

Electronic Supplementary Information

Representative spectral data for:

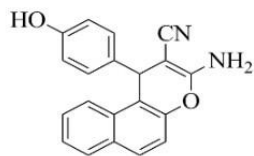
Ball milling for the quantitative and specific solvent-free Knoevenagel condensation + Michael addition cascade in the synthesis of various 2-amino-4-aryl-3-cyano-4*H*-chromenes without heating

Omid Hosseinchi Qareaghaj^a, Sara Mashkouri^a, M. Reza Naimi-Jamal^{a*}, Gerd Kaupp^b

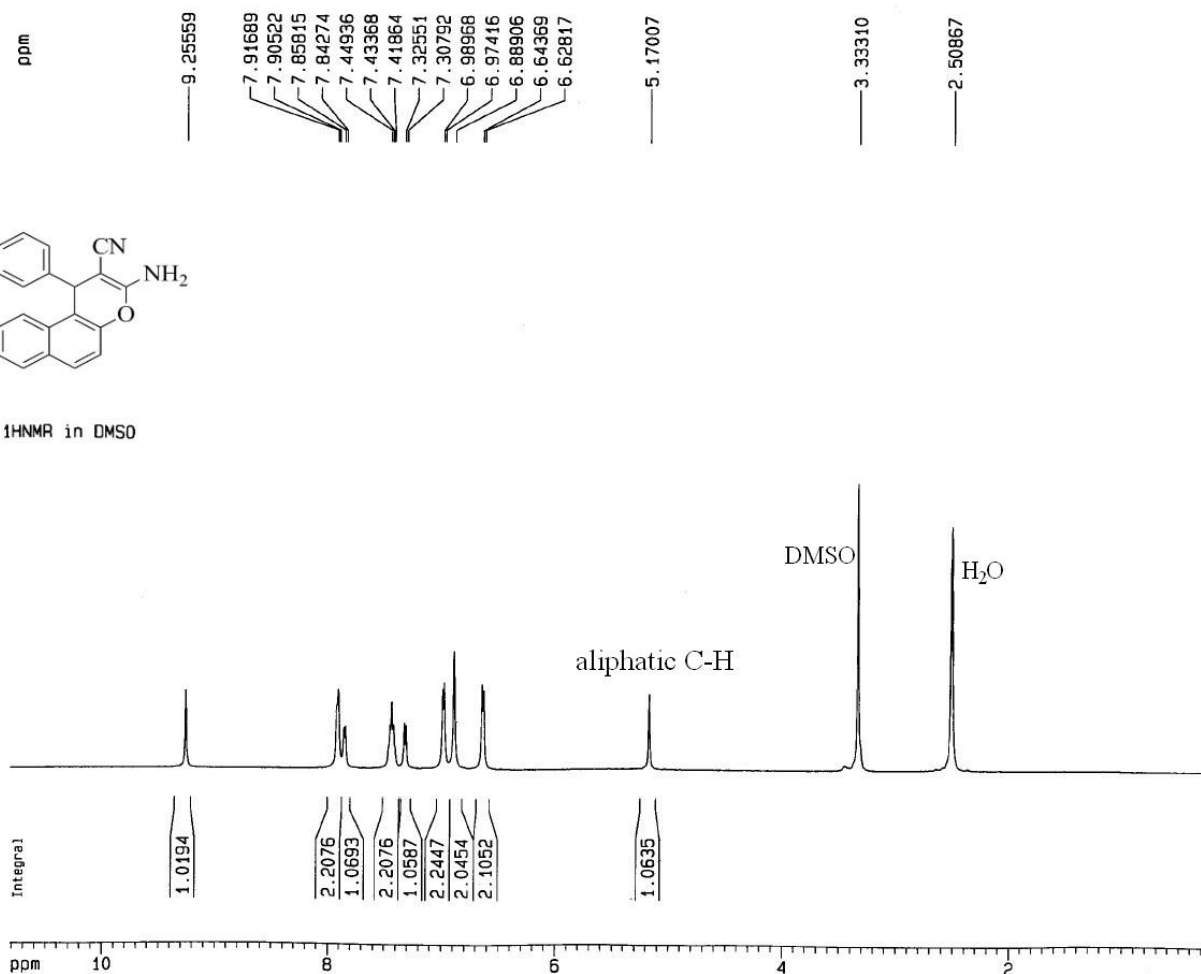
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E-mail address: naimi@iust.ac.ir (M. R. Naimi-Jamal).*

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A 1H NMR in DMSO at 298 K



1H NMR in DMSO



```

INSTRUM      spect
PROBHD      5 mm QNP 1H/13
PULPROG      zg30
TD          32768
SOLVENT      DMSO
NS           16
DS           1
SWH         10330.578 Hz
FIDRES      0.315264 Hz
AQ          1.5860696 sec
RG           574.7
DW          48.400 usec
DE           6.50 usec
TE           298.0 K
D1           5.0000000 sec
MCREST      0.0000000 sec
MCWFK       0.0150000 sec
    
```

```

***** CHANNEL f1 *****
NUC1         1H
P1           10.50 usec
PL1          -3.00 dB
SF01        500.1330885 MHz
    
```

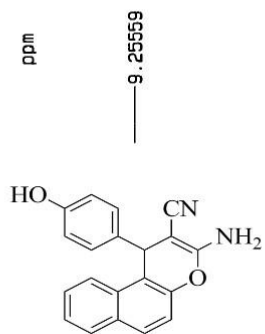
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F2 - Processing parameters
SI           32768
SF           500.1300000 MHz
WDW          EM
SSB          0
LB           0.30 Hz
GB           0
PC           1.00
    
```

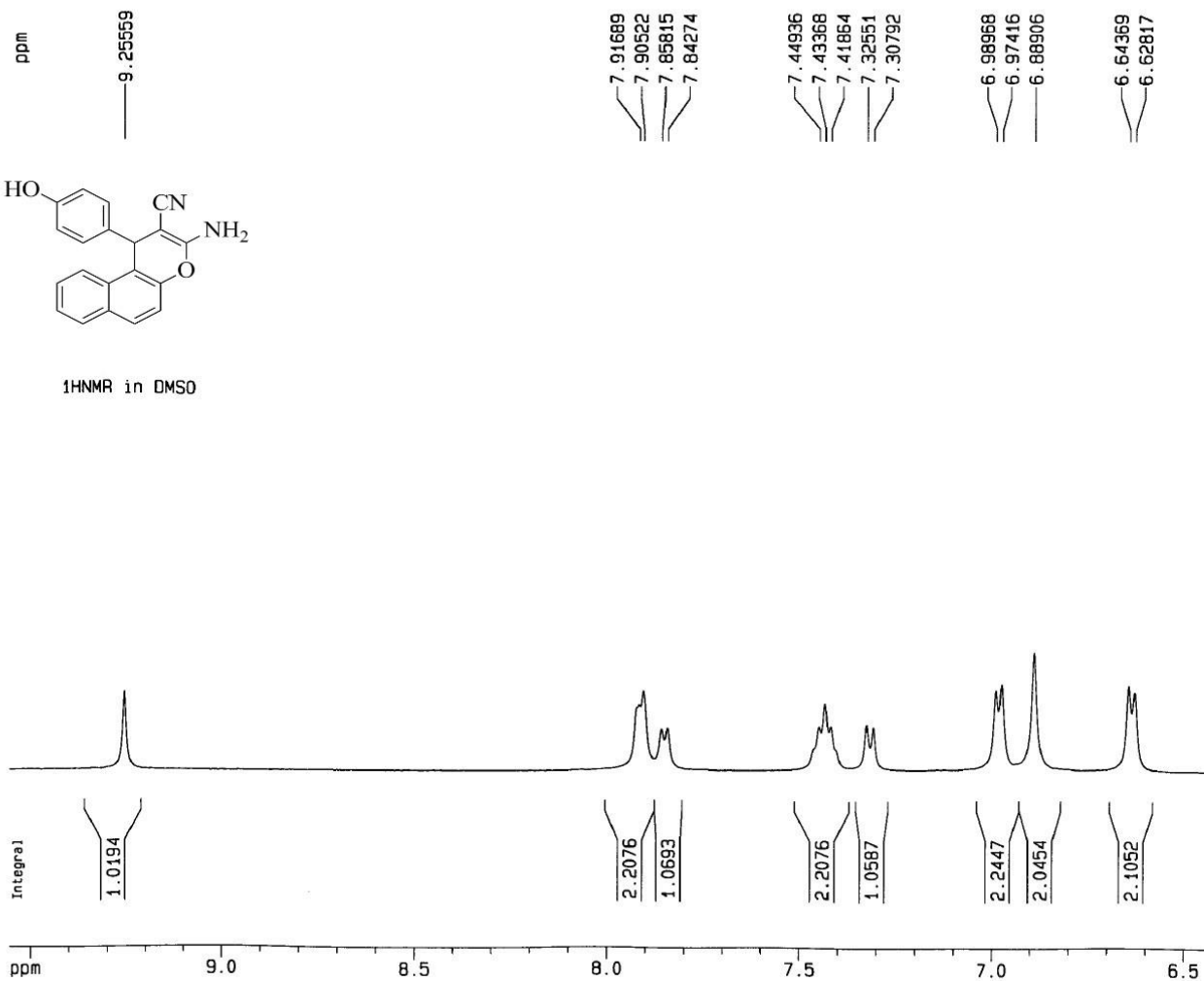
```

1D NMR plot parameters
CX           20.00 cm
CY           4.82 cm
F1P         10.809 ppm
F1           5405.97 Hz
F2P         0.251 ppm
F2           125.38 Hz
PPMCM       0.52792 ppm/cm
HZCM        264.02930 Hz/cm
    
```

A ¹H NMR in DMSO at 298 K



¹H NMR in DMSO



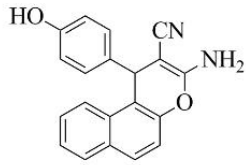
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 16
DS 1
SWH 10330.578 Hz
FIDRES 0.315264 Hz
AQ 1.5860696 sec
RG 574.7
DM 48.400 usec
DE 6.50 usec
TE 298.0 K
D1 5.0000000 sec
MCREST 0.0000000 sec
MCWAK 0.0150000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 10.50 usec
PL1 -3.00 dB
SF01 500.1330885 MHz

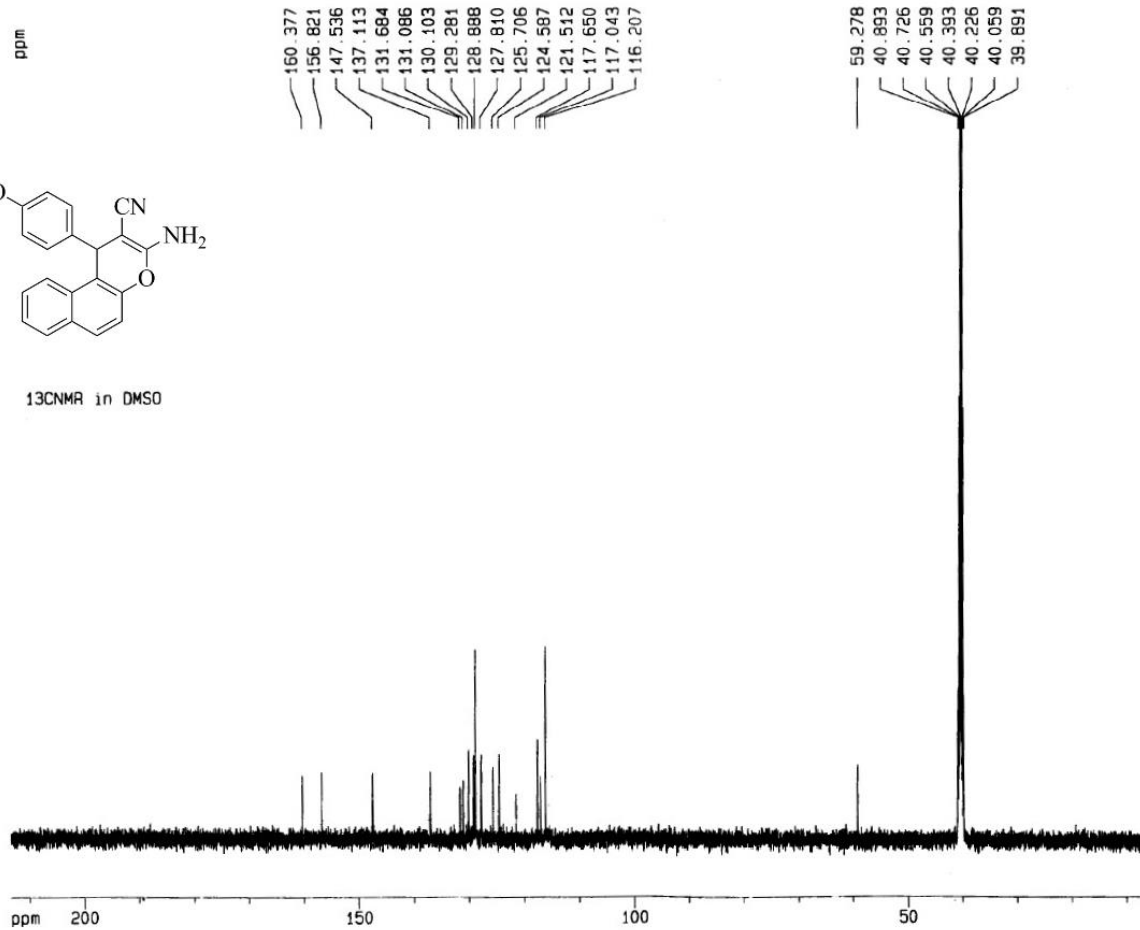
F2 - Processing parameters
SI 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 4.82 cm
F1P 9.554 ppm
F1 4778.32 Hz
F2P 6.411 ppm
F2 3206.39 Hz
PPMCM 0.15715 ppm/cm
HZCM 78.59666 Hz/cm

P-OH+B 13CNMR in DMSO at 298 K



13CNMR in DMSO



160.377
156.821
147.536
137.113
131.684
131.086
130.103
129.281
128.888
127.810
125.706
124.587
121.512
117.650
117.043
116.207

59.278
40.893
40.726
40.559
40.393
40.226
40.059
39.891

```

INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 430
DS 4
SWH 30030.029 Hz
FIDRES 0.456222 Hz
AQ 1.0912410 sec
RG 6502
DM 16.650 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
MCREST 0.00000000 sec
MCHWK 0.01500000 sec
    
```

```

----- CHANNEL f1 -----
NUC1 13C
P1 9.10 usec
PL1 3.00 dB
SF01 125.7703643 MHz
    
```

```

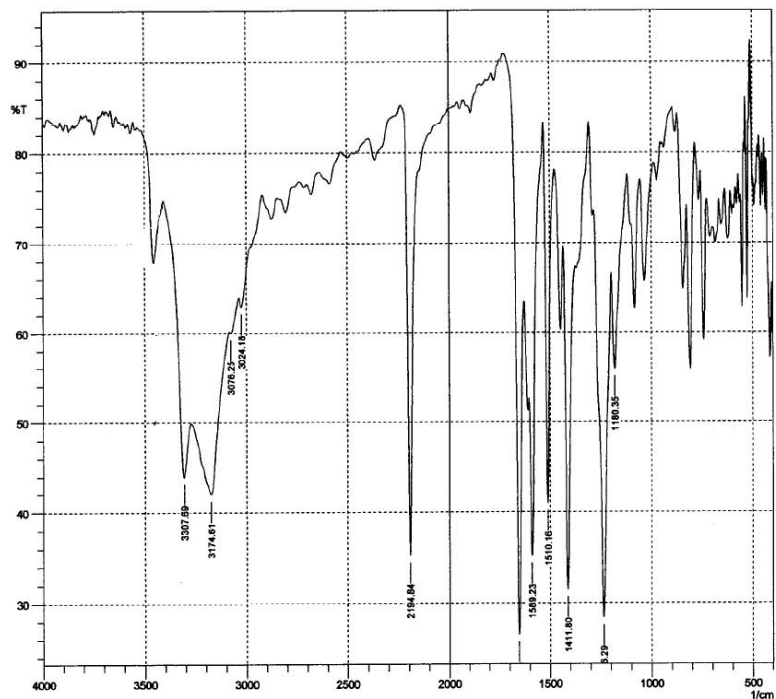
----- CHANNEL f2 -----
CPOPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 15.50 dB
PL13 15.50 dB
SF02 500.1320005 MHz
    
```

```

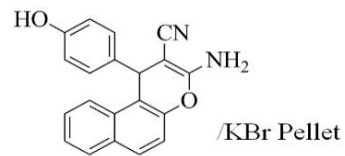
F2 - Processing parameters
SI 32768
SF 125.7577390 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
    
```

```

1D NMR plot parameters
CX 20.00 cm
CY 18.30 cm
F1P 213.506 ppm
F1 26850.06 Hz
F2P 6.134 ppm
F2 771.34 Hz
PPMCM 10.36863 ppm/cm
HZCM 1303.93555 Hz/cm
    
```



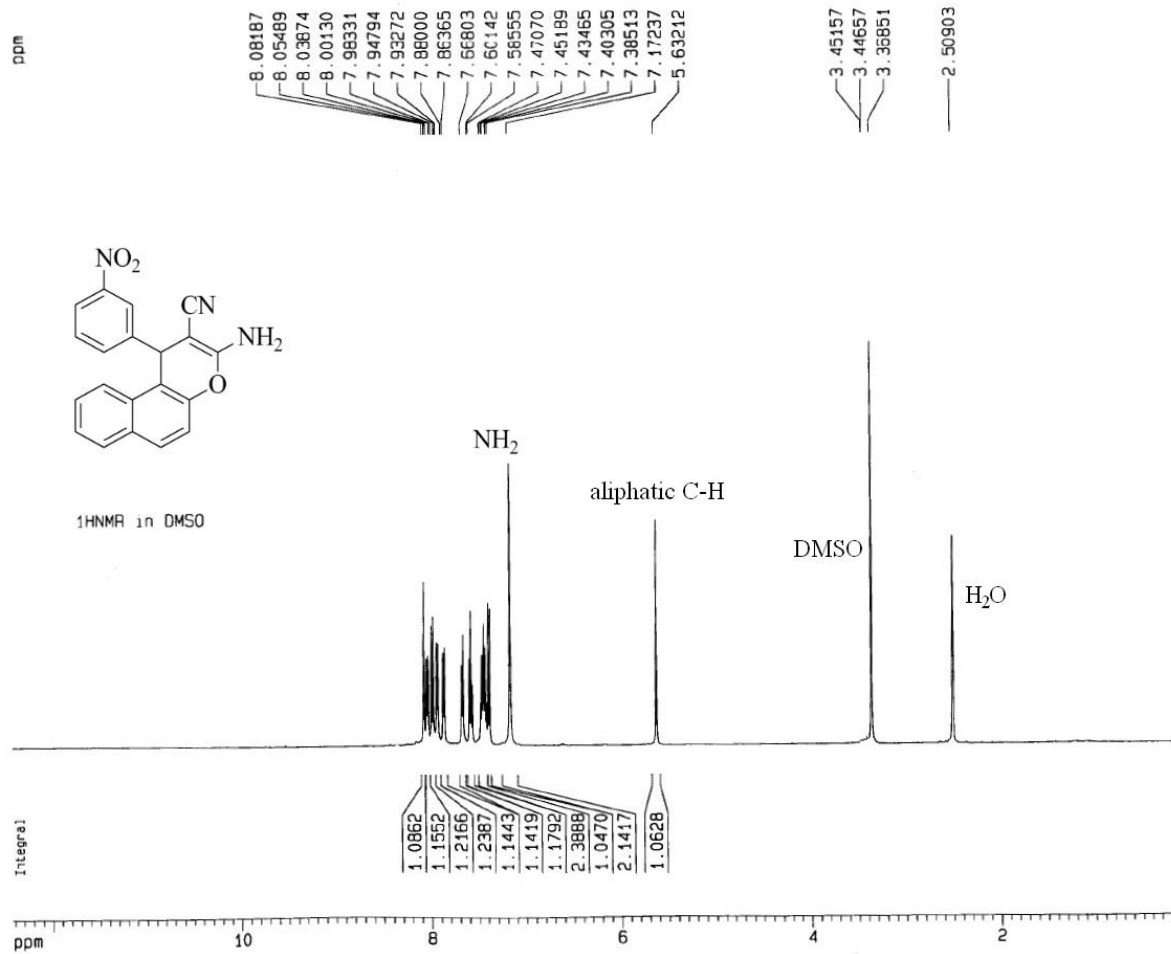
1180.35	55.865	12.914	1105.78	1118.64	13.684	2.681
1236.29	28.418	41.294	1282.57	1197.71	26.517	13.301
1411.8	31.532	37.207	1433.01	1375.15	16.835	7.292
1510.16	41.011	40.315	1529.45	1477.37	10.517	5.615
1589.23	35.239	23.082	1602.74	1531.37	15.455	3.333
1652.88	26.584	44.328	1720.39	1627.81	18.599	8.394
2194.84	35.305	49.415	2235.34	2069.48	22.838	10.406
3024.18	62.893	2.161	3035.75	2920.03	18.674	0.479
3076.25	60.008	0.777	3083.96	3037.68	9.764	0.181
3174.61	42.015	13.261	3272.98	3085.89	58.772	9.939
3307.69	43.945	12.139	3406.05	3274.9	30.782	3.497
3452.34	48.978	29.125	3512.13	3407.98	15.941	5.011



Comment;

No. of Scans;
Resolution;

A4 1HNMR in DMSO at 298 K



----- CHANNEL f1 -----
NUC1 1H
P1 10.50 usec
PL1 -3.00 dB
SF01 500.1330885 MHz

F2 - Processing parameters
SI 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

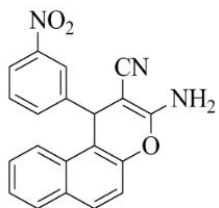
1D NMR plot parameters
CX 20.00 cm
CY 6.93 cm
F1P 12.426 ppm
F1 6214.87 Hz
F2P 0.151 ppm
F2 75.42 Hz
PPMCM 0.61378 ppm/cm
HZCM 305.97226 Hz/cm

A4 1HNMR in DMSO at 298 K

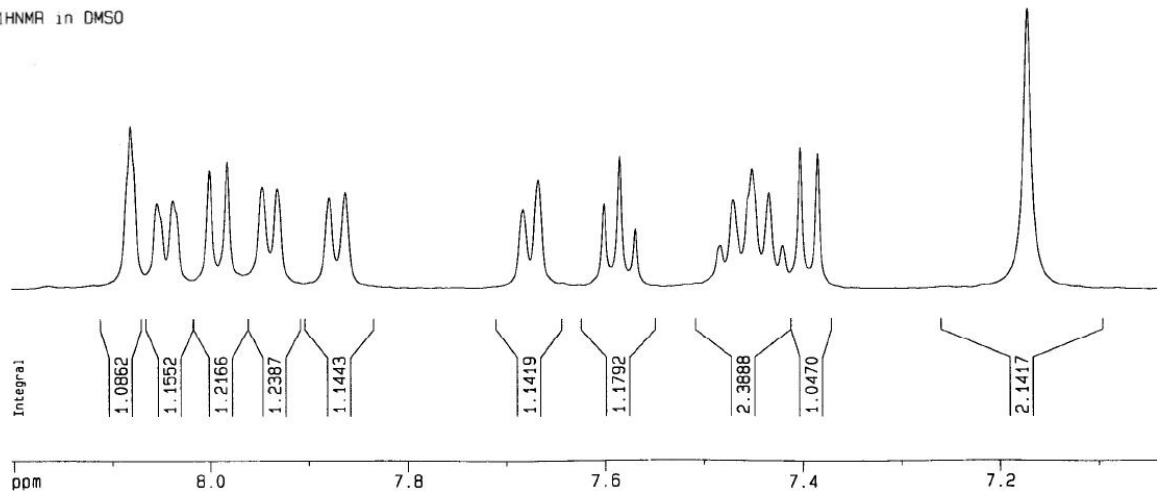
ppm
8.08187
8.05489
8.03874
8.00130
7.98331
7.94794
7.93272
7.88000
7.86365

7.68342
7.66803
7.60142
7.58555
7.56980
7.48380
7.47070
7.45189
7.43465
7.42095
7.40305
7.38513

7.17237



1HNMR in DMSO



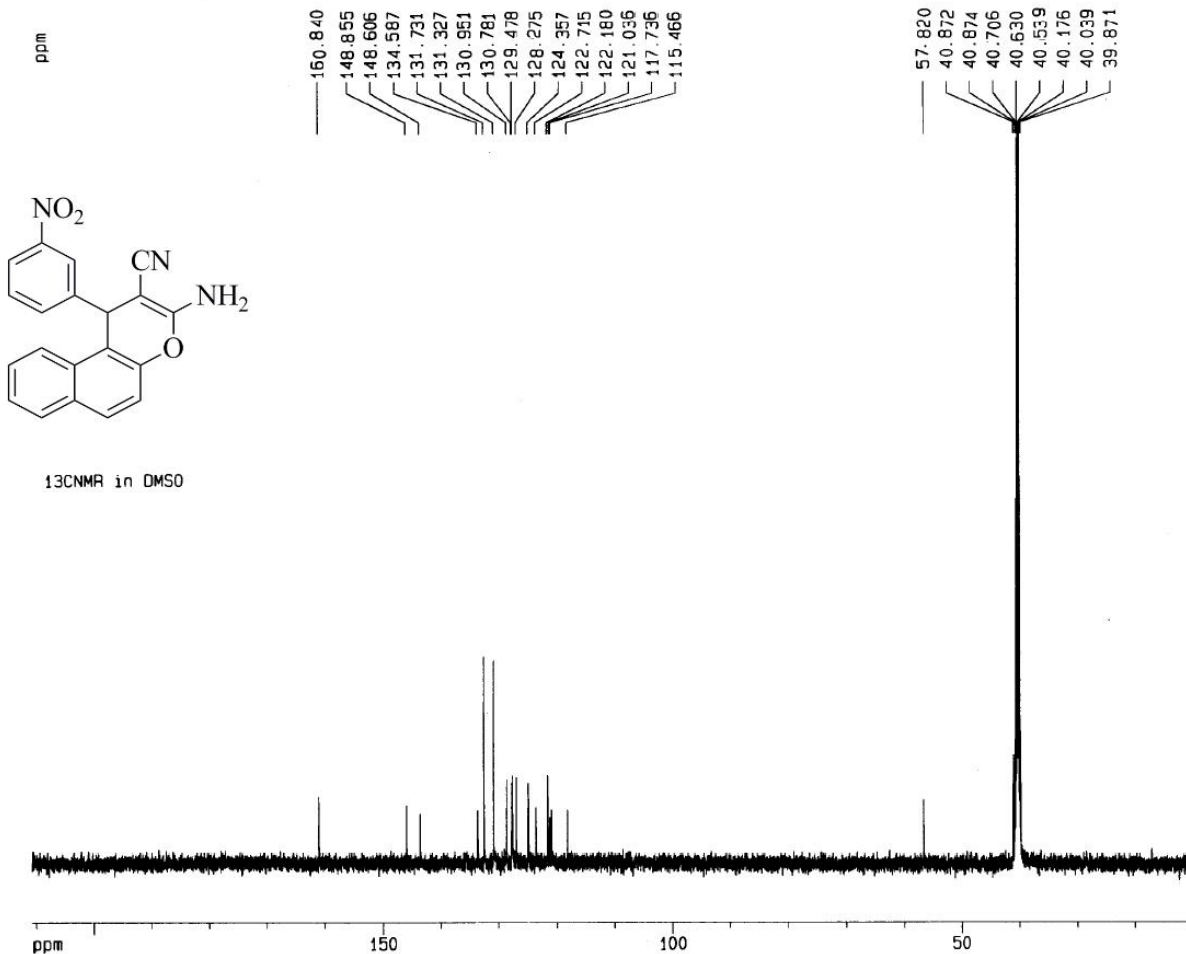
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 16
DS 1
SWH 10330.578 Hz
FIDRES 0.315264 Hz
AQ 1.5860596 sec
RG 287.4
DW 48.400 usec
DE 6.50 usec
TE 294.6 K
D1 5.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 10.50 usec
PL1 -3.00 dB
SFO1 500.1330985 MHz

F2 - Processing parameters
SI 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 6.93 cm
F1P 8.203 ppm
F1 4102.42 Hz
F2P 7.028 ppm
F2 3514.86 Hz
PPMCM 0.05874 ppm/cm
HZCM 29.37802 Hz/cm

¹³CNMR in DMSO at 298 K



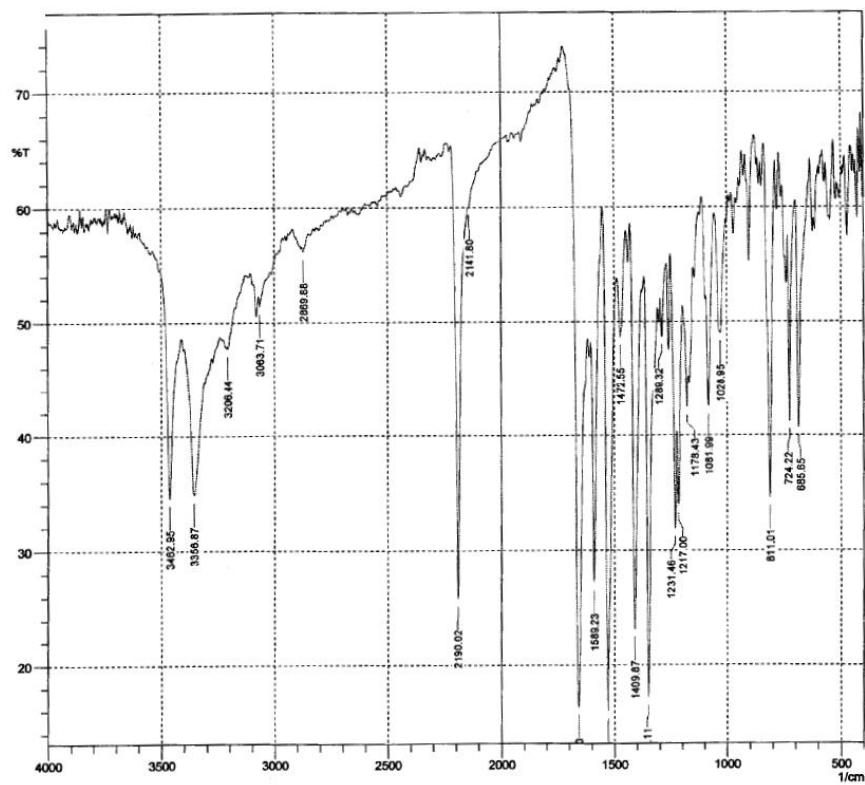
```
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 32768
SOLVENT DMSO
NS 746
DS 4
SMH 30030.029 Hz
FIDRES 0.916444 Hz
AQ 0.5456539 sec
RG 5792.5
DM 16.650 usec
DE 6.50 usec
TE 294.3 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
```

```
----- CHANNEL f1 -----
NUC1 13C
P1 9.10 usec
PL1 3.00 dB
SFO1 125.7703643 MHz
```

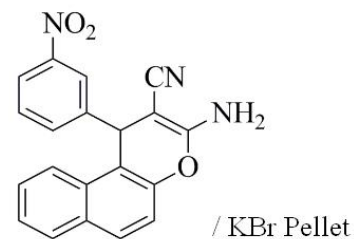
```
----- CHANNEL f2 -----
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 15.50 dB
PL13 15.50 dB
SFO2 500.1320005 MHz
```

```
F2 - Processing parameters
SI 32768
SF 125.7577390 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
```

```
1D NMR plot parameters
CX 20.00 cm
CY 14.88 cm
F1P 210.626 ppm
F1 26487.85 Hz
F2P 9.537 ppm
F2 1199.41 Hz
PPMCM 10.05443 ppm/cm
HZCM 1264.42236 Hz/cm
```

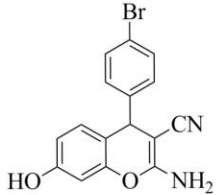



685.65	40.691	19.27	697.22	655.75	12.063	2.733
724.22	41.229	16.708	731.94	701.08	8.538	1.428
811.01	34.546	30.026	836.08	788.83	14.009	5.042
1028.95	48.976	10.168	1054.03	1006.77	12.737	1.963
1081.99	42.604	11.528	1091.63	1054.99	10.671	1.414
1178.43	42.448	4.175	1196.75	1172.64	7.925	0.274
1217	33.954	9.23	1223.75	1197.71	9.843	1.006
1231.46	31.892	12.268	1250.75	1224.71	9.878	1.35
1289.32	48.687	4.081	1296.08	1270.04	7.385	0.319
1349.11	17.243	35.756	1367.44	1308.61	24.934	8.451
1409.87	23.114	33.183	1429.15	1379.97	20.083	7.521
1472.55	48.726	6.67	1487.98	1448.44	11.099	1.102
1528.48	18.039	41.679	1550.66	1488.94	26.492	11.291
1589.23	27.311	23.887	1602.74	1551.63	18.049	4.271
1657.7	16.349	43.544	1693.38	1618.17	31.32	13.644
2141.8	60.034	0.161	2143.73	2102.26	8.676	0.009
2190.02	25.851	37.426	2218.95	2143.73	22.77	7.46
2869.88	56.2	0.705	2877.6	2845.77	7.783	0.07
3063.71	51.43	1.128	3070.46	3038.64	8.883	0.114
3206.44	47.734	0.339	3208.36	3175.58	10.049	0.066
3356.87	34.875	11.48	3398.34	3309.62	34.503	4.767
3462.95	34.626	17.432	3502.49	3409.91	61.781	5.101

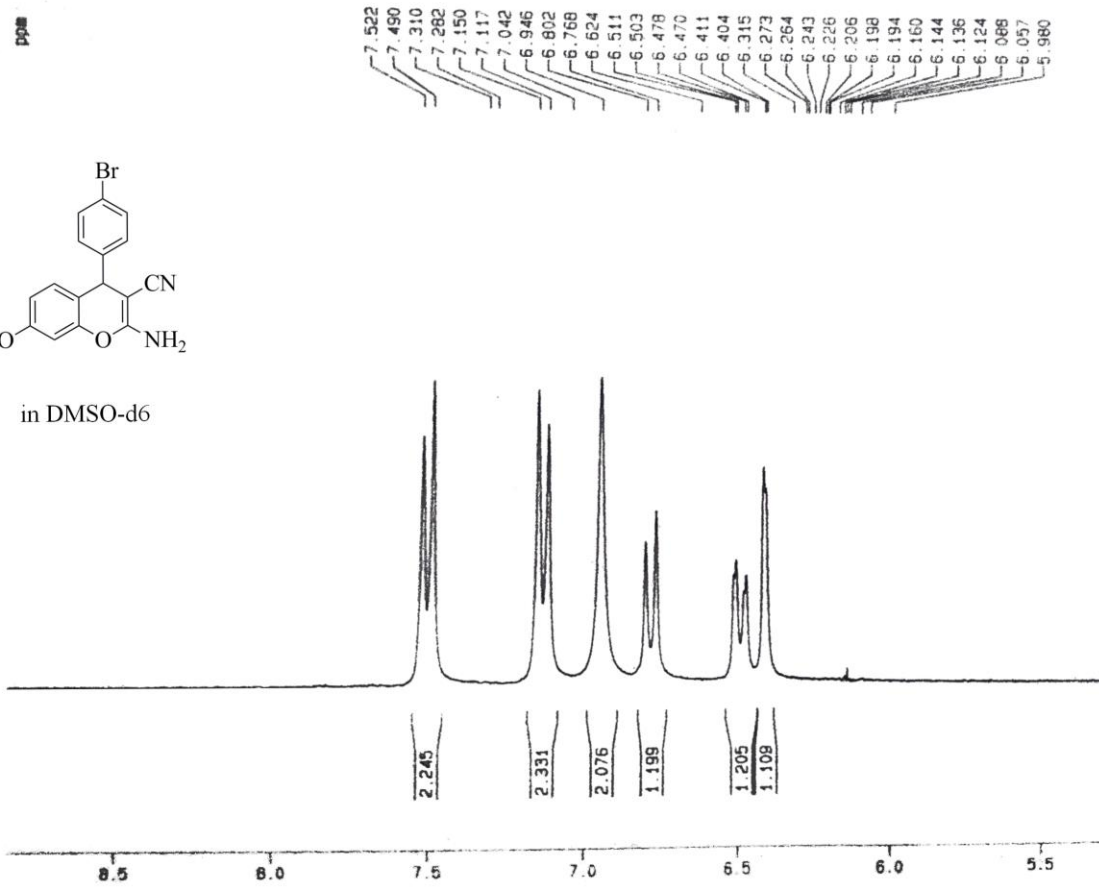


Comment;

No. of Scans;
Resolution;



in DMSO-d6



F2 - Acquisition Parameters
 Date_ 20131203
 Time 9.45
 INSTRUM spect
 PROBRD 5 mm QNP 1H/1
 PULPROG zg
 TD 65536
 SOLVENT DMSO
 NS 18
 DS 2
 SSB 6265.864 Hz
 FIDRES 0.095606 Hz
 AQ 5.2298226 sec
 RG 40.3
 DW 79.800 usec
 DE 6.00 usec
 TE 300.0 K
 SI 1.0000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 10.50 usec
 PL1 -4.00 dB
 SFO1 250.1300024 MHz
 F2 - Processing parameters
 SI 32768
 SF 250.1300024 MHz
 NSW 136
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 6.828 ppm
 F1 2208.23 Hz
 F2P 6.290 ppm
 F2 1329.21 Hz
 PPMCH 0.17691 ppm/cm
 -QCN 44.25126 Hz/cm