Electronic supplementary information (ESI)

KF/alumina catalyzed regioselective benzylation and benzoylation using solvent-free grind-stone chemistry

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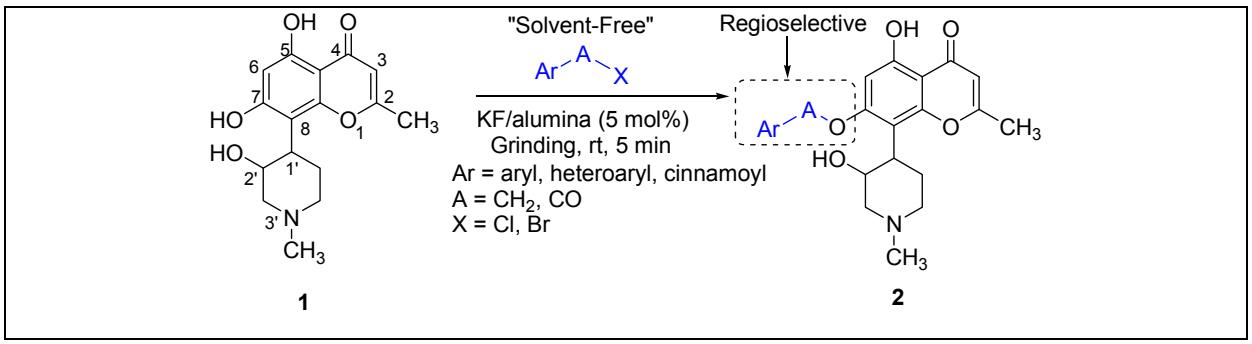


CONTENTS

S1. General

All chemicals were obtained from Sigma-Aldrich Company and used as received. ^{1}H, ^{13}C and DEPT NMR spectra were recorded on Brucker-Avance DPX FT-NMR 500 and 400 MHz instruments. Chemical data for protons are reported in parts per million (ppm) downfield from tetramethylsilane and are referenced to the residual proton in the NMR solvent ($CDCl_3$, 7.26 ppm; CD_3OD, 3.31 ppm). Carbon nuclear magnetic resonance spectra (^{13}C NMR) were recorded at 125 MHz or 100 MHz: chemical data for carbons are reported in parts per million (ppm, δ scale) downfield from tetramethylsilane and are referenced to the carbon resonance of the solvent ($CDCl_3$, 77.16 ppm; CD_3OD, 49.0 ppm). ESI-MS spectra were recorded on Agilent 1100 LC-Q-TOF. IR spectra were recorded on Perkin-Elmer IR spectrophotometer. Melting points were recorded on digital melting point apparatus.

S2. Procedure for preparation of KF-alumina

A mixture of potassium fluoride (45 g) and basic alumina (55 g, type T, Merck) in water (100 mL) was stirred at room temperature for 10 min. The resulting suspension was concentrated on rotary evaporator at 50 ^{o}C, and then dried in calcium chloride desicator under vacuum for 15 h. Use of basic alumina in the solid support gave better results relative to the neutral one.

S3. General procedure for *ON/S*-benzylation/benzoylation of phenols, other aromatics and heteroaromatics

Hydroxy aromatic compounds/ aromatic amines/ NH-heteroarenes/ thiophenols (**1**, **3** or **5**, 1 mmol), KF/Al_2O_3 (5 mol%) and benzyl/benzoyl halides (1.2 mmol) were mixed in a mortar and grinded intermittently using a pestle. The mixture changed to mushy state within a proper reaction time and then gets solidified itself. Formation of product was monitored by TLC. After filtration of the catalyst, the filtrate was washed with aqueous NaOH (10%) and the organic phase was evaporated under reduced pressure to furnish the desired product. The crude product was sufficiently pure (as observed by TLC and ^{1}H NMR), and further purification was performed by column chromatography. All products were characterized by melting point, MS and ^{1}H NMR

data and their physical data were similar to those reported in the literature. Spectral characterization data for new compounds is provided:

7-(4-Methoxybenzyloxy)-5-hydroxy-8-(3-hydroxy-1-methylpiperidin-4-yl)-2-methyl-4H-chromen-4-one (2a): White powder; m.p. 208-210 ^{o}C; ^{1}H NMR (200 MHz, D_2O, ppm): δ 12.39 (s, 1H, H-bonded), 7.71 (d, J = 8.2 Hz, 2H), 7.09 (d, J = 8.2 Hz, 2H), 5.82 (s, 1H), 5.57 (s, 1H), 5.29 (d, J = 12.7 Hz, 1H), 4.71 (d, J = 12.7 Hz, 1H), 4.15 (brs, 1H), 3.80 (s, 3H), 3.57-3.35 (m, 6H of piperidine), 2.83 (s, 3H), 2.27 (s, 3H), 1.54 (m, 1H); ^{13}C NMR (100 MHz, CD_3OD, ppm): δ 179.62, 176.46, 163.71, 159.99, 159.69, 155.30, 134.37, 120.21, 113.72, 106.80, 106.12, 102.14, 98.04, 68.37, 62.58, 62.27, 61.80, 54.84, 51.53, 35.55, 19.85, 19.38; IR (KBr): ν_{max} 3415, 2854, 1617, 1352, 1258, 1180 cm^{-1}; ESI-MS: *m/z* 448.20 $[M+Na]^+$.

7-(Benzyloxy)-5-hydroxy-8-(3-hydroxy-1-methylpiperidin-4-yl)-2-methyl-4H-chromen-4-one (2b): Light yellowish powder; m.p. 218-220 °C; ^{1}H-NMR (400 MHz, CD_3OD, ppm): δ 7.16 (m, 5H, ArH), 6.64 (s, 1H, H-6), 6.13 (s, 1H, H-3), 5.22 (brs, 2H), 4.29 (brs, 1H, H-3'), 3.80-3.45 (m, 6H of piperidine), 2.99 (s, 3H), 2.47 (s, 3H) 1.96 (m, 1H); IR (KBr): ν_{max} 3325, 2923, 1658, 1588, 1420, 1270, 1120, 1028 cm^{-1}; ESI-MS: *m/z* 396.2103 $[M+H]^+$.

7-(4-Bromobenzyloxy)-5-hydroxy-8-(3-hydroxy-1-methylpiperidin-4-yl)-2-methyl-4H-chromen-4-one (2c): Off-white powder; m.p. 248-250 °C; ^{1}H NMR (400 MHz, CD_3OD, ppm): 7.68 (m, 4H), 5.97 (s, 1H), 5.37 (s, 1H), 5.33 (d, J = 12 Hz, 1H), 4.84 (d, J = 12 Hz, 1H), 4.33 (brs, 1H), 3.73-3.30 (m, 6H of piperidine), 2.89 (s, 3H), 2.43 (s, 3H), 1.71 (m, 1H); ESI-MS: *m/z* 476.0895 $[M+H]^+$.

7-(4-Nitrobenzyloxy)-5-hydroxy-8-(3-hydroxy-1-methylpiperidin-4-yl)-2-methyl-4H-chromen-4-one (2d): White powder; m.p. 234-236 °C; ^{1}H NMR (400 MHz, CD_3OD, ppm): δ 8.33 (d, J = 8.4 Hz, 2H), 8.08 (d, J = 8.4 Hz, 2H), 6.17 (m, 2H), 5.97 (d, J = 12.8 Hz, 1H), 4.80 (d, J = 12.8 Hz, 1H), 4.13 (brs, 1H), 3.77-3.35 (m, 6H of piperidine), 2.95 (s, 3H), 2.35 (s, 3H), 1.74 (m, 1H). ^{13}C NMR (100 MHz, CD_3OD, ppm): δ 183.46, 176.22, 167.33, 162.22, 157.66, 149.16, 136.55 (2C), 124.89 (2C), 107.94, 107.88, 104.36, 104.03, 101.85, 70.89, 65.17, 64.55, 62.78, 57.29, 37.71, 21.73, 20.28; ESI-MS: *m/z* 441.1692 $[M+H]^+$.

7-(2-Bromobenzyloxy)-5-hydroxy-8-(3-hydroxy-1-methylpiperidin-4-yl)-2-methyl-*4H*-chromen-4-one (2e): White powder; m.p. 238-241 °C; ^{1}H NMR (400 MHz, CD_3OD, ppm): δ 8.10 (d, J = 8.0 Hz, 1H), 7.83 (m, 1H), 7.48 (m, 2H), 5.98 (brs, 2H), 5.48 (d, J = 13.2 Hz, 1H), 5.17 (d, J = 13.3 Hz, 1H), 4.37 (brs, 1H), 3.95-3.59 (m, 6H of piperidine), 2.41 (s, 3H), 2.01 (s, 3H), 1.84 (m, 1H); ^{13}C NMR (100 MHz, CD_3OD, ppm): δ 184.43, 169.35, 164.14, 161.97, 158.39, 137.24, 136.47, 135.66, 133.73, 129.69, 128.96, 108.81, 106.27, 105.51, 100.37, 72.14, 68.83, 66.74, 64.61, 53.39, 38.00, 21.31, 20.76; ESI-MS: *m/z* 474 $[M+1]^+$.

7-(Cinnamyloxy)-5-hydroxy-8-(3-hydroxy-1-methylpiperidin-4-yl)-2-methyl-*4H*-chromen-4-one (2f): Yellow powder; m.p. 242-246 °C; ^{1}H NMR (400 MHz, CD_3OD, ppm): δ 7.57 (m, 2H), 7.37 (m, 3H), 7.08 (d, J = 15.6, 1H), 6.51 (m, J = 15.6, 1H), 6.07 (s, 1H) 5.97 (s, 1H), 4.85 (m, 1H), 4.51 (m, 1H), 4.28 (brs, 1H), 3.78-3.33 (m, 6H of piperidine ring) 3.10 (s, 3H), 2.36 (s, 3H), 1.74 (m, 1H).). ^{13}C NMR (100 MHz, CD_3OD, ppm): δ 180.75, 165.22, 164.62, 159.96, 155.80, 140.84, 135.21, 128.6 (2C), 127.03(2C), 117.2, 106.71, 106.60, 101.68, 101.22, 68.25, 64.8, 63.7, 62.4, 52.2, 36.1, 19.95, 19.76; ESI-MS: *m/z* 423.0771 $[M+H]^+$.

7-((1H-Benzo[d]imidazol-2-yl)methoxy)-5-hydroxy-8-(3-hydroxy-1-methylpiperidin-4-yl)-2-methyl-*4H*-chromen-4-one (2g): White powder; m.p. 217-220 °C; ^{1}H NMR (400 MHz, CD_3OD, ppm): δ 7.61 (dd, J = 3.2, 6 Hz, 2H), 7.52 (dd, J = 2.8, 6 Hz, 2H), 5.90 (s, 1H), 5.82 (s, 1H), 5.50 (d, J = 13.6 Hz, 1H), 4.99 (d, J = 13.6 Hz, 1H), 4.27 (brs, 1H), 3.91-3.26 (m, 6H of piperidin), 2.58 (s, 3H), 2.23 (s, 3H), 1.54 (m, 1H); ESI-MS: *m/z* 436.1835 $[M+H]^+$.

5-Hydroxy-8-(3-hydroxy-1-methylpiperidin-4-yl)-2-methyl-4-oxo-4H-chromen-7-yl-furan-2-carboxylate (2h): White powder; m.p. 184-186 ^{o}C; ^{1}H NMR (400 MHz, CD_3OD, ppm): δ 7.73 (dd, J = 0.4, 1.6 Hz, 1H), 7.49 (dd, J = 0.8, 3.6 Hz, 1H), 6.60 (dd, J = 1.6, 3.6 Hz, 1H), 6.22 (s, 1H), 5.96 (s, 1H), 5.58 (brs, 1H), 3.89-3.33 (m, 6H of piperidine), 2.97 (s, 3H), 2.14 (s, 3H), 2.10 (m, 1H); ^{13}C NMR (100 MHz, CD_3OD, ppm): δ 184.16, 169.03, 164.07, 162.34, 158.66, 158.21, 148.77, 145.03, 120.90, 113.27, 108.67, 105.18, 104.62, 99.79, 70.35, 58.27, 56.92, 44.67, 36.52, 24.37, 20.47; IR (KBr): ν_{max} 3416, 2924, 1617, 1560, 1424, 1290, 1177, 1113 cm^{-1}; ESI-MS: *m/z* 400.16 $[M+H]^+$.

5-Hydroxy-8-(3-hydroxy-1-methylpiperidin-4-yl)-2-methyl-4-oxo-4H-chromen-7-yl-2-methylbenzoate (2i): White powder; m.p. 244-246 °C; ^{1}H-NMR (400 MHz, CD_3OD, ppm): δ 8.03 (d, J = 7.2 Hz, 1H), 7.36 (dd, J = 7.2, 7.6 Hz, 1H), 7.23-7.13 (m, 2H), 6.187 (s, 1H), 5.83 (s, 1H), 5.61 (brs, 1H), 3.84-3.40 (m, 6H of piperidin), 2.94 (s, 3H), 2.17 (s, 3H), 2.08 (s, 3H), 2.03 (m, 1H); ESI-MS: *m/z* 424.4592 $[M+H]^+$.

1-((4-Sec-butylphenoxy) methyl)benzene (4d): White solid; m.p. 121-124 ^{o}C; ^{1}H NMR (200 MHz, $CDCl_3$, ppm): δ 7.64-6.43 (m, 9H), 5.03 (s, 2H), 2.52 (m, 1H), 1.53 (m, 2H), 1.23 (m, 3H), 0.93 (m, 3H); ESI-MS: *m/z* 239 $[M-H]^+$.

1-((4-Isopropyl-3-methylphenoxy)methyl)benzene (4e): White solid; 131-133 ^{o}C; ^{1}H NMR (500 MHz, $CDCl_3$, ppm): δ 7.44-7.13 (m, 6H), 6.83 (d, J = 6.8 Hz, 2H), 5.02 (s, 2H), 3.13 (m, 1H), 2.33 (s, 3H), 1.23 (d, J = 7.6 Hz, 6H); ESI-MS: *m/z* 239 $[M-H]^+$.

4-(Benzyloxy)-6-bromoquinazoline (6d): White crystalline solid; m.p. 127-129 ^{o}C; ^{1}H NMR (500 MHz, $CDCl_3$, ppm): δ 8.46 (s, 1H), 8.10 (s, 1H), 7.84 (d, J = 8.2 Hz, 1H), 7.59 (d, J = 8.2 Hz, 1H), 7.36 (m, 5H), 5.19 (s, 2H); IR (KBr): ν_{max} 3434, 3064, 2953, 1673, 1605.97, 1468.39, 1363, 1318, 1256, 1151, 1077 cm^{-1}; ESI-MS: *m/z* 315.12 $[M+H]^+$.

2-(Dibenzylamino)-4-nitrophenol (6g): Red solid; m.p. 185-187 ^{o}C; ^{1}H NMR (200 MHz, $CDCl_3$, ppm): δ 7.78 (m, 1H), 7.61 (brs,1H), 7.25 (m, 10H), 6.85 (m, 1H), 4.30 (s, 4H); ^{13}C NMR (125 MHz, $CDCl_3$, ppm): δ 105.5, 112.9, 114.51, 116.01, 119.3, 120.90, 128.15, 129.2, 129.64, 129.9, 139.15, 141.58, 57.07; IR (KBr): ν_{max} 3410, 1584, 1527, 1497, 1332, 1277, 1100 cm^{-1}; ESI-MS: *m/z* 335.23 $[M+H]^+$.

1-Benzyl-5-nitro-1H-indazole (6ja): Cream colored solid; m.p. 118-121 ^{o}C; ^{1}H NMR ($CDCl_3$, 200 MHz): δ 8.69 (d, J = 1.5 Hz, 1H), 8.17 (s, 1H), 8.09 (dd, J = 2.0, 9.5 Hz, 1H), 7.77 (d, J = 9.0 Hz, 1H), 7.42-7.36 (m, 3H), 7.33-7.31 (m, 2H), 5.64 (s, 2H); ^{13}C NMR ($CDCl_3$, 125 MHz): 142.36 (C-5), 141.17 (C-7a), 136.04 (C-3), 135.7 (C-1′), 128.93 (C-4′), 128.23 (C-2′,6′), 127.24 (C-3′,5′), 123.45 (3a), 121.48 (C-6), 118.92 (C-4), 109.61 (C-7), 53.51 (C-7′); IR ($CHCl_3$): ν_{max} 3436, 3032, 1622, 1498, 1339, 1331, 1068 cm^{-1}; ESI-MS: *m/z* 275.14 $[M+Na]^+$ (^{1}H NMR data is in accordance with literature values: *Org. Process Res. Dev.* 2011, **15**, 565–569).

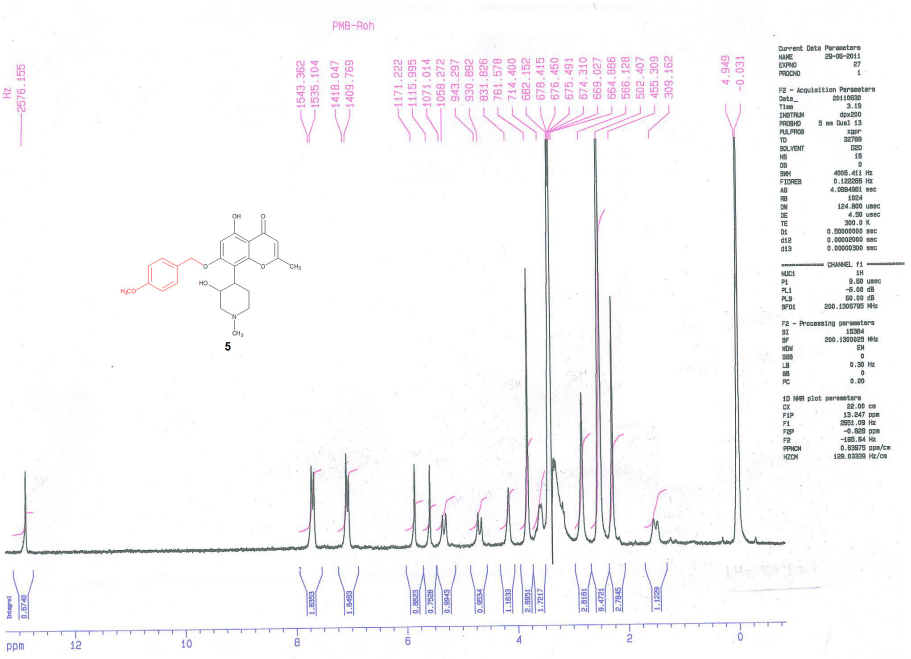
2-Benzyl-5-nitro-2H-indazole (6jb): Light yellow solid; m.p. 128-131 ^{o}C; ^{1}H NMR ($CDCl_3$, 200 MHz): δ 8.73 (s, 1H), 8.25 (s, 1H), 8.22 (d, J = 9.5 Hz, 1H), 7.40 (d, J = 9.0 Hz, 1H), 7.34-7.28 (m, 3H), 7.22-7.19 (m, 2H), 5.65 (s, 2H); ^{13}C NMR ($CDCl_3$, 125 MHz): 149.90 (C-5), 143.08 (C-7a), 134.60 (C-1'), 129.16 (C-3′,5′), 128.90 (C-4′), 128.28 (C-2′,6′), 127.07 (C-3), 120.24 (C-3a), 120.25 (C-6), 128.90 (C-4), 118.49 (C-7), 58.16 (C-7′); IR ($CHCl_3$): ν_{max} 3436, 3147, 3111, 1542, 1488, 1339, 1285, 1124 cm^{-1}; ESI-MS: *m/z* 275.14 $[M+Na]^+$.

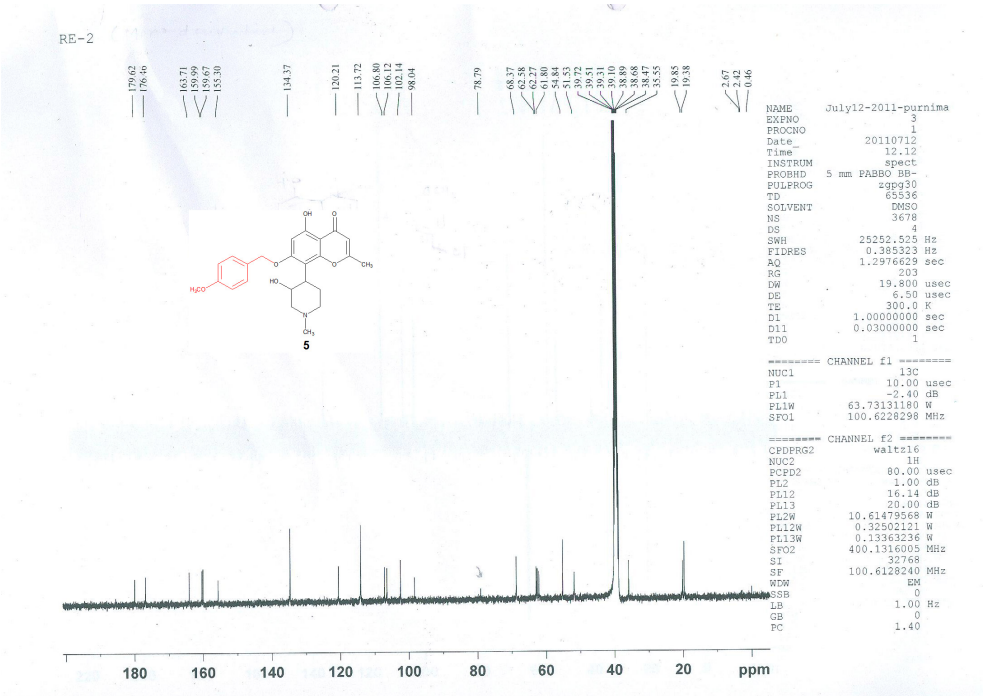
S4. Recyclability studies of KF/alumina catalyst

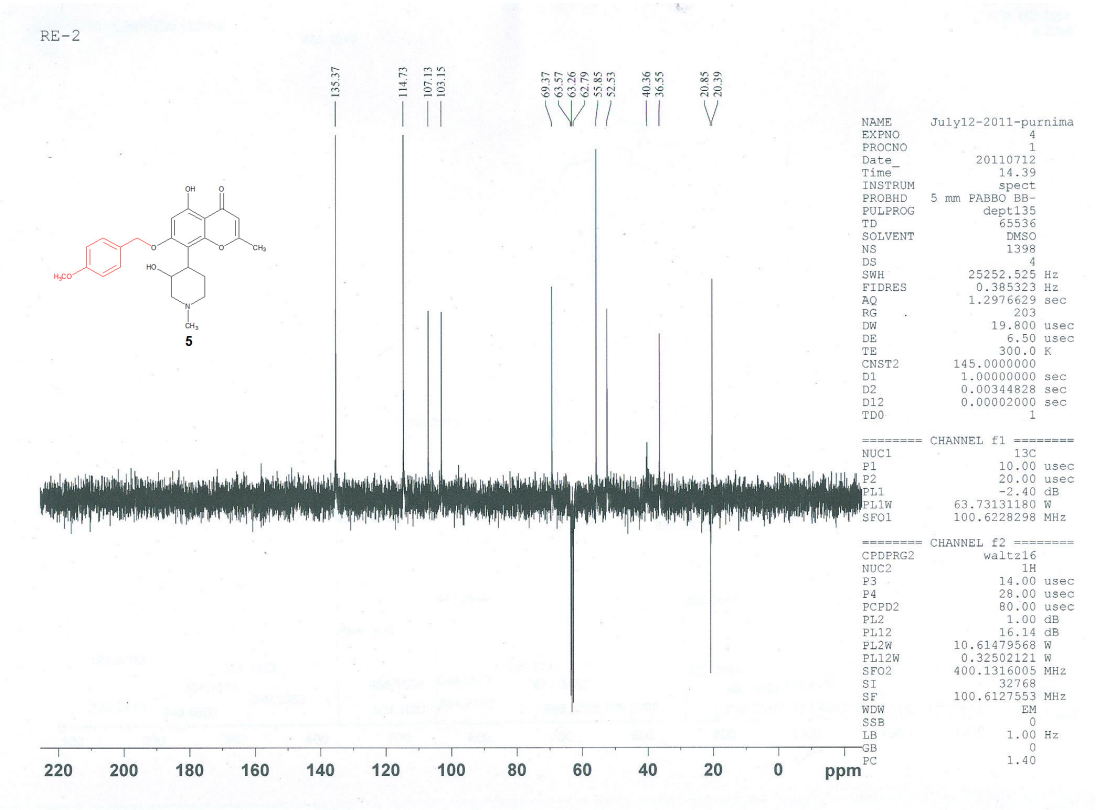
A mixture of rohitukine (**1**, 500 mg, 1 mmol), KF/Al_2O_3 (5 mol%) and PMB-Cl (1.2 mmol) was mixed in a mortar and grinded intermittently using a pestle for 20 min. After completion of reaction, organic solvent was added to the reaction mixture and filtered. The residual KF/Al_2O_3 catalyst obtained from the reaction mixture was washed with ethyl acetate then dried under vacuum at 100 ^{o}C for 10 h and reused in next cycle. The catalyst was recycled 5 times and the amount of catalyst recovered and percentage yield of **2a** was determined.

S5. Scanned spectra's of representative compounds

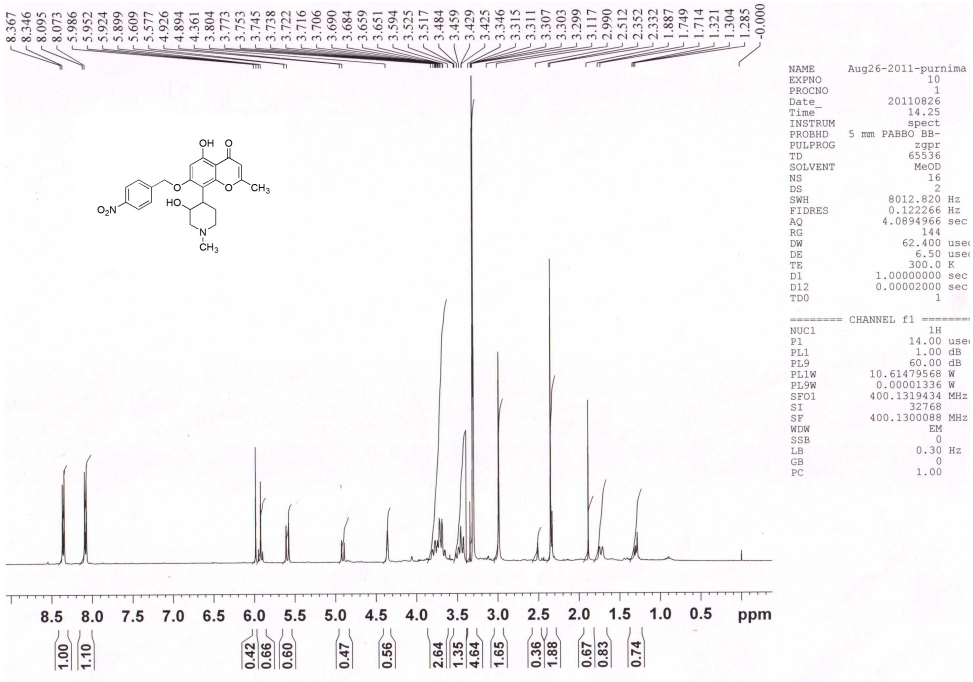
1. ^{1}H NMR, ^{13}C NMR and DEPT-135 spectrum of 7-(4-Methoxybenzyloxy)-5-hydroxy-8-(3-hydroxy-1-methylpiperidin-4-yl)-2-methyl-4H-chromen-4-one (**2a**) in DMSO-d_6

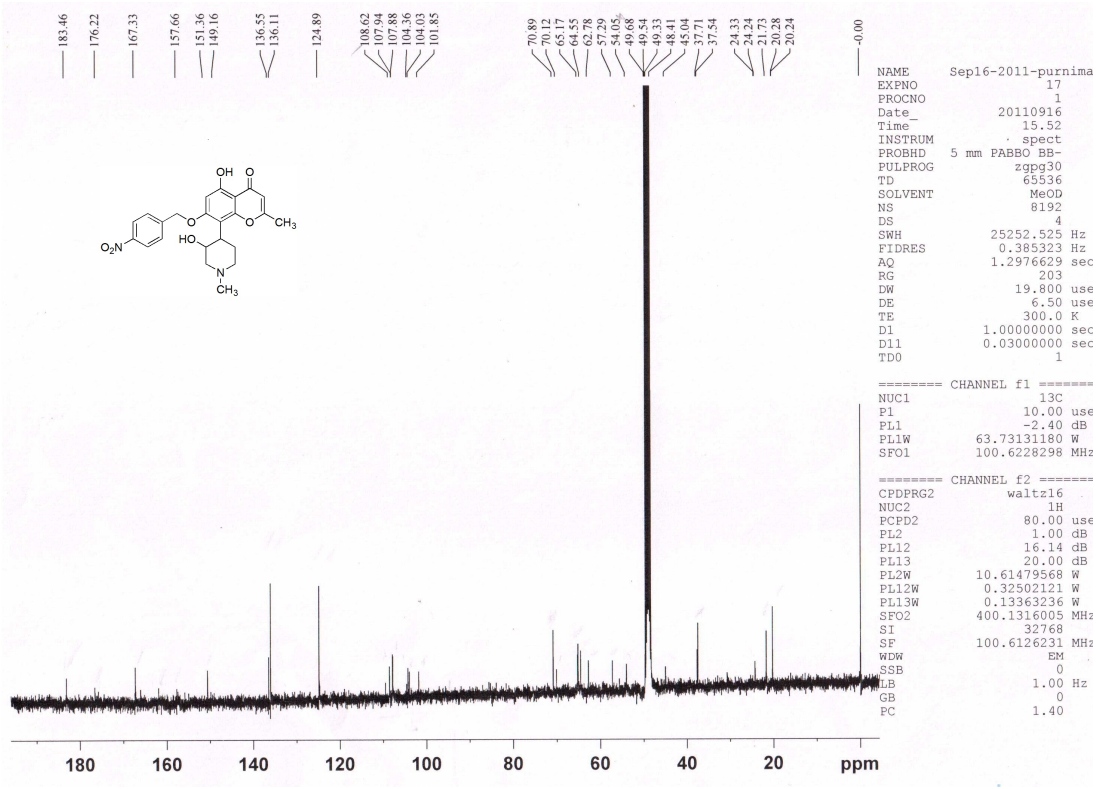






2. ^{1}H NMR, ^{13}C NMR and DEPT-135 spectrum of 7-(4-nitrobenzyloxy)-5-hydroxy-8-(3-hydroxy-1-methylpiperidin-4-yl)-2-methyl-4H-chromen-4-one **(2d)** in CD_3OD

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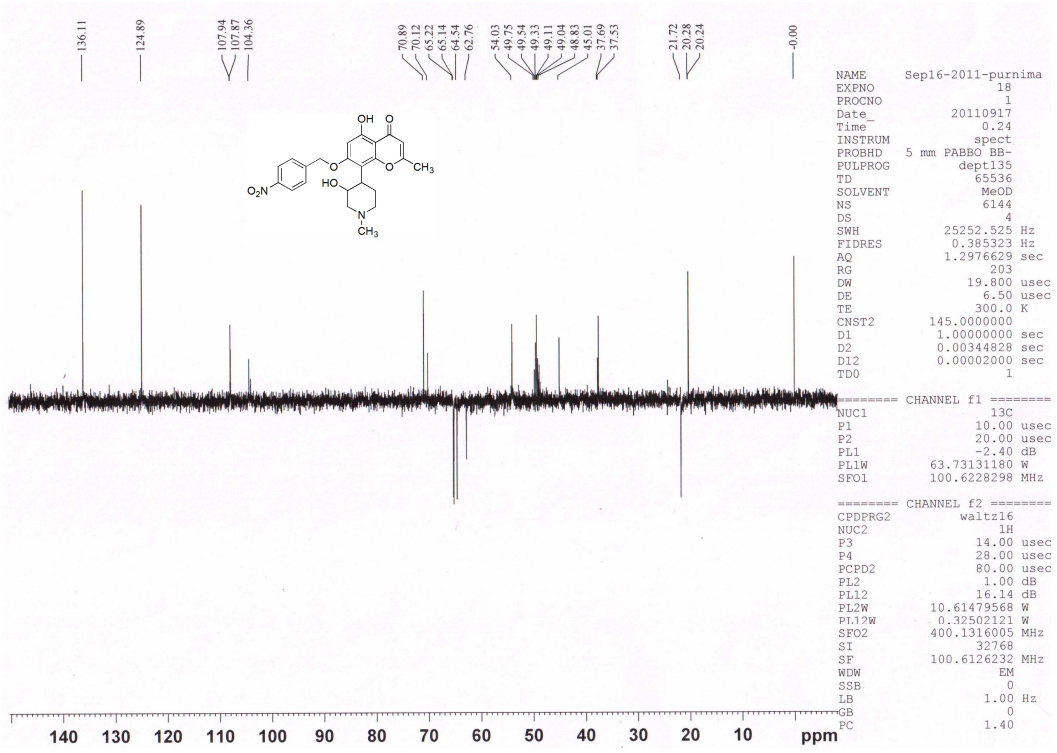
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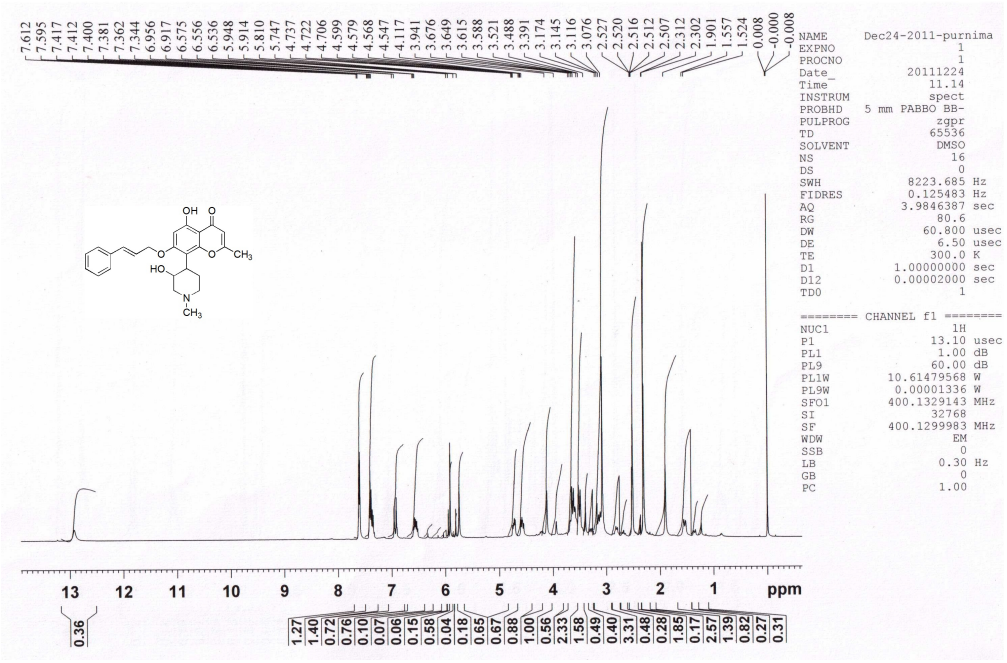
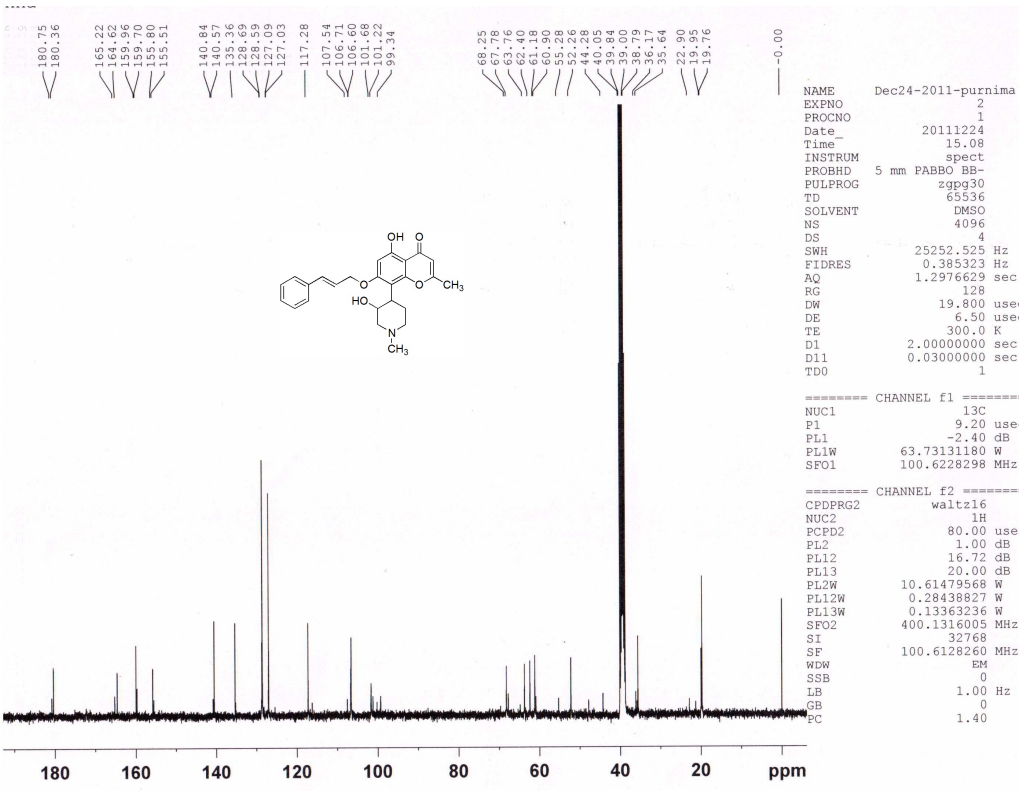
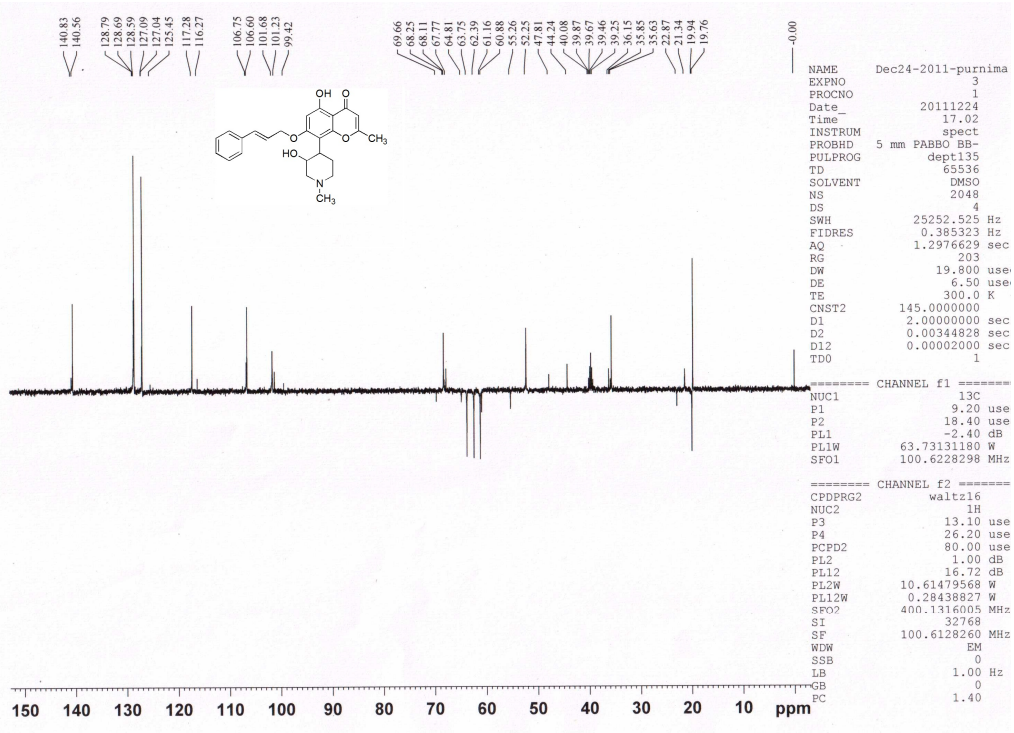
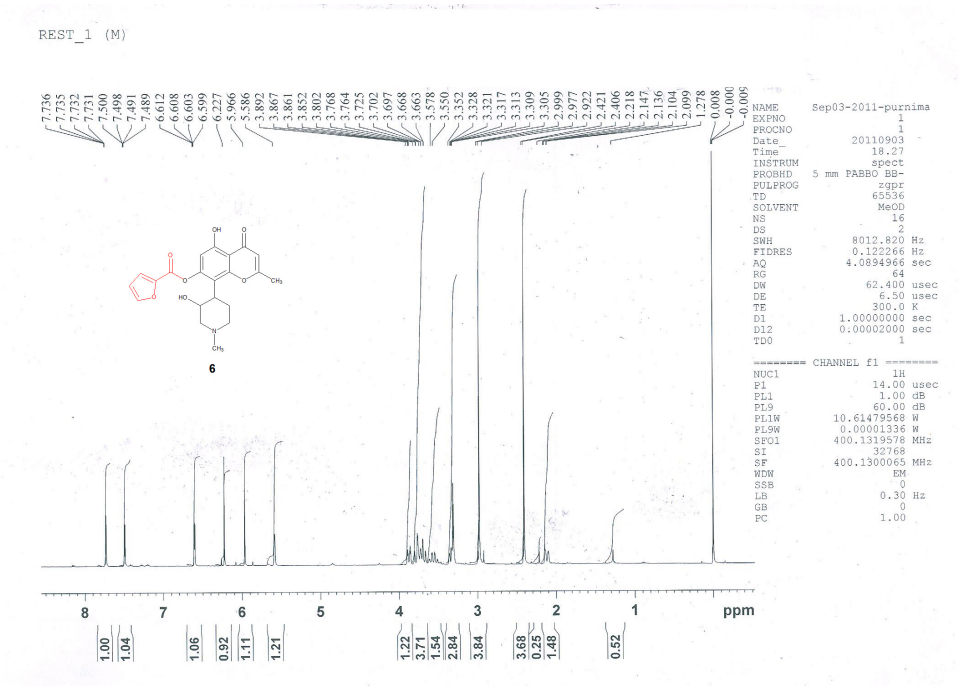
3. ^{1}H NMR, ^{13}C NMR and DEPT-135 spectrum of 7-(cinnamyloxy)-5-hydroxy-8-(3-hydroxy-1-methylpiperidin-4-yl)-2-methyl-4H-chromen-4-one (**2f**) in DMSO-d_6

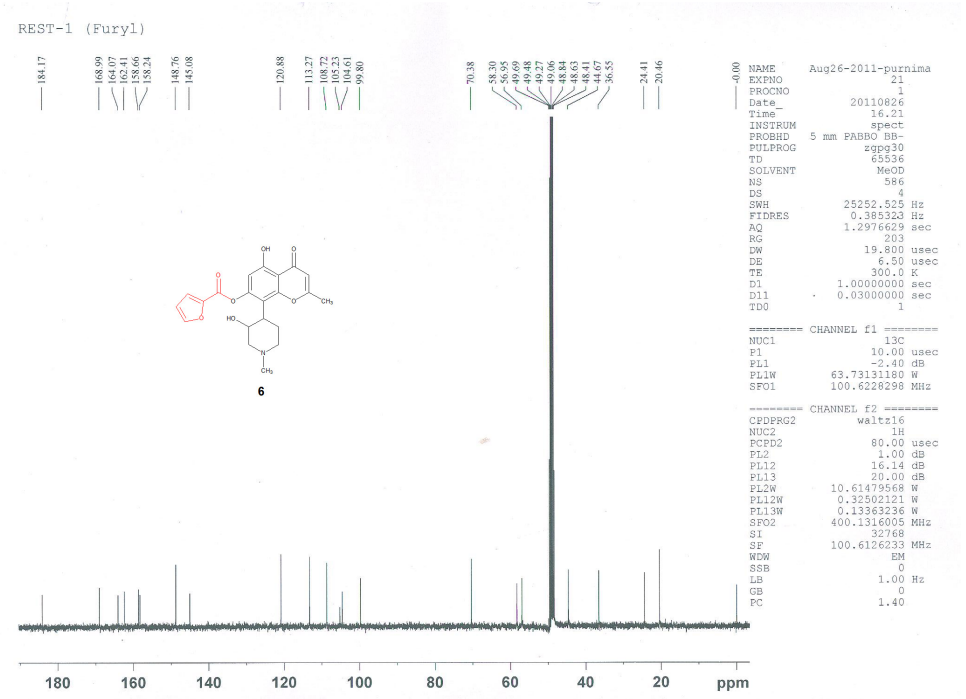






4. ^{1}H NMR, ^{13}C NMR and DEPT-135 spectrum of 5-hydroxy-8-(3-hydroxy-1-methylpiperidin-4-yl)-2-methyl-4-oxo-4H-chromen-7-yl-furan-2-carboxylate (**2h**) in CD_3OD

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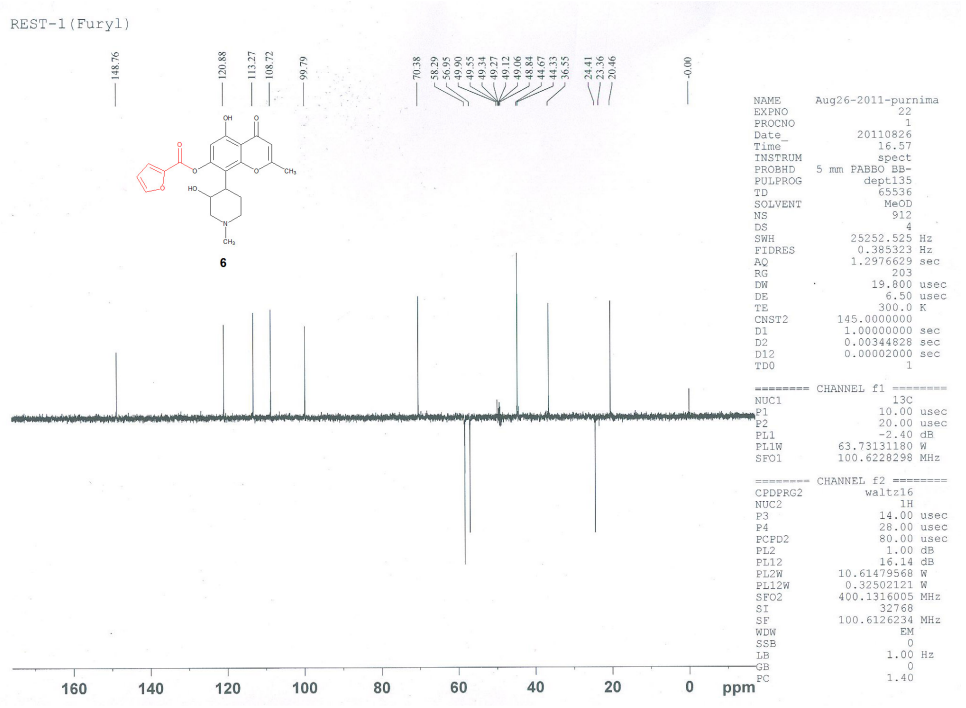
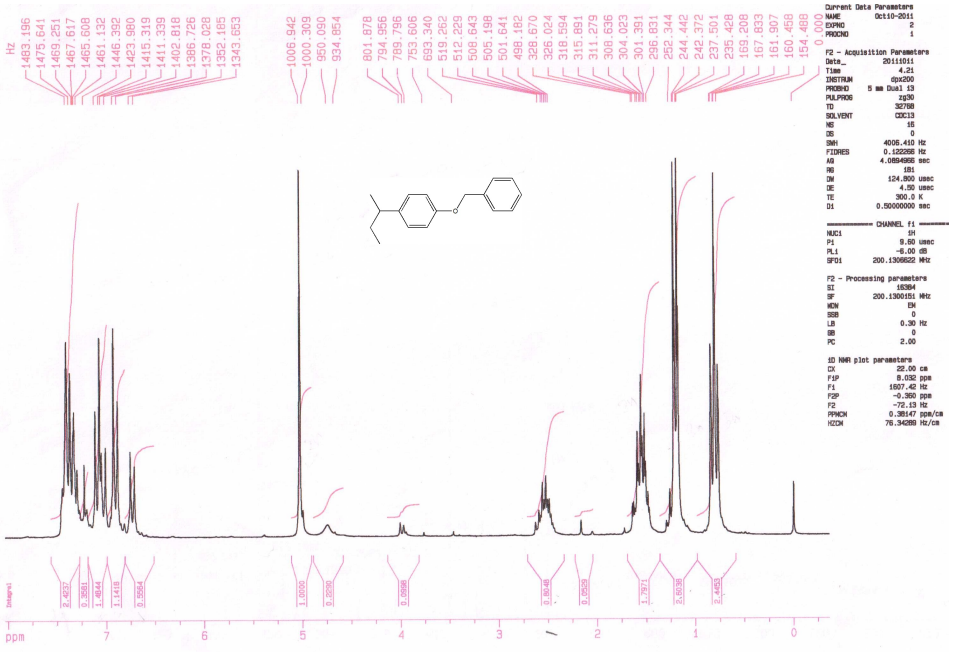
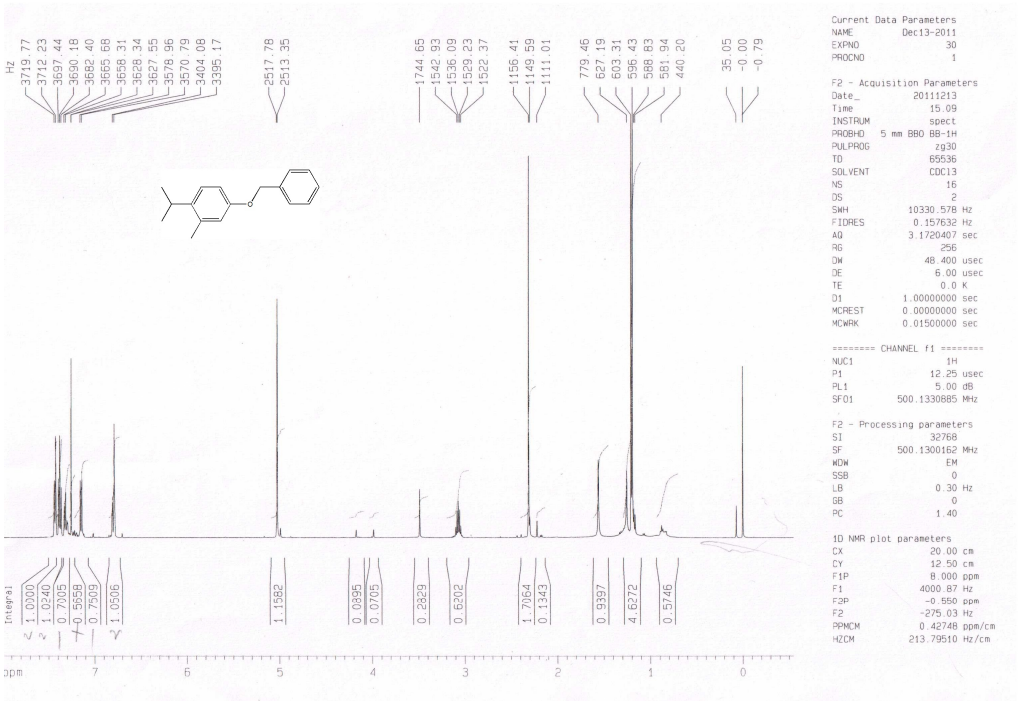
99.80
70.38
58.30
56.95
49.69
49.48
49.27
49.06
48.84
48.63
48.41
44.67
36.55
24.41
20.46
-0.00
6
NAME Aug26-2011-purnima
EXPNO 21
PROCNO 1
Date_ 20110826
Time 16.21
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 586
DS 4
SWH 25252.525 Hz
FIDRES 0.385323 Hz
AQ 1.2976629 sec
RG 203
DW 19.800 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 -2.40 dB
PL1W 63.73131180 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 16.14 dB
PL13 20.00 dB
PL2W 10.61479568 W
PL12W 0.32502121 W
PL13W 0.13363236 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6126233 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
180
160
140
120
100
80
60
40
20
ppm


REST-1(Furyl)
148.76
120.88
113.27
108.72
99.79
70.38
58.29
56.95
49.90
49.55
49.34
49.27
49.12
49.06
48.84
44.67
44.33
36.55
24.41
23.36
20.46
-0.00
6
NAME Aug26-2011-purnima
EXPNO 22
PROCNO 1
Date_ 20110826
Time 16.57
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG dept135
TD 65536
SOLVENT MeOD
NS 912
DS 4
SWH 25252.525 Hz
FIDRES 0.385323 Hz
AQ 1.2976629 sec
RG 203
DW 19.800 usec
DE 6.50 usec
TE 300.0 K
CNST2 145.0000000
D1 1.00000000 sec
D2 0.00344828 sec
D12 0.00002000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
P2 20.00 usec
PL1 -2.40 dB
PL1W 63.73131180 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
P3 14.00 usec
P4 28.00 usec
PCPD2 80.00 usec
PL2 1.00 dB
PL12 16.14 dB
PL2W 10.61479568 W
PL12W 0.32502121 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6126234 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
160
140
120
100
80
60
40
20
0
ppm

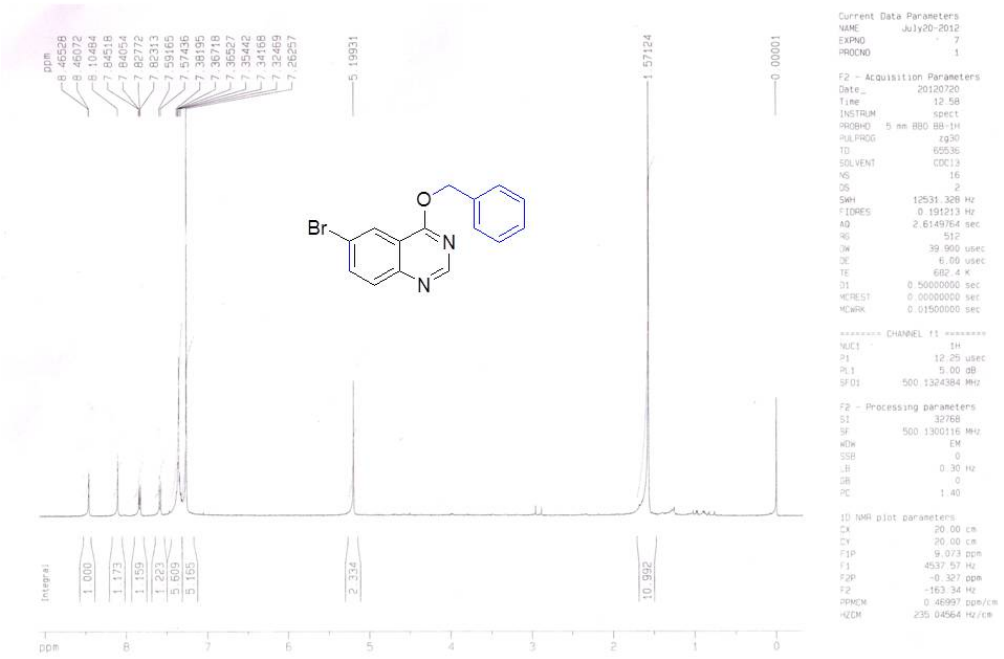
5. ^{1}H NMR of 1-((4-Sec-butylphenoxy) methyl)benzene **(4d)** in $CDCl_3$



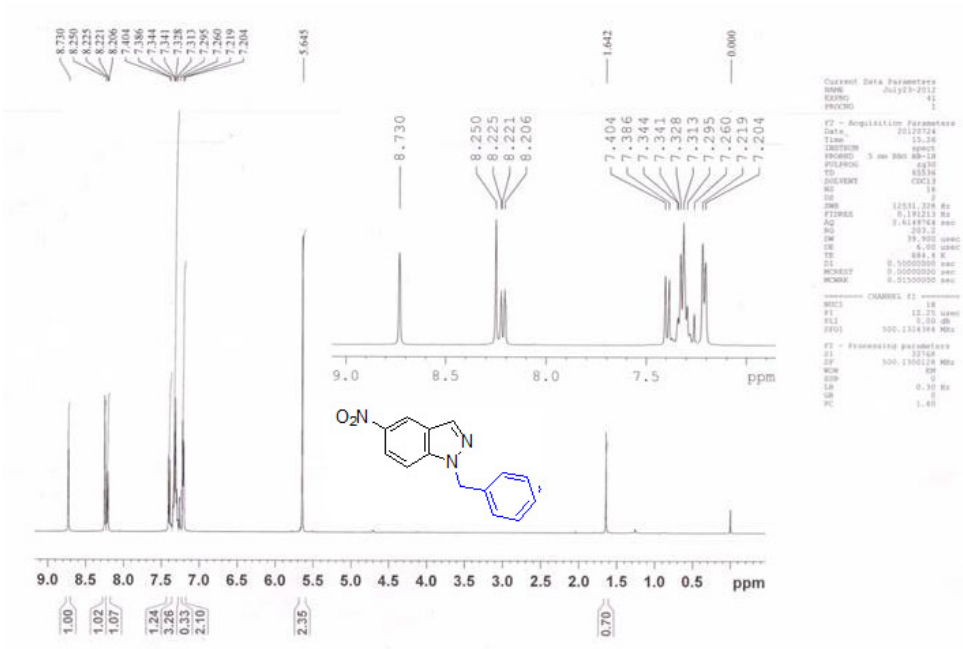
6. ^{1}H NMR of 1-((4-isopropyl-3-methylphenoxy)methyl)benzene **(4e)** in $CDCl_3$

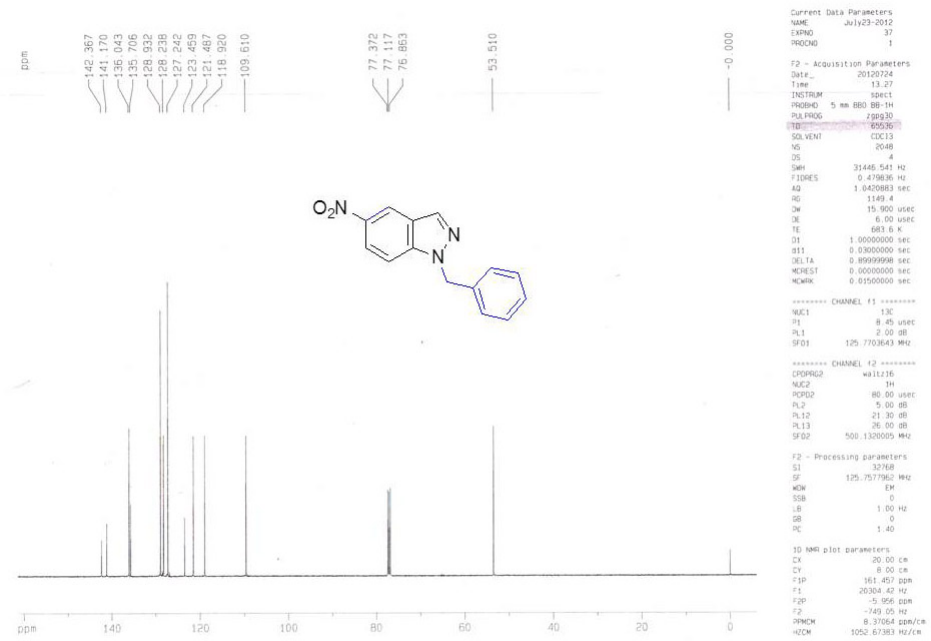


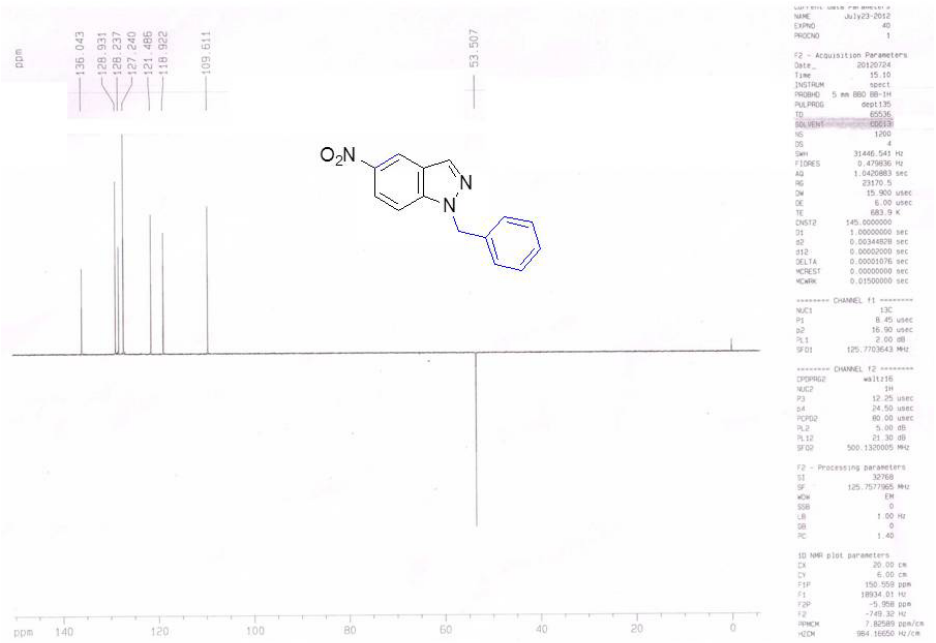
7. ^{1}H NMR of 4-(benzyloxy)-6-bromoquinazoline (**6d**) in $CDCl_3$

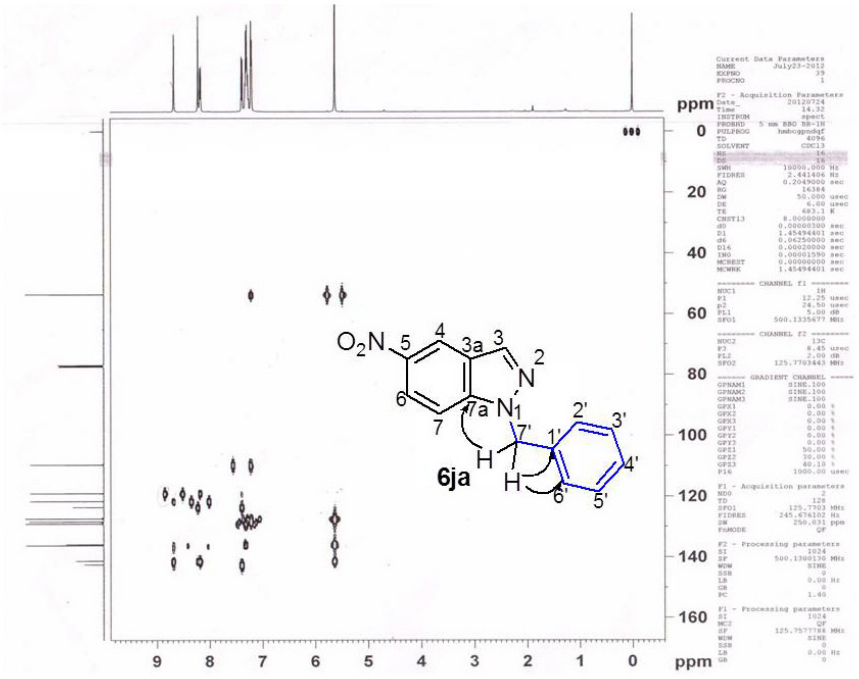


8. ^{1}H, ^{13}C, DEPT135 NMR and HMBC of 1-benzyl-5-nitro-1H-indazole (**6ja**) in $CDCl_3$

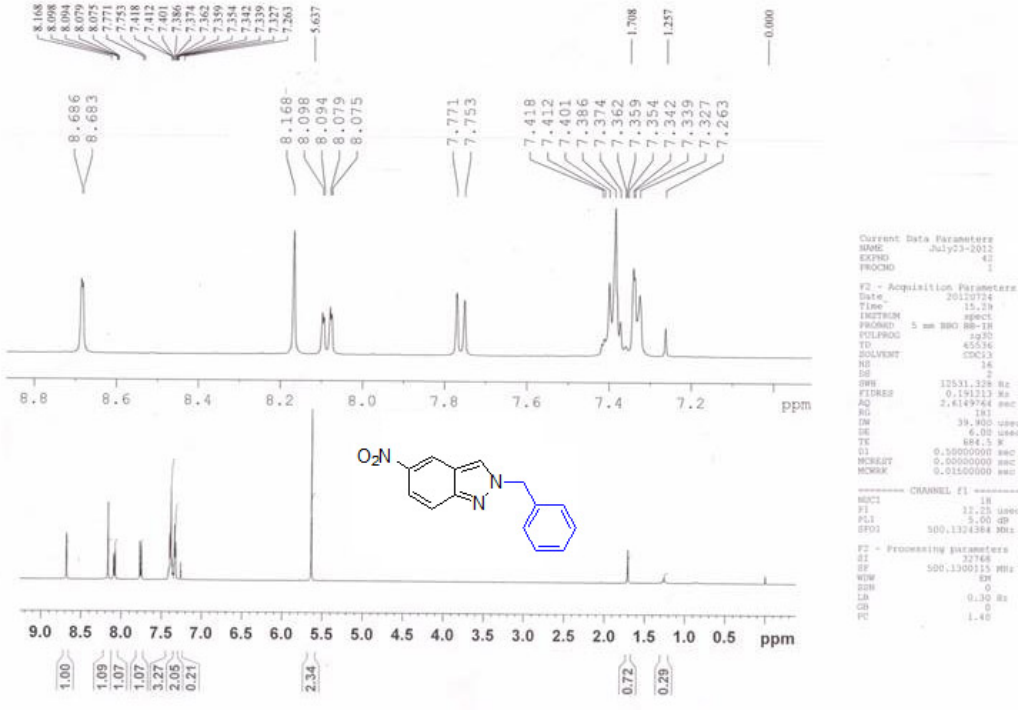


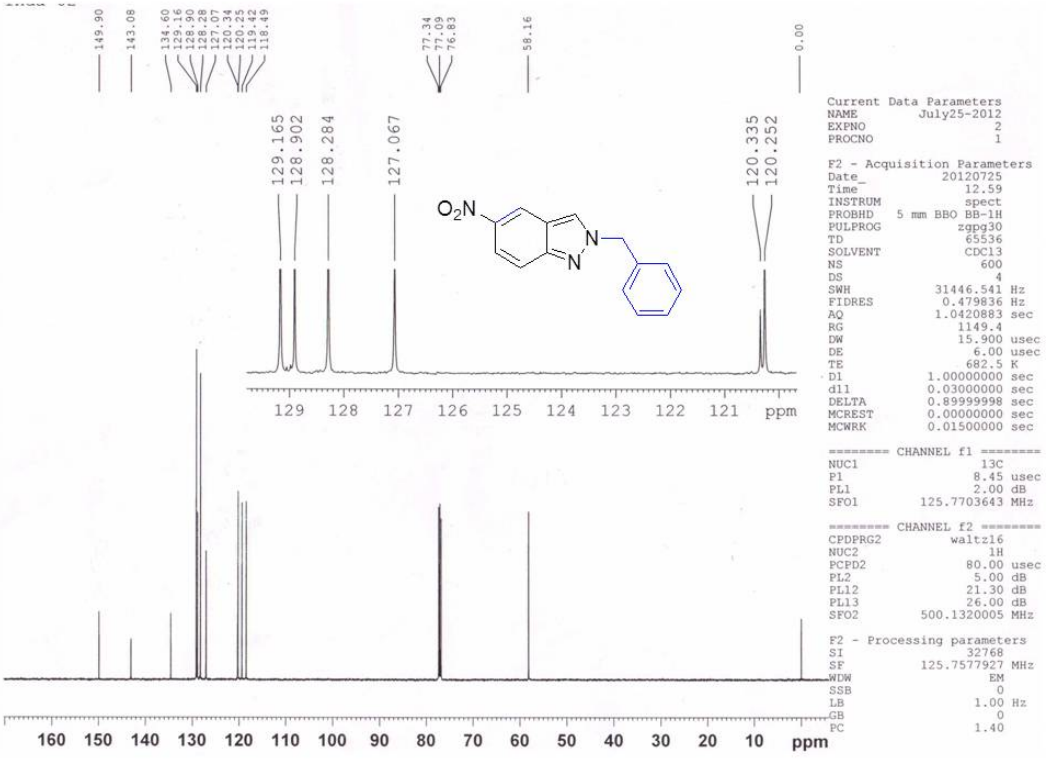

142.367
141.170
136.043
135.706
128.932
128.238
127.242
123.459
121.467
118.920
109.610
77.372
77.117
76.863
53.510
-0.000
O2N
N
N
ppm
140
120
100
80
60
40
20
0

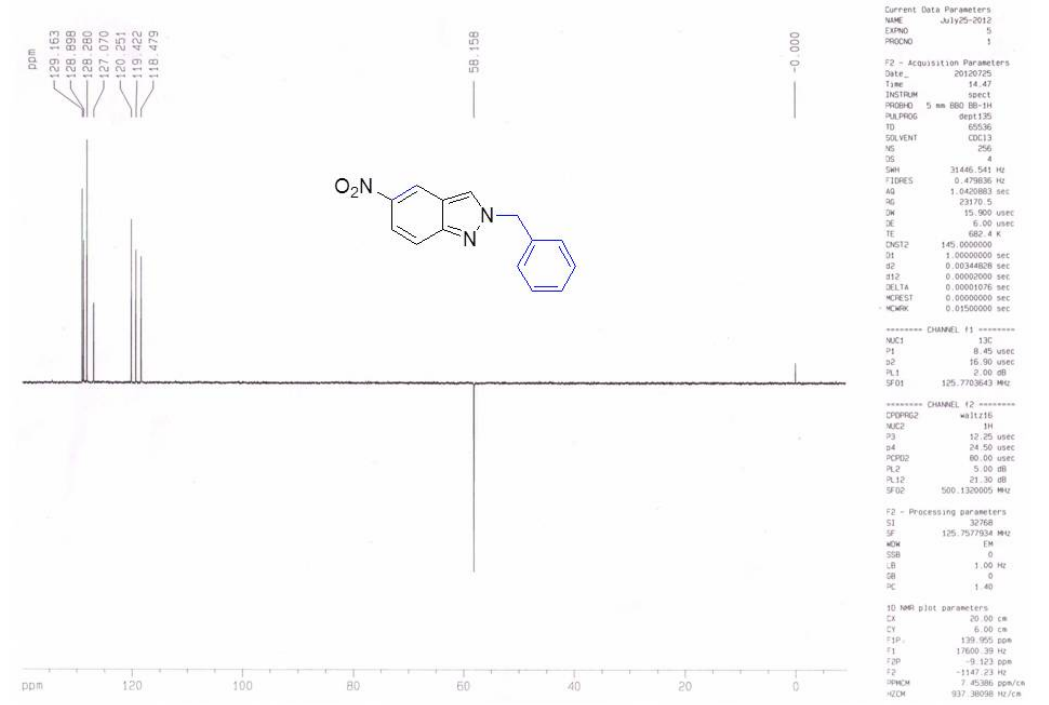

136.043
128.931
128.237
127.240
121.486
118.922
109.611
53.507
O2N
N
N
ppm
140
120
100
80
60
40
20
0

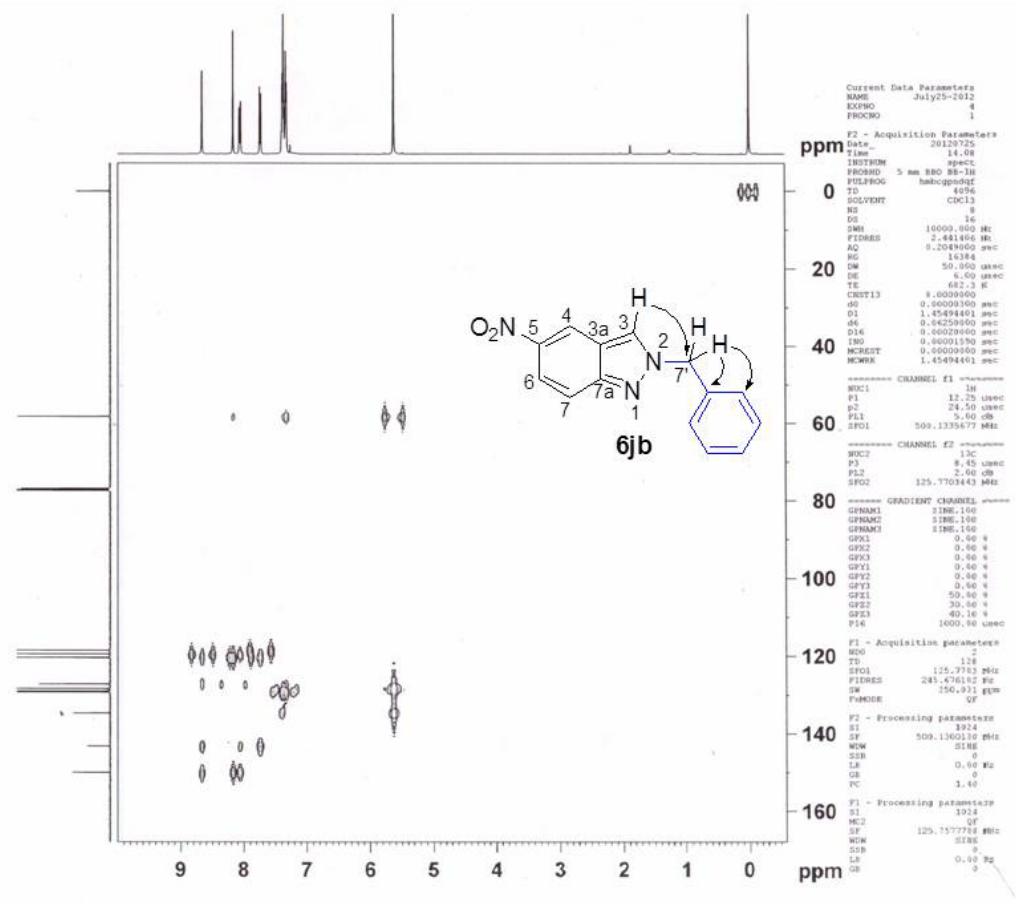


9. ^{1}H, ^{13}C, DEPT135 NMR and HMBC of 2-benzyl-5-nitro-2H-indazole (**6jb**) in $CDCl_3$




149.90
143.08
134.60
129.16
128.90
128.28
127.07
120.34
120.25
119.42
118.49
77.34
77.09
76.83
58.16
0.00
129.165
128.902
128.284
127.067
120.335
120.252
O2N
N
N
129 128 127 126 125 124 123 122 121 ppm
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm
Current Data Parameters
NAME July25-2012
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120725
Time 12.59
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 600
DS 4
SWH 31446.541 Hz
FIDRES 0.479836 Hz
AQ 1.0420883 sec
RG 1149.4
DW 15.900 usec
DE 6.00 usec
TE 682.5 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 8.45 usec
PL1 2.00 dB
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 5.00 dB
PL12 21.30 dB
PL13 26.00 dB
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 32768
SF 125.7577927 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


ppm
129.163
128.898
128.280
127.070
120.251
119.422
118.479
58.158
-0.000
O2N
N
N
ppm 120 100 80 60 40 20 0
Current Data Parameters
NAME July25-2012
EXPNO 5
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120725
Time 14.47
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG dept135
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 31446.541 Hz
FIDRES 0.479836 Hz
AQ 1.0420883 sec
RG 23170.5
DW 15.900 usec
DE 6.00 usec
TE 682.4 K
CNST2 145.0000000
D1 1.00000000 sec
d2 0.00344828 sec
d12 0.00002000 sec
DELTA 0.00001076 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 8.45 usec
p2 16.90 usec
PL1 2.00 dB
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
P3 12.25 usec
p4 24.50 usec
PCPD2 80.00 usec
PL2 5.00 dB
PL12 21.30 dB
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 32768
SF 125.7577934 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
CY 6.00 cm
F1P 139.955 ppm
F1 17600.39 Hz
F2P -9.123 ppm
F2 -1147.23 Hz
PPMCM 7.45386 ppm/cm
HZCM 937.38098 Hz/cm


ppm
0
20
40
60
80
100
120
140
160
9
8
7
6
5
4
3
2
1
0
ppm
O2N
5
4
3a
3
H
2
N
H
H
7'
6
7
7a
N
1
6jb