

SUPPORTING INFORMATION

Discovery of Inhibitors of *Burkholderia pseudomallei* Methionine Aminopeptidase with Antibacterial Activity

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1. Sequence Alignment

M.Tuberculosis MetAp1	-----	
B.Pseudomallei MetAp1	-----	
Human MetAp2	MAGVVEVAASGSHLNGDLDPDDRREGAATAEEAAKKRRKKKKSKGPSA 50	
M.Tuberculosis MetAp1	-----	
B.Pseudomallei MetAp1	-----	
Human MetAp2	AGEQEPDKESGASVDEVARQLERSALEDKERDEDEDGDDGGDGATGKKK 100	
M.Tuberculosis MetAp1	-----MASMPSTRALSPGULSPTRPVPNWIAPEYVVGKPAAQEGSEPWVO 45	
B.Pseudomallei MetAp1	-----MRNSPHFRHRLMAITLKNED----- 21	
Human MetAp2	KKKKKKRGPKVQTDPPSVPICDLYPNGVFPKQGECEYPTQDGRATAAWRT 150	
M.Tuberculosis MetAp1	TPE-----VIEKMRVAGHIAAGALAEAGKAVAPGVTTDELDRIAHEYL 88	
B.Pseudomallei MetAp1	-----IAQMRIACRLASEVLDYITPTFVAGVTTGELDRLCHEYM 60	
Human MetAp2	TSEKKALDQASEEIWNDFREAAEHQVRKYVMSWIKPGMTMIEICEKL 200	
M.Tuberculosis MetAp1	VDN-GAYPSTLGYKG-----FPKSCCTSLNEVICHGIE---DSTVITDGD 129	
B.Pseudomallei MetAp1	THVQGITVPAPLNYQPPGYPPFPAICTSVNDVICHGIE---GEKTLKNGD 107	
Human MetAp2	EDCSRKLKENGKLNAG-----LAFPTGCSLNNCAHYTNAGDITVLQYDD 246	
M.Tuberculosis MetAp1	IVNIDVTAYIGGVHGDNTATFPAGDVADHRLVVDRTREATMRAINTVKP 179	
B.Pseudomallei MetAp1	ALNIDITVIKNGYFGDTSRMFIIGEGSILAKRLVQITTYECMWLGIDQVRP 157	
Human MetAp2	ICKIDFGTHISGRIIDCAFTVTFN---PKYDILLKAVKDATNTGKICAGI 293	
M.Tuberculosis MetAp1	GRALSVIGRVIESYAN-----RFGYNVVRDFTGHCIGITTFHNLGVV 220	
B.Pseudomallei MetAp1	GAHIGDIGHAIQKHAE-----AQGYSVVREYCGHIGITVFHEDPQV 198	
Human MetAp2	DVRLCDVGEAQQEVMESYEVEIDGKTYQVKPIRNLNHSGIQYRIHAGKT 343	
M.Tuberculosis MetAp1	LHYDQPAVETIMQPGMTFTIEPMINLGA----- 248	
B.Pseudomallei MetAp1	VHYGRPGTGLELKPMTFTIEPMINAGK----- 226	
Human MetAp2	VPIVKGGEATRMEEGEVYALTFGSTGKGVVHDDMECSHYMKNFVGHVP 393	
M.Tuberculosis MetAp1	-----LDYEIWDD 256	
B.Pseudomallei MetAp1	-----RDIRTMPD 234	
Human MetAp2	IRLPRTKHLNINENFGTLAFCRRLDRLGESKYLMAKLNCLGLGIVDP 443	
M.Tuberculosis MetAp1	GWTVVVKDRKWTIAQFHTILVVIDTGVEILTCL----- 288	
B.Pseudomallei MetAp1	QWTVKTRDRSLSAQWHTILVVIDTGHEVLTVSAGTFARPPTFAAPATA 282	
Human MetAp2	YPLCDIKGSYTAQFHTILLRPTCKEVVSRGDDY----- 478	

Table 1. Alignment of the amino acid sequences of *B. pseudomallei* MetAP1, human (*Homo sapiens*) MetAP2 and *M. tuberculosis* MetAP1. There are five conserved metal-ligating residues (Asp 131, Asp 142, His 205, Glu 238, and Glu 269; numbering based on PDBcode:3IU9 ;highlighted in yellow). Other identical residues among all three MetAPs are shaded grey.

2. Docking Studies

The amino acid sequence of *Burkholderia pseudomallei* methionine aminopeptidase (*BpMetAP*) was obtained from SSGCID. The structure of the mycobacterium tuberculosis methionine aminopeptidase 1 (PDB code: 3IU9)¹ was chosen as a structural template to model the *BpMetAP* structure for docking studies as the amino acids within the ligand binding site were found to be nearly identical to that of *BpMetAP*. A homology model of *BpMetAP* was generated using Sybyl (version 8.1, Tripos, St. Louis, MO USA) and Pymol (1.3r1 edu, Sept 2010). To provide a ligand template for docking studies, the crystal

structure of *Aeromonas proteolytica* aminopeptidase in complex with 8-quinolinol (PDB code: 3VH9),² was aligned to the active site of the MtMetAp/BpMetAp model using active site atoms. Prior to the docking experiments, water was removed from the model. All docking was performed in Sybyl (version 2.0, Tripos, St. Louis, MO USA). The site targeted in the docking calculations were defined by the positions of 8-quinolinol with penalty for deviating from the template fragment of 95%. The energy minimization was performed to generate final structures.

3. Assay protocols

Enzyme Activity Assay.

The fluorogenic peptide substrate, H-Met-Gly-Pro-7-amino-4-methylcoumarin hydrochloride (H-met-Gly-Pro-AMC), and recombinant human DPPIV/CD26 (rhDPPIV) were purchased from R&D Systems, Inc. (Minneapolis, MN). The purified recombinant Burkholderia Pseudomallei was obtained from Seattle Structural Genomics Center For Infectious Disease (SSGCID). 8-hydroxy-5-nitroquinoline and Furan inhibitors were purchased from Sigma Aldrich (St. Louis, MO) with purity of >95% and their purity and identity were confirmed by ¹H and ¹³C NMR. Triazole inhibitor and sulfonamide inhibitors were synthesized according to the procedure mentioned in the synthesis section.

The enzyme inhibition assay was performed similar to an established recombinant human methionine aminopeptidase 2 activity assay by Zhou et al.³ The assay was modified substituting H-Met-Gly-Pro-AMC and rhCD26/dppIV as the substrate and secondary enzyme, respectively. Enzymatic activity of *BpMetAP* was followed using a Synergy 2 Plate reader (Biotek, Winooski, Vt). All kinetic experiments were carried out on a Nunc flat-bottom maxisorp 96-well plates (Thermo Fisher Scientific, Rochester, NY). Each well contained 100 µL of assay mixture with 50 mM HEPES, 0.1 mM CoCl₂, 100 mM NaCl, pH 7.5, 250 µM H-Met-Gly-Pro-AMC, 0.100 µg rhCD26/ dppIV, and 0.5 µg apoenzyme in 1.0 mg/mL BSA in the assay buffer. Assays were initiated with the addition of the substrate. The reaction was incubated at room temperature for 30 minutes before the initial rates were recorded. The fluorescent signal was measured for 10 min, using excitation and emission wavelengths of 380 nm and 460 nm, respectively. The initial rates were corrected for the background rate determined in the absence of *BpMetAP*. For IC₅₀ determination, the inhibitors were screened at six or more concentrations.

Kinetic Analysis

The IC₅₀ values were calculated from nonlinear regression curve fitting of percent inhibitions as a function of inhibitor concentration to eq 1 reported by Grant et al⁴, using Origin 7 (OriginLab, Northampton, MA):

$$y = (A-D)/(1+(x/C)^B) + D; \quad (1)$$

where y is the initial rates with and without the inhibitor, x is inhibitor concentration, A is uninhibited enzyme activity, B is the slope of the inflection (Hill coefficient), C is the central inflection point (or IC_{50}), and D is the fully inhibited enzyme activity.

Bacterial Growth Inhibition Assay.

Potential inhibitor compounds were tested against *Burkholderia thailandensis* strain E264⁵ and *Burkholderia pseudomallei* strains 1026b⁶ and Bp340⁷. All manipulations of *B. pseudomallei* were done in a CDC-approved biosafety level 3 (BSL3) facility at the University of North Carolina. Bacteria were maintained on low-salt lysogeny broth (LSLB) agar plates (10 g/L tryptone, 5 g/L yeast extract, 1.25 g/L NaCl, 7.5 g/L agar) at 37°C. For the assay, bacterial cultures were prepared in 3 mL of LSLB medium and were incubated 16 h at 37°C with aeration. Cells were washed in PBS and a suspension was prepared at an $OD_{600} = 0.2$ in LSLB.

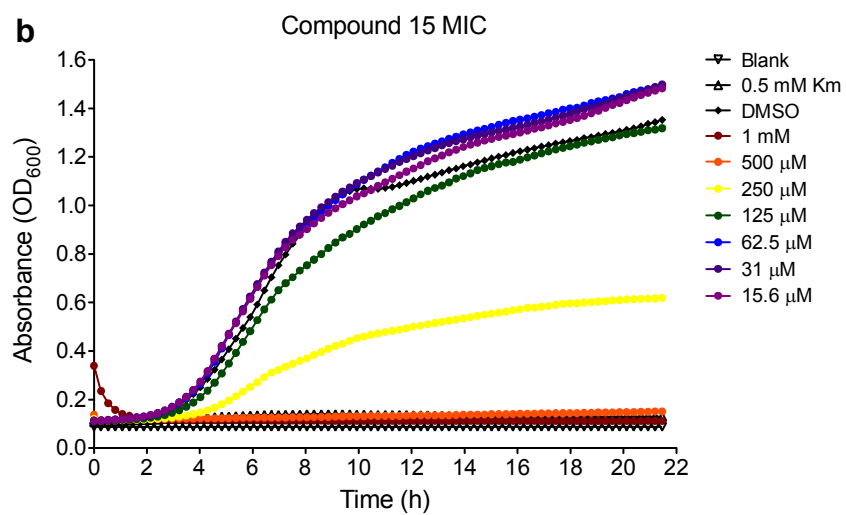
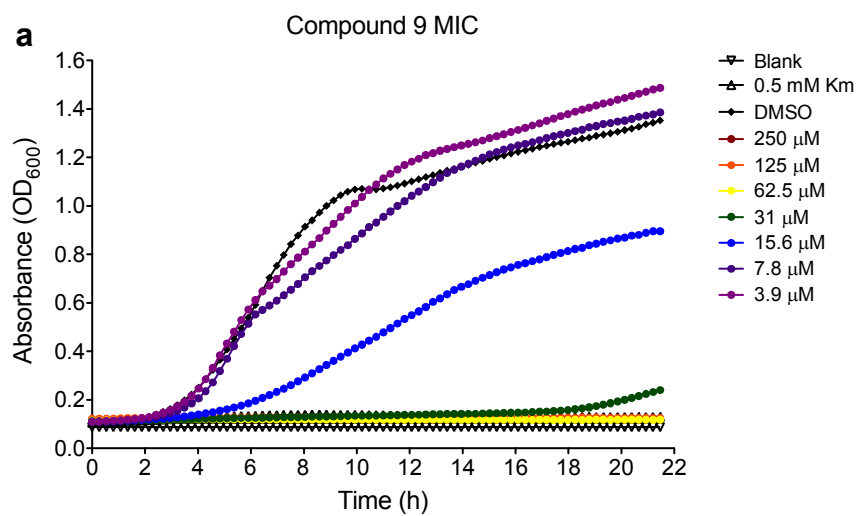
Stocks of each compound were made at 40 mM in DMSO and stored at -20°C. Compounds were diluted in LSLB to 1.25 mM, and 160 μ L of each diluted compound was added to wells of a 96-well microtiter plate in triplicate. To each well was added 40 μ L of bacterial suspension ($OD_{600} = 0.2$) for a final $OD_{600} = 0.04$ and a final inhibitor concentration of 1 mM. For some experiments, including determination of minimum inhibitory concentration (MIC) and minimum bactericidal concentration (MBC), two-fold dilutions of the compound were prepared in LSLB and the assay was performed as described above. As positive controls, bacteria were incubated in medium alone and in medium containing DMSO. As a negative control for growth, kanamycin (final concentration 300 μ g/mL) was added to the medium in certain wells. Plates were incubated approximately 22 h at 37°C with shaking in an Infinite M200 plate reader (Tecan Group Ltd., Männedorf, Switzerland). The OD_{600} was read every 15 min and was graphed as a function of time. The area under the curve (AUC) was determined for each growth curve using Prism 5 (GraphPad Software, Inc.). The MIC for compounds **9** and **15** was defined as the lowest concentration of compound at which no growth was observed (indicated by no change in the OD_{600} over the course of the assay). To assess the MBC for compounds **9** and **15**, 10 μ L aliquots from triplicate wells were spotted onto LSLB plates, and plates were incubated overnight at 37°C. The MBC was defined as the lowest concentration of compound at which no colonies were visible after 24 h of growth at 37°C.

Table 2. Growth inhibition of *B. thailandensis* by compounds 1-25

Compound	Growth as % E264 + DMSO ^a	% Inhibition ^b
Blank	0.00 ± 0.00	NA
DMSO	100.00 ± 0.00	NA
Km	3.0 ± 0.5	97.0 ± 0.5
1	91 ± 8	9 ± 8
2	4.2 ± 0.7	95.8 ± 0.7
3	5.8 ± 0.8	94.2 ± 0.8
4	48 ± 3 ^c	52 ± 3
5	6 ± 2	94 ± 2
6	7.1 ± 0.7	92.9 ± 0.7
7	38 ± 8 ^c	62 ± 8
8	8.7 ± 0.5	91.3 ± 0.5
9	16 ± 5	84 ± 5
10	98 ± 8	2 ± 8
11	25 ± 2 ^c	75 ± 2
12	39 ± 8 ^c	61 ± 8
13	88 ± 2 ^c	12 ± 2
14	56 ± 5 ^c	44 ± 5
15	4.3 ± 0.5	95.7 ± 0.5
16	82.5 ± 0.6	17.5 ± 0.6
17	74 ± 2	26 ± 2
18	72.1 ± 0.9	27.9 ± 0.9
19	84 ± 5	16 ± 5
20	97 ± 8	3 ± 8
21	90 ± 6	10 ± 6
22	98.3 ± 0.2	1.7 ± 0.2
23	100.5 ± 0.3	0.00 ± 0.00
24	78.1 ± 0.7	21.9 ± 0.7
25	84.5 ± 0.3	15.6 ± 0.3

^aGrowth calculated as AUC relative to blank. ^bPercent inhibition determined relative to DMSO.

^cCompounds showed inhibition of growth, but had high background (OD₆₀₀ > 0.2) due to solubility limitations.



MIC determination for compounds **9** and **15** against *B. thailandensis* E264.

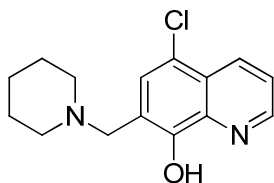
4. Synthesis and Characterization of the compounds

Chemistry. All reagents and solvents were purchased from Sigma Aldrich (St. Louis, MO). For chromatography, silica gel TLC plates with UV254 from Sorbent Technologies were used (Atlanta, GA). Carousel reactions were performed on carousel 12 plus reaction station from Radleys Discovery Technologies (Essex, United Kingdom). Purification with HPLC was performed using Biotage Isolera One separation system (Charlotte, NC) with Biotage SNAP 50 g column using a gradient of Ethyl Acetate/Hexane as eluant and UV detection at 254/280nm. For analytical purposes, HPLC data were obtained from DELTA HPLC SYSTEM XXI. ESI MS mass spectra were taken on a Finnigan Mat LCQ Mass spectrometer. Elemental analysis was performed by PerkinElmer 2400 Series II CHN Elemental Analyzer. ^1H NMR and ^{13}C NMR were measured at 300 MHz, respectively, on Avance III (Bruker Biospin from Billenca, using BBFO probe, 5 mm). IR spectra were recorded on Mattson Gausis ATI FT-IR (600-4000 cm^{-1}). Melting points were determined on MEL-TEMP II from LABORATORY DEVICES INC, USA.

General Procedure.

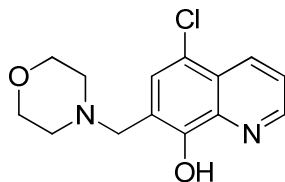
The synthesis of the clioquinol and nitroxoline inhibitors were modified according to the procedure done by Chen et al.⁸

5-chloro-7-(piperidin-1-ylmethyl)quinolin-8-ol (5)



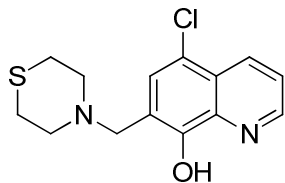
In a carousel reaction tube, 0.41 g (2.28 mmol) of 5-chloroquinolin-8-ol was mixed with 300 μL formaldehyde in 20 mL dry ethanol. To this solution was added 310 μL piperidine (2.4 mmol). The mixture was refluxed at 80°C for 24 hr. The precipitate was filtered. The crude product was purified by recrystallization from 1:1 EtOH-H₂O to yield the final product (0.60 g, 95.1%). mp 63-65 °C; FT-IR: ν = 3330.88, 2931.81, 2852.33, 2751.57, 1497.01, 1464.31, 1408.09, 1371.76, 1333.84 cm^{-1} ; ^1H NMR (300 MHz, DMSO-*d*₆) δ 8.92 (s, 1H), 8.46 (d, 1H), 7.59 (t, 1H), 3.46 (d, 2H), 2.47 (d, 2H), 1.54 (m, 3H); ^{13}C NMR (300 MHz, DMSO-*d*₆) δ 151.41, 148.90, 136.68, 132.17, 126.16, 124.89, 120.32, 118.04, 88.30, 63.07, 61.49, 55.99, 53.62, 50.15, 25.40, 23.63, 18.47, 15.20. Anal. HPLC (Ret time: 4.982 min Height: 222.08 mAUArea%: 89.68%). Anal Calcd for C₁₅H₁₇ClN₂O: C, 65.10; H, 6.19; N, 10.12; Found C, 64.70; H, 6.16; N, 9.72.

5-chloro-7-(morpholinomethyl)quinolin-8-ol (6)



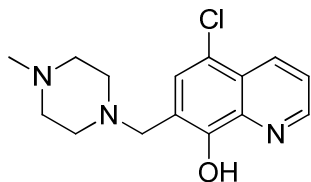
In a carousel reaction tube, 0.40 g (2.23 mmol) of 5-chloroquinolin-8-ol was mixed with 300 μ L formaldehyde in 20 mL dry ethanol. To this solution was added 260 μ L morpholine (2.4 mmol). The mixture was refluxed at 80°C for 24 hr. The precipitate was filtered. The crude product was purified by recrystallization from 1:1 EtOH-H₂O to yield the final product (0.42 g, 67.6%). mp 89-91°C; FT-IR: ν = 1494.45, 1453.19, 1405.94, 1369.28 cm^{-1} ; ¹H NMR (300 MHz, DMSO-*d*₆) δ 8.93 (d, 1H), 8.47 (d, 1H), 7.64 (d, 1H), 3.71 (d, 2H), 3.60 (s, 2H), 2.46 (d, 2H); ¹³C NMR (300 MHz, DMSO-*d*₆) δ 150.82, 148.87, 138.78, 138.56, 132.43, 132.30, 128.54, 127.18, 124.98, 124.89, 122.74, 122.59, 121.72, 120.31, 118.37, 118.25, 66.20, 55.99, 53.10, 15.02. Anal. HPLC (Ret time: 3.692 min Height: 343.34 mAU Area%: 100%). Anal Calcd for C₁₄H₁₅ClN₂O₂: C, 60.33; H, 5.42; N, 10.05; Found C, 60.07; H, 5.52; N, 9.62.

5-chloro-7-(thiomorpholinomethyl)quinolin-8-ol (7)



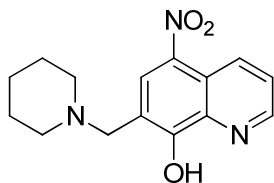
In a carousel reaction tube, 0.50 g (2.78 mmol) of 5-chloroquinolin-8-ol was mixed with 300 μ L formaldehyde in 20 mL dry ethanol. To this solution was added 303 μ L thiomorpholine (2.4 mmol). The mixture was refluxed at 80°C for 24 hr. The precipitate was filtered. The crude product was purified by recrystallization from 1:1 EtOH-H₂O to yield the final product (0.54 g, 73.7%). mp 101-104 °C; FT-IR: ν = 3345.82, 1497.57, 1455.46, 1409.39, 1367.13, 1330.70 cm^{-1} ; ¹H NMR (300 MHz, DMSO-*d*₆) δ 8.93 (d, 1H), 8.47 (d, 1H), 7.62 (d, 1H), 3.73 (s, 2H), 2.72 (d, 2H), 2.64 (d, 2H); ¹³C NMR (300 MHz, DMSO-*d*₆) δ 148.91, 138.74, 132.32, 128.43, 124.86, 122.62, 120.51, 118.23, 56.19, 54.37, 27.13. Anal. HPLC (Ret time: 4.448 min Height: 354.30 mAU Area%: 100%). Anal Calcd for C₁₄H₁₅ClN₂OS: C, 57.04; H, 5.13; N, 9.50; Found C, 57.07; H, 5.03; N, 9.10.

5-chloro-7-((4-methylpiperazin-1-yl)methyl)quinolin-8-ol (8)



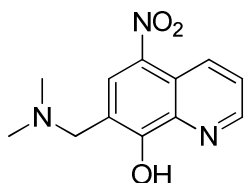
In a carousel reaction tube, 0.44 g (2.45 mmol) of 5-chloroquinolin-8-ol was mixed with 300 μ L formaldehyde in 20 mL dry ethanol. To this solution was added 333 μ L 1-methylpiperazine (2.4 mmol). The mixture was refluxed at 80°C for 24 hr. The precipitate was filtered. The crude product was purified by recrystallization from 1:1 EtOH-H₂O to yield the final product (0.58 g, 79.5%). mp 126-129 °C; FT-IR: ν = 2835.35, 1495.86, 1455.47, 1403.42, 1369.44, 1327.96 cm^{-1} ; ¹H NMR (300 MHz, DMSO-*d*₆) δ 8.93 (d, 1H), 8.47 (d, 1H), 7.61 (d, 1H), 3.59 (s, 2H), 2.50 (d, 2H), 2.38 (d, 2H), 2.19 (s, 3H); ¹³C NMR (300 MHz, DMSO-*d*₆) δ 150.97, 149.54, 148.89, 138.83, 132.45, 132.24, 128.32, 127.19, 124.99, 124.88, 122.76, 122.53, 121.74, 120.54, 118.19, 65.62, 55.91, 54.61, 52.34, 45.49, 15.02. Anal. HPLC (Ret time: 3.126 min Height: 277.99 mAUArea%: 100%). Anal Calcd for C₁₅H₁₈ClN₃O: C, 61.75; H, 6.22; N, 14.40; Found C, 61.35; H, 5.96; N, 14.0.

5-nitro-7-(piperidin-1-ylmethyl)quinolin-8-ol (10)



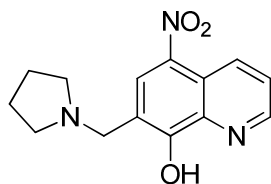
In a carousel reaction tube, 0.90 g (4.73 mmol) of 5-chloroquinolin-8-ol was mixed with 743 μ L formaldehyde in 20 mL dry ethanol. To this solution was added 561 μ L piperidine (5.68 mmol). The mixture was refluxed at 80°C for 24 hr. The precipitate was filtered. The crude product was purified by recrystallization from 1:1 EtOH-H₂O to yield the final product (0.58 g, 42.7%). mp 225 °C; FT-IR: ν = 1597.43, 1547.95, 1506.81, 1444.30, 1386.92 cm^{-1} ; ¹H NMR (300 MHz, DMSO-*d*₆) δ 9.23 (d, 1H), 8.50 (d, 2H), 7.50 (t, 1H), 4.10 (s, 2H), 2.43 (s, 2H), 1.66 (s, 1H), 1.49 (d, 2H); ¹³C NMR (300 MHz, DMSO-*d*₆) δ 155.06, 150.10, 146.03, 133.86, 131.95, 124.41, 58.21, 52.12, 22.74, 21.72. Anal. HPLC (Ret time: 7.243 min Height: 489.21 mAUArea%: 97.67%). Anal Calcd for C₁₅H₁₇N₃O₃: C, 62.71; H, 5.96; N, 14.63; Found C, 62.39; H, 5.96; N, 14.31.

7-((dimethylamino)methyl)-5-nitroquinolin-8-ol (11)



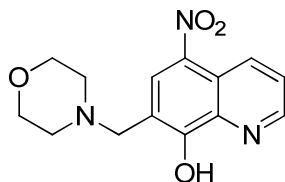
In a carousel reaction tube, 0.47 g (2.48 mmol) of 5-chloroquinolin-8-ol was mixed with 300 μ L formaldehyde in 20 mL dry ethanol. To this solution was added 380 μ L dimethylamine (2.4 mmol). The mixture was refluxed at 80°C for 24 hr. The precipitate was filtered. The crude product was purified by recrystallization from 1:1 EtOH-H₂O to yield the final product (0.53 g, 86.8%). mp 235-237 °C; FT-IR: ν = 1596.37, 1558.06, 1528.28, 1511.03, 1471.67, 1449.67, 1386.31, 1366.81 cm^{-1} ; ¹H NMR (300 MHz, DMSO-*d*₆) δ 9.38 (d, 1H), 8.67 (d, 1H), 7.60 (d, 1H), 6.60 (d, 1H), 4.10 (s, 2H), 2.69 (s, 6H); ¹³C NMR (300 MHz, DMSO-*d*₆) δ 150.87, 141.98, 134.46, 131.15, 127.71, 124.40, 122.14, 114.14, 52.49, 40.66. Anal. HPLC (Ret time: 2.742 min Height: 561.70 mAUArea%: 92.96%). Anal Calcd for C₁₂H₁₃N₃O₃: C, 58.29; H, 5.30; N, 16.99; Found C, 57.84; H, 5.36; N, 16.75.

5-nitro-7-(pyrrolidin-1-ylmethyl)quinolin-8-ol (12)



In a carousel reaction tube, 0.40 g (2.10 mmol) of 5-chloroquinolin-8-ol was mixed with 300 μ L formaldehyde in 20 mL dry ethanol. To this solution was added 245 μ L pyrrolidine (2.4 mmol). The mixture was refluxed at 80°C for 24 hr. The precipitate was filtered. The crude product was purified by recrystallization from 1:1 EtOH-H₂O to yield the final product (0.42 g, 73.1%). mp 227-230°C; FT-IR: ν = 1594.09, 1529.65, 1448.41, 1390.08 cm^{-1} ; ¹H NMR (300 MHz, DMSO-*d*₆) δ 9.36 (d, 1H), 8.62 (d, 2H), 7.58 (d, 1H), 4.20 (s, 2H), 2.50 (d, 2H), 1.93 (d, 2H); ¹³C NMR (300 MHz, DMSO-*d*₆) δ 155.0, 151.3, 142.4, 137.9, 131.6, 126.1, 124.3, 119.3, 60.3, 56.9, 23.6. Anal. HPLC (Ret time: 3.406 min Height: 673.64 mAUArea%: 100%). Anal Calcd for C₁₄H₁₅N₃O₃: C, 61.53; H, 5.53; N, 15.38; Found C, 61.13; H, 5.44; N, 15.09.

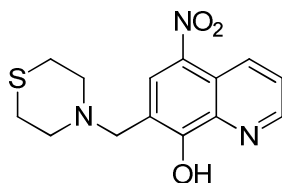
7-(morpholinomethyl)-5-nitroquinolin-8-ol (13)



In a carousel reaction tube, 0.40 g (2.10 mmol) of 5-chloroquinolin-8-ol was mixed with 300 μ L formaldehyde in 20 mL dry ethanol. To this solution was added 260 μ L morpholine (2.4 mmol). The mixture was refluxed at 80°C for 24 hr. The precipitate was filtered. The crude product was purified by recrystallization from 1:1 EtOH-H₂O to yield the final product (0.55 g, 91.2%). mp 221-224 °C; FT-IR: ν = 1597.26, 1576.57, 1546.49, 1524.96, 1450.25, 1387.93, 1328.50 cm^{-1} ; ¹H NMR (300 MHz, DMSO-*d*₆) δ 9.17 (d, 1H), 8.83 (d, 1H), 8.54 (d, 1H), 7.71 (d, 1H), 4.60 (s, 2H), 3.61 (d, 2H), 2.63 (t, 2H); ¹³C NMR (300 MHz, DMSO-*d*₆) δ 148.14, 138.45, 132.38, 130.75, 124.74, 122.79, 117.50, 65.52, 55.96, 52.63. Anal.

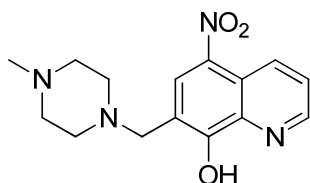
HPLC (Ret time: 2.655 min Height: 510.56 mAUArea%: 94.10%). Anal Calcd for $C_{14}H_{15}N_3O_4$: C, 58.13; H, 5.23; N, 14.53; Found C, 57.81; H, 4.95; N, 14.32.

5-nitro-7-(thiomorpholinomethyl)quinolin-8-ol (14)



In a carousel reaction tube, 0.40 g (2.10 mmol) of 5-chloroquinolin-8-ol was mixed with 300 μ L formaldehyde in 20 mL dry ethanol. To this solution was added 303 μ L thiomorpholine (2.4 mmol). The mixture was refluxed at 80°C for 24 hr. The precipitate was filtered. The crude product was purified by recrystallization from 1:1 EtOH-H₂O to yield the final product (0.58 g, 90.6%). mp 190-193 °C; FT-IR: ν = 1592.18, 1575.17, 1539.23, 1522.94, 1507.11, 1455.12, 1418.88, 1390.07 cm^{-1} ; ^1H NMR (300 MHz, DMSO-*d*₆) δ 9.18 (d, 1H), 8.86 (d, 1H), 8.57 (d, 1H), 7.76 (d, 1H), 3.66 (s, 2H), 2.77 (d, 2H), 2.50 (d, 2H); ^{13}C NMR (300 MHz, DMSO-*d*₆) δ 147.82, 138.86, 132.33, 130.89, 124.62, 123.13, 117.85, 81.07, 56.31, 54.09, 52.86, 26.39. Anal. HPLC (Ret time: 3.066 min Height: 477.26 mAUArea%: 94.68%). Anal Calcd for $C_{14}H_{15}N_3O_3S$: C, 55.07; H, 4.95; N, 13.76; Found C, 54.68; H, 4.45; N, 13.40.

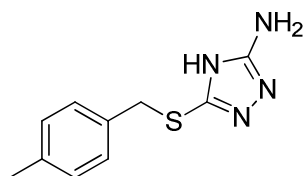
7-((4-methylpiperazin-1-yl)methyl)-5-nitroquinolin-8-ol (15)



In a carousel reaction tube, 0.41 g (2.16 mmol) of 5-chloroquinolin-8-ol was mixed with 300 μ L formaldehyde in 20 mL dry ethanol. To this solution was added 333 μ L 1-methylpiperazine (2.4 mmol). The mixture was refluxed at 80°C for 24 hr. The precipitate was filtered. The crude product was purified by recrystallization from 1:1 EtOH-H₂O to yield the final product (0.48 g, 73.5%). mp 209-211 °C; FT-IR: ν =1595.09, 1573.24, 1546.49, 1526.32, 1499.95, 1458.46, 1433.85, 1382.61, 1358.19 cm^{-1} ; ^1H NMR (300 MHz, DMSO-*d*₆) δ 9.29 (d, 1H), 8.77 (d, 1H), 8.55 (d, 1H), 7.68 (d, 1H), 3.91 (s, 2H), 2.80 (d, 2H), 2.62 (d, 2H), 2.32 (s, 3H); ^{13}C NMR (300 MHz, DMSO-*d*₆) δ . Anal. HPLC (Ret time: 2.366 min Height: 604.64 mAUArea%: 95.23%). Anal Calcd for $C_{15}H_{18}N_4O_3$: C, 59.59; H, 6.00; N, 18.53; Found C, 59.19; H, 5.85; N, 18.28.

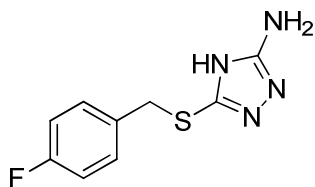
The synthesis of the triazole inhibitors were modified according to the procedure done by Novinson et al.⁹

5-((4-methylbenzyl)thio)-4H-1,2,4-triazol-3-amine (16)



In a 250-mL Erlenmeyer flask, 2.32 g (0.02 mol) of 3-amino-5-thio-1,2,4-triazole was mixed with 20 mL of 1 M NaOH solution (0.04 mol). To this solution was added 3.75g (0.02 mol) of 1-(bromomethyl)-4-methylbenzene dissolved in 40 mL of absolute ethanol. The yellowish green mixture was refluxed in the oil bath at 75°C for 20 min until the turbidity disappeared and the color of the solution turned to amber. The solution was evaporated under the reduced pressure to about 25 mL, diluted with 5 mL distilled water, and refrigerated overnight. The precipitate was filtered, mixed with toluene and was azeotroped with the toluene under the reduced pressure. The crude residue was recrystallized from hot chloroform to yield a white solid (2.93 g, 66.1%). mp 109-110 °C; FT-IR: ν = 3439.64, 3329.11, 3226.21, 3063.36, 2919.17, 2851.98, 1636.92, 1500.70, 1445.72, 1365.40 cm^{-1} ; ^1H NMR (300 MHz, DMSO- d_6) δ 7.20 (d, 2H), 7.10 (d, 2H), 4.20 (s, 2H), 2.30 (s, 3H); ^{13}C NMR (300 MHz, DMSO- d_6) δ 158.81, 156.62, 136.83, 134.20, 128.82, 34.45, 21.33. MS (ESI) 220.9 (M-H)⁺. Anal. HPLC (Ret time: 7.14 min Height: 529. 96 mAUArea%: 95.91%). Anal Calcd for C₁₀H₁₂N₄S: C, 52.52; H, 5.49; N, 25.43; Found C, 54.38; H, 5.49; N, 24.27.

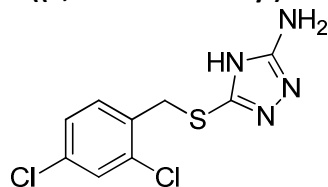
5-((4-fluorobenzyl)thio)-4H-1,2,4-triazol-3-amine (17)



In a 250-mL Erlenmeyer flask, 2.32 g (0.02 mol) of 3-amino-5-thio-1,2,4-triazole was mixed with 20 mL of 1 M NaOH solution (0.04 mol). To this solution was added 2.5 mL (0.02) of 4-fluorobenzyl bromide dissolved in 40 mL of absolute ethanol. The yellowish green mixture was refluxed in the oil bath at 75°C for 20 min until the turbidity disappeared and the color of the solution turned to amber. The solution was evaporated under the reduced pressure to about 25 mL, diluted with 5 mL distilled water, and refrigerated overnight. The precipitate was filtered, mixed with toluene and was azeotroped with the toluene under the reduced pressure. The crude residue was recrystallized from hot chloroform to yield a white solid (2.10 g, 46.9 %). mp 123-125 °C; FT-IR: ν = 3479.79, 3457.19, 3305.21, 3201.82, 3049.00,

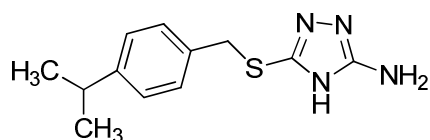
2930.64, 2846.91, 2759.84, 2705.46, 2341.14, 1898.12, 1641.18, 1587.28, 1495.74, 1345.29 cm^{-1} ; ^1H NMR (300 MHz, $\text{DMSO}-d_6$) δ 7.35 (t, 2H), 7.00 (t, 2H), 4.22 (s, 2H); ^{13}C NMR (300 MHz, $\text{DMSO}-d_6$) δ 161.32, 158.85, 156.13, 135.50, 132.71, 115.55, 34.42. MS (ESI) (M-H) $^+$ 224.26. Anal. HPLC (Ret time: 5.66 min Height: 520.52 mAU Area%: 94.76%). Anal. Calcd for $\text{C}_9\text{H}_9\text{FN}_4\text{S}$: C, 48.20; H, 4.05; N, 24.98; Found C, 47.65; H, 3.95; N, 24.51.

5-((2,4-dichlorobenzyl)thio)-4H-1,2,4-triazol-3-amine (18)



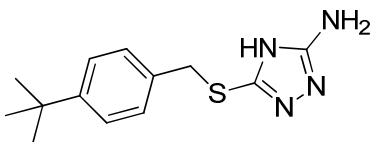
In a 250-mL Erlenmeyer flask, 2.32 g (0.02 mol) of 3-amino-5-thio-1,2,4-triazole was mixed with 20 mL of 1 M NaOH solution (0.04 mol). To this solution was added 2.8 mL g (0.02 mol) of 1-(bromomethyl)-2,4-dichlorobenzene dissolved in 40 mL of absolute ethanol. The yellowish green mixture was refluxed in the oil bath at 75°C for 20 min until the turbidity disappeared and the color of the solution turned to amber. The solution was evaporated under the reduced pressure to about 25 mL, diluted with 5 mL distilled water, and refrigerated overnight. The precipitate was filtered, mixed with toluene and was azeotroped with the toluene under the reduced pressure. The crude residue was recrystallized from hot chloroform to yield a white solid (5.06 g, 92%). mp 147-149 °C; FT-IR: ν = 3422.73, 3330.42, 3211.84, 3054.68, 2928.38, 2762.10, 1629.39, 1599.81, 1469.28 cm^{-1} ; ^1H NMR (300 MHz, $\text{DMSO}-d_6$) δ 7.60 (s, 1H), 7.48 (d, 1H), 7.36 (d, 1H), 6.09 (s, 1H), 4.27 (s, 2H). ; ^{13}C NMR (300 MHz, $\text{DMSO}-d_6$) δ 163.27, 160.05, 157.83, 135, 25, 131, 14, 131.03, 115.63, 34.33. MS (ESI) 274.9 (M-H) $^+$. Anal. HPLC: (Ret time: 10.02 min Height: 429.71 mAU Area%: 92.63%). Anal. Calcd for $\text{C}_9\text{H}_8\text{Cl}_2\text{N}_4\text{S}$: C, 39.29; H, 2.93; N, 20.36; Found C, 39.17; H, 2.95; N, 20.00.

5-((4-isopropylbenzyl)thio)-4H-1,2,4-triazol-3-amine (19)



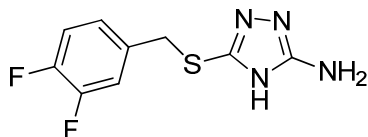
In a carousel reaction tube, 2.00 g (0.017 mol) of 3-amino-5-thio-1,2,4-triazole was mixed with 8.6 mL of 1 M NaOH solution (0.034 mol). To this solution was added 2.84 mL (0.017 mol) of 4-isopropylbenzyl bromide dissolved in 17 mL of absolute ethanol. The mixture was refluxed at 75°C for 20 min. The solution was evaporated under the reduced pressure to yield a desired produce (2.30 g, 55%). mp 109-111 °C, FT-IR: ν =3413.12, 3260.06, 2917.63, 1657.69, 1626.20, 1503.56, 1431.24 cm^{-1} ; ^1H NMR(300 MHz, $\text{DMSO}-d_6$) δ 7.26 (d, 2H), 7.18 (d, 2H), 4.26 (s, 2H), 2.85 (m, 1H), 1.18 (d, 6H); ^{13}C NMR (300 MHz, $\text{DMSO}-d_6$) δ 154.87, 151.07, 147.47, 134.64, 128.72, 126.29, 35.04, 33.03, 23.75. Anal. HPLC: (Ret time: 5.755 min Height: 602.25 mAU Area%: 97.10%). Anal. Calcd for $\text{C}_{12}\text{H}_{16}\text{N}_4\text{S}$: C, 58.04; H, 6.49; N, 22.56; Found C, 58.44; H, 6.09; N, 22.10.

5-((4-(tert-butyl)benzyl)thio)-4H-1,2,4-triazol-3-amine (20)



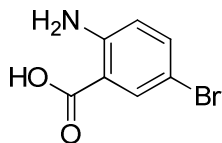
In a carousel reaction tube, 2.00 g (0.017 mol) of 3-amino-5-thio-1,2,4-triazole was mixed with 8.6 mL of 1 M NaOH solution (0.034 mol). To this solution was added 3.12 mL (0.017 mol) of 4-tert-butyl benzyl bromide dissolved in 17 mL of absolute ethanol. The mixture was refluxed at 75°C for 20 min. The solution was evaporated under the reduced pressure to yield a desired produce (3.50 g, 85%). mp 118-120 °C, FT-IR: ν = 3377.29, 3280.47, 3065.55, 1678.54, 1660.39, 1517.46, 1492.09, 1435.76, 1403.62 cm^{-1} ; ^1H NMR(300 MHz, $\text{DMSO}-d_6$) δ 7.31 (d, 2H), 7.15 (d, 2H), 4.26 (s, 2H), 1.25 (s, 9H); ^{13}C NMR (300 MHz, $\text{DMSO}-d_6$) δ 154.85, 151.07, 149.74, 134.27, 128.46, 125.14, 38.01, 37.10, 31.06. Anal. HPLC: (Ret time: 7.243 min Height: 489.21 mAU Area%: 97.67%). Anal. Cald for $\text{C}_{13}\text{H}_{18}\text{N}_4\text{S}$: C, 59.51; H, 6.91; N, 21.35; Found C, 59.11; H, 6.88; N, 21.32.

5-((3,4-difluorobenzyl)thio)-4H-1,2,4-triazol-3-amine (21)



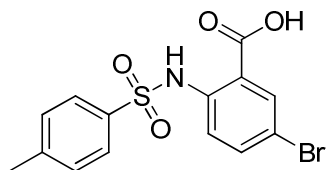
In a carousel reaction tube, 2.00 g (0.017 mol) of 3-amino-5-thio-1,2,4-triazole was mixed with 8.6 mL of 1 M NaOH solution (0.034 mol). To this solution was added 2.18 mL (0.017 mol) of 3,4-difluorobenzyl bromide dissolved in 17 mL of absolute ethanol. The mixture was refluxed at 75°C for 20 min. The solution was evaporated under the reduced pressure to yield a desired produce (5.98 g, 76%). mp 98-100 °C, FT-IR: ν = 2950.84, 1684.09, 1639.16, 1581.27, 1534.08, 1497.47, 1412.16 cm^{-1} ; ^1H NMR(300 MHz, $\text{DMSO}-d_6$) δ 7.37 (m, 1H), 7.22 (s, 1H), 7.03 (d, 1H), 4.28 (s, 2H); ^{13}C NMR (300 MHz, $\text{DMSO}-d_6$) δ 154.65, 150.27, 146.92, 135.66, 125.66, 117.86, 34.13. Anal. HPLC: (Ret time: 2.736 min Height: 193.94 mAU Area%: 100%). Anal. Cald for $\text{C}_9\text{H}_8\text{F}_2\text{N}_4\text{S}$: C, 44.62; H, 3.33; N, 23.13; Found C, 44.58; H, 3.73; N, 23.53.

2-amino-5-bromobenzoic acid



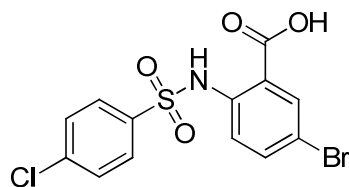
2-aminobenzoic acid (1.00 g, 7.29 mmol) was dissolved in glacial acetic acid (17 mL). The temperature was cooled to below 17 °C. Bromine solution (1.47 g, 9.30 mmol) was then added dropwise until the precipitate forms. The solid product was filtered and boiled up in a solution of deionized water (33 mL) and concentrated HCl (2 mL). The solution was subjected to vacuum filtration. The precipitate was collected and extracted twice with boiling water (38 mL). In the next step, the filtrate was cooled to room temperature to yield the desired product (0.45 g, 28.6%). mp 213-215 °C; FT-IR: ν = 3475.54, 3358.81, 1659.47, 1580.13, 1544.64, 1478.81, 1419.97, 1308.55, 1231.59 cm^{-1} ; ^1H NMR (300 MHz, $\text{DMSO}-d_6$) δ 7.81 (s, 1H), 7.69 (d, 1H), 7.32 (d, 1H), 6.75(d, 1H,); ^{13}C NMR (75 MHz, $\text{DMSO}-d_6$) δ 169.35, 150.22, 137.63, 134.60, 118.41, 112.45, 104.23. Anal. HPLC (Ret time: 3.431 Height: 236.19 mAUArea%: 85.93%). Anal Calcd for $\text{C}_7\text{H}_6\text{BrNO}_2$: C, 38.92; H, 2.80; N, 6.48; Found: C, 38.80; H, 2.63; N, 6.19.

5-bromo-2-(4-methylphenylsulfonamido)benzoic acid (24)



2-amino-5-bromobenzoic acid (0.202 g, 1 mmol) was suspended in distilled water (20 mL). The pH of the solution was adjusted to 8 with 1 M Na_2CO_3 until the solution became clear. To this solution, 0.231 g (1.2 mmol) of 4-methylbenzene-1-sulfonyl chloride was added. The reaction was stirred at room temperature overnight. The pH of the solution was then adjusted to 1 using 1N HCl dropwise until the white precipitate formed. The solid was filtered and washed with deionized water. The solid was subjected to recrystallization in methanol to yield the desired white solid (0.28 g, 75.68 %). mp 192-199 °C; FT-IR: ν = 3211.70, 2919.64, 2861.81, 2541.09, 1659.52, 1597.21, 1571.70, 1480.37, 1436.00, 1389.79, 1341.29, 1307.34, 1289.84, 1240.53, 1174.35 cm^{-1} ; ^1H NMR (300 MHz, $\text{DMSO}-d_6$) δ 7.81(s, 1H), 7.74(d, 2H), 7.69(d, 1H), 7.40(d, 2H), 2.34(d, 1H); ^{13}C NMR (75 MHz, $\text{DMSO}-d_6$) δ 168.65, 143.22, 139.91, 137.71, 135.53, 133.30, 130.10, 127.75, 121.10, 119.19, 115.52, 22.25. Anal. HPLC (Ret time: 11.94 Height: 401.88 mAUArea%: 100%). Anal Calcd for $\text{C}_{14}\text{H}_{12}\text{BrNO}_4\text{S}$: C, 45.42; H, 3.27; N, 3.78; Found: C, 45.02; H, 3.66; N, 3.33.

5-bromo-2-(4-chlorophenylsulfonamido)benzoic acid (25)



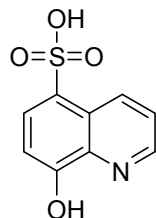
2-amino-5-bromobenzoic acid (0.300 g, 1.39 mmol) was suspended in distilled water (20 mL). The pH of the solution was adjusted to 8 with 1 M Na_2CO_3 until the solution became clear. To this solution, 0.322 g (1.528 mmol) of 4-chlorobenzene-1-sulfonyl chloride was added. The reaction was stirred at room temperature overnight. The pH of the solution was then adjusted to 1 using 1N HCl dropwise until the white precipitate formed. The solid was filtered and washed with deionized water. The desired compound was dried under the vacuum to yield a white solid (0.5 g, 92 %). Mp 222-223 °C; FT-IR: ν = 3217.57, 3093.65, 2541.29, 1659.47, 1567.68, 1474.08, 1436.12, 1391.53, 1349.44, 1308.15, 1281.26, 1166.98 cm^{-1} ; ^1H NMR (300 MHz, $\text{DMSO}-d_6$) δ 7.81 (s, 1H), 7.80(d, 2H), 7.69(d, 1H), 7.62(d, 2H); ^{13}C NMR (75 MHz, $\text{DMSO}-d_6$) δ 169.35, 138.20, 135.23, 133.22, 130.19, 129.35, 122.23, 119.90, 116.18. Anal. HPLC (Ret time: 12.55 Height: 331.32 mAUArea%: 100%). Anal Calcd for $\text{C}_{13}\text{H}_9\text{BrClNO}_4\text{S}$: C, 39.97; H, 2.32; N, 3.59; Found: C, 39.84; H, 2.21; N, 3.58.

References

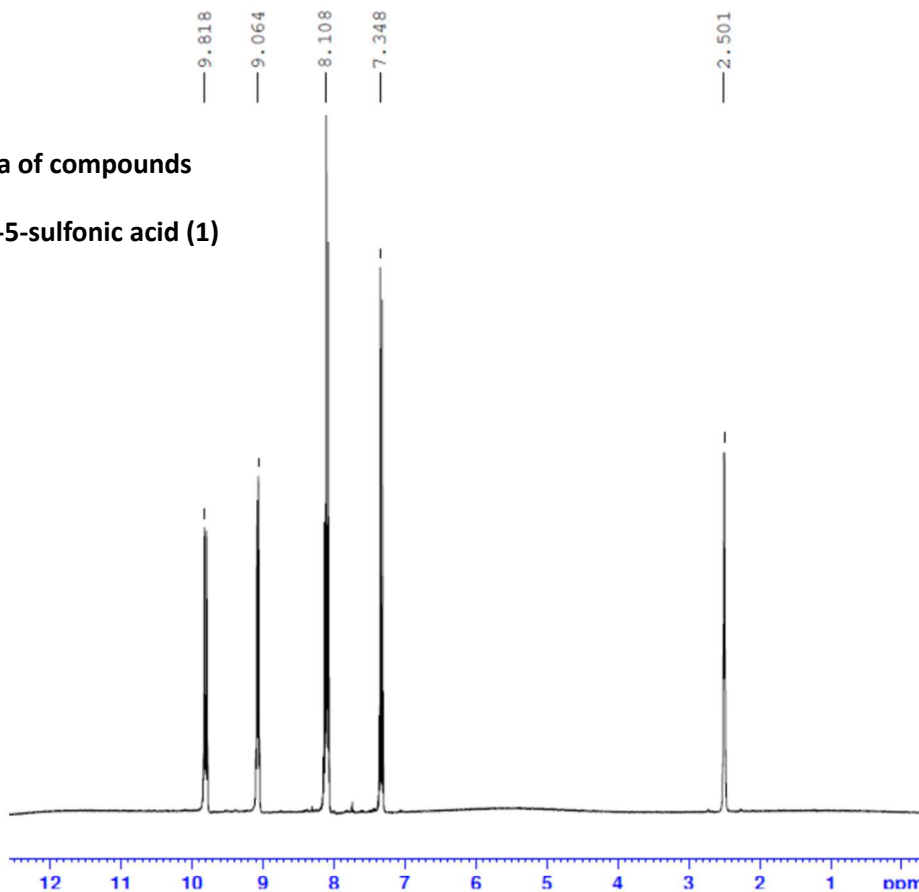
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5. NMR spectra of compounds

8-hydroxyquinoline-5-sulfonic acid (1)



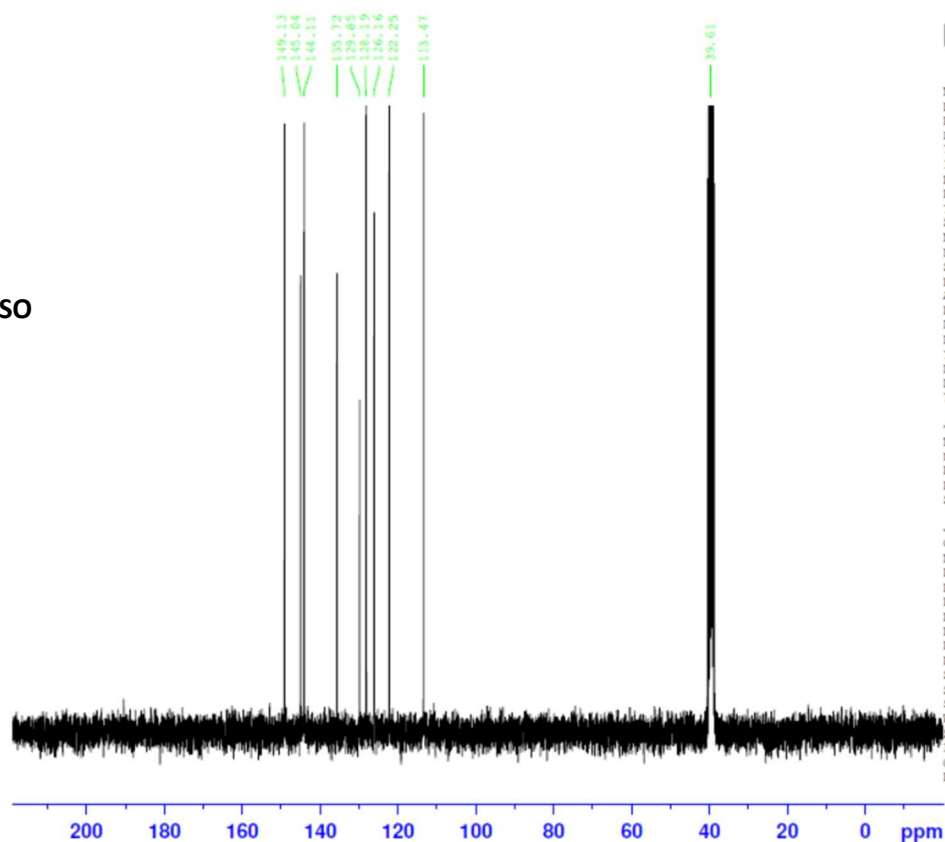
^1H NMR (CD_3) $_2\text{SO}$



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PROCNO    1
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PULPROG   zg30
TD         9806
SOLVENT   DMSO
NS         8
DS         2
SWH        3898.129 Hz
FIDRES     0.397525 Hz
AQ         1.2578329 sec
RG         203
DE         128.267 usec
TE         313.0 K
D1         3.00000000 sec
D0         1
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----- CHANNEL f1 -----
NUC1      1H
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PL1       1.10 dB
PL1W      8.87179279 W
SFO1      300.1518535 MHz
SI        32768
SF        300.1500000 MHz
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SSB       0
LB        0.30 Hz
GB        0
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^{13}C NMR (CD_3) $_2\text{SO}$

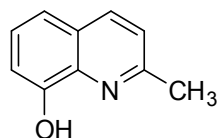


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Time      20.18
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PULPROG   zgpg30
TD         65536
SOLVENT   DMSO
NS         1024
DS         4
SWH        18028.846 Hz
FIDRES     0.275098 Hz
AQ         1.8175818 sec
RG         203
DE         27.733 usec
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D11        0.03000000 sec
D0         1
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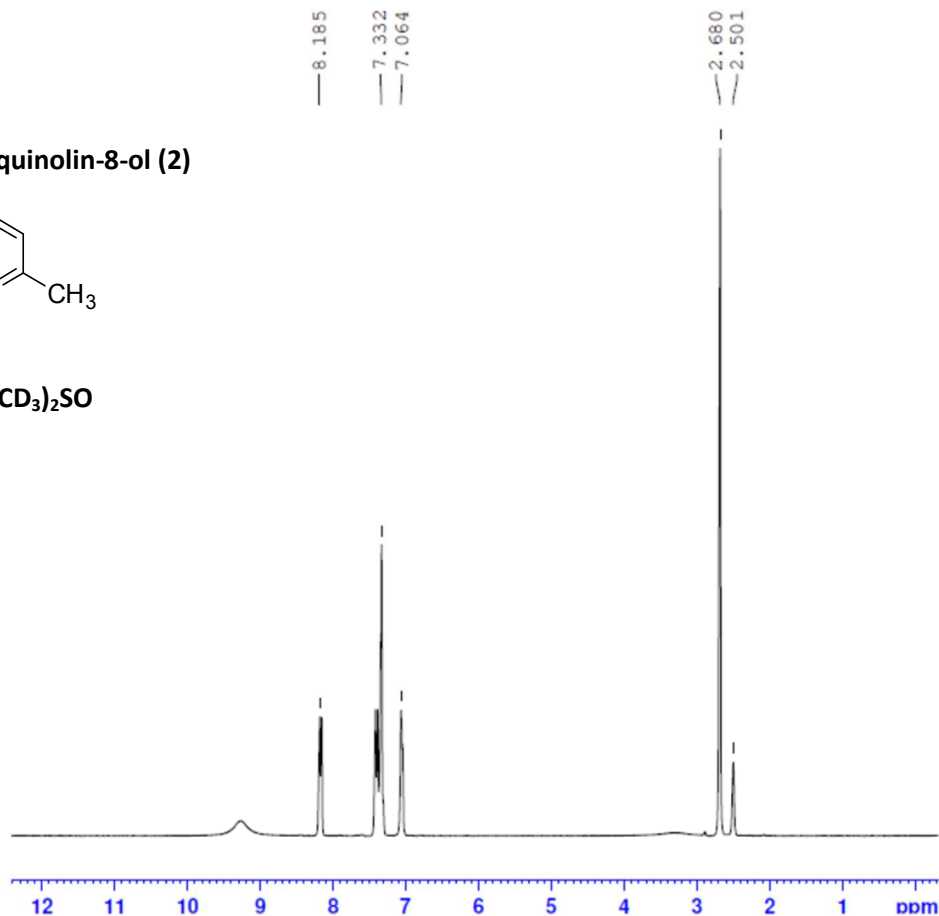
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PL1       -0.20 dB
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SFO1      75.4803248 MHz
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----- CHANNEL f2 -----
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NUC2      1H
PCPD2     100.00 usec
PL2       1.50 dB
PL12      18.40 dB
PL13      21.40 dB
PL2W      8.09117126 W
PL12W     0.16520053 W
PL13W     0.08279639 W
SFO2      300.1512006 MHz
SI        32768
SF        75.4728157 MHz
WDW       EM
SSB       0
LB        1.00 Hz
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PC        1.40
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2-methylquinolin-8-ol (2)



¹H NMR (CD₃)₂SO



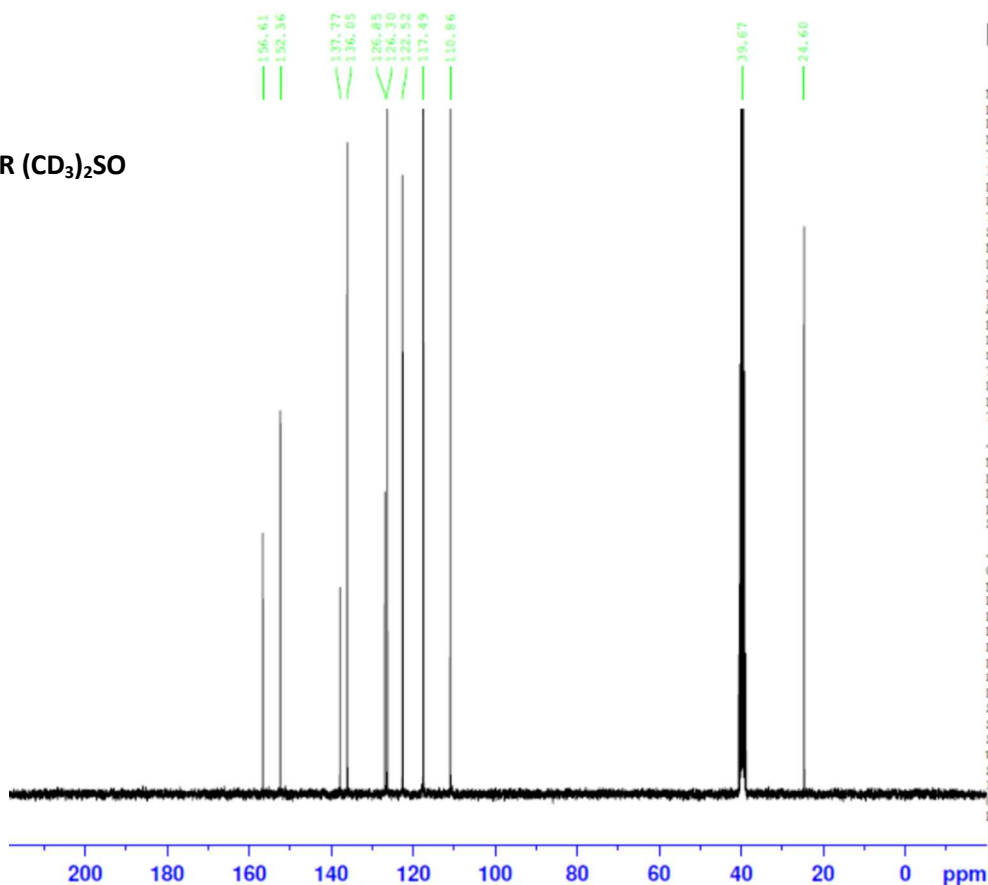
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PULPROG   zg30
TD         9806
SOLVENT   DMSO
NS         8
DS         2
SWH        3898.129 Hz
FIDRES     0.397525 Hz
AQ         1.2578329 sec
RG         144
DW         128.267 usec
DE         8.00 usec
TE         313.0 K
D1         3.00000000 sec
TD0        1
    
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----- CHANNEL f1 -----
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P1         14.60 usec
PL1        1.10 dB
PL1W       8.87179279 W
SFO1       300.1518535 MHz
SI         32768
SF         300.1500000 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
    
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¹³C NMR (CD₃)₂SO



```

NAME      Mar17-2013
EXPNO     20
PROCNO    1
Date_     20130317
Time      21.30
INSTRUM   spect
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PULPROG   zgpg30
TD         65536
SOLVENT   DMSO
NS         1024
DS         4
SWH        18028.846 Hz
FIDRES     0.275098 Hz
AQ         1.8175818 sec
RG         203
DW         27.733 usec
DE         8.50 usec
TE         323.0 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1
    
```

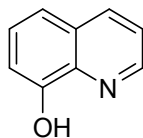
```

----- CHANNEL f1 -----
NUC1       13C
P1         9.80 usec
PL1        -0.20 dB
PL1W       32.35229492 W
SFO1       75.4803248 MHz
    
```

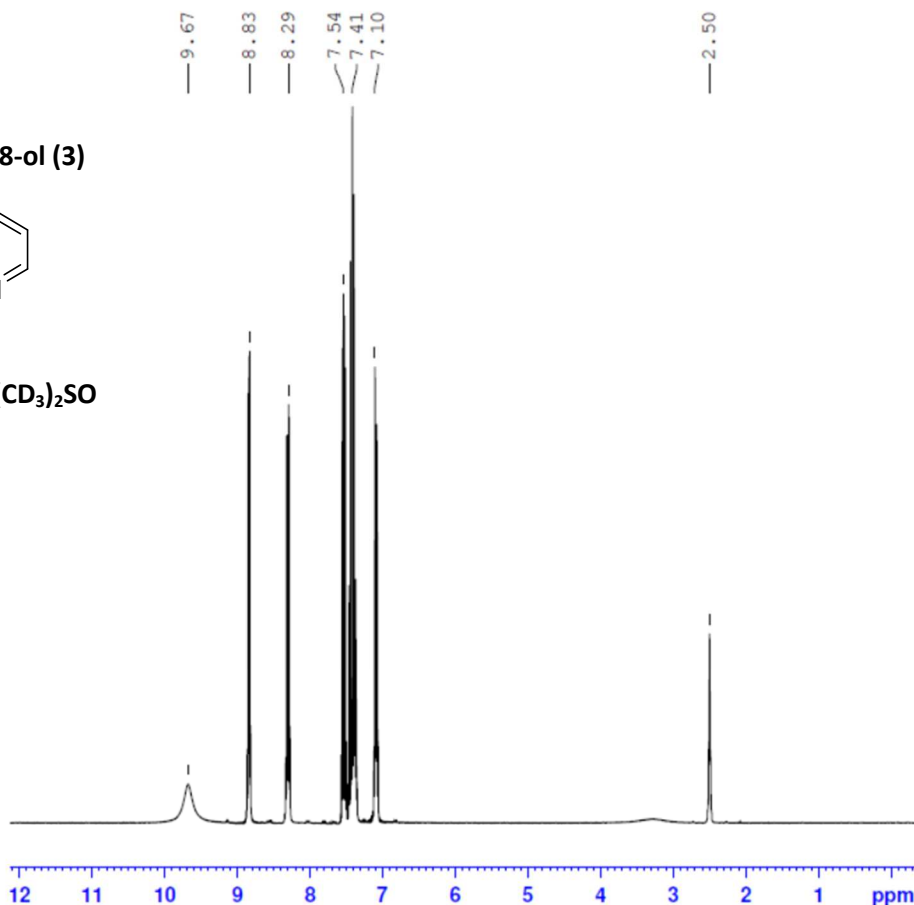
```

----- CHANNEL f2 -----
CPDPRG2   waltz16
NUC2       1H
PCPD2     100.00 usec
PL2        1.50 dB
PL12       18.40 dB
PL13       21.40 dB
PL12W     8.09117126 W
PL13W     0.16520053 W
PL13W     0.08279639 W
SFO2       300.1512006 MHz
SI         32768
SF         75.4728157 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
    
```

quinolin-8-ol (3)



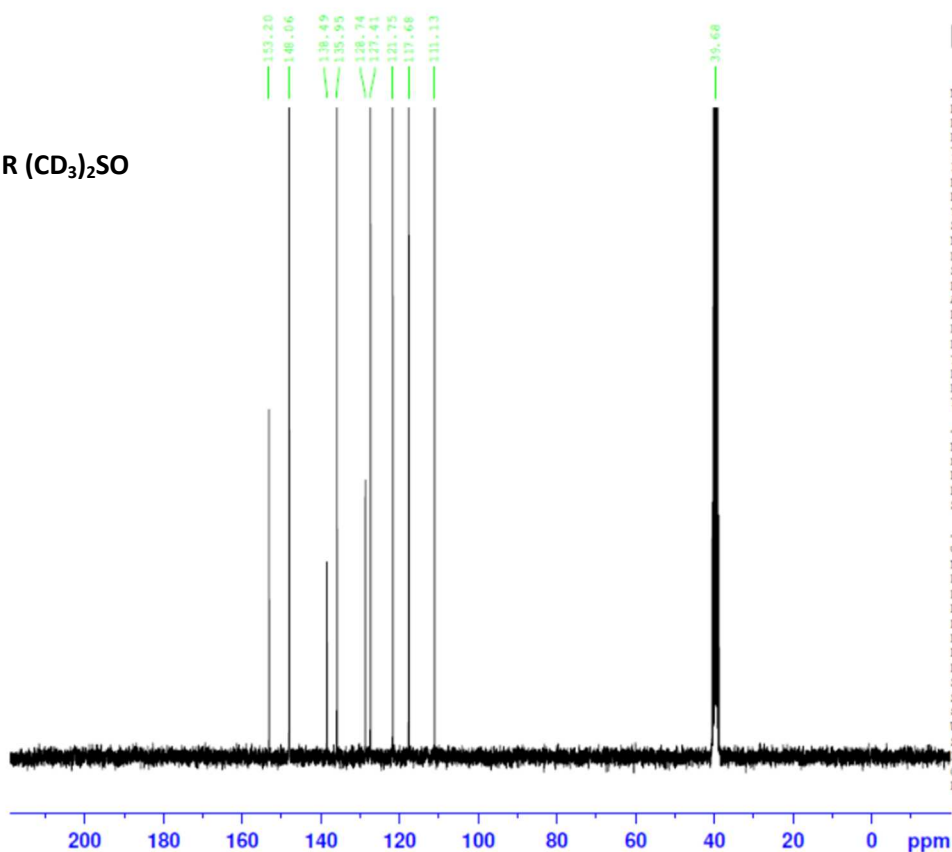
^1H NMR (CD_3) $_2\text{SO}$



NAME Mar07-2013
EXPNO 31
PROCNO 1
Date_ 20130307
Time_ 18.54
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 9806
SOLVENT DMSO
NS 8
DS 2
SWH 3898.129 Hz
FIDRES 0.397525 Hz
AQ 1.2578329 sec
RG 161
DW 128.267 usec
DE 8.00 usec
TE 313.0 K
D1 3.0000000 sec
TD0 1

----- CHANNEL f1 -----
NUC1 ^1H
P1 14.60 usec
PL1 1.10 dB
PL1W 8.87179279 W
SFO1 300.1518535 MHz
SI 32768
SF 300.1500000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

^{13}C NMR (CD_3) $_2\text{SO}$

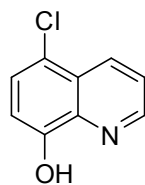


NAME Mar17-2013
EXPNO 30
PROCNO 1
Date_ 20130317
Time_ 22.42
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 1024
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 203
DW 27.733 usec
DE 8.50 usec
TE 323.0 K
D1 2.0000000 sec
D11 0.0300000 sec
TD0 1

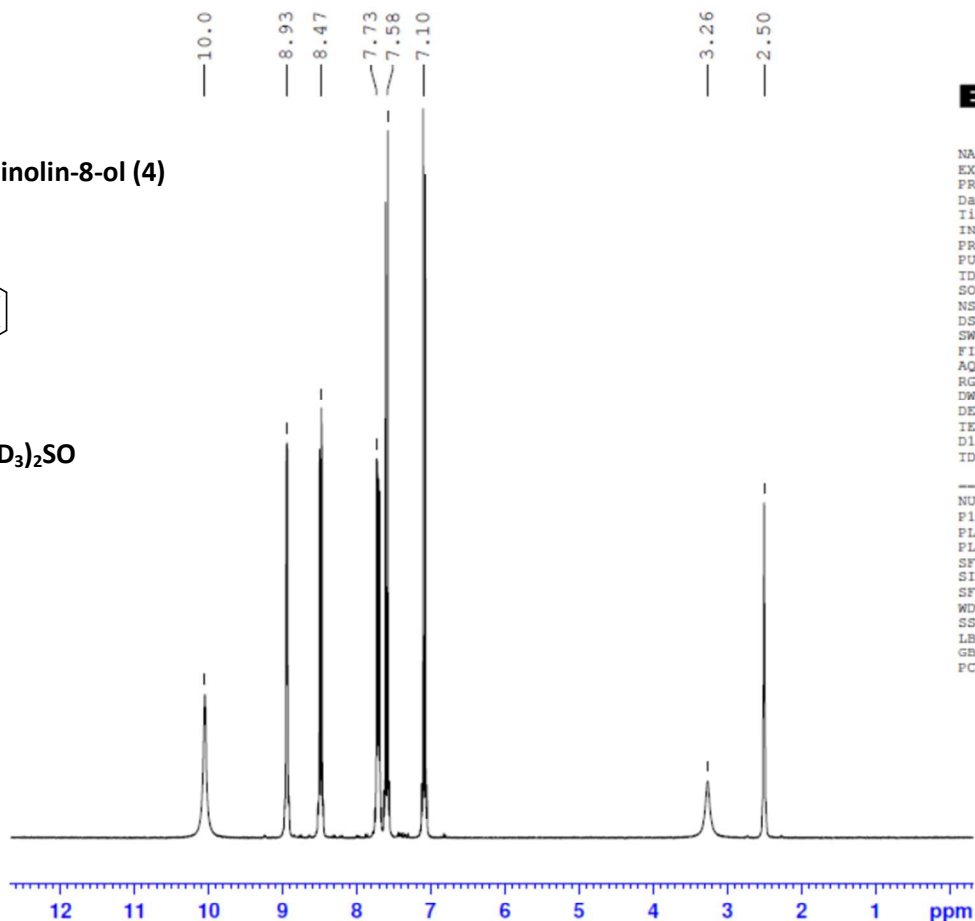
----- CHANNEL f1 -----
NUC1 ^{13}C
P1 9.80 usec
PL1 -0.20 dB
PL1W 32.35229492 W
SFO1 75.4803248 MHz

----- CHANNEL f2 -----
CPDPRG2 waltz16
NUC2 ^1H
PCPD2 100.00 usec
PL2 1.50 dB
PL12 18.40 dB
PL13 21.40 dB
PL2W 8.09117126 W
PL12W 0.16520053 W
PL13W 0.08279639 W
SFO2 300.1512006 MHz
SI 32768
SF 75.4728157 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

5-chloroquinolin-8-ol (4)



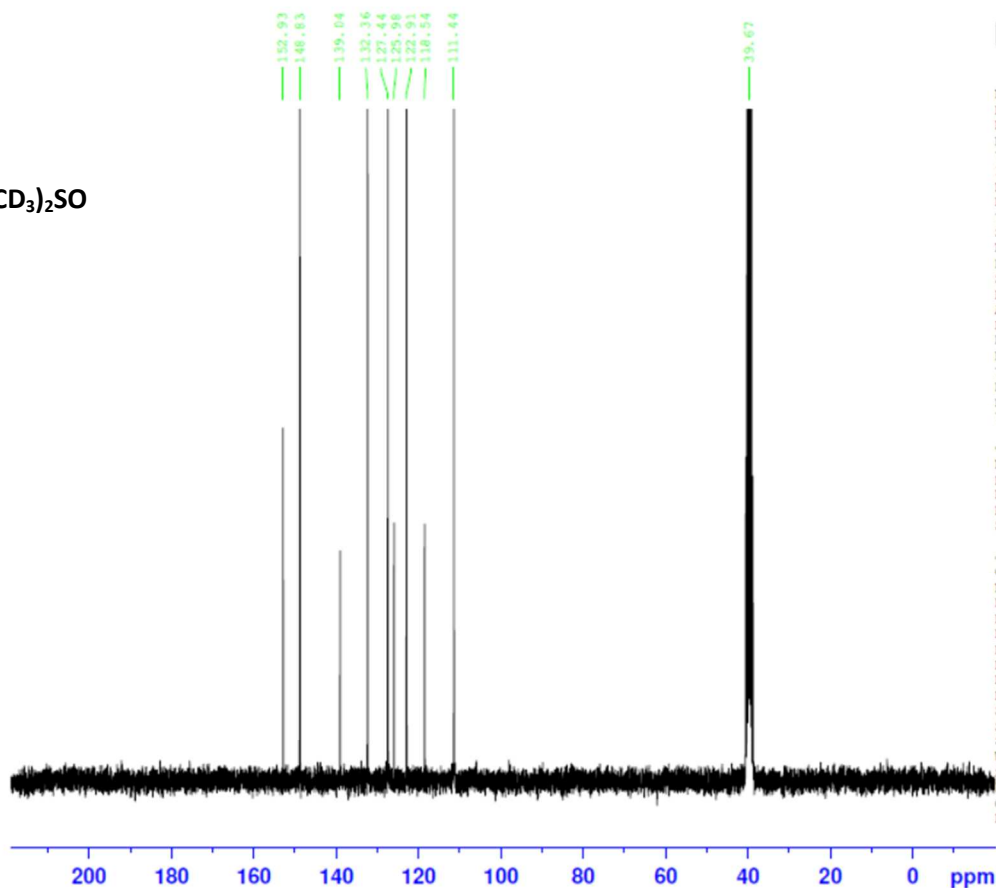
¹H NMR (CD₃)₂SO



NAME Mar07-2013
EXPNO 41
PROCNO 1
Date_ 20130307
Time 20.06
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 9806
SOLVENT DMSO
NS 8
DS 2
SWH 3898.129 Hz
FIDRES 0.397525 Hz
AQ 1.2578329 sec
RG 203
DW 128.267 usec
DE 8.00 usec
TE 313.0 K
D1 3.0000000 sec
TD0 1

----- CHANNEL f1 -----
NUC1 1H
P1 14.60 usec
PL1 1.10 dB
PL1W 8.87179279 W
SFO1 300.1518535 MHz
SI 32768
SF 300.1500000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C NMR (CD₃)₂SO

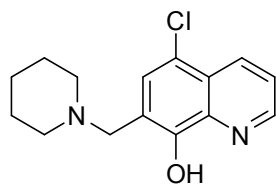


NAME Mar17-2013
EXPNO 40
PROCNO 1
Date_ 20130317
Time 23.55
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 1024
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 203
DW 27.733 usec
DE 8.50 usec
TE 323.0 K
D1 2.0000000 sec
D11 0.0300000 sec
TD0 1

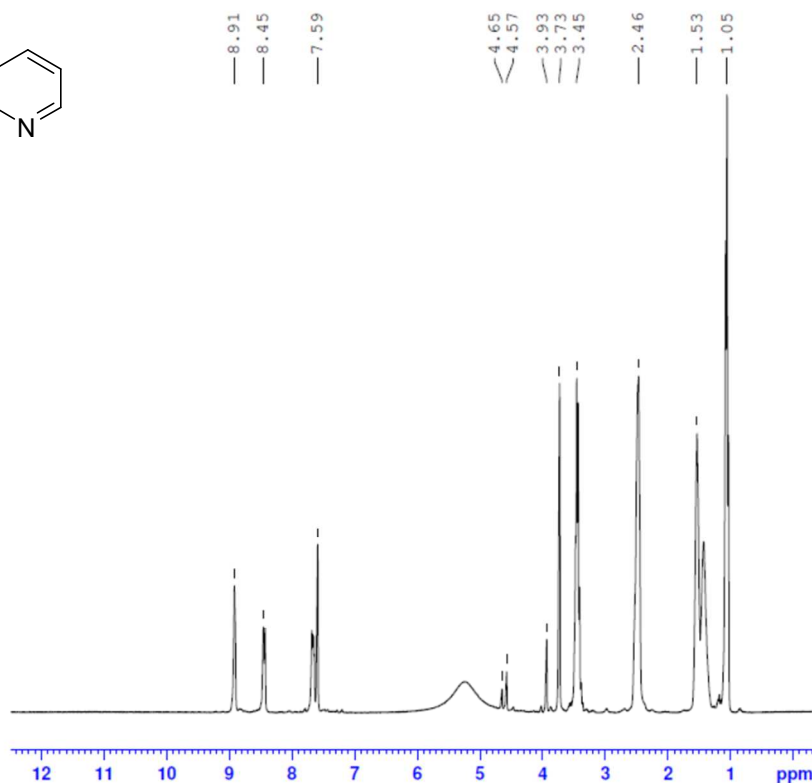
----- CHANNEL f1 -----
NUC1 13C
P1 9.80 usec
PL1 -0.20 dB
PL1W 32.35229492 W
SFO1 75.4803248 MHz

----- CHANNEL f2 -----
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.50 dB
PL12 18.40 dB
PL13 21.40 dB
PL2W 8.09117126 W
PL12W 0.16520053 W
PL13W 0.08279639 W
SFO2 300.1512006 MHz
SI 32768
SF 75.4728157 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

5-chloro-7-(piperidin-1-ylmethyl)quinolin-8-ol (5)



¹H NMR (CD₃)₂SO

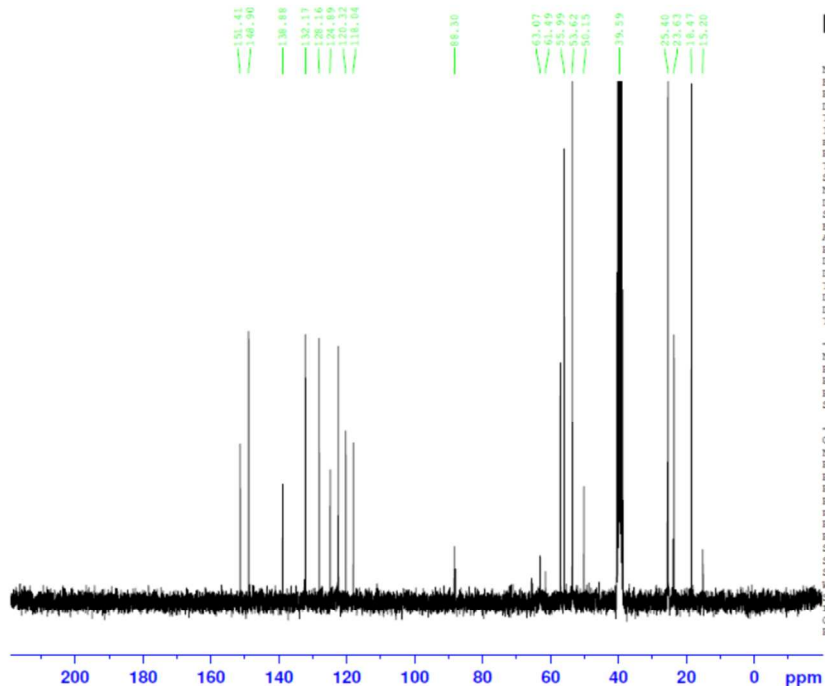


```

NAME      Mar07-2013
EXPNO     111
PROCNO    1
Date_     20130308
Time      4.28
INSTRUM   spect
PROBHD    5 mm F4BBO BB-
PULPROG   zg30
TD         9806
SOLVENT   DMSO
NS         8
DS         2
SWH        3898.129 Hz
FIDRES     0.397525 Hz
AQ         1.2578329 sec
RG         101
DW         128.267 usec
DE         8.00 usec
TE         312.9 K
D1         3.00000000 sec
TD0        1

----- CHANNEL f1 -----
NUC1       1H
P1         14.60 usec
PL1        1.10 dB
PL1W       8.87179279 W
SFO1       300.1518535 MHz
SI         32768
SF         300.1500000 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
    
```

¹³C NMR (CD₃)₂SO



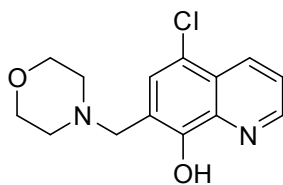
```

NAME      Mar07-2013
EXPNO     110
PROCNO    1
Date_     20130308
Time      4.26
INSTRUM   spect
PROBHD    5 mm F4BBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   DMSO
NS         1024
DS         4
SWH        18028.846 Hz
FIDRES     0.275098 Hz
AQ         1.8175818 sec
RG         203
DW         27.733 usec
DE         8.50 usec
TE         312.9 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1

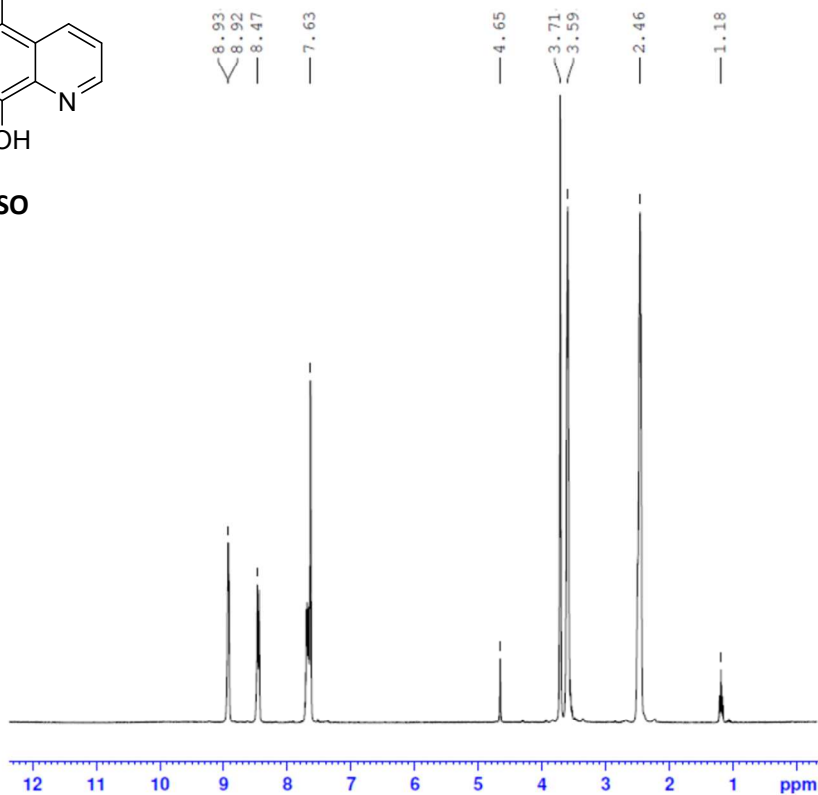
----- CHANNEL f1 -----
NUC1       13C
P1         9.80 usec
PL1        -0.20 dB
PL1W       32.35229492 W
SFO1       75.4803248 MHz

----- CHANNEL f2 -----
CPDPRG2   waltz16
NUC2       1H
PCPD2     100.00 usec
PL2        1.50 dB
PL12       18.40 dB
PL13       21.40 dB
PL12W      0.09117126 W
PL13W      0.16520053 W
SFO2       300.1512006 MHz
SI         32768
SF         75.4728157 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
    
```


5-chloro-7-(morpholinomethyl)quinolin-8-ol (6)



¹H NMR (CD₃)₂SO



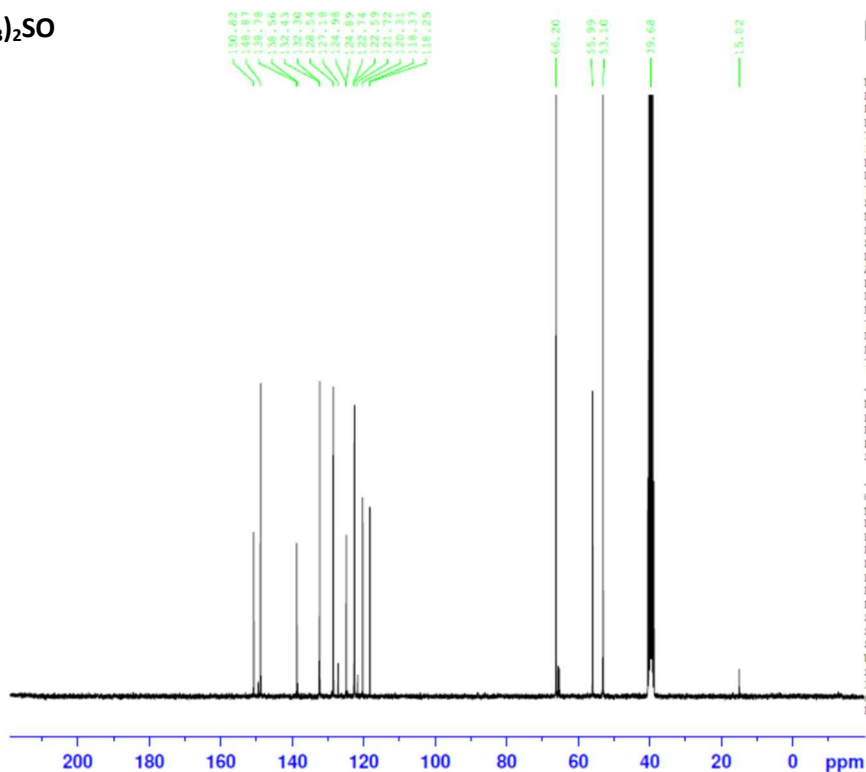
```

NAME      Mari4-2013
EXPNO     11
PROCNO    1
Date_     20130315
Time      3.58
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD         9806
SOLVENT   DMSO
NS         8
DS         2
SWH        3898.129 Hz
FIDRES     0.397525 Hz
AQ         1.2578329 sec
RG         144
DW         128.267 usec
DE         8.00 usec
TE         322.9 K
D1         3.00000000 sec
TD0        1
  
```

```

----- CHANNEL f1 -----
NUC1      1H
P1        14.60 usec
PL1       1.10 dB
PL1W      8.87179279 W
SFO1      300.1518535 MHz
SI        32768
SF        300.1500000 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00
  
```

¹³C NMR (CD₃)₂SO



```

NAME      Mari4-2013
EXPNO     10
PROCNO    1
Date_     20130315
Time      3.52
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   DMSO
NS         10240
DS         4
SWH        18028.846 Hz
FIDRES     0.275098 Hz
AQ         1.8175818 sec
RG         203
DW         27.733 usec
DE         8.50 usec
TE         323.0 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1
  
```

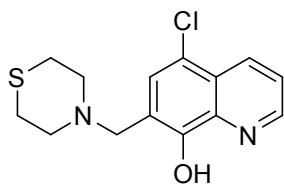
```

----- CHANNEL f1 -----
NUC1      13C
P1        9.80 usec
PL1       -0.20 dB
PL1W      32.35229492 W
SFO1      75.4803248 MHz
  
```

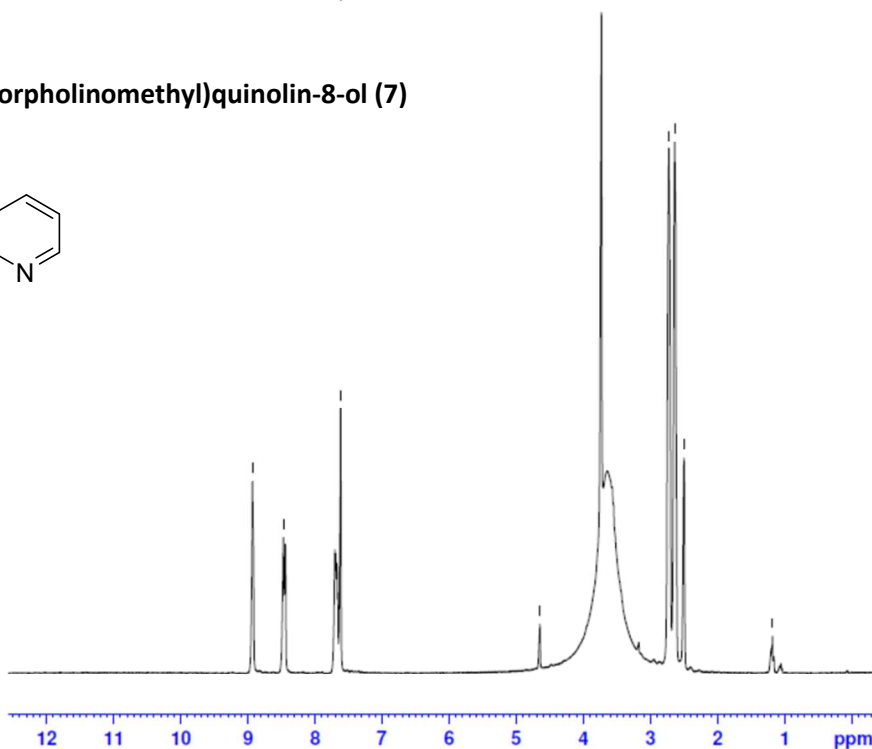
```

----- CHANNEL f2 -----
CPDPRG2   waltz16
NUC2      1H
PCPD2     100.00 usec
PL2       1.50 dB
PL12      18.40 dB
PL13      21.40 dB
PL2W      8.09117126 W
PL12W     0.16520053 W
PL13W     0.08279639 W
SFO2      300.1512006 MHz
SI        32768
SF        75.4728157 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40
  
```

5-chloro-7-(thiomorpholinomethyl)quinolin-8-ol (7)



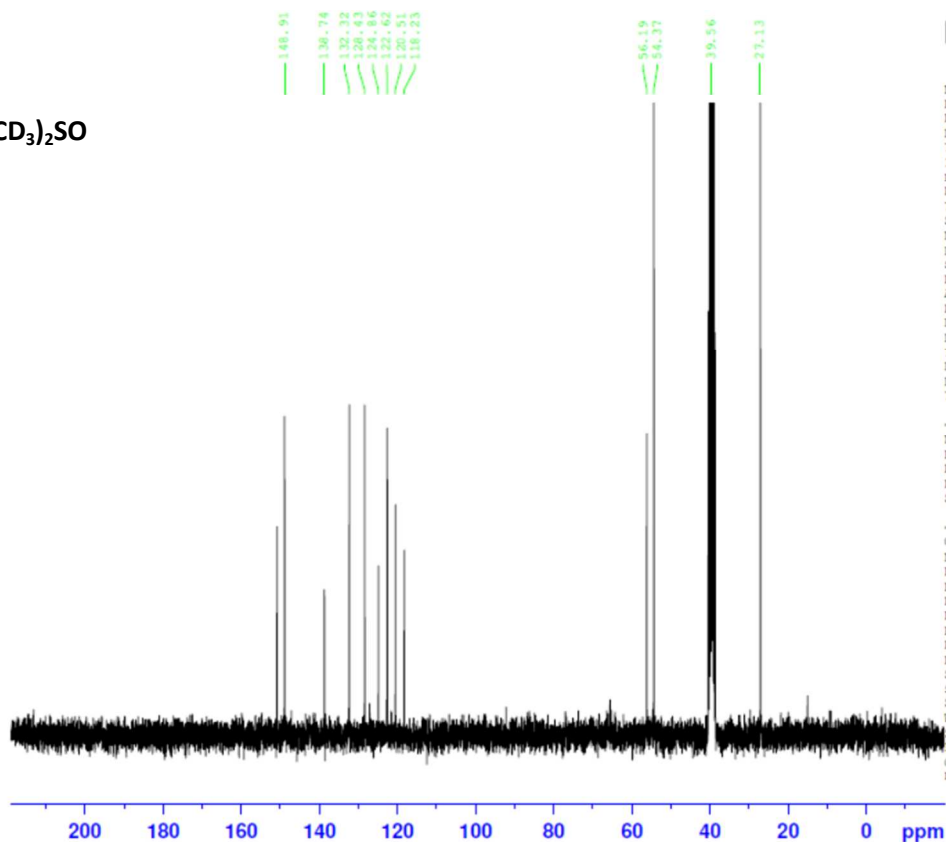
^1H NMR (CD_3) $_2\text{SO}$



NAME Mar07-2013
EXPNO 51
PROCNO 1
Date_ 20130308
Time 6.51
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 9806
SOLVENT DMSO
NS 8
DS 2
SWH 3896.129 Hz
FIDRES 0.397525 Hz
AQ 1.2578329 sec
RG 128
DW 128.267 usec
DE 8.00 usec
TE 313.0 K
D1 3.0000000 sec
TD0 1

----- CHANNEL f1 -----
NUC1 1H
P1 14.60 usec
PL1 1.10 dB
PL1W 8.87179279 W
SFO1 300.1518535 MHz
SI 32768
SF 300.1500000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

^{13}C NMR (CD_3) $_2\text{SO}$

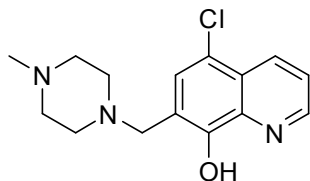


NAME Mar07-2013
EXPNO 50
PROCNO 1
Date_ 20130308
Time 6.49
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 1024
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 203
DW 27.733 usec
DE 8.50 usec
TE 313.1 K
D1 2.0000000 sec
D11 0.0300000 sec
TD0 1

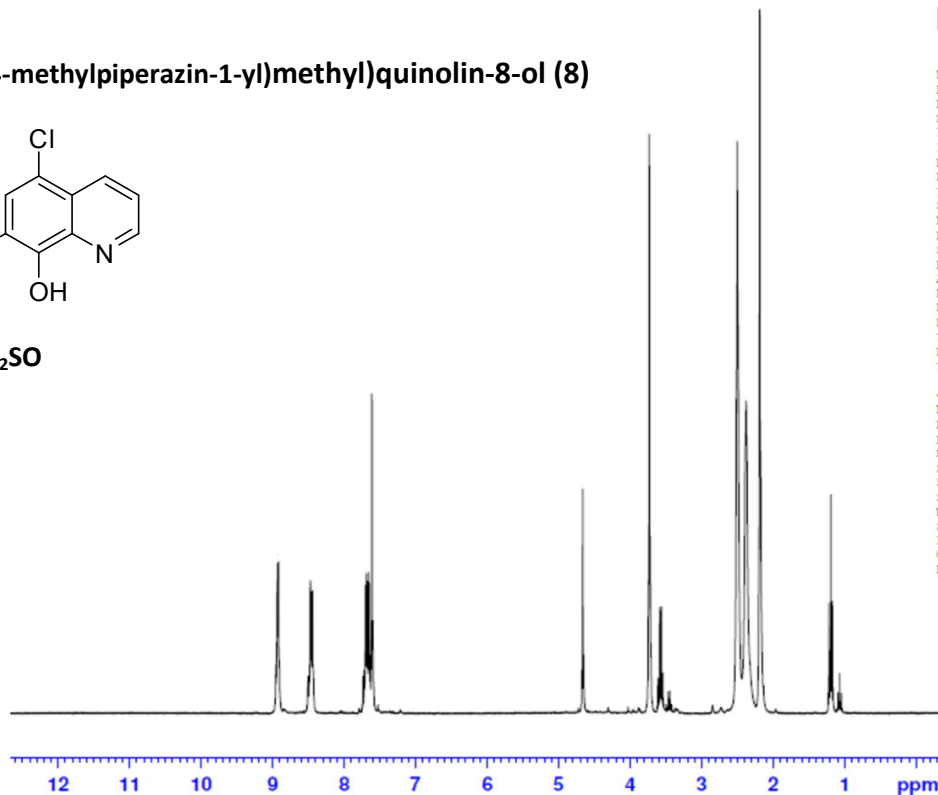
----- CHANNEL f1 -----
NUC1 13C
P1 9.80 usec
PL1 -0.20 dB
PL1W 32.35229492 W
SFO1 75.4803248 MHz

----- CHANNEL f2 -----
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.50 dB
PL12 18.40 dB
PL13 21.40 dB
PL2W 8.09117126 W
PL12W 0.16520053 W
PL13W 0.08279639 W
SFO2 300.1512006 MHz
SI 32768
SF 75.4728157 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

5-chloro-7-((4-methylpiperazin-1-yl)methyl)quinolin-8-ol (8)



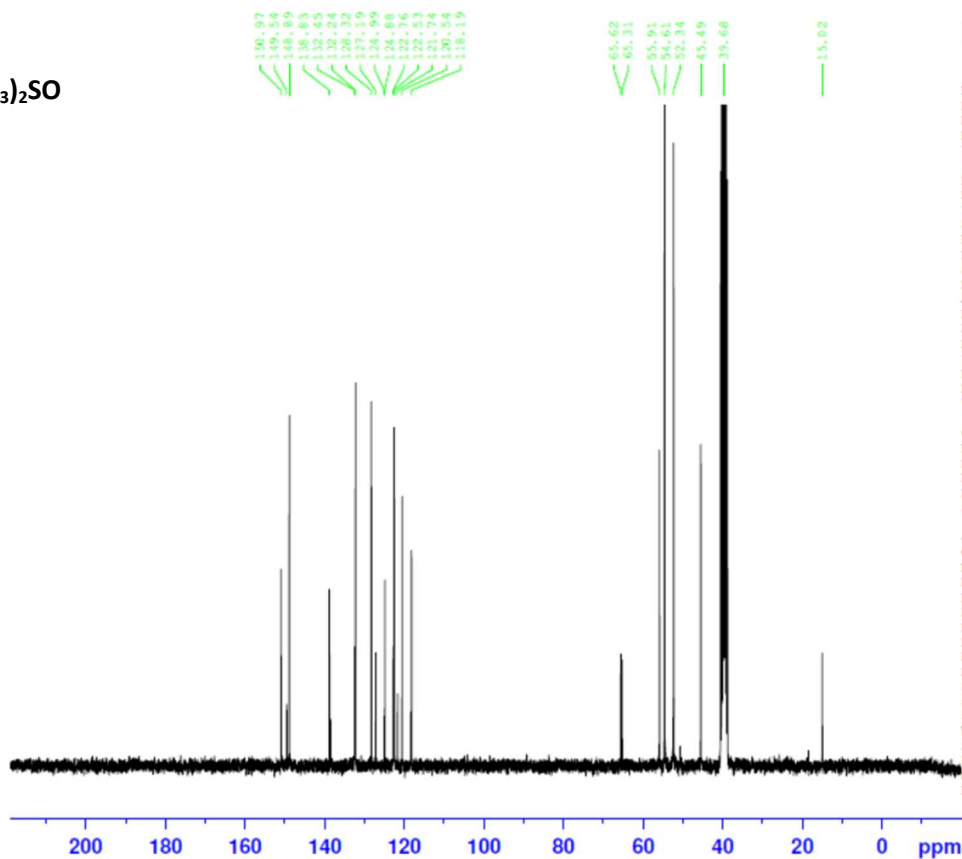
^1H NMR (CD_3) $_2\text{SO}$



NAME Mar15-2013
EXPNO 11
PROCNO 1
Date_ 20130316
Time 4.00
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 9806
SOLVENT DMSO
NS 8
DS 2
SWH 3898.129 Hz
FIDRES 0.397525 Hz
AQ 1.2578329 sec
RG 181
DW 128.267 usec
DE 8.00 usec
TE 323.0 K
D1 3.0000000 sec
TD0 1

CHANNEL f1
NUC1 1H
P1 14.60 usec
PL1 1.10 dB
PL1W 8.87179279 W
SFO1 300.1518535 MHz
SI 32768
SF 300.1500000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

^{13}C NMR (CD_3) $_2\text{SO}$

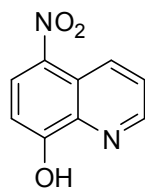


NAME Mar15-2013
EXPNO 10
PROCNO 1
Date_ 20130316
Time 3.55
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 10240
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 203
DW 27.733 usec
DE 8.50 usec
TE 322.8 K
D1 2.0000000 sec
D11 0.0300000 sec
TD0 1

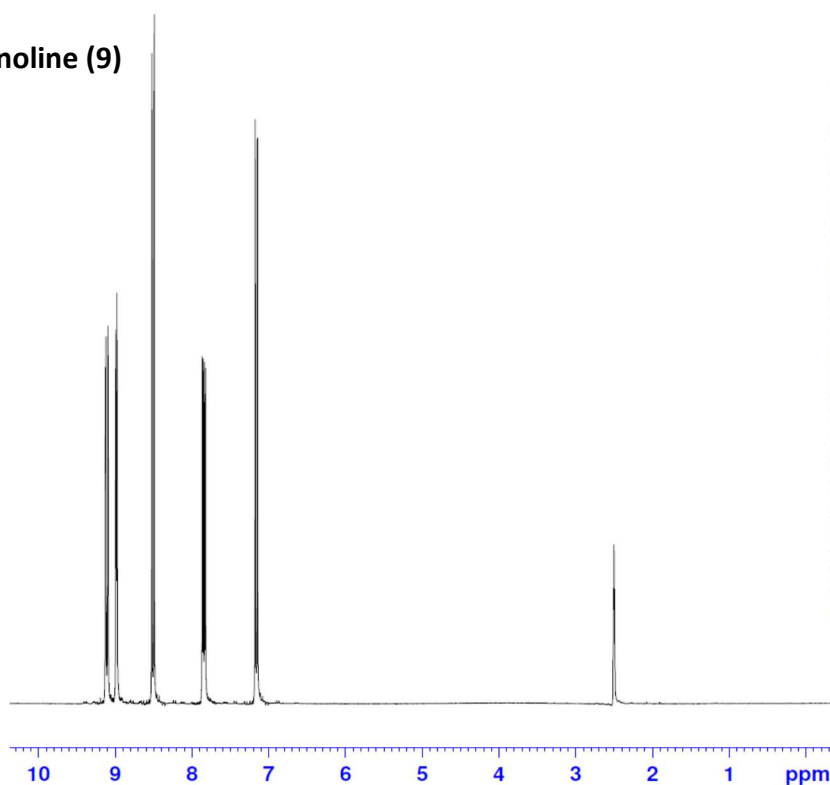
CHANNEL f1
NUC1 13C
P1 9.80 usec
PL1 -0.20 dB
PL1W 32.35229492 W
SFO1 75.4803248 MHz

CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.50 dB
PL12 18.40 dB
PL13 21.40 dB
PL2W 8.09117126 W
PL12W 0.16520053 W
PL13W 0.08279639 W
SFO2 300.1512006 MHz
SI 32768
SF 75.4728157 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

8-hydroxy-5-nitroquinoline (9)



¹H NMR (CD₃)₂SO

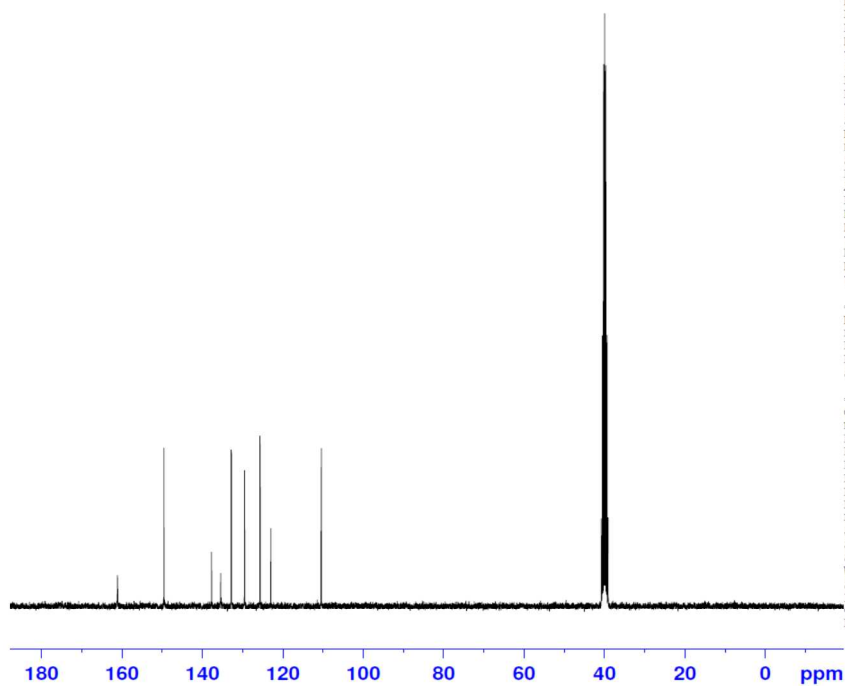


```

NAME      BP manuscript
EXPNO     1
PROCNO    1
Date_     20120802
Time      15.48
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD        9806
SOLVENT   DMSO
NS         8
DS         2
SWH        3898.129 Hz
FIDRES     0.397525 Hz
AQ         1.2578329 sec
RG         161
DW         128.267 usec
DE         8.00 usec
TE         296.7 K
D1         3.0000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       1H
P1         14.60 usec
PL1        1.10 dB
PL1W       8.87179279 W
SFO1       300.1518535 MHz
SI         32768
SF         300.1500000 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
    
```

¹³C NMR (CD₃)₂SO



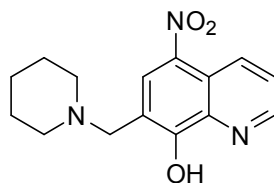
```

NAME      BP manuscript
EXPNO     2
PROCNO    2
Date_     20120802
Time      16.14
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD        65536
SOLVENT   DMSO
NS         4
DS         4
SWH        18028.846 Hz
FIDRES     0.275098 Hz
AQ         1.8175818 sec
RG         203
DW         27.733 usec
DE         8.50 usec
TE         297.0 K
D1         2.0000000 sec
D11        0.0300000 sec
TD0        1

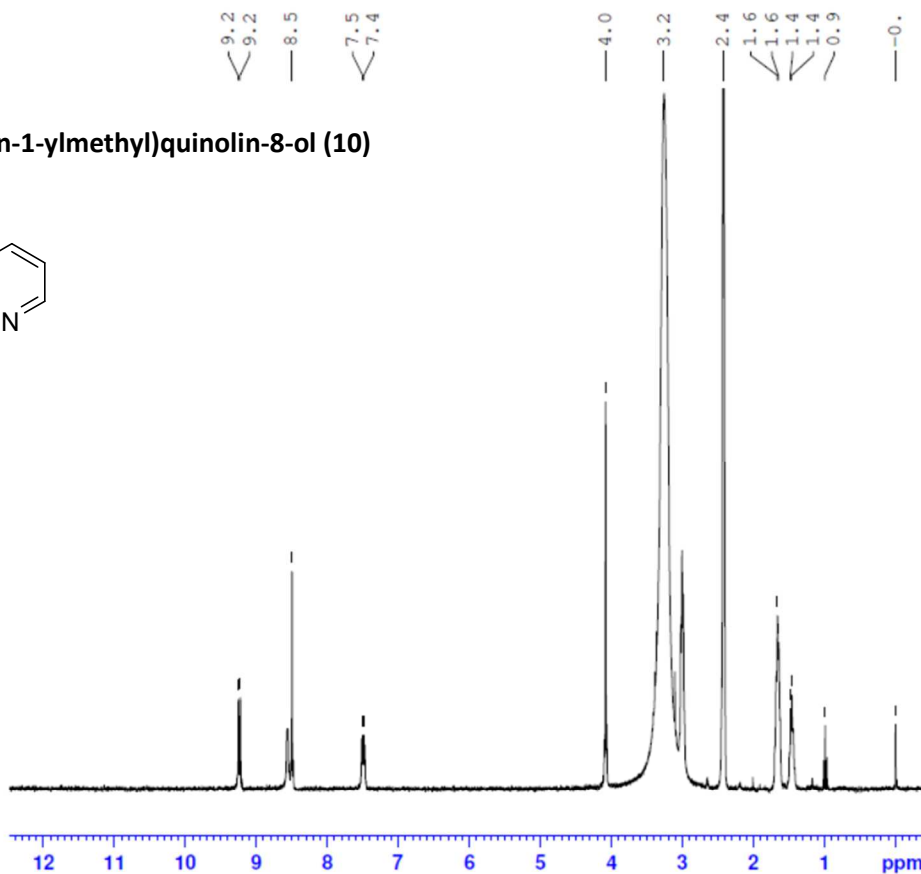
===== CHANNEL f1 =====
NUC1       13C
P1         9.80 usec
PL1        -0.20 dB
PL1W       32.35229492 W
SFO1       75.4803248 MHz

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2       1H
PCPD2     100.00 usec
PL2        1.50 dB
PL12       18.40 dB
PL13       21.40 dB
PL2W       8.09117126 W
PL12W      0.16520053 W
PL13W      0.00279639 W
SFO2       300.1512006 MHz
SI         32768
SF         75.4727780 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.00
    
```

5-nitro-7-(piperidin-1-ylmethyl)quinolin-8-ol (10)



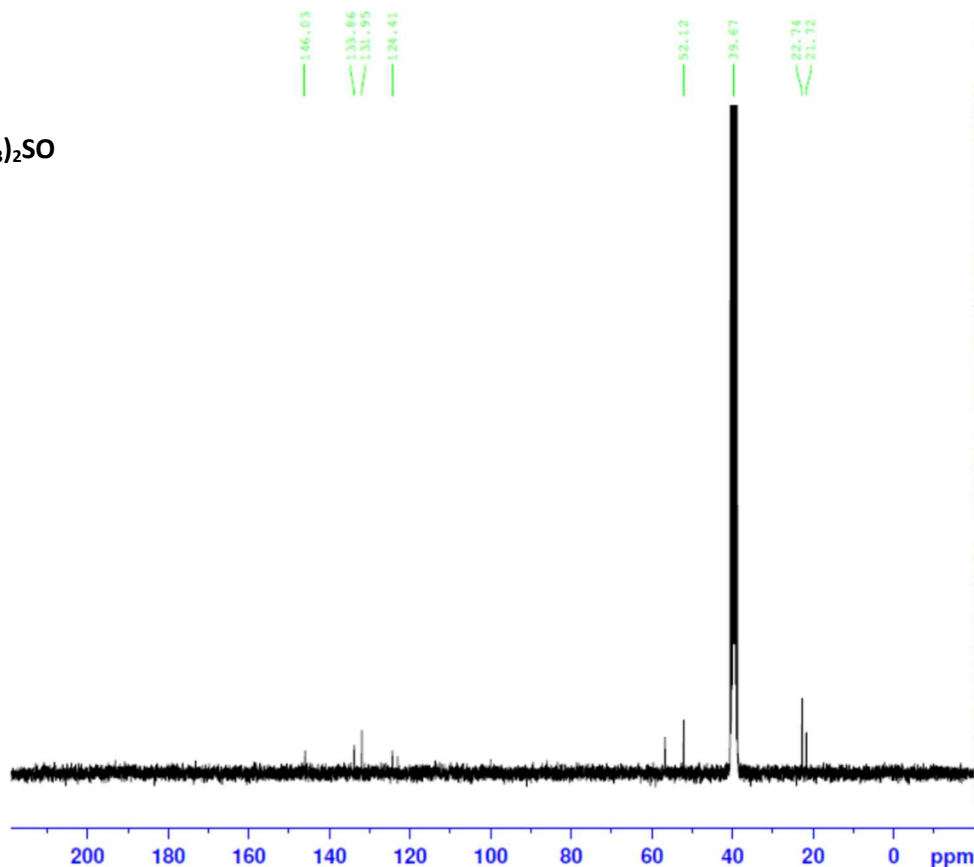
¹H NMR (CD₃)₂SO



NAME Mar09-2013
EXPNO 11
PROCNO 1
Date_ 20130309
Time_ 23.32
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 9806
SOLVENT DMSO
NS 8
DS 2
SWH 3898.129 Hz
FIDRES 0.397525 Hz
AQ 1.2578329 sec
RG 203
DW 128.267 usec
DE 8.00 usec
TE 323.0 K
D1 3.00000000 sec
TD0 1

CHANNEL f1
NUC1 1H
P1 14.60 usec
PL1 1.10 dB
PL1W 8.87179279 W
SFO1 300.1518535 MHz
SI 32768
SF 300.1500228 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C NMR (CD₃)₂SO

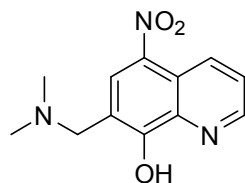


NAME Mar09-2013
EXPNO 10
PROCNO 1
Date_ 20130309
Time_ 23.29
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 10240
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 203
DW 27.733 usec
DE 8.50 usec
TE 322.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

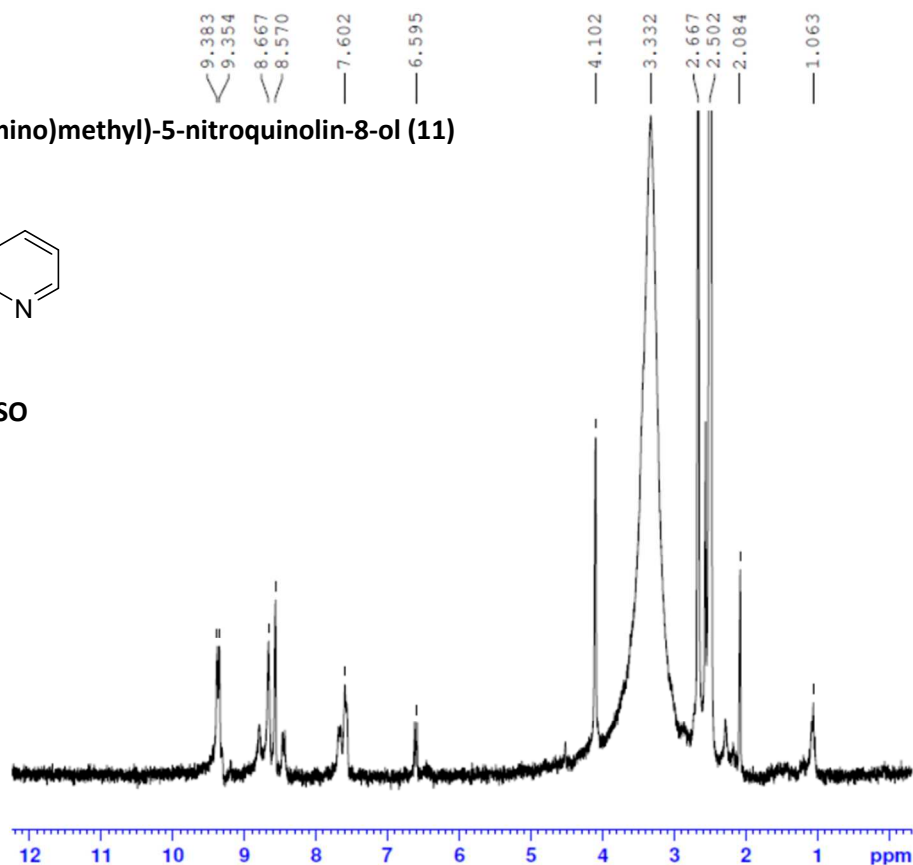
CHANNEL f1
NUC1 13C
P1 9.80 usec
PL1 -0.20 dB
PL1W 32.35229492 W
SFO1 75.4803248 MHz

CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.50 dB
PL12 18.40 dB
PL13 21.40 dB
PL2W 8.09117126 W
PL12W 0.16520053 W
PL13W 0.08279639 W
SFO2 300.1512006 MHz
SI 32768
SF 75.4728157 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

7-((dimethylamino)methyl)-5-nitroquinolin-8-ol (11)



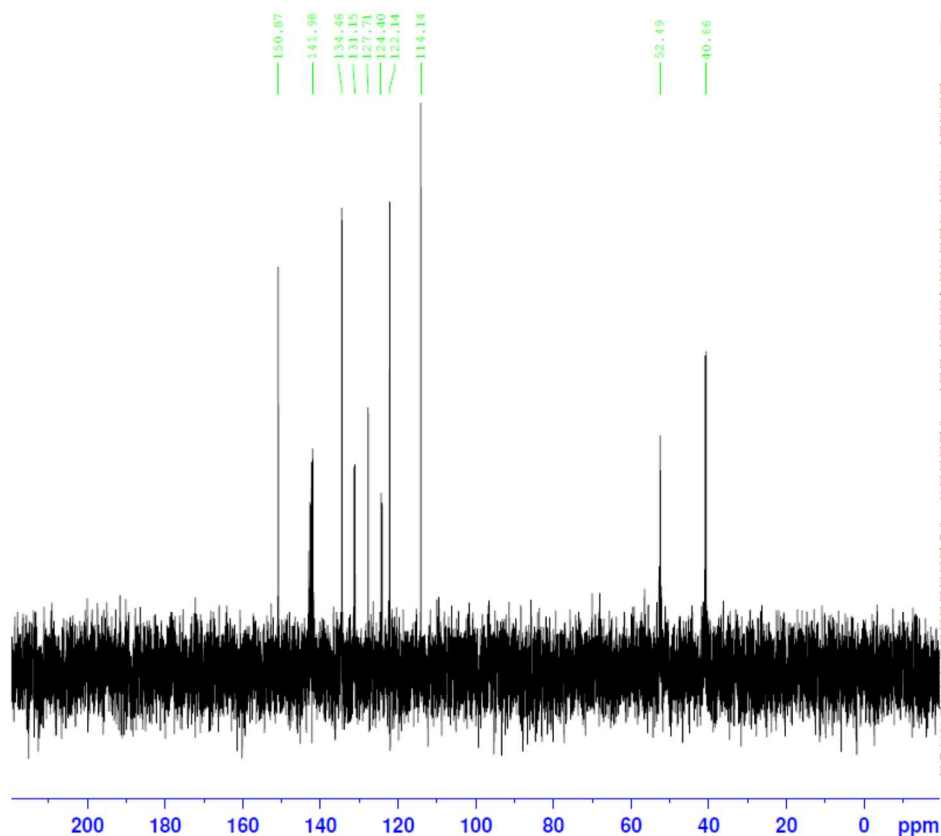
¹H NMR (CD₃)₂SO



NAME Mar09-2013
EXPNO 21
PROCNO 1
Date_ 20130310
Time 11.41
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 9806
SOLVENT DMSO
NS 8
DS 2
SWH 3898.129 Hz
FIDRES 0.397525 Hz
AQ 1.2578329 sec
RG 203
DW 128.267 usec
DE 8.00 usec
TE 323.0 K
D1 3.0000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 14.60 usec
PL1 1.10 dB
PL1W 8.87179279 W
SFO1 300.1518535 MHz
SI 32768
SF 300.1500000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C NMR D₂O

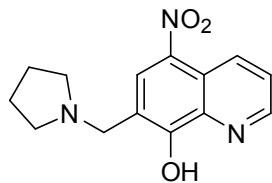


NAME Mar22-2013-beau
EXPNO 10
PROCNO 1
Date_ 20130322
Time 18.26
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT D2O
NS 1024
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 203
DW 27.733 usec
DE 8.50 usec
TE 296.2 K
D1 2.0000000 sec
D11 0.0300000 sec
TD0 1

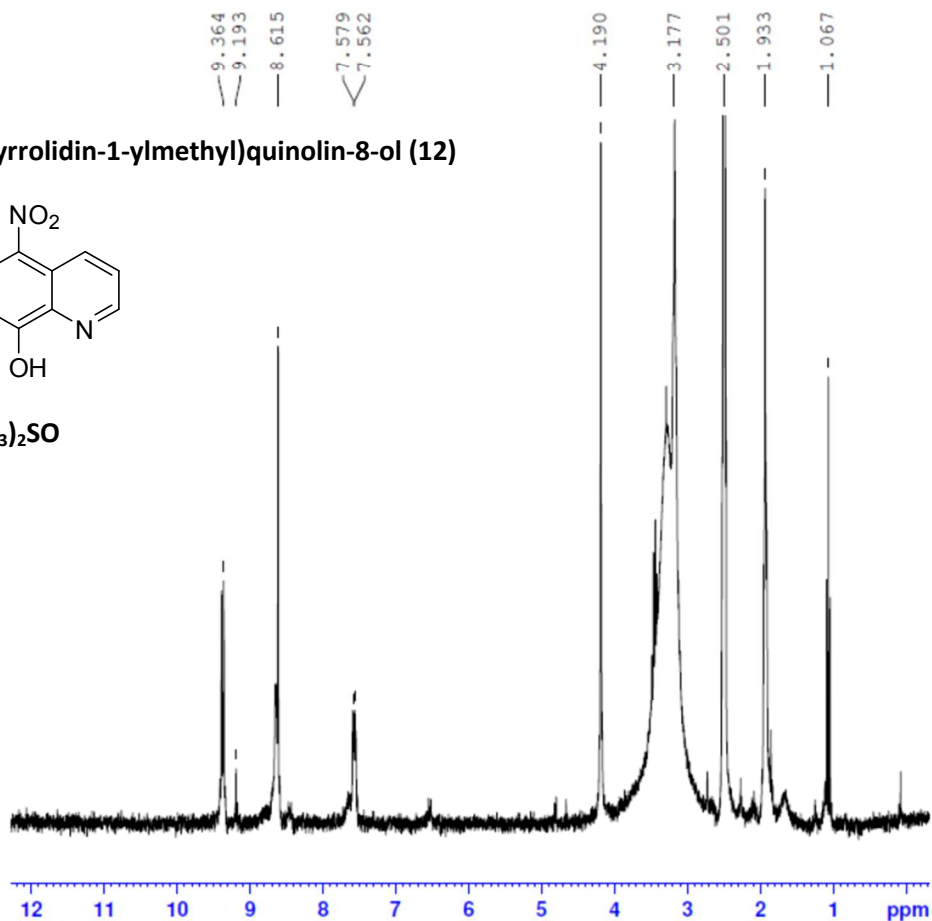
===== CHANNEL f1 =====
NUC1 13C
P1 9.80 usec
PL1 -0.20 dB
PL1W 32.35229492 W
SFO1 75.4803248 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.50 dB
PL12 18.40 dB
PL13 21.40 dB
PL2W 8.09117126 W
PL12W 0.16520053 W
PL13W 0.08279639 W
SFO2 300.1512006 MHz
SI 32768
SF 75.4727780 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

5-nitro-7-(pyrrolidin-1-ylmethyl)quinolin-8-ol (12)



¹H NMR (CD₃)₂SO



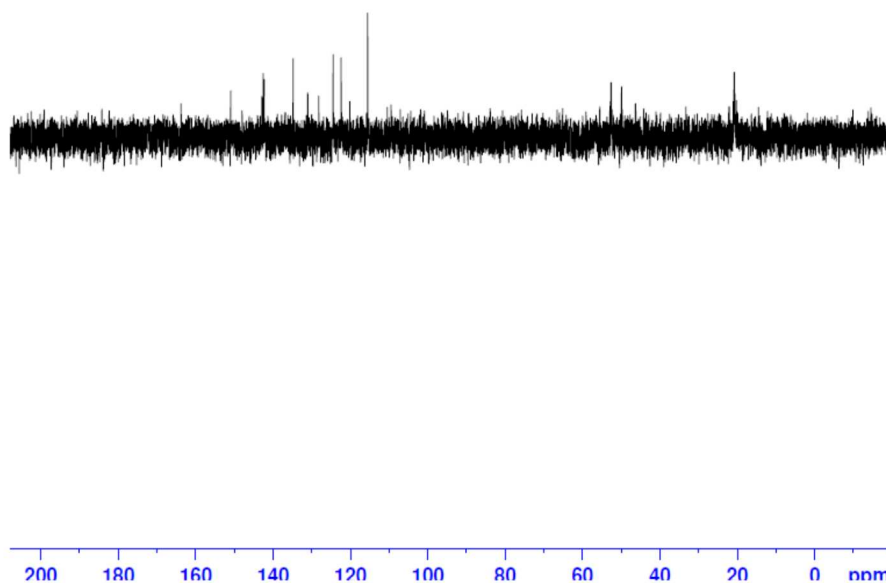
```

NAME      Mar09-2013
EXPNO     31
PROCNO    1
Date_     20130310
Time      22.49
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD         9806
SOLVENT   DMSO
NS         8
DS         2
SWH        3898.129 Hz
FIDRES     0.397525 Hz
AQ         1.2578329 sec
RG         203
DW         128.267 usec
DE         8.00 usec
TE         323.0 K
D1         3.00000000 sec
TD0        1
    
```

```

===== CHANNEL f1 =====
NUC1       1H
P1         14.60 usec
PL1        1.10 dB
PL1W       8.87179279 W
SFO1       300.1518535 MHz
SI         32768
SF         300.1500000 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
    
```

¹³C NMR D₂O



```

NAME      Mar22-2013-beau
EXPNO     20
PROCNO    1
Date_     20130322
Time      20.12
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   D2O
NS         1024
DS         4
SWH        18028.846 Hz
FIDRES     0.275098 Hz
AQ         1.8175818 sec
RG         203
DW         27.733 usec
DE         8.50 usec
TE         296.1 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1
    
```

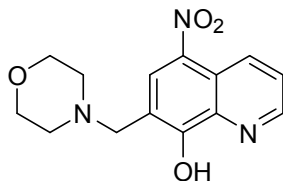
```

===== CHANNEL f1 =====
NUC1       13C
P1         9.80 usec
PL1        -0.20 dB
PL1W       32.35229492 W
SFO1       75.4803248 MHz
    
```

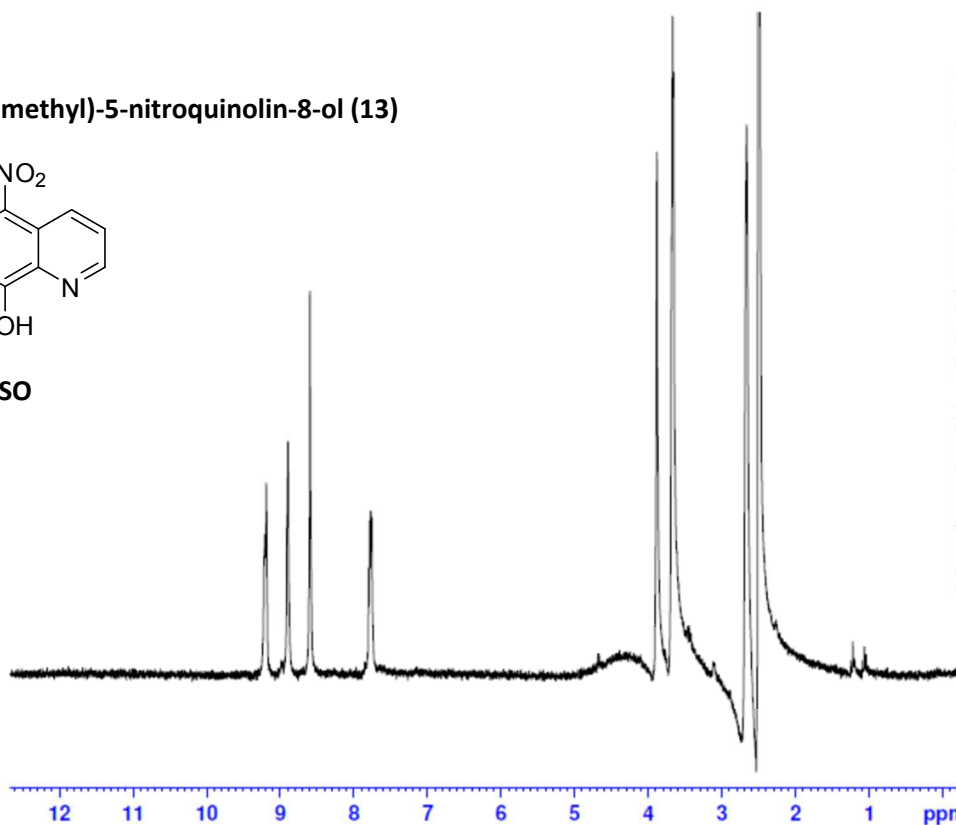
```

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2        1H
PCPD2      100.00 usec
PL2         1.50 dB
PL12       18.40 dB
PL13       21.40 dB
PL2W       8.09117126 W
PL12W      0.16520053 W
PL13W      0.08279639 W
SFO2       300.1512006 MHz
SI         32768
SF         75.4727780 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
    
```


7-(morpholinomethyl)-5-nitroquinolin-8-ol (13)



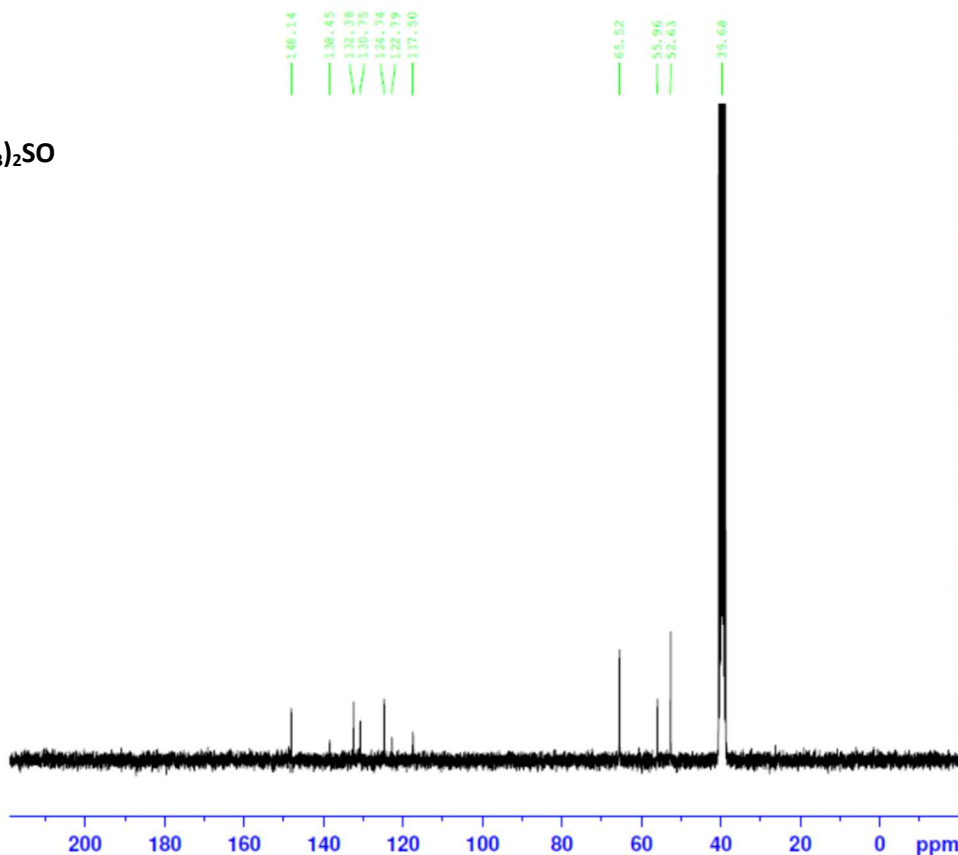
^1H NMR (CD_3) $_2\text{SO}$



NAME Mar09-2013
EXPNO 41
PROCNO 1
Date_ 20130311
Time 9.57
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 9806
SOLVENT DMSO
NS 8
DS 2
SWH 3898.129 Hz
FIDRES 0.397525 Hz
AQ 1.2578329 sec
RG 203
DW 128.267 usec
DE 8.00 usec
TE 322.8 K
D1 3.0000000 sec
TD0 1

----- CHANNEL f1 -----
NUC1 ^1H
P1 14.60 usec
PL1 1.10 dB
PL1W 8.87179279 W
SFO1 300.1518535 MHz
SI 32768
SF 300.1500000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

^{13}C NMR (CD_3) $_2\text{SO}$

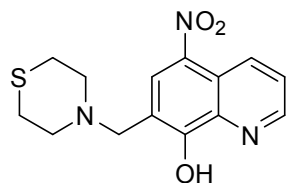


NAME Mar09-2013
EXPNO 40
PROCNO 1
Date_ 20130311
Time 9.55
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 10240
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 203
DW 27.733 usec
DE 8.50 usec
TE 322.9 K
D1 2.0000000 sec
D11 0.03000000 sec
TD0 1

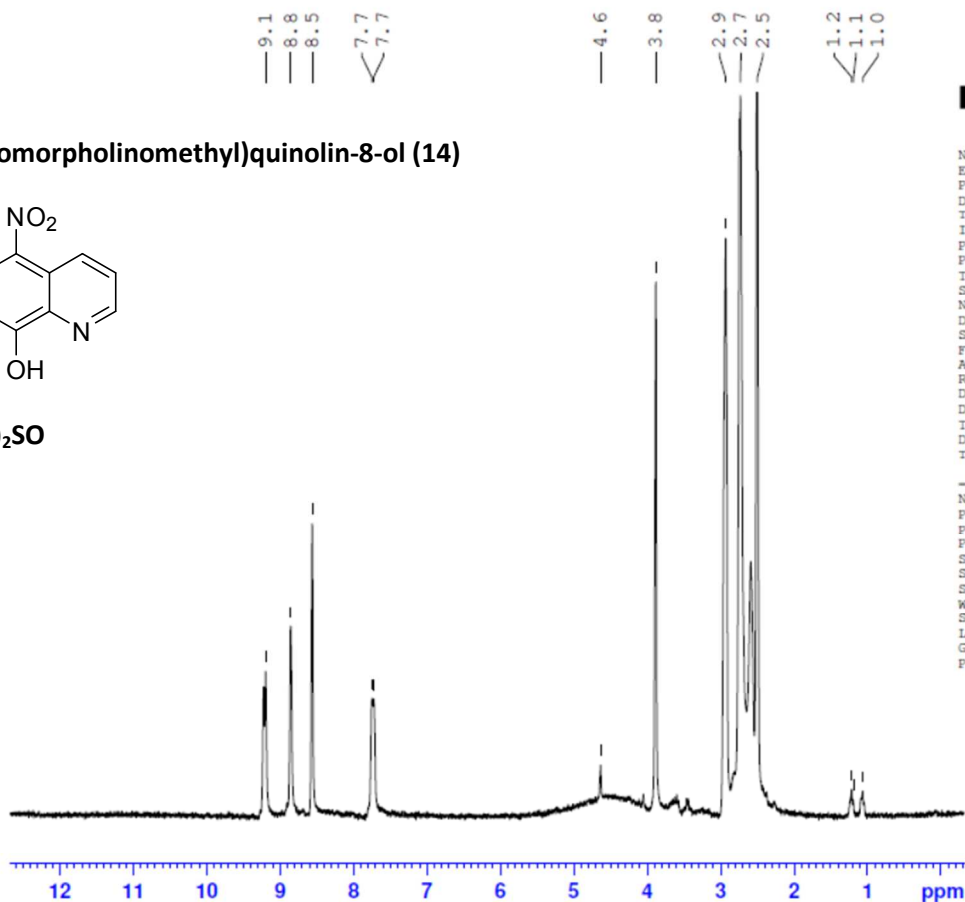
----- CHANNEL f1 -----
NUC1 ^{13}C
P1 9.80 usec
PL1 -0.20 dB
PL1W 32.35229492 W
SFO1 75.4803248 MHz

----- CHANNEL f2 -----
CPDPRG2 waltz16
NUC2 ^1H
PCPD2 100.00 usec
PL2 1.50 dB
PL12 18.40 dB
PL13 21.40 dB
PL1W 8.09117126 W
PL12W 0.16520053 W
PL13W 0.08279639 W
SFO2 300.1512006 MHz
SI 32768
SF 75.4728157 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

5-nitro-7-(thiomorpholinomethyl)quinolin-8-ol (14)



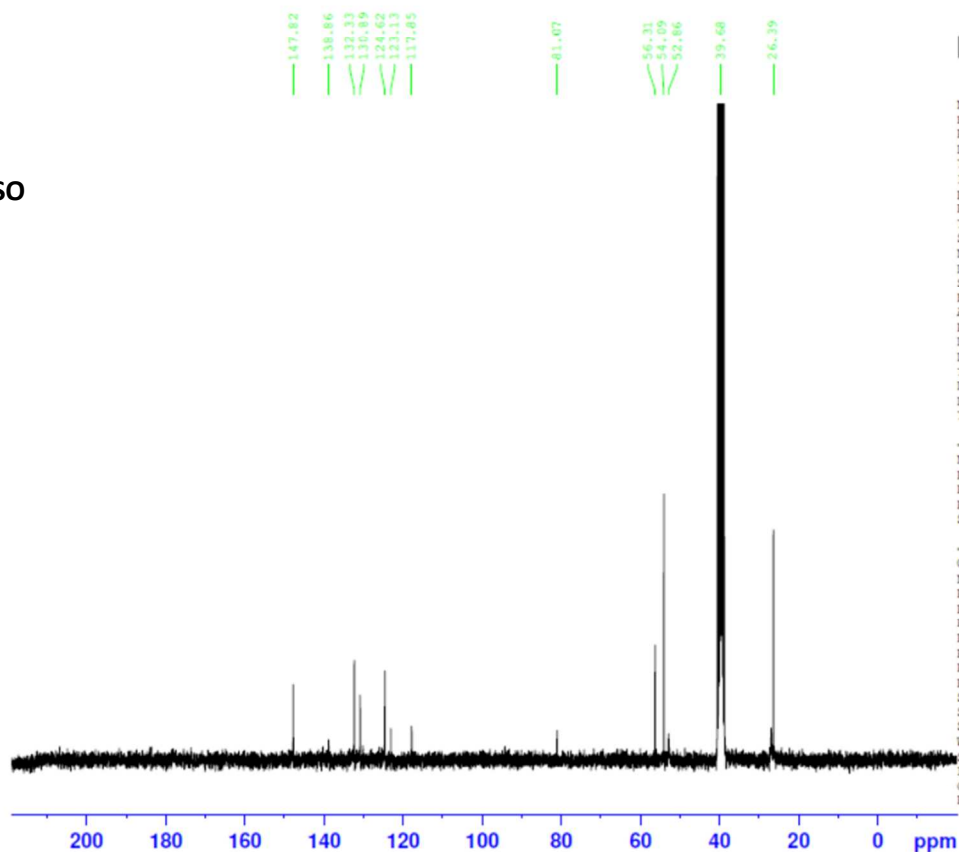
¹H NMR (CD₃)₂SO



NAME Mar11-2013
EXPNO 11
PROCNO 1
Date_ 20130312
Time 4.27
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 9806
SOLVENT DMSO
NS 8
DS 2
SWH 3898.129 Hz
FIDRES 0.397525 Hz
AQ 1.2578329 sec
RG 203
DW 128.267 usec
DE 8.00 usec
TE 322.8 K
D1 3.0000000 sec
TD0 1

CHANNEL f1
NUC1 1H
P1 14.60 usec
PL1 1.10 dB
PL1W 8.87179279 W
SFO1 300.1518535 MHz
SI 32768
SF 300.1500000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C NMR (CD₃)₂SO

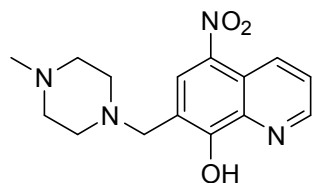


NAME Mar11-2013
EXPNO 10
PROCNO 1
Date_ 20130312
Time 4.24
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 10240
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 203
DW 27.733 usec
DE 8.50 usec
TE 323.0 K
D1 2.0000000 sec
D11 0.03000000 sec
TD0 1

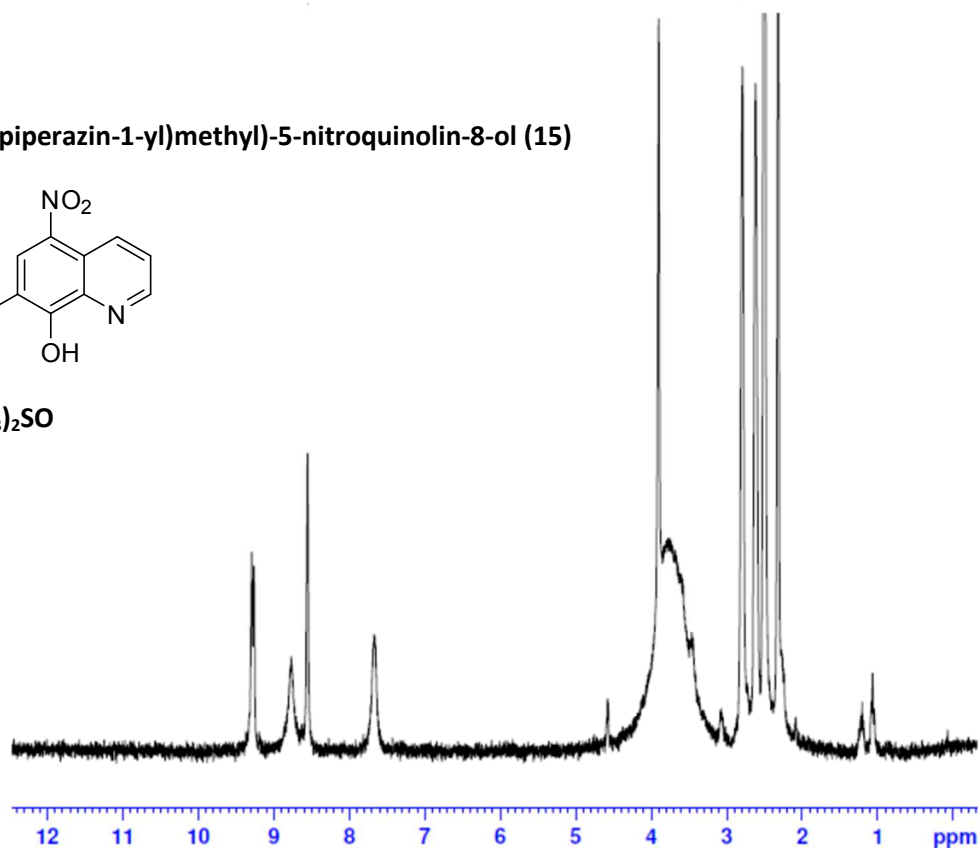
CHANNEL f1
NUC1 13C
P1 9.80 usec
PL1 -0.20 dB
PL1W 32.35229492 W
SFO1 75.4803248 MHz

CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.50 dB
PL12 18.40 dB
PL13 21.40 dB
PL2W 8.09117126 W
PL12W 0.16520053 W
PL13W 0.08279639 W
SFO2 300.1512006 MHz
SI 32768
SF 75.4728157 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

7-((4-methylpiperazin-1-yl)methyl)-5-nitroquinolin-8-ol (15)



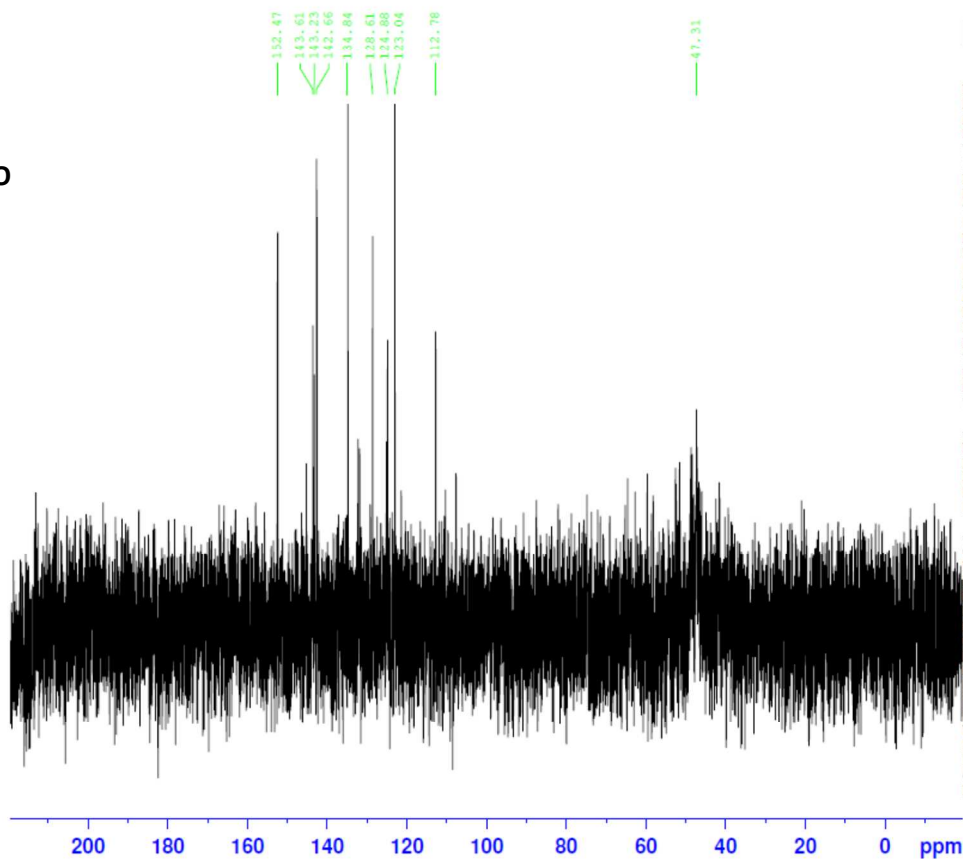
¹H NMR (CD₃)₂SO



NAME Mar12-2013
EXPNO 11
PROCNO 1
Date_ 20130313
Time_ 3.58
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 9806
SOLVENT DMSO
NS 8
DS 2
SWH 3898.129 Hz
FIDRES 0.397525 Hz
AQ 1.2578329 sec
RG 203
DW 128.267 usec
DE 8.00 usec
TE 323.0 K
D1 3.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 14.60 usec
PL1 1.10 dB
PL1W 8.87179279 W
SFO1 300.1518535 MHz
SI 32768
SF 300.1500000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C NMR D₂O

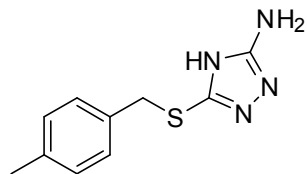


NAME Mar22-2013-beau
EXPNO 30
PROCNO 1
Date_ 20130322
Time_ 21.56
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT D2O
NS 1024
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 203
DW 27.733 usec
DE 8.50 usec
TE 296.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

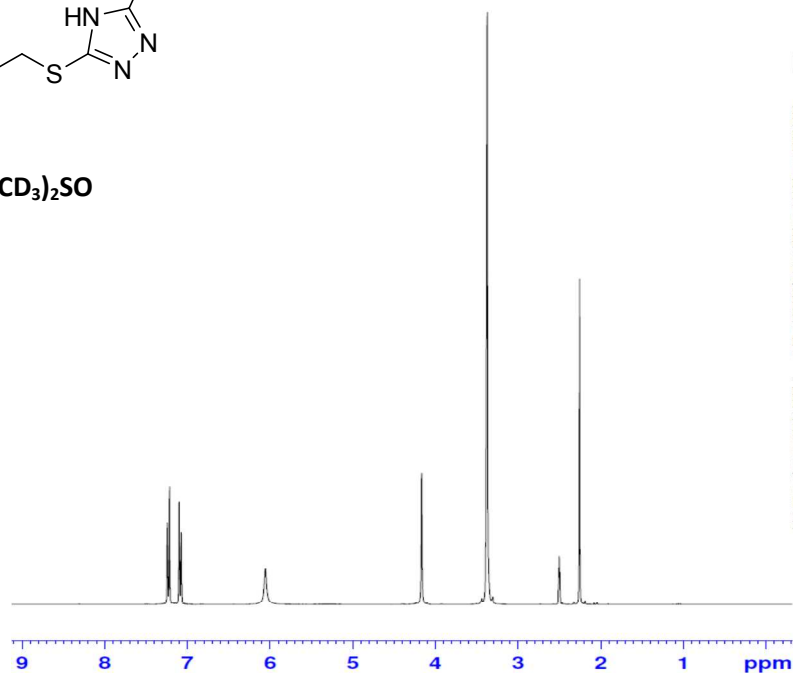
===== CHANNEL f1 =====
NUC1 13C
P1 9.80 usec
PL1 -0.20 dB
PL1W 32.35229492 W
SFO1 75.4803248 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.50 dB
PL12 18.40 dB
PL13 21.40 dB
PL2W 8.09117126 W
PL12W 0.16520053 W
PL13W 0.08279639 W
SFO2 300.1512006 MHz
SI 32768
SF 75.4727780 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

5-((4-methylbenzyl)thio)-4H-1,2,4-triazol-3-amine (16)



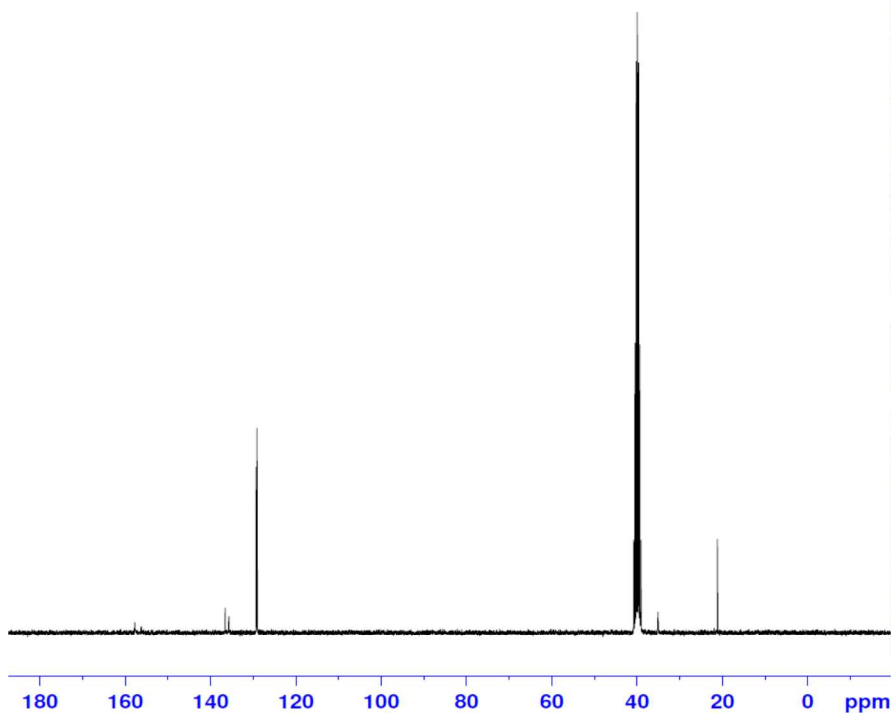
^1H NMR (CD_3) $_2\text{SO}$



```
NAME      BP manuscript
EXPNO     2
PROCNO    3
Date_     20120809
Time      17.02
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD         9806
SOLVENT   DMSO
NS         8
DS         2
SWH        3898.129 Hz
FIDRES     0.397525 Hz
AQ         1.2578329 sec
RG         144
DW         128.267 usec
DE         8.00 usec
TE         296.3 K
D1         3.00000000 sec
TD0        1
```

```
----- CHANNEL f1 -----
NUC1      1H
P1         14.60 usec
PL1        1.10 dB
PL1W       8.87179279 W
SFO1      300.1518535 MHz
SI         32768
SF         300.1500000 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
```

^{13}C NMR (CD_3) $_2\text{SO}$

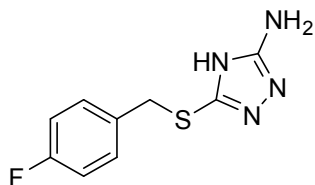


```
NAME      BP manuscript
EXPNO     4
PROCNO    4
Date_     20120809
Time      18.03
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   DMSO
NS         846
DS         4
SWH        18028.846 Hz
FIDRES     0.275098 Hz
AQ         1.8175818 sec
RG         203
DW         27.733 usec
DE         8.50 usec
TE         296.6 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1
```

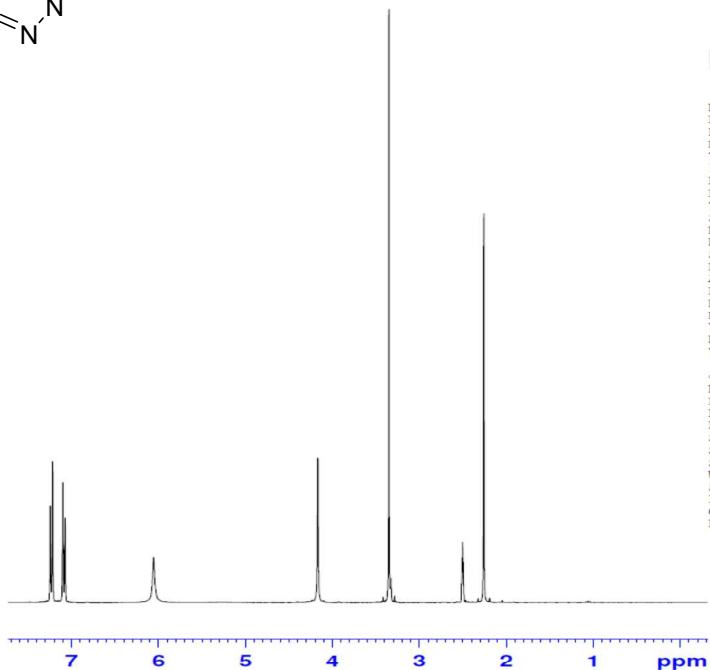
```
----- CHANNEL f1 -----
NUC1      13C
P1         9.80 usec
PL1        -0.20 dB
PL1W       32.35229492 W
SFO1      75.4803248 MHz
```

```
----- CHANNEL f2 -----
CPDPRG2   waltz16
NUC2      1H
PCPD2     100.00 usec
PL2        1.50 dB
PL12       18.40 dB
PL13       21.40 dB
PL2W       8.09117126 W
PL12W      0.16520053 W
PL13W      0.08279639 W
SFO2      300.1512006 MHz
SI         32768
SF         75.4727780 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.00
```

5-((4-fluorobenzyl)thio)-4H-1,2,4-triazol-3-amine (17)



^1H NMR (CD_3) $_2\text{SO}$



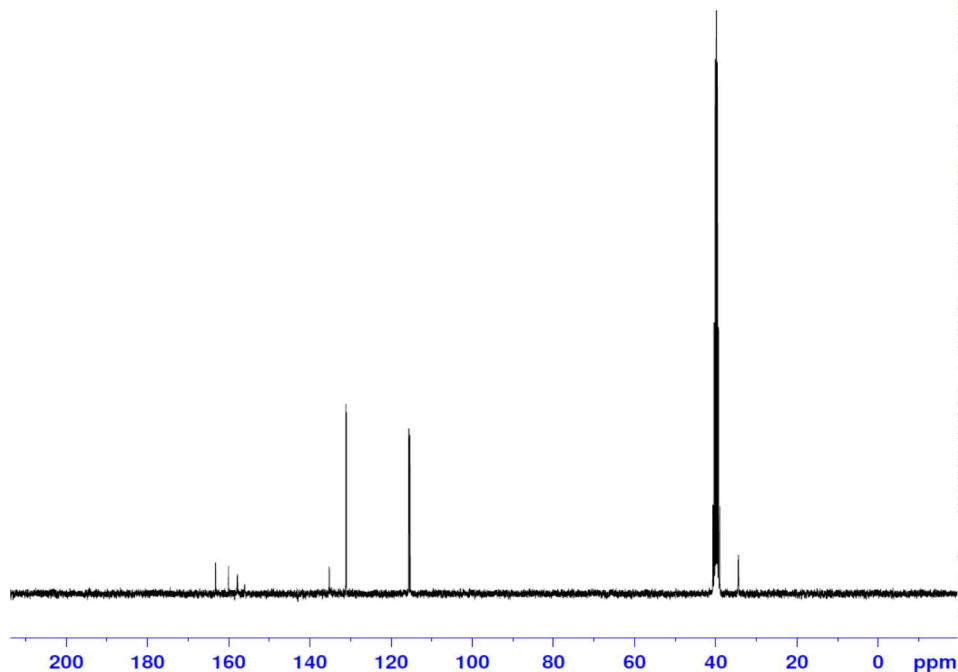
```

NAME      BP manuscript
EXPNO     1
PROCNO    5
Date_     20120802
Time      17.21
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD         9806
SOLVENT   DMSO
NS         2
DS         2
SWH        3898.129 Hz
FIDRES     0.397525 Hz
AQ         1.2578329 sec
RG         181
DW         128.267 usec
DE         8.00 usec
TE         296.9 K
D1         3.0000000 sec
TD0        1
    
```

```

----- CHANNEL f1 -----
NUC1      1H
P1         14.60 usec
PL1        1.10 dB
PL1W       8.87179279 W
SFO1      300.1518535 MHz
SI         32768
SF         300.1500000 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
    
```

^{13}C NMR (CD_3) $_2\text{SO}$



```

NAME      BP manuscript
EXPNO     2
PROCNO    1
Date_     20120809
Time      16.52
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   DMSO
NS         4
DS         4
SWH        18028.846 Hz
FIDRES     0.275098 Hz
AQ         1.8175818 sec
RG         203
DW         27.733 usec
DE         8.50 usec
TE         296.6 K
D1         2.0000000 sec
D11        0.0300000 sec
TD0        1
    
```

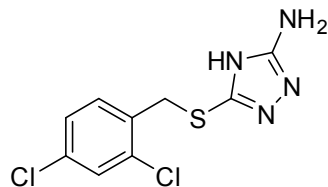
```

----- CHANNEL f1 -----
NUC1      13C
P1         9.80 usec
PL1        -0.20 dB
PL1W       32.35229492 W
SFO1      75.4803248 MHz
    
```

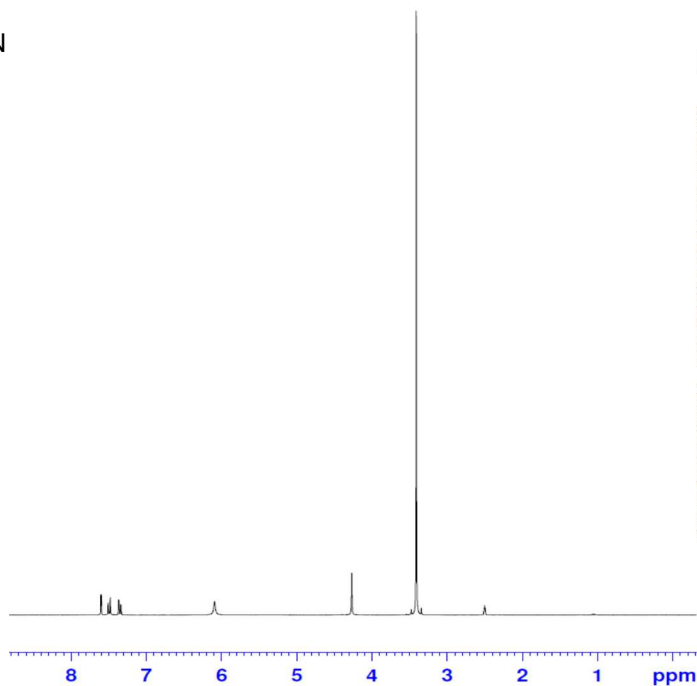
```

----- CHANNEL f2 -----
CPDPRG2   waltz16
NUC2       1H
PCPD2     100.00 usec
PL2        1.50 dB
PL12       18.40 dB
PL13       21.40 dB
PL12W      0.16520053 W
PL13W      0.08279639 W
SFO2      300.1512006 MHz
SI         32768
SF         75.4727780 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.00
    
```

5-((2,4-dichlorobenzyl)thio)-4H-1,2,4-triazol-3-amine (18)



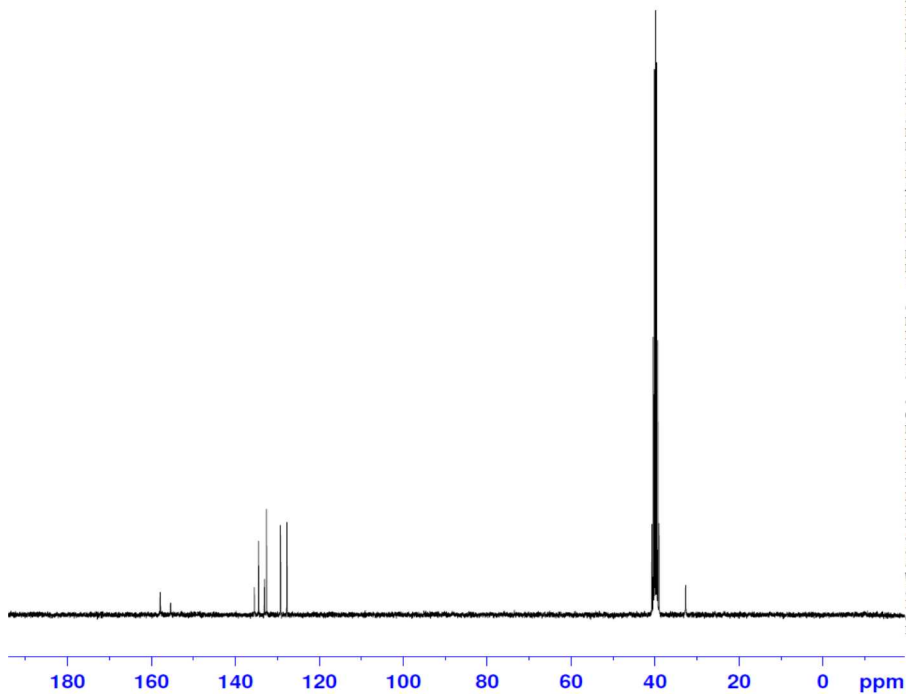
¹H NMR (CD₃)₂SO



```
NAME BP manuscript
EXPNO 1
PROCNO 3
Date_ 20120802
Time 16.23
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 9806
SOLVENT DMSO
NS 8
DS 2
SWH 3898.129 Hz
FIDRES 0.397525 Hz
AQ 1.2578329 sec
RG 114
DW 128.267 usec
DE 8.00 usec
TE 296.7 K
D1 3.0000000 sec
TD0 1
```

```
===== CHANNEL f1 =====
NUC1 1H
P1 14.60 usec
PL1 1.10 dB
PL1W 8.87179279 W
SFO1 300.1518535 MHz
SI 32768
SF 300.1500000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
```

¹³C NMR (CD₃)₂SO

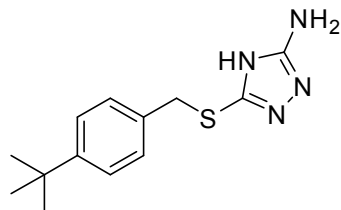


```
NAME BP manuscript
EXPNO 1
PROCNO 4
Date_ 20120802
Time 17.17
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 766
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 203
DW 27.733 usec
DE 8.50 usec
TE 297.1 K
D1 2.0000000 sec
D11 0.03000000 sec
ID0 1
```

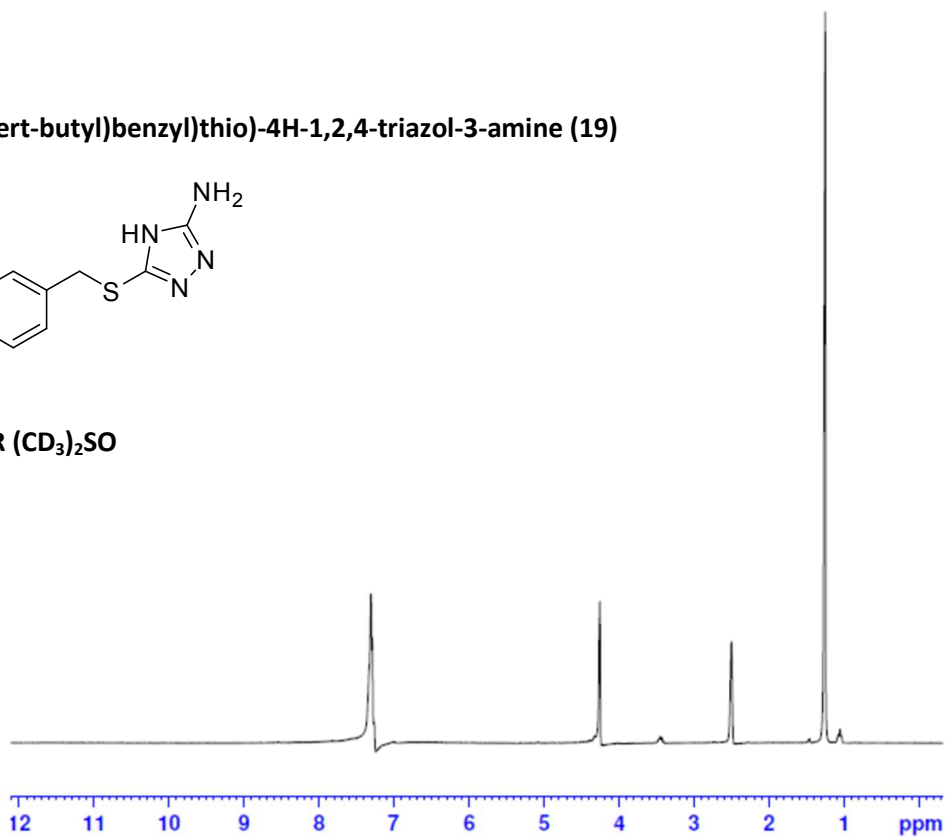
```
===== CHANNEL f1 =====
NUC1 13C
P1 9.80 usec
PL1 -0.20 dB
PL1W 32.35229492 W
SFO1 75.4803248 MHz
```

```
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.50 dB
PL12 18.40 dB
PL13 21.40 dB
PL2W 8.09117126 W
PL12W 0.16520053 W
PL13W 0.08279639 W
SFO2 300.1512006 MHz
SI 32768
SF 75.4727780 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
```

5-((4-(tert-butyl)benzyl)thio)-4H-1,2,4-triazol-3-amine (19)



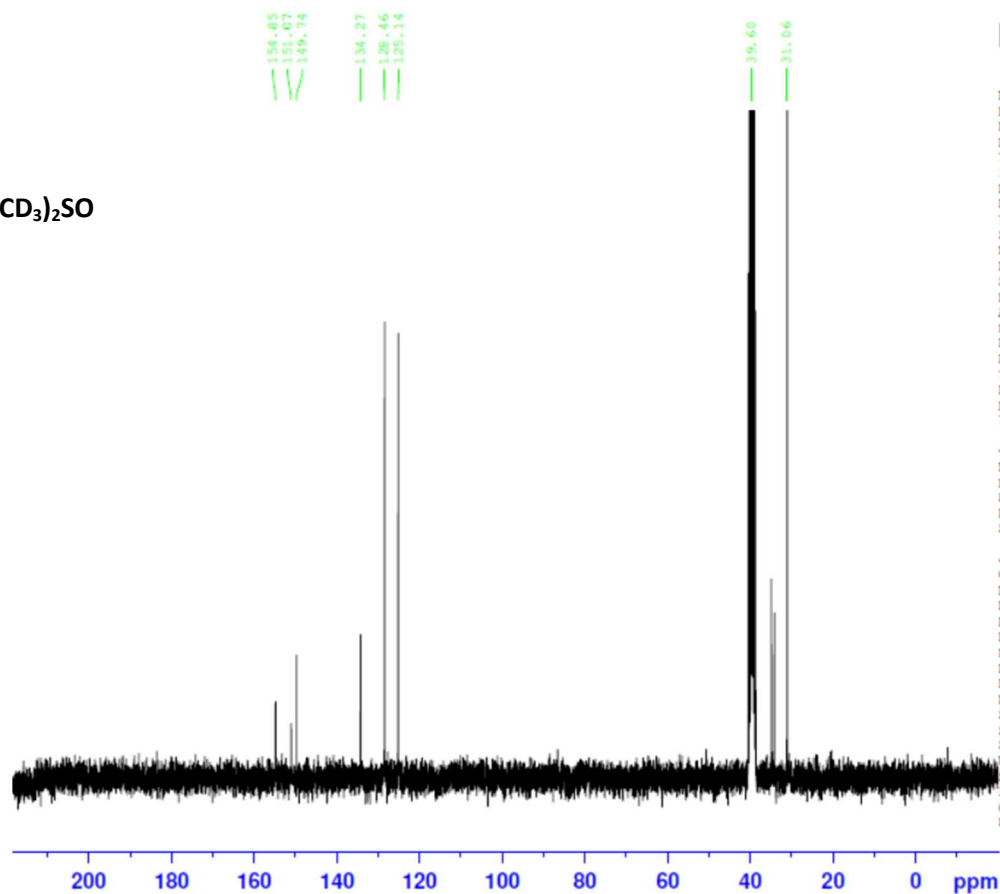
^1H NMR (CD_3) $_2\text{SO}$



```
NAME      Mar07-2013
EXPNO     141
PROCNO    1
Date_     20130308
Time      9.14
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD         9806
SOLVENT   DMSO
NS         8
DS         2
SWH        3898.129 Hz
FIDRES     0.397525 Hz
AQ         1.2578329 sec
RG         203
DW         128.267 usec
DE         8.00 usec
TE         313.0 K
D1         3.0000000 sec
TD0        1
```

```
----- CHANNEL f1 -----
NUC1      1H
P1        14.60 usec
PL1       1.10 dB
PL1W      8.87179279 W
SFO1      300.1518535 MHz
SI        32768
SF        300.1500000 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00
```

^{13}C NMR (CD_3) $_2\text{SO}$

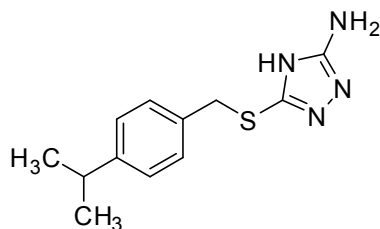


```
NAME      Mar07-2013
EXPNO     140
PROCNO    1
Date_     20130308
Time      8.14
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   DMSO
NS         1024
DS         4
SWH        18028.846 Hz
FIDRES     0.275098 Hz
AQ         1.8175818 sec
RG         203
DW         27.733 usec
DE         8.50 usec
TE         313.0 K
D1         2.0000000 sec
D11        0.0300000 sec
TD0        1
```

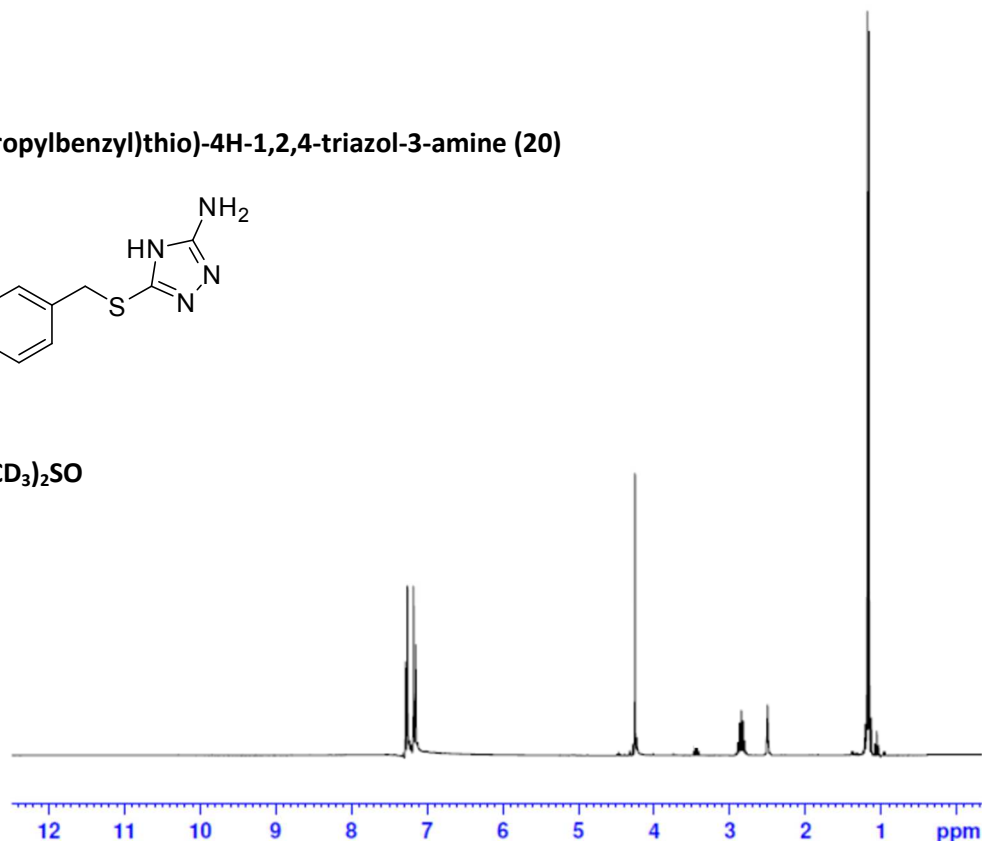
```
----- CHANNEL f1 -----
NUC1      13C
P1        9.80 usec
PL1       -0.20 dB
PL1W      32.35229492 W
SFO1      75.4803248 MHz
```

```
----- CHANNEL f2 -----
CPDPRG2   waltz16
NUC2      1H
PCPD2     100.00 usec
PL2       1.50 dB
PL12      18.40 dB
PL13      21.40 dB
PL2W      8.09117126 W
PL12W     0.16520053 W
PL13W     0.08279639 W
SFO2      300.1512006 MHz
SI        32768
SF        75.4728157 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40
```

5-((4-isopropylbenzyl)thio)-4H-1,2,4-triazol-3-amine (20)



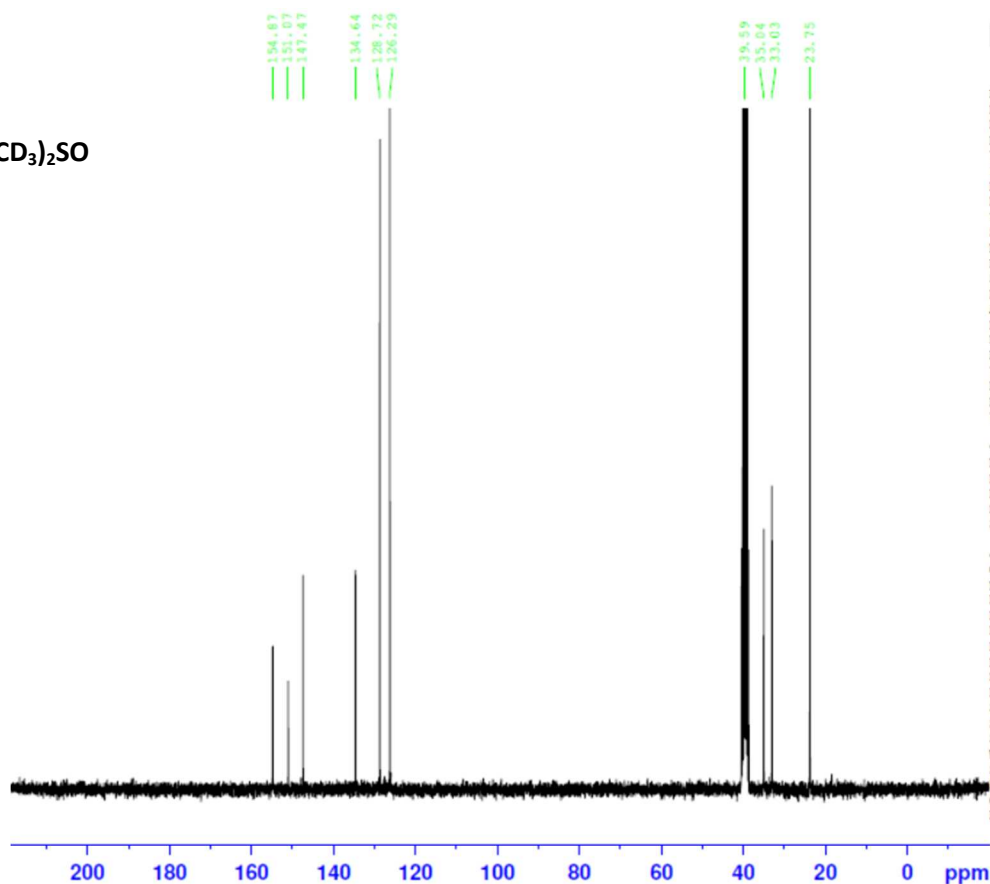
^1H NMR (CD_3SO)



NAME Mar07-2013
EXPNO 161
PROCNO 1
Date_ 20130308
Time 11.37
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 9806
SOLVENT DMSO
NS 8
DS 2
SWH 3898.129 Hz
FIDRES 0.397525 Hz
AQ 1.2578329 sec
RG 128
DW 128.267 usec
DE 8.00 usec
TE 313.0 K
D1 3.00000000 sec
TD0 1

----- CHANNEL f1 -----
NUC1 1H
P1 14.60 usec
PL1 1.10 dB
PL1W 8.87179279 W
SFO1 300.1518535 MHz
SI 32768
SF 300.1500000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

^{13}C NMR (CD_3SO)

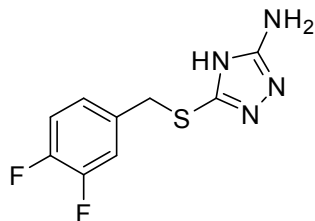


NAME Mar07-2013
EXPNO 160
PROCNO 1
Date_ 20130308
Time 11.35
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 1024
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 203
DW 27.733 usec
DE 8.50 usec
TE 312.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

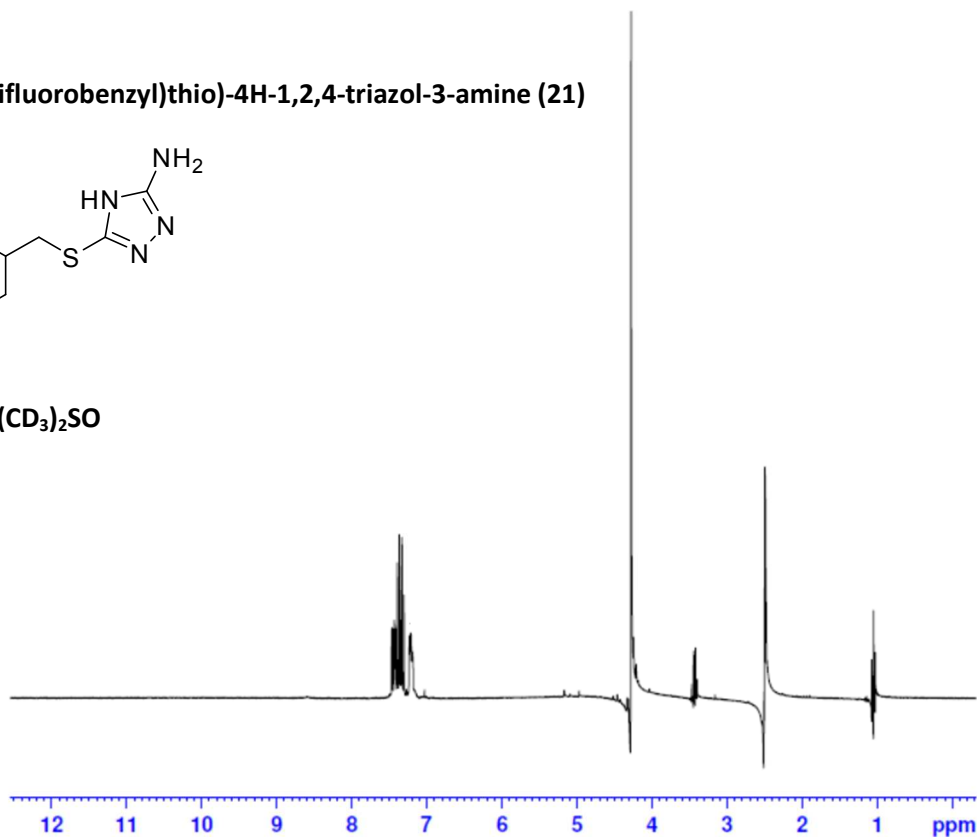
----- CHANNEL f1 -----
NUC1 13C
P1 9.80 usec
PL1 -0.20 dB
PL1W 32.35229492 W
SFO1 75.4803248 MHz

----- CHANNEL f2 -----
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.50 dB
PL12 18.40 dB
PL13 21.40 dB
PL2W 8.09117126 W
PL12W 0.16520053 W
PL13W 0.08279639 W
SFO2 300.1512006 MHz
SI 32768
SF 75.4728157 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

5-((3,4-difluorobenzyl)thio)-4H-1,2,4-triazol-3-amine (21)



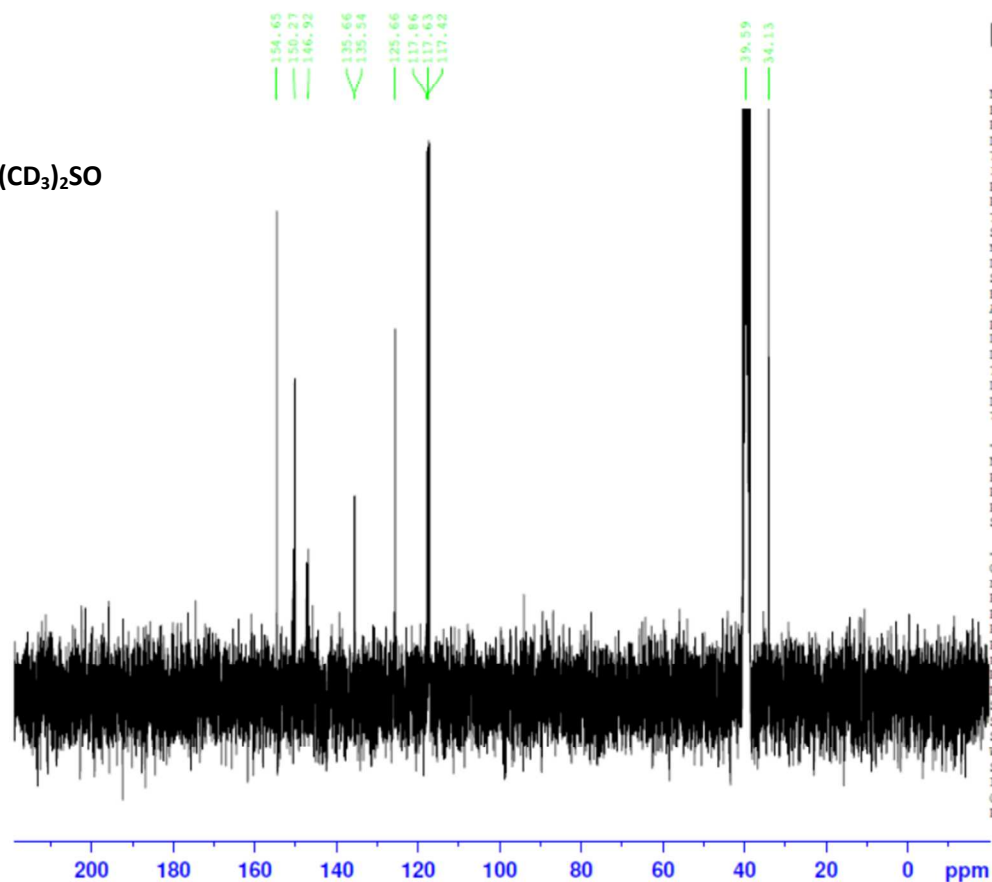
^1H NMR (CD_3) $_2\text{SO}$



NAME Mar07-2013
EXPNO 151
PROCNO 1
Date_ 20130308
Time 10.25
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 9806
SOLVENT DMSO
NS 8
DS 2
SWH 3898.129 Hz
FIDRES 0.397525 Hz
AQ 1.2578329 sec
RG 203
DW 128.267 usec
DE 8.00 usec
TE 312.9 K
D1 3.0000000 sec
TD0 1

----- CHANNEL f1 -----
NUC1 ^1H
P1 14.60 usec
PL1 1.10 dB
PL1W 8.87179279 W
SFO1 300.1518535 MHz
SI 32768
SF 300.1500000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

^{13}C NMR (CD_3) $_2\text{SO}$

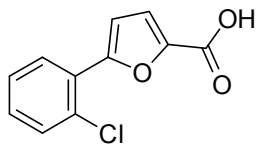


NAME Mar07-2013
EXPNO 150
PROCNO 1
Date_ 20130308
Time 10.23
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 1024
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 203
DW 27.733 usec
DE 8.50 usec
TE 313.0 K
D1 2.0000000 sec
D11 0.03000000 sec
TD0 1

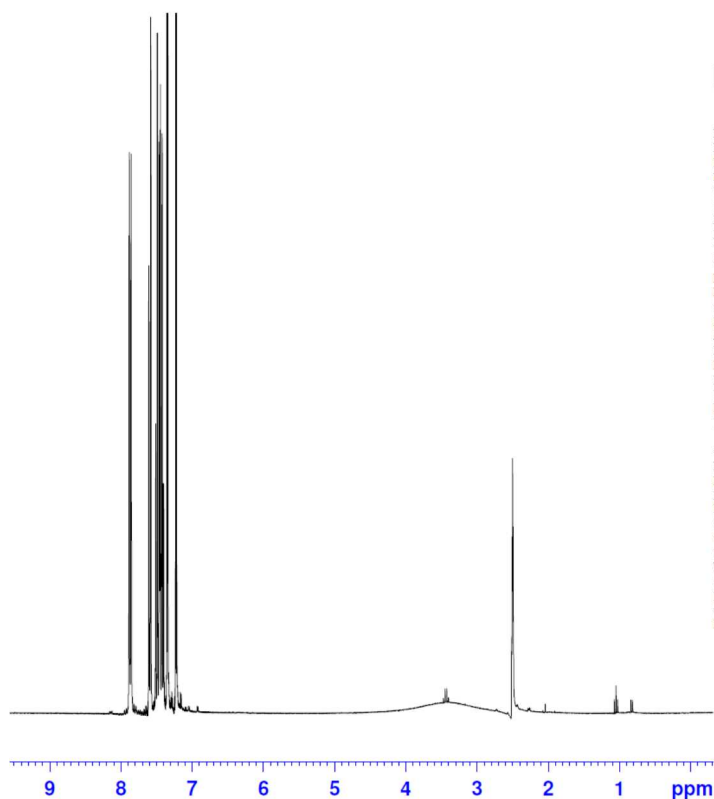
----- CHANNEL f1 -----
NUC1 ^{13}C
P1 9.80 usec
PL1 -0.20 dB
PL1W 32.35229492 W
SFO1 75.4803248 MHz

----- CHANNEL f2 -----
CPDPRG2 waltz16
NUC2 ^1H
PCPD2 100.00 usec
PL2 1.50 dB
PL12 18.40 dB
PL13 21.40 dB
PL2W 8.09117126 W
PL12W 0.16520053 W
PL13W 0.08279639 W
SFO2 300.1512006 MHz
SI 32768
SF 75.4728157 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

5-(2-chlorophenyl)furan-2-carboxylic acid (22)



^1H NMR $(\text{CD}_3)_2\text{SO}$

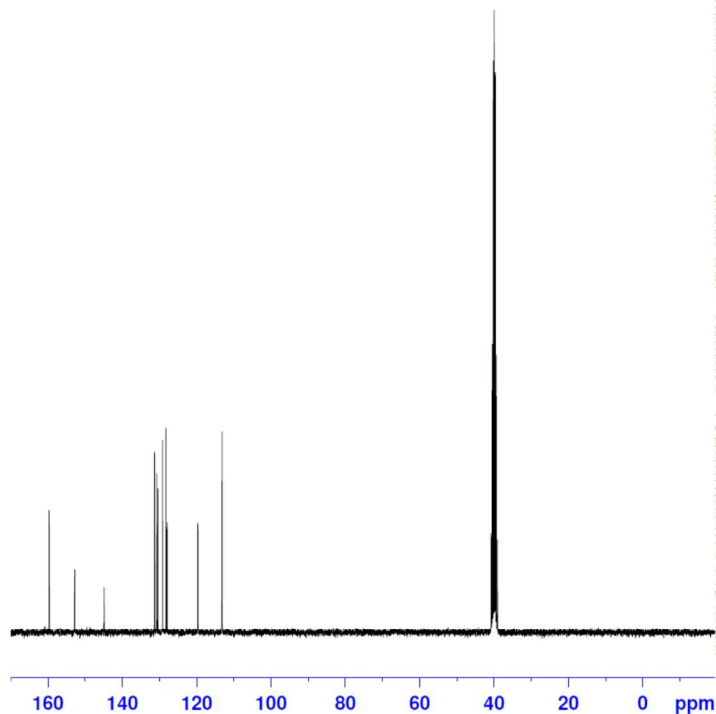


```

NAME      BP manuscript
EXPNO     1
PROCNO    13
Date_     20120809
Time      15.35
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD        9806
SOLVENT   DMSO
NS         8
DS         2
SWH        3898.129 Hz
FIDRES     0.397525 Hz
AQ         1.2578329 sec
RG         144
DW         128.267 usec
DE         8.00 usec
TE         296.2 K
D1         3.0000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       1H
P1         14.60 usec
PL1        1.10 dB
PL1W       8.87179279 W
SFO1       300.1518535 MHz
SI         32768
SF         300.1500000 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
    
```

^{13}C NMR $(\text{CD}_3)_2\text{SO}$



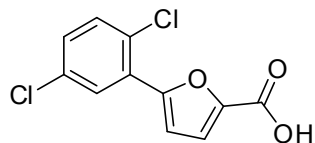
```

NAME      BP manuscript
EXPNO     14
PROCNO    14
Date_     20120809
Time      16.10
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD        65536
SOLVENT   DMSO
NS         4
DS         4
SWH        18028.846 Hz
FIDRES     0.275098 Hz
AQ         1.8175818 sec
RG         203
DW         27.733 usec
DE         8.50 usec
TE         296.6 K
D1         2.0000000 sec
D11        0.0300000 sec
TD0        1

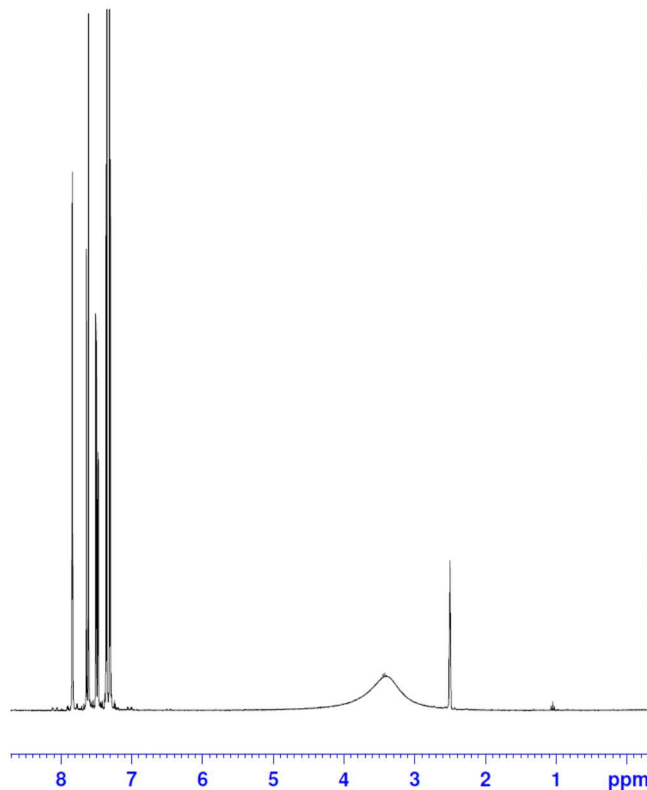
===== CHANNEL f1 =====
NUC1       13C
P1         9.80 usec
PL1        -0.20 dB
PL1W       32.35229492 W
SFO1       75.4803248 MHz

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2       1H
PCPD2     100.00 usec
PL2        1.50 dB
PL12       18.40 dB
PL13       21.40 dB
PL2W       8.09117126 W
PL12W      0.16520053 W
PL13W      0.08279639 W
SFO2       300.1512006 MHz
SI         32768
SF         75.4727780 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.00
    
```

5-(2,5-dichlorophenyl)furan-2-carboxylic acid (23)



¹H NMR (CD₃)₂SO



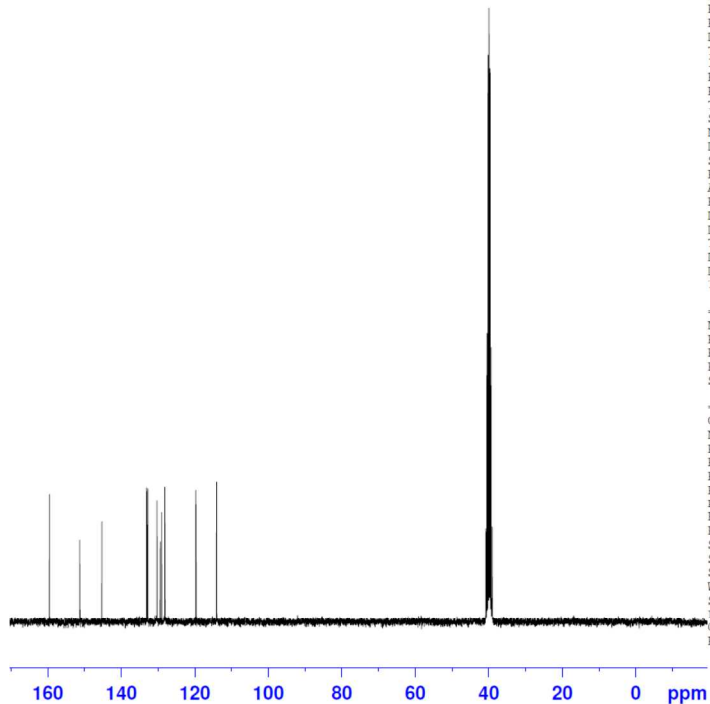
```

NAME      BP manuscript
EXPNO     1
PROCNO    11
Date_     20120809
Time      15.02
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD        9806
SOLVENT   DMSO
NS         8
DS         2
SWH        3898.129 Hz
FIDRES     0.397525 Hz
AQ         1.2578329 sec
RG         161
DW         128.267 usec
DE         8.00 usec
TE         296.2 K
D1         3.00000000 sec
TD0        1
  
```

```

===== CHANNEL f1 =====
NUC1      1H
P1        14.60 usec
PL1       1.10 dB
PL1W      8.87179279 W
SFO1      300.1518535 MHz
SI        32768
SF        300.1500000 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00
  
```

¹³C NMR (CD₃)₂SO



```

NAME      BP manuscript
EXPNO     1
PROCNO    12
Date_     20120809
Time      15.31
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD        65536
SOLVENT   DMSO
NS         399
DS         4
SWH        18028.846 Hz
FIDRES     0.275098 Hz
AQ         1.8175818 sec
RG         203
DW         27.733 usec
DE         8.50 usec
TE         296.5 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1
  
```

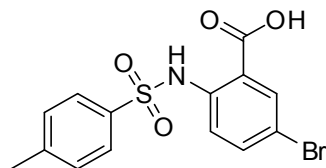
```

===== CHANNEL f1 =====
NUC1      13C
P1        9.80 usec
PL1       -0.20 dB
PL1W      32.35229492 W
SFO1      75.4803248 MHz
  
```

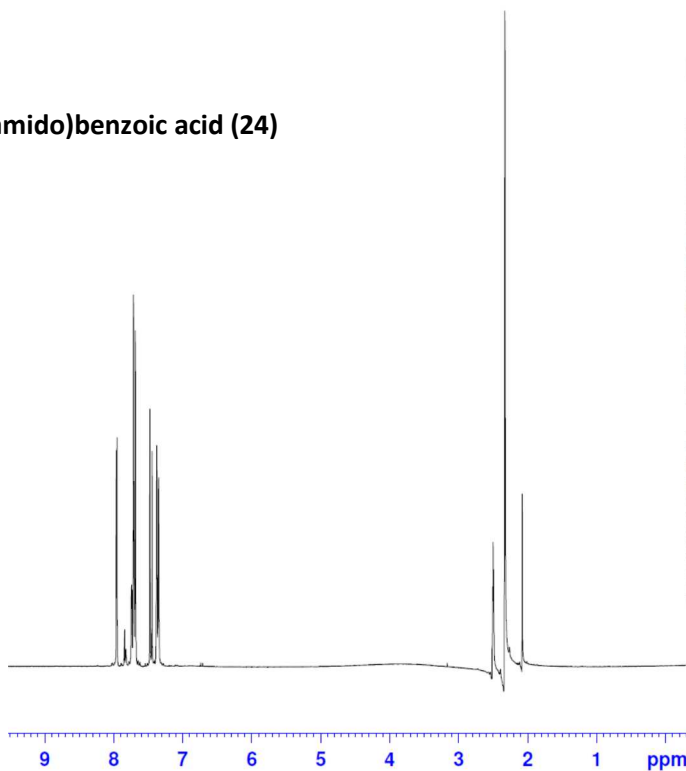
```

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2      1H
PCPD2     100.00 usec
PL2       1.50 dB
PL12      18.40 dB
PL13      21.40 dB
PL2W      8.09117126 W
PL12W     0.16520053 W
PL13W     0.08279639 W
SFO2      300.1512006 MHz
SI        32768
SF        75.4727780 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.00
  
```

5-bromo-2-(4-methylphenylsulfonamido)benzoic acid (24)



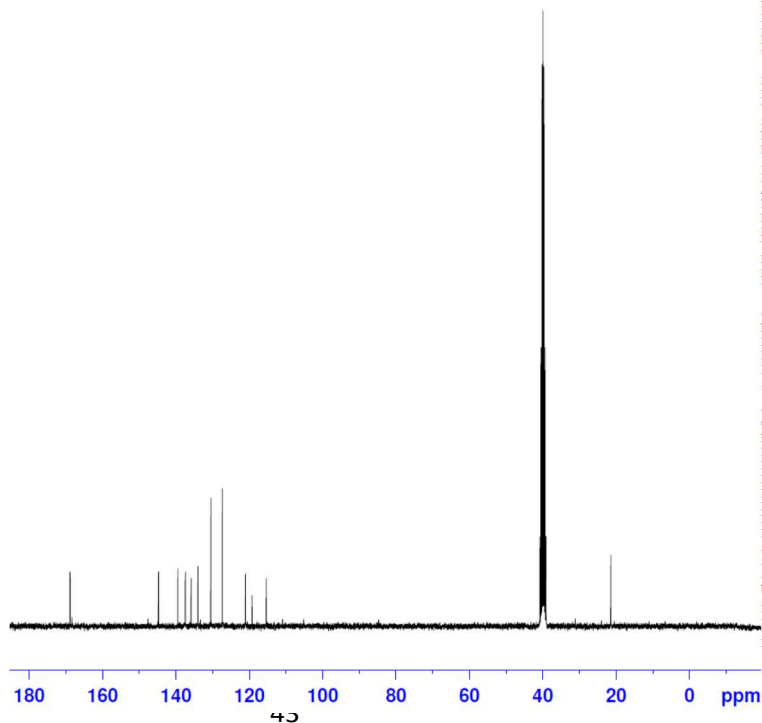
^1H NMR (CD_3) $_2\text{SO}$



```
NAME BP manuscript
EXPNO 1
PROCNO 7
Date_ 20120809
Time 13.40
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 9806
SOLVENT DMSO
NS 8
DS 2
SWH 3898.129 Hz
FIDRES 0.397525 Hz
AQ 1.2578329 sec
RG 161
DW 128.267 usec
DE 8.00 usec
TE 296.1 K
D1 3.0000000 sec
TD0 1
```

```
===== CHANNEL f1 =====
NUC1 1H
P1 14.60 usec
PL1 1.10 dB
PL1W 8.87179279 W
SFO1 300.1518535 MHz
SI 32768
SF 300.1500000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
```

^{13}C NMR (CD_3) $_2\text{SO}$

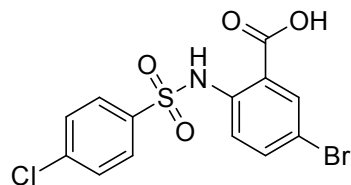


```
NAME BP manuscript
EXPNO 1
PROCNO 8
Date_ 20120809
Time 14.15
INSTRUM spect
PROBHD 5 mm PABDO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 485
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 203
DW 27.733 usec
DE 8.50 usec
TE 296.4 K
D1 2.0000000 sec
D11 0.0300000 sec
TD0 1
```

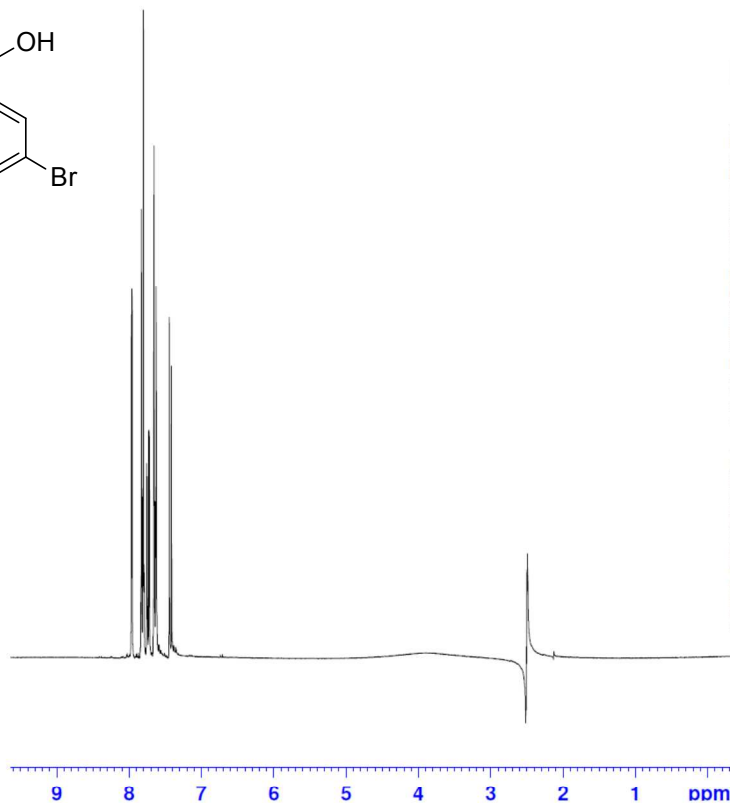
```
===== CHANNEL f1 =====
NUC1 13C
P1 9.80 usec
PL1 -0.20 dB
PL1W 32.35229492 W
SFO1 75.4803248 MHz
```

```
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.50 dB
PL12 18.40 dB
PL13 21.40 dB
PL2W 8.09117126 W
PL12W 0.16520053 W
PL13W 0.08279639 W
SFO2 300.1512006 MHz
SI 32768
SF 75.4727780 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
```

5-bromo-2-(4-chlorophenylsulfonamido)benzoic acid (25)



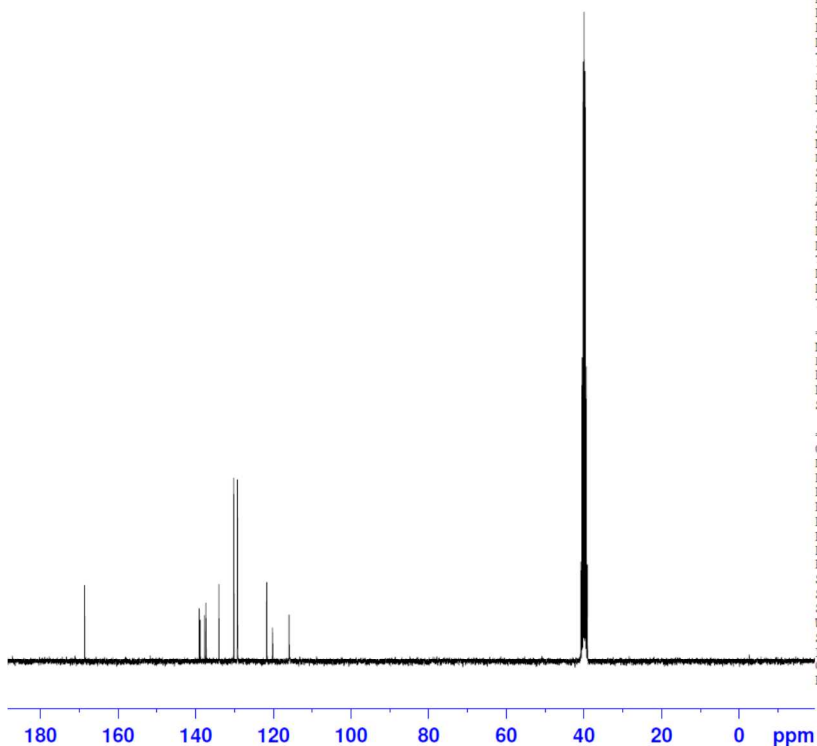
^1H NMR (CD_3) $_2\text{SO}$



```
NAME      BP manuscript
EXPNO     1
PROCNO    9
Date_     20120809
Time      14.20
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD         9806
SOLVENT   DMSO
NS         8
US         2
SWH        3898.129 Hz
FIDRES     0.397525 Hz
AQ         1.2578329 sec
RG         161
DW         128.267 usec
DE         8.00 usec
TE         296.2 K
D1         3.0000000 sec
TD0        1
```

```
===== CHANNEL f1 =====
NUC1      1H
P1         14.60 usec
PL1        1.10 dB
PL1W       8.87179279 W
SFO1      300.1518535 MHz
SI         32768
SF         300.1500000 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
```

^{13}C NMR
(CD_3) $_2\text{SO}$



```
NAME      BP manuscript
EXPNO     1
PROCNO    10
Date_     20120809
Time      14.57
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   DMSO
NS         505
DS         4
SWH        18028.846 Hz
FIDRES     0.275098 Hz
AQ         1.8175818 sec
RG         203
DW         27.733 usec
DE         8.50 usec
TE         296.5 K
D1         2.0000000 sec
D11        0.03000000 sec
TD0        1
```

```
===== CHANNEL f1 =====
NUC1      13C
P1         9.80 usec
PL1        -0.20 dB
PL1W       32.35229492 W
SFO1      75.4803248 MHz
```

```
===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2      1H
PCPD2     100.00 usec
PL2        1.50 dB
PL12       18.40 dB
PL13       21.40 dB
PL2W       8.09117126 W
PL12W      0.16520053 W
PL13W      0.08279639 W
SFO2      300.1512006 MHz
SI         32768
SF         75.4727780 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.00
```