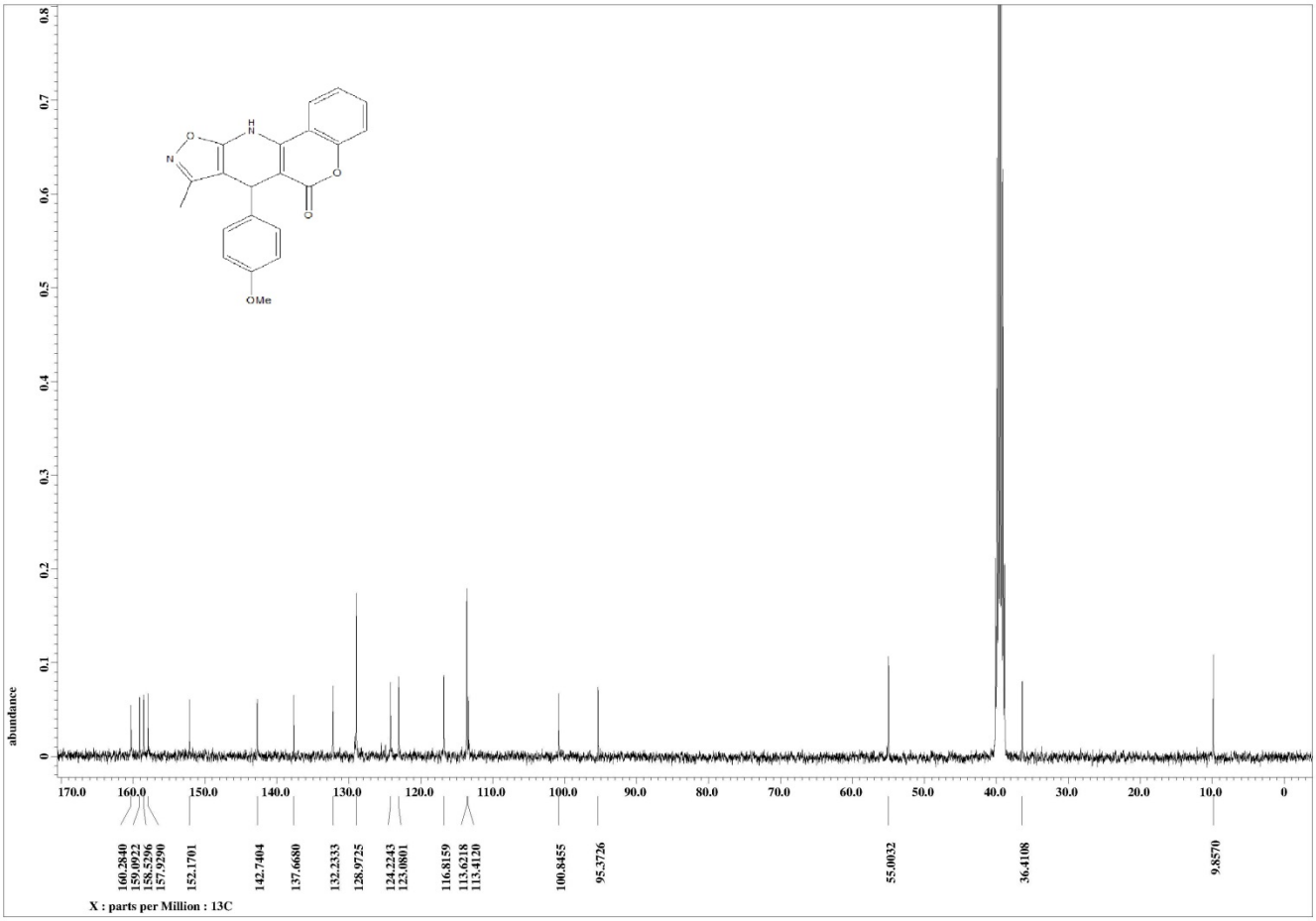
OMe
abundance
11.4045
8.1462
7.6126
7.5935
7.5763
7.4008
7.3817
7.3645
7.3263
7.1279
6.7824
5.0372
3.6470
1.8617
X : parts per Million : 1H

^{13}C NMR of 4b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0.2
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0.4
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0.6
0.7
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170.0
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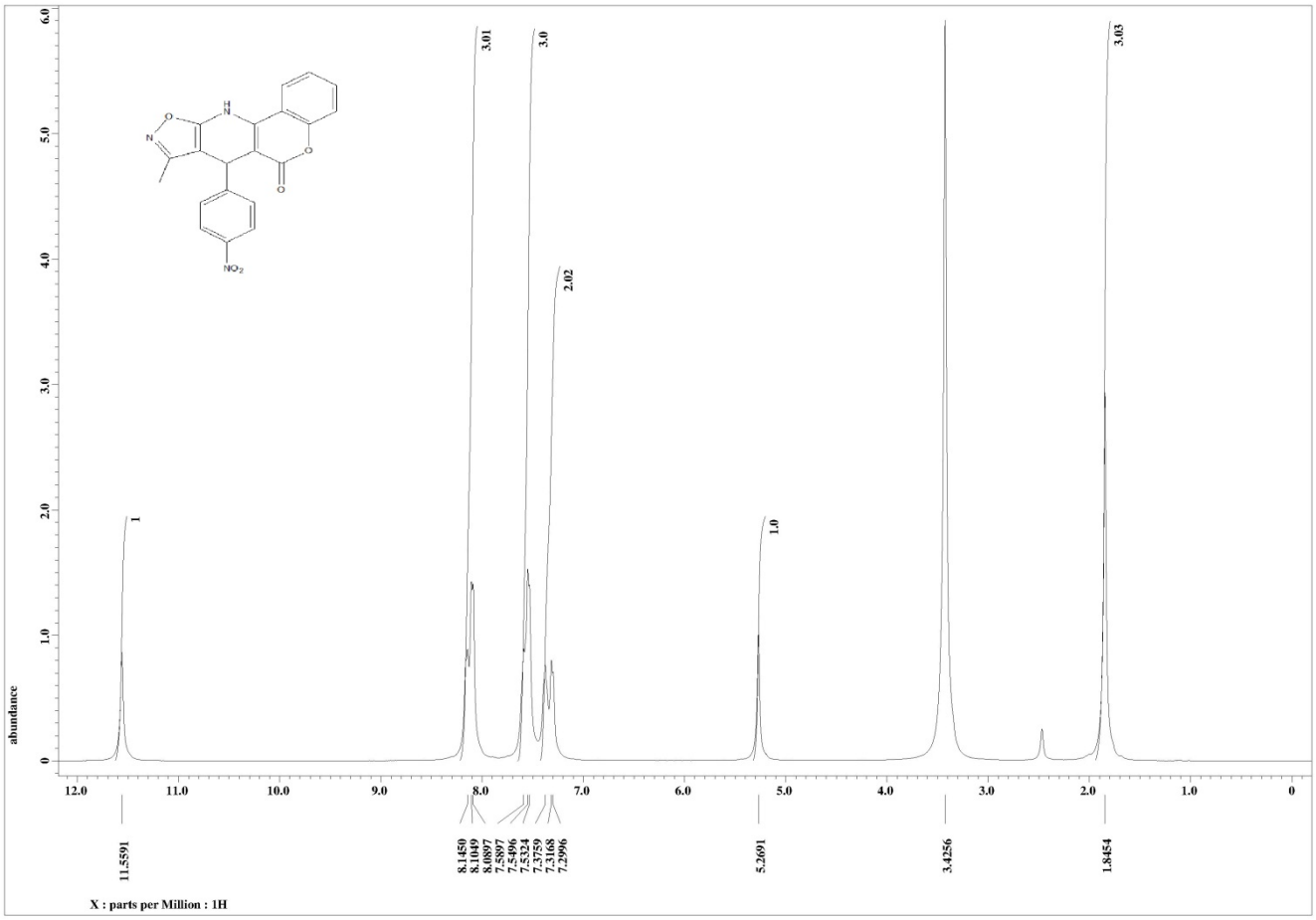
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
160.2840
159.0922
158.5296
157.9290
152.1701
142.7404
137.6680
132.2333
128.9725
124.2243
123.0801
116.8159
113.6218
113.4120
100.8455
95.3726
55.0032
36.4108
9.8570
X : parts per Million : 13C
O
N
H
O
O
OMe

^{1}H NMR of 4c


abundance
X : parts per Million : 1H
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8.1049
8.0897
7.5897
7.5496
7.5324
7.3759
7.3168
7.2996
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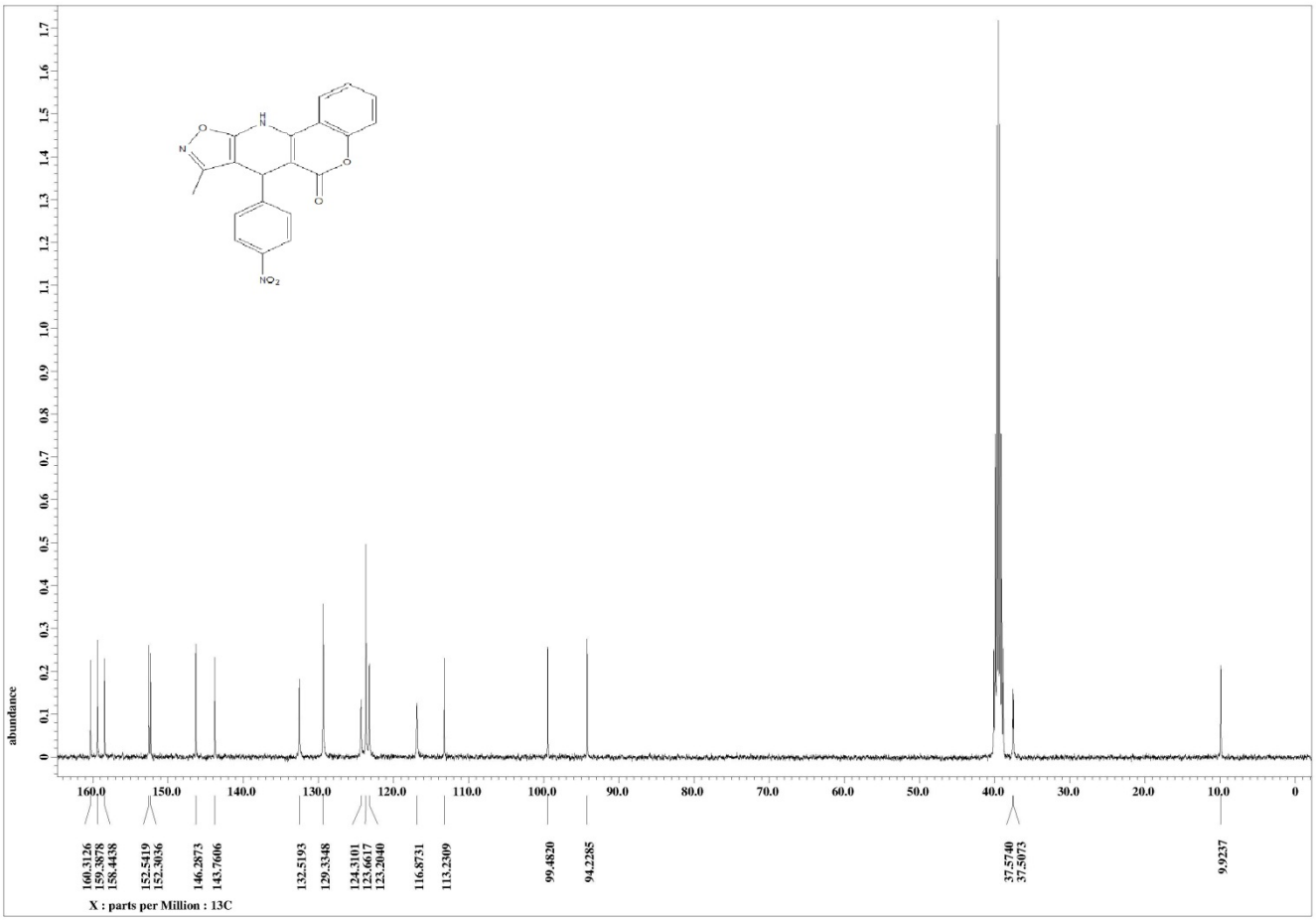
^{13}C NMR of 4c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159.3878
158.4438
152.5419
152.3036
146.2873
143.7606
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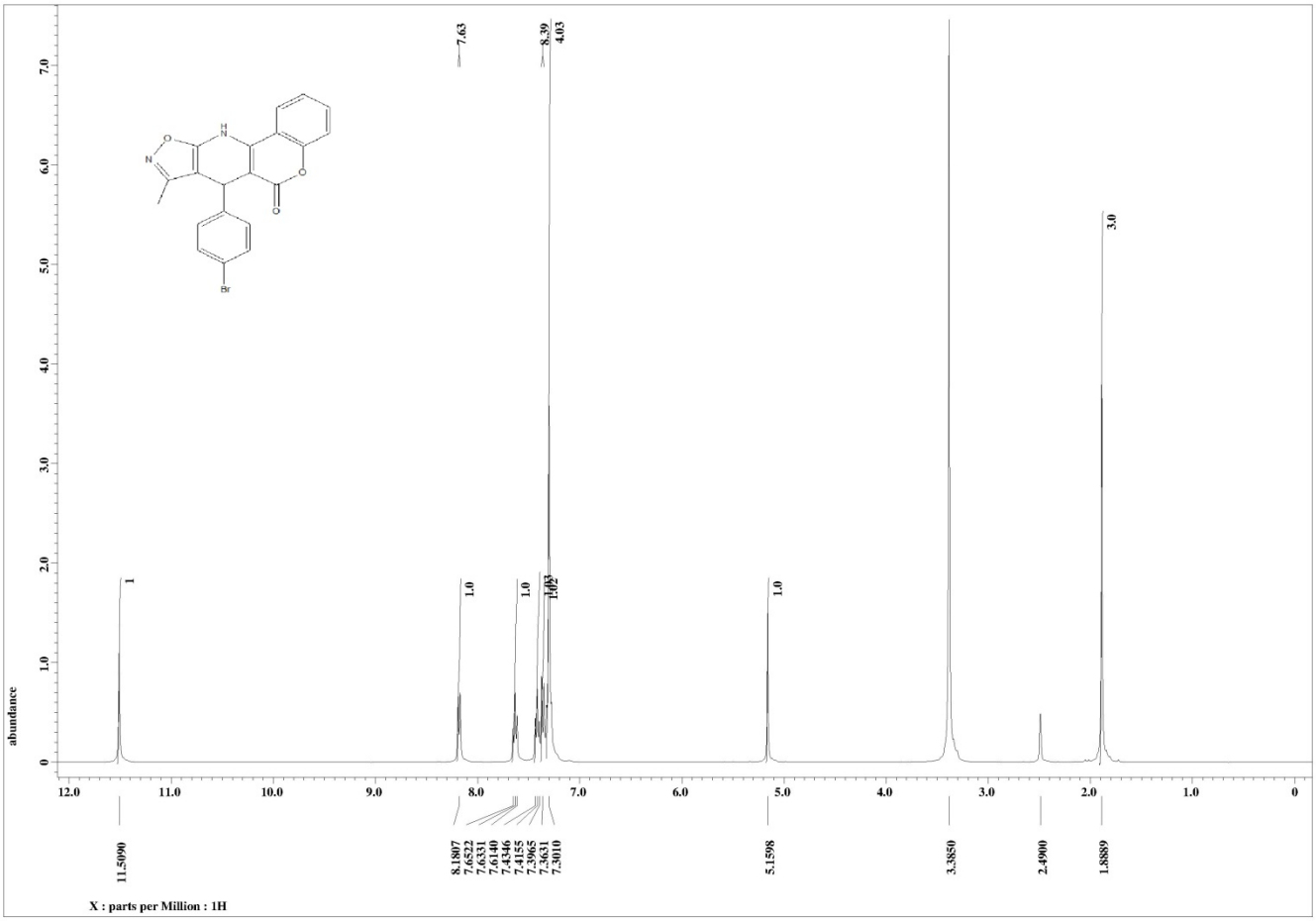
129.3348
124.3101
123.6617
123.2040
116.8731
113.2309
99.4820
94.2285
37.5740
37.5073
9.9237
X : parts per Million : 13C

^{1}H NMR of 4d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7.3774
7.3583
7.3010
5.1598
3.3850
2.4900
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7.63
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4.03
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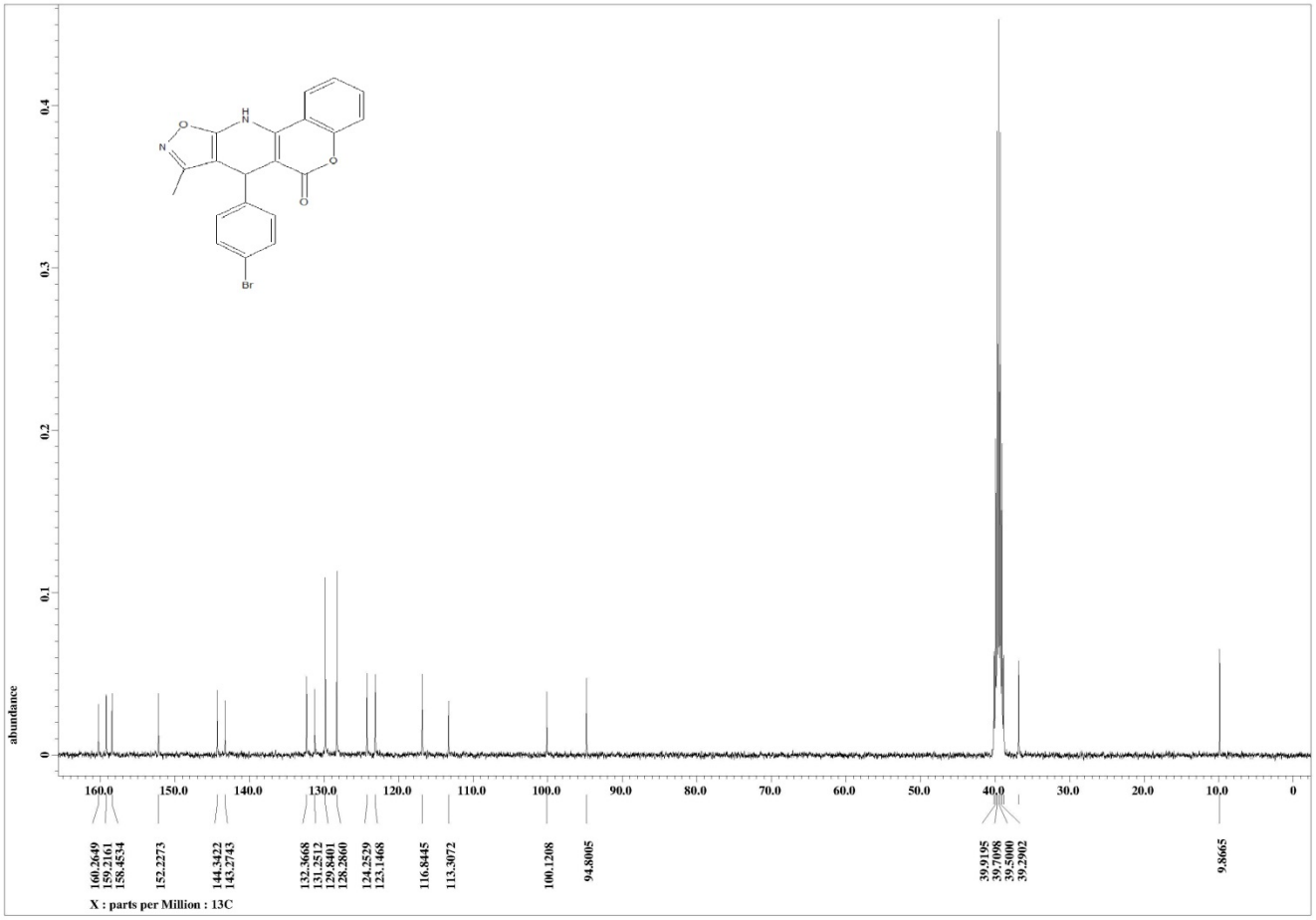
1.0
1.0
Br

^{13}C NMR of 4d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0.4
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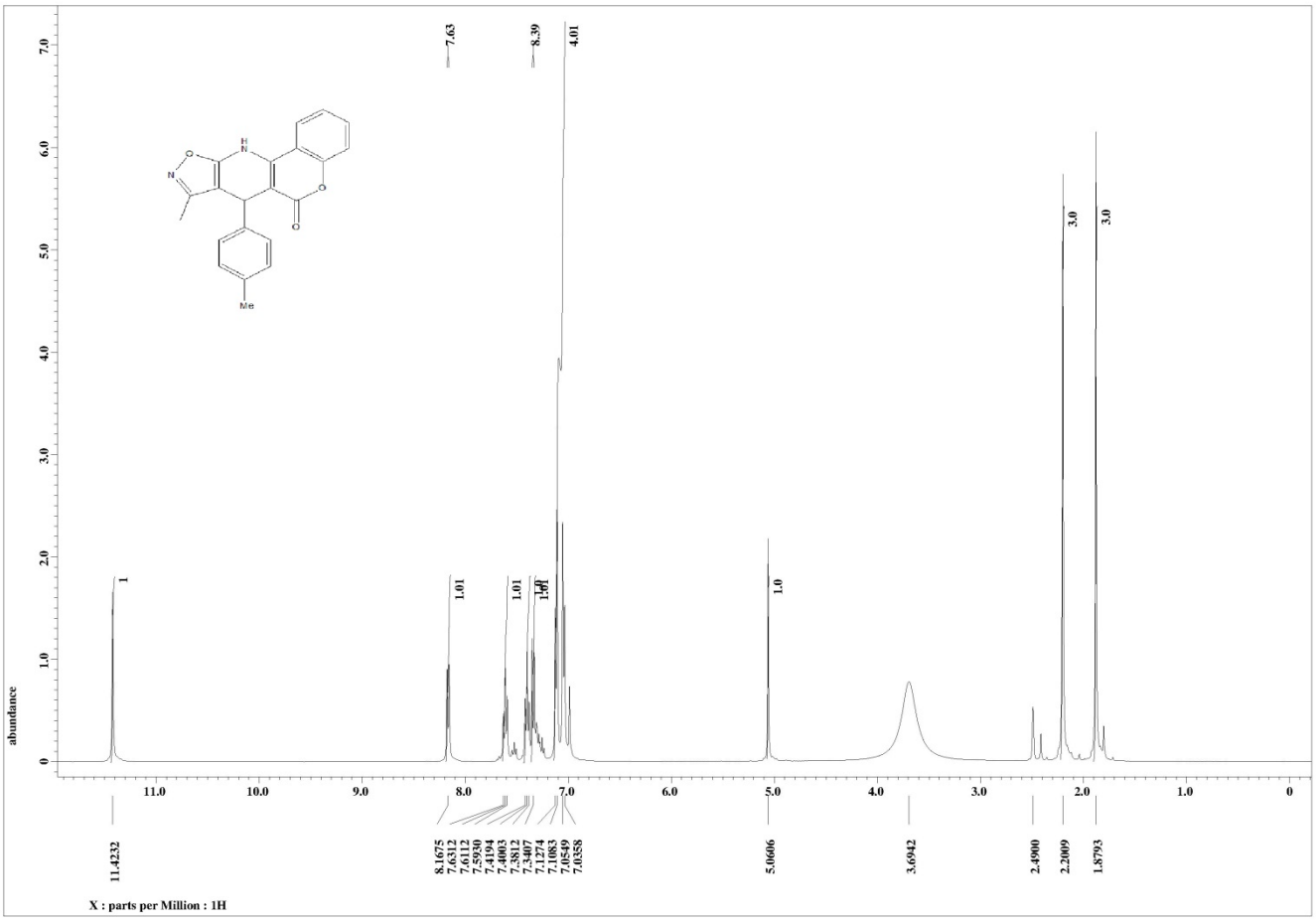
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
160.2649
159.2161
158.4534
152.2273
144.3422
143.2743
132.3668
131.2512
129.8401
128.2860
124.2539
123.1468
116.8445
113.3072
100.1208
94.8005
39.9195
39.7098
39.5000
39.2902
9.8665
X : parts per Million : 13C
O
N
H
N
O
O
O
Br

^{1}H NMR of 4e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7.4003
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7.3407
7.1274
7.1083
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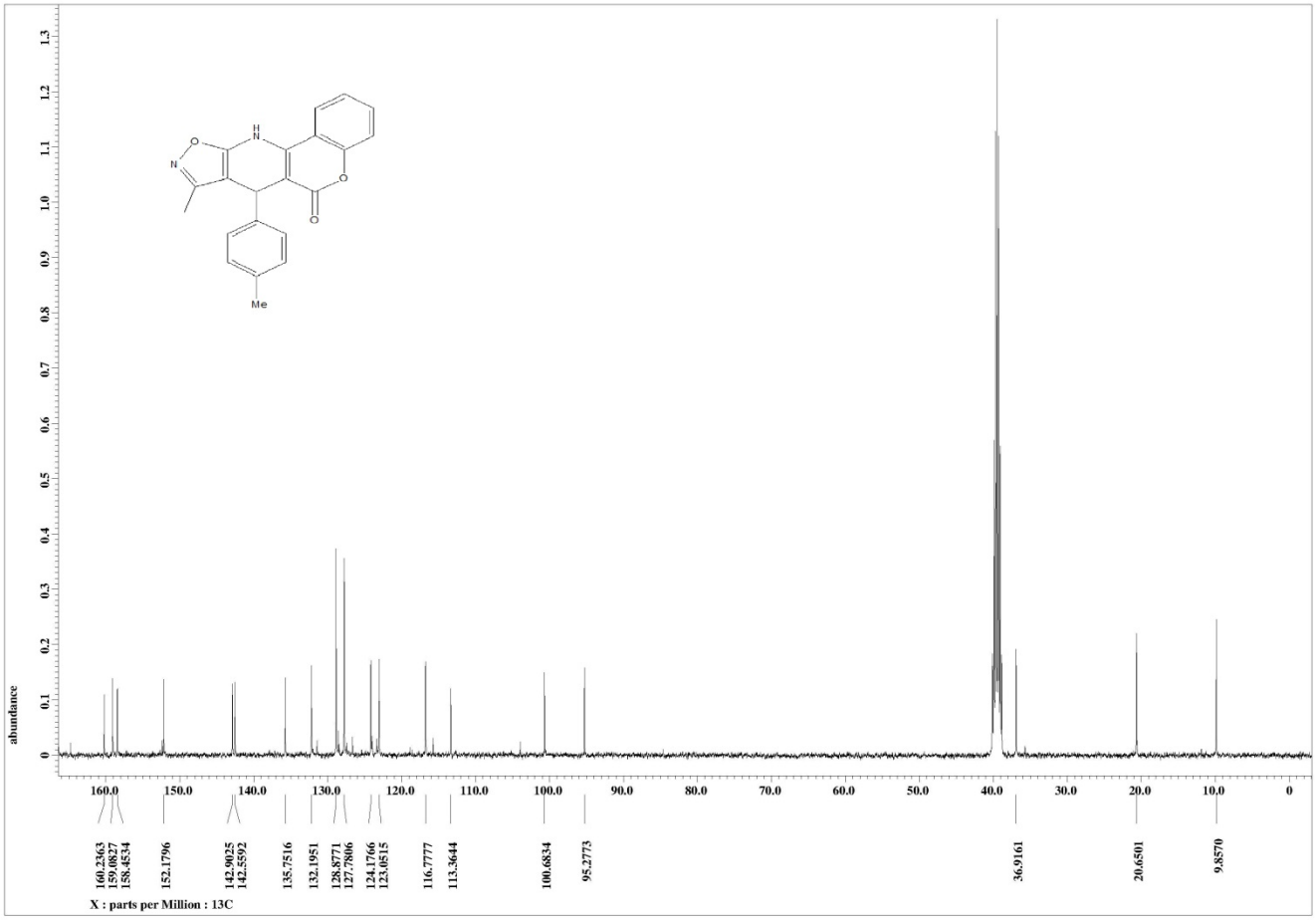
7.0358
5.0606
3.6942
2.4900
2.2009
1.8793
Me

^{13}C NMR of 4e


abundance
Me
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142.5592
135.7516
132.1951
128.8771
127.7806
124.1766
123.0515
116.7777
113.3644
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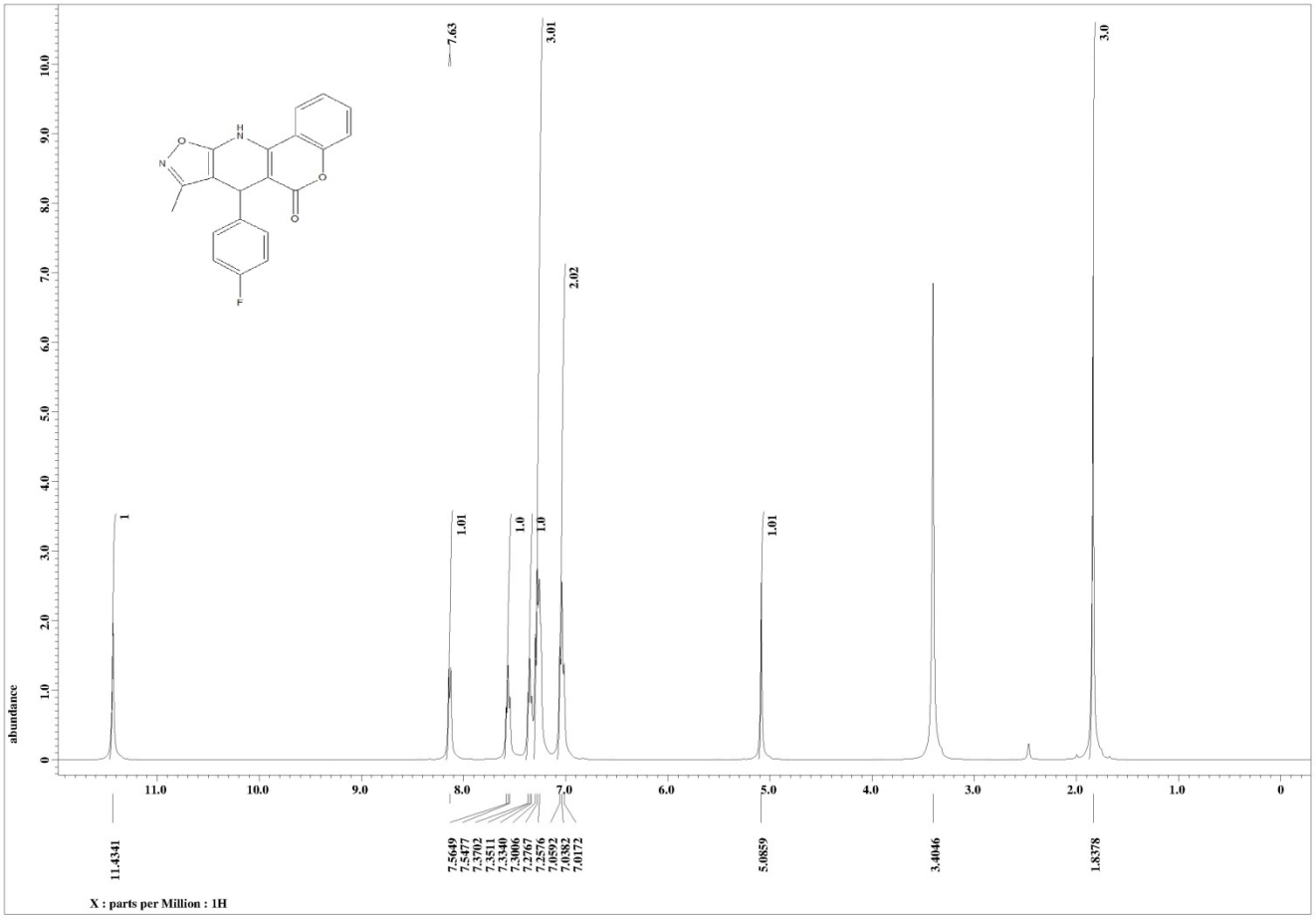
95.2773
36.9161
20.6501
9.8570
X : parts per Million : 13C

^{1}H NMR of 4f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1.01
1.0
1.0
1.01
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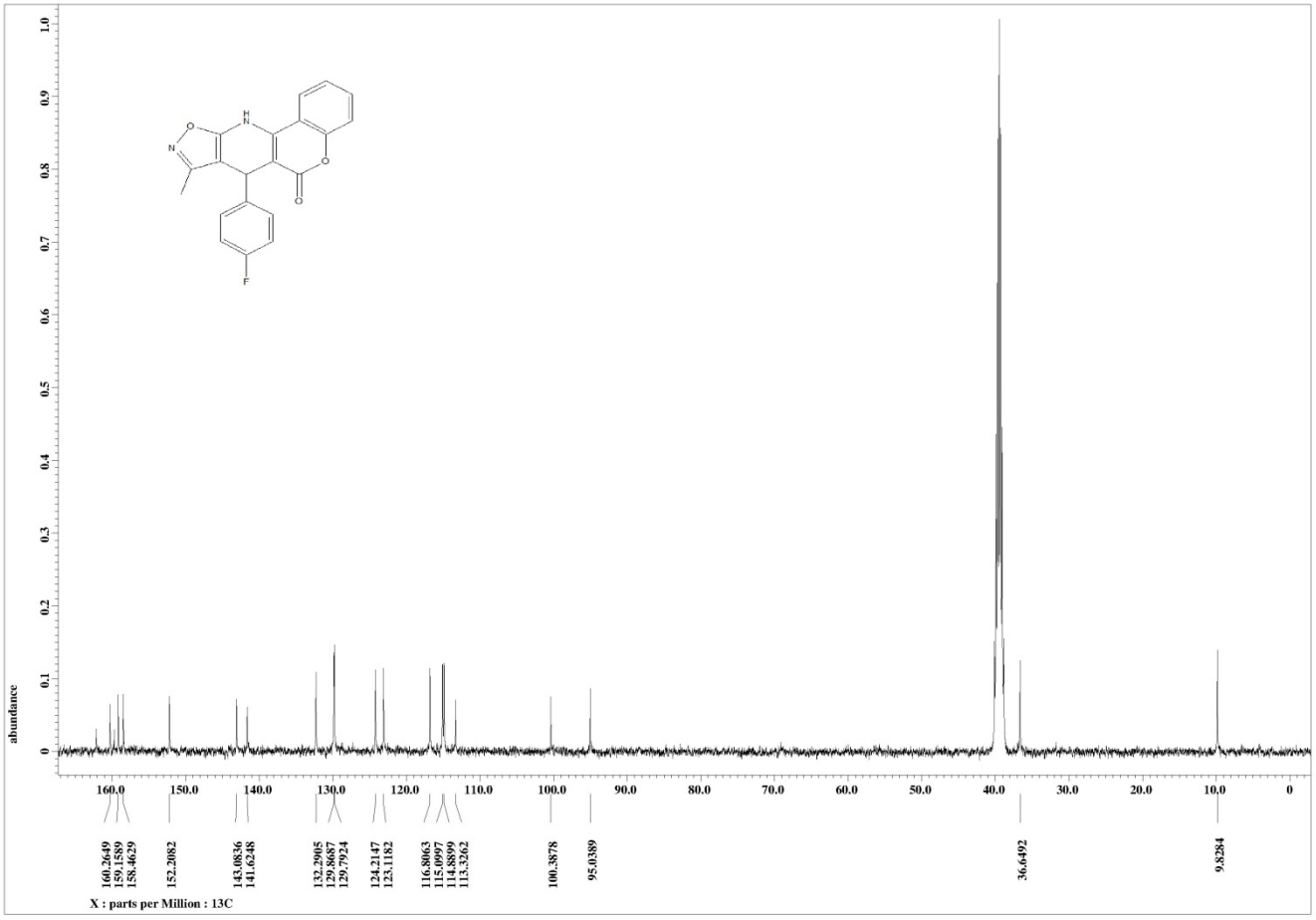
7.3702
7.3511
7.3340
7.3006
7.2767
7.2576
7.0592
7.0382
7.0172
5.0859
3.4046
1.8378
X : parts per Million : 1H

^{13}C NMR of 4f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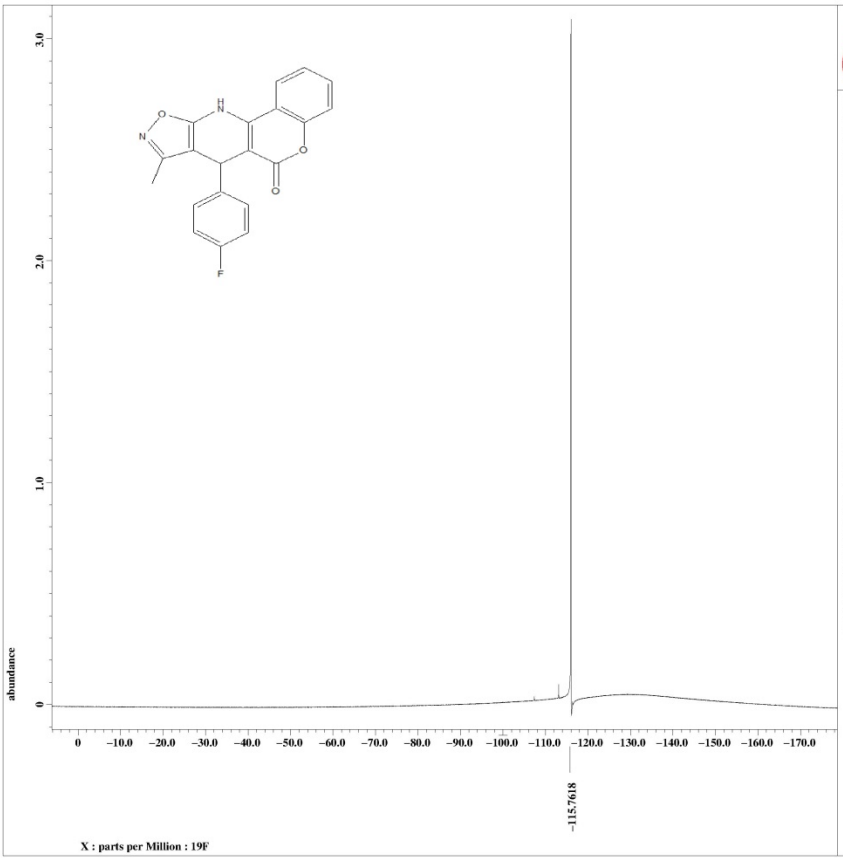
159.1589
158.4629
152.2082
143.0836
141.6348
132.2905
129.8687
129.7924
124.2147
123.1182
116.8063
115.0997
114.8999
113.3262
100.3878
95.0389
36.6492
9.8284
X : parts per Million : 13C

^{19}F NMR of 4f

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Revision_time     = 20-NOV-2015 13:02:31
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Tri_offset        = 5[ppm]
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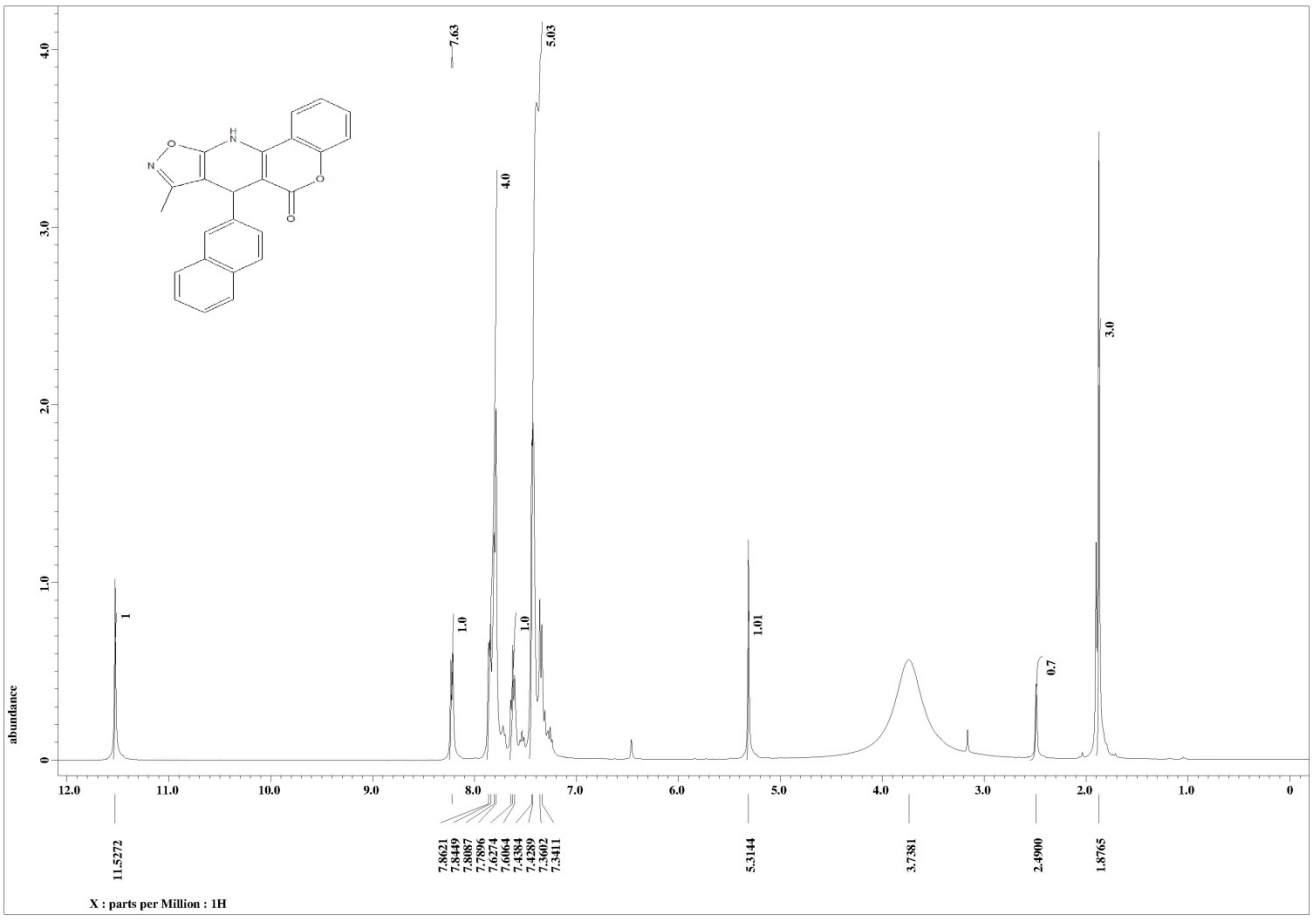
^{1}H NMR of 4g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X : parts per Million : 1H
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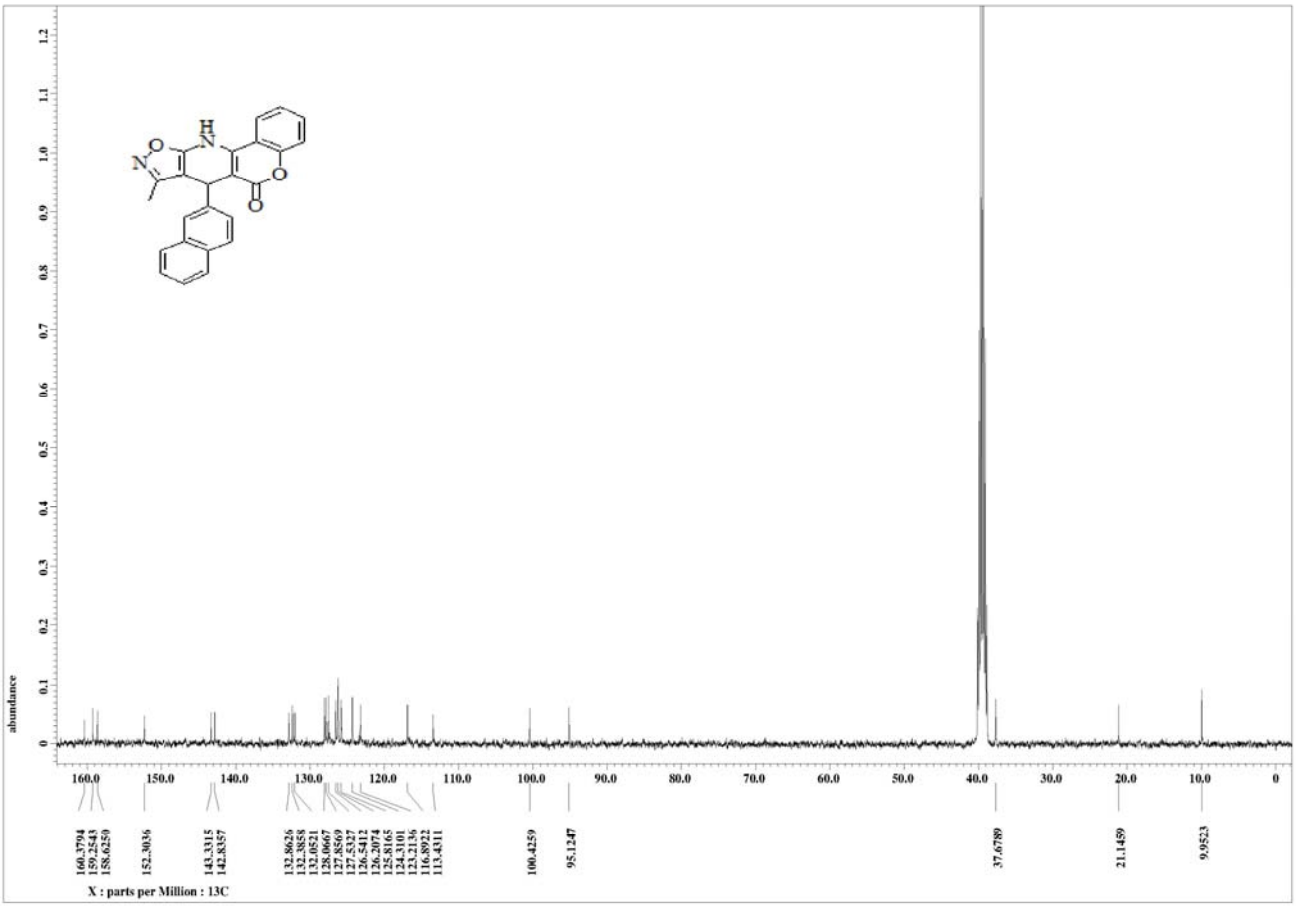
1.8765

^{13}C NMR of 4g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143.3315
142.8357
132.8626
132.3858
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127.8569
127.5327
126.5412
126.2074
125.8165
124.3101
123.2136
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100.4259
95.1247
37.6789
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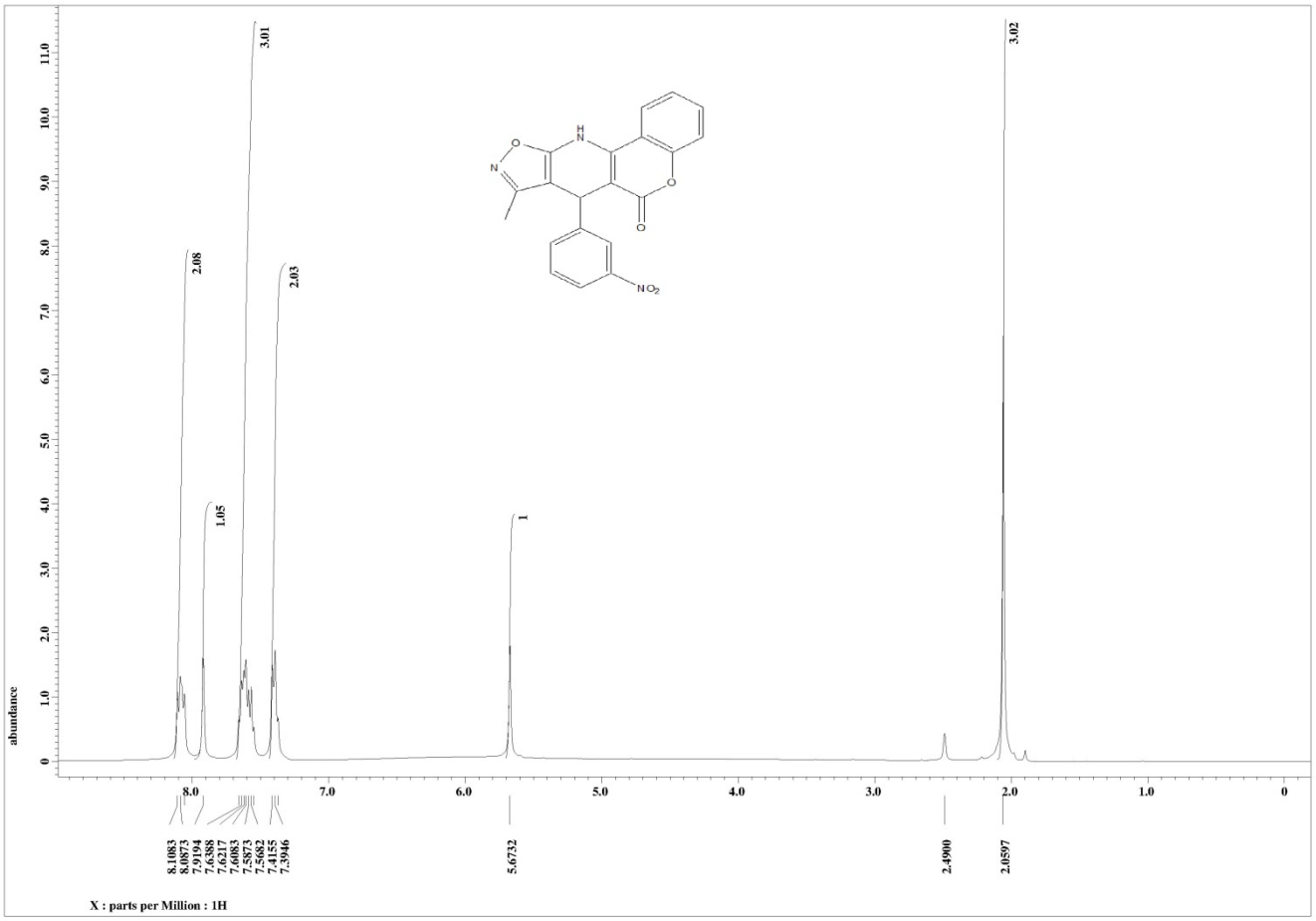
9.9523
X : parts per Million : 13C

^{1}H NMR of 4h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2.03
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3.02
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7.6083
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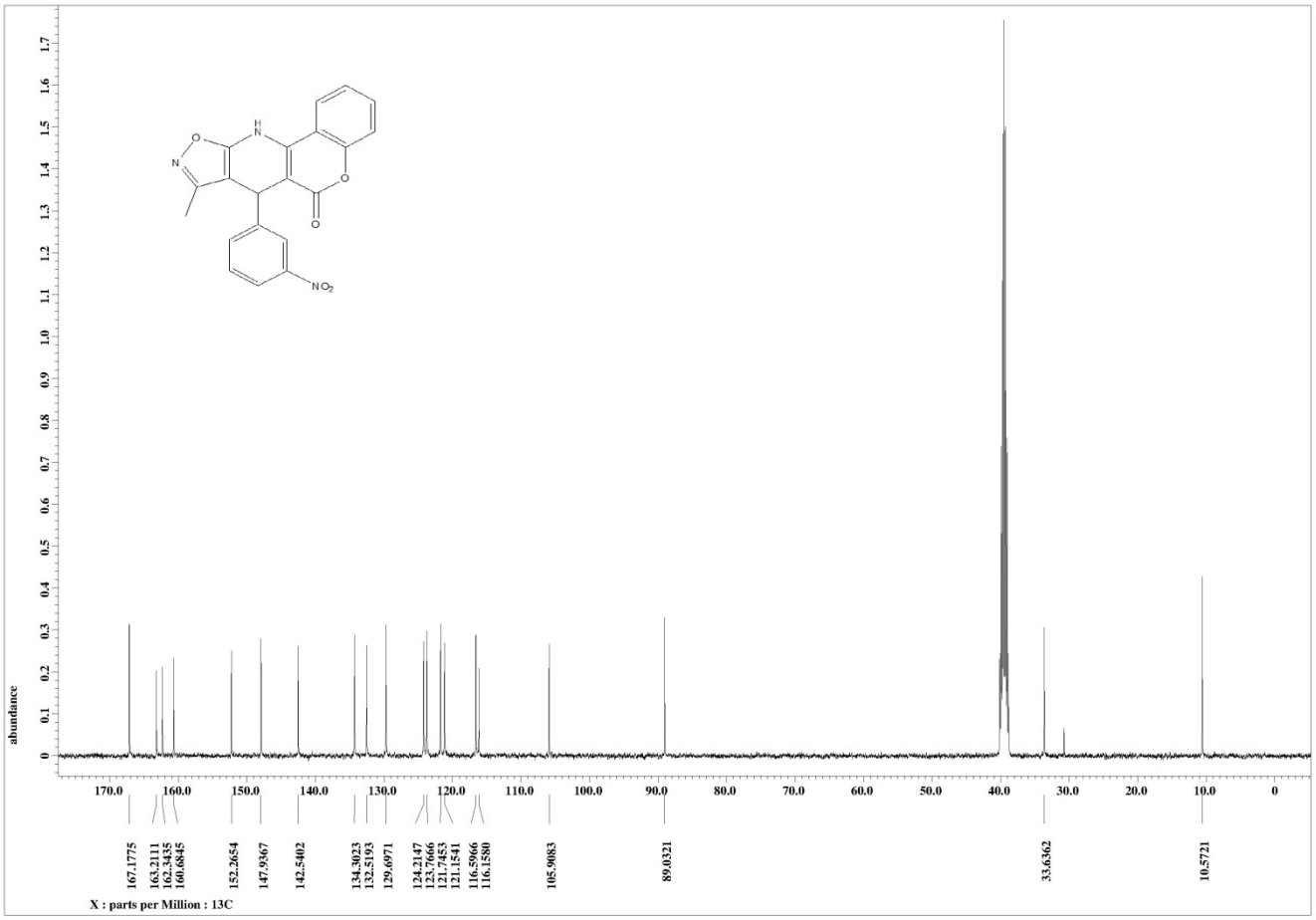
X : parts per Million : 1H

^{13}C NMR of 4h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160.6845
152.2654
147.9367
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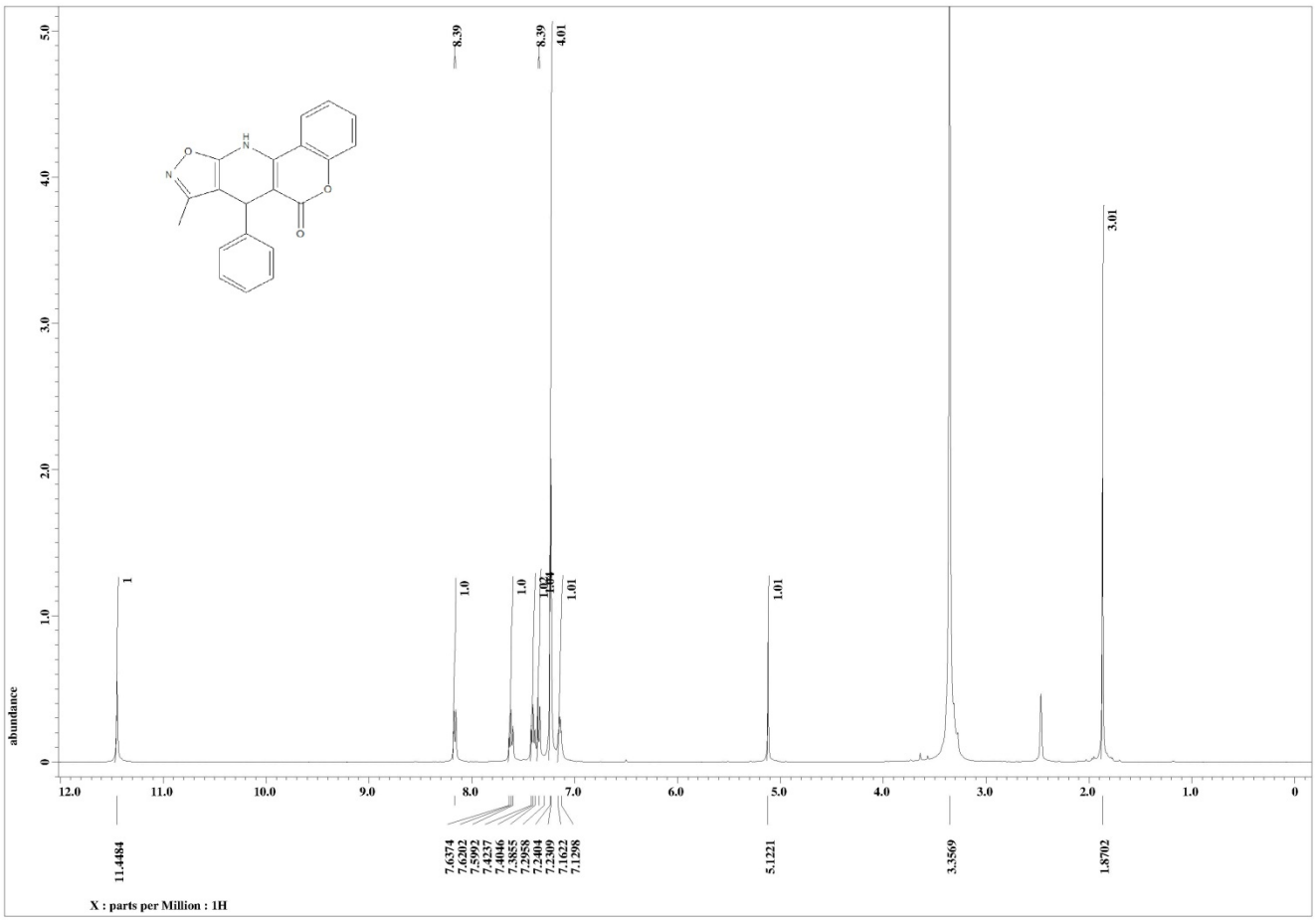
134.3023
132.5193
129.6971
124.2147
123.7666
121.7453
121.1541
116.5966
116.1580
105.9083
89.0321
33.6362
10.5721
X : parts per Million : 13C

^{1}H NMR of 4i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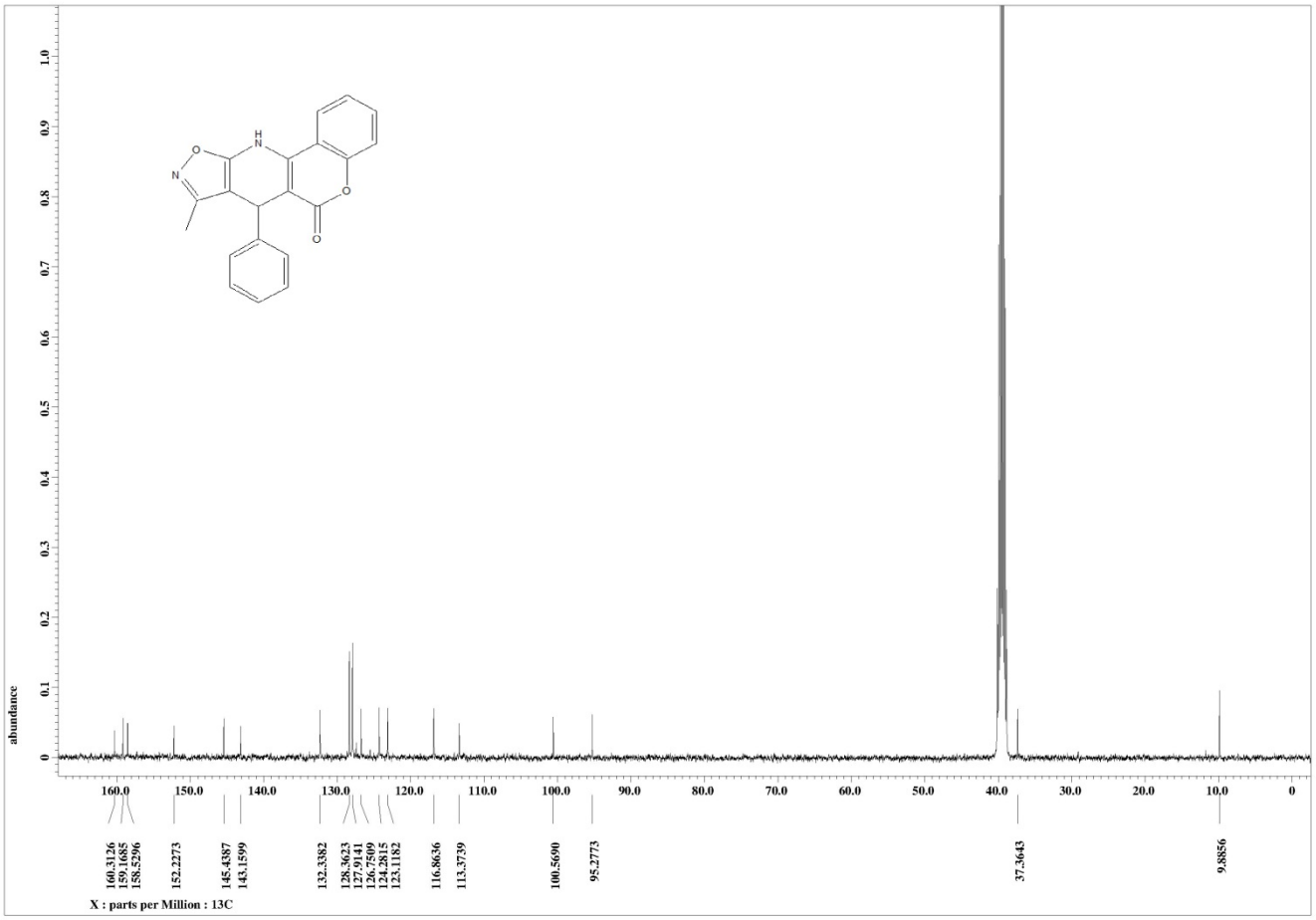
5.1221
3.3569
1.8702

^{13}C NMR of 4i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124.2815
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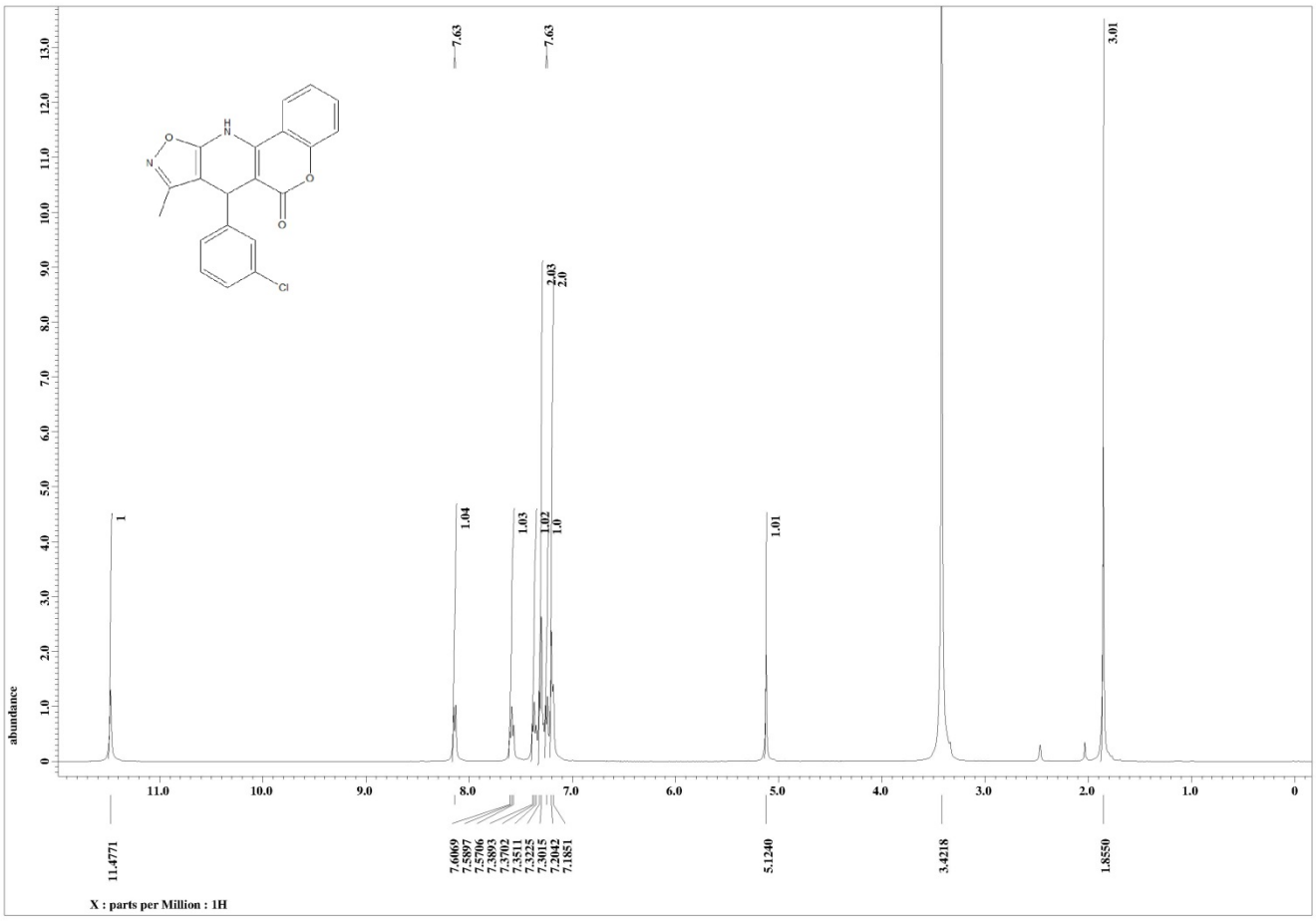
^{1}H NMR of 4j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X : parts per Million : 1H
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7.3015
7.2042
7.1851
5.1240
3.4218
1.8550
7.63
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1.03
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1.01
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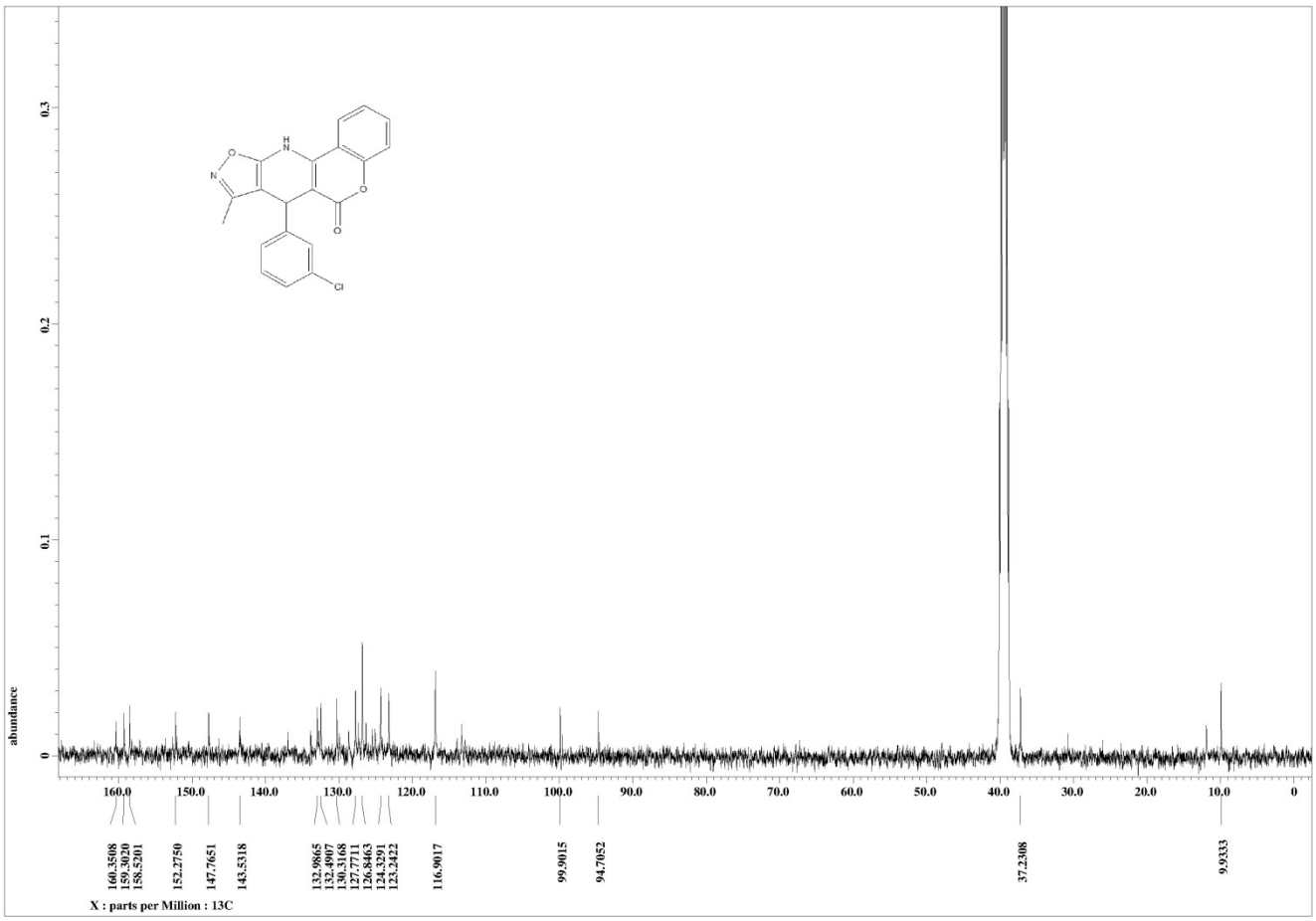
Cl

^{13}C NMR of 4j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90.0
80.0
70.0
60.0
50.0
40.0
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20.0
10.0
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159.3020
158.5201
152.2750
147.7651
143.5318
132.9865
132.4907
130.3168
127.7711
126.8463
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99.9015
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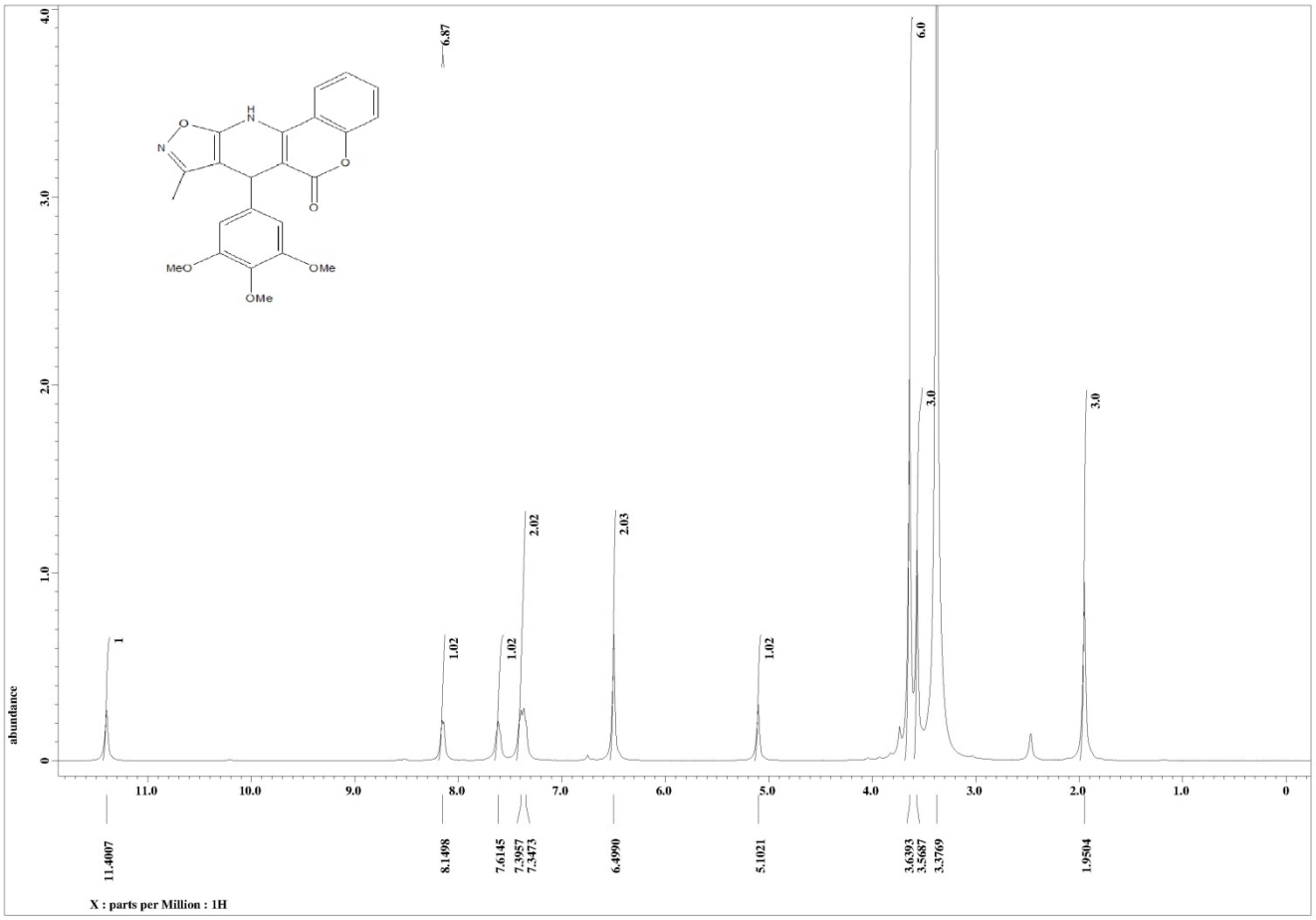
9.9333
X : parts per Million : 13C

^{1}H NMR of 4k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X : parts per Million : 1H
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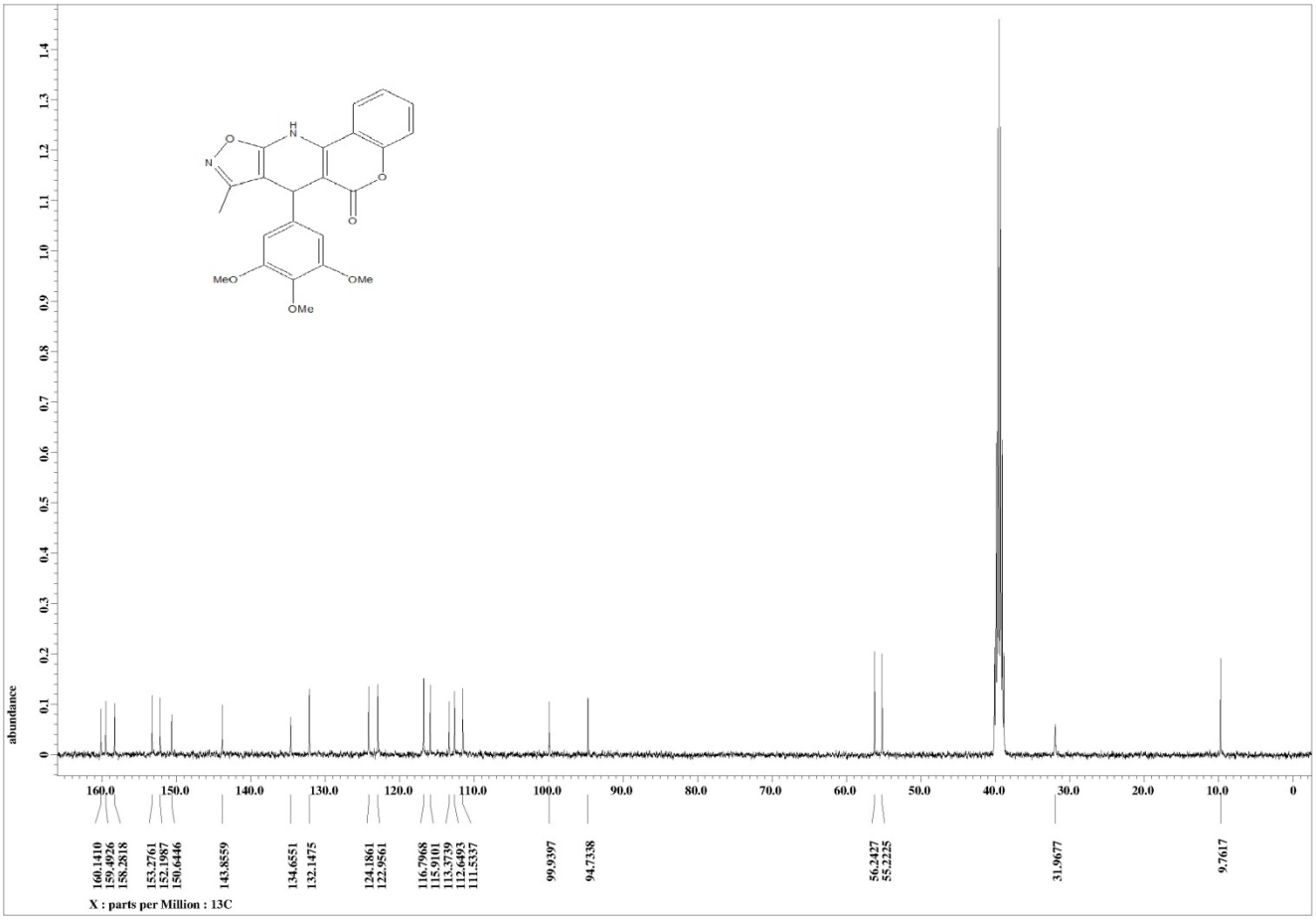
MeO
OMe
OMe

^{13}C NMR of 4k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152.1987
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143.8559
134.6551
132.1475
124.1861
122.9561
116.7968
115.9101
113.3739
112.6493
111.5337
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94.7338
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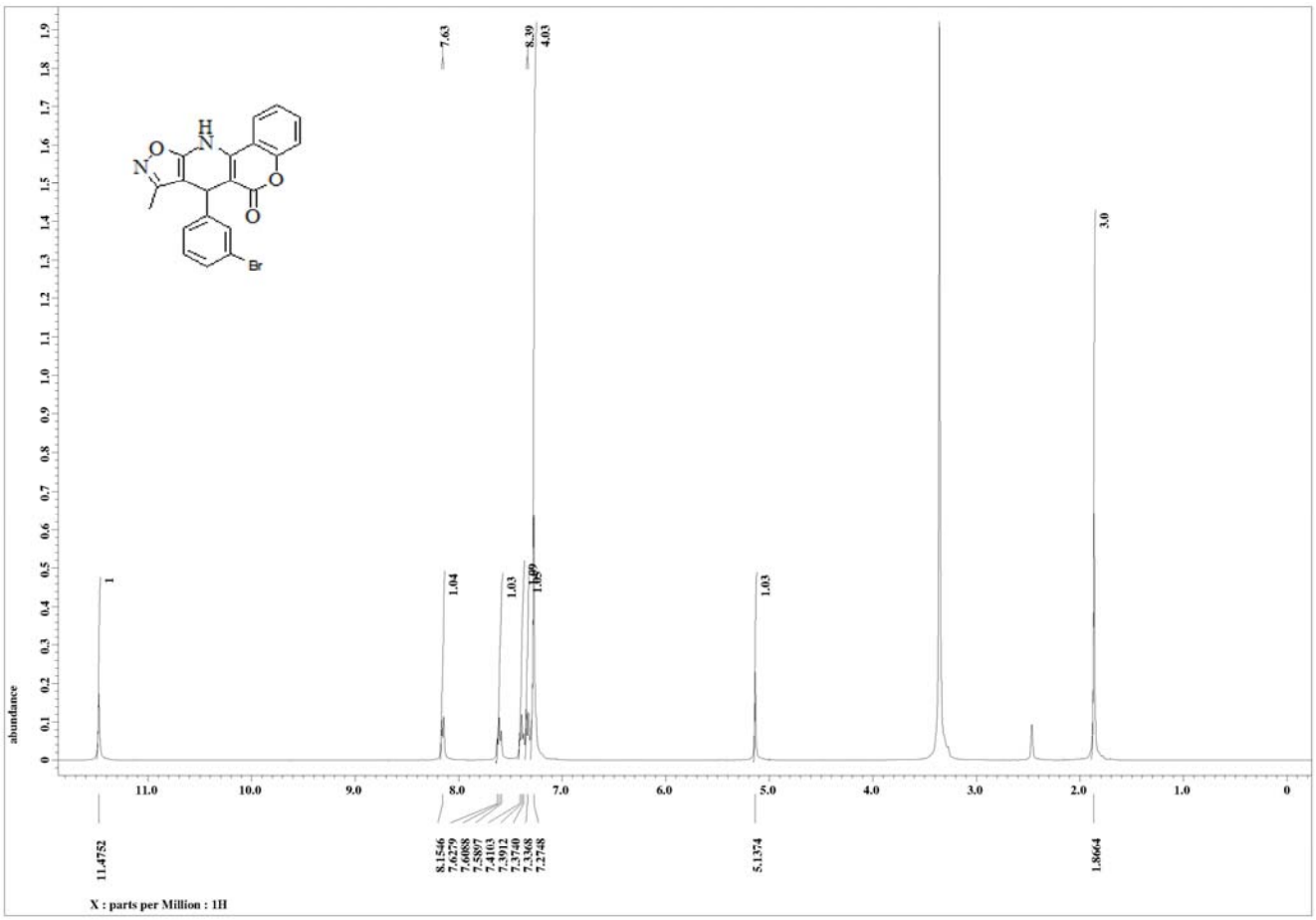
9.7617
X : parts per Million : 13C

^{1}H NMR of 4l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7.3368
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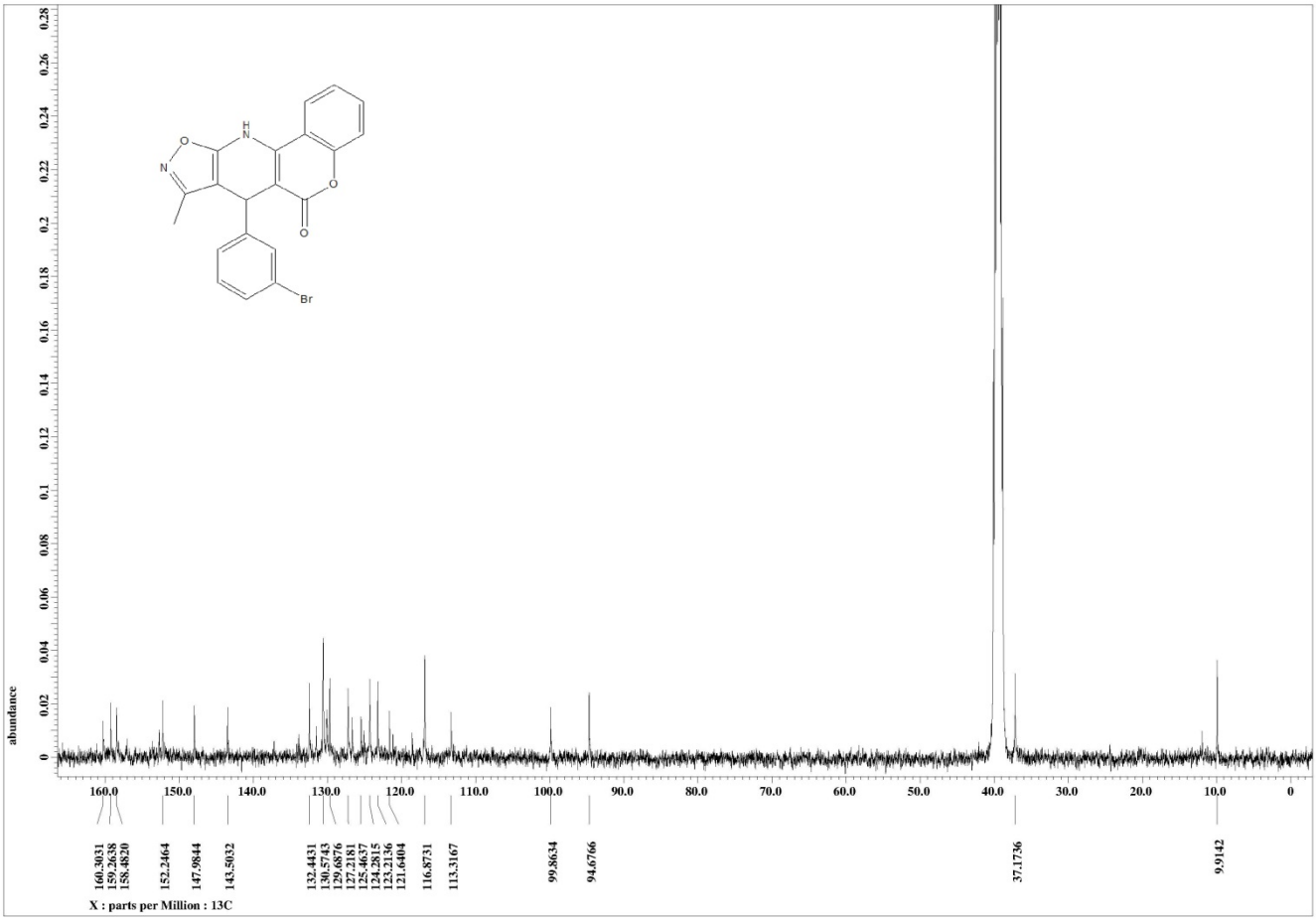
Br

^{13}C NMR of 4l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0.24
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129.6876
127.7151
125.4637
124.2815
123.2136
121.6404
116.8731
113.3167
99.8634
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9.9142
X : parts per Million : 13C
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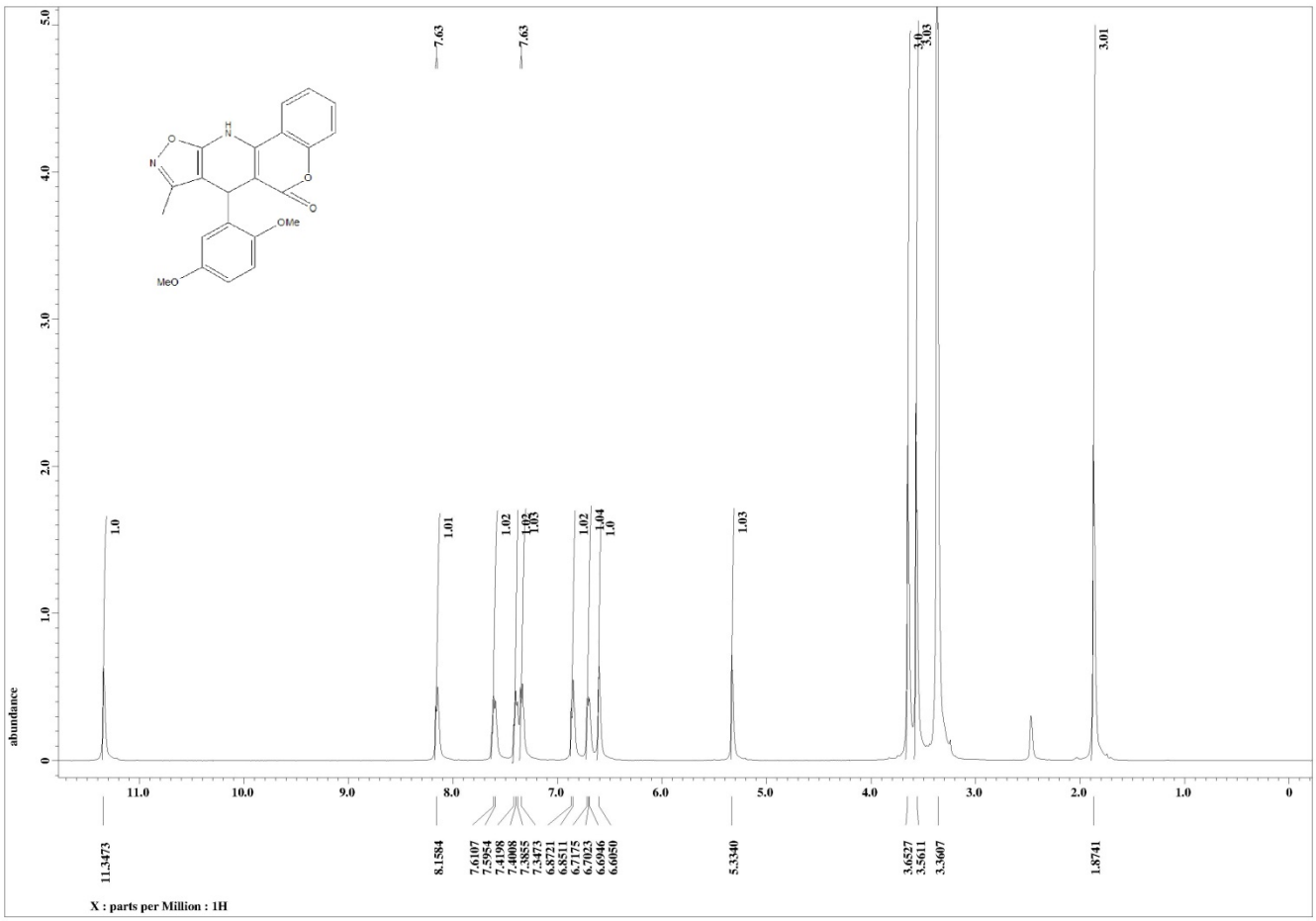
H
N
O
O
Br

^{1}H NMR of 4m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6.7023
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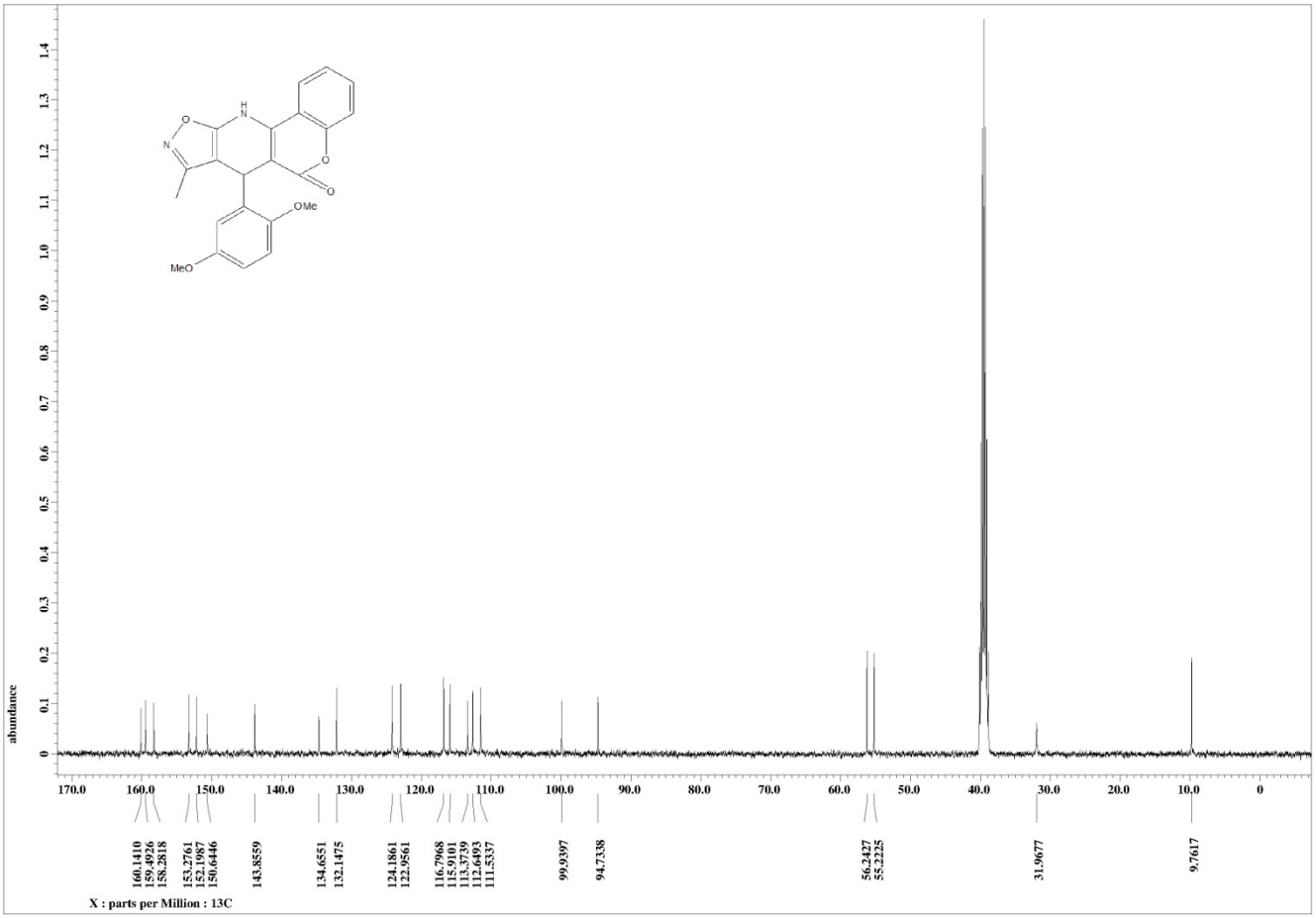
1.8741
OMe
MeO

^{13}C NMR of 4m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132.1475
124.1861
122.9561
116.7968
115.9101
113.3739
112.6493
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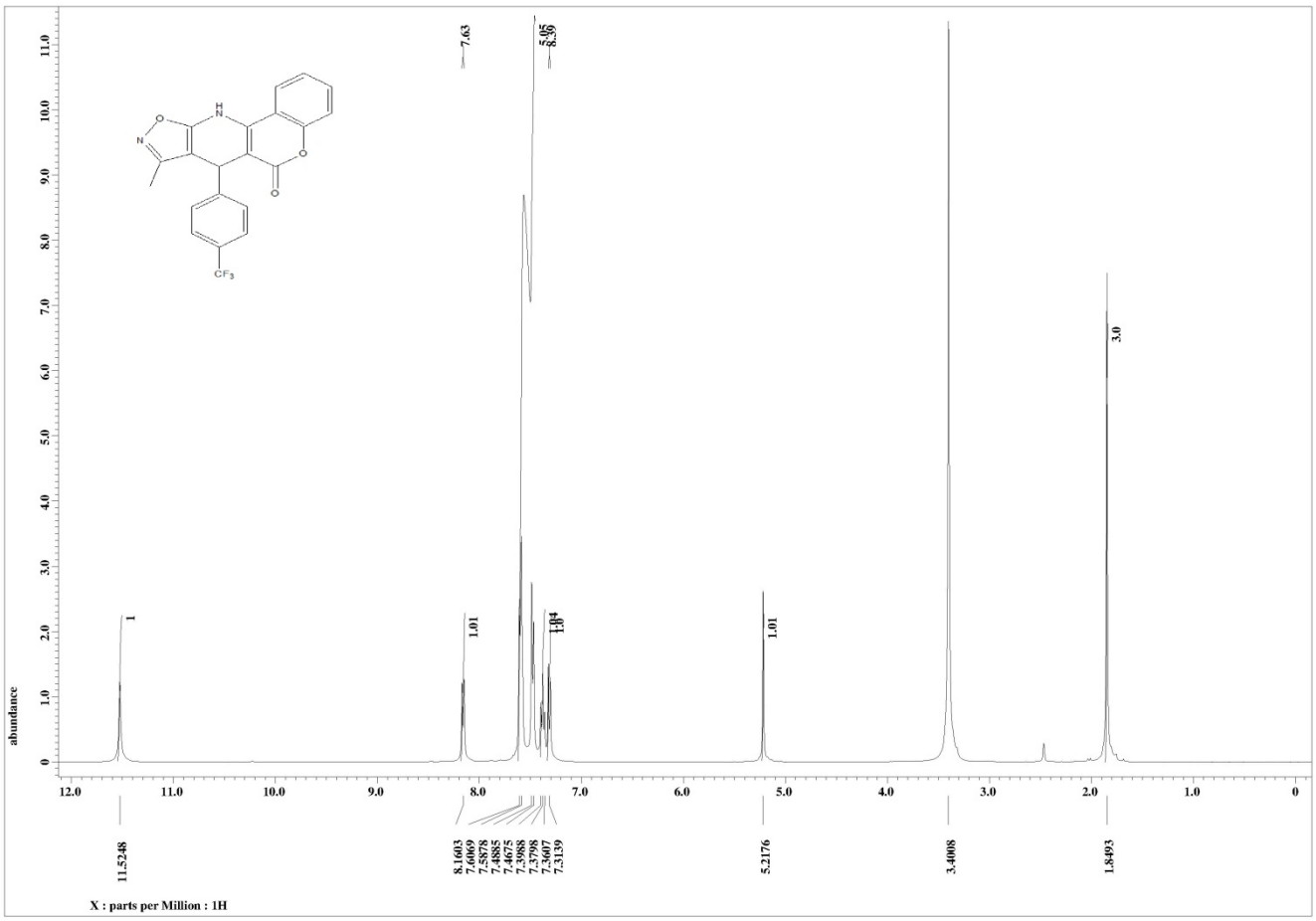
OMe
MeO

^{1}H NMR of 4n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7.3617
7.3139
5.2176
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8.39
1.01
1.01
3.0
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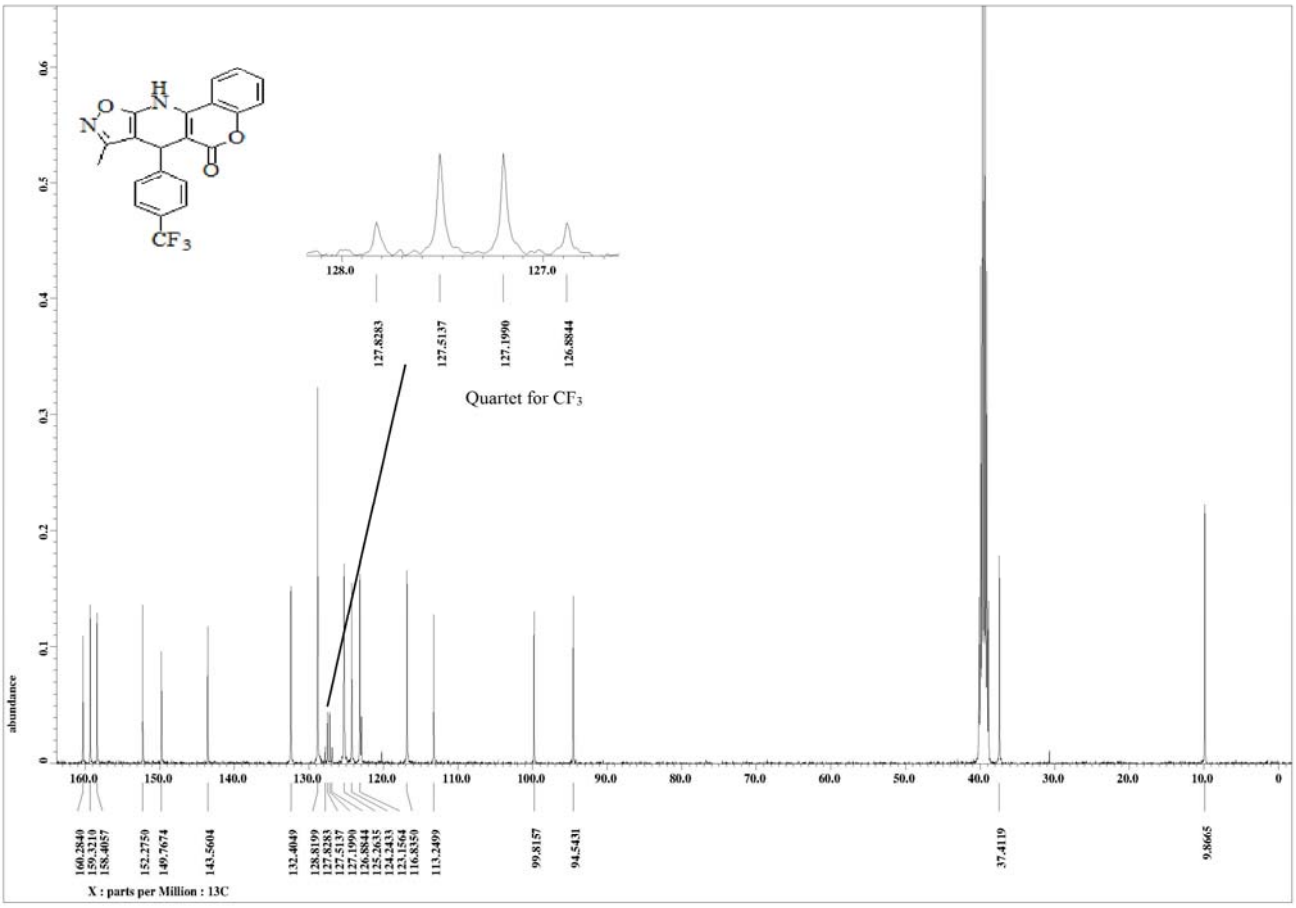
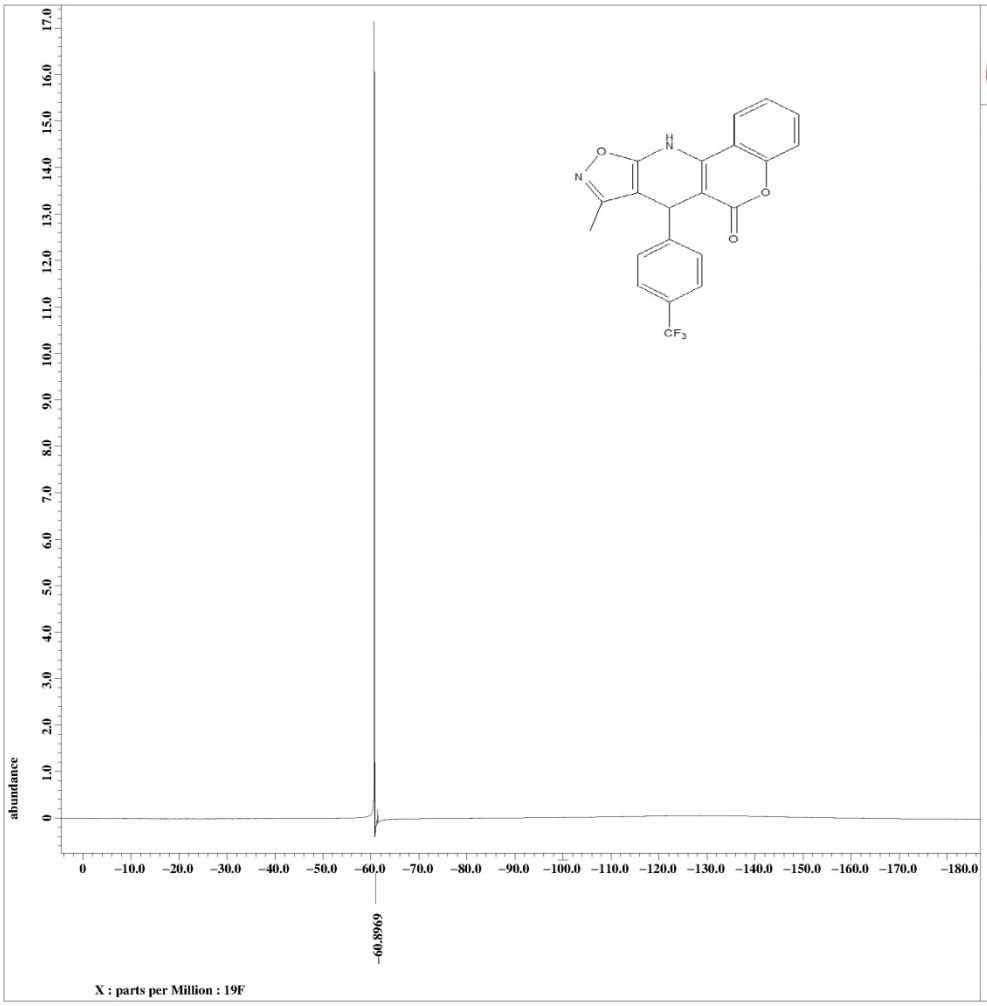
CF3

^{13}C NMR of 4n



^{19}F NMR of 4n

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Solvent           = DMSO-D6
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Revision_time     = 20-NOV-2015 12:21:02
Current_time      = 20-NOV-2015 12:21:14

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Dim_size          = 26214
Dim_title         = 19F
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 400P
Spectrometer      = DELTA2_NMR

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Tri_offset        = 5[ppm]
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Mod_return        = 1
Scans             = 32
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X_pulse           = 6.16[us]
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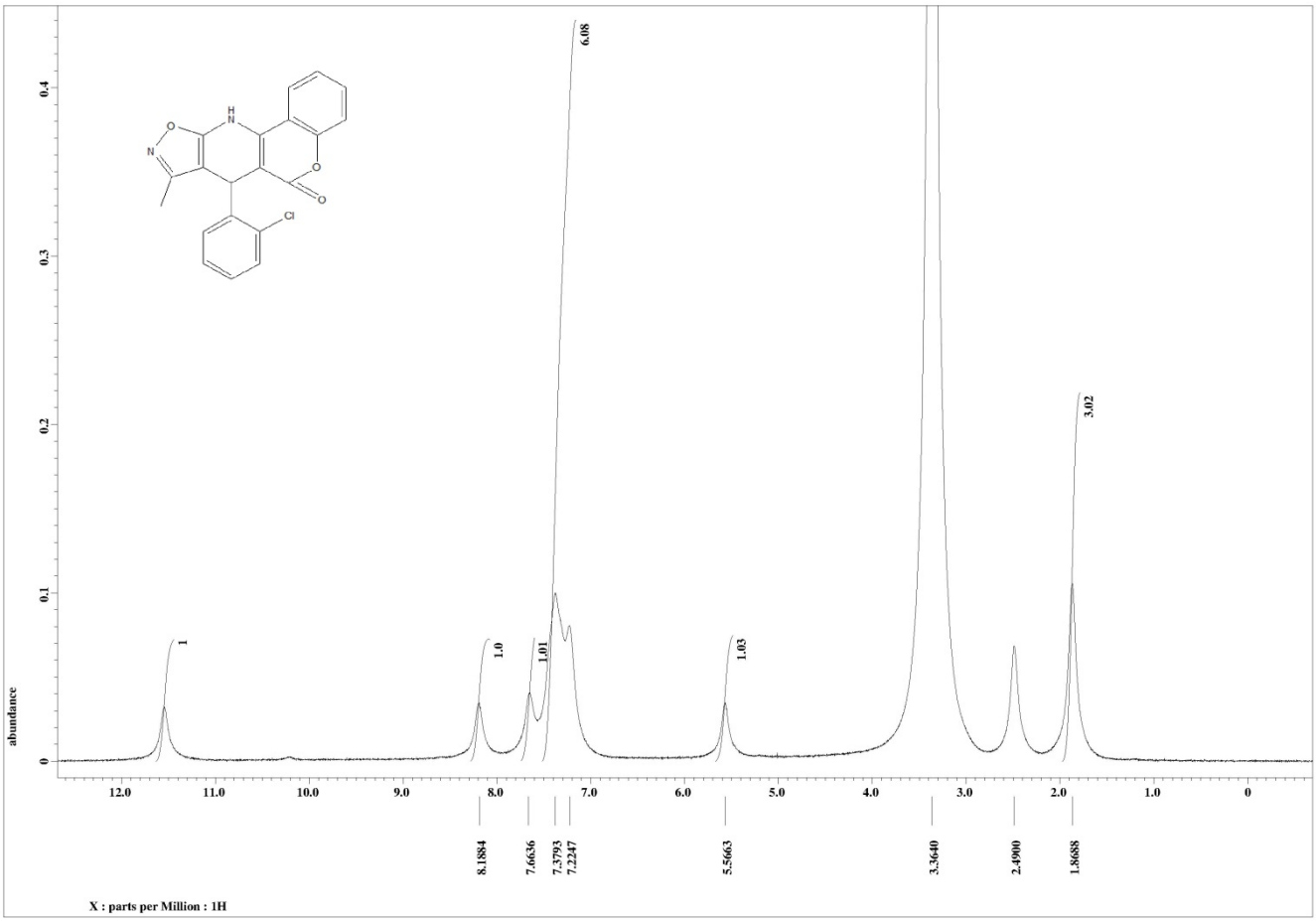
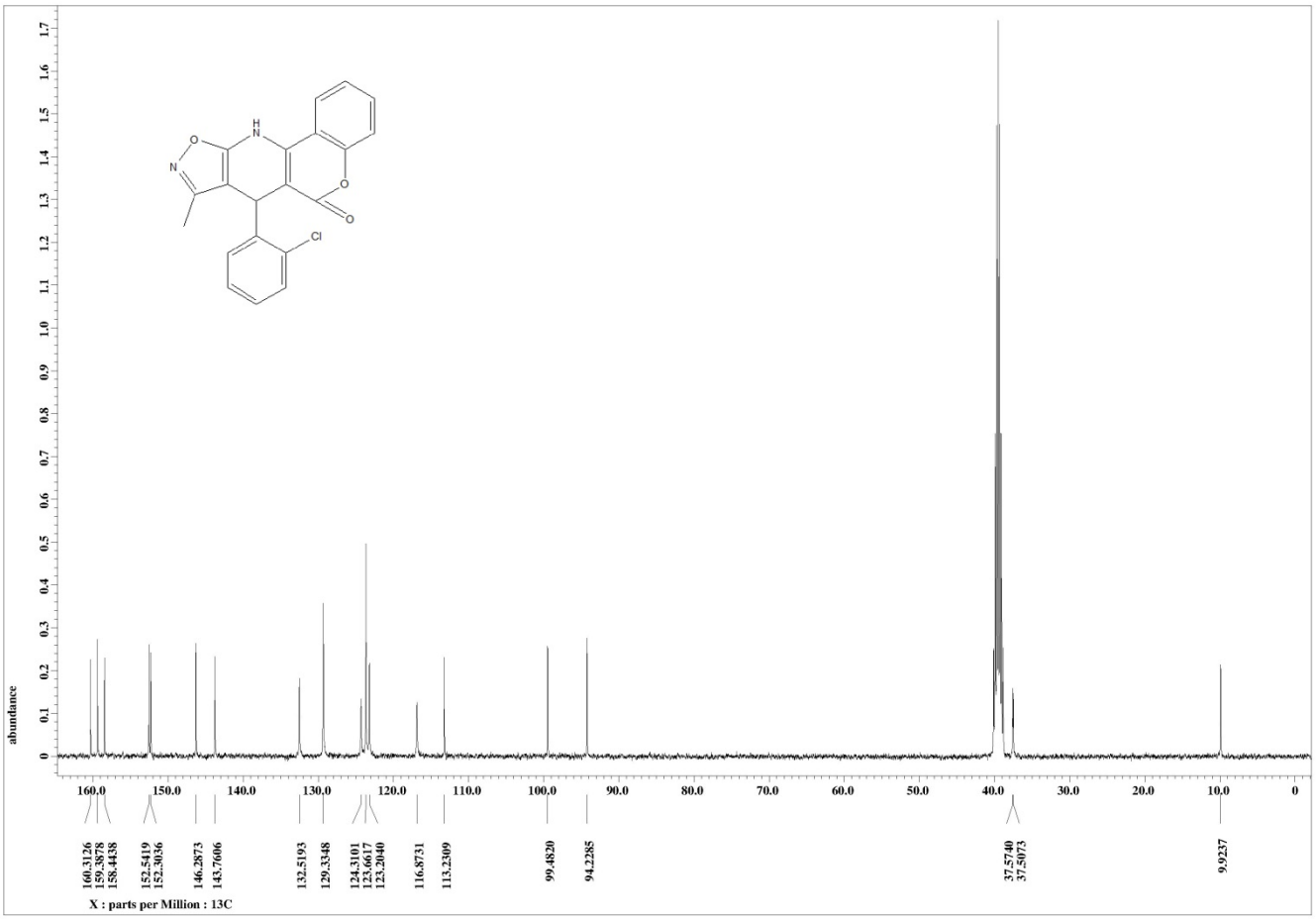
^{1}H NMR of 4o


6.08
3.02
1
1.0
1.01
1.03
abundance
0
0.1
0.2
0.3
0.4
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
8.1884
7.6636
7.3793
7.2247
5.5663
3.3640
2.4900
1.8688
X : parts per Million : 1H
H
N
O
N
O
O
Cl

^{13}C NMR of 4o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159.3878
158.4438
152.5419
152.3036
146.2873
143.7606
132.5193
129.3348
124.3101
123.6617
123.2040
116.8731
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99.4820
94.2285
37.5740
37.5073
9.9237
X : parts per Million : 13C

Crystallographic study

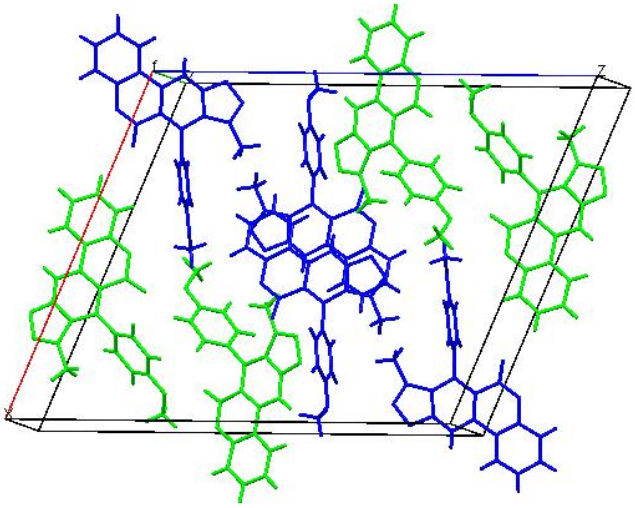

Figure S1: View of crystal packing of **4b** shown along b axis according to symmetry equivalence

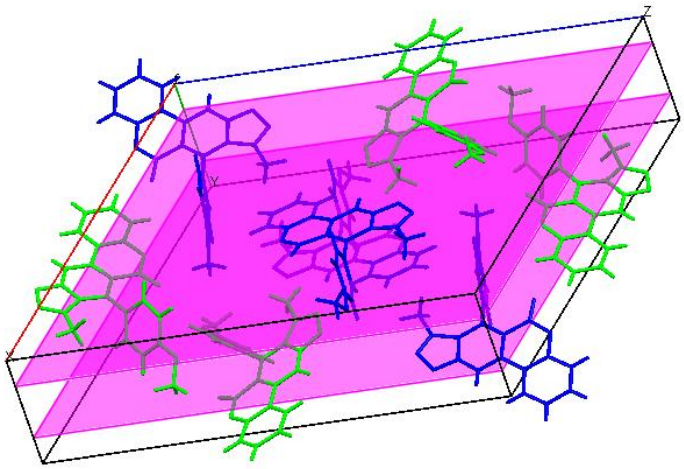

Figure S2: View of crystal packing of **4b** showing glide planes

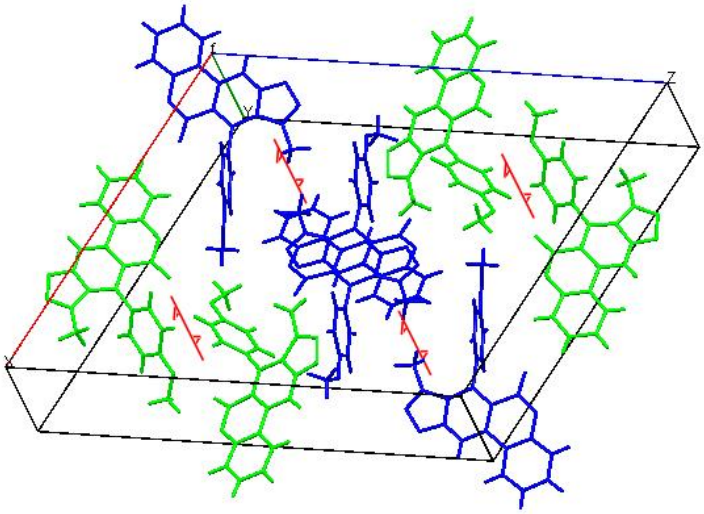

Figure S3: View of crystal packing of **4b** showing four Screw axes

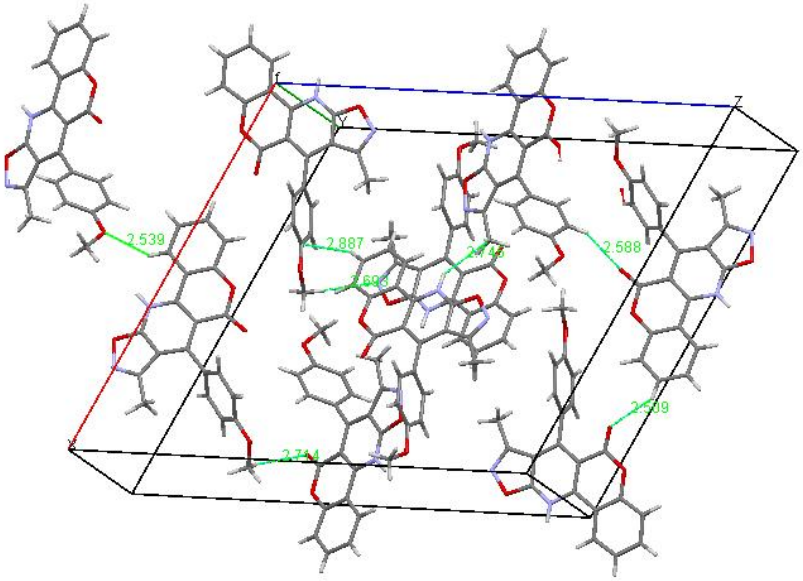

Figure S4: Intermolecular C-H…..O, C-H…..N and C-H…..C interactions in the crystal packing of **4b**

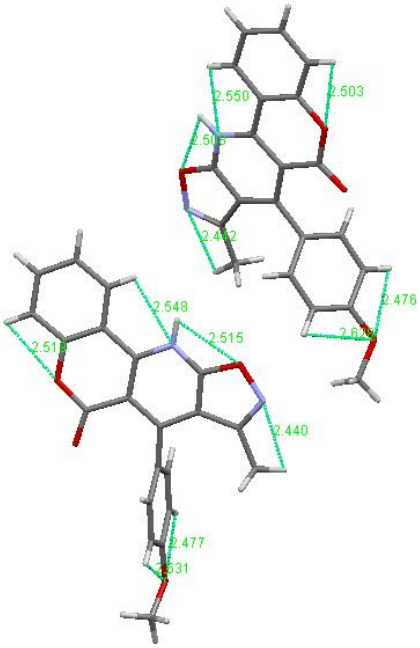

Figure S5: Intramolecular C-H…..O and C-H…..N interactions in the crystal structure of **4b**