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Synthesis of 3-(*N*-arylthiocarbamoyl)chromones from 2-hydroxyphenyl amino enones and isothiocyanates

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General Experimental Details

NMR spectra (for compounds **8a-j** and **13a-d**) were recorded on Bruker AM-300 (^{1}H: 300 MHz; ^{13}C: 75 MHz) using $CDCl_3$ and DMSO-d_6. Data for ^{1}H NMR are reported as chemical shifts (δ ppm), multiplicity (s = singlet, d = doublet, dd = doublet of doublets, t = triplet, m = multiplet), coupling constant(s) in hertz (Hz), and integration. Data for ^{13}C NMR are reported as chemical shift. Chemical shift were referenced to the residual solvent peak. IR spectra were registered on a Bruker ALPHA spectrometer in KBr pellets. Elemental analysis were recorded on Eurovector EA 3000 with autoanaliser. High-resolution mass spectra (HRMS) were recorded on Bruker micrOTOF II using electrospray. Mass spectra were obtained on the mass spectrometer (70 eV) with direct sample injection into the ion source. Melting points were measured using Boetius

melting point apparatus and are uncorrected. Reactions monitored by thin-layer chromatography using Merck 60 F254 UV-254 precoated silica gel plates.

^{1}H NMR and 2D correlation spectra for compound **10** were recorded on a Bruker Avance 600 spectrometer operated at 600 and 150 MHz for ^{1}H and ^{13}C, respectively, in $CDCl_3$ at 293 K. The ^{13}C NMR spectrum for compound **10** was measured on a Bruker DRX500 spectrometer operated at 125 MHz in $CDCl_3$ at room temperature. The residual proton signal from the solvent at δ 7.26 was used as the internal standard for the ^{1}H NMR spectrum; the signal at δ 77.2, for the ^{13}C NMR spectra. The 2D correlation spectra were recorded using standard procedures included in the Bruker software.

Experimental Procedures

Synthesis of starting material

3-Dimethylamino-1-(2-hydroxyphenyl)prop-2-en-1-one [S1], and 1-(2-hydroxyphenyl)-3-(phenylamino)prop-2-en-1-one [S2] were prepared according to the reported methods.

All isothiocyanates were synthesized by reaction of amines with thiophosgene according to the reported methods [S3-S7].

General procedure for the synthesis of 3-thiocarbamoylchromone (8a-k, 13a-d)

A mixture of amino enone (1 mmol) and aryl isothiocyanate (2.2 mmol) in dry DMF (2 ml) was heated at 120 °C. After 4-16 hours (the reaction progress was monitored by TLC) the precipitate that formed was filtered off and washed with solution DCM/hexane (1:10) and then the crude product was purified on column chromatography (DCM/hexane 2:1).

4-Oxo-*N*-phenyl-4*H*-chromene-3-carbothioamide (8a)

Yield 68%. Yellow crystals. Mp. 122–123 °C; lit. [S8] 121-127 °C. IR: 2923, 1614, 1555, 1463, 1402, 1304, 757 cm-1; ^{1}H-NMR (300 MHz, $CDCl_3$) δ 13.72 (c, 1HNH), 9.76 (s, 1H), 8.37 (d, J = 8.0 Hz, 1H), 7.92 (d, J = 7.9 Hz, 2H), 7.83 (t, J = 7.8 Hz, 1H), 7.65 – 7.52 (m, 2H), 7.47 (t, J = 7.8 Hz, 2H), 7.32 (t, J = 7.4 Hz, 1H); ^{13}C-NMR (75.47 MHz, DMSO-d_6) δ 178.6, 175.5, 163.4, 155.0, 138.9 , 138.1, 135.3, 128.7 (2C), 126.6, 126.5, 125.7, 123.3 (2C), 122.7, 118.5; MS m/z (I, %) 281 (100), 247 [M – S]+ (87), 189 [M – NHC_6H_5]+ (7), 121 [Ph(CO)O]+ (28). Calculated for $C_{16}H_{11}NO_2S$ %: C, 68.31; H, 3.94; N, 4.98; S, 11.40. Found %: C, 68.21; H, 3.97; N, 5.00; S, 11.33.

***N*-(4-Bromophenyl)-4-oxo-4*H*-chromene-3-carbothioamide (8b)**

Yield 53%. Yellow crystals. Mp. 184-186 °C. IR: 3430, 1612, 1576, 1548, 1487, 1462, 1391, 1304, 764 cm-1; ^{1}H-NMR (300 MHz, $CDCl_3$) δ 13.75 (s, 1HNH), 9.69 (s, 1H), 8.34 (d, J = 8.0 Hz, 1H), 7.86 – 7.79 (m, J = 12.8, 8.5 Hz, 3H), 7.63 – 7.54 (m, J = 18.5, 8.6 Hz, 4H); ^{13}C-NMR (75 MHz, CDCl3) δ 188.4, 177.7, 166.2, 155.5, 138.0, 135.1, 131.8, 126.7, 126.5, 125.6, 123.8, 119.9, 119.8, 118.4; HRMS (micrOTOF) m/z calcd. for $C_{16}H_{10}BrNO_2S$: 360.9595, found 360.9608.

***N*-(3-Methylphenyl)-4-oxo-4*H*-chromene-3-carbothioamide (8c)**

Yield 42%. Yellow crystals. Mp. 122-124 °C. IR: 3425, 2924, 1606, 1551, 1462, 1391, 1312, 763 cm-1; 1H-NMR (300 MHz, CDCl3) δ 13.64 (s, 1HNH), 9.75 (s, 1H), 8.36 (d, J = 8.1 Hz, 1H), 7.85 – 7.73 (m, 2H), 7.68 – 7.53 (m, 3H), 7.34 (t, J = 7.8 Hz, 1H), 7.12 (d, J = 7.7 Hz, 1H), 2.42 (s, 3H); 13C-NMR (75 MHz, CDCl3) δ 188.1, 177.7, 166.2, 155.5, 138.9, 138.8, 135.0, 128.6, 127.7, 126.6, 126.5, 124.7, 123.9 121.3, 120.0, 118.4, 21.4; HRMS (micrOTOF) m/z calcd. for C17H13NO2S+Na: 318.0559, found: 318.0551.

***N*-(4-Fluorophenyl)-4-oxo-4*H*-chromene-3-carbothioamide (8d)**

Yield 21%. Yellow crystals. Mp. 159-160 °C. IR: 3424, 1613, 1559, 1507, 1463, 1423, 1396, 1309, 1215, 836, 761 cm-1; ^{1}H-NMR (300 MHz, $CDCl_3$) δ 13.69 (s, 1HNH), 9.75 (s, 1H), 8.37 (d, J = 8.0 Hz, 1H), 7.90 – 7.80 (m, J = 14.6, 9.2 Hz, 3H), 7.71 – 7.47 (m, 2H), 7.15 (t, J = 8.6 Hz, 2H); ^{13}C-NMR (75 MHz, $CDCl_3$) δ 188.7, 177.8, 166.3, 155.5, 135.1, 126.7, 126.5, 126.2, 126.1, 123.9, 119.8, 118.4, 115.8, 115.5; HRMS (micrOTOF) m/z calcd. for $C_{16}H_{10}FNO_2S$: 300.0489, found 300.0498.

***N*-(2,4-Dichlorophenyl)-4-oxo-4*H*-chromene-3-carbothioamide (8e)**

Yield 61%. Yellow crystals. Mp. 201-203 °C. IR: 3429, 2926, 1646, 1533, 1466, 1401, 1310, 764 cm-1; ^{1}H-NMR (300 MHz, $CDCl_3$) δ 13.71 (s, 1HNH), 9.70 (s, 1H), 8.48 – 8.32 (m, 2H), 7.82 (t, J = 7.7 Hz, 1H), 7.69 – 7.48 (m, 3H), 7.34 (dd, J = 8.8, 2.2 Hz, 1H); ^{13}C-NMR (75 MHz, CDCl3) δ 190.1, 177.4, 166.2, 155.5, 135.0, 134.9, 132.6, 129.5, 129.4, 127.9, 126.9, 126.7, 126.6, 124.0, 120.0, 118.3; HRMS (micrOTOF) m/z calcd. for $C_{16}H_9Cl_2NO_2S$: 349.9803, found 349.9809.

***N*-(3,5-Dichlorophenyl)-4-oxo-4*H*-chromene-3-carbothioamide (8f)**

Yield 63%. Yellow crystals. Mp. 222-224 °C. IR: 3428, 2832, 1614, 1574, 1547, 1460, 1390, 1307, 760 cm-1; ^{1}H-NMR (300 MHz, DMSO-d_6) δ 13.01 (s, 1H), 9.26 (s, 1H), 8.21 (d, J = 7.6

Hz, 1H), 7.99 (s, 2H), 7.90 (t, J = 7.6 Hz, 1H), 7.74 (d, J = 8.4 Hz, 1H), 7.60 (t, J = 7.4 Hz, 1H), 7.45 (s, 1H); ^{13}C-NMR low solubility; HRMS (micrOTOF) m/z calcd. for $C_{16}H_9Cl_2NO_2S$: 349.9803, found 349.9809.

_N_-(2-Methoxyphenyl)-4-oxo-4_H_-chromene-3-carbothioamide (8g)

Yield 49%. Yellow crystals. Mp. 115-117 °C. IR: 3428, 1655, 1638, 1618, 1544, 1390, 768 cm-1; ^{1}H-NMR (300 MHz, $CDCl_3$) δ 13.74 (s, 1HNH), 9.74 (s, 1H), 8.90 (d, J = 8.0 Hz, 1H), 8.40 (d, J = 7.9 Hz, 1H), 7.80 (t, J = 7.8 Hz, 1H), 7.67 – 7.49 (m, 2H), 7.28 (s, 1H), 7.05 (dd, J = 14.3, 7.9 Hz, 2H), 4.01 (s, 3H); ^{13}C-NMR (75 MHz, CDCl3) δ 187.2, 177.3, 166.1, 155.4, 151.6, 134.8, 128.5, 127.2, 126.6, 126.4, 124.4, 124.0, 120.7, 120.0, 118.3, 110.9, 56.1; HRMS (micrOTOF) m/z calcd. for $C_{17}H_{13}NO_3S$: 311.0616, found 311.0610.

_N_-(4-Nitrophenyl)-4-oxo-4_H_-chromene-3-carbothioamide (8h)

Yield 75%. Yellow crystals. Mp. >260 °C. IR: 3431, 1617, 1592, 1561, 1344, 1301, 751 cm-1; ^{1}H-NMR (300 MHz, $CDCl_3$) δ 13.99 (s, 1HNH), 9.43 (s, 1H), 8.21 – 7.97 (m, 5H), 7.65 (d, J = 7.2 Hz, 1H), 7.52 – 7.34 (m, 2H); ^{13}C-NMR low solubility; HRMS (micrOTOF) m/z calcd. for $C_{16}H_{10}N_2O_4S$: 327.0395, found 327.0389.

Ethyl 4-{[(4-oxo-4_H_-chromen-3-yl)carbonothioyl]amino}benzoate (8i)

Yield 74%. Yellow crystals. Mp. 176-178 °C. IR: 2984, 1707, 1548, 1466, 1391, 1277, 764 cm-1; ^{1}H-NMR (300 MHz, $CDCl_3$) δ 13.98 (s, 1HNH), 9.71 (s, 1H), 8.37 (dd, J = 7.9, 1.0 Hz, 1H), 8.12 (q, J = 8.9 Hz, 4H), 7.88 – 7.79 (m, 1H), 7.68 – 7.54 (m, 2H), 4.41 (q, J = 7.1 Hz, 2H), 1.42 (t, J = 7.1 Hz, 3H); ^{13}C-NMR (75 MHz, CDCl3) 188.6, 177.7, 166.3, 165.9, 155.5, 142.8, 135.2, 130.3, 128.3, 126.8, 126.5, 123.8, 123.3, 120.0, 118.4, 61.0, 14.3; HRMS (micrOTOF) m/z calcd. for $C_{19}H_{15}NO_4S$: 353.0722, found: 353.0795.

4-Oxo-_N_-(3-trifluoromethylphenyl)-4_H_-chromene-3-carbothioamide (8j)

Yield 70%. Yellow crystals. Mp. 185-187 °C. IR: 3447, 3070, 2959, 1635, 1524, 1466, 1396, 1310, 1164, 1109, 762 cm-1; ^{1}H-NMR (300 MHz, $CDCl_3$) δ 13.60 (s, 1HNH), 9.74 (s, 1H), 8.40 (d, J = 8.0 Hz, 1H), 7.91 (d, J = 8.0 Hz, 1H), 7.87 – 7.75 (m, J = 15.0, 7.7 Hz, 2H), 7.71 – 7.52 (m, J = 22.7, 7.6 Hz, 3H), 7.47 (t, J = 7.6 Hz, 1H); ^{13}C-NMR (75 MHz, CDCl3) δ 192.1, 177.6, 166.4, 155.5, 136.7, 135.1, 132.0, 130.2, 127.5, 126.7, 126.5, 126.4, 126.0, 125.6, 124.0, 119.4, 118.4; HRMS (micrOTOF) m/z calcd. for $C_{19}H_{15}NO_4S$: 350.0418, found: 350.0426.

2-(Furan-2-yl)-4-oxo-*N*-phenylchroman-3-carbothioamide (13a)

Yield 35%. Yellow crystals. Mp. 145-146 °C. IR: 3365, 3146, 2976, 1595, 1540, 1495, 1413, 1379, 1353, 12299, 1245, 1178, 971, 744, 694 cm-1; ^{1}H-NMR (300 MHz, DMSO-d_6) δ 12.18 (s, 1HNH), 8.11 (d, J = 9.4 Hz, 2H), 8.00 (d, J = 7.3 Hz, 2H), 7.94 – 7.84 (m, 1H), 7.77 (d, J = 8.0 Hz, 1H), 7.59 – 7.43 (m, 3H), 7.39 (s, 1H), 7.33 (d, J = 6.7 Hz, 1H), 6.81 (s, 1H); ^{13}C-NMR (75 MHz, DMSO-d6) δ 190.1, 174.0, 155.2, 148.1, 144.9, 139.9, 135.3, 129.3, 126.9, 126.3, 125.7, 124.2, 123.4, 123.4, 122.9, 118.7, 117.1, 113.5; HRMS (micrOTOF) m/z calcd. for $C_{20}H_{13}NO_3S$: 348.0689, found: 348.0696.

2-(Furan-2-yl)-4-oxo-*N*-(3-methylphenyl)chroman-3-carbothioamide (13b)

Yield 30%. Yellow crystals. Mp. 162-164 °C. IR: 3448, 3247, 3012, 1606, 1568, 1460, 1379, 751 cm-1; ^{1}H-NMR (300 MHz, $CDCl_3$) δ 10.76 (s, 1HNH), 8.06 (d, J = 8.0 Hz, 1H), 7.88 (s, 1H), 7.85 (s, 1H), 7.71 (s, 1H), 7.54 (t, J = 7.7 Hz, 1H), 7.33 (d, J = 3.6 Hz, 1H), 7.26 – 7.12 (m, 3H), 6.99 (d, J = 7.7 Hz, 1H), 6.64 (s, 1H), 2.30 (s, 3H); ^{13}C-NMR (75 MHz, CDCl3) δ 188.5, 175.7, 154.7, 150.4, 146.8, 146.4, 144.8, 139.2, 138.5, 134.1, 128.1, 127.1, 125.1, 125.1, 124.2, 123.1, 119.3, 117.6, 117.5, 112.9, 21.3; HRMS (micrOTOF) m/z calcd. for $C_{21}H_{15}NO_3S$: 362.0845, found: 361.0836.

2-(Furan-2-yl)-*N*-(4-nitrophenyl)-4-oxochroman-3-carbothioamide (13c)

Yield 50%. Yellow crystals. Mp. decomp. 237°C. IR: 3451, 3214, 3048, 1617, 1573, 1515, 1459, 1375, 1341, 759 cm-1; ^{1}H NMR (300 MHz, DMSO-d_6) δ 12.62 (s, 1H), 8.35 (s, 4H), 8.10 (d, J = 8.1 Hz, 1H), 8.05 (s, 1H), 7.90 (t, J = 7.8 Hz, 1H), 7.78 (d, J = 8.5 Hz, 1H), 7.56 (t, J = 8.0 Hz, 1H), 7.41 (s, 1H), 6.80 (s, 1H); ^{13}C-NMR low solubility; HRMS (micrOTOF) m/z calcd. for $C_{20}H_{12}N_2O_5S$: 392.0467, found: 392.0469.

***N*-(2,4-Dichlorophenyl)-2-(furan-2-yl)-4-oxochroman-3-carbothioamide (13d)**

Yield 32%. Yellow crystals. Mp. decomp. 180 °C. IR: 3424, 3185, 2923, 1612, 1574, 1526, 1460, 1370, 759 cm-1; ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.63 (s, 1HNH), 8.60 (d, J = 8.8 Hz, 1H), 8.18 (d, J = 7.8 Hz, 1H), 7.77 – 7.63 (m, 2H), 7.55 – 7.29 (m, 5H), 6.64 (d, J = 1.7 Hz, 1H); ^{13}C-NMR (75 MHz, CDCl3) δ 191.9, 174.9, 155.0, 151.0, 146.6, 145.0, 134.3, 134.2, 132.5, 129.5, 128.3, 127.4, 126.9, 126.0, 125.5, 123.4, 123.3, 117.8, 117.2, 112.7; HRMS (micrOTOF) m/z calcd. for $C_{20}H_{11}Cl_2NO_3S$: 415.9909, found: 415.9903.

(2*Z*,3*E*)-3-(Phenylaminomethylidene)-2-(phenylimino)chroman-4-one (10)

A mixture of 1-(2-hydroxyphenyl)-3-(phenylamino)prop-2-en-1-one (1 mmol) and isothiocyanatobenzene (2.2 mmol) in dry DMF (2 ml) was heated at 110 °C. In 5 hours (the reaction progress was monitored by TLC) the precipitate that formed was filtered off and washed with EtOAc. Yield 32%. Yellow crystals. Mp. 182-184 °C. IR: 3056, 1670, 1609, 1464, 1425, 1298, 1254, 1212, 1126, 754, 689 cm-1; HRMS (micrOTOF) m/z calcd. for $C_{22}H_{16}N_2O_2$: 341.1279, found: 341.1285. ^{1}H-NMR (600 MHz, $CDCl_3$) δ: 13.97 (d, J = 9.2 Hz, 1H, NH), 9.11 (d, J = 9.2 Hz, 1H, CH), 8.18 (dd, J = 7.8, 1.6 Hz, 1H, H5), 7.55 (ddd, J = 8.6, 7.8, 1.7 Hz, 1H, H7), 7.45 – 7.40 (m, 4H, Hm′, Hm″), 7.40 – 7.35 (m, 2H, Ho″), 7.32 (d, J = 7.8 Hz, 2H, Ho′), 7.28 (t, J = 7.5 Hz, 1H, H6), 7.24 (t, J = 7.4 Hz, 1H, Hp′), 7.21 (t, J = 7.3 Hz, 1H, Hp″), 7.15 (d, J = 8.3 Hz, 1H, H8); ^{13}C-NMR (126 MHz, Chloroform-d) δ: 177.2 (C4), 155.4 (C2), 154.5 (C8a), 150.7 (CH), 142.4 (Ci″), 142.2 (Ci′), 134.1 (C7), 129.9 (Cm′), 129.2 (Cm″), 126.6 (C5), 126.3 (Cp′), 124.7 (Cp″), 124.5 (C6), 123.4 (Co″), 121.5 (C4a), 119.2 (Co′), 116.9 (C8), 98.5 (C3);

The complete assignment of all signals in the ^{1}H and ^{13}C NMR spectra was made using 2D ^{1}H-^{1}H COSY, ^{1}H-^{13}C HSQC, and ^{1}H-^{13}C HMBC homo- and heteronuclear correlation techniques (Table S1, Figures S1-S3).

The observed spin-spin coupling (J of about 9 Hz) between protons of NH and CH groups initially seemed to be inconsistent with the structure. However, we hypothesized that only one tautomeric form of the proposed structure is manifested in the spectrum.

Table S1 ^{1}H and ^{13}C NMR assignment of compound **10**.

Position	δ_C, ppm	δ_H, ppm	HMBC (C→H)
2	155.4	–	CH
3	98.5	–	NH, CH
4	177.2	–	CH, H^5, H^8
4a	121.5	–	H^7, H^6, H^8
5	126.6	8.18	H^7
6	124.5	7.28	H^8
7	134.1	7.55	H^5, H^6
8	116.9	7.15	H^5, H^6, H^7
8a	154.5	–	H^5, H^7, H^6, H^8
i'	142.2	–	CH, $H^{m'}$, $H^{o'}$
o'	119.2	7.37	NH, $H^{m'}$, $H^{o'}$, $H^{p'}$
m'	129.9	7.42	$H^{m'}$, $H^{o'}$
p'	126.3	7.24	$H^{o'}$
i''	142.4	–	$H^{m''}$,$H^{o'}$
o''	123.4	7.32	$H^{m''}$, $H^{o''}$, $H^{p''}$
m''	129.2	7.43	$H^{m''}$
p''	124.7	7.24	$H^{o''}$
CH	150.7	9.11	–
NH	–	13.97	–

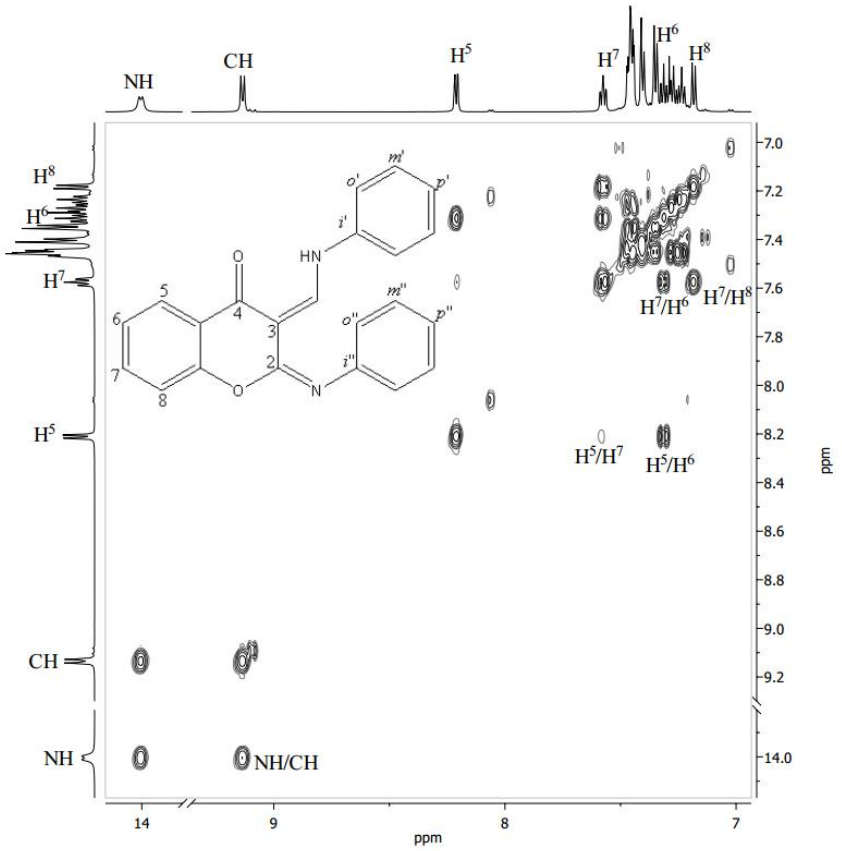

Figure S1. Fragment of ^{1}H-^{1}H COSY spectrum of compound **10**

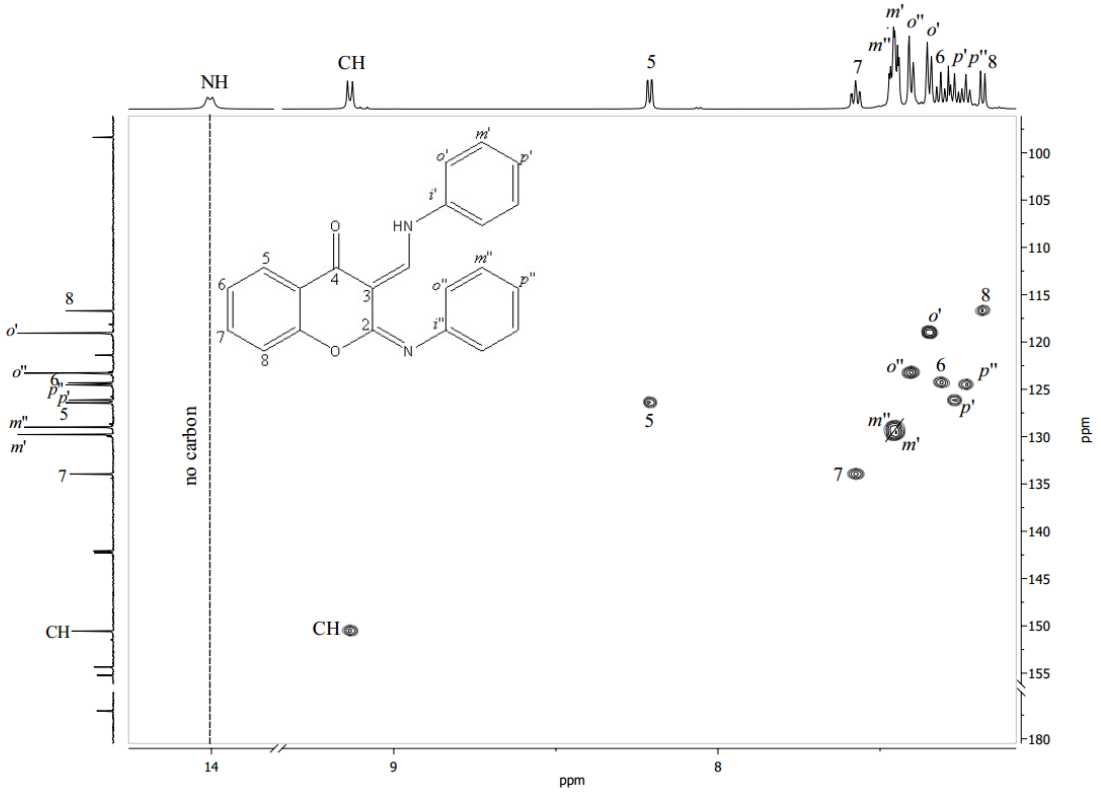

Figure S2. Fragment of ^{1}H-^{13}C HSQC spectrum of compound **10**

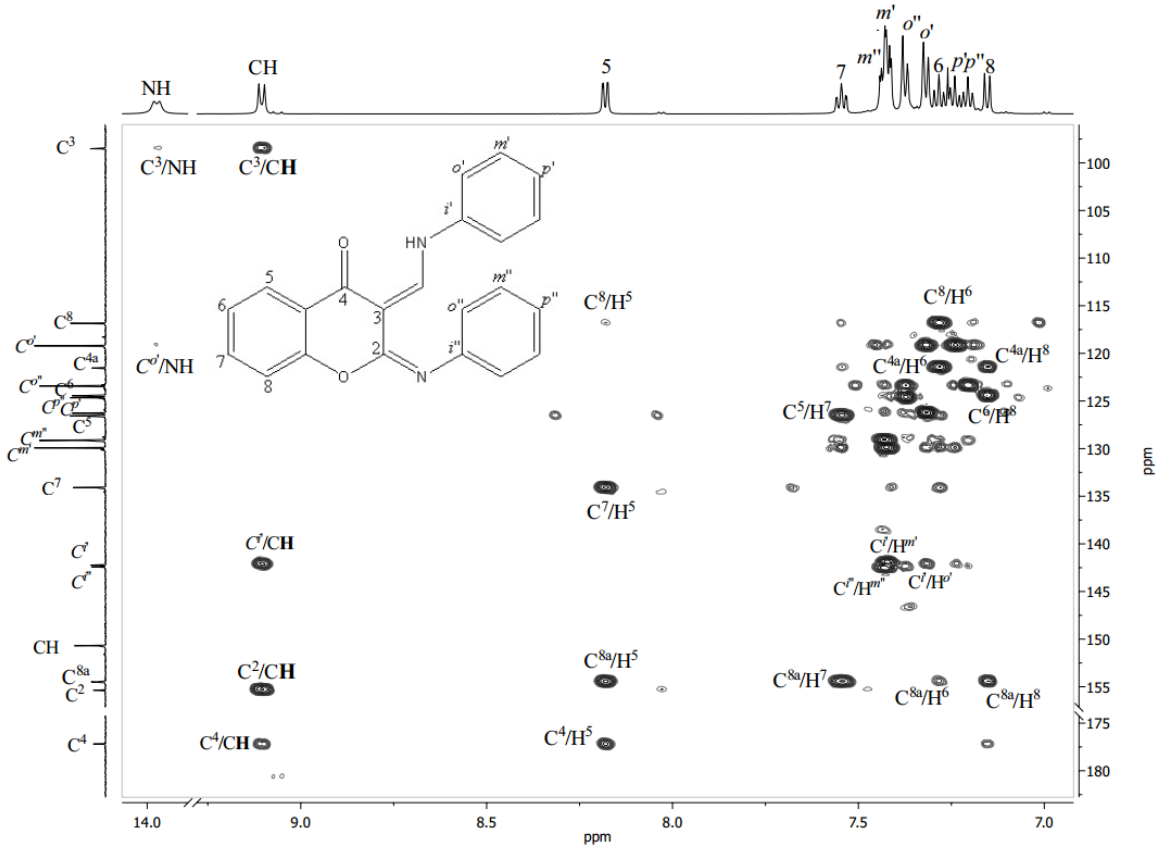

Figure S3. Fragment of ^{1}H-^{13}C HMBC spectrum of compound **10**

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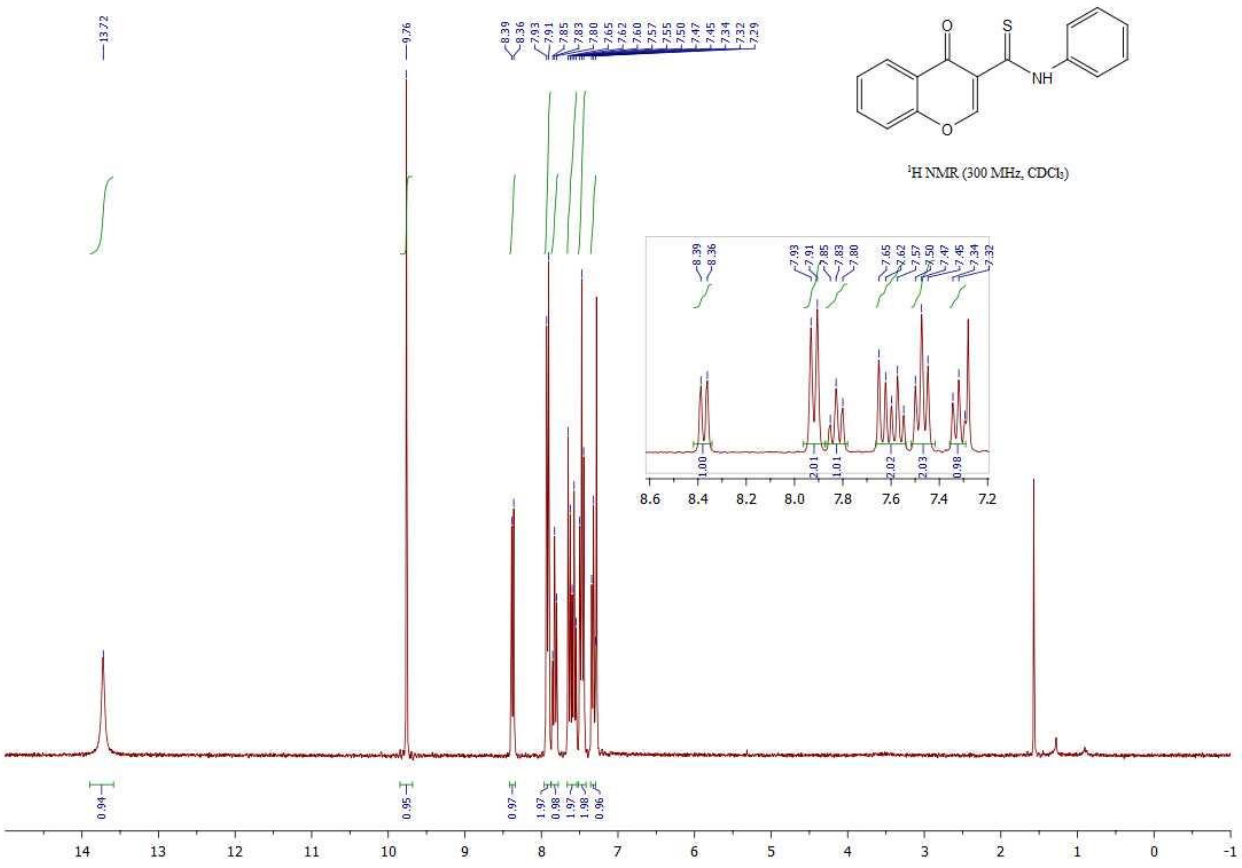
Spectra of compounds

8a



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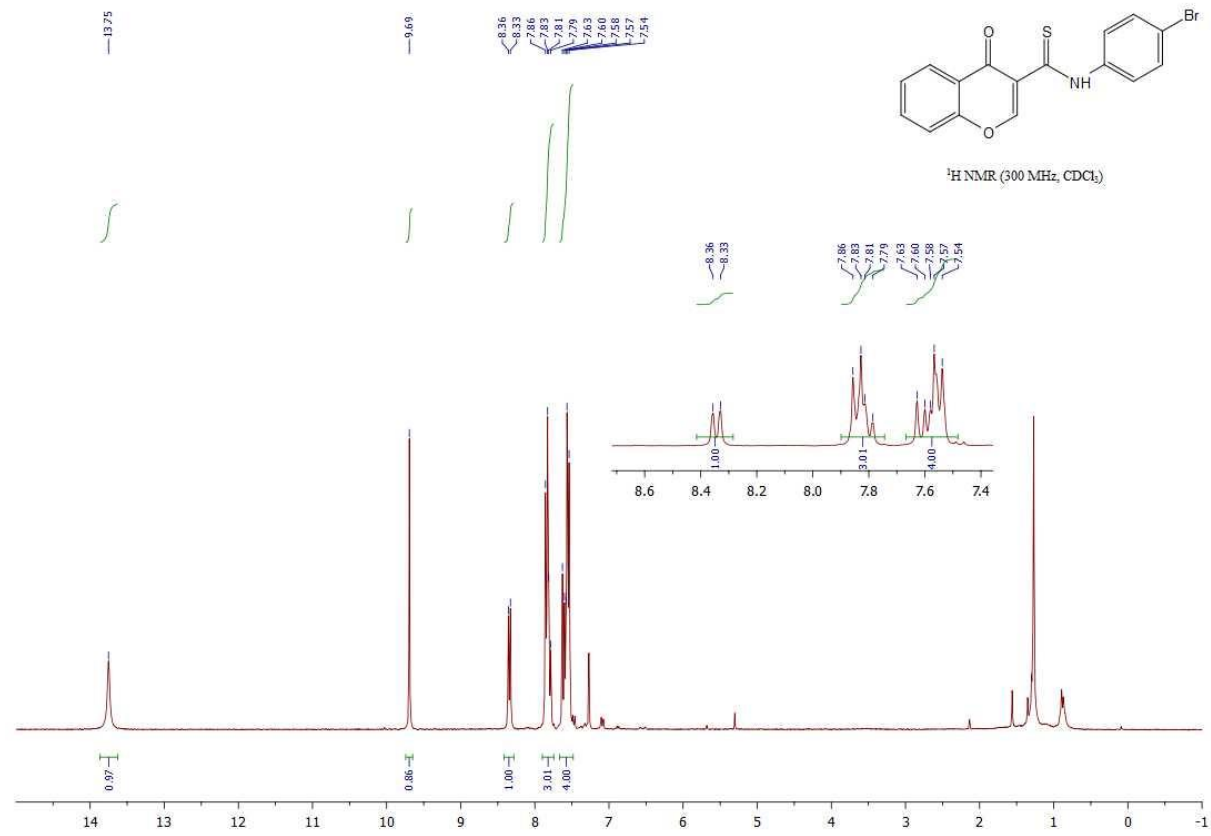
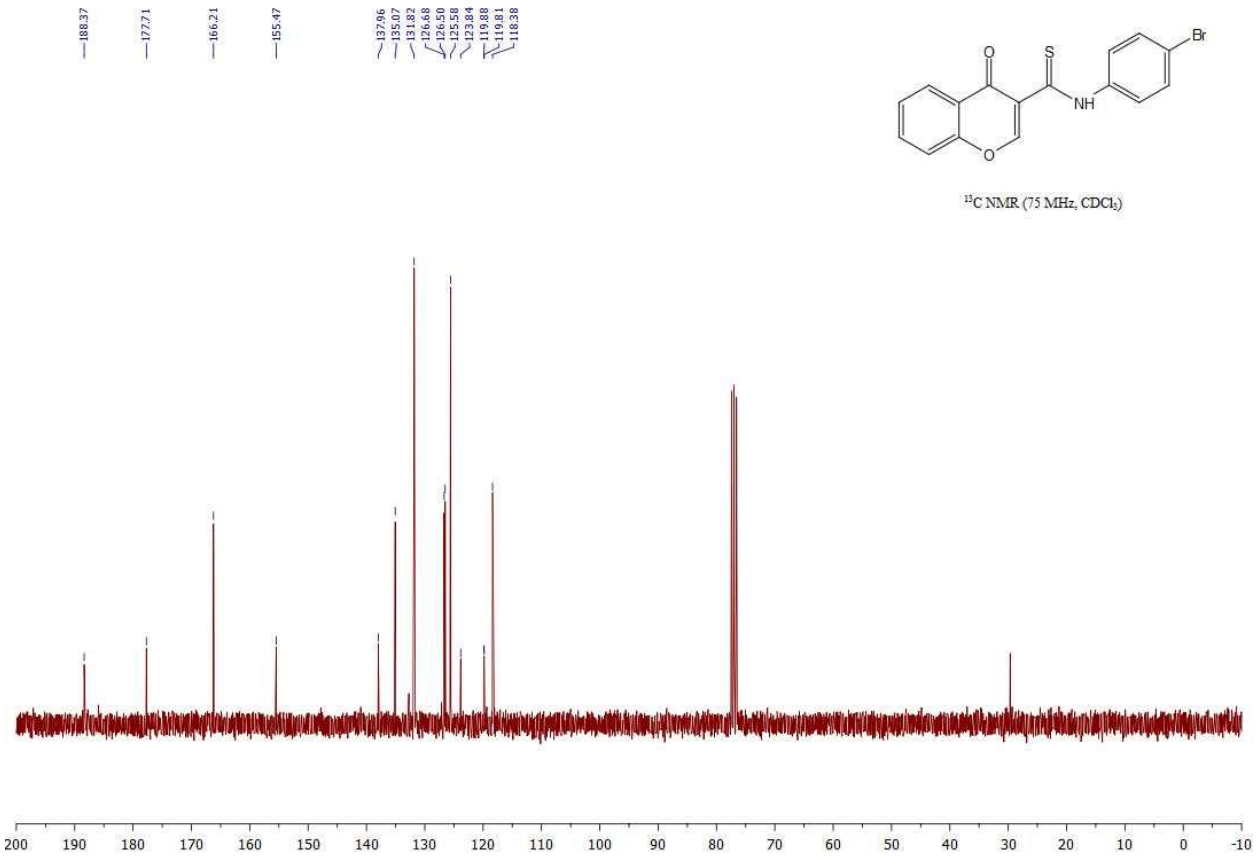
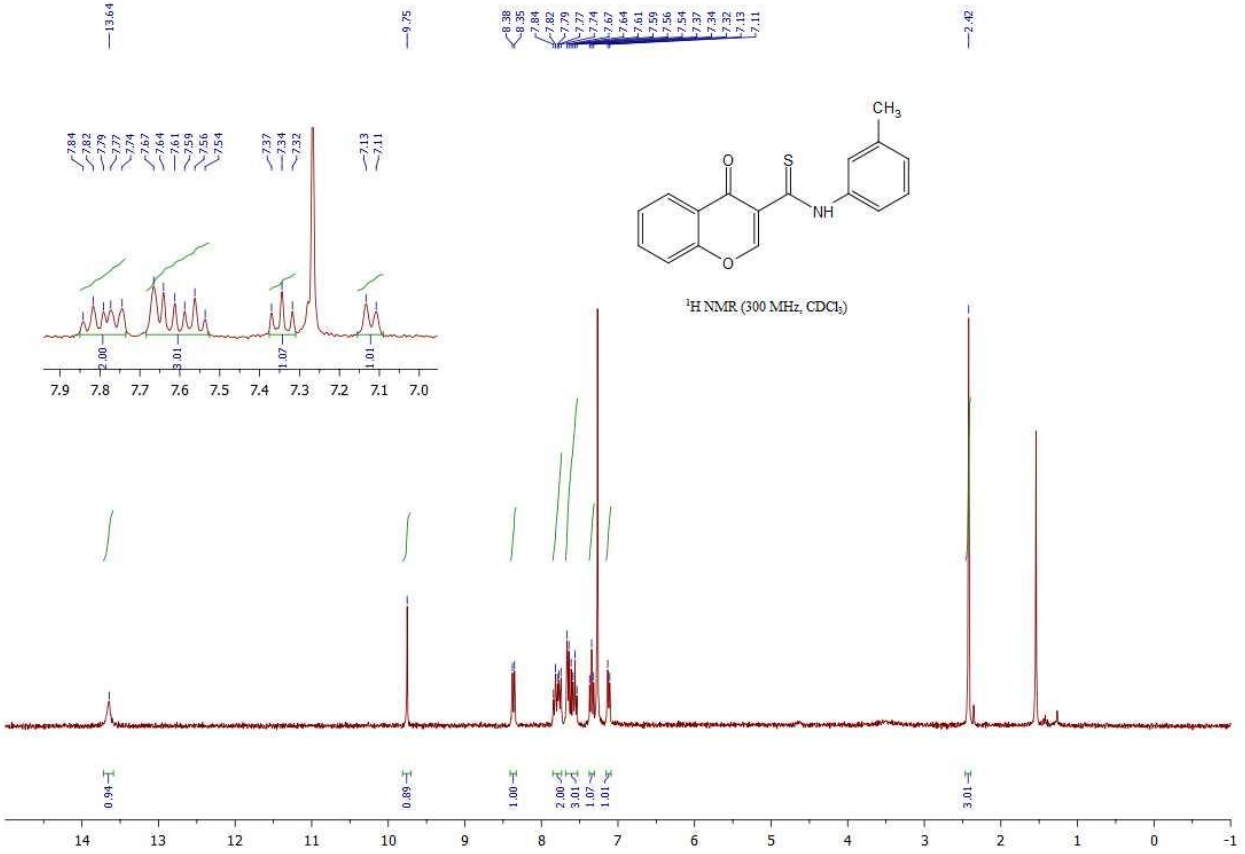


^{1}H NMR (300 MHz, $CDCl_3$)



^{13}C NMR (75 MHz, $CDCl_3$)

8c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7.37
7.34
7.32
7.13
7.11
2.42
CH3
O
S
NH
O
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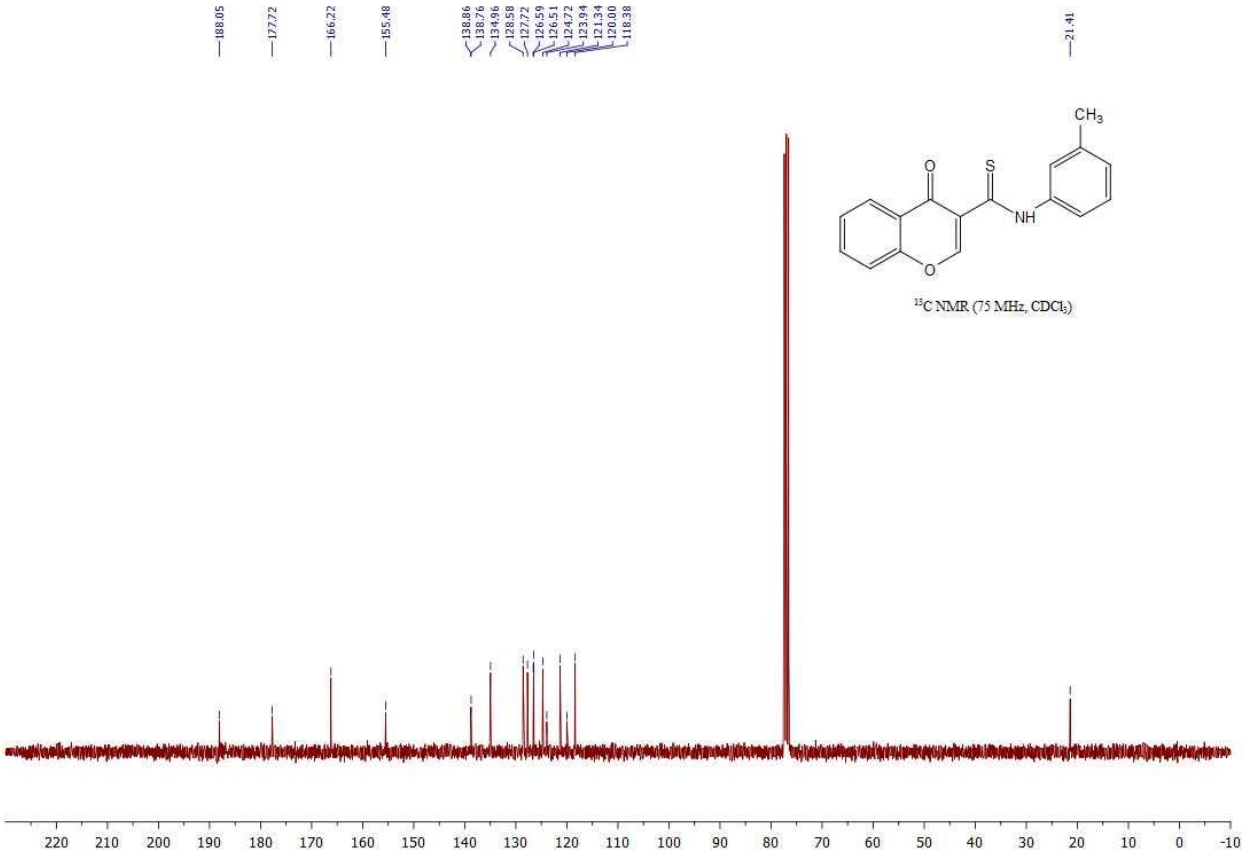
2.00
3.01
1.07
1.01
3.01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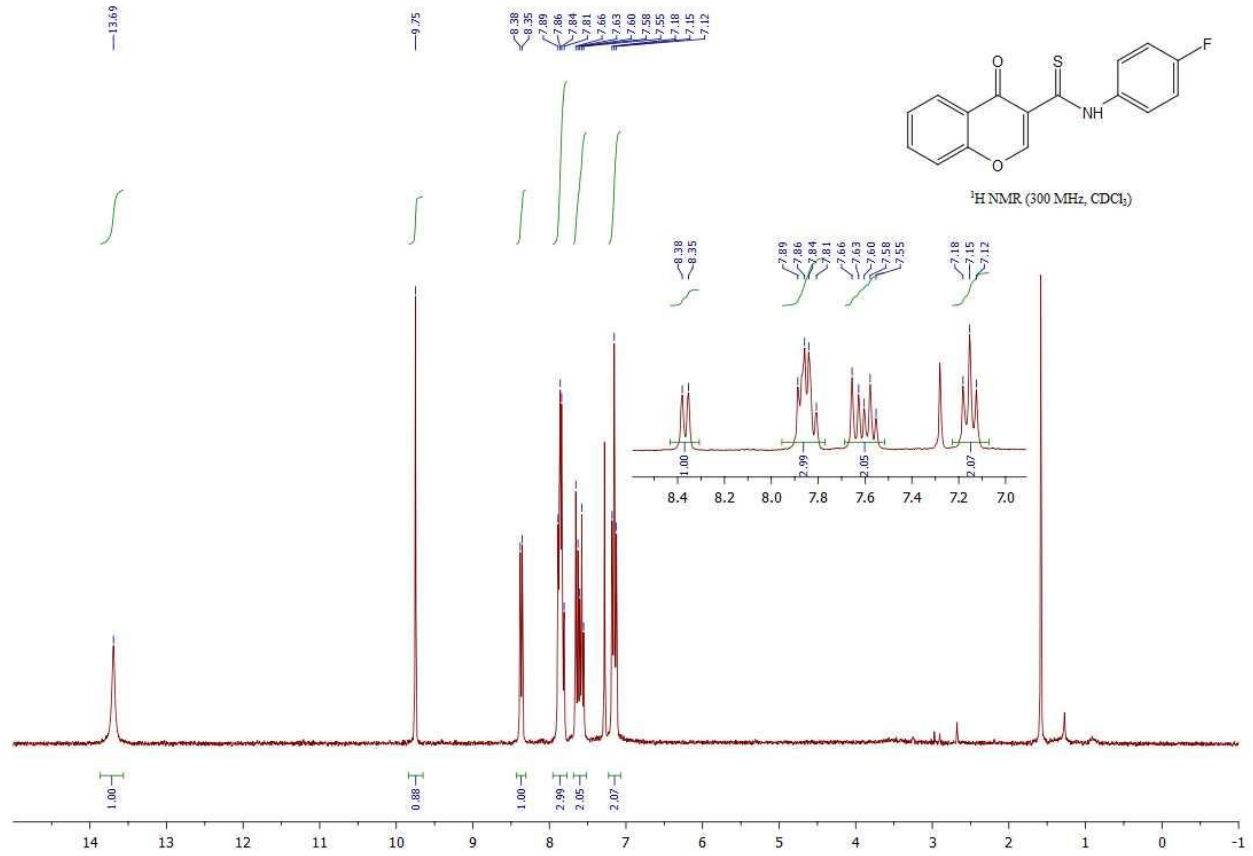
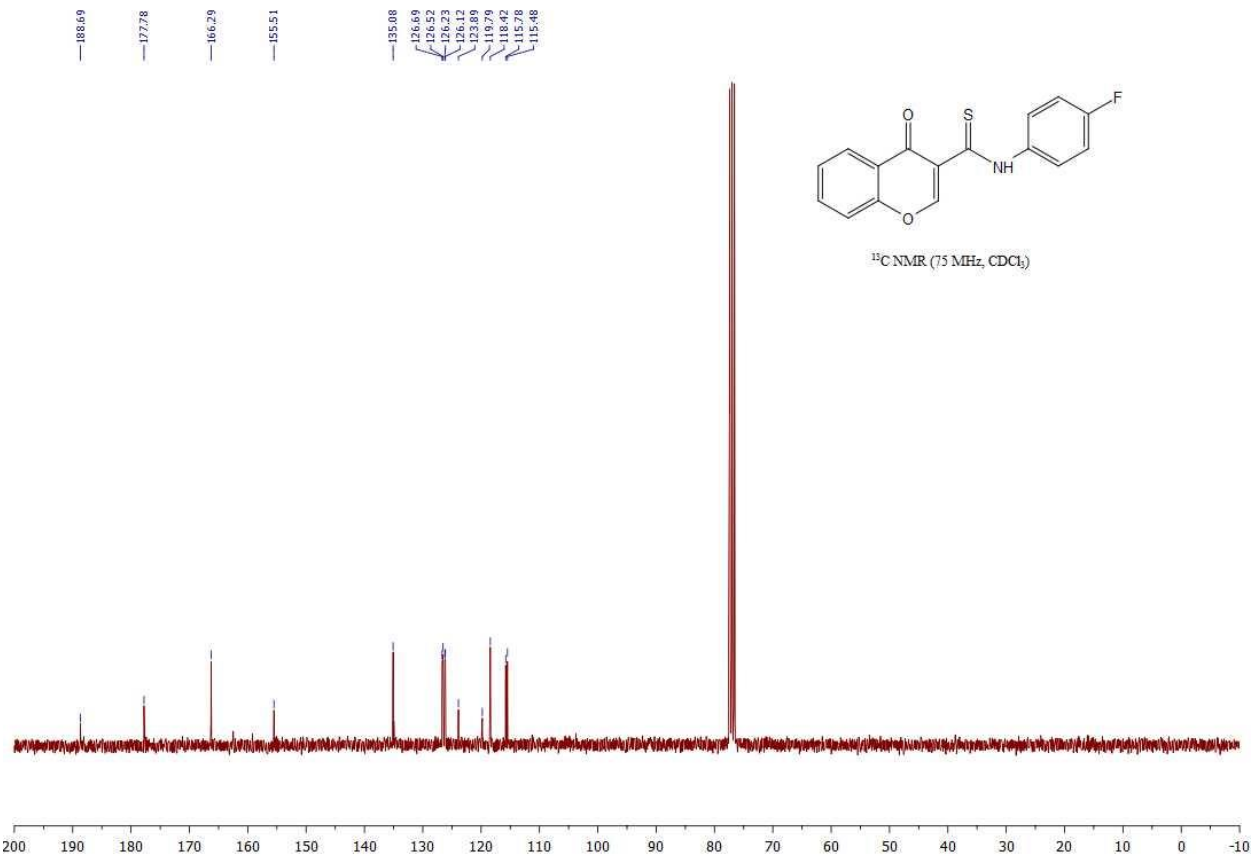
128.58
127.72
126.59
126.51
124.72
123.94
121.34
120.00
118.38
21.41
CH3
O
S
NH
O
13C NMR (75 MHz, CDCl3)

8d

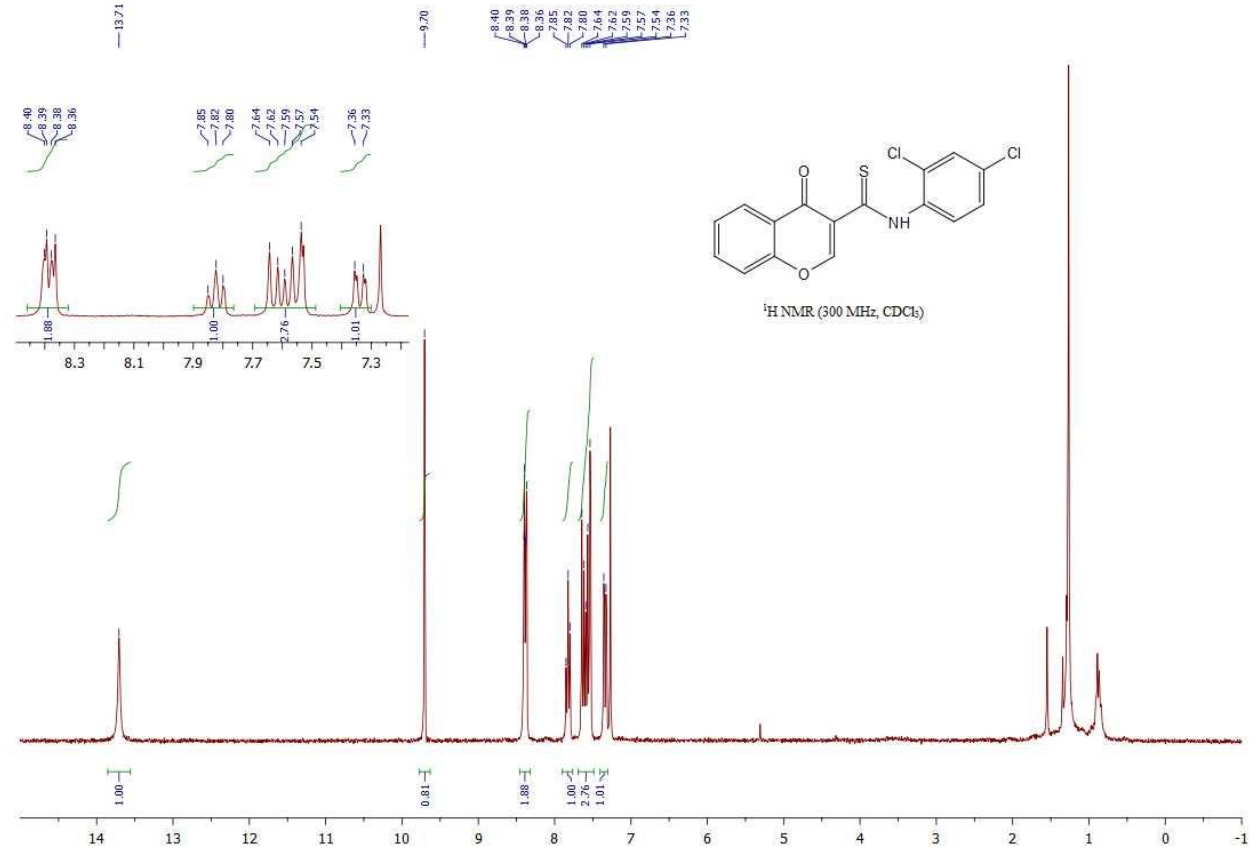


^{1}H NMR (300 MHz, $CDCl_3$)

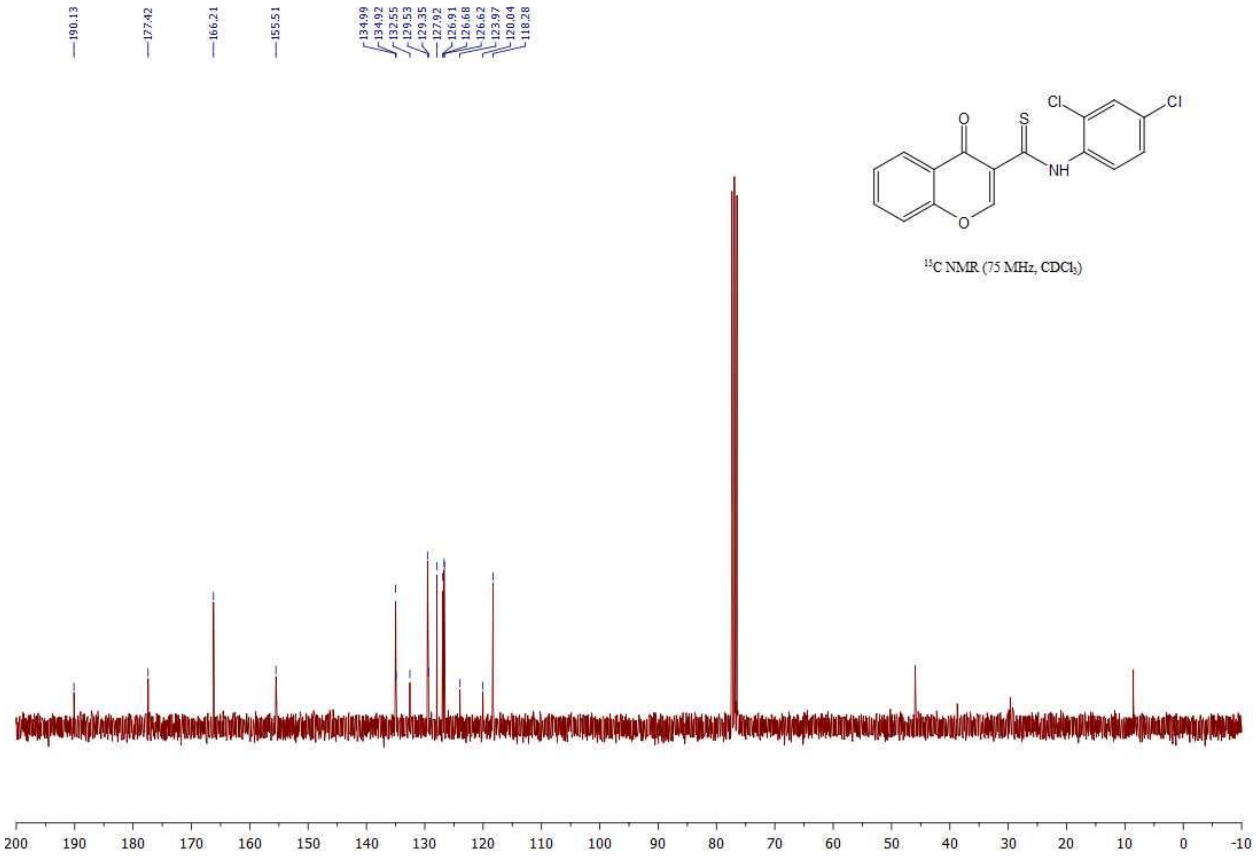


^{13}C NMR (75 MHz, $CDCl_3$)

8e

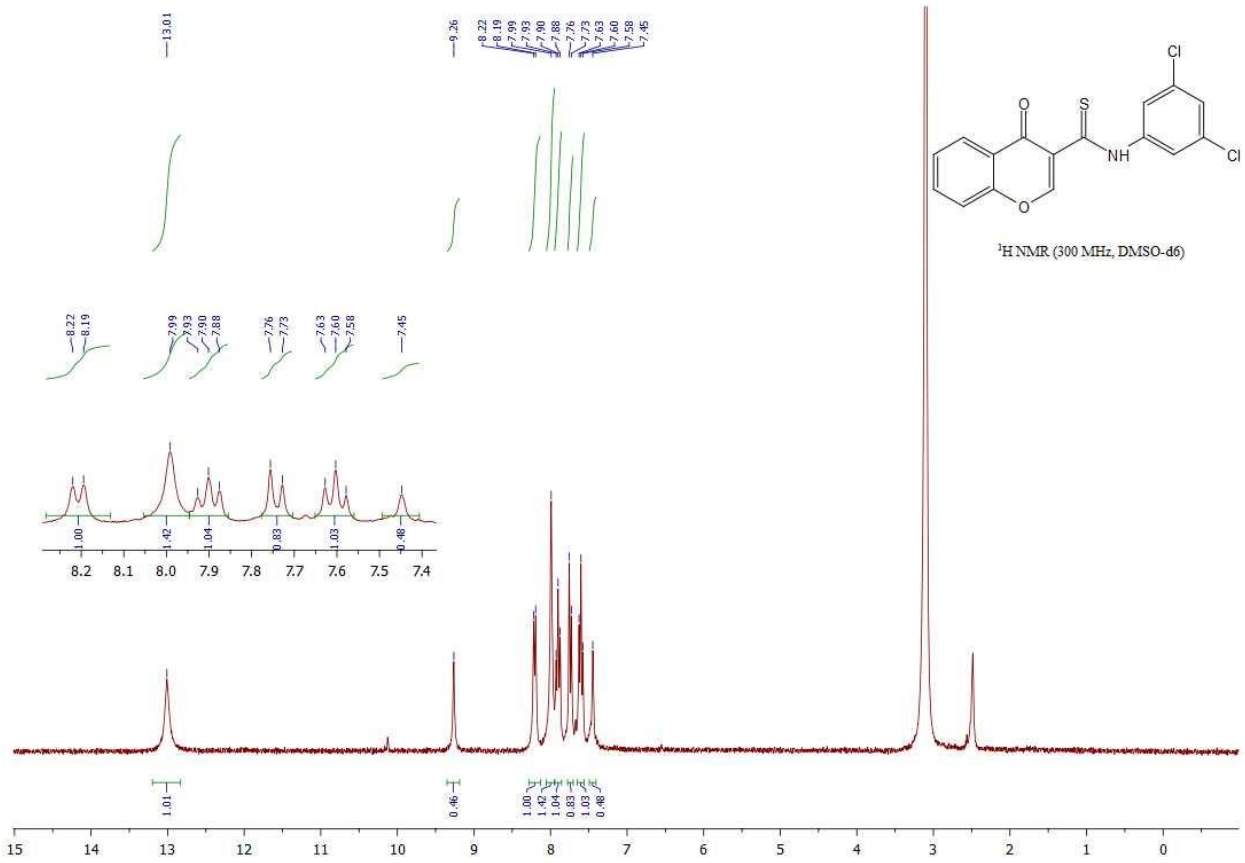


^{1}H NMR (300 MHz, $CDCl_3$)

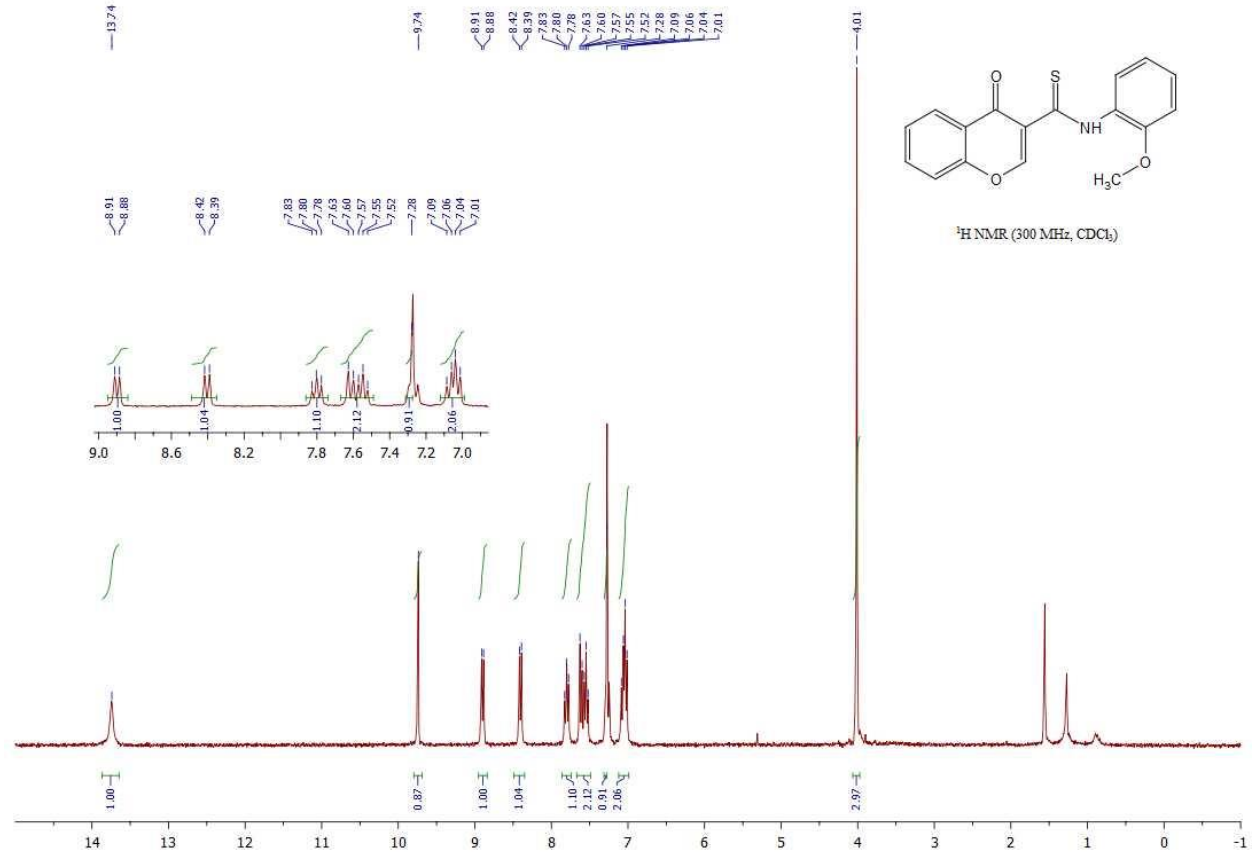


^{13}C NMR (75 MHz, $CDCl_3$)

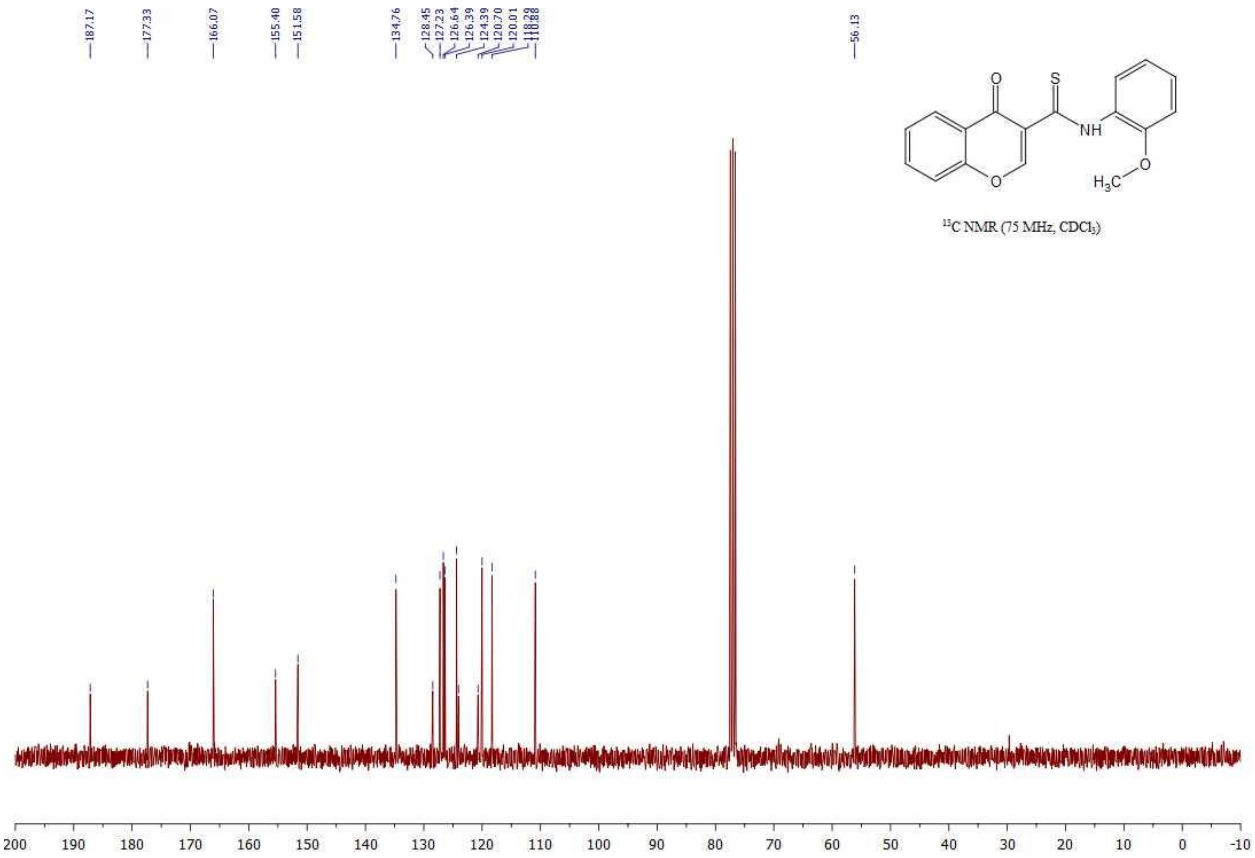
8f


13.01
9.26
8.22
8.19
7.99
7.93
7.90
7.88
7.76
7.73
7.63
7.60
7.58
7.45
Cl
O
S
NH
Cl
O
1H NMR (300 MHz, DMSO-d6)
1.00
1.42
1.04
0.83
1.03
0.48
8.2
8.1
8.0
7.9
7.8
7.7
7.6
7.5
7.4
1.01
0.46
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0

8g

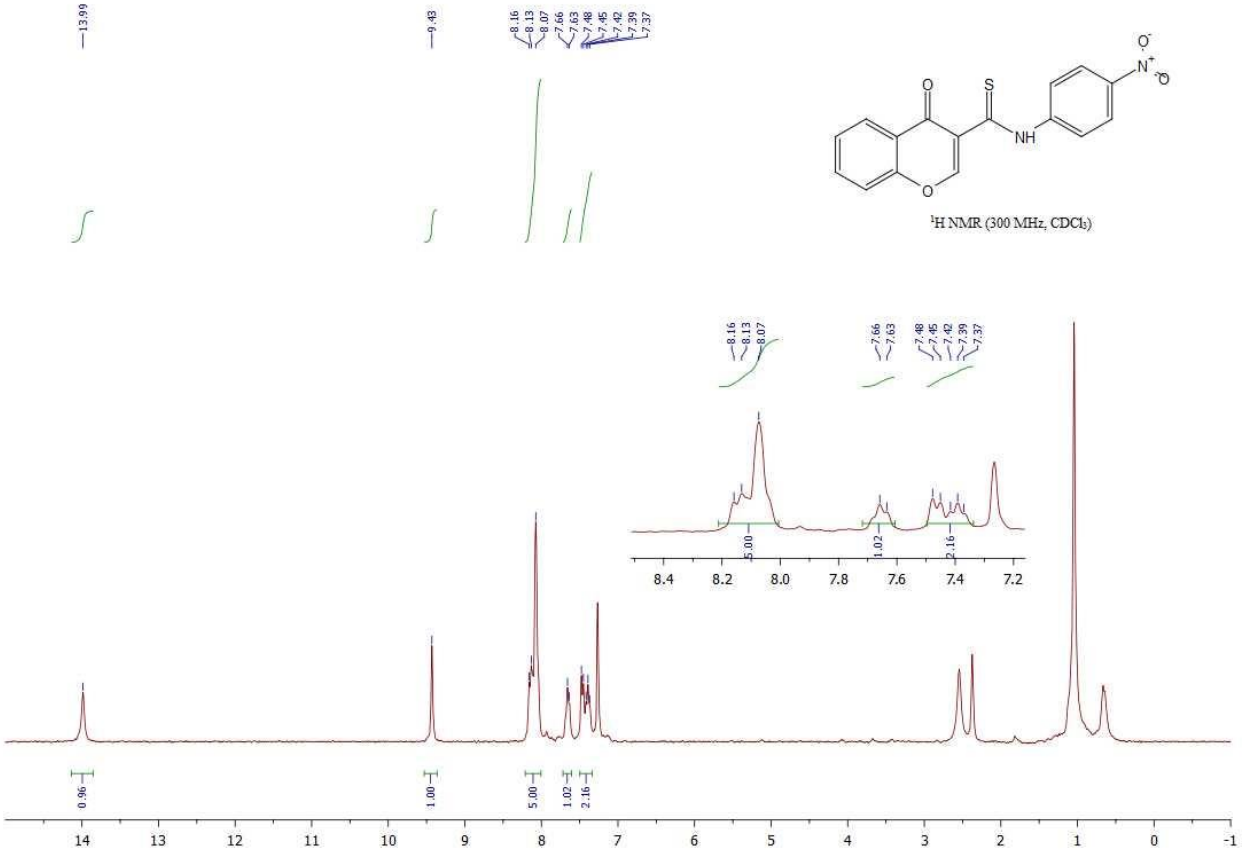


^{1}H NMR (300 MHz, $CDCl_3$)

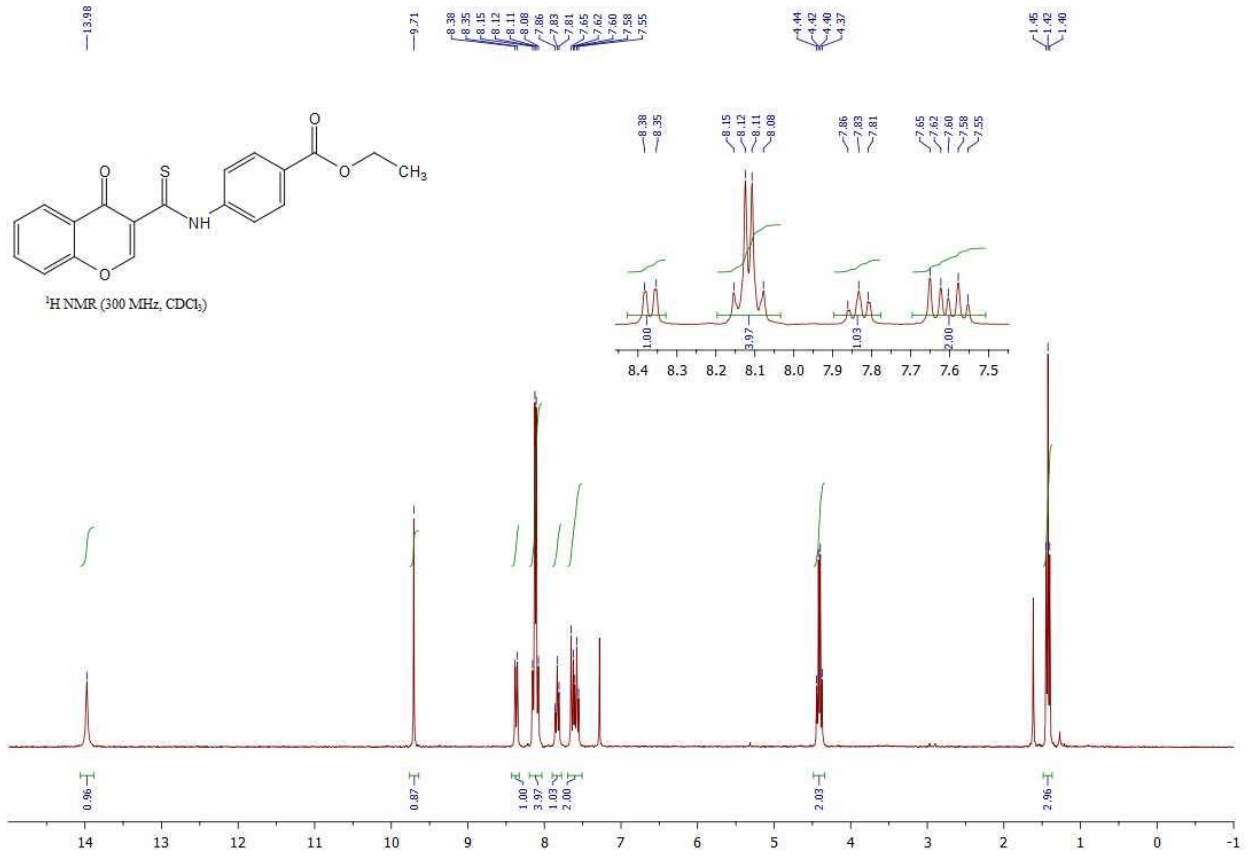


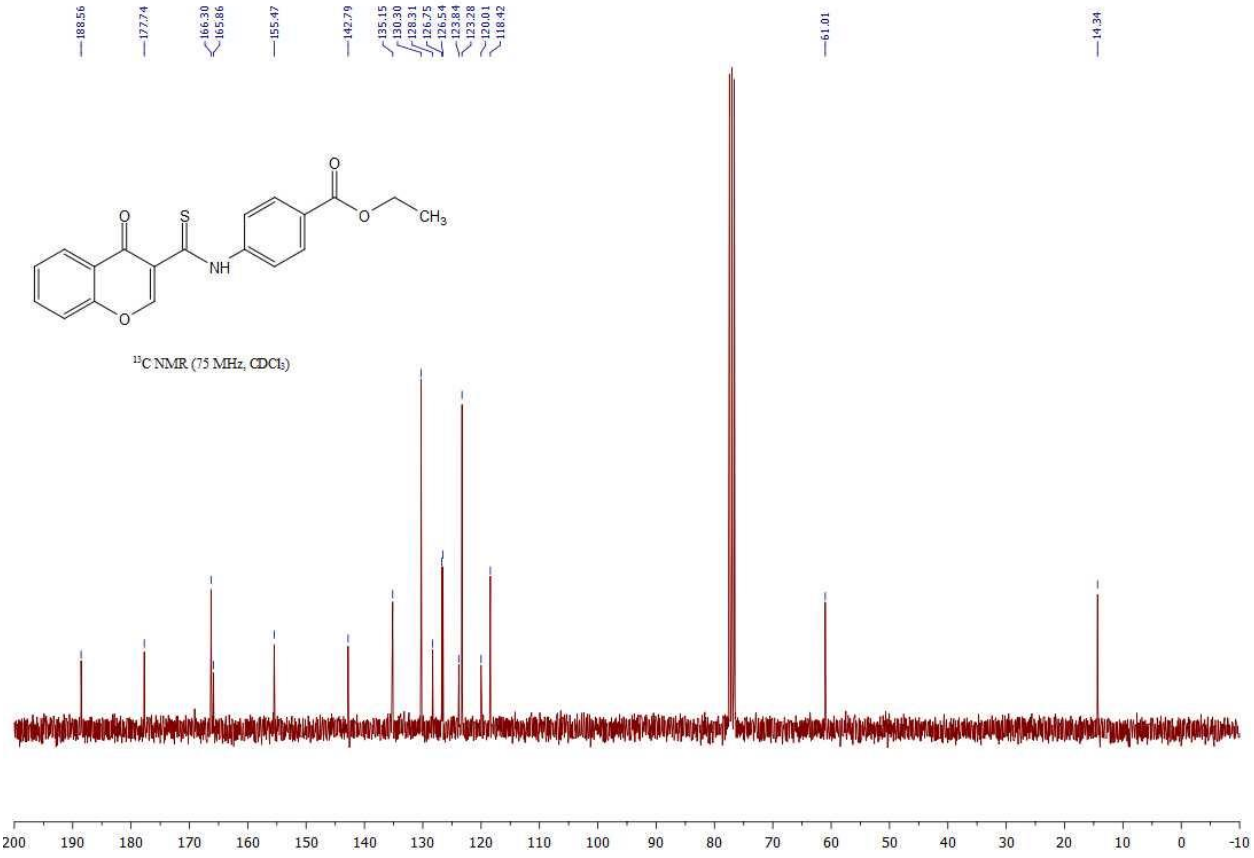
^{13}C NMR (75 MHz, $CDCl_3$)

8h

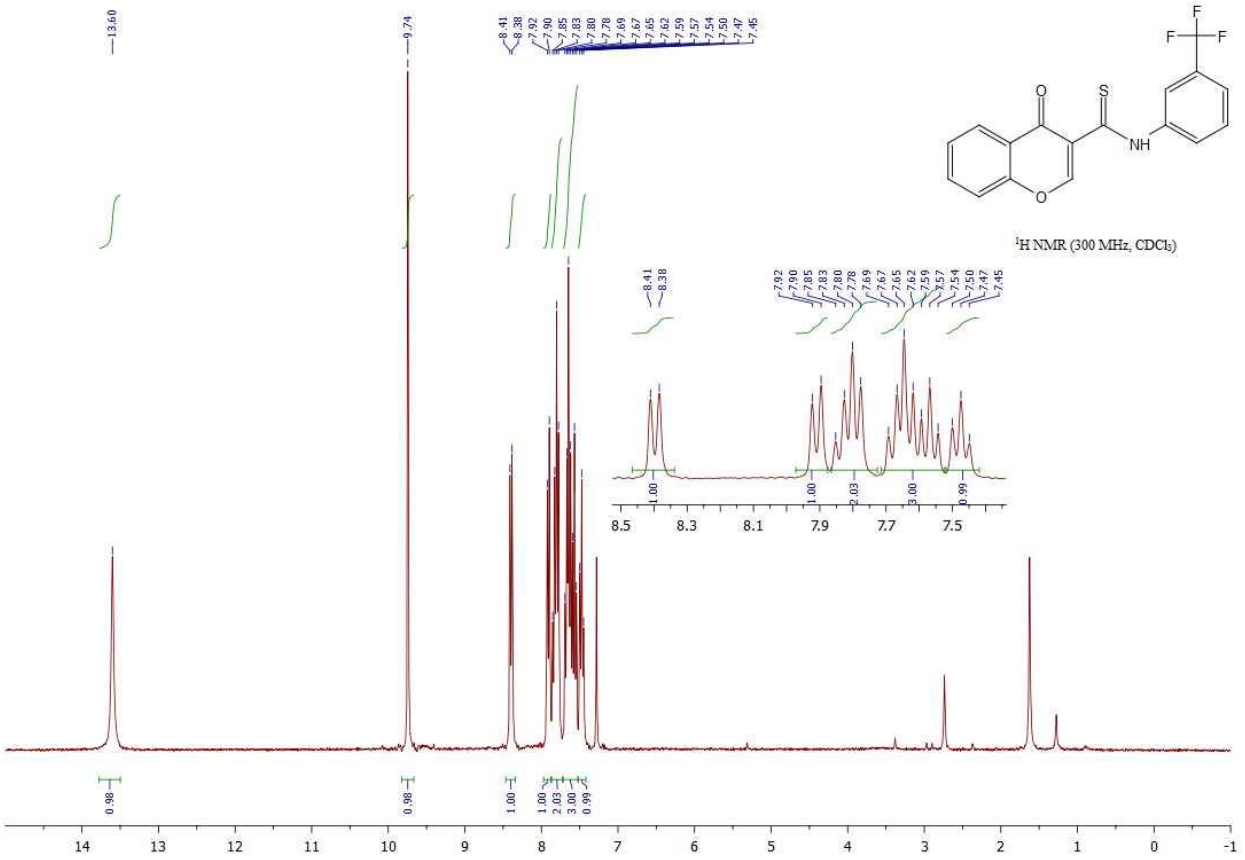

13.99
9.43
8.16
8.13
8.07
7.66
7.63
7.48
7.45
7.42
7.39
7.37
O
S
NH
O
N+
O-
O-
1H NMR (300 MHz, CDCl3)
0.96
1.00
5.00
1.02
2.16
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
8.4
8.2
8.0
7.8
7.6
7.4
7.2

8i

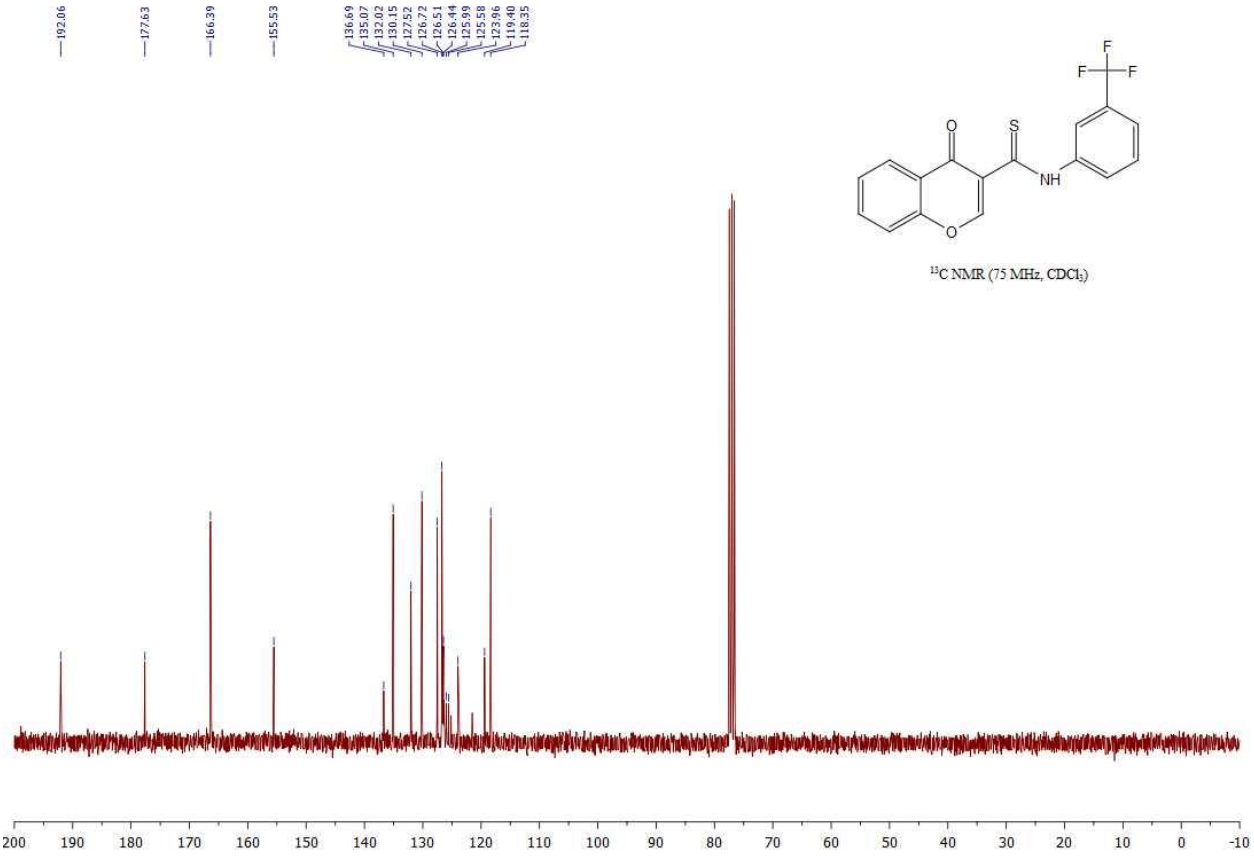

13.98
9.71
8.38
8.35
8.15
8.12
8.11
8.08
7.86
7.83
7.81
7.65
7.62
7.60
7.58
7.55
4.44
4.42
4.40
4.37
1.45
1.42
1.40
O
S
NH
O
O
CH3
O
1H NMR (300 MHz, CDCl3)
1.00
3.97
1.03
2.00
0.96
0.87
2.03
2.96


188.56
177.74
166.30
165.86
155.47
142.79
135.15
130.30
128.31
126.75
126.54
123.84
123.28
120.01
118.42
61.01
14.34
O
S
NH
O
O
CH3
O
13C NMR (75 MHz, CDCl3)

8j

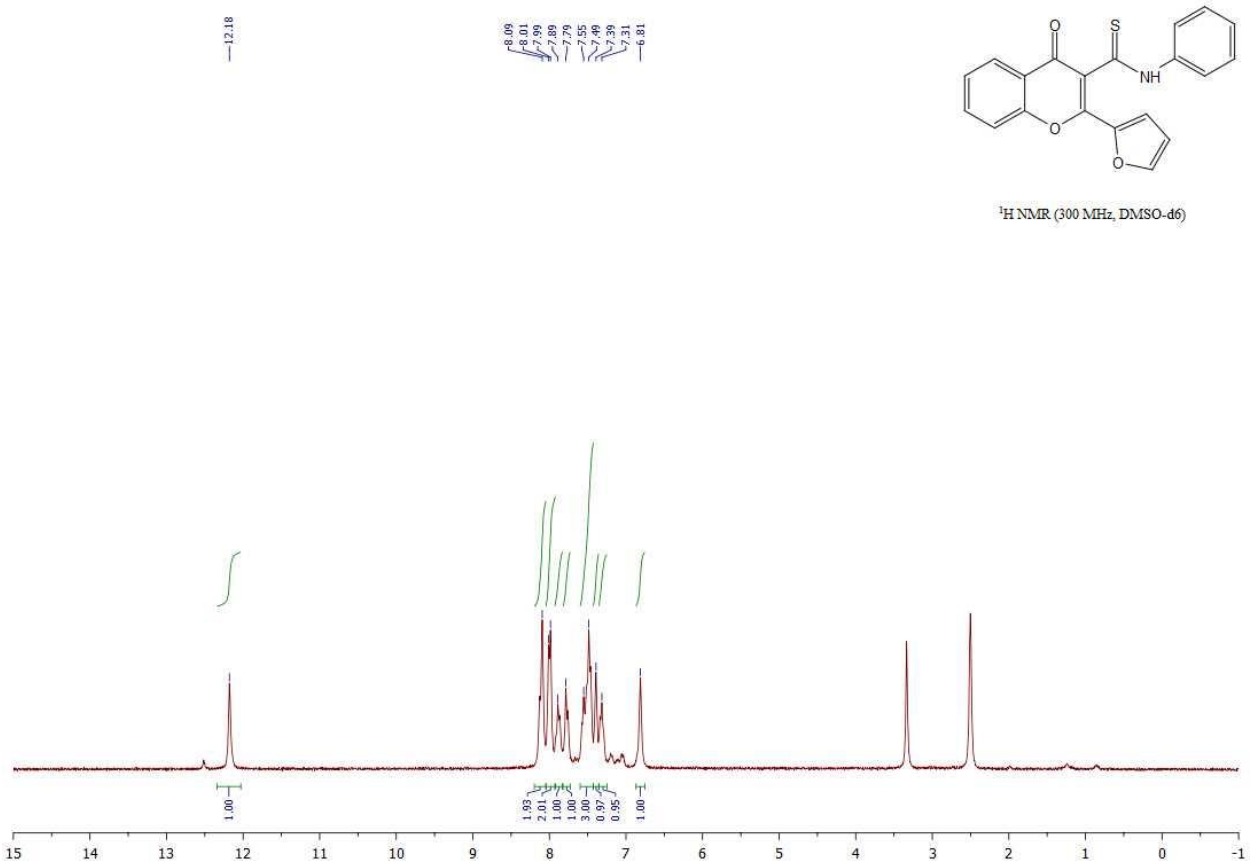


^{1}H NMR (300 MHz, $CDCl_3$)

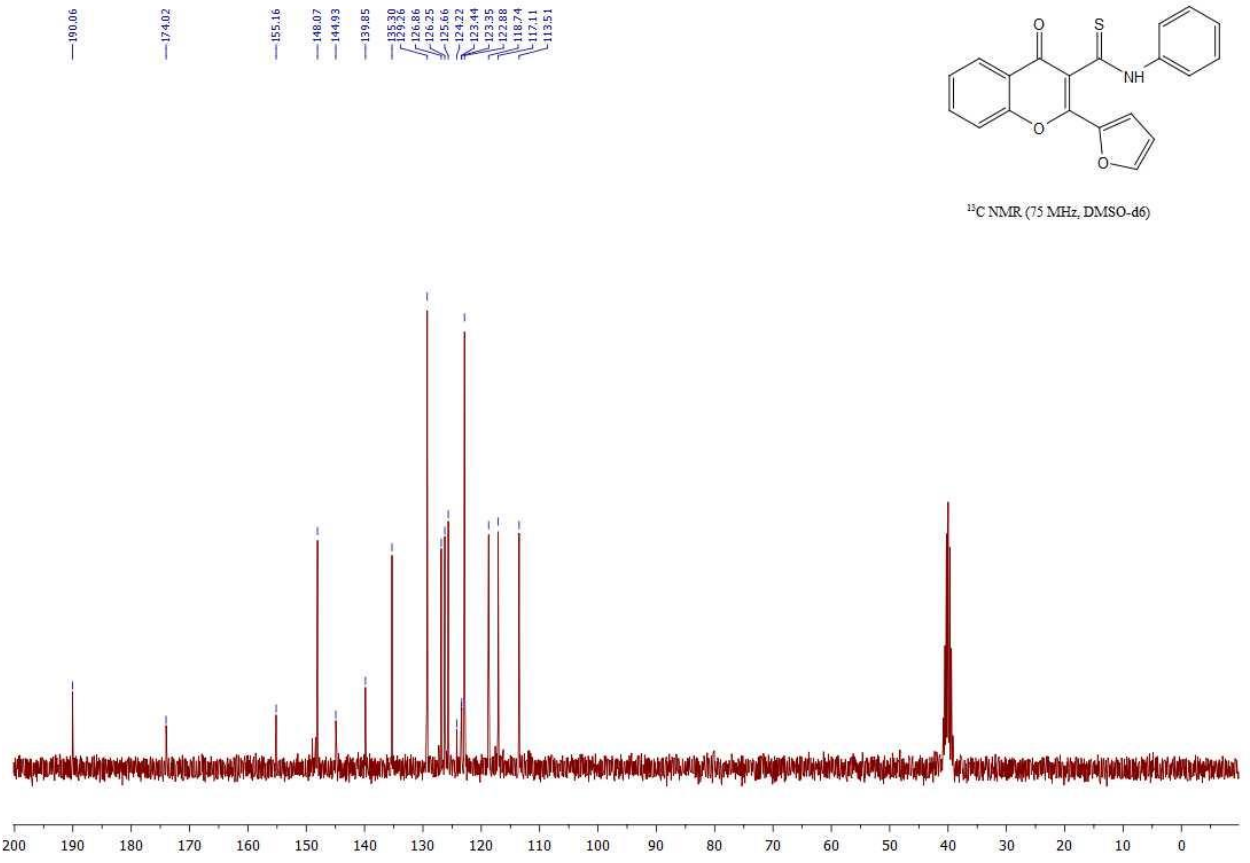


^{13}C NMR (75 MHz, $CDCl_3$)

13a

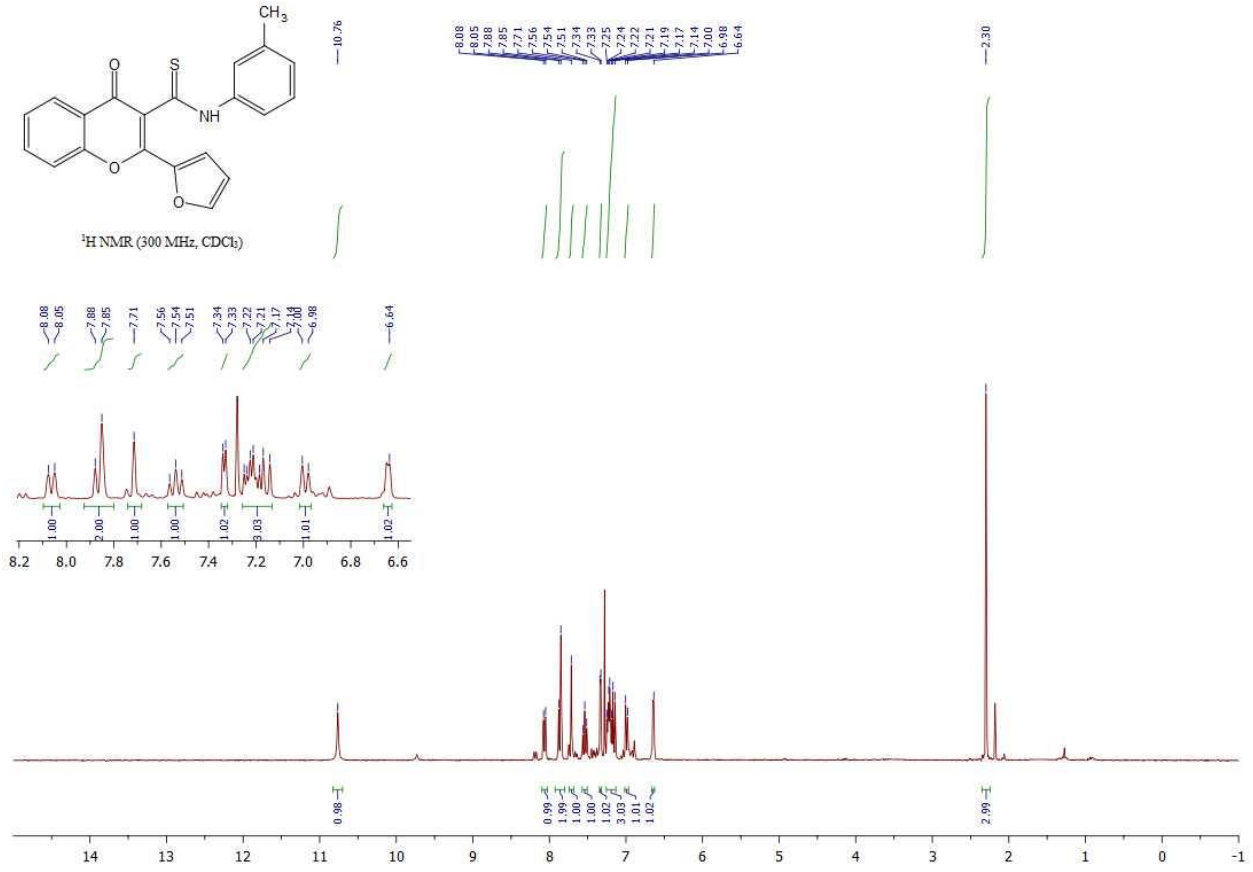


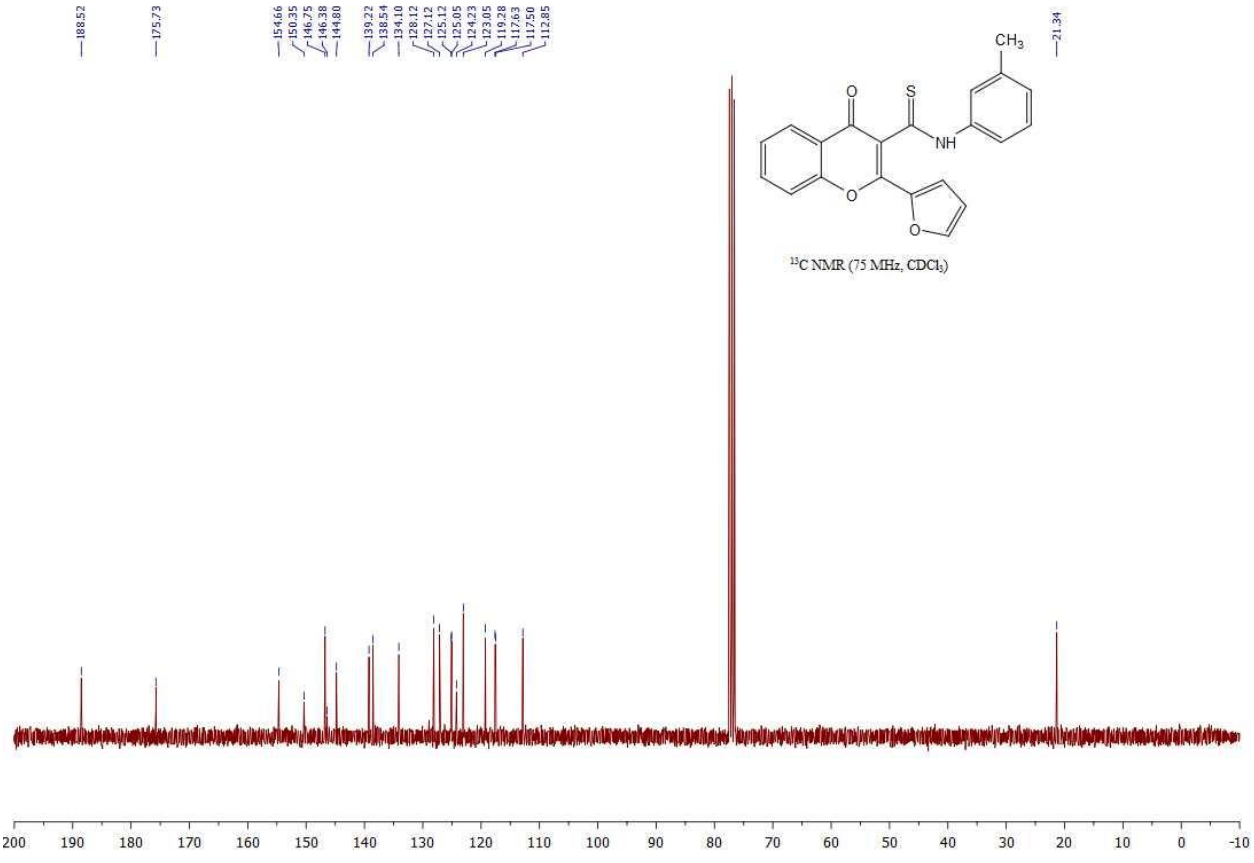
^{1}H NMR (300 MHz, DMSO-d6)



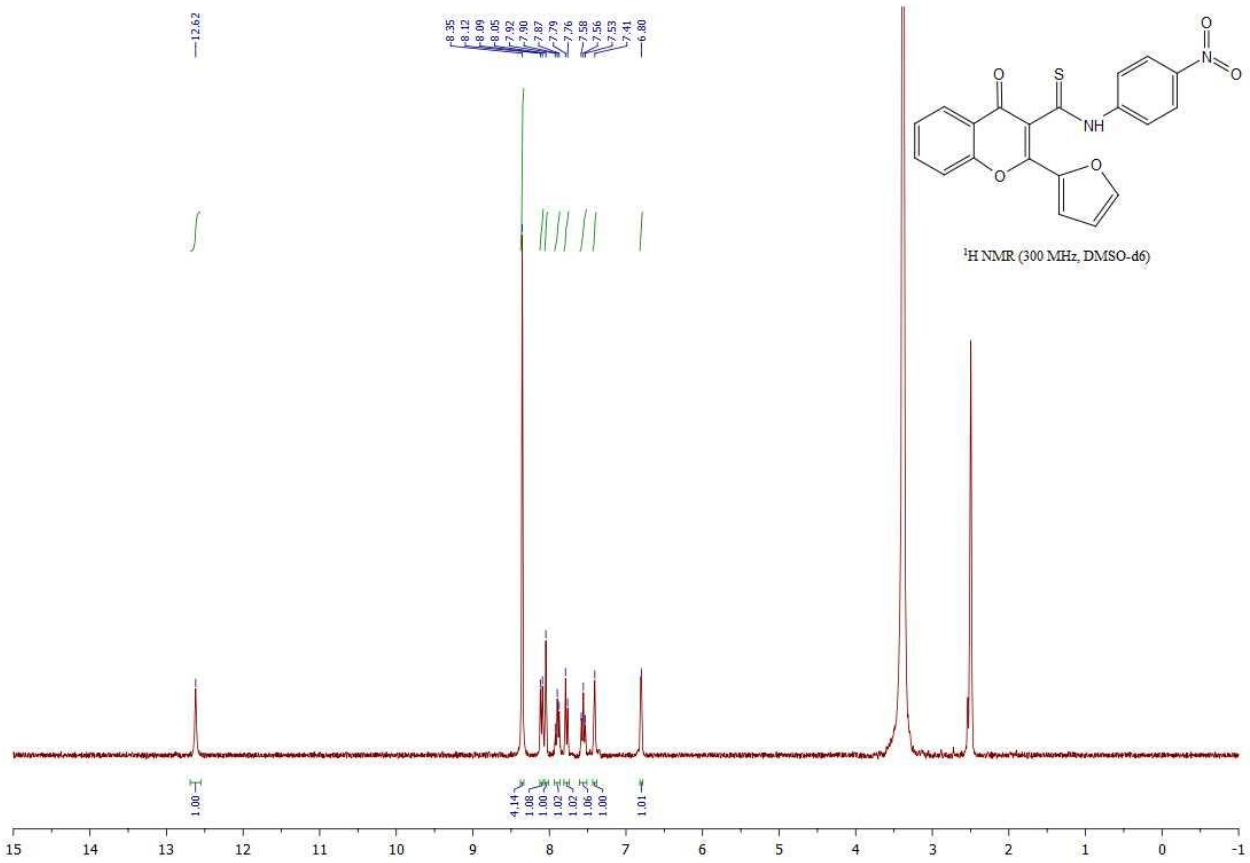
^{13}C NMR (75 MHz, DMSO-d6)

13b

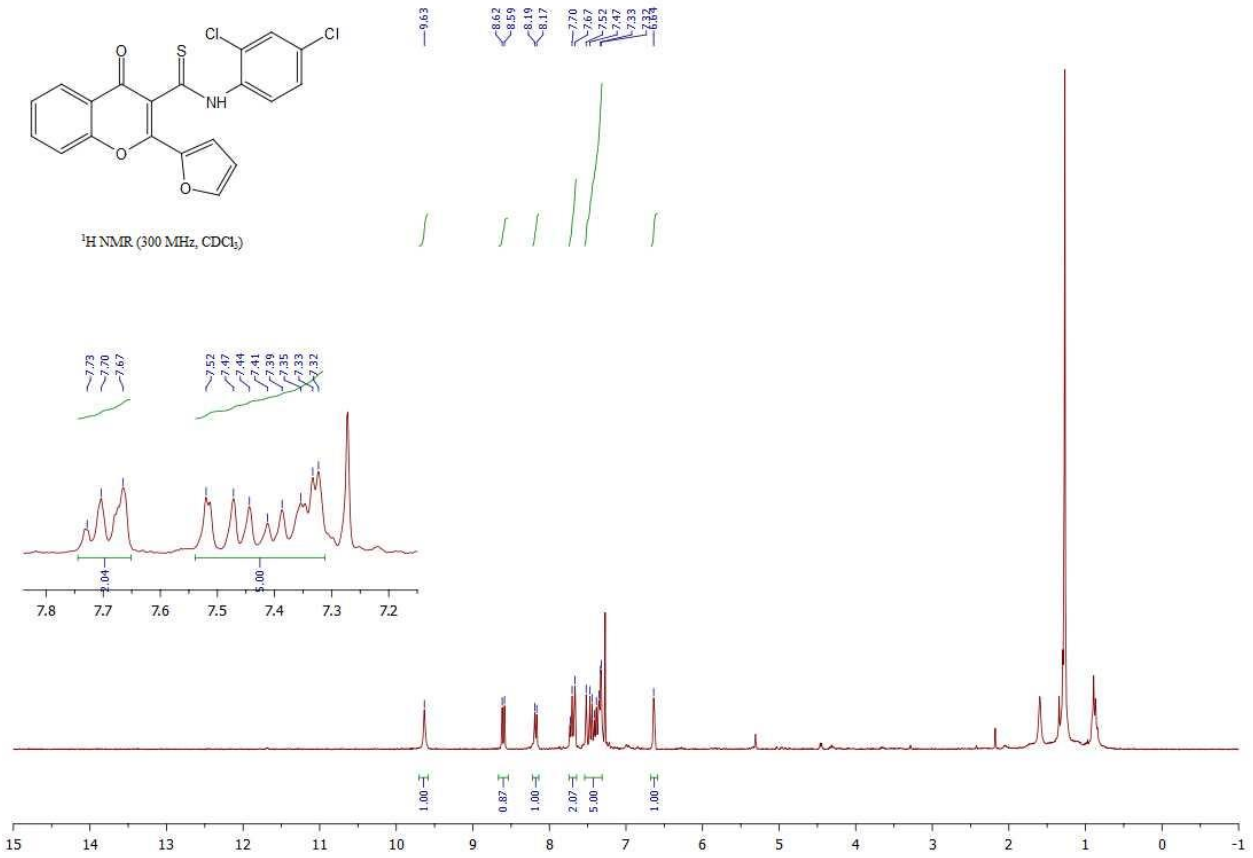

CH3
O
S
NH
O
O
1H NMR (300 MHz, CDCl3)


CH3
O
S
NH
O
O
13C NMR (75 MHz, CDCl3)

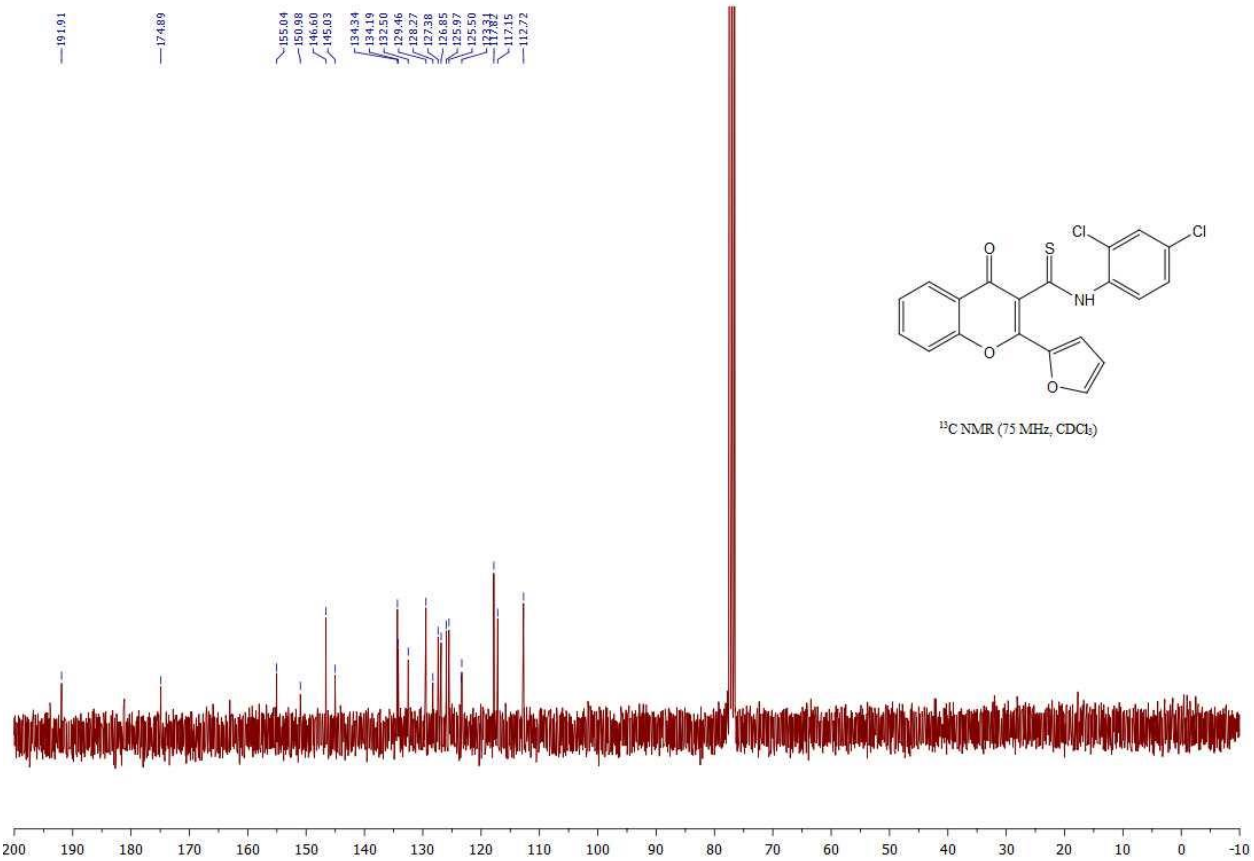
13c


12.62
8.35
8.12
8.09
8.05
7.92
7.90
7.87
7.79
7.76
7.58
7.56
7.53
7.41
6.80
O
S
NH
O
O
O
N
O
1H NMR (300 MHz, DMSO-d6)
1.00
4.14
1.08
1.00
1.02
1.02
1.06
1.00
1.01
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1

13d

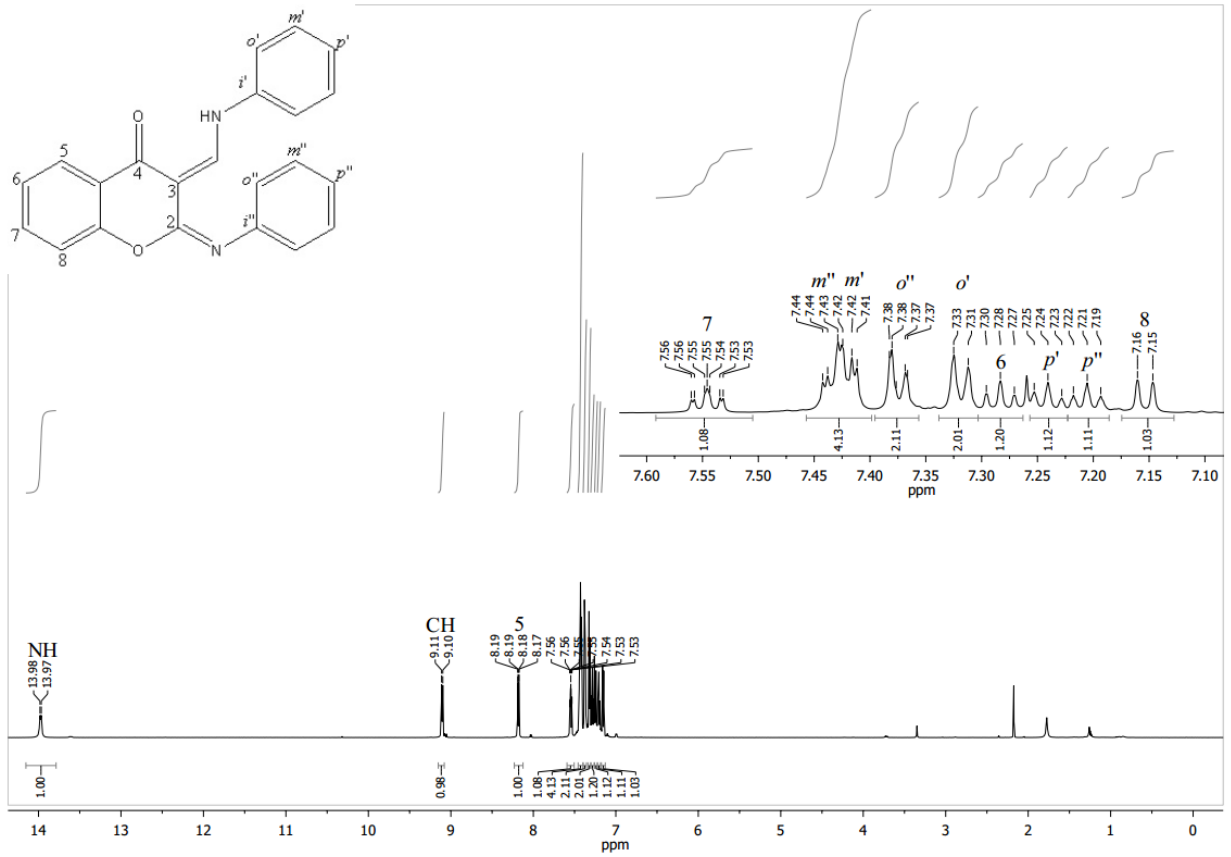


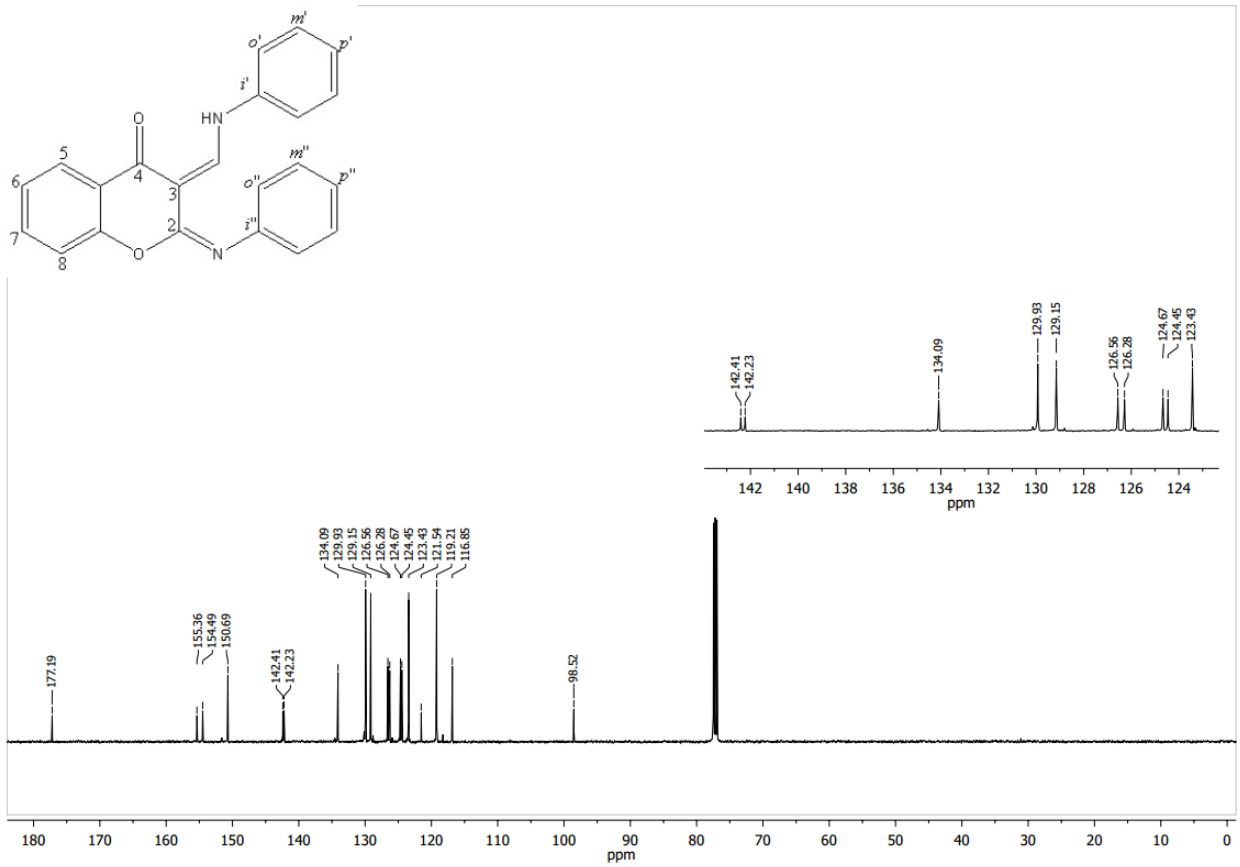
^{1}H NMR (300 MHz, $CDCl_3$)

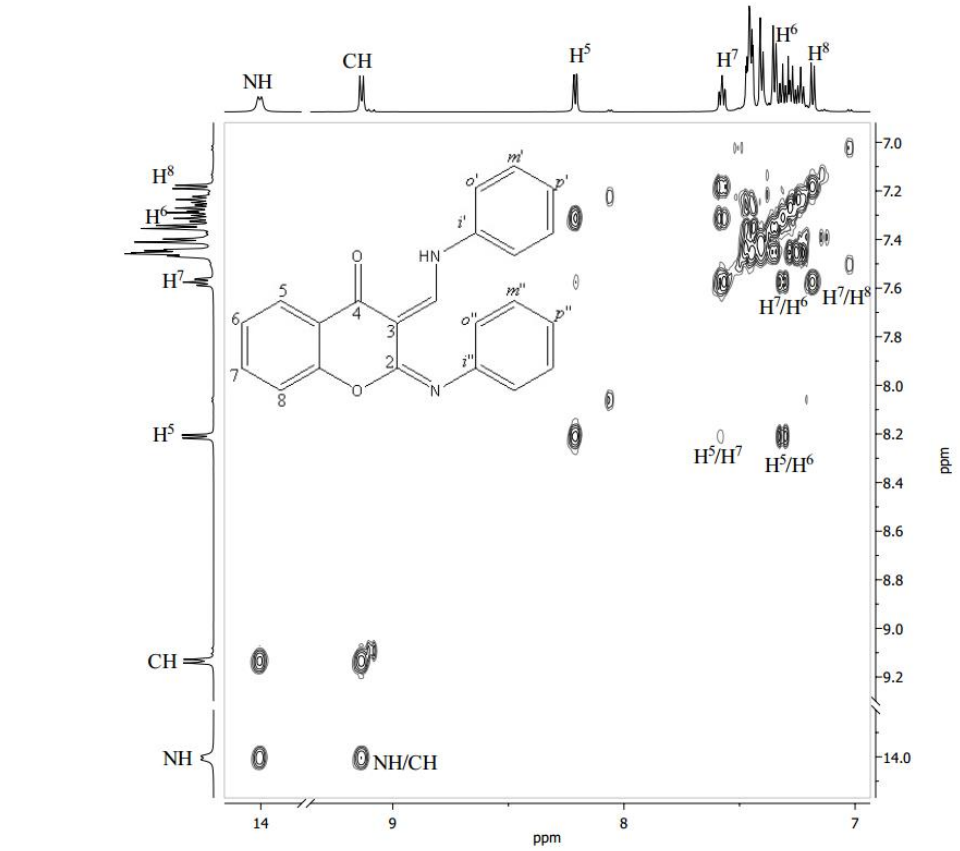


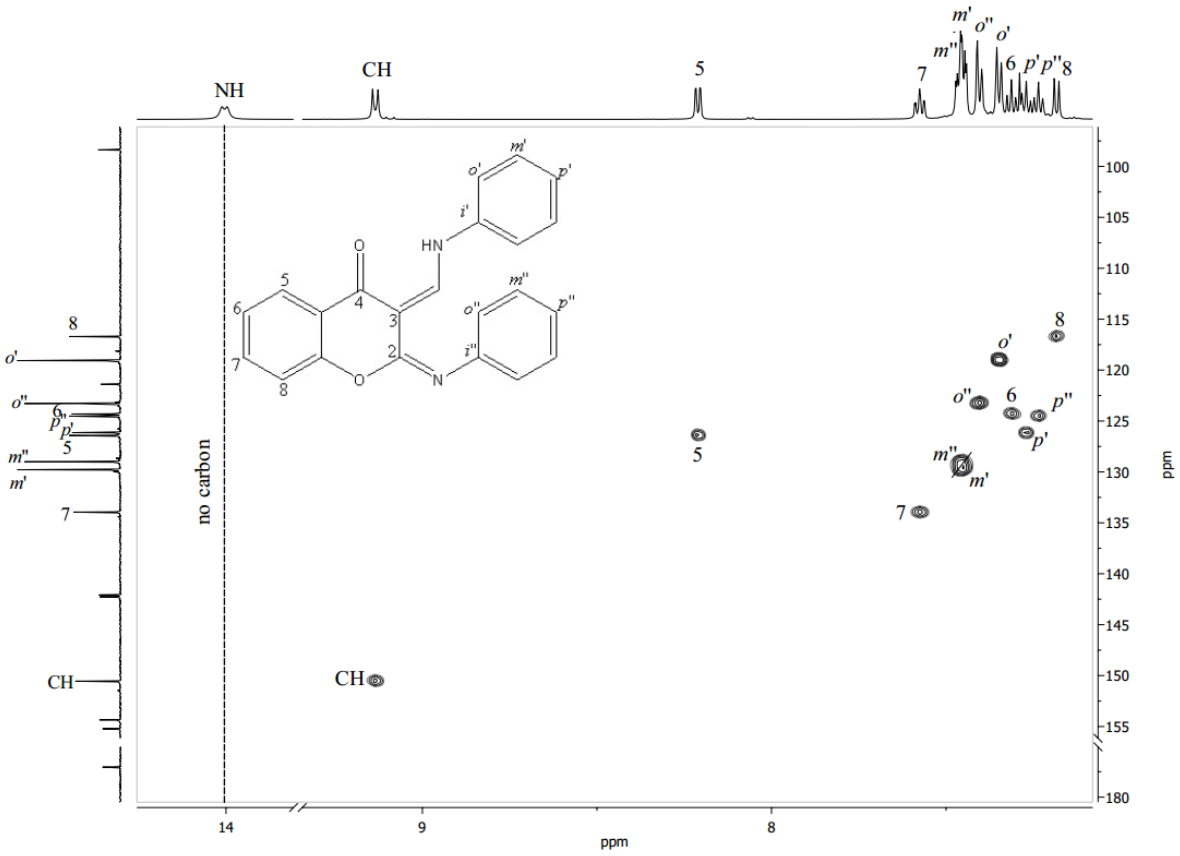
^{13}C NMR (75 MHz, $CDCl_3$)

10


NH
CH
5
7
6
m''
m'
o''
o'
p'
p''
8
ppm


177.19
155.36
154.49
150.69
142.41
142.23
134.09
129.93
129.15
126.56
126.28
124.67
124.45
123.43
121.54
119.21
116.85
98.52
ppm


NH
CH
H5
H7
H6
H8
H7/H6
H7/H8
H5/H7
H5/H6
NH/CH
ppm


NH
CH
5
7
no carbon
CH
ppm