Supporting Information

Brønsted acidic ionic liquid catalyzed tandem reaction of 4-hydroxy-1-methyl-2-quinolone with chalcone: regioselective synthesis of pyrano[3,2-*c*]quinolin-2-ones

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EXPERIMENTAL

General: ^{1}H NMR (400 MHz) and ^{13}C NMR (100 MHz) spectra were run in $CDCl_3$ & DMSO-d_6 solutions (Bruker 400). Chemical shift were recorded as δ values in parts per million (ppm), and the signals were reported as s (singlet), d (doublet), t (triplet), m (multiplet) and coupling constants *J* were given in Hz. TLC was done on silica gel coated glass slide (Merck, Silica gel G for TLC). 4-hydroxy-1-methyl-2-quinolone was purchased from TCI and the unsaturated ketones were prepared by following literature report.

General procedure for the synthesis of *α,β*-unsaturated ketones:

The substituted acetophenone (0.05 mmol) and NaOH (0.01 mmol) were dissolved in 15 mL ethanol, and taken in a 50 mL round bottom of flask equipped with stirrer. The reaction was agitated at 0-5 ^{0}C; then the aldehyde was dissolved in 10 mL ethanol and added dropwise for 30 minutes, then the reaction was allowed to continue for 5 h at room temperature. The residual mass was quenched in the ice-water mixture and neutralized with 10% HCl solution. Then chromatography separation was followed by recrystallization from 95% ethanol (rectified spirit).

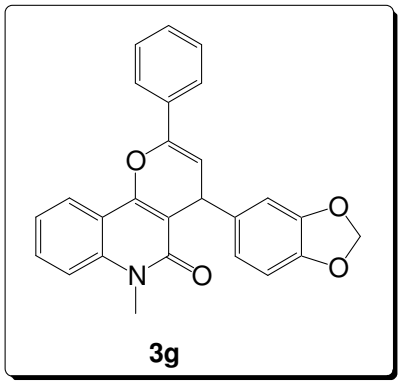
General procedure for the synthesis of pyrano[3,2-*c*]quinolin-2-ones:

To a mixture of 4-hydroxy-1-methyl-2-quinolone (1 mmol) and *α,β*-unsaturated ketone (1 mmol), the 2 mol% acidic ionic liquid was added and the mixture was stirred at 110 ºC for 6h (TLC). After completion of the reaction, water was added to the reaction mixture. Then the product was filtered off and the ionic liquid was recovered by evaporating the water. The recovered ionic liquid was reused for a subsequent fresh batch of the reaction after reactivation. The crude product was recrystallized from hot ethanol to afford the pure product.

Single crystal XRD data of 4-Benzo[1,3]dioxol-5-yl-6-methyl-2-phenyl-4,6-dihydro-pyrano[3,2-*c*]quinolin-5-one (3g):

Single crystal suitable for X-ray diffraction of (**3g**) was grown from ethanol. The crystals were carefully chosen using a stereo zoom microscope supported by a rotatable polarizing stage. The data was collected at room temperature on Bruker-APEX II SMART CCD diffractometer with graphite monochromated Mo-Kα radiation (0.71073 Å).

Crystal data of 3g:



3g

Molecular formula = $C_{26}H_{19}NO_4$, Formula weight = 409.43, Crystal system = Triclinic, space group = P-1, a = 7.6235(9)Å, b = 10.5395(12)Å, c = 13.1263(16)Å, V = 976.7(2)Å^3, T = 296 K, Dx = 1.392g cm^{-3}, Mu (Mo-Kα) = 0.094mm^{-1}, 2162 reflections measured, R_1_obs = 0.0464. Crystallographic data (excluding structure factors) for **3g** have been deposited with the Cambridge Crystallographic Data Center as supplementary publication number CCDC 964212.

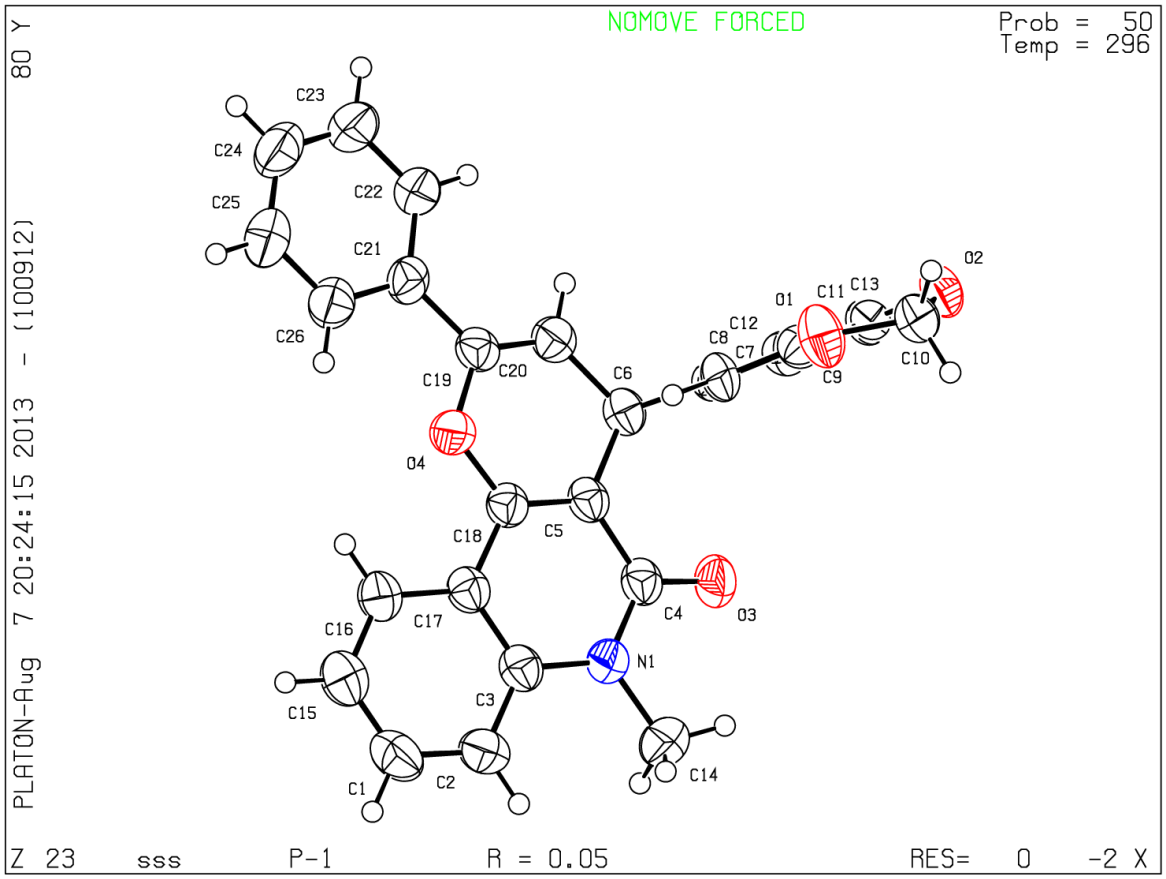
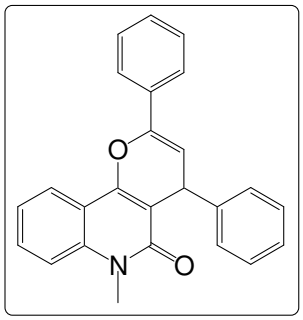


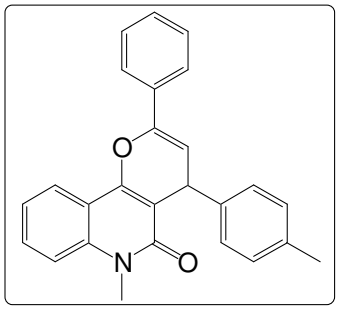
Figure 1: ORTEP view of the compound **3g.**

Characterization data of compounds (3):



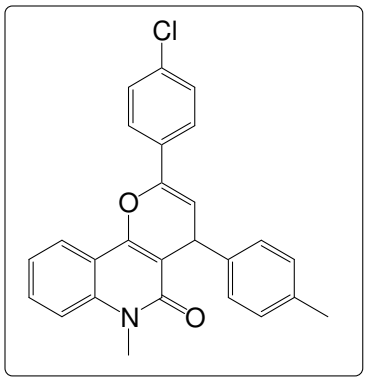
6-Methyl-2,4-diphenyl-4,6-dihydro-pyrano[3,2-*c*]quinolin-5-one (3a):

Isolated yield: 81%; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.23 (d, *J* = 8.4 Hz, 1H), 7.80 (d, *J* = 8.4 Hz, 2H), 7.63-7.59 (m, 1H), 7.49-7.32 (m, 9H), 7.25-7.21 (m, 1H), 5.86 (d, *J* = 5.2 Hz, 1H), 4.87 (d, *J* = 4.8 Hz, 1H), 3.65 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 162.0, 152.7, 146.9, 145.0, 139.0, 133.3, 130.8, 128.9, 128.6, 128.5, 126.8, 124.7, 123.0, 122.0, 114.9, 114.1, 108.9, 103.7, 37.3, 29.5. Anal. Calcd. For $C_{25}H_{19}NO_2$: C, 82.17; H, 5.24; N, 3.83%; Found: C, 82.10; H, 5.18; N, 3.76%.



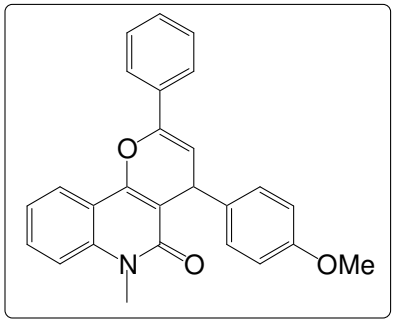
6-Methyl-2-phenyl-4-p-tolyl-4,6-dihydro-pyrano[3,2-*c*]quinolin-5-one (3b):

Isolated yield: 80%; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.23 (d, *J* = 8.4 Hz, 1H), 7.80 (d, *J* = 7.2 Hz, 2H), 7.60 (m, 1H), 7.49-7.34 (m, 7H), 7.14 (d, *J* = 8.0 Hz, 2H), 5.85 (d, *J* = 4.8 Hz, 1H), 4.83 (d, *J* = 5.2 Hz, 1H), 3.66 (s, 3H), 2.32 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 162.1, 152.7, 146.7, 142.1, 139.0, 136.4, 133.4, 130.8, 129.3, 128.9, 128.6, 128.4, 124.7, 123.0, 122.0, 115.0, 114.1, 109.0, 103.9, 36.9, 29.5, 21.2. Anal. Calcd. For $C_{26}H_{21}NO_2$: C, 82.30; H, 5.58; N, 3.69%; Found: C, 82.20; H, 5.48; N, 3.66%.



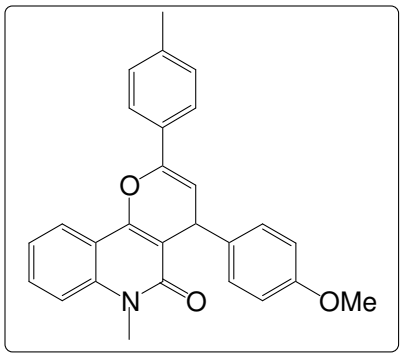
2-(4-Chloro-phenyl)-6-methyl-4-p-tolyl-4,6-dihydro-pyrano[3,2-*c*]quinolin-5-one (3c):

Isolated yield: 79%; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.21 (d, *J* = 7.6 Hz, 1H), 7.95 (d, *J* = 8.0 Hz, 1H), 7.68-7.58 (m, 2H), 7.39-7.26 (m, 8H), 5.74 (d, *J* = 4.8 Hz, 1H), 4.81 (d, *J* = 4.4 Hz, 1H), 3.66 (s, 3H), 2.42 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 162.0, 152.9, 147.3, 143.9, 143.7, 139.1, 133.6, 132.5, 131.0, 129.9, 129.4, 128.6, 124.7, 123.1, 122.1, 114.9, 114.2, 108.5, 102.4, 36.8, 29.5, 21.4. Anal. Calcd. For $C_{26}H_{20}ClNO_2$: C, 75.45; H, 4.87; N, 3.38%; Found: C, 75.36; H, 4.78; N, 3.36%.



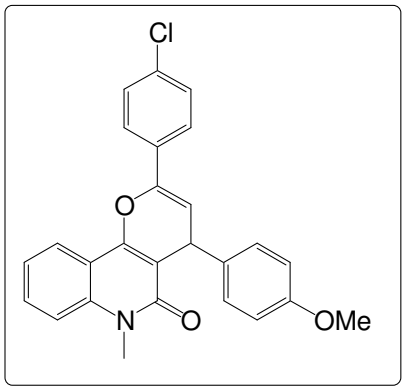
4-(4-Methoxy-phenyl)-6-methyl-2-phenyl-4,6-dihydro-pyrano[3,2-*c*]quinolin-5-one (3d):

Isolated yield: 79%; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.21 (d, *J* = 8.0 Hz, 1H), 7.79 (d, *J* = 7.2 Hz, 2H), 7.61 (m, 1H), 7.48-7.34 (m, 7H), 6.86 (d, *J* = 8.4 Hz, 2H), 5.84 (d, *J* = 4.8 Hz, 1H), 4.80 (d, *J* = 4.8 Hz, 1H), 3.78 (s, 3H), 3.66 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 162.1, 158.4, 152.5, 146.8, 139.0, 137.3, 133.4, 130.8, 129.6, 128.9, 128.6, 124.7, 123.0, 122.0, 115.0, 114.1, 113.9, 109.1, 103.9, 55.3, 36.4, 29.5. Anal. Calcd. For $C_{26}H_{21}NO_3$: C, 78.97; H, 5.35; N, 3.54%; Found: C, 78.88; H, 5.19; N, 3.46%.



4-(4-Methoxy-phenyl)-6-methyl-2-*p*-tolyl-4,6-dihydro-pyrano[3,2-*c*]quinolin-5-one (3e):

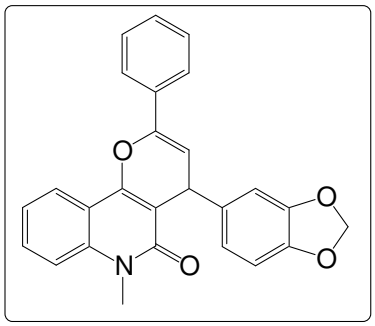
Isolated yield: 78%; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.21 (d, *J* = 8.0 Hz, 1H), 7.68 (d, *J* = 8.0 Hz, 2H), 7.61 (t, *J* = 8.0 Hz, 1H), 7.39-7.33 (m , 4H), 7.27 (d, *J* = 8.0 Hz, 2H), 6.85 (d, *J* = 8.8 Hz, 2H), 5.78 (d, *J* = 4.8 Hz, 1H), 4.80 (d, *J* = 5.2 Hz, 1H), 3.77 (s, 3H), 3.66 (s, 3H), 2.42 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 162.2, 158.4, 152.6, 146.9, 139.0, 138.9, 137.4, 130.8, 130.7, 129.6, 129.3, 124.6, 123.0, 122.0, 115.0, 114.1, 113.9, 109.2, 103.0, 55.3, 36.4, 29.5, 21.4. Anal. Calcd. For $C_{27}H_{23}NO_3$: C, 79.20; H, 5.66; N, 3.42%; Found: C, 79.12; H, 5.51; N, 3.35%.



2-(4-Chloro-phenyl)-4-(4-methoxy-phenyl)-6-methyl-4,6-dihydro-pyrano[3,2-*c*]quinolin-5-one (3f):

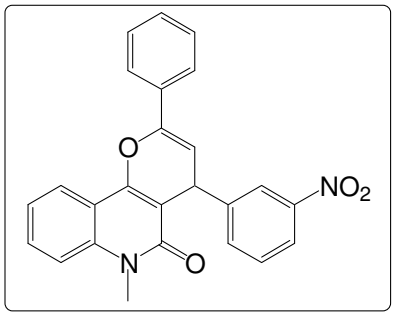
Isolated yield: 80%; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.17 (d, *J* = 7.2 Hz, 1H), 7.70 (d, *J* = 8.4 Hz, 2H), 7.64-7.60 (M, 1H), 7.43-7.34 (m, 6H), 6.85 (d, *J* = 8.4 Hz, 2H), 5.82 (d, *J* = 5.2 Hz, 1H), 4.78 (d, *J* = 4.8 Hz, 1H), 3.77 (s, 3H), 3.66 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 162.0, 158.5, 152.4, 145.9, 139.0, 137.0, 134.7, 131.9, 130.9, 129.5, 128.8, 126.0, 122.9, 122.1, 114.8, 114.2,

114.0, 109.1, 104.4, 55.3, 36.4, 29.5. Anal. Calcd. For $C_{26}H_{20}ClNO_3$: C, 72.64; H, 4.69; N, 3.26%; Found: C, 72.51; H, 4.50; N, 3.16%.



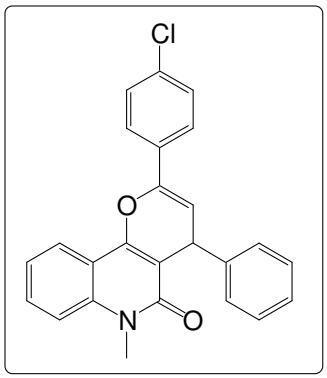
4-Benzo[1,3]dioxol-5-yl-6-methyl-2-phenyl-4,6-dihydro-pyrano[3,2-*c*]quinolin-5-one (3g):

Isolated yield: 86%; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.21 (d, *J* = 7.6 Hz, 1H), 7.78 (d, *J* = 7.2 Hz, 2H), 7.63 (t, *J* = 7.2 Hz, 1H), 7.46-7.34 (m, 5H), 6.94-6.91 (m, 2H), 6.76 (d, *J* = 7.6 Hz, 1H), 5.90 (s, 2H), 5.81 (d *J* = 4.0 Hz, 1H). 4.77 (d, *J* = 4.4 Hz, 1H), 3.67 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 162.1, 152.7, 147.8, 146.8, 146.4, 139.2, 139.0, 133.3, 130.9, 129.0, 128.6, 124.7, 123.0, 122.1, 121.6, 114.9, 114.1, 109.0, 108.8, 108.3, 103.8, 101.0, 36.9, 29.5. Anal. Calcd. For $C_{26}H_{19}NO_4$: C, 76.27; H, 4.68; N, 3.42%; Found: C, 76.20; H, 4.59; N, 3.35%.



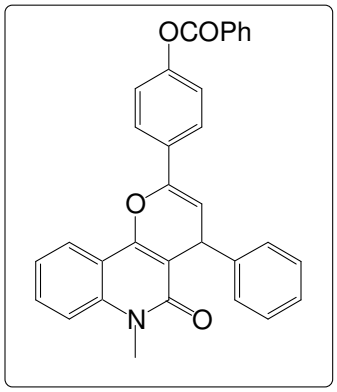
6-Methyl-4-(3-nitro-phenyl)-2-phenyl-4,6-dihydro-pyrano[3,2-*c*]quinolin-5-one (3h):

Isolated yield: 84%; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.25-8.22 (m, 2H), 8.07 (d, *J* = 8.0 Hz, 1H), 7.82-7.78 (m, 3H), 7.68-7.60 (m, 1H), 7.50-7.28 (m, 6H), 5.77 (d, *J* = 4.4 Hz, 1H), 4.96 (d, *J* = 4.4 Hz, 1H), 3.64 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 161.8, 153.3, 148.6, 147.6, 147.2, 139.2, 134.8, 132.9, 131.4, 129.4, 129.3, 128.7, 124.8, 123.4, 123.2, 122.3, 122.0, 114.6, 114.3, 107.5, 102.3, 37.3, 29.6. Anal. Calcd. For $C_{25}H_{18}N_2O_4$: C, 73.16; H, 4.42; N, 6.83%; Found: C, 73.11; H, 4.31; N, 6.74%.



2-(4-Chloro-phenyl)-6-methyl-4-phenyl-4,6-dihydro-pyrano[3,2-*c*]quinolin-5-one (3i):

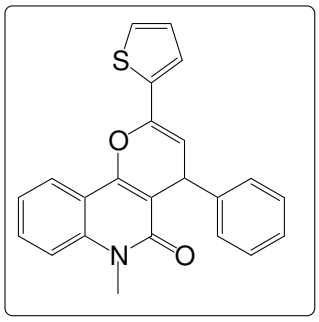
Isolated yield: 82%; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.21 (d, *J* = 7.2 Hz, 1H), 7.78 (d, *J* = 7.2 Hz, 2H), 7.65-7.61 (m, 1H), 7.49-7.35 (m, 7H), 7.28 (d, *J* = 8.4 Hz, 2H), 5.79 (d, *J* = 4.8 Hz, 1H), 4.83 (d, *J* = 4.8 Hz, 1H), 3.66 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 162.0, 152.8, 147.1, 143.5, 139.1, 133.2, 132.6, 131.1, 129.9, 129.1, 128.7, 124.8, 123.1, 122.1, 114.8, 114.2, 108.5, 103.2, 36.8, 29.5. Anal. Calcd. For $C_{25}H_{18}ClNO_2$: C, 75.09; H, 4.54; N, 3.50%; Found: C, 74.98; H, 4.43; N, 3.43%.



Benzoic acid 4-(6-methyl-5-oxo-4-phenyl-5,6-dihydro-4H-pyrano[3,2-*c*]quinolin-2-yl)-phenyl ester (3j):

Isolated yield: 83%; ^{1}H NMR (400 MHz, $CDCl_3$ + DMSO-d_6): δ 8.26-8.15 (m, 3H), 7.97 (d, *J* = 8.4 Hz, 2H), 7.79-7.73 (m, 2H), 7.65-7.58 (m, 3H), 7.45-7.40 (m, 3H), 7.34-7.27 (m, 4H), 7.20-7.17 (m, 1H), 6.17 (d, *J* = 5.2 Hz, 1H), 4.76 (d, *J* = 5.2 Hz, 1H), 3.58 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$ + DMSO-d_6): δ 164.4, 160.6, 152.0, 150.9, 145.2, 144.8, 138.6, 134.0, 131.2, 130.2, 129.8, 128.9, 128.7, 128.2, 128.0, 126.4, 125.6, 122.4, 122.2, 114.7, 113.7, 107.9, 103.8,

36.34, 29.1. Anal. Calcd. For $C_{32}H_{23}NO_4$: C, 79.16; H, 4.77; N, 2.88%; Found: C, 79.08; H, 4.62; N, 2.77%.



6-Methyl-4-phenyl-2-thiophen-2-yl-4,6-dihydro-pyrano[3,2-*c*]quinolin-5-one (3k):

Isolated yield: 70%; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.04 (d, *J* = 8.0 Hz, 1H),7.49-7.45 (m, 1H), 7.33-7.29 (m, 3H), 7.23-7.07 (m, 6H), 6.68-6.96 (m, 1H), 5.62 (d, *J* = 4.8 Hz, 1H), 4.68 (d, *J* = 5.2 Hz, 1H), 3.50 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 161.9, 152.5, 144.7, 143.0, 139.0, 136.9, 130.9, 128.5, 128.2, 127.6, 126.9, 125.5, 123.9, 123.0, 122.0, 114.6, 114.0, 108.8, 102.7, 37.1, 29.5. Anal. Calcd. For $C_{23}H_{17}NO_2S$: C, 74.37; H, 4.61; N, 3.77%; Found: C, 74.29; H, 4.55; N, 3.68%.

E-factor Calculations[1]

Table 1 Tandem reaction between 4-hydroxy-1-methyl-2-quinolone and chalcones: E-factor value for this protocol.[a]

Entry	4-hydroxy-1-methyl-2-quinolone (mmol)	Chalcones (mmol)	Product	Isolated yield (%)[b]	Time (h)	E factor (kg waste/kg product)[c]
1	1	Chalcone (1)	**3a**	81	6	0.29
2	1	Chalcone (1)	**3f**	80	6	0.30

[a] All reactions are performed with acidic ionic liquid (2 mol%) at110 ºC under solvent free conditions.

[b] Isolated yields.

[c] Exclusion of ethyl acetate used for work up procedure, exclusion of the amount of the acidic ionic liquid used, and exclusion of ingredients used for chromatography.

Note (Regarding Table 1, SI): When the Authors have not reported the amount of solvent used in the work-up procedure, we have not accounted for solvent and considered that solvent can be recovered. By considering the acidic ionic liquid catalyst is recyclable and hence, waste is essentially eliminated.

For Entry 1, Table1 (SI)

E = [0.175 g (4-hydroxy-1-methyl-2-quinolone) + 0.208 g (chalcone) – 0.296 g (product × yield)] / 0.296 g

= 0.29

For Entry 2, Table1 (SI)

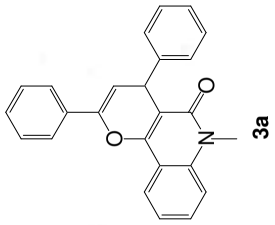
E = [0.175 g (4-hydroxy-1-methyl-2-quinolone) + 0.272 g (chalcone) – 0.343 g (product × yield)] / 0.343 g

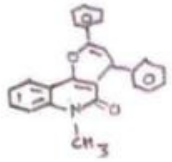
= 0.30

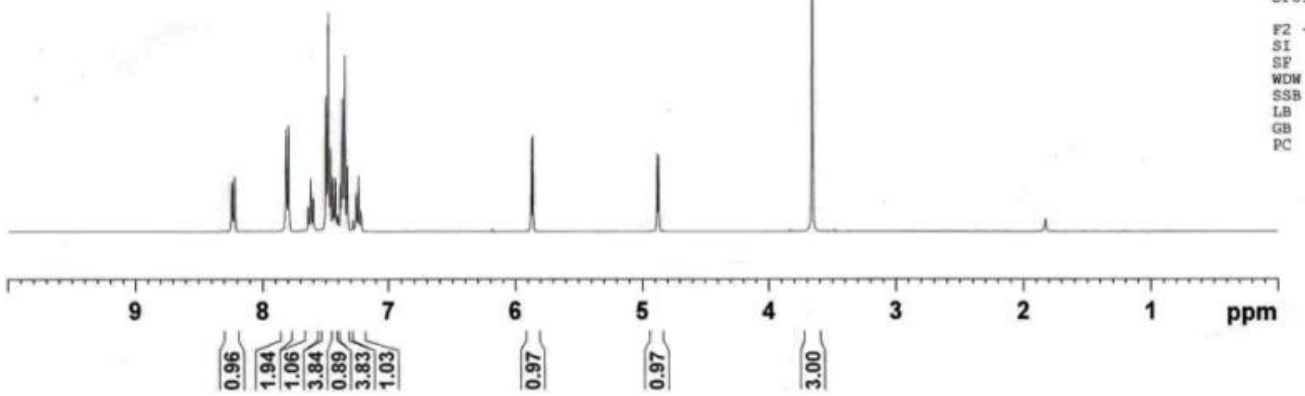
References:

1 A. Kamal, V. Srinivasulu, B. N. Seshadri, N. Markandeya, A. Alarifi and N. Shankaraiah, *Green Chem.*, 2012, **14**, 2513.

1H of VBAKB-783/13







BRUKER

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DE                6.50 usec
TE               296.6 K
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TD0                   1

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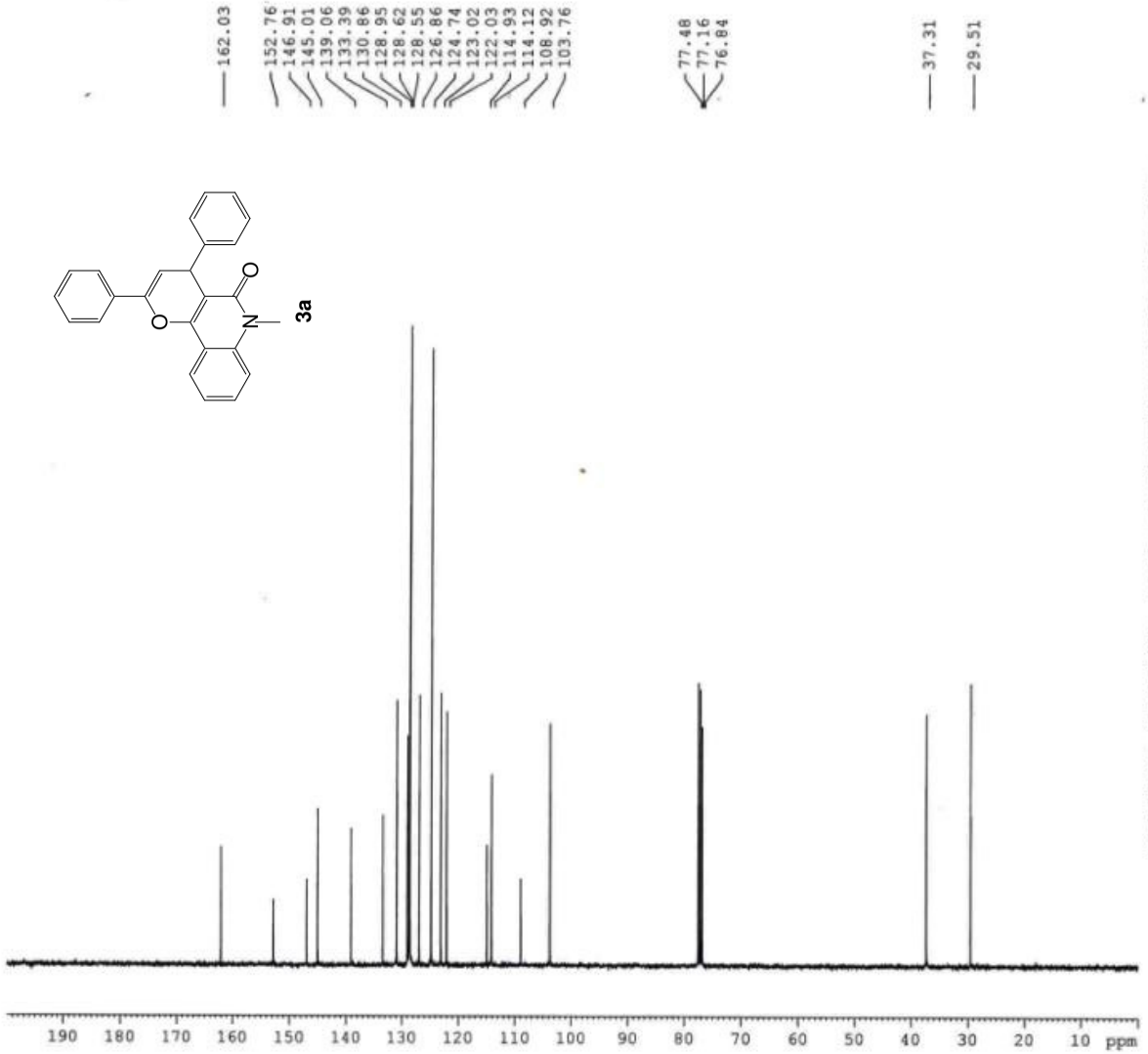

BRUKER

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PROCNO                  1

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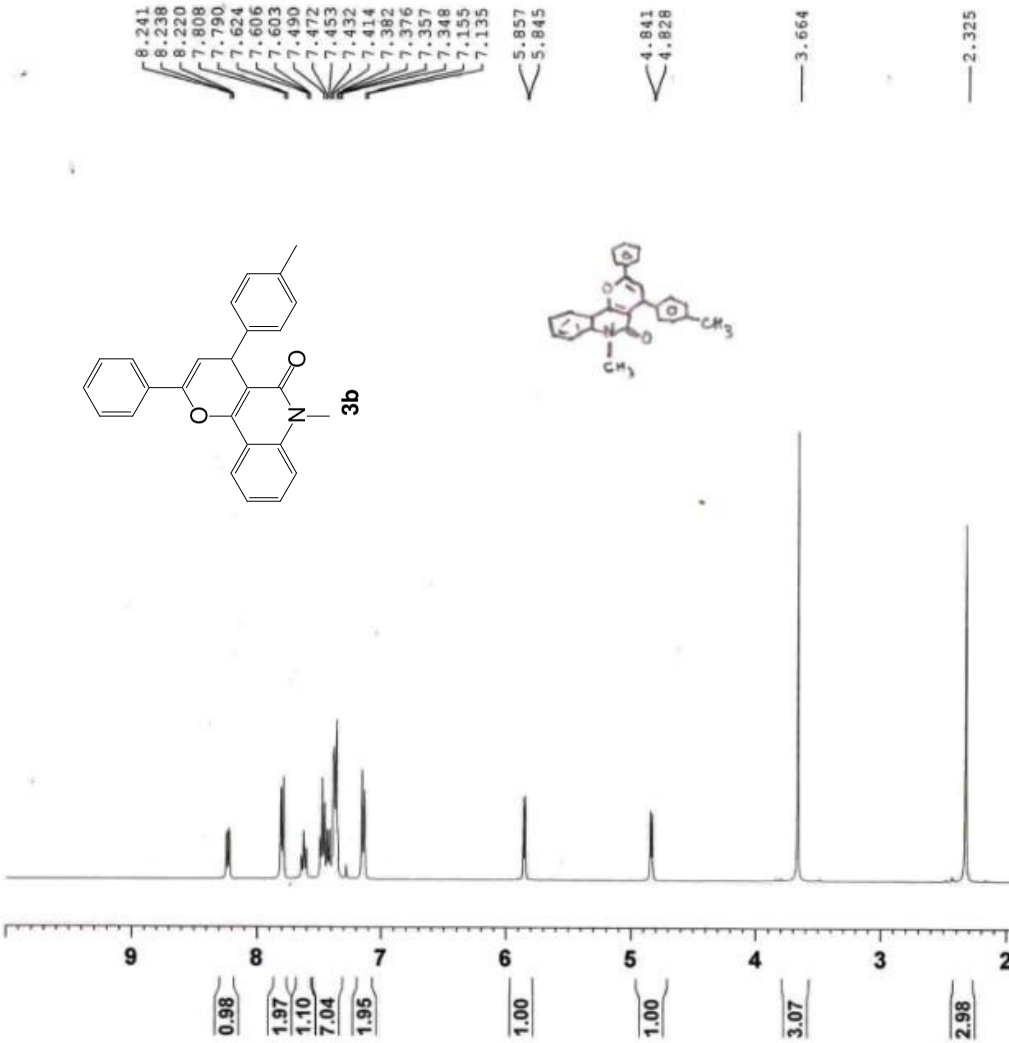
AQ             0.6816244 sec
RG                 47.25
DW                20.800 usec
DE                 6.50 usec
TE                297.5 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1                 8.90 usec
PLW1        54.00000000 W
SFO1       100.6278588 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2             80.00 usec
PLW2        12.00000000 W
PLW12        0.40792999 W
PLW13        0.26107001 W
SFO2       400.1516006 MHz

F2 - Processing parameters
SI                 16384
SF         100.6177939 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

1H of VBAKB-789/13



BRUKER

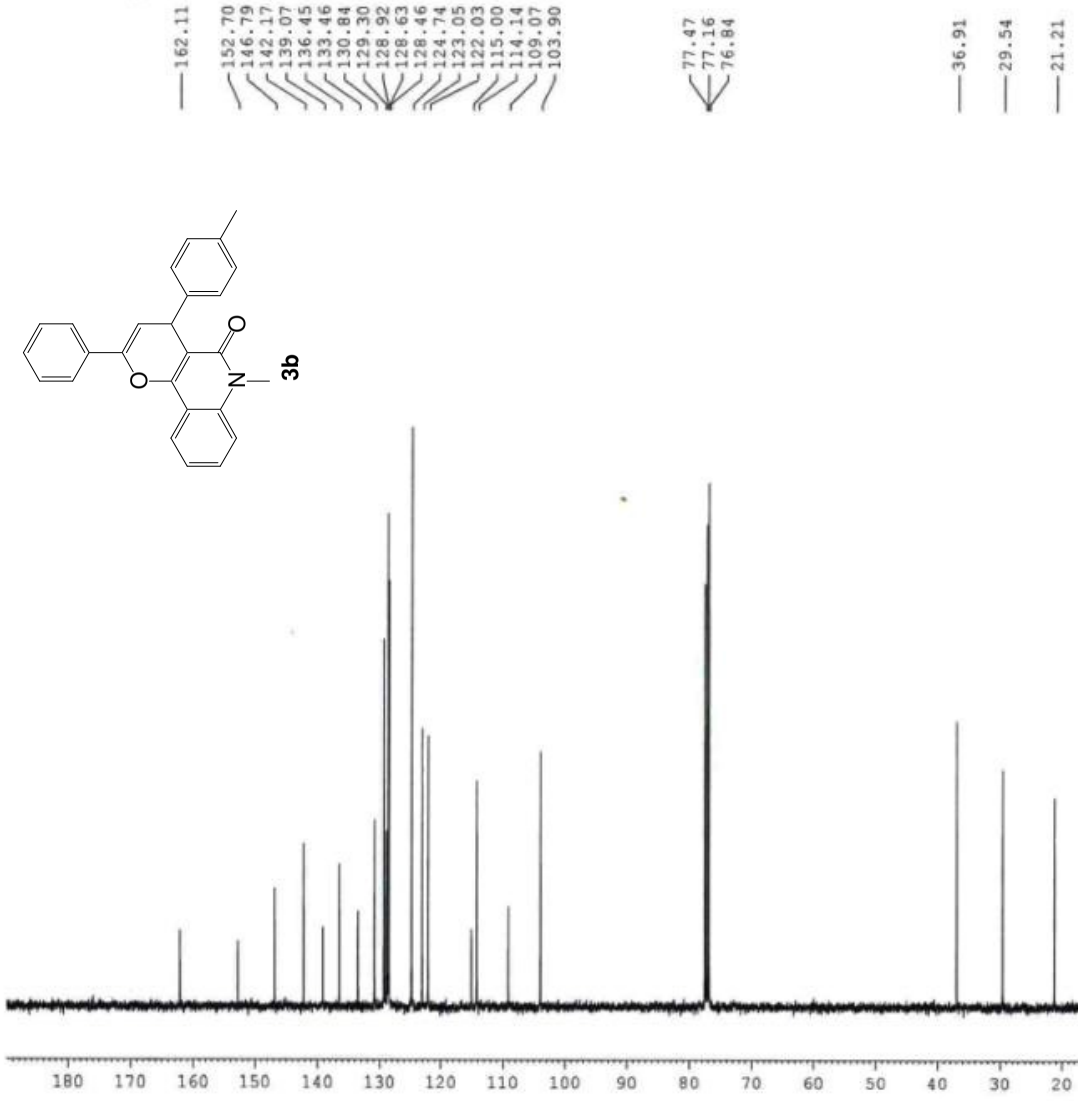
```
Current Data Parameters
NAME          Dr. A MAJEE
EXPNO               753
PROCNO                1

F2 - Acquisition Parameters
Date_          20130527
Time              20.33
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG           zg30
TD               32768
SOLVENT          CDCl3
NS                  24
DS                   2
SWH           8223.685 Hz
FIDRES        0.250967 Hz
AQ            1.9923444 sec
RG                57.28
DW               60.800 usec
DE                6.50 usec
TE               296.2 K
D1            1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                 1H
P1                14.75 usec
PLW1         11.99499989 W
SFO1         400.1524711 MHz

F2 - Processing parameters
SI                16384
SF          400.1500000 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

13C of VBAKB-789



BRUKER

```
Current Data Parameters
NAME            Dr. A MAJEE
EXPNO                   785
PROCNO                    1

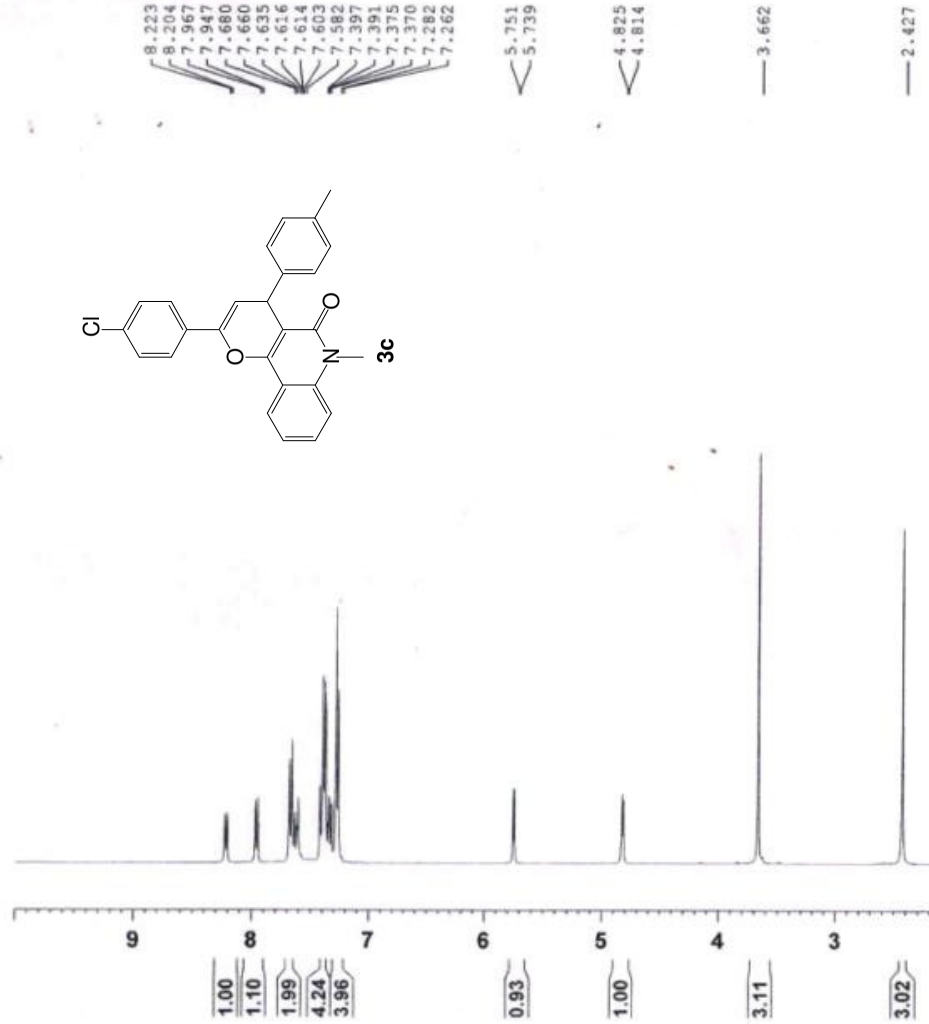
F2 - Acquisition Parameters
Date_            20130531
Time                21.36
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                 32768
SOLVENT            CDCl3
NS                   320
DS                     2
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ             0.6816244 sec
RG                 57.28
DW                20.800 usec
DE                 6.50 usec
TE                295.1 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                13C
P1                 8.90 usec
PLW1        54.00000000 W
SFO1       100.6278588 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2             80.00 usec
PLW2        12.00000000 W
PLW12        0.40792999 W
PLW13        0.26107001 W
SFO2       400.1516006 MHz

F2 - Processing parameters
SI                 16384
SF         100.6177909 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

1H of VBAKB-794/13



BRUKER

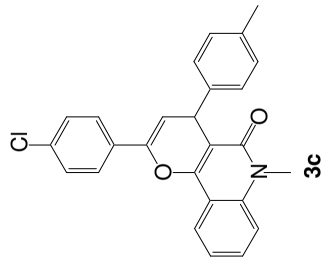
Current Data Parameters
NAME Dr. A MAJEE
EXPNO 836
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130609
Time 13.16
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 2
SWH 8223.685 Hz
FIDRES 0.250967 Hz
AQ 1.9923444 sec
RG 67.81
DW 60.800 usec
DE 6.50 usec
TE 293.7 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 14.75 usec
PLW1 11.99499989 W
SFO1 400.1524711 MHz

F2 - Processing parameters
SI 16384
SF 400.1500000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

13C of VBAKB-794



BRUKER

Current Data Parameters
NAME Dr. A MAJEE
EXPNO 858
PROCNO 1

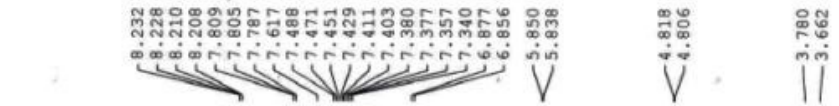
F2 - Acquisition Parameters
Date 20130611
Time 12.50
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 512
DS 2
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6816244 sec
RG 67.81
DW 20.800 usec
DE 6.50 usec
TE 296.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

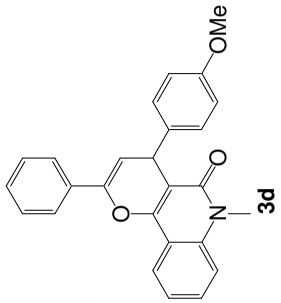
======== CHANNEL f1 ========
NUC1 13C
P1 8.90 usec
PLW1 54.00000000 W
SFO1 100.6278588 MHz

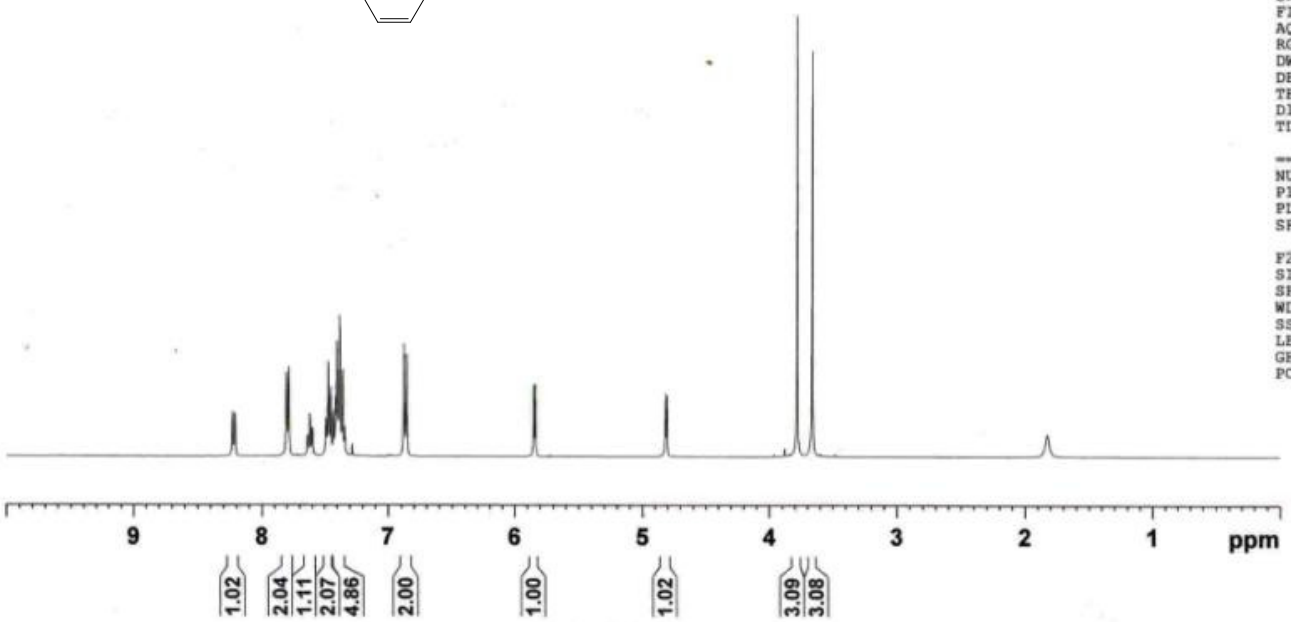
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PLW2 12.00000000 W
PLW12 0.40792999 W
PLW13 0.26107001 W
SFO2 400.1516006 MHz

F2 - Processing parameters
SI 16384
SF 100.6177866 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1H of VBAKB-788







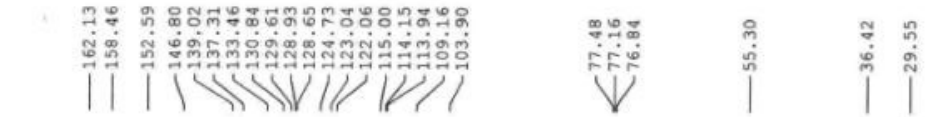
Current Data Parameters
NAME Dr. A MAJEE
EXPNO 752
PROCNO 1

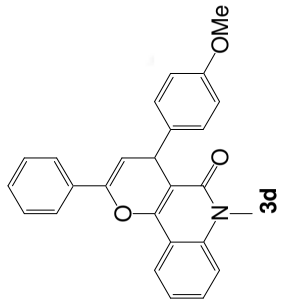
F2 - Acquisition Parameters
Date_ 20130527
Time 20.29
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 24
DS 2
SWH 8223.685 Hz
FIDRES 0.250967 Hz
AQ 1.9923444 sec
RG 57.28
DW 60.800 usec
DE 6.50 usec
TE 296.3 K
D1 1.00000000 sec
TD0 1

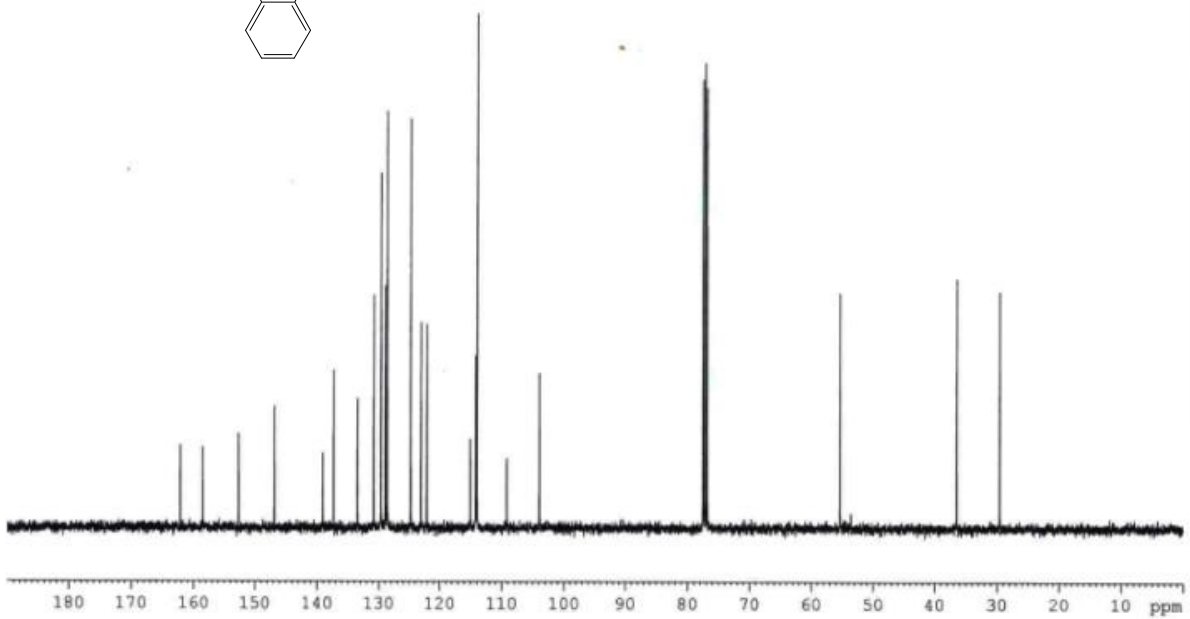
======== CHANNEL f1 ========
NUC1 1H
P1 14.75 usec
PLW1 11.99499989 W
SFO1 400.1524711 MHz

F2 - Processing parameters
SI 16384
SF 400.1500000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

13C of VBAKB-788







BRUKER

```
Current Data Parameters
NAME          Dr. A MAJEE
EXPNO               784
PROCNO                1

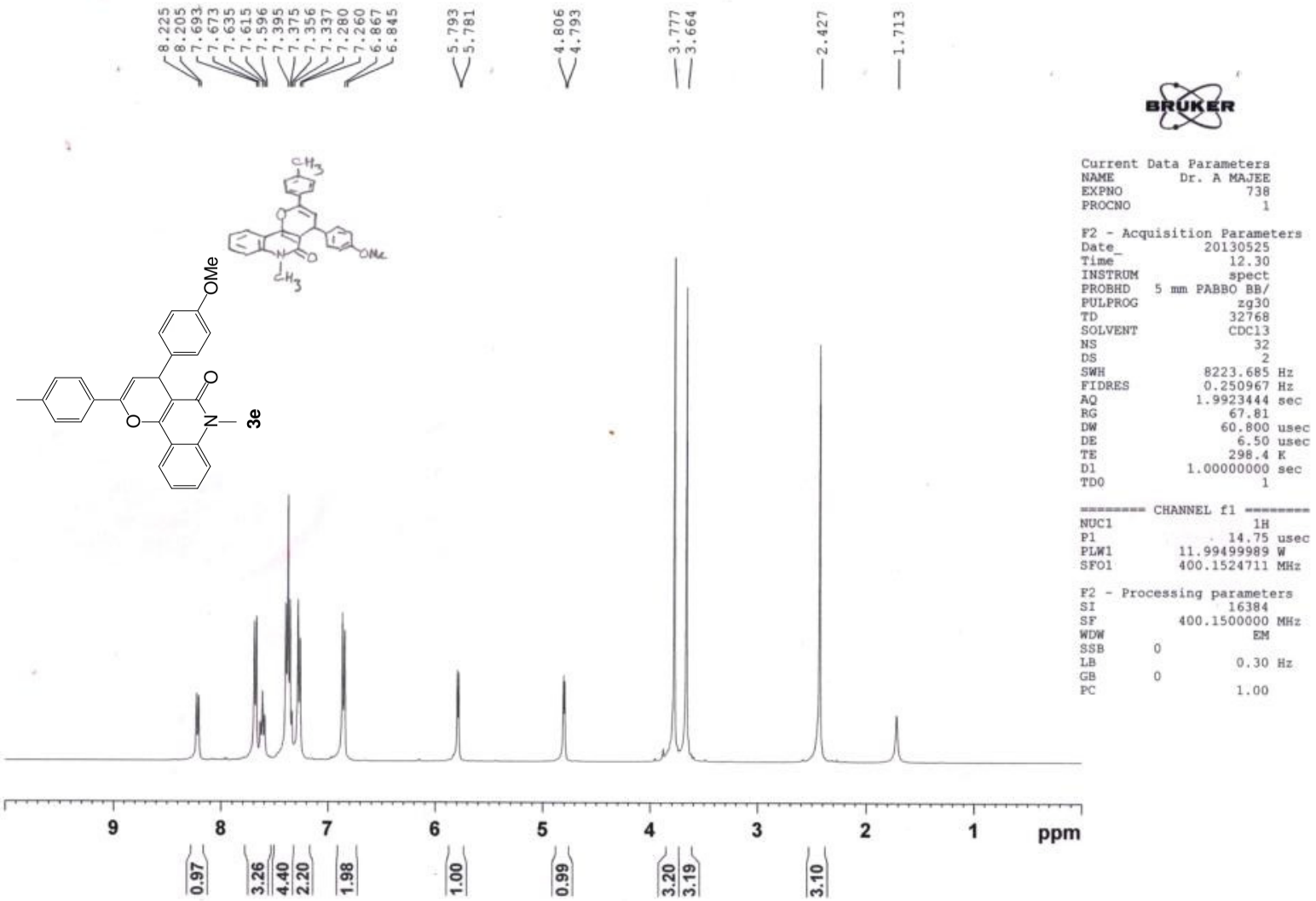
F2 - Acquisition Parameters
Date_          20130531
Time              21.19
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD               32768
SOLVENT          CDCl3
NS                 209
DS                   2
SWH          24038.461 Hz
FIDRES        0.733596 Hz
AQ            0.6816244 sec
RG                57.28
DW               20.800 usec
DE                6.50 usec
TE               295.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1               13C
P1                8.90 usec
PLW1        54.00000000 W
SFO1       100.6278588 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2               1H
PCPD2            80.00 usec
PLW2        12.00000000 W
PLW12        0.40792999 W
PLW13        0.26107001 W
SFO2       400.1516006 MHz

F2 - Processing parameters
SI                16384
SF         100.6177910 MHz
WDW                EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

1H of VBAKB-786/13



BRUKER

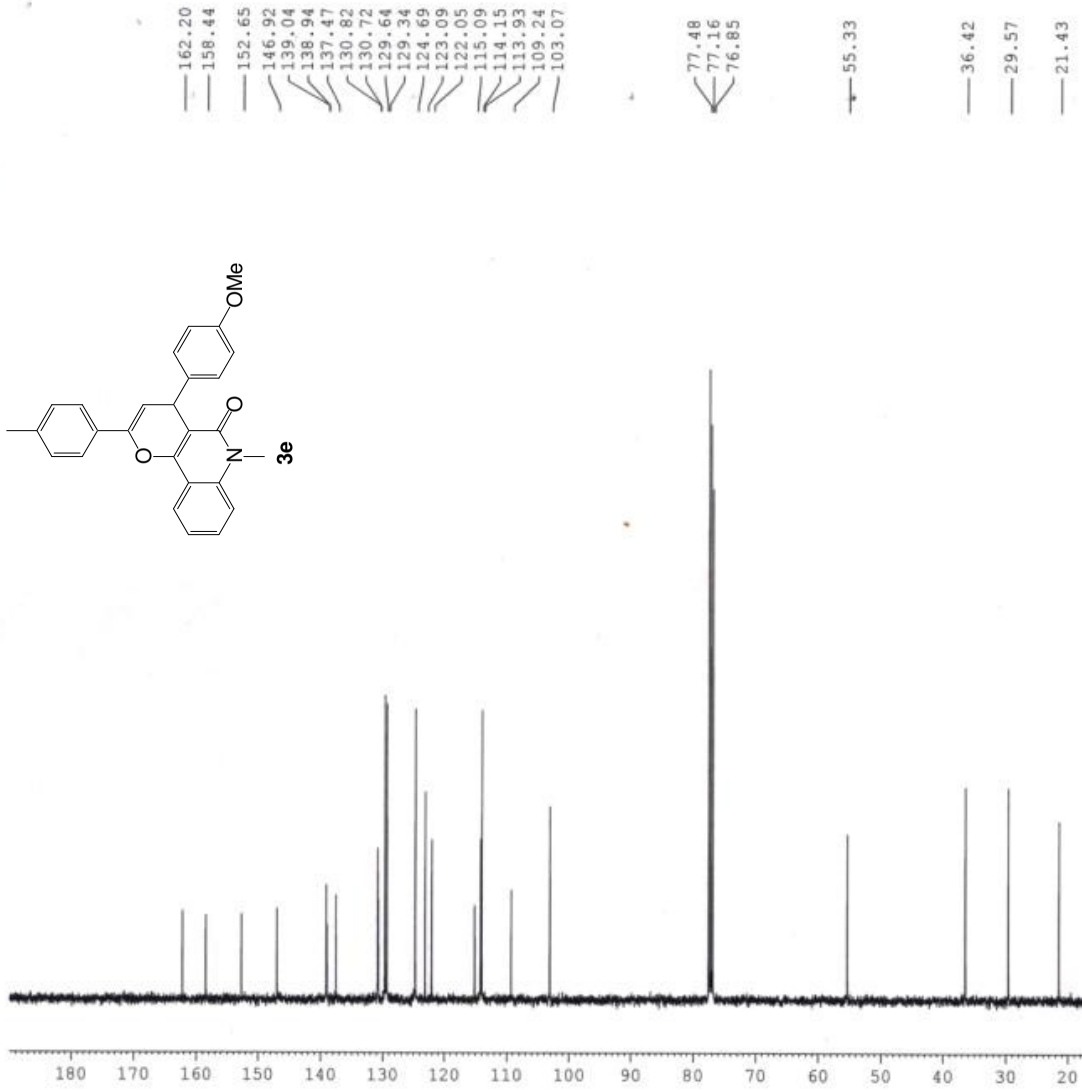
```
Current Data Parameters
NAME         Dr. A MAJEE
EXPNO              738
PROCNO               1

F2 - Acquisition Parameters
Date_          20130525
Time              12.30
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG           zg30
TD               32768
SOLVENT          CDC13
NS                  32
DS                   2
SWH            8223.685 Hz
FIDRES        0.250967 Hz
AQ            1.9923444 sec
RG                67.81
DW               60.800 usec
DE                6.50 usec
TE               298.4 K
D1            1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                1H
P1                14.75 usec
PLW1        11.99499989 W
SFO1       400.1524711 MHz

F2 - Processing parameters
SI                16384
SF         400.1500000 MHz
WDW                EM
SSB       0
LB               0.30 Hz
GB        0
PC               1.00
```

13C of VBAKB-786/1



BRUKER

```
Current Data Parameters
NAME          Dr. A MAJEE
EXPNO                 749
PROCNO                  1

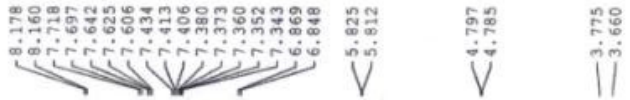
F2 - Acquisition Parameters
Date_           20130525
Time              20.14
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD                32768
SOLVENT           CDCl3
NS                  400
DS                    2
SWH           24038.461 Hz
FIDRES        0.733596 Hz
AQ            0.6816244 sec
RG                67.81
DW               20.800 usec
DE                6.50 usec
TE               295.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

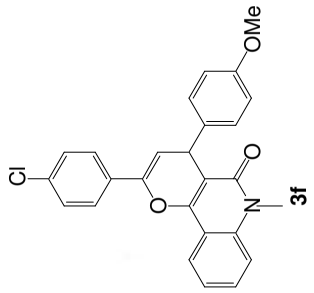
======== CHANNEL f1 ========
NUC1                 13C
P1                 8.90 usec
PLW1        54.00000000 W
SFO1       100.6278588 MHz

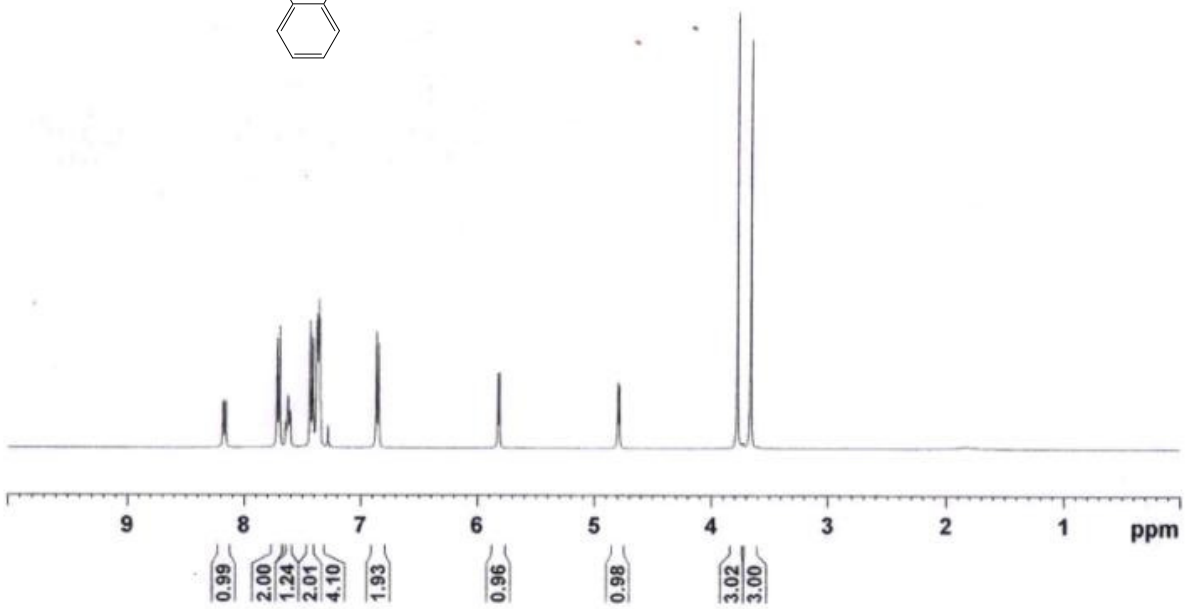
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             80.00 usec
PLW2        12.00000000 W
PLW12        0.40792999 W
PLW13        0.26107001 W
SFO2       400.1516006 MHz

F2 - Processing parameters
SI                16384
SF         100.6177880 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

1H of VBAKB-784/13







BRUKER

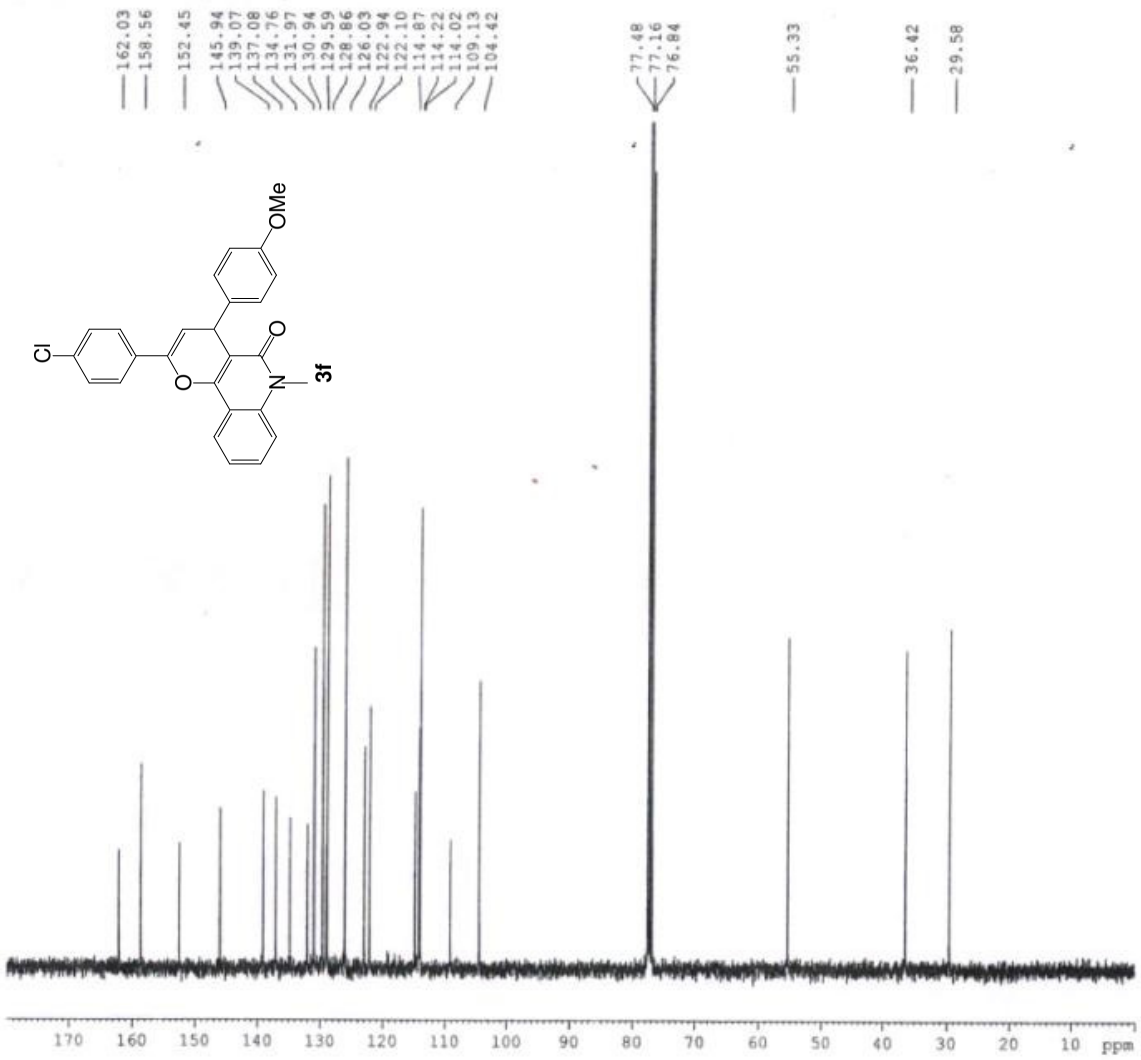
```
Current Data Parameters
NAME          Dr. A MAJEE
EXPNO               732
PROCNO                1

F2 - Acquisition Parameters
Date_          20130523
Time              16.19
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG           zg30
TD               32768
SOLVENT          CDCl3
NS                   24
DS                    2
SWH           8223.685 Hz
FIDRES        0.250967 Hz
AQ            1.9923444 sec
RG                77.59
DW                60.800 usec
DE                 6.50 usec
TE               295.8 K
D1            1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                14.75 usec
PLW1        11.99499989 W
SFO1        400.1524711 MHz

F2 - Processing parameters
SI                16384
SF          400.1500000 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

13C of VBAKB-784/1:



BRUKER

```
Current Data Parameters
NAME                  Dr. A MAJEE
EXPNO                         733
PROCNO                          1

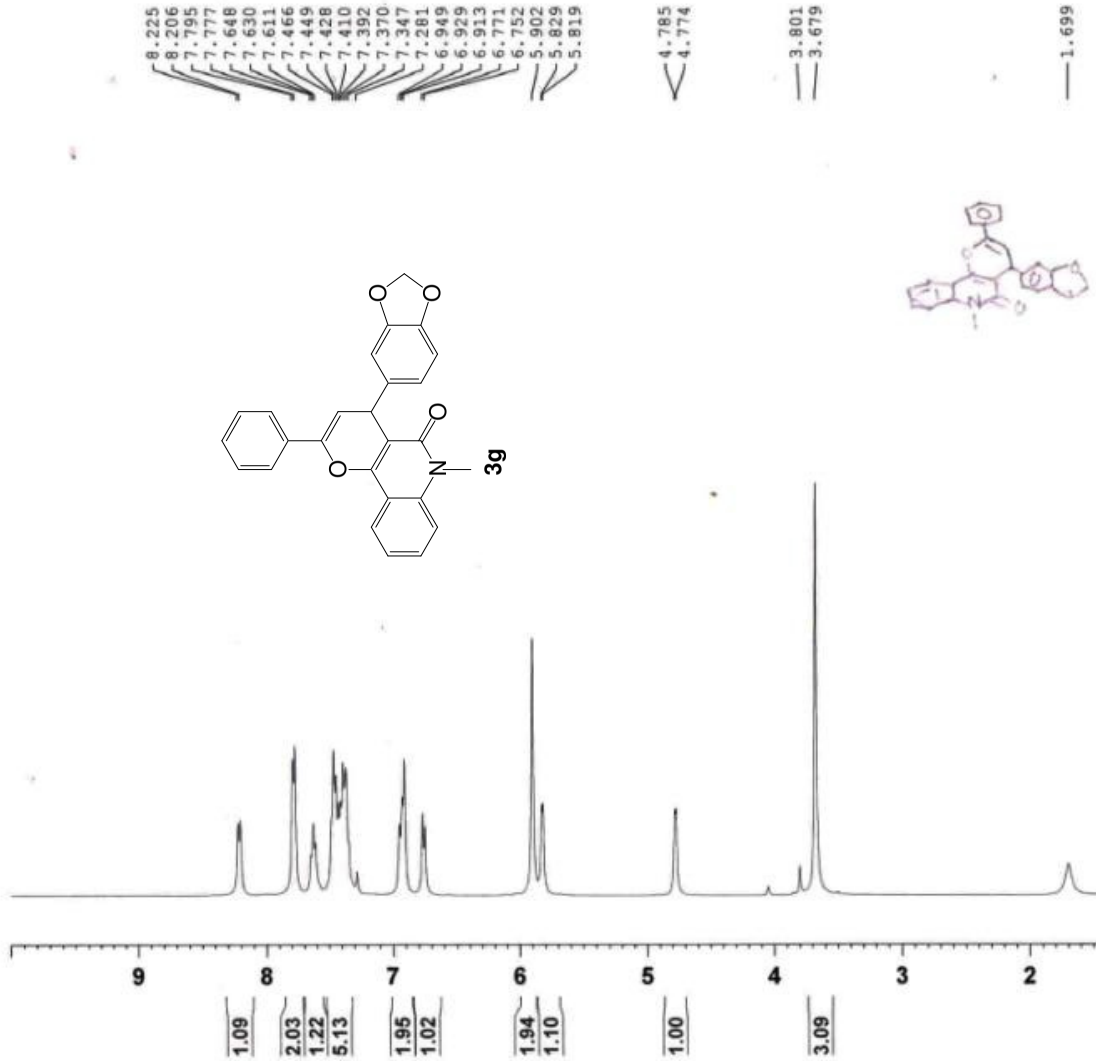
F2 - Acquisition Parameters
Date_                    20130523
Time                        16.41
INSTRUM                     spect
PROBHD    5 mm PABBO BB/
PULPROG                   zgpg30
TD                         32768
SOLVENT                    CDCl3
NS                           400
DS                             2
SWH                  24038.461 Hz
FIDRES                0.733596 Hz
AQ                    0.6816244 sec
RG                        77.59
DW                       20.800 usec
DE                        6.50 usec
TE                       296.9 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                         13C
P1                          8.90 usec
PLW1                54.00000000 W
SFO1               100.6276588 MHz

======== CHANNEL f2 ========
CPDPRG2                  waltz16
NUC2                          1H
PCPD2                      80.00 usec
PLW2                12.00000000 W
PLW12                0.40792999 W
PLW13                0.26107001 W
SFO2               400.1516006 MHz

F2 - Processing parameters
SI                           16384
SF                 100.6177880 MHz
WDW                             EM
SSB                0
LB                            1.00 Hz
GB                 0
PC                            1.40
```

1H of VBAKB-795/13



BRUKER

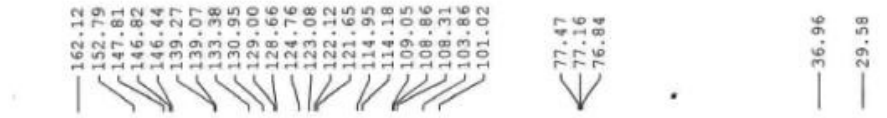
Current Data Parameters
NAME Dr. A MAJEE
EXPNO 834
PROCNO 1

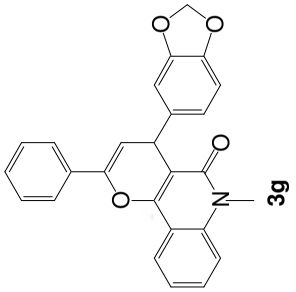
F2 - Acquisition Parameters
Date_ 20130607
Time 20.38
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 2
SWH 8223.685 Hz
FIDRES 0.250967 Hz
AQ 1.9923444 sec
RG 77.59
DW 60.800 usec
DE 6.50 usec
TE 296.6 K
D1 1.00000000 sec
TD0 1

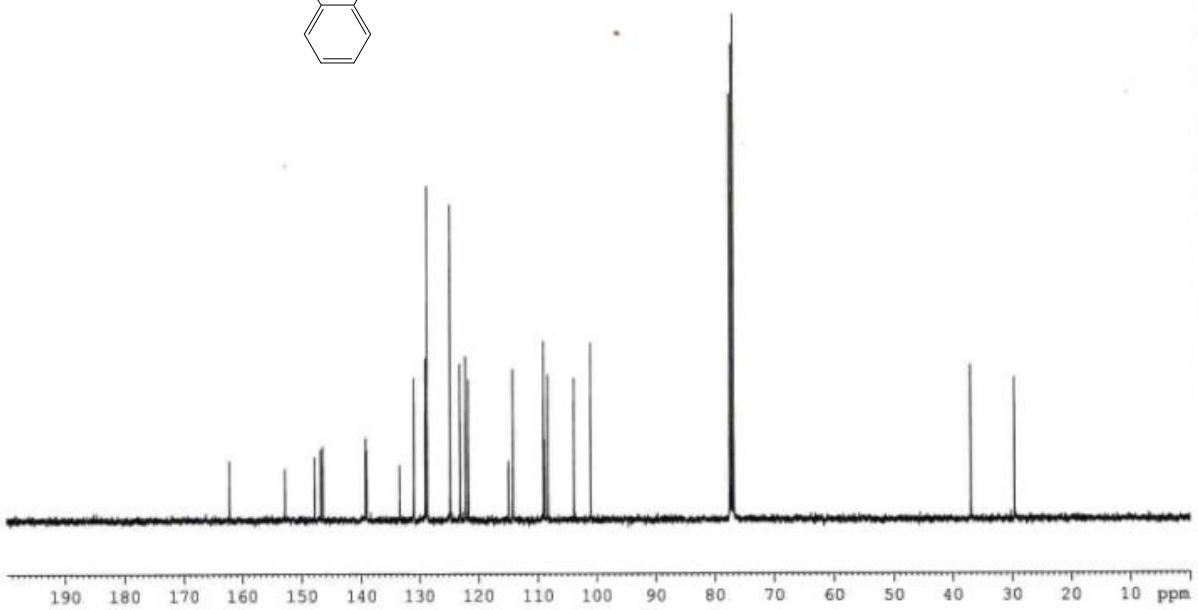
======== CHANNEL f1 ========
NUC1 1H
P1 14.75 usec
PLW1 11.99499989 W
SFO1 400.1524711 MHz

F2 - Processing parameters
SI 16384
SF 400.1500000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

13C of VBAKB-795/1:







BRUKER

```
Current Data Parameters
NAME            Dr. A MAJEE
EXPNO                838
PROCNO                 1

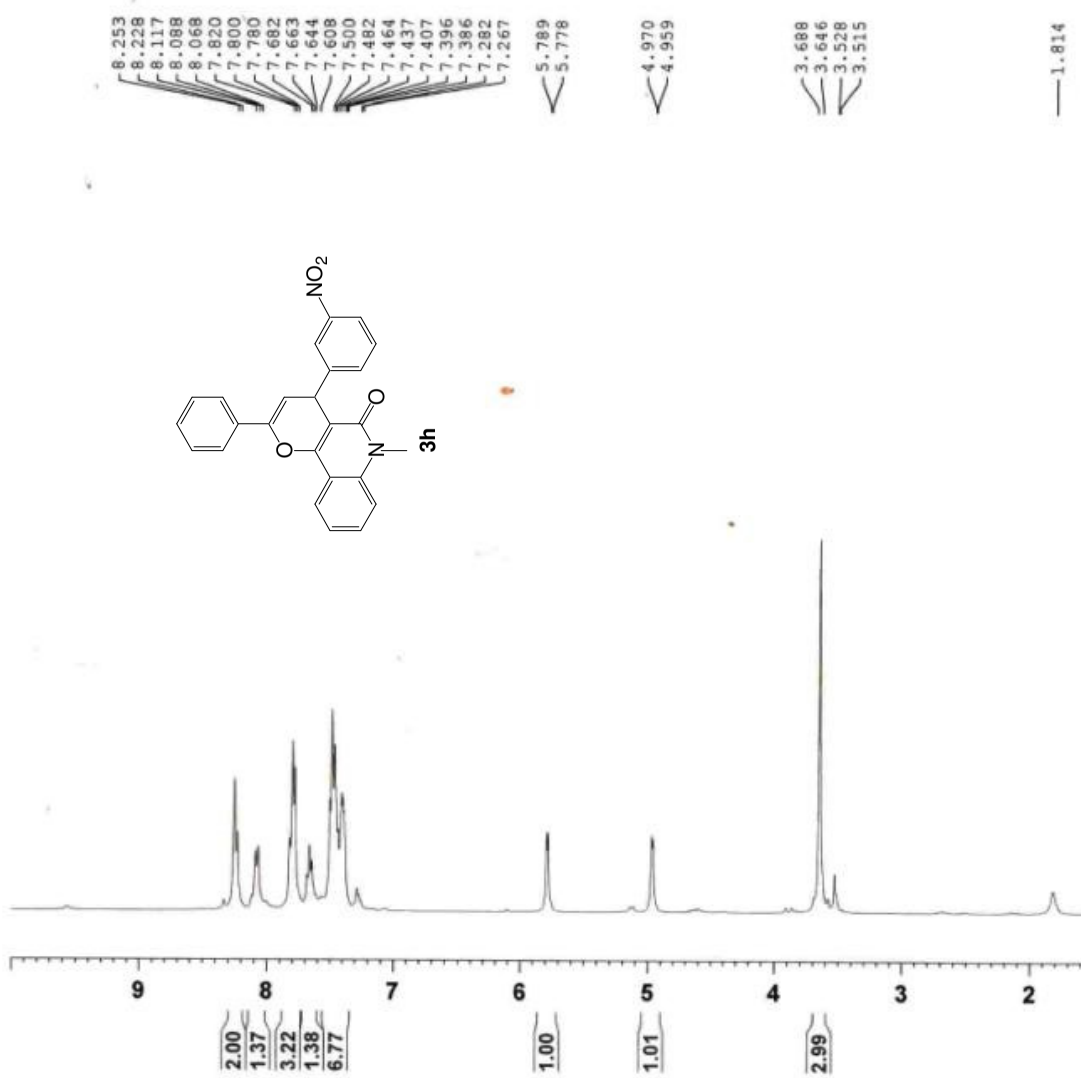
F2 - Acquisition Parameters
Date_           20130608
Time                19.26
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG             zgpg30
TD                  32768
SOLVENT             CDCl3
NS                    512
DS                      2
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ             0.6816244 sec
RG                  77.59
DW                 20.800 usec
DE                  6.50 usec
TE                294.9 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                   8.90 usec
PLW1          54.00000000 W
SFO1         100.6278588 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                   1H
PCPD2               80.00 usec
PLW2          12.00000000 W
PLW12          0.40792999 W
PLW13          0.26107001 W
SFO2         400.1516006 MHz

F2 - Processing parameters
SI                  16384
SF           100.6177895 MHz
WDW                    EM
SSB      0
LB                  1.00 Hz
GB       0
PC                  1.40
```

1H of VBAKB-798/13



BRUKER

Current Data Parameters
NAME Dr. A MAJEE
EXPNO 883
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130614
Time 18.57
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 2
SWH 8223.685 Hz
FIDRES 0.250967 Hz
AQ 1.9923444 sec
RG 62.69
DW 60.800 usec
DE 6.50 usec
TE 294.6 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 14.75 usec
PLW1 11.99499989 W
SFO1 400.1524711 MHz

F2 - Processing parameters
SI 16384
SF 400.1500000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

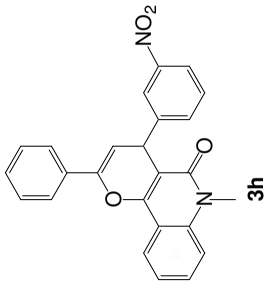
13C of VBAKB-798/1.

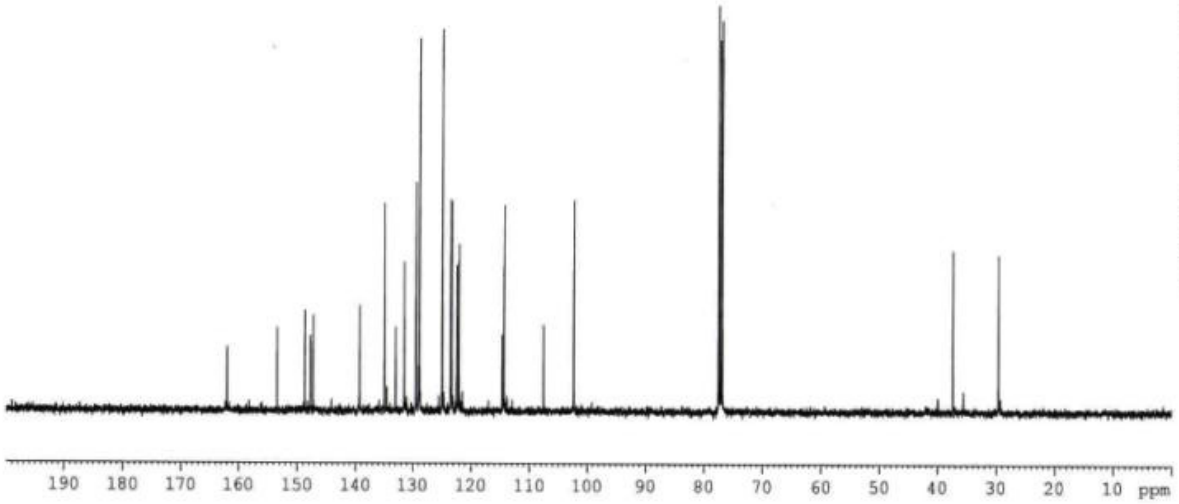
161.89
153.31
148.62
147.68
147.22
139.21
134.88
132.90
131.42
129.40
129.36
128.76
124.88
123.41
123.26
122.39
122.04
114.66
114.34
107.53
102.31

77.48
77.16
76.84

37.35

29.60





BRUKER

Current Data Parameters
NAME Dr. A MAJEE
EXPNO 884
PROCNO 1

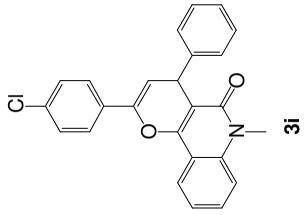
F2 - Acquisition Parameters
Date_ 20130614
Time 19.18
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 400
DS 2
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6816244 sec
RG 62.69
DW 20.800 usec
DE 6.50 usec
TE 295.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

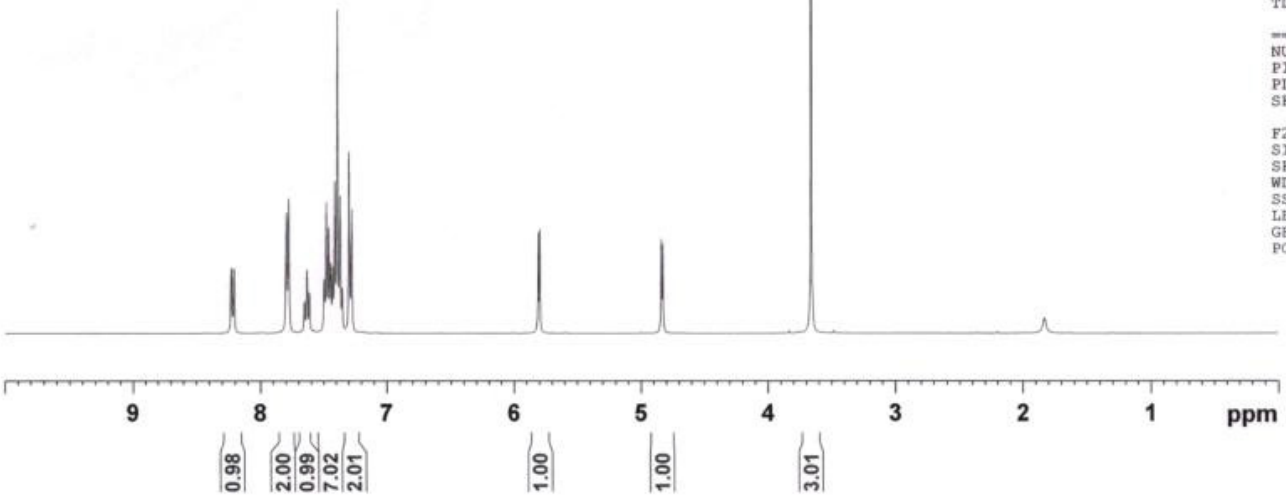
======== CHANNEL f1 ========
NUC1 13C
P1 8.90 usec
PLW1 54.00000000 W
SFO1 100.6278588 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PLW2 12.00000000 W
PLW12 0.40792999 W
PLW13 0.26107001 W
SFO2 400.1516006 MHz

F2 - Processing parameters
SI 16384
SF 100.6177902 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1H of VBAKB-793/13





BRUKER

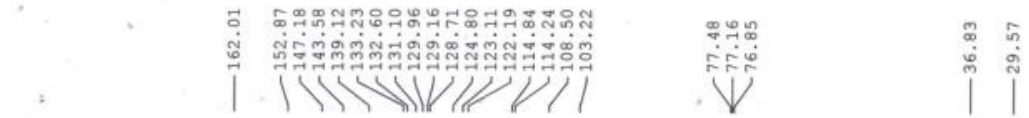
```
Current Data Parameters
NAME         Dr. A MAJEE
EXPNO                835
PROCNO                 1

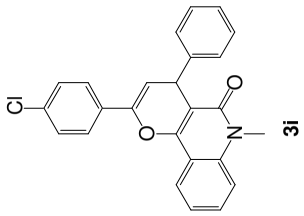
F2 - Acquisition Parameters
Date_           20130607
Time               20.43
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     2
SWH             8223.685 Hz
FIDRES          0.250967 Hz
AQ              1.9923444 sec
RG                 57.28
DW                60.800 usec
DE                 6.50 usec
TE                296.6 K
D1              1.00000000 sec
TD0                    1

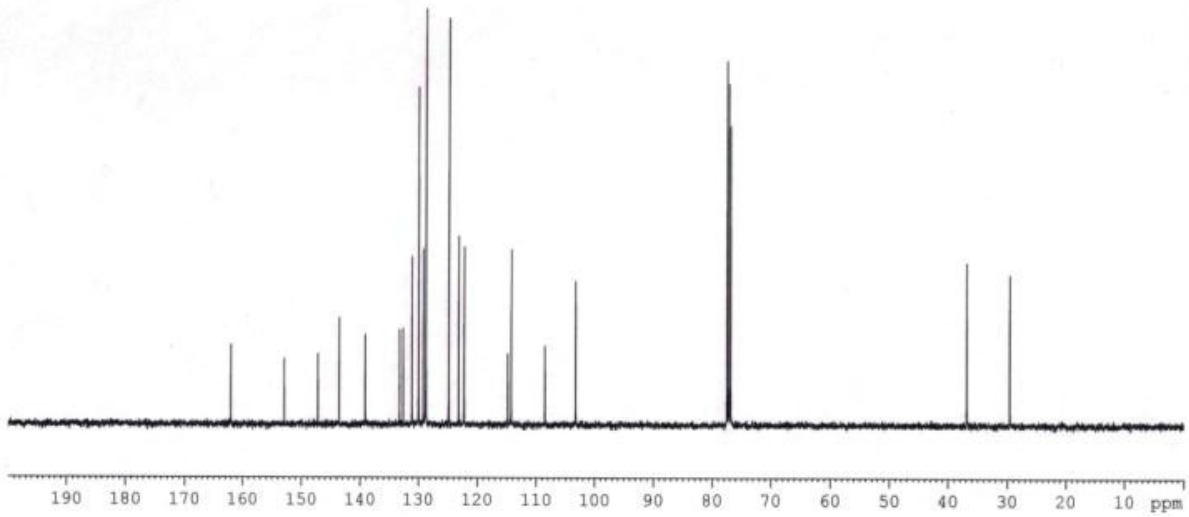
======== CHANNEL f1 ========
NUC1                  1H
P1                 14.75 usec
PLW1         11.99499989 W
SFO1         400.1524711 MHz

F2 - Processing parameters
SI                 16384
SF          400.1500000 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

13C of VBAKB-793







BRUKER

Current Data Parameters
NAME Dr. A MAJEE
EXPNO 839
PROCNO 1

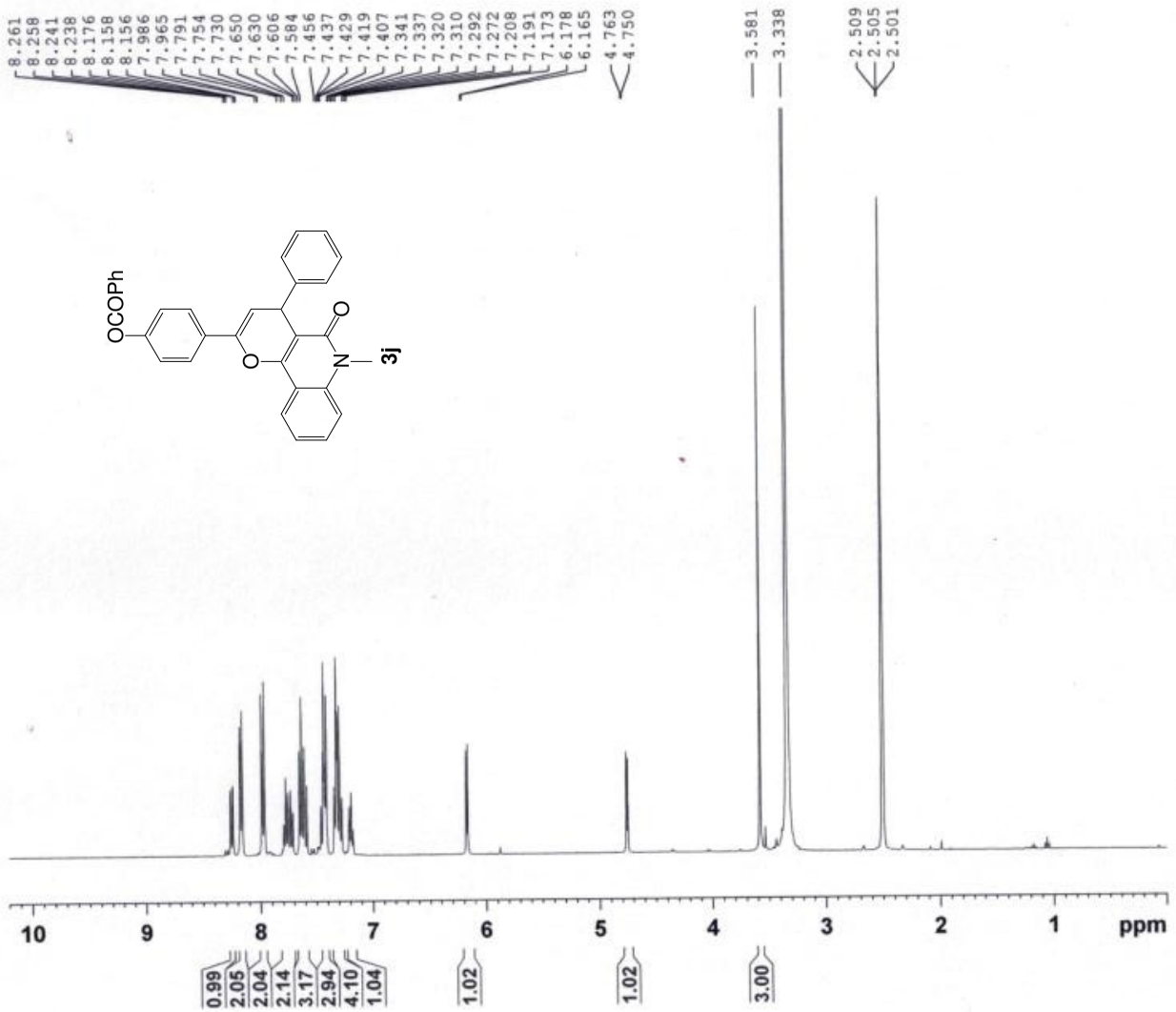
F2 - Acquisition Parameters
Date_ 20130608
Time 20.05
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 320
DS 2
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6816244 sec
RG 57.28
DW 20.800 usec
DE 6.50 usec
TE 295.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 8.90 usec
PLW1 54.00000000 W
SFO1 100.6278588 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PLW2 12.00000000 W
PLW12 0.40792999 W
PLW13 0.26107001 W
SFO2 400.1516006 MHz

F2 - Processing parameters
SI 16384
SF 100.6177895 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1H of VBAKB-799-1



Current Data Parameters

NAME	Dr. A HAJRA
EXPNO	884
PROCNO	1

F2 - Acquisition Parameters

Date_	20130731
Time	18.34
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zg30
TD	32768
SOLVENT	DMSO
NS	40
DS	2
SWH	8223.685 Hz
FIDRES	0.250967 Hz
AQ	1.9923444 sec
RG	87.66
DW	60.800 usec
DE	6.50 usec
TE	298.5 K
D1	1.00000000 sec
TD0	1

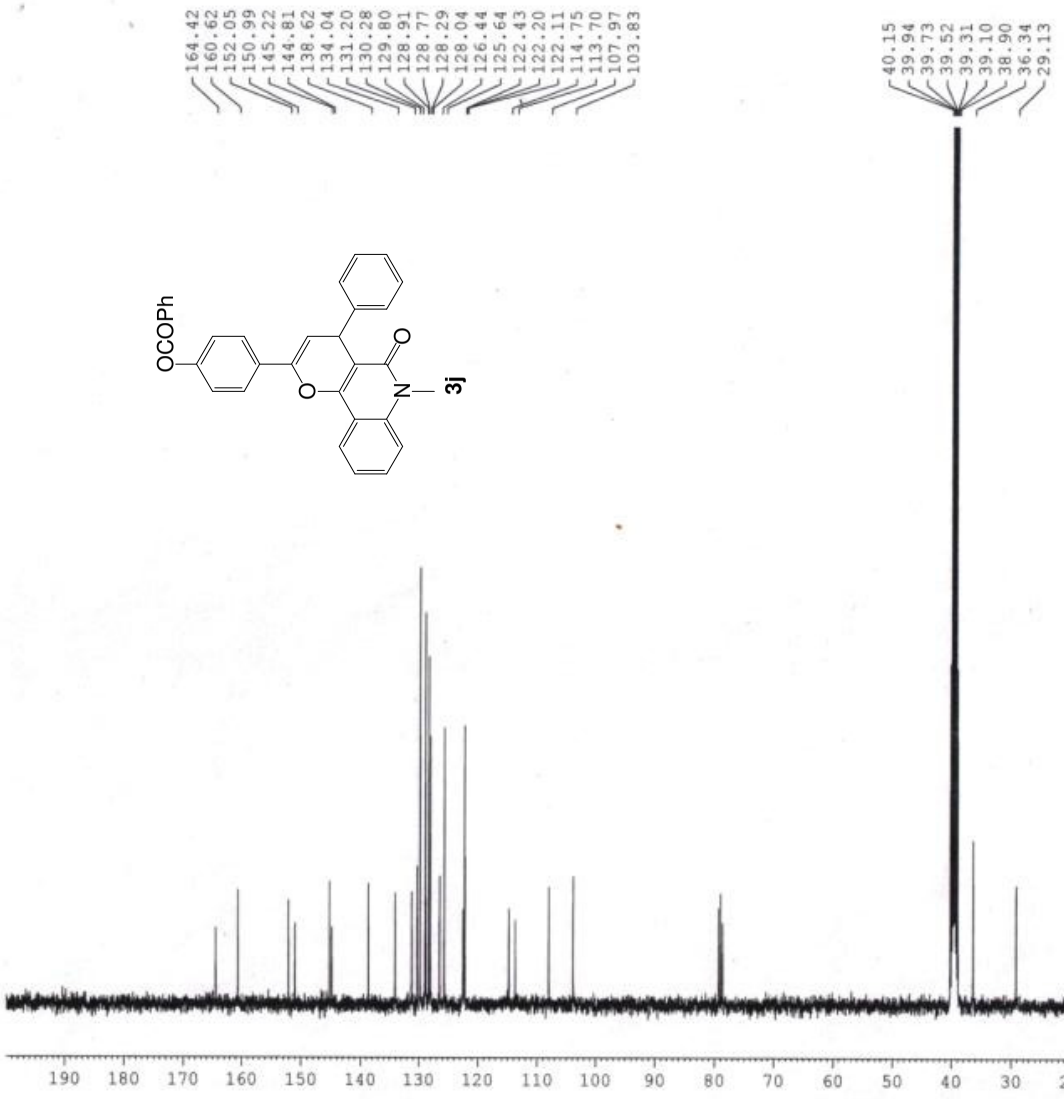
======== CHANNEL f1 ========

NUC1	1H
P1	14.75 usec
PLW1	11.99499989 W
SFO1	400.1524711 MHz

F2 - Processing parameters

SI	16384
SF	400.1500000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

13C of VBAKB-799-1



BRUKER

Current Data Parameters
NAME Dr. A HAJRA
EXPNO 890
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130731
Time 21.20
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT DMSO
NS 640
DS 2
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6816244 sec
RG 87.66
DW 20.800 usec
DE 6.50 usec
TE 299.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 8.90 usec
PLW1 54.00000000 W
SFO1 100.6278588 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PLW2 12.00000000 W
PLW12 0.40792999 W
PLW13 0.26107001 W
SFO2 400.1516006 MHz

F2 - Processing parameters
SI 16384
SF 100.6178471 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1H of VBAKB-823

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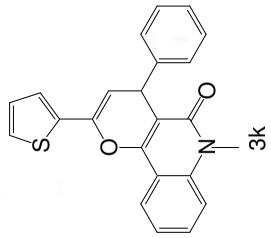


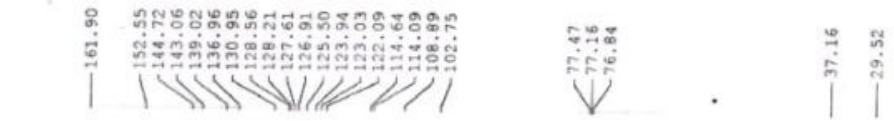

```
Current Data Parameters
NAME      Dr. A HAJRA 1
EXPNO              286
PROCNO               1

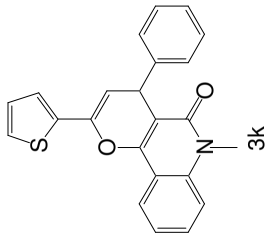
F2 - Acquisition Parameters
Date_          20131022
Time              12.17
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG           zg30
TD               32768
SOLVENT          CDCl3
NS                  24
DS                   2
SWH            8223.685 Hz
FIDRES         0.250967 Hz
AQ             1.9923444 sec
RG                54.07
DW               60.800 usec
DE                6.50 usec
TE               295.1 K
D1            1.00000000 sec
TD0                  1

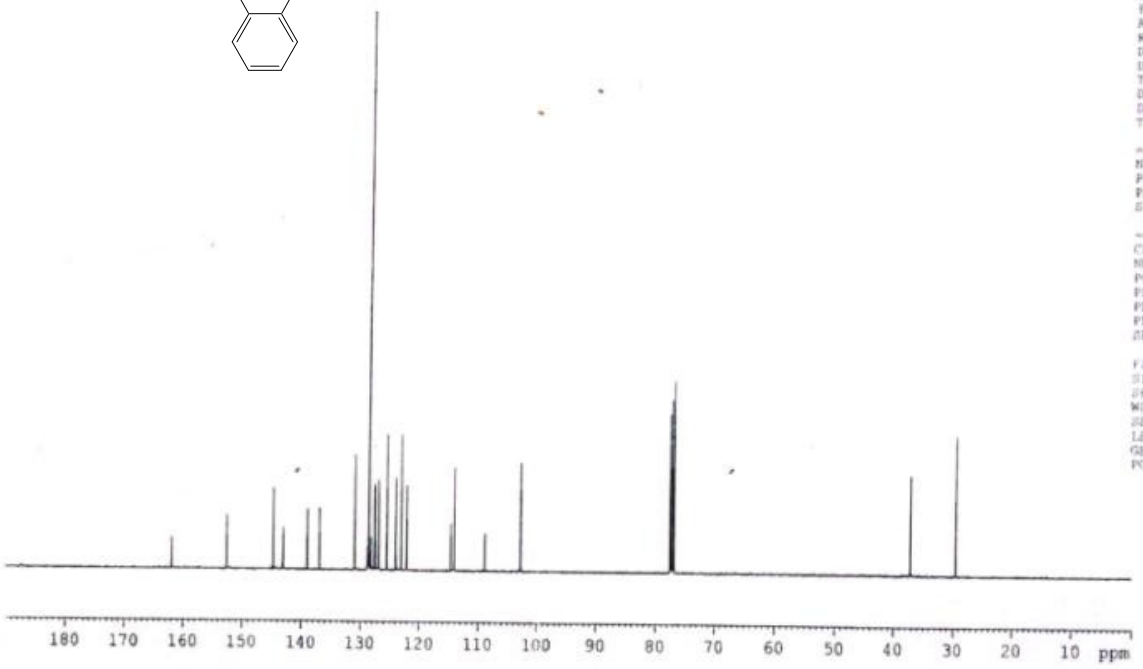
======== CHANNEL f1 ========
NUC1                 1H
P1                14.75 usec
PLW1         11.99499989 W
SFO1         400.1524711 MHz

F2 - Processing parameters
SI                16384
SF           400.1500544 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

13C of VBAKB-823/1:







BRUKER

```
Current Data Parameters
NAME        Dr. A RAJNA 1
EXPNO              289
PROCNO               1

F2 - Acquisition Parameters
Date          20131022
Time              13.34
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD              32768
SOLVENT           CDCl3
NS                 240
DS                   2
SWH         24038.461 Hz
FIDRES       0.733596 Hz
AQ           0.6816244 sec
RG              54.07
DW             20.800 usec
DE              6.50 usec
TE            294.8 K
D1          2.00000000 sec
D11         0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1              13C
P1              8.90 usec
PLW1       54.00000000 W
SFO1      100.6278588 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2               1H
PCPD2           60.00 usec
PLW2       12.00000000 W
PLW12       0.40792999 W
PLW13       0.26107001 W
SFO2      400.1516004 MHz

F2 - Processing parameters
SI              16384
SF        100.6127939 MHz
WDW                EM
SSB     0
LB              1.00 Hz
GB      0
PC              1.40
```