SUPPORTING INFORMATION

Acetyltrimethylsilane: A Novel Reagent for the Transformation of 2*H*-Pyran-2-ones to Unsymmetrical Biaryls

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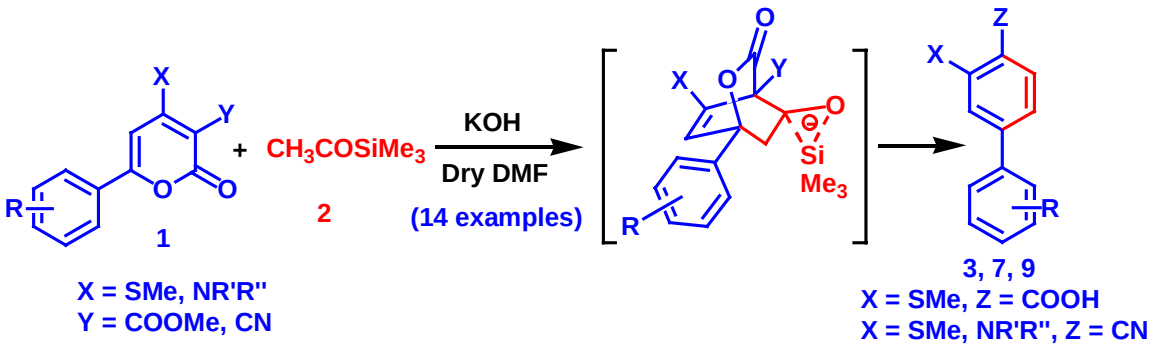

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Experimental Section

General procedure for the synthesis of 1a-f: A mixture of methyl 2-methoxycarbonyl-3,3-di(methylsulfanyl)acrylate (prepared from dimethylmalonate, CS_2 and MeI) **1** (10 mmol), acetophenone (11 mmol) and powdered KOH (12 mmol) in dry DMSO (50 mL) was stirred at room temperature for 10h. After completion, the reaction mixture was poured into ice water with constant stirring. The precipitate thus obtained was filtered and purified on a silica gel column using chloroform as eluent. The spectroscopic analysis data for all the synthesized 2*H*-pyran-2-ones were compared with reported data.

General procedure for the synthesis of 3a-f: A mixture of 6-aryl-3-methoxycarbonyl-4-methylsulfanyl-2*H*-pyran-2-ones **1** (1 mmol), acetyltrimethylsilane (1.1 mmol) and powdered KOH (1.2 mmol) in dry DMF (5 mL) was stirred at room temperature for 4-6h. At the end reaction mixture was poured into ice water with vigorous stirring and finally neutralized with dilute HCl. The solid thus obtained was filtered and purified on a silica gel column using chloroform-hexane (3:1) as eluent.

3-Methylsulfanyl-biphenyl-4-carboxylic acid (3a)
White solid; mp 219-220 ^{0}C; ^{1}H NMR (200 MHz, DMSO-d_6) δ 2.49 (s, 3H, SCH_3), 7.36-7.54 (m, 5H, ArH), 7.72-7.78 (m, 2H, ArH), 7.98 (d, 1H, *J*=5.2 Hz, ArH), 13.01 (brs, 1H, COOH); IR (KBr) 1680 cm^{-1} (CO); MS (FAB) 244 (M^+).

4'-Methyl-3-methylsulfanyl-biphenyl-4-carboxylic acid (3b)
White solid; mp 205-206 ^{0}C; ^{1}H NMR (200 MHz, DMSO-d_6) δ 2.38 (s, 3H, CH_3), 2.51 (s, 3H, SCH_3), 7.34 (d, 2H, *J*=8.0 Hz, ArH), 7.42-7.52 (m, 2H, ArH), 7.68 (d, 2H, *J*=8.0 Hz, ArH), 7.99 (d, 1H, *J*=8.2 Hz, ArH); IR (KBr)1682 cm^{-1} (CO); MS (FAB) 258 (M^+).

4'-Methoxy-3-methylsulfanyl-biphenyl-4-carboxylic acid (3c)
White solid; mp 222-223 ^{0}C; ^{1}H NMR (200 MHz, DMSO-d_6) δ 2.27 (s, 3H, SCH_3), 3.59 (s, 3H, OCH_3), 6.84 (d, 2H, *J*=8.6 Hz, ArH), 7.18-7.24 (m, 2H, ArH), 7.49 (d, 2H, *J*=8.6 Hz, ArH), 7.74 (d, 1H, *J*=8.2 Hz, ArH); IR (KBr) 1678 cm^{-1} (CO); MS (FAB) 274 (M^+).

4-Benzo[1,3]dioxol-5-yl-2-methylsulfanyl-benzoic acid (3d)

White solid; mp 218-219 ^{0}C; ^{1}H NMR (200 MHz, DMSO-d_6) δ 2.31 (s, 3H, SCH_3), 5.90 (s, 2H, CH_2), 6.85 (d, 1H, *J*=8.0 Hz, ArH), 7.05-7.09 (m, 1H, ArH), 7.18-7.24 (m, 3H, ArH), 7.76 (d, 1H, *J*=8.6 Hz, ArH); IR (KBr) 1679 cm^{-1} (CO); MS (FAB) 288 (M^+).

4'-Bromo-3-methylsulfanyl-biphenyl-4-carboxylic acid (3e)
White solid; mp 214-216 ^{0}C; ^{1}H NMR (200 MHz, DMSO-d_6) δ 2.53 (s, 3H, SCH_3), 7.45-7.53 (m, 2H, ArH), 7.66-7.80 (m, 4H, ArH), 8.01 (d, 1H, *J*=8.2 Hz, ArH); IR (KBr) 1681 cm^{-1} (CO); MS (FAB) 324 (M^++2), 322 (M^+).

4'-Chloro-3-methylsulfanyl-biphenyl-4-carboxylic acid (3f)
White solid; mp 218-219 ^{0}C; ^{1}H NMR (200 MHz, DMSO-d_6) δ 2.31 (s, 3H, SCH_3), 7.26-7.32 (m, 2H, ArH), 7.37 (d, 2H, *J*=8.4 Hz, ArH), 7.62 (d, 2H, *J*=8.4 Hz, ArH), 7.81 (d, 1H, *J*=8.6 Hz, ArH); IR (KBr) 1663 cm^{-1} (CO); MS (FAB) 278 (M^+).

4-Furan-2-yl-2-methylsulfanyl-benzoic acid (5a)
White solid; mp 192-194 ^{0}C; ^{1}H NMR (200 MHz, DMSO-d_6) δ 2.57 (s, 3H, SCH_3), 6.71-6.75 (m, 1H, CH), 7.26 (d, 1H, *J*=3.2 Hz, CH), 7.55-7.64 (m, 2H, ArH), 7.91 (s, 1H, CH), 8.02 (d, 1H, *J*=8.0 Hz, ArH); IR (KBr) 1679 cm^{-1} (CO); MS (FAB) 234 (M^+).

2-Methylsulfanyl-4-thiophen-2-yl-benzoic acid (5b)
White solid; mp 194-196 ^{0}C; ^{1}H NMR (200 MHz, DMSO-d_6) δ 2.56 (s, 3H, SCH_3), 7.26 (dd, 1H, CH), 7.48-7.58 (m, 2H, ArH), 7.70-7.80 (m, 2H, CH), 8.00 (d, 1H, *J*=8.0 Hz, ArH); IR (KBr) 1677 cm^{-1} (CO); MS (FAB) 250 (M^+).

4-Methylsulfanyl-2-oxo-6-*p*-tolyl-2H-pyran-3-carbonitrile (6b)
Yellow solid; mp 215-216 ^{0}C; ^{1}H NMR (200 MHz, DMSO-d_6) δ 2.43 (s, 3H, CH_3), 2.85 (s, 3H, SCH_3), 7.21 (s, 1H, CH), 7.42 (d, 2H, *J*=8.0 Hz, ArH), 7.96 (d, 2H, *J*=8.0 Hz, ArH); IR (KBr) 1716 (CO), 2195 cm^{-1} (CN); MS (FAB) 258 (M^++1).

General procedure for the synthesis of 7a-c: A mixture of 6-aryl-4-methylsulfanyl-2-oxo-2*H*-pyran-3-carbonitrile **6a-c** (1 mmol), acetyltrimethylsilane (1 mmol) and powdered KOH (1.2 mmol) in dry DMF (5 mL) was stirred at room temperature for 6-8h in dark under highly inert atmosphere. At the end reaction mixture was poured into ice water with vigorous stirring and finally neutralized with dilute HCl. The solid thus obtained was filtered and purified on a silica gel column using chloroform-hexane (1:9) as eluent.

3-Methylsulfanyl-biphenyl-4-carbonitrile (7a)
Light yellow solid; mp 84-86 ^{0}C; ^{1}H NMR (200 MHz, $CDCl_3$) δ 2.62 (s, 3H, SCH_3), 7.36-7.70 (m, 8H, ArH); IR (KBr) 2221 cm^{-1} (CN); MS (FAB) 226 (M^++1).

4'-Methyl-3-methylsulfanyl-biphenyl-4-carbonitrile (7b)

Light yellow solid; mp 100-102 ^{0}C; ^{1}H NMR (200 MHz, $CDCl_3$) δ 2.42 (s, 3H, CH_3), 2.61 (s, 3H, SCH_3), 7.28 (d, 2H, *J*=8.0 Hz, ArH), 7.39 (d, 1H, *J*=8.1 Hz, ArH), 7.45-7.50 (m, 2H, ArH), 7.64 (d, 2H, *J*=8.0 Hz, ArH); IR (KBr) 2219 cm^{-1} (CN); MS (FAB) 240 (M^++1).

4'-Bromo-3-methylsulfanyl-biphenyl-4-carbonitrile (7c)
Light yellow solid; mp 132-134 ^{0}C; ^{1}H NMR (200 MHz, $CDCl_3$) δ 2.62 (s, 3H, SCH_3), 7.32-7.48 (m, 4H, ArH), 7.58-7.70 (m, 3H, ArH); IR (KBr) 2216 cm^{-1} (CN); MS (FAB) 306, 304 (M^++1).

2-Oxo-6-phenyl-4-piperidin-1-yl-2H-pyran-3-carbonitrile (8a)
White solid; mp 218-219 ^{0}C (Lit.[21]: 211 ^{0}C); ^{1}H NMR (200 MHz, DMSO-d_6) δ 1.56-1.58 (m, 6H, 3CH_2), 3.74-3.76 (m, 4H, 2CH_2), 6.92 (s, 1H, CH), 7.40-7.44 (m, 3H, ArH), 7.81-7.86 (m, 2H, ArH); IR (KBr) 1692 (CO), 2196 cm^{-1} (CN); MS (FAB) 281 (M^++1).

2-Oxo-4-piperidin-1-yl-6-p-tolyl-2H-pyran-3-carbonitrile (8b)
White solid; mp 198-199 ^{0}C; ^{1}H NMR (200 MHz, DMSO-d_6) δ 1.70-1.72 (m, 6H, 3CH_2), 2.53 (s, 3H, CH_3), 3.88-3.90 (m, 4H, 2CH_2), 7.02 (s, 1H, CH), 7.37 (d, 2H, *J*=8.0 Hz, ArH), 7.89 (d, 2H, *J*=8.0 Hz, ArH); IR (KBr) 1693 (CO), 2195 cm^{-1} (CN); MS (FAB) 295 (M^++1).

6-(4-Bromophenyl)-2-oxo-4-piperidin-1-yl-2H-pyran-3-carbonitrile (8c)
White solid; mp 247-248 ^{0}C (Lit.[21]: 248 ^{0}C); ^{1}H NMR (200 MHz, DMSO-d_6) δ 1.63-1.65 (m, 6H, 3CH_2), 3.80-3.83 (m, 4H, 2CH_2), 7.04 (s, 1H, CH), 7.69 (d, 2H, *J*=8.6 Hz, ArH), 7.87 (d, 2H, *J*=8.6 Hz, ArH); IR (KBr) 1691 (CO), 2196 cm^{-1} (CN); MS (FAB) 361, 359 (M^++1).

6-(4-Methoxyphenyl)-2-oxo-4-piperidin-1-yl-2H-pyran-3-carbonitrile (8d)
White solid; mp 189-190 ^{0}C; ^{1}H NMR (200 MHz, DMSO-d_6) δ 1.69-1.71 (m, 6H, 3CH_2), 3.84-3.87 (m, 7H, OCH_3&2CH_2), 6.93 (s, 1H, CH), 7.08 (d, 2H, *J*=8.9 Hz, ArH), 7.93 (d, 2H, *J*=8.9 Hz, ArH); IR (KBr) 1693 (CO), 2199 cm^{-1} (CN); MS (FAB) 311 (M^++1).

6-Benzo[1,3]dioxol-5-yl-2-oxo-4-piperidin-1-yl-2H-pyran-3-carbonitrile (8e)
White solid; mp 217-218 ^{0}C; ^{1}H NMR (200 MHz, DMSO-d_6) δ 1.54-1.58 (m, 6H, 3CH_2), 3.72-3.75 (m, 4H, 2CH_2), 6.02 (s, 2H, CH_2), 6.79 (s, 1H, CH), 6.94 (d, 1H, *J*=8.2 Hz, ArH), 7.38 (d, 1H, *J*=8.2 Hz, ArH), 7.48 (s, 1H, ArH); IR (KBr) 1693 (CO), 2201 cm^{-1} (CN); MS (FAB) 325 (M^++1).

3-Piperidin-1-yl-biphenyl-4-carbonitrile (9a)
Viscous Oil; ^{1}H NMR (200 MHz, $CDCl_3$) δ 1.60-1.68 (m, 2H, CH_2), 1.78-1.87 (m, 4H, 2CH_2), 3.18-3.26 (m, 4H, 2CH_2), 7.14-7.18 (m, 2H, ArH), 7.31-7.64 (m, 6H, ArH); IR (neat) 2220 cm^{-1} (CN); MS (FAB) 263 (M^++1).

4'-Methyl-3-piperidin-1-yl-biphenyl-4-carbonitrile (9b)
Viscous Oil; ^{1}H NMR (200 MHz, $CDCl_3$) δ 1.56-1.68 (m, 2H, CH_2), 1.72-1.86 (m, 4H, 2CH_2), 2.40 (s, 3H, CH_3), 3.19-3.24 (m, 4H, 2CH_2), 7.10-7.16 (m, 2H, ArH), 7.26 (d, 2H, *J*=8.0 Hz, ArH), 7.46 (d, 2H, *J*=8.0 Hz, ArH), 7.57 (d, 1H, *J*=8.4 Hz, ArH); IR (neat) 2217 cm^{-1} (CN); MS (FAB) 277 (M^++1).

4'-Bromo-3-piperidin-1-yl-biphenyl-4-carbonitrile (9c)
White solid; mp 120-122^{o}C; ^{1}H NMR (200 MHz, $CDCl_3$) δ 1.60-1.65 (m, 2H, CH_2), 1.76-1.82 (m, 4H, 2CH_2), 3.20-3.26 (m, 4H, 2CH_2), 7.06-7.18 (m, 2H, ArH), 7.42 (d, 2H, *J*=8.4 Hz, ArH), 7.56-7.61 (m, 3H, ArH); IR (KBr) 2219 cm^{-1} (CN); MS (FAB) 343, 341 (M^++1).

4'-Methoxy-3-piperidin-1-yl-biphenyl-4-carbonitrile (9d)

Viscous Oil; ^{1}H NMR (200 MHz, $CDCl_3$) δ 1.52-1.61 (m, 2H, CH_2), 1.68-1.78 (m, 4H, $2CH_2$), 3.10-3.18 (m, 4H, $2CH_2$), 3.77 (s, 3H, OCH_3), 6.90 (d, 2H, *J*=8.8 Hz, ArH), 7.00-7.06 (m, 2H, ArH), 7.40-7.50 (m, 3H, ArH); IR (neat) 2217 cm^{-1} (CN); MS (FAB) 293 (M^++1).

4-Benzo[1,3]dioxol-5-yl-2-piperidin-1-yl-benzonitrile (9e)
Viscous Oil; ^{1}H NMR (200 MHz, $CDCl_3$) δ 1.61-1.68 (m, 2H, CH_2), 1.78-1.90 (m, 4H, $2CH_2$), 3.20-3.28 (m, 4H, $2CH_2$), 6.01 (s, 2H, CH_2), 6.85 (d, 1H, *J*=8.0 Hz, ArH), 7.00-7.12 (m, 3H, ArH), 7.18 (s, 1H, ArH), 7.55 (d, 1H, *J*=8.0 Hz, ArH); IR (neat) 2216 cm^{-1} (CN); MS (FAB) 307 (M^++1).

Crystal structure of 3a (ORTEP view): Crystals suitable for X-ray structure were obtained by slow evaporation of chloroform-methanol solution of **3a** at room temperature. The crystal data of **3a**: $C_{14}H_{12}O_2S$, *M* = 244.30, triclinic, *P* -1, *a* = 7.215(1) Å , *b* = 11.234(1) Å, *c* = 14. 4730(1) Å, α = 84.09(1) *β* = 88.61(1)°, γ = 87.87(1), *V* = 1165.8(2) Å^3, *Z* = 4, D_c = 1.392 g cm^{-3}, *μ* (Mo-Kα) = 0.263 mm^{-1}, *F*(000) = 512, rectangular block, colourless, size = 0.25 x 0.3 x 0.125 mm, 5174 reflections measured (R_{int} = 0.0209), 4110 unique, wR_2 = 0.1217 for all data, conventional *R* = 0.0455 [$(\Delta/\sigma)_{max}$ = 000)] on F-values of 2996 reflections with $I>2\sigma(I)$, *S* = 1.087 for all data and 311 parameters. Unit cell determination and intensity data collection (2θ= 50°) was performed on a Bruker P4 diffractometer at 293(2) K. Structure solutions by direct methods and refinements by full-matrix least-squares methods on F^2. Programs: XSCANS [Siemens Analytical X-ray Instrument Inc.: Madison, Wisconsin, USA 1996], SHELXTL-NT [Bruker AXS Inc.: Madison, Wisconsin, USA 1997]. CCDC (deposit No: 290318) contains the supplementary crystallographic data. These data can be obtained free of charge from www.ccdc.cam.uk/conts/retrieving.html [or from the Cambridge Crystallographic Data Center, 12 Union Road, Cambridge, CB2 1EZ, U. K; Fax: (Internet) + 44-1223/336-033; E-mail: deposit@ccdc.cam.ac.uk.

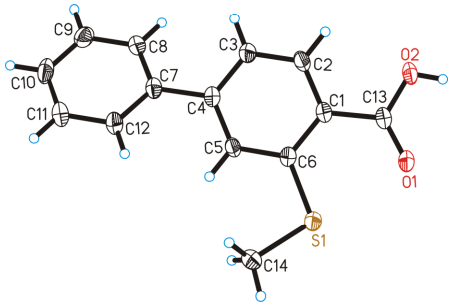
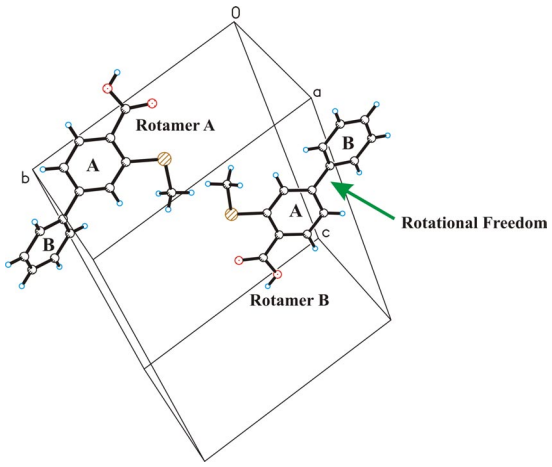


Figure 1. ORTEP diagram of compound **3a** with arbitrary numbering

The ORTEP diagram, Fig. 1 shows the crystal structure of **3a** and its conformation with atomic numbering scheme. The structural studies of the compound **3a** revealed the existence of two rotamers in the unit cell (Figure 2). The twisting angle of ring A and ring B around biaryl axis is 23.84° for one molecule and 36.68° for other molecule. The crystal packing of **3a** showed the most robust and reliable supramolecular pattern for aromatic carboxylic acid, which forms a stable eight-membered ring by O-H…O

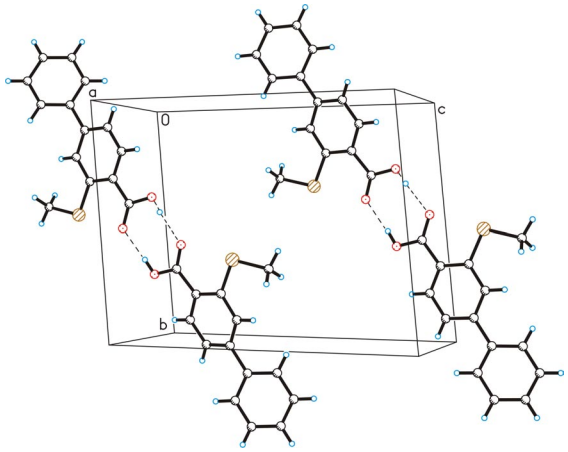
interactions [O2-H2A…O3: 2.634 Å and O4-H4A…O3: 2.667 Å] as indicated by dashed lines in Figure 3. There are two intramolecular H-bonds, C2-H2…O2, with H-bonding parameters C2-O2: 2.6892Å; H2…O2: 2.34Å, and C2-H2-O2: 102° and C16-H16…O4, with H-bonding parameters C16-O4: 2.6763Å; H16…O4: 2.33Å, and C16-H16-O4: 102°. The packing further shows S…O (S2…O3) close contact of distance 2.746 Å.

Figure 2: Unit cell packing diagram of 3a showing structures of two rotamers formed due to rotational degree of freedom along biaryl axis

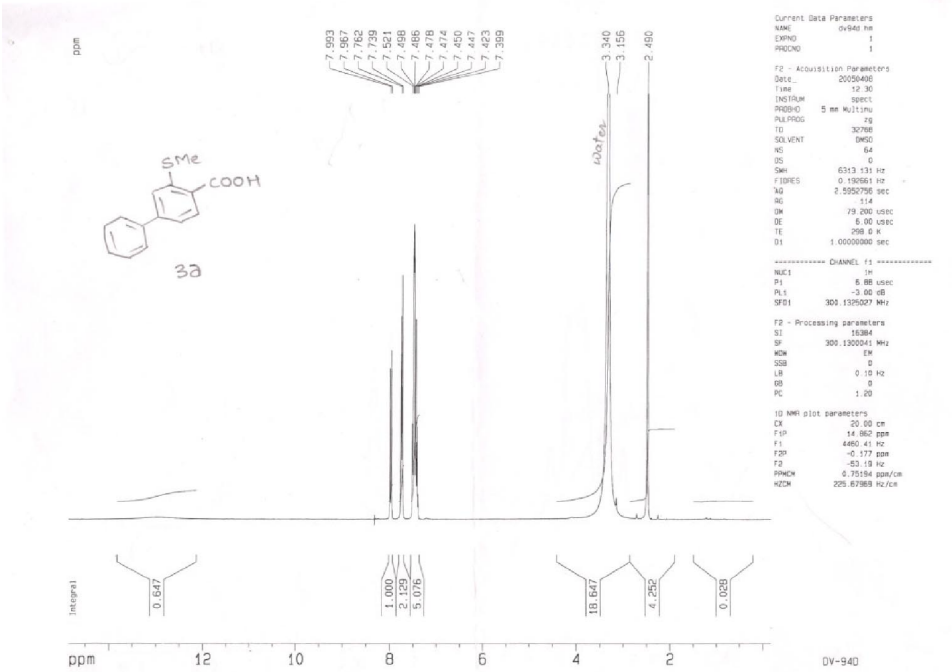


The twisting angle of ring A and ring B around biaryl axis is 23.84° for one molecule and 36.68° for other molecule.

Figure 3: Stable eight-membered ring by O-H…O interactions of carboxylic acid



Packing diagram of **3a** showing carboxylic acid dimerisation due to stable eight-membered ring formed by intermolecular strong H-bonding through O-H…O interactions [O2-H2A…O3: 2.634 Å, ∠ OHO = 173.87° and O4-H4A…O3: 2.667 Å, ∠ OHO = 175.27°]. Symmetry codes [x, y, z+1 and x, y, z-1]. There are two intramolecular H-bonds, C2-H2…O2, with H-bonding parameters C2-O2: 2.6892Å; H2…O2: 2.34Å, and C2-H2-O2: 102° and C16-H16…O4, with H-bonding parameters C16-O4: 2.6763 Å; H16…O4: 2.33 Å, and C16-H16-O4: 102°.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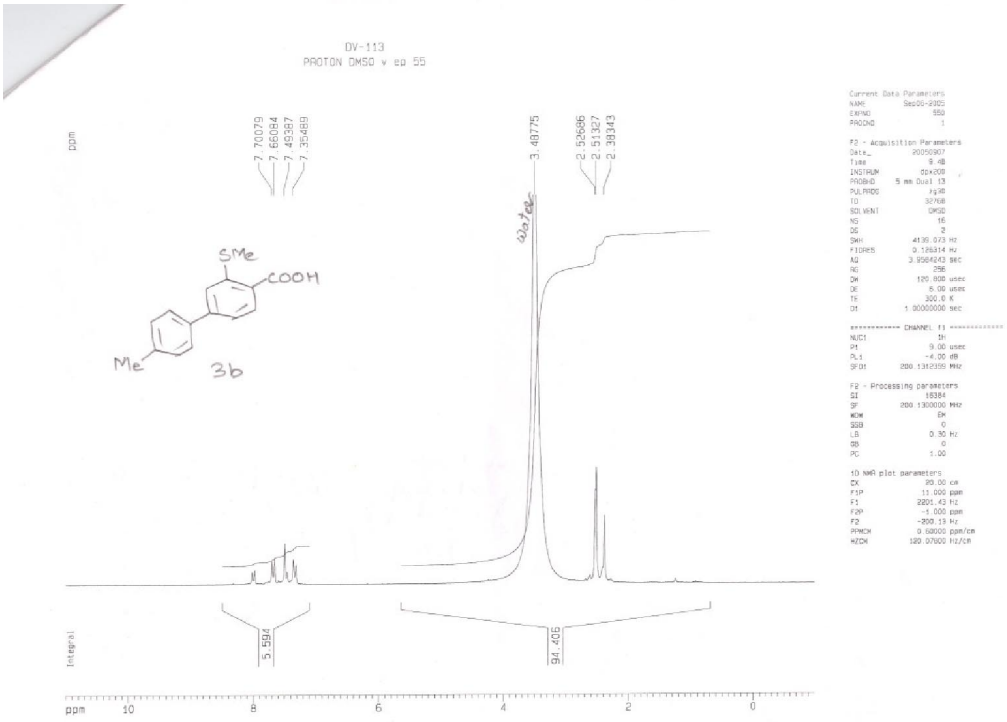
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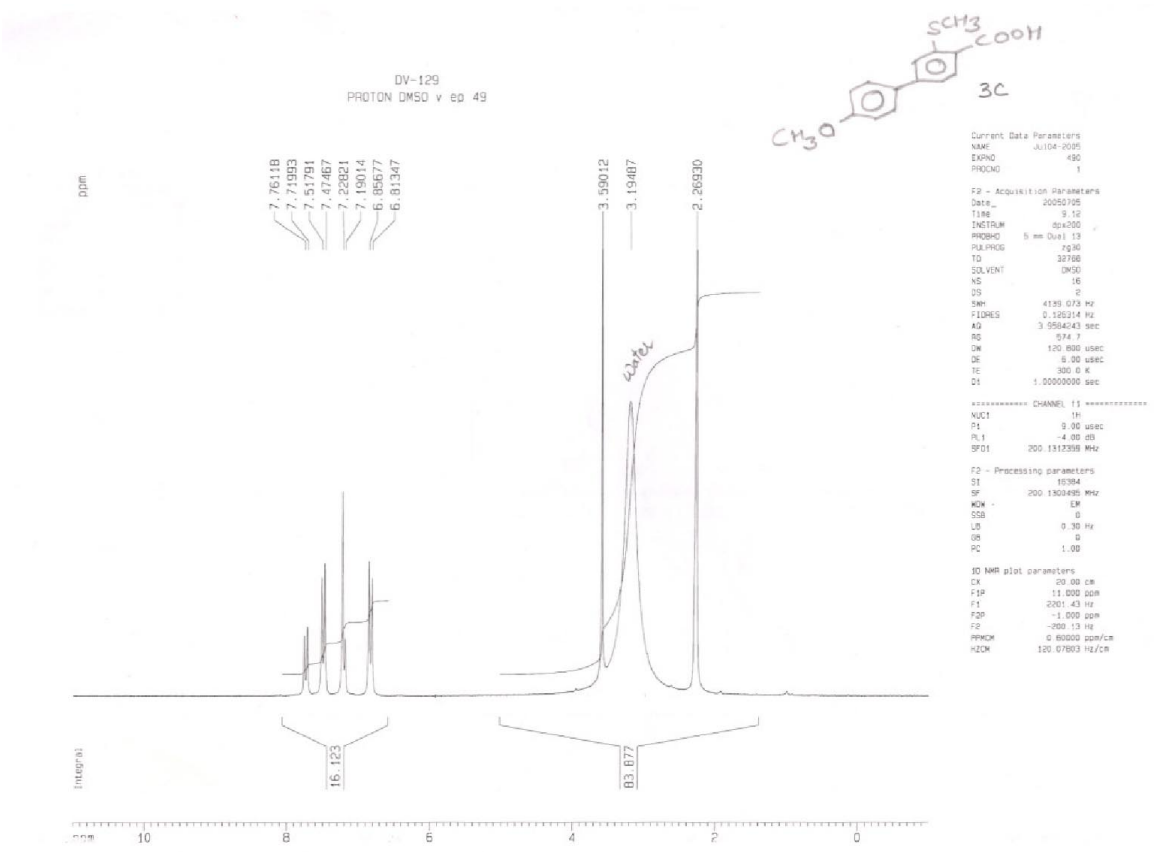
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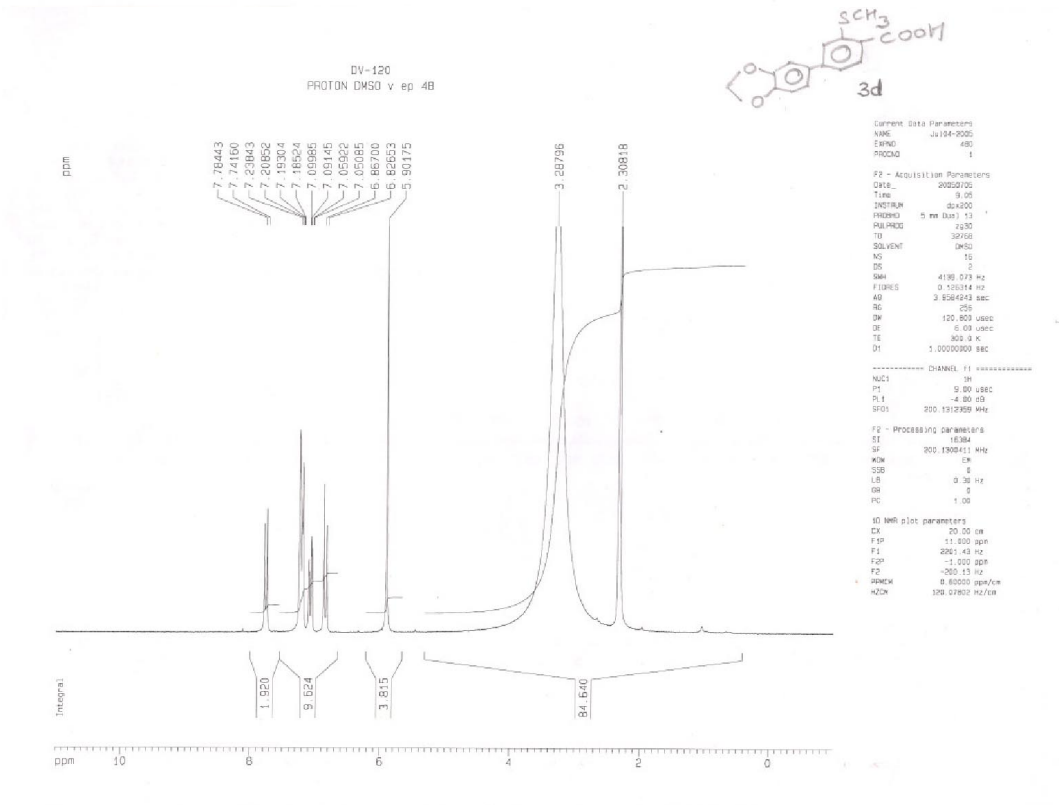
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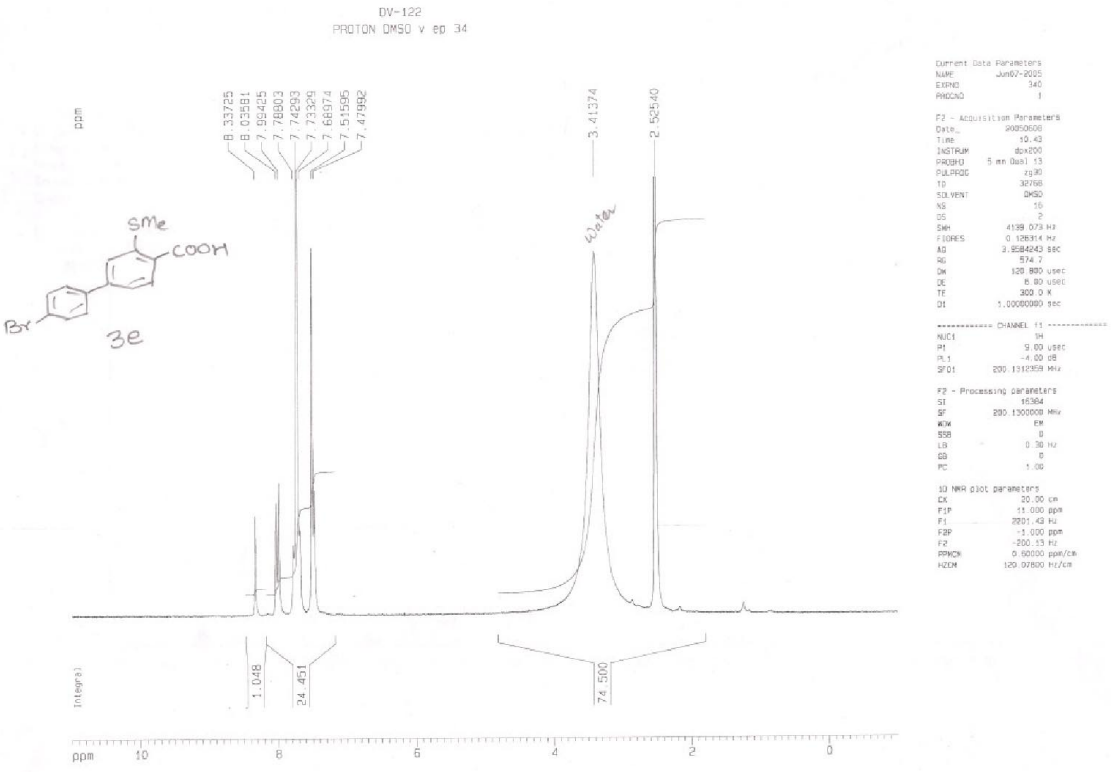

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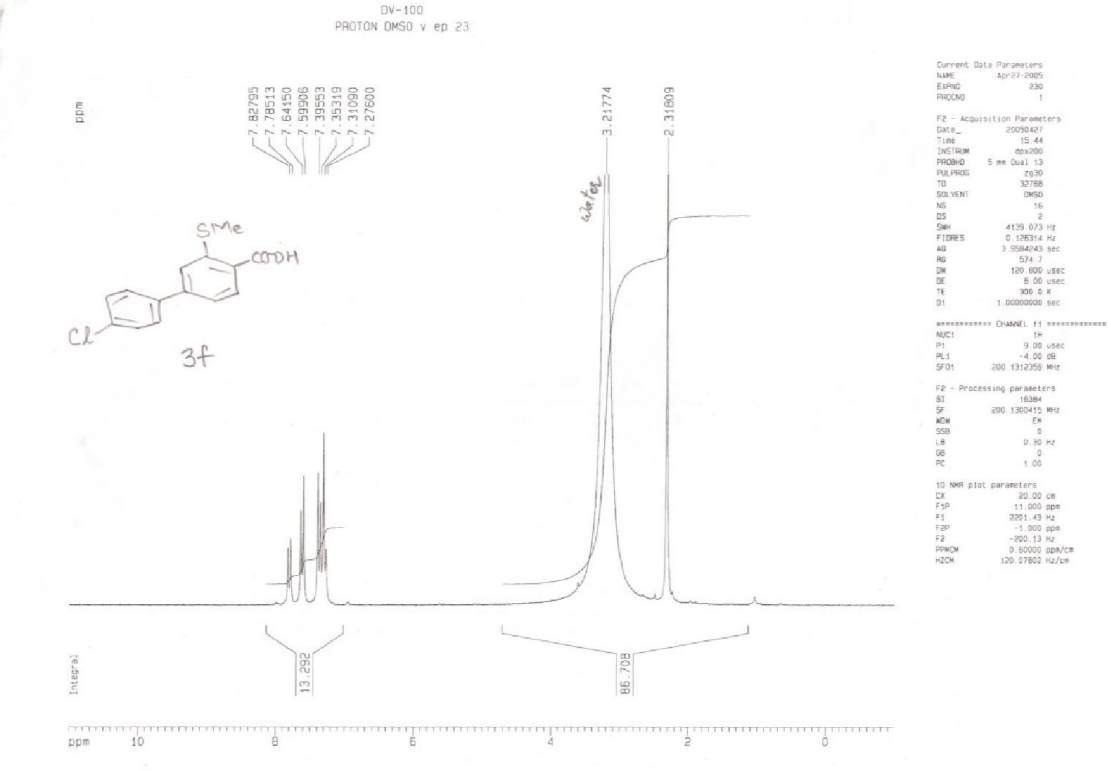

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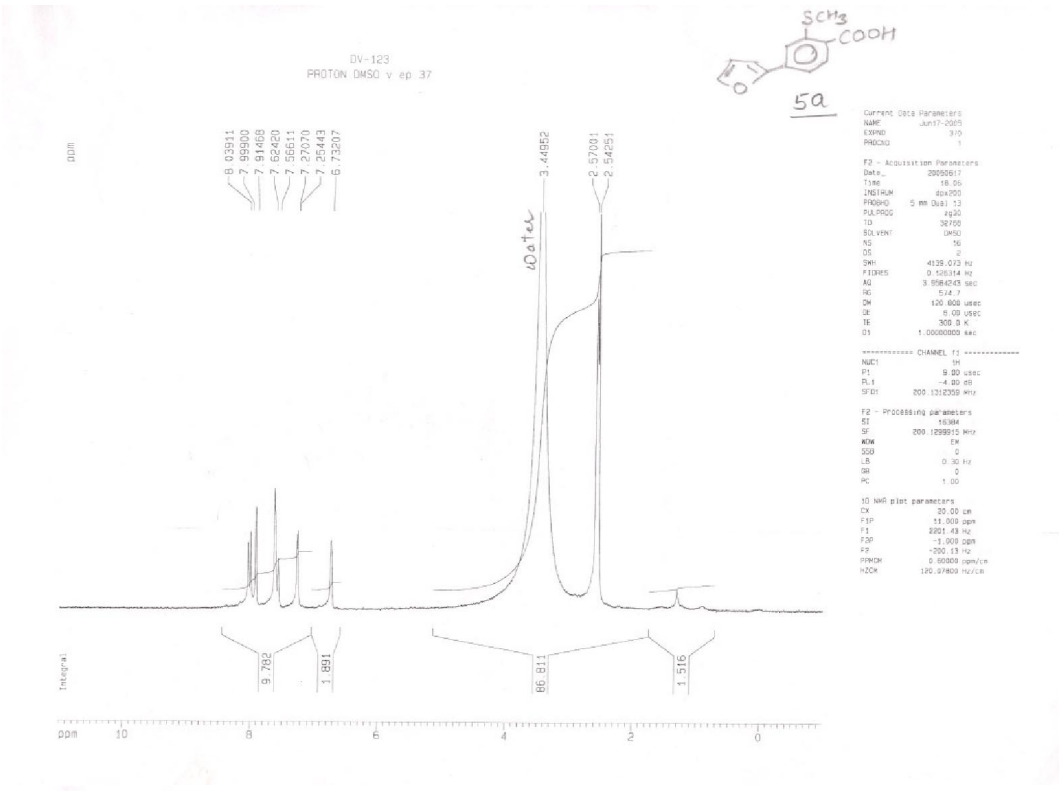
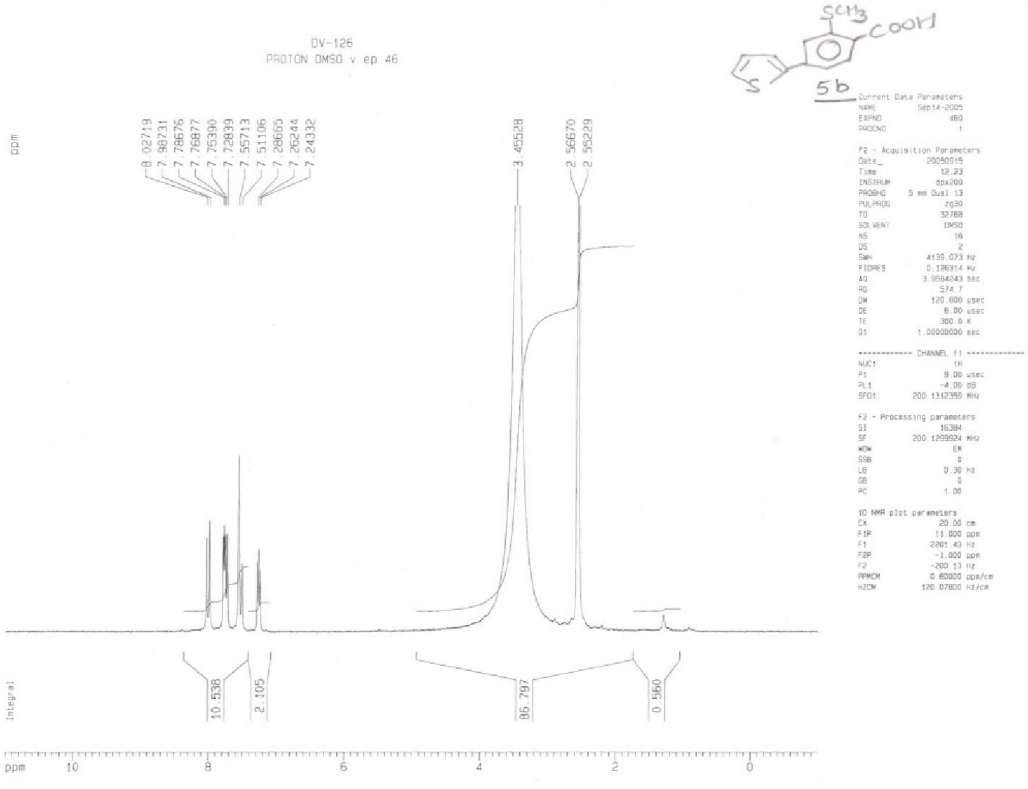
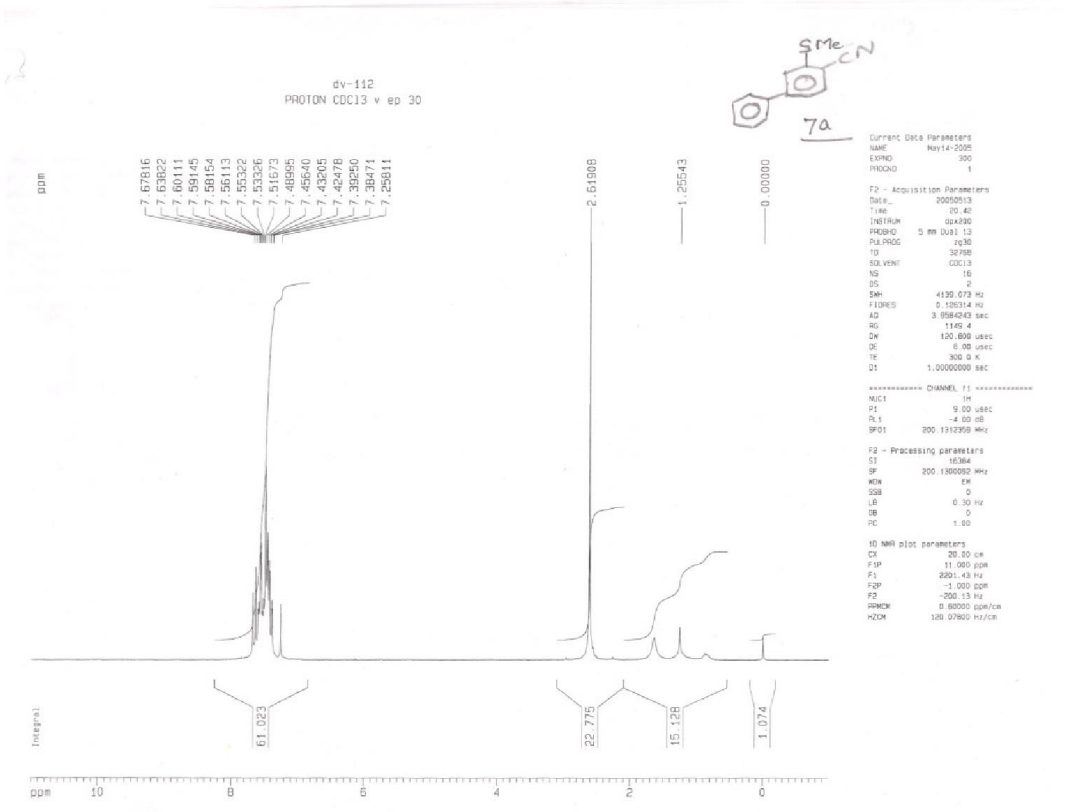
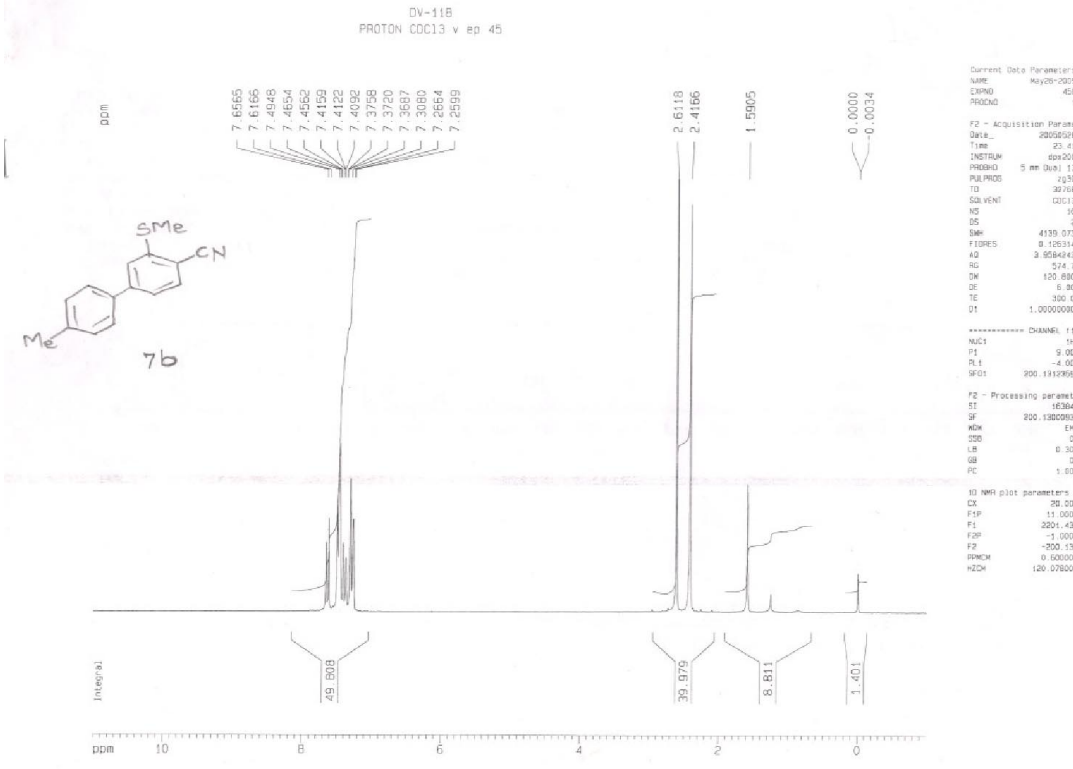
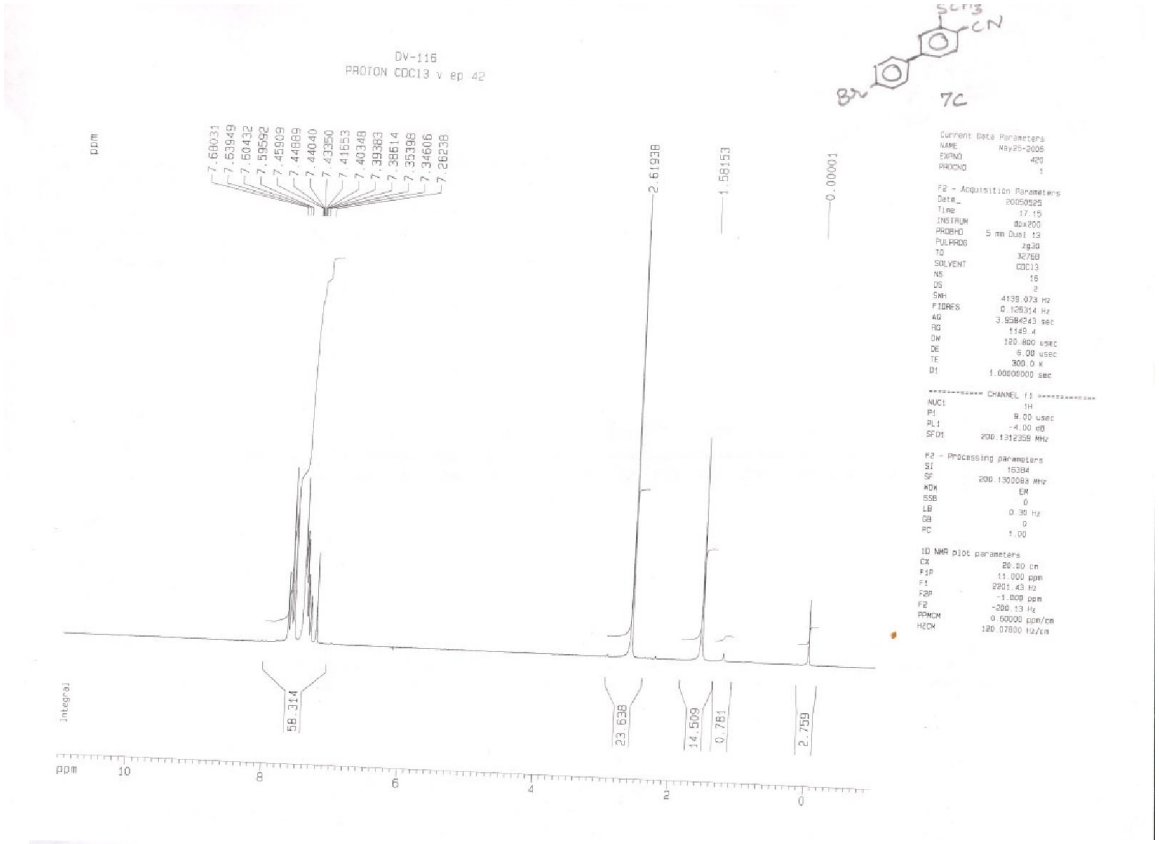
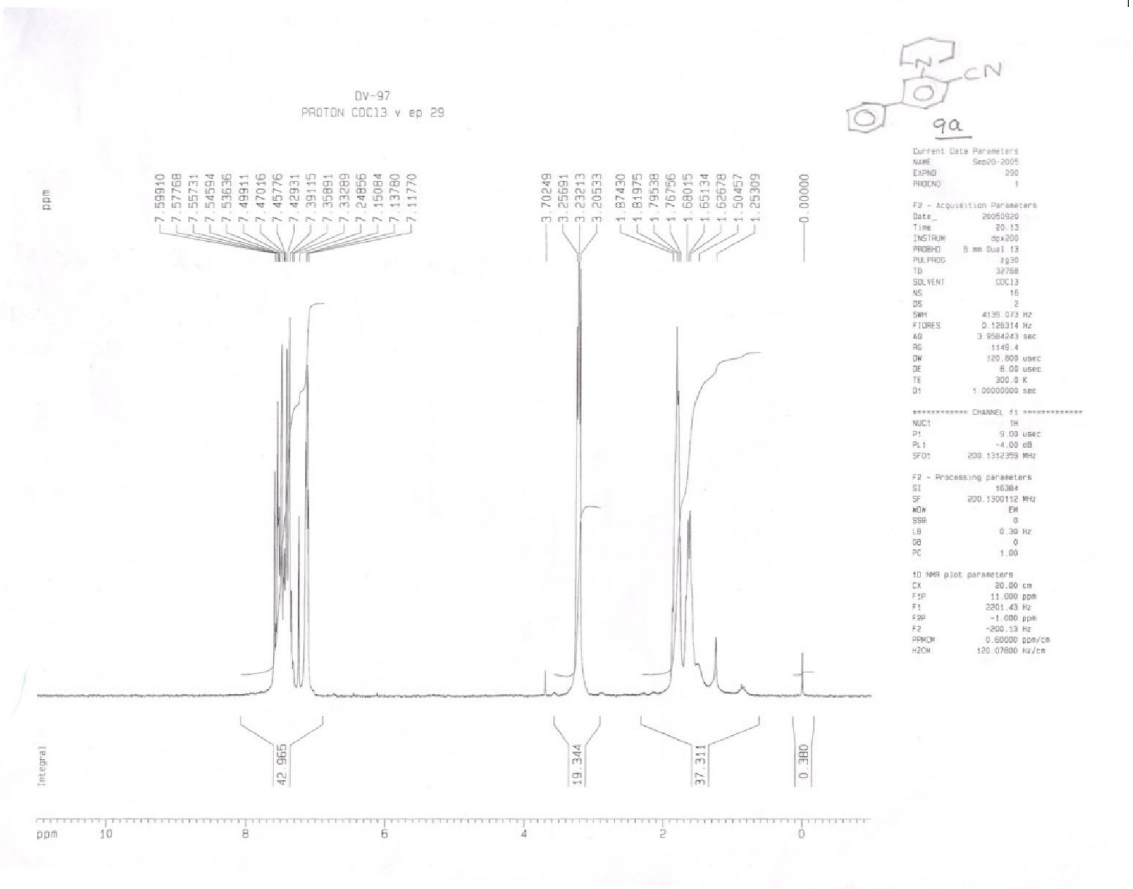

DV-123
PROTON DMSO v ep 37
SCH3
COOH
5a
ppm
8.03991
7.99900
7.91468
7.62420
7.56611
7.27070
7.25443
6.73207
3.44952
water
2.57001
2.54251
Integral
9.782
1.891
86.811
1.516
ppm 10 8 6 4 2 0
Current Data Parameters
NAME Jun17-2005
EXPNO 310
PROCNO 1
F2 - Acquisition Parameters
Date_ 20050617
Time 18.06
INSTRUM spx200
PROBHD 5 mm Dual 13
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 16
DS 2
SWH 4139.073 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 574.7
DW 120.600 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
CHANNEL f1
NUC1 1H
P1 9.00 usec
PL1 -4.00 dB
SFO1 200.1312359 MHz
F2 - Processing parameters
SI 16384
SF 200.1299915 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
F1P 11.000 ppm
F1 2201.43 Hz
F2P -1.000 ppm
F2 -200.13 Hz
PPMCM 0.60000 ppm/cm
HZCM 120.07800 Hz/cm

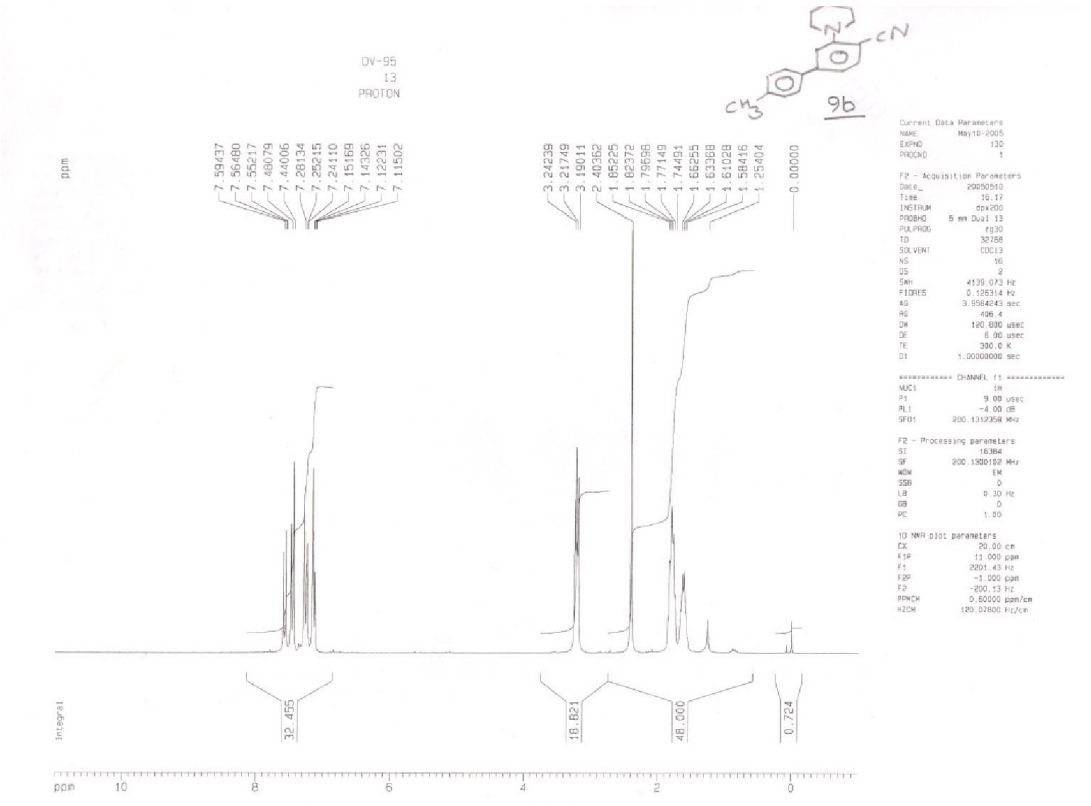

DV-126
PROTON DMSO v ep 46
SCH3
COOH
5b
ppm
8.02719
7.98731
7.78676
7.76877
7.75390
7.72839
7.55713
7.51106
7.28665
7.26244
7.24332
3.45528
2.56670
2.55229
Integral
10.536
2.105
86.797
0.560
ppm 10 8 6 4 2 0
Current Data Parameters
NAME Sep14-2005
EXPNO 460
PROCNO 1
F2 - Acquisition Parameters
Date_ 20050915
Time 12.23
INSTRUM spx200
PROBHD 5 mm Dual 13
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 16
DS 2
SWH 4139.073 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 574.7
DW 120.600 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
CHANNEL f1
NUC1 1H
P1 9.00 usec
PL1 -4.00 dB
SFO1 200.1312359 MHz
F2 - Processing parameters
SI 16384
SF 200.1299924 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
F1P 11.000 ppm
F1 2201.43 Hz
F2P -1.000 ppm
F2 -200.13 Hz
PPMCM 0.60000 ppm/cm
HZCM 120.07800 Hz/cm

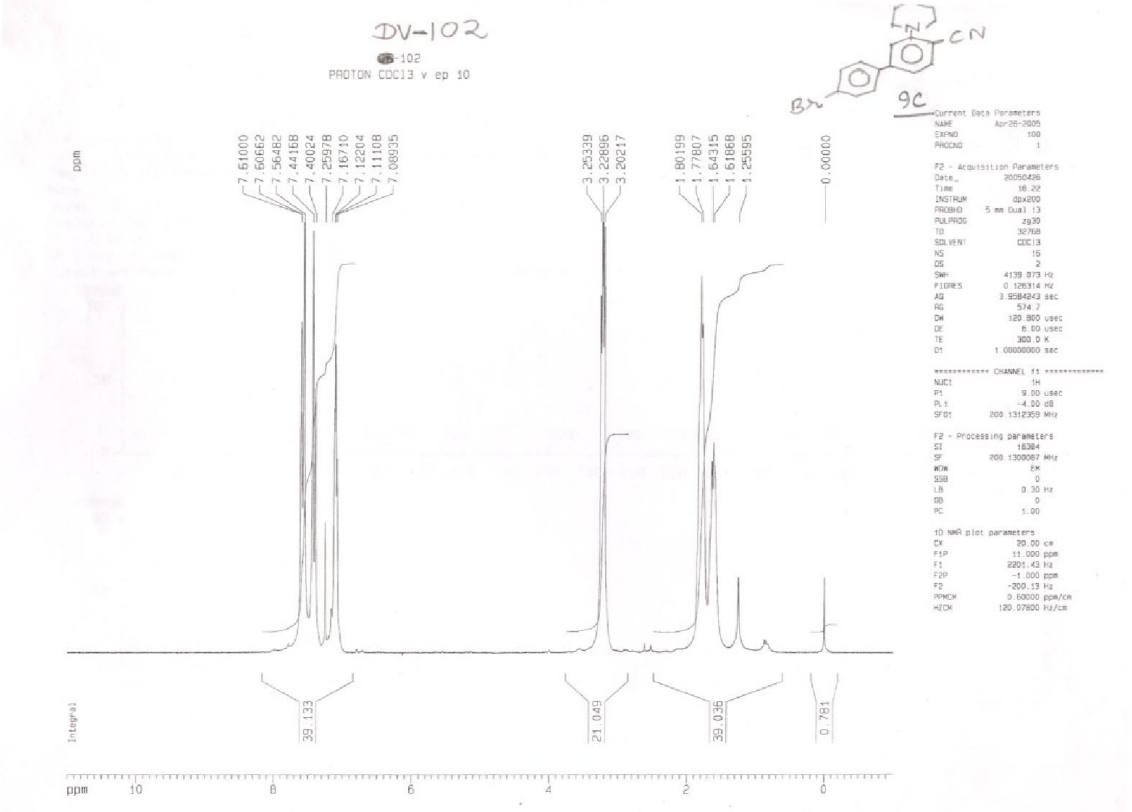

dv-112
PROTON CDCl3 v ep 30
SMe
CN
7a
ppm
7.67816
7.63822
7.60111
7.59145
7.58154
7.56113
7.55322
7.53326
7.51673
7.48995
7.45640
7.43205
7.42478
7.39250
7.38471
7.25841
2.61908
1.25543
0.00000
Integral
61.023
22.775
15.128
1.074
ppm 10 8 6 4 2 0

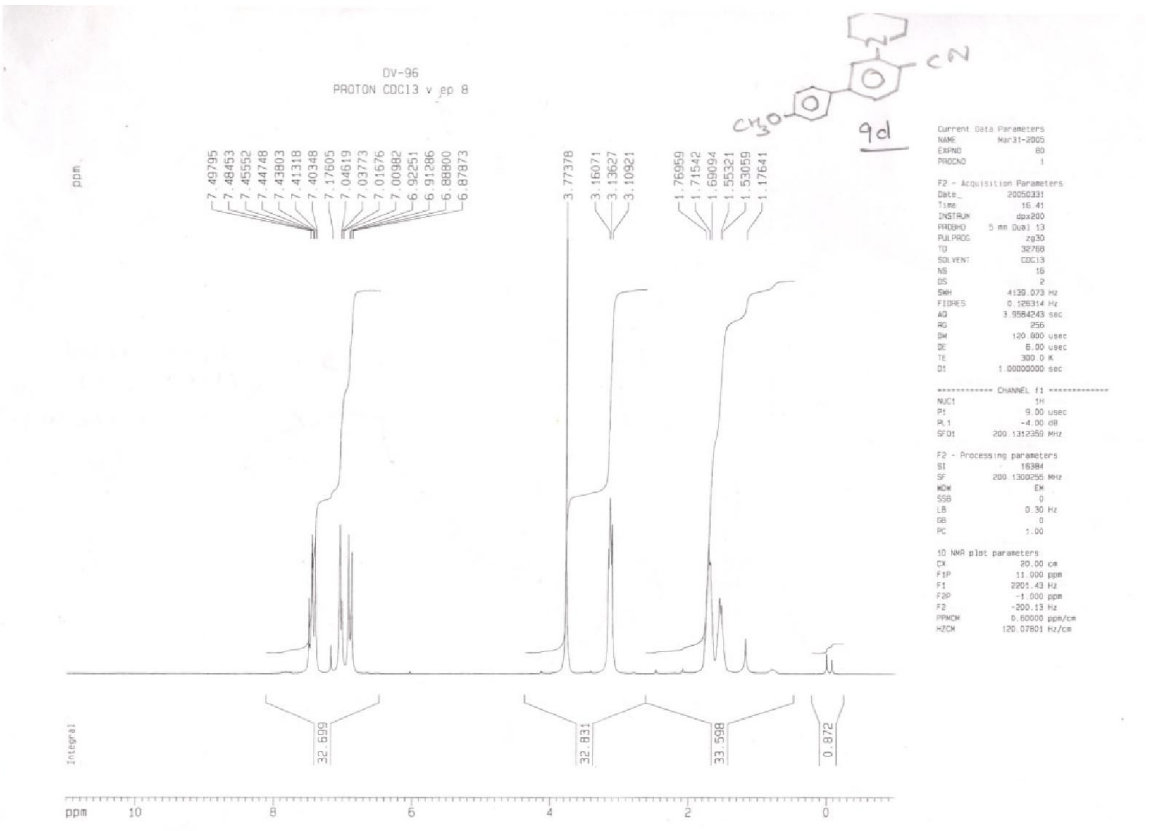

DV-118
PROTON CDCl3 v ep 45
SMe
CN
Me
7b
ppm
7.6565
7.6166
7.4948
7.4654
7.4562
7.4159
7.4122
7.4092
7.3758
7.3720
7.3687
7.3080
7.2664
7.2599
2.6118
2.4166
1.5905
0.0000
-0.0034
Integral
49.808
39.979
8.611
1.401
ppm 10 8 6 4 2 0

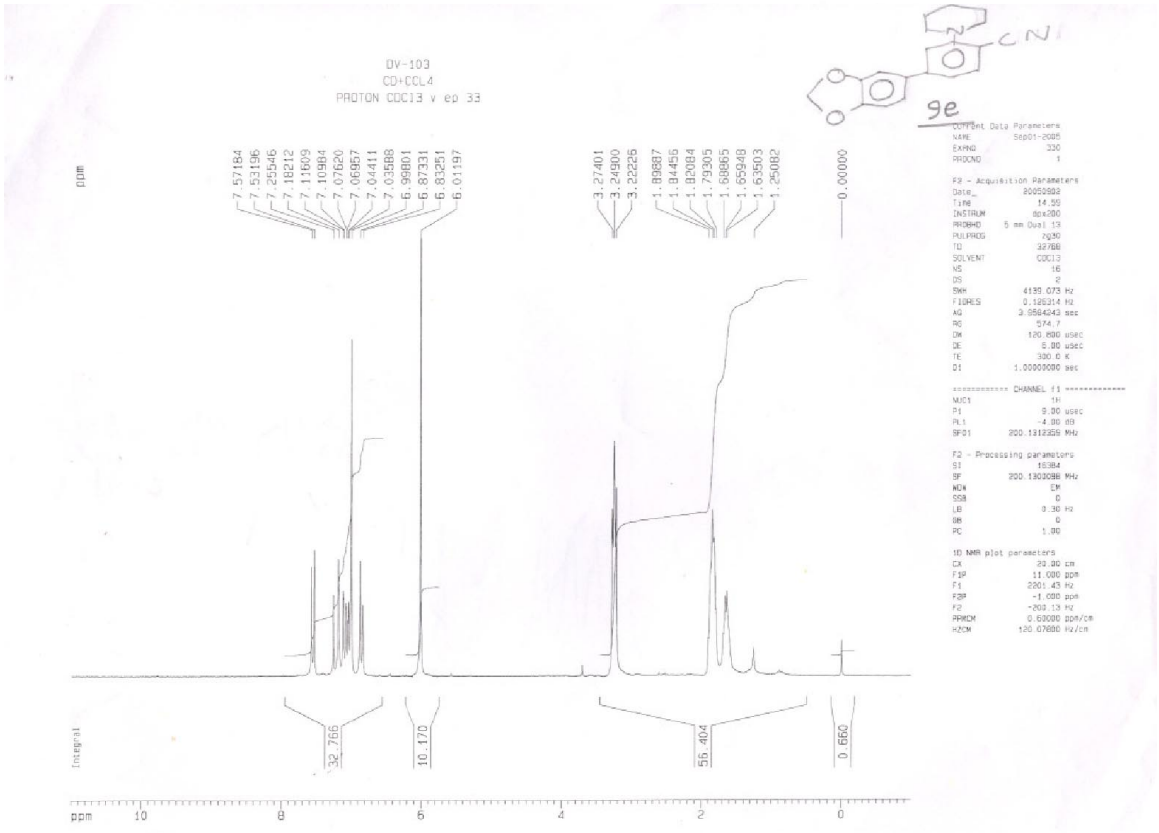

DV-116
PROTON CDCl3 v ep 42
7c
ppm
integral


DV-97
PROTON CDCl3 v ep 29
9a
ppm
integral


DV-95
13
PROTON
9b
ppm
7.59437
7.56490
7.35217
7.48079
7.44006
7.28134
7.25215
7.24110
7.15169
7.14326
7.12231
7.11502
3.24239
3.21749
3.19011
2.40362
1.85225
1.82372
1.79698
1.77149
1.74491
1.66255
1.63368
1.61028
1.58416
1.25404
0.00000
Integral
32.455
16.821
48.000
0.724
ppm 10 8 6 4 2 0


DV-102
DV-102
PROTON CDCl3 v ep 10
9c
ppm
7.61000
7.60662
7.56482
7.44168
7.40024
7.25978
7.16710
7.12204
7.11108
7.08935
3.25339
3.22896
3.20217
1.90199
1.77807
1.64315
1.61068
1.25595
0.00000
Integral
39.133
21.049
39.036
0.781
ppm 10 8 6 4 2 0


DV-96
PROTON CDCl3 v ep 8
9d
ppm
7.49795
7.48453
7.45552
7.44748
7.43803
7.41318
7.40348
7.17605
7.04619
7.03773
7.01676
7.00982
6.92251
6.91286
6.88800
6.87873
3.77378
3.16071
3.13627
3.10921
1.76959
1.71542
1.69094
1.55321
1.53059
1.17641
Integral
32.699
32.831
33.598
0.872
ppm 10 8 6 4 2 0


DV-103
CD+CCL4
PROTON CDCl3 v ep 33
9e
ppm
7.57184
7.53196
7.25546
7.18212
7.16609
7.10984
7.07620
7.06957
7.04411
7.03588
6.99801
6.87331
6.83251
6.01197
3.27401
3.24900
3.22226
1.89687
1.84456
1.82084
1.79305
1.68865
1.65948
1.63503
1.25082
0.00000
Integral
32.766
10.170
56.404
0.660
ppm 10 8 6 4 2 0