

**An expedient solvent-free C-benylation of 4-hydroxycoumarin with styrenes**

**Rana Chatterjee, Anindita Mukherjee, Sougata Santra, Grigory V. Zyryanov, Oleg N. Chupakhin and Adinath Majee**

**General Information:**

<sup>1</sup>H NMR spectra were determined on a Bruker 400 (400 MHz) spectrometer as solutions in CDCl<sub>3</sub>. Chemical shifts are expressed in parts per million (δ) and the signals were reported as s (singlet), d (doublet), t (triplet), m (multiplet) and coupling constants *J* were given in Hz. <sup>13</sup>C NMR spectra were recorded at 100 MHz in CDCl<sub>3</sub> solution. Chemical shifts are expressed in parts per million (δ) and are referenced to CDCl<sub>3</sub> (δ = 77.16) as internal standard. TLC was done on silica gel coated glass slide (Merck, Silica gel G for TLC). Silica gel (60-120 mesh, SRL, India) was used for column chromatography. Petroleum ether refers to the fraction boiling in the range of 60-80 °C unless otherwise mentioned. All solvents were dried and distilled before use. Commercially available substrates were freshly distilled before the reaction. Solvents, reagents and chemicals were purchased from Aldrich, Merck, and Spectrochem Chemicals. All reactions involving moisture-sensitive reactants were executed using oven-dried glassware.

**General procedure for the synthesis of compounds 3a-h:**

A mixture of 4-hydroxycoumarin **1** (1, 162 mg, 1 mmol), styrene **2a-h** (1.5 mmol) and TsOH (0.034 g, 20 mol%) was stirred at 80 °C for 4-6 h (TLC control). After completion, the reaction mixture was cooled and extracted with ethyl acetate (3×15 ml) and water (2×10 ml). The organic layer was separated and dried over Na<sub>2</sub>SO<sub>4</sub>. After evaporating the solvent, the residue was purified by column chromatography on silica gel using petroleum ether/ethyl acetate (6→8%) to afford the pure products.

To the best of our knowledge, in the present study, we have encountered one new compound **3g** which was fully characterized by elemental analysis, <sup>1</sup>H and <sup>13</sup>C NMR data. For known compounds (**3a**,<sup>S1</sup> **3b**,<sup>S2</sup> **3c**,<sup>S1</sup> **3d**,<sup>S1,S2</sup> **3e**,<sup>S1,S2</sup> **3f**<sup>S3</sup> and **3h**<sup>S4</sup>), the data were cross-checked with literature and found matching with reported analytical data.

**4-Hydroxy-3-(1-phenylethyl)-2H-chromen-2-one (3a):** Yield: 207 mg (78%); mp: 203-205 °C (lit.<sup>S1</sup> 204-205 °C). <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz):  $\delta$  7.65-7.63 (m, 1H, ArH), 7.53-7.49 (m, 3H, ArH), 7.47-7.43 (m, 2H, ArH), 7.38-7.31 (m, 2H, ArH), 7.24-7.20 (m, 1H, ArH), 5.92 (s, 1H, OH), 4.76-4.71 (m, 1H, CH), 1.66 (d,  $J = 7.2$  Hz, 3H, CH<sub>3</sub>); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz):  $\delta$  163.6, 159.8, 152.6, 141.6, 132.0, 129.9, 128.1, 127.4, 124.0, 123.0, 116.5, 116.2, 110.2, 34.7, 16.6. Anal. Calcd. For C<sub>17</sub>H<sub>14</sub>O<sub>3</sub>: C, 76.68; H, 5.30%; Found: C, 76.78; H, 5.34%.

**4-Hydroxy-3-[1-(*o*-tolyl)ethyl]-2H-chromen-2-one (3b):** Yield: 196 mg (70%), mp: 163-165 °C. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz):  $\delta$  7.71-7.65 (m, 2H, ArH), 7.55-7.50 (m, 1H, ArH), 7.45-7.41 (m, 1H, ArH), 7.37-7.29 (m, 3H, ArH), 7.26-7.22 (m, 1H, ArH), 6.37 (s, 1H, OH), 4.73-4.68 (m, 1H, CH), 2.27 (s, 3H, CH<sub>3</sub>), 1.72 (d,  $J = 7.2$  Hz, 3H, CH<sub>3</sub>); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz):  $\delta$  163.2, 159.8, 152.6, 140.1, 139.0, 132.1, 131.8, 128.3, 127.6, 124.7, 123.9, 123.0, 116.4, 116.1, 108.2, 33.4, 19.7, 17.4. Anal. Calcd. For C<sub>18</sub>H<sub>16</sub>O<sub>3</sub>: C, 77.12; H, 5.75%; Found: C, 77.04; H, 5.78%. NMR data are close to literature.<sup>S2</sup>

**4-Hydroxy-3-[1-(*p*-tolyl)ethyl]-2H-chromen-2-one (3c):** Yield: 210 mg (75%); mp: 166-168 °C (lit.<sup>S1</sup> 165-166 °C). <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz):  $\delta$  7.70-7.68 (m, 1H, ArH), 7.52-7.47 (m, 1H, ArH), 7.38 (d,  $J = 8$  Hz, 2H, ArH), 7.30-7.27 (m, 1H, ArH), 7.24-7.20 (m, 3H, ArH), 6.45 (s, 1H, OH), 4.72-4.66 (m, 1H, CH), 2.35 (s, 3H, CH<sub>3</sub>), 1.65 (d,  $J = 7.2$  Hz, 3H, CH<sub>3</sub>); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz):  $\delta$  163.7, 159.9, 152.6, 138.5, 137.7, 131.8, 130.5, 127.3, 123.9, 123.0, 116.4, 116.3, 110.2, 34.3, 21.1, 16.7. Anal. Calcd. For C<sub>18</sub>H<sub>16</sub>O<sub>3</sub>: C, 77.12; H, 5.75%; Found: C, 77.17; H, 5.82%

**4-Hydroxy-3-[1-(4-methoxyphenyl)ethyl]-2H-chromen-2-one (3d):** Yield: 227 mg (77%); mp: 168-170 °C (lit.<sup>S1</sup> 169-171 °C). <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz):  $\delta$  7.73-7.71 (m, 1H, ArH), 7.53-7.49 (m, 1H, ArH), 7.43-7.41 (m, 2H, ArH), 7.31-7.29 (m, 1H, ArH), 7.26-7.22 (m, 1H, ArH), 6.96-6.94 (m, 2H, ArH), 6.64 (s, 1H, OH), 4.71-4.65 (m, 1H, CH), 3.82 (s, 3H, OCH<sub>3</sub>), 1.67 (d,  $J = 7.2$  Hz, 3H, CH<sub>3</sub>); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz):  $\delta$  163.6, 159.8, 159.4, 152.7, 133.0, 131.9, 128.6, 123.9, 123.0, 116.5, 116.3, 115.3, 110.2, 55.5, 34.0, 16.9. Anal. Calcd. For C<sub>18</sub>H<sub>16</sub>O<sub>4</sub>: C, 72.96; H, 5.44%; Found: C, 72.90; H, 5.35%. NMR data are close to literature.<sup>S2</sup>

**3-[1-(4-Chlorophenyl)ethyl]-4-hydroxy-2H-chromen-2-one (3e):** Yield: 222 mg (74%); mp: 185-187 °C. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz):  $\delta$  7.72-7.70 (m, 1H, ArH), 7.55-7.50 (m, 1H, ArH), 7.42-7.35 (m, 4H), 7.31-7.27 (m, 1H, ArH), 7.25-7.23 (m, 1H, ArH), 6.36 (s, 1H, OH), 4.71-4.66 (m, 1H, CH), 1.66 (d,  $J = 7.2$  Hz, 3H, CH<sub>3</sub>); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz):  $\delta$  163.5, 159.9, 152.6, 140.4, 133.7, 132.2, 129.7, 128.8, 124.1, 123.0, 116.6, 116.0, 109.8, 34.0, 16.7. Anal. Calcd. For: C<sub>17</sub>H<sub>13</sub>ClO<sub>3</sub>: C, 67.90; H, 4.36%; Found: C, 67.81; H, 4.45%. NMR data are close to literature.<sup>S1,S2</sup>

**3-[1-(3-Bromophenyl)ethyl]-4-hydroxy-2H-chromen-2-one (3f):** Yield: 247 mg (72%); mp: 205-207 °C (lit.<sup>S3</sup> 203-204 °C). <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz):  $\delta$  7.72-7.70 (m, 1H, ArH), 7.61-7.60 (m, 1H, ArH), 7.55-7.51 (m, 1H, ArH), 7.46-7.40 (m, 2H, ArH), 7.32-7.23 (m, 3H, ArH), 6.26 (s, 1H, OH), 4.73-4.68 (m, 1H, CH), 1.66 (d,  $J = 7.2$  Hz, 3H, CH<sub>3</sub>); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz):  $\delta$  163.3, 159.8, 152.7, 144.5, 132.2, 131.1, 130.9, 130.6, 126.0, 124.1, 123.9, 122.9, 116.7, 116.0, 109.7, 34.3 16.6. Anal. Calcd. for C<sub>17</sub>H<sub>13</sub>BrO<sub>3</sub>: C, 59.15; H, 3.80%; Found: C, 59.07; H, 3.88%.

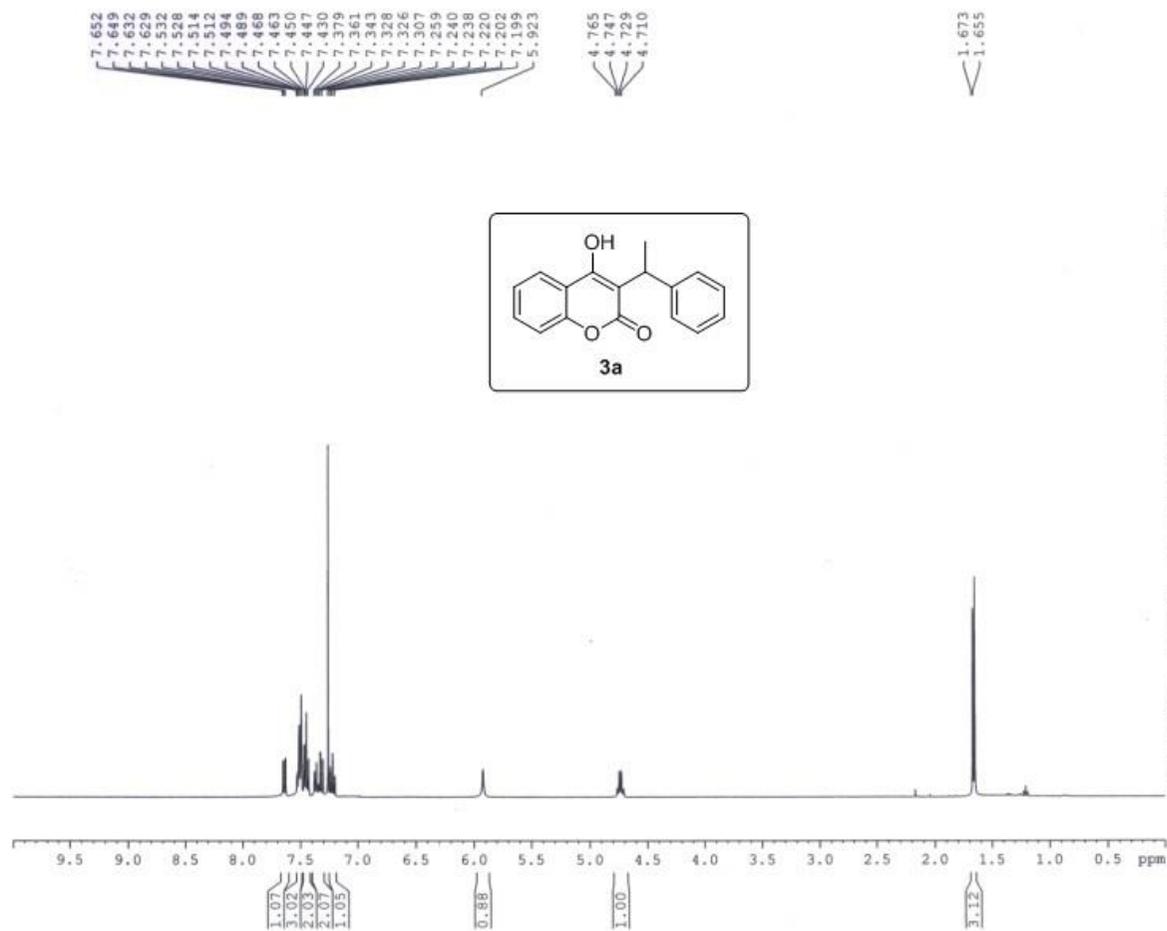
**3-[1-(4-Fluorophenyl)ethyl]-4-hydroxy-2H-chromen-2-one (3g):** 207 mg, mp: 184-186 °C, Yield: 73%; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz):  $\delta$  7.75-7.73 (m, 1H, ArH), 7.60-7.55 (m, 1H, ArH), 7.53-7.49 (m, 2H, ArH), 7.36 (d,  $J = 7.6$  Hz, 1H, ArH), 7.35-7.28 (m, 1H, ArH), 7.19-7.14 (m, 2H, ArH), 6.15 (s, 1H, OH), 4.78-4.72 (m, 1H, CH), 1.71 (d,  $J = 7.2$  Hz, 3H, CH<sub>3</sub>); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz):  $\delta$  162.3 (d,  $J = 246$  Hz), 159.8, 152.6, 137.3 (d,  $J = 2$  Hz), 132.1, 129.1 (d,  $J = 7$  Hz), 124.1, 123.0, 116.7 (d,  $J = 19$  Hz), 116.6, 116.0, 109.9, 34.0, 16.9. Anal. Calcd. for C<sub>17</sub>H<sub>13</sub>FO<sub>3</sub>: C, 71.82; H, 4.61%; Found C, 71.76; H, 4.68%.

**4-Hydroxy-3-(1-phenylpropyl)-2H-chromen-2-one (3h):** Yield: 190 mg (68%); mp: 176-178 °C (lit.<sup>S3</sup> 174-176 °C). <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz):  $\delta$  7.66-7.64 (m, 1H, ArH), 7.53-7.47 (m, 3H, ArH), 7.44-7.40 (m, 2H, ArH), 7.34-7.30 (m, 2H, ArH), 7.24-7.20 (m, 1H, ArH), 6.12 (s, 1H, OH), 4.53 (t,  $J = 15.2$  Hz, 1H, CH), 2.29-2.22 (m, 1H, CH<sub>2</sub>), 2.12-2.05 (m, 1H, CH<sub>2</sub>), 1.08 (t,  $J = 14.4$  Hz, 3H, CH<sub>3</sub>); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz):  $\delta$  163.6, 159.1, 151.0, 138.5, 132.0, 129.9, 127.9, 128.8, 124.0, 122.9, 116.8, 116.5, 108.3, 41.8, 24.1 12.4. Anal. Calcd. for C<sub>18</sub>H<sub>16</sub>O<sub>3</sub>: C, 77.12; H, 5.75%; Found: C, 77.20; H, 5.87%. NMR data are close to literature.<sup>S4</sup>

#### References:

- S1. X. Lin, X. Dai, Z. Mao and Y. Wang, *Tetrahedron*, 2009, **65**, 9233.
- S2. M. Rueping, B. J. Nachtsheim and E. Sugiono, *Synlett*, 2010, **10**, 1549.
- S3. P. Theerthagiri and A. Lalitha, *Tetrahedron Lett.*, 2010, **51**, 5454.
- S4. V. V. Angeles-Dunham, D. M. Nickerson, D. M. Ray and A. E. Mattson, *Angew. Chem., Int. Ed.*, 2014, **53**, 14538.

<sup>1</sup>H of VBRC-306

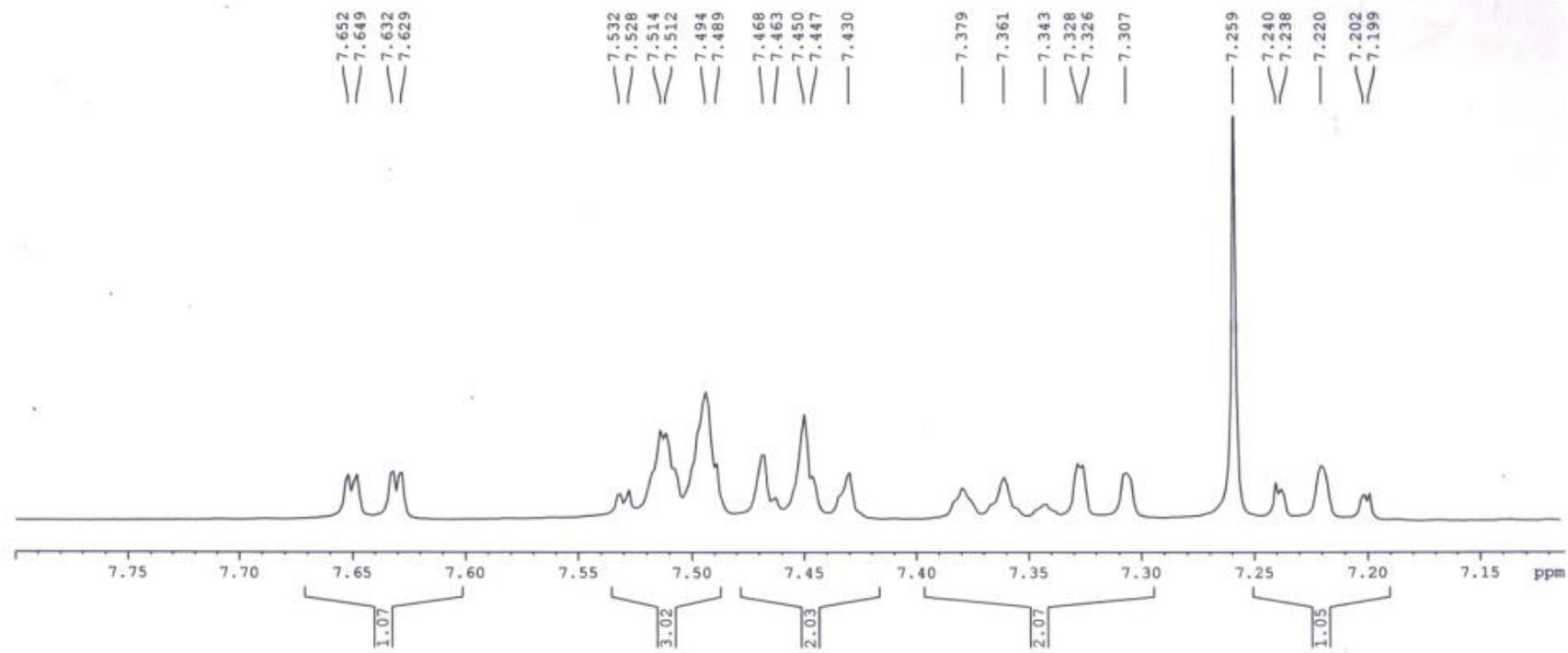


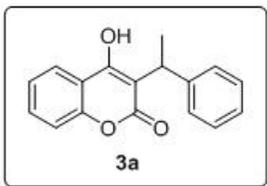
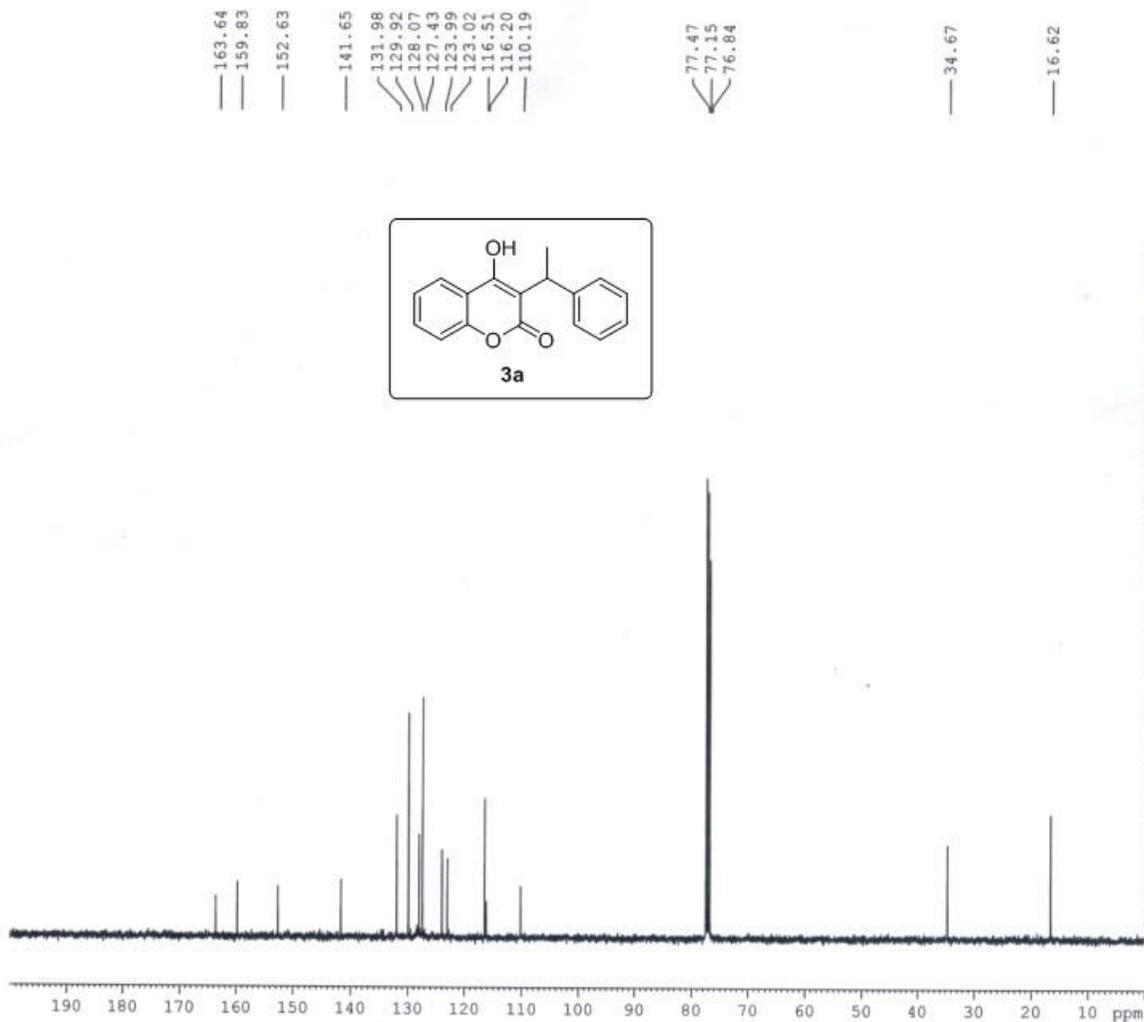
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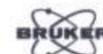
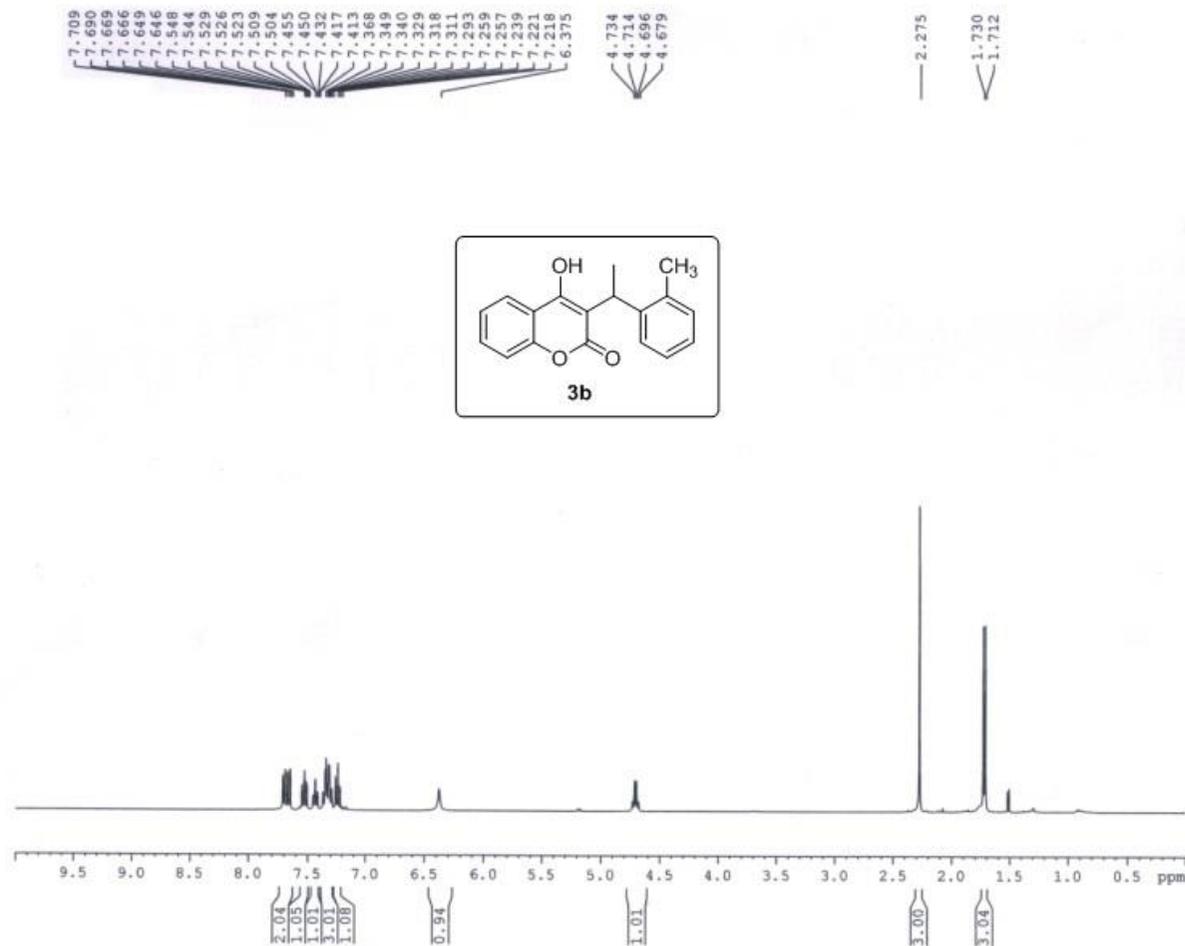
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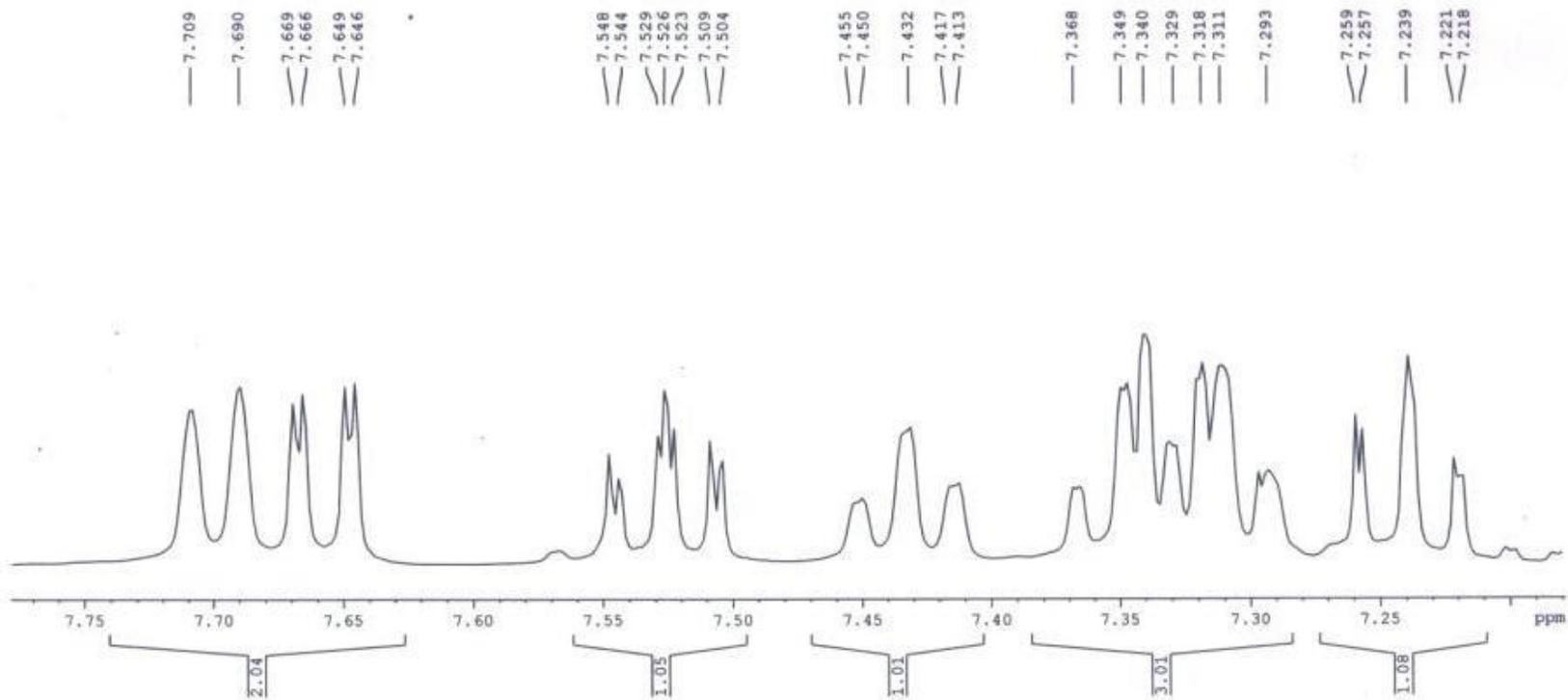


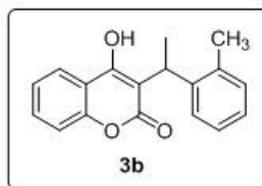
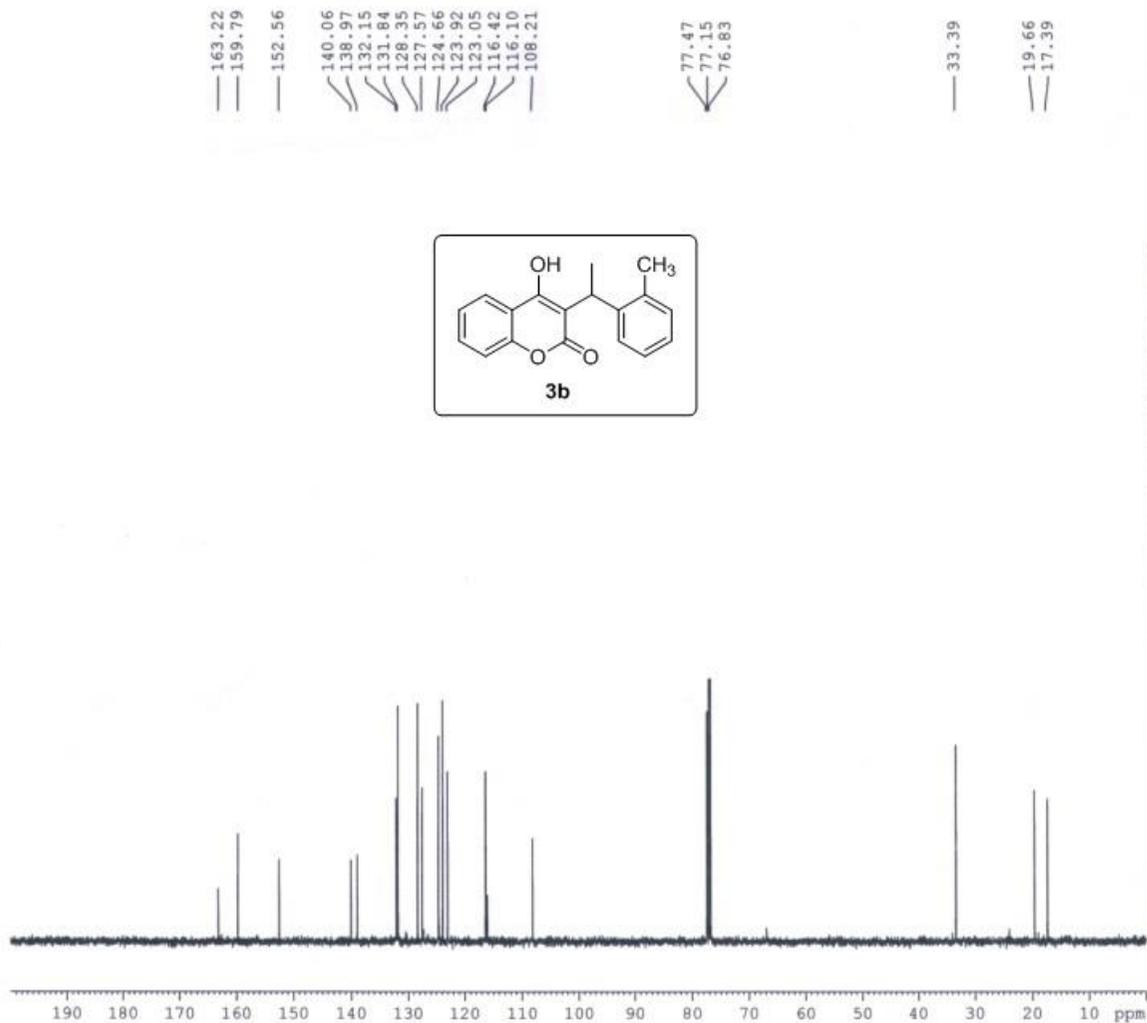
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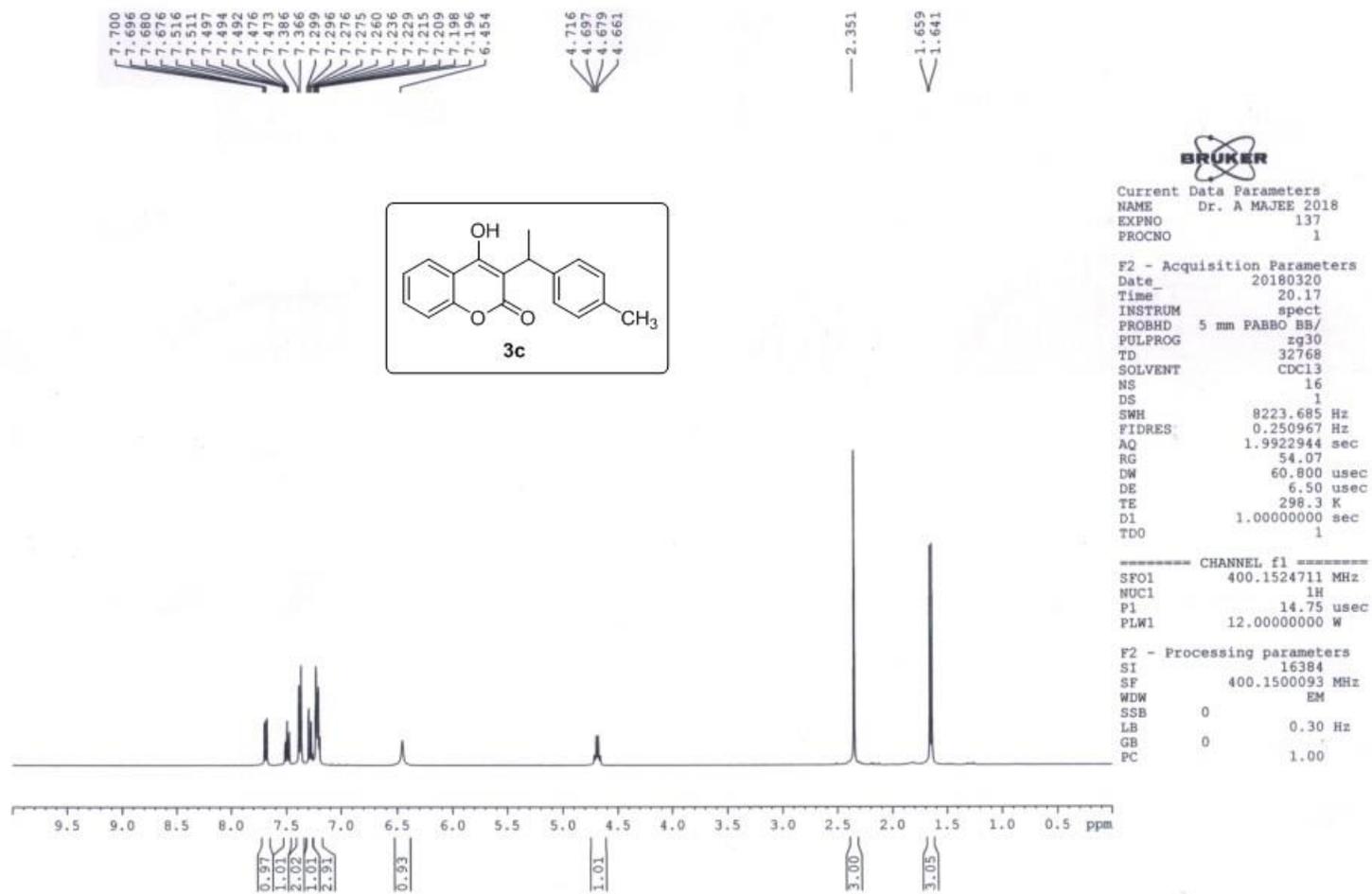
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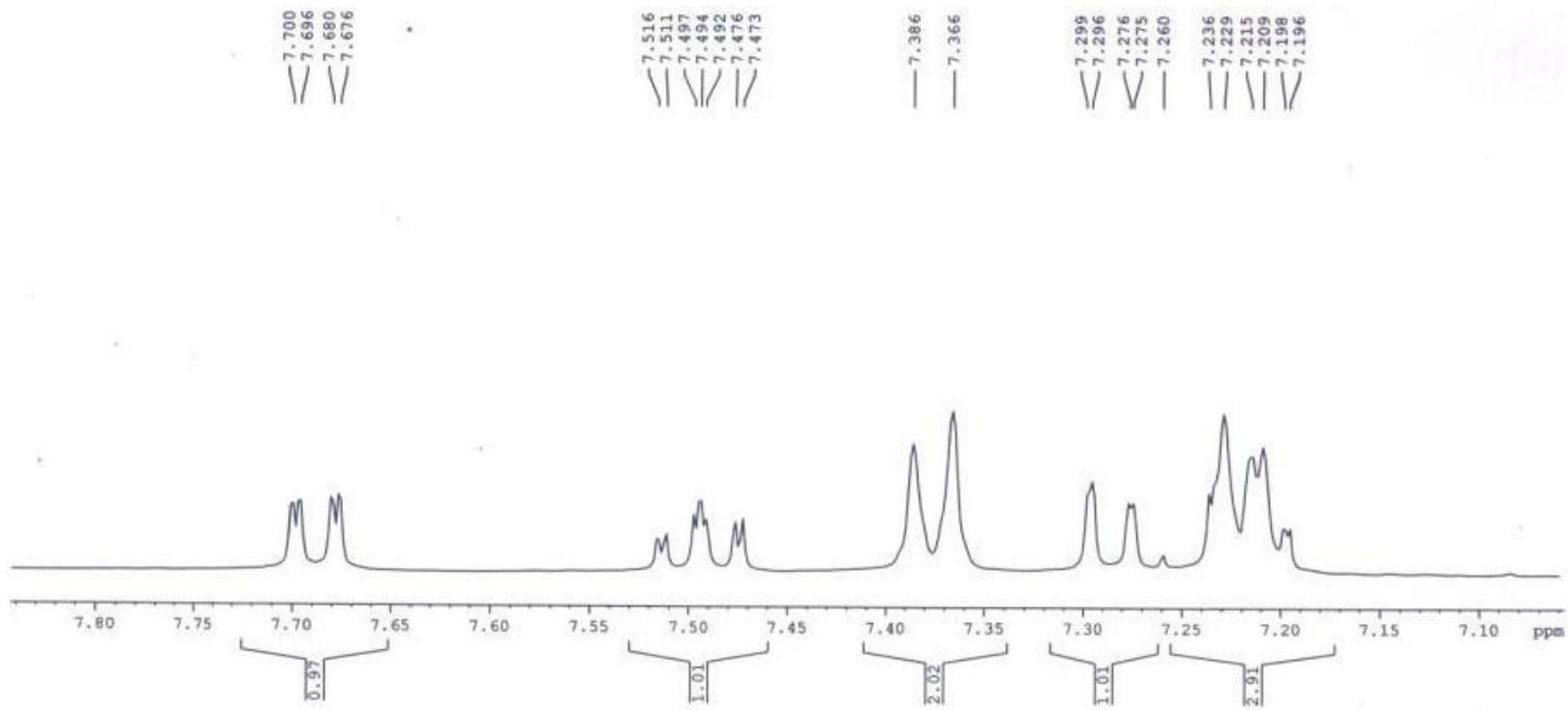
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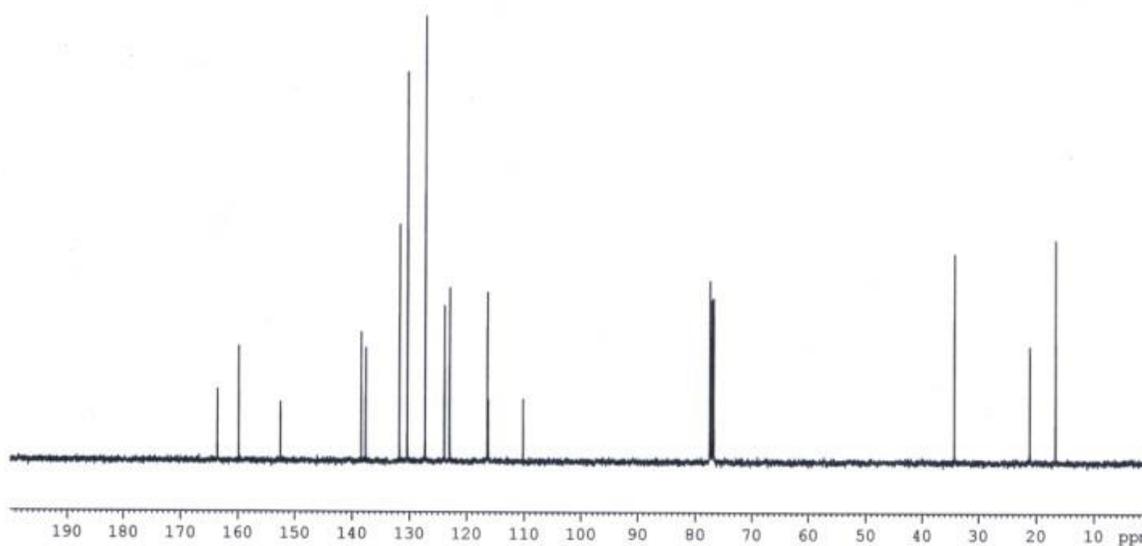
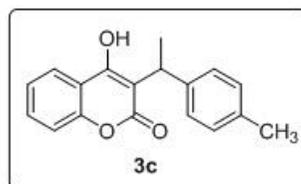
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13C of VBRC-328

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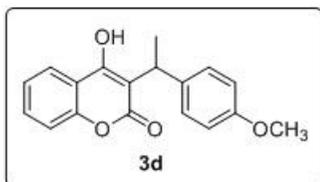
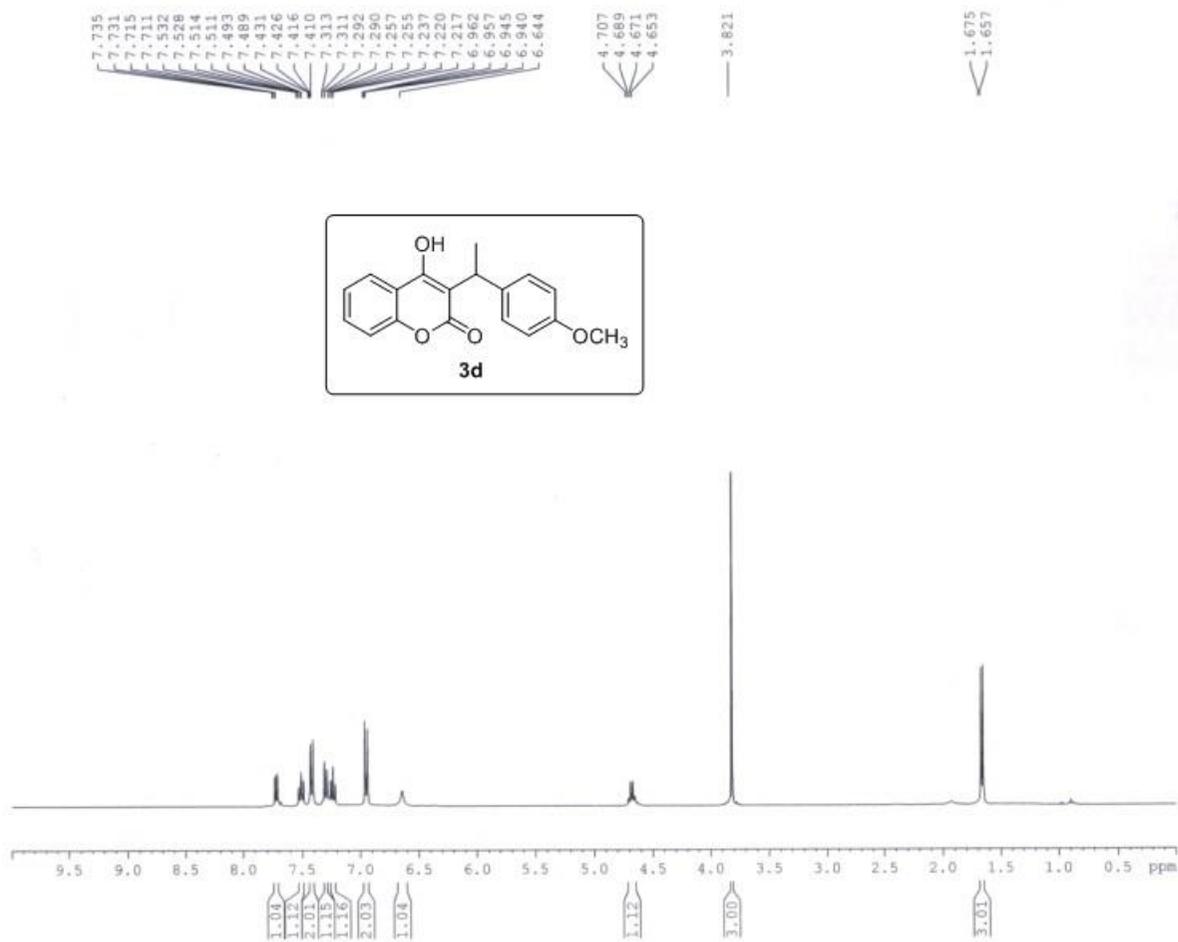
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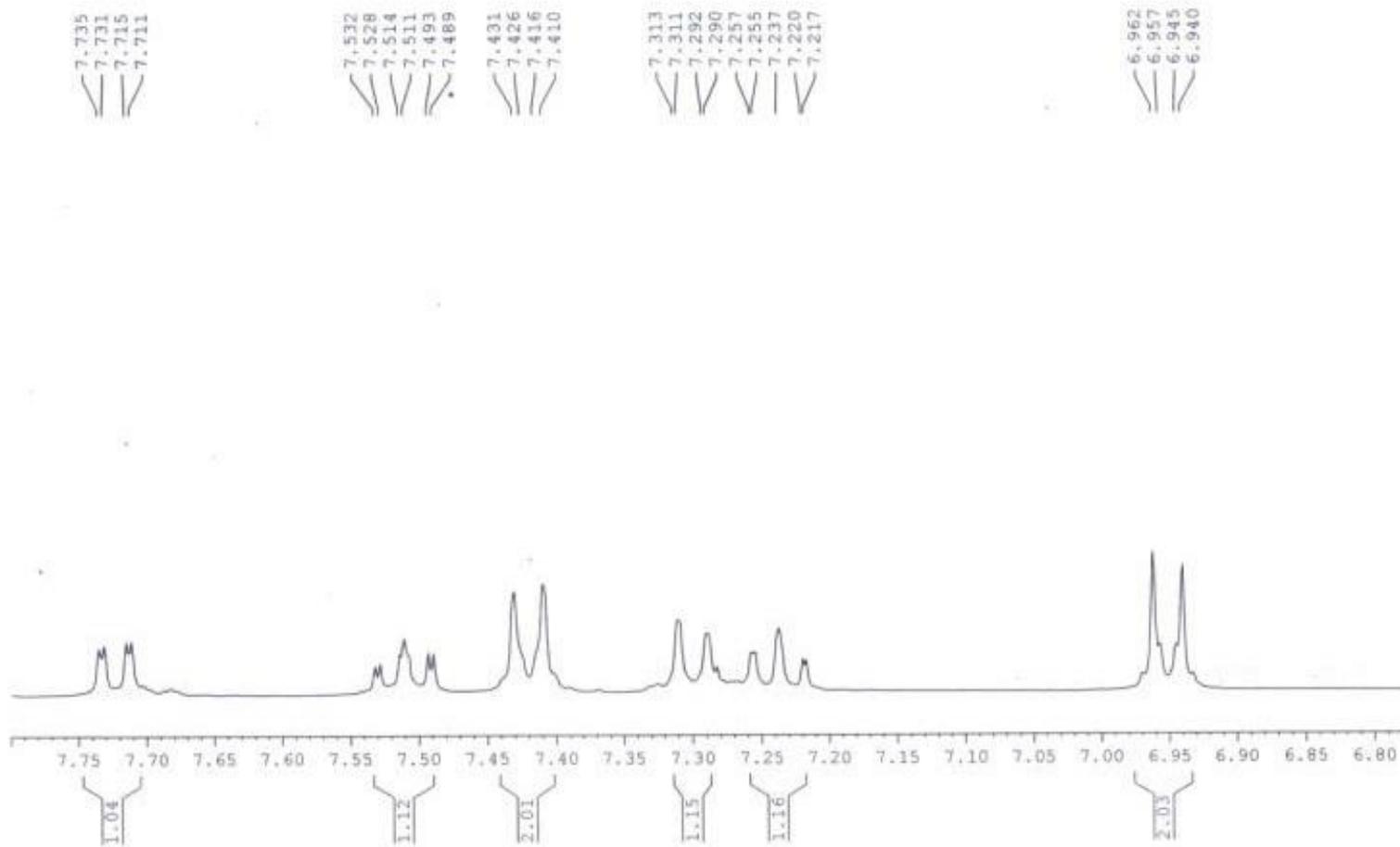


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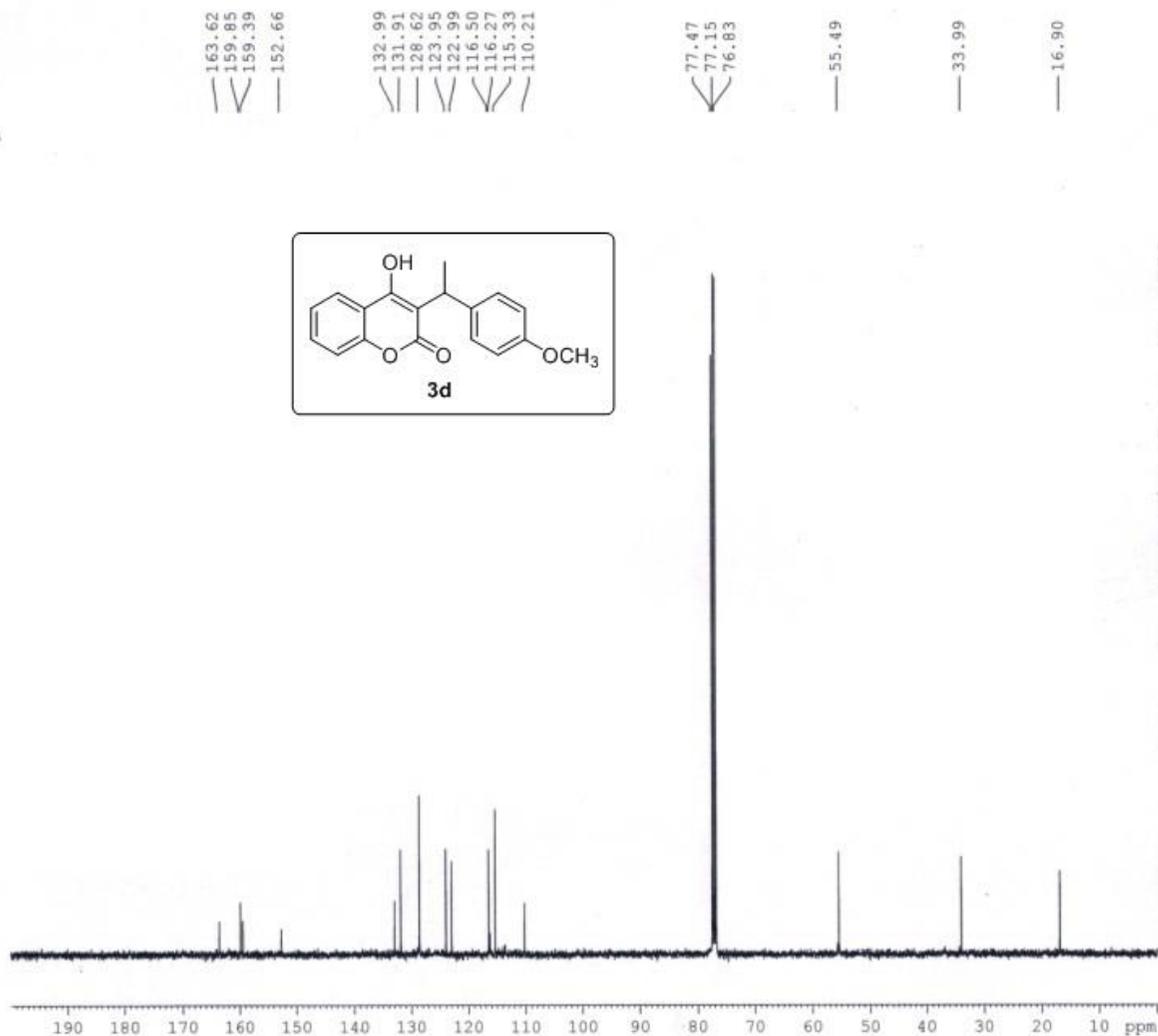
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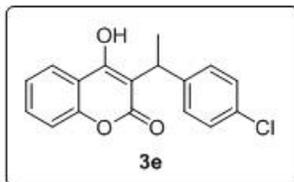
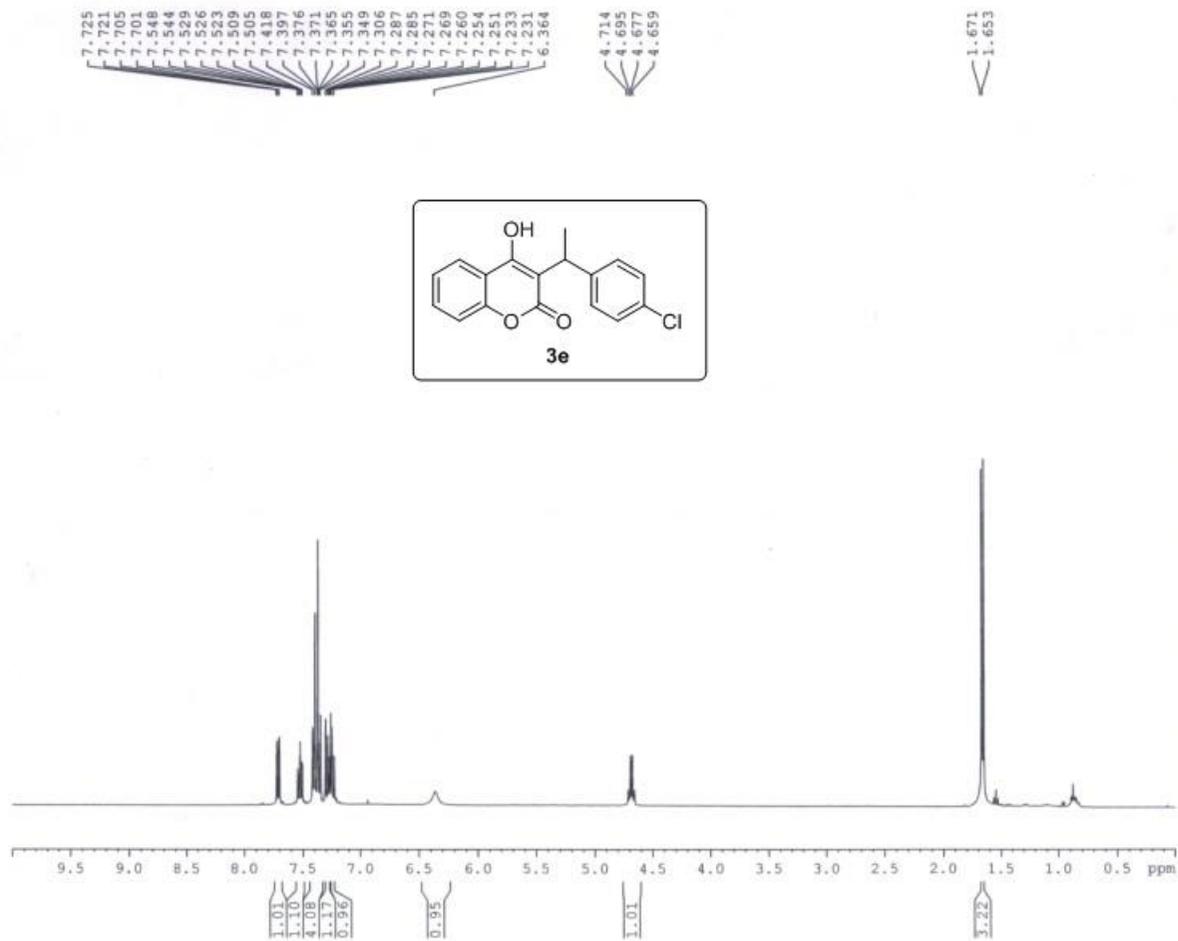
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TD0 1

----- CHANNEL f1 -----  
SFO1 100.6278588 MHz  
NUC1 13C  
P1 8.90 usec  
PLW1 54.0000000 W

----- CHANNEL f2 -----  
SFO2 400.1516006 MHz  
NUC2 1H  
CPDPRG[2] waltz16  
PCPD2 90.00 usec  
PLW2 12.0000000 W  
PLW12 0.32231000 W  
PLW13 0.16212000 W

F2 - Processing parameters  
SI 16384  
SF 100.6177842 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

<sup>1</sup>H of VBRC-327

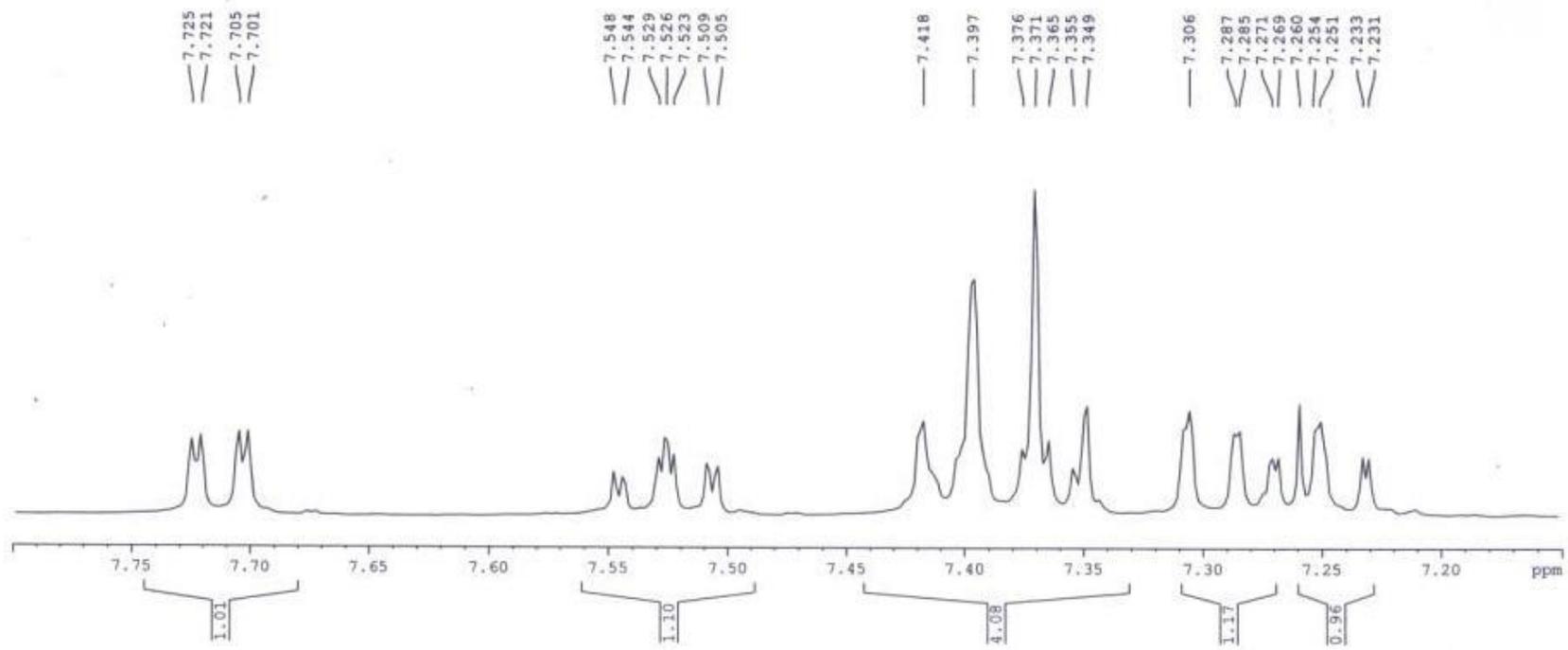


Current Data Parameters  
NAME Dr. A MAJEE 2018  
EXPNO 146  
PROCNO 1

F2 - Acquisition Parameters  
Date 20180223  
Time 17.51  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zg30  
TD 32768  
SOLVENT CDCl3  
NS 16  
DS 1  
SWH 8223.685 Hz  
FIDRES 0.250967 Hz  
AQ 1.9922944 sec  
RG 106.66  
DW 60.800 usec  
DE 6.50 usec  
TE 296.0 K  
D1 1.00000000 sec  
TDO 1

----- CHANNEL f1 -----  
SFO1 400.1524711 MHz  
NUC1 1H  
P1 14.75 usec  
PLW1 12.00000000 W

F2 - Processing parameters  
SI 16384  
SF 400.1500096 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00



13C of VBRC-327



Current Data Parameters  
NAME Dr. A MAJEE 2018  
EXPNO 93  
PROCNO 1

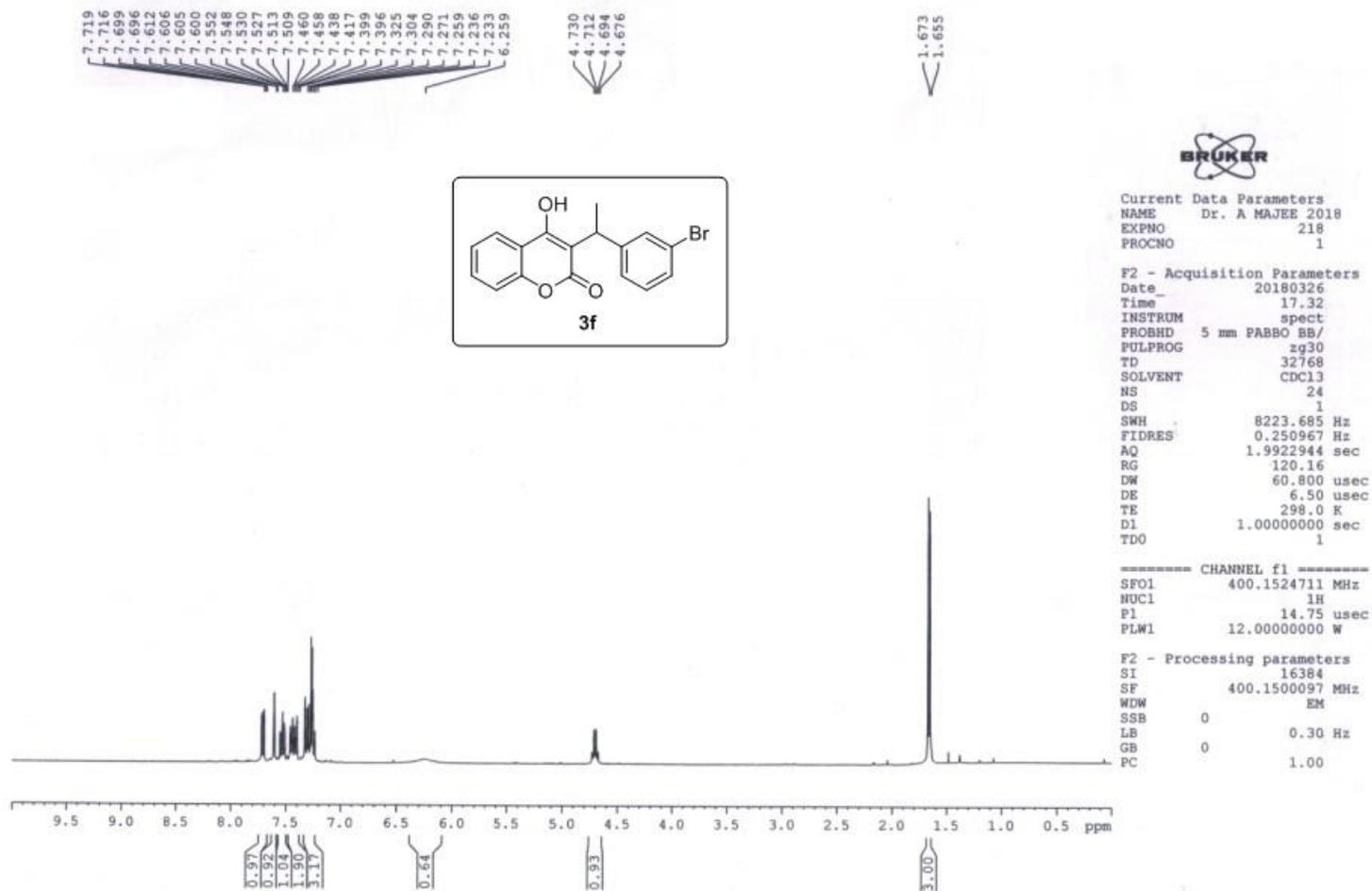
F2 - Acquisition Parameters  
Date\_ 20180223  
Time 18.39  
INSTRUM spect  
PROBHD 5 mm FAPBO BB/  
PULPROG zgpg30  
TD 32768  
SOLVENT CDCl3  
NS 480  
DS 2  
SWH 24038.461 Hz  
FIDRES 0.733596 Hz  
AQ 0.6815744 sec  
RG 186.42  
DW 20.800 usec  
DE 6.50 usec  
TE 296.5 K  
D1 2.00000000 sec  
D11 0.03000000 sec  
TDO 1

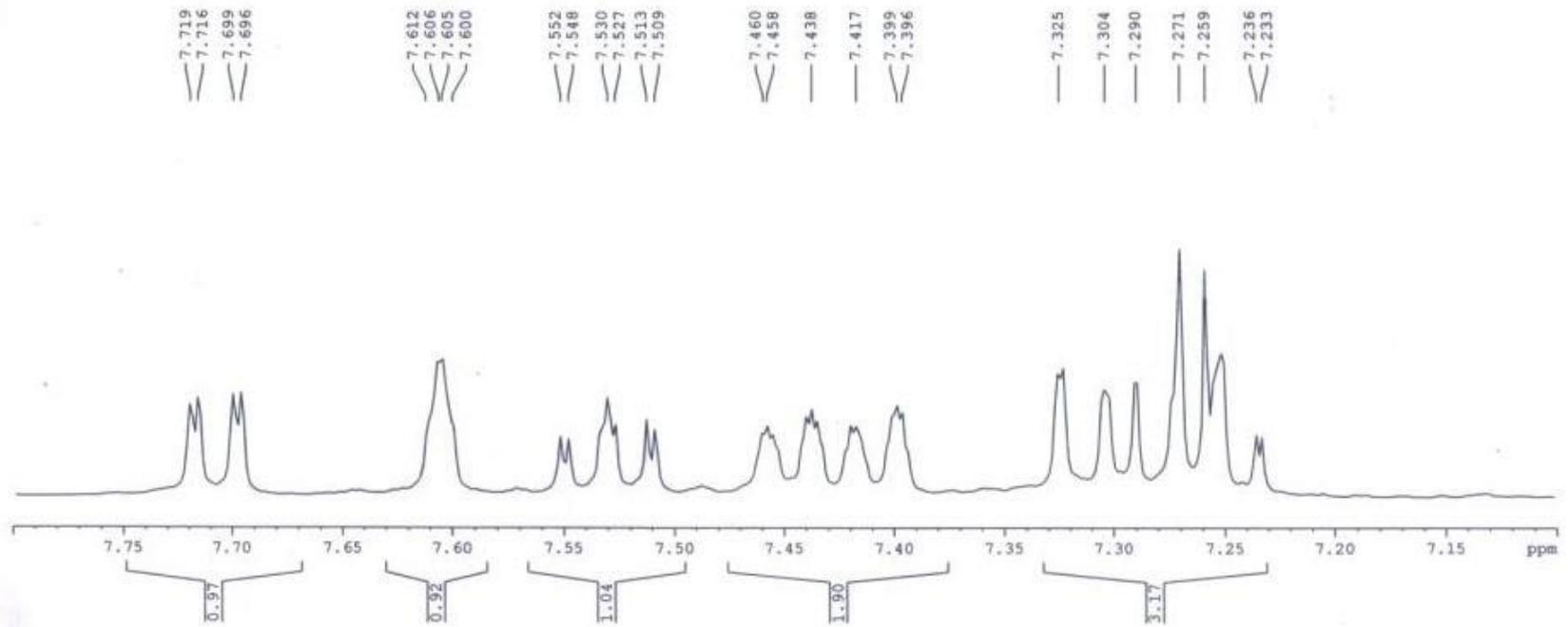
----- CHANNEL f1 -----  
SFO1 100.6278588 MHz  
NUC1 13C  
P1 8.90 usec  
PLW1 54.00000000 W

----- CHANNEL f2 -----  
SFO2 400.1516006 MHz  
NUC2 1H  
CPCPRG[2] waltz16  
PCPD2 90.00 usec  
PLW2 12.00000000 W  
PLW12 0.32231000 W  
PLW13 0.16212000 W

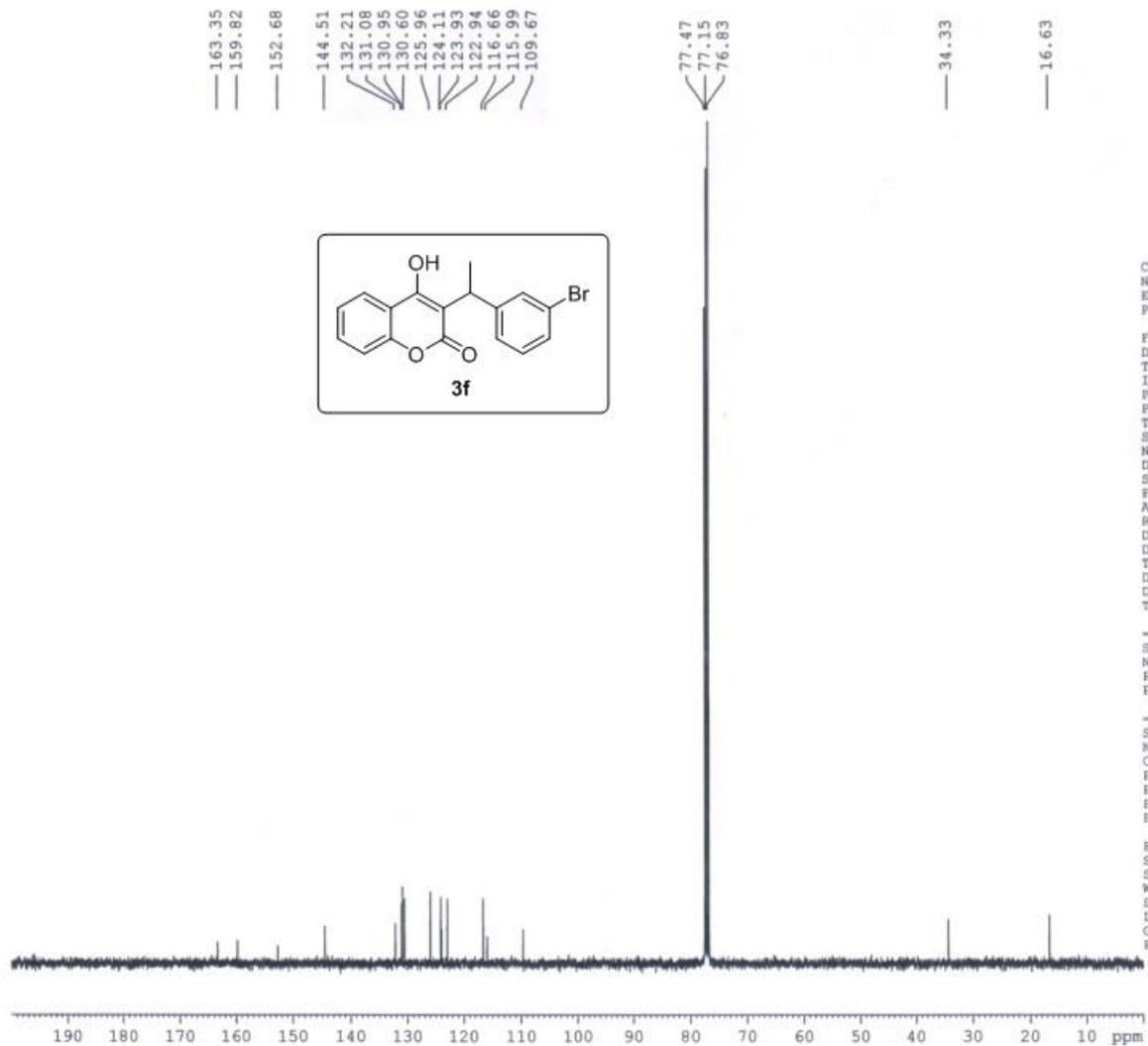
F2 - Processing parameters  
SI 16384  
SF 100.6177857 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

1H of VBRC-339





13C of VBRC-339



Current Data Parameters  
NAME Dr. A MAJEE 2018  
EXPNO 219  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20180326  
Time 18.08  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zgpg30  
TD 32768  
SOLVENT CDCl3  
NS 580  
DS 2  
SWH 24038.461 Hz  
FIDRES 0.733596 Hz  
AQ 0.6815744 sec  
RG 120.16  
DM 20.800 usec  
DE 6.50 usec  
TE 298.7 K  
D1 2.0000000 sec  
D11 0.0300000 sec  
TDO 1

----- CHANNEL f1 -----  
SFO1 100.6278588 MHz  
NUC1 13C  
P1 8.90 usec  
PLW1 54.0000000 W

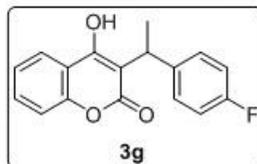
----- CHANNEL f2 -----  
SFO2 400.1516006 MHz  
NUC2 1H  
CPDPRG[2] waltz16  
PCPD2 90.00 usec  
PLW2 12.0000000 W  
PLW12 0.32231000 W  
PLW13 0.16212000 W

F2 - Processing parameters  
SI 16384  
SF 100.6177841 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
CB 0  
PC 1.40

<sup>1</sup>H of VBRC-4-F

7.750  
7.746  
7.740  
7.730  
7.726  
7.598  
7.593  
7.579  
7.576  
7.573  
7.558  
7.554  
7.528  
7.515  
7.511  
7.508  
7.495  
7.371  
7.352  
7.350  
7.316  
7.313  
7.297  
7.286  
7.276  
7.270  
7.194  
7.186  
7.181  
7.165  
7.148  
7.143  
6.150  
4.781  
4.758  
4.741  
4.722

1.727  
1.709

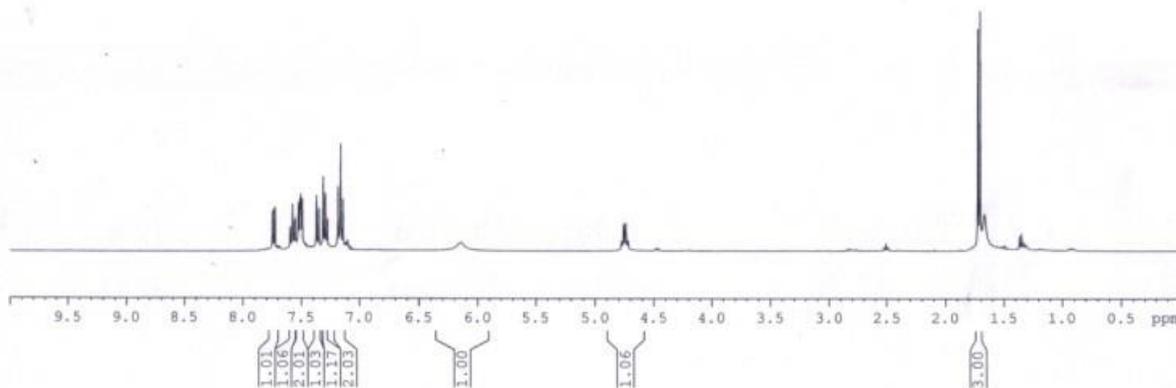


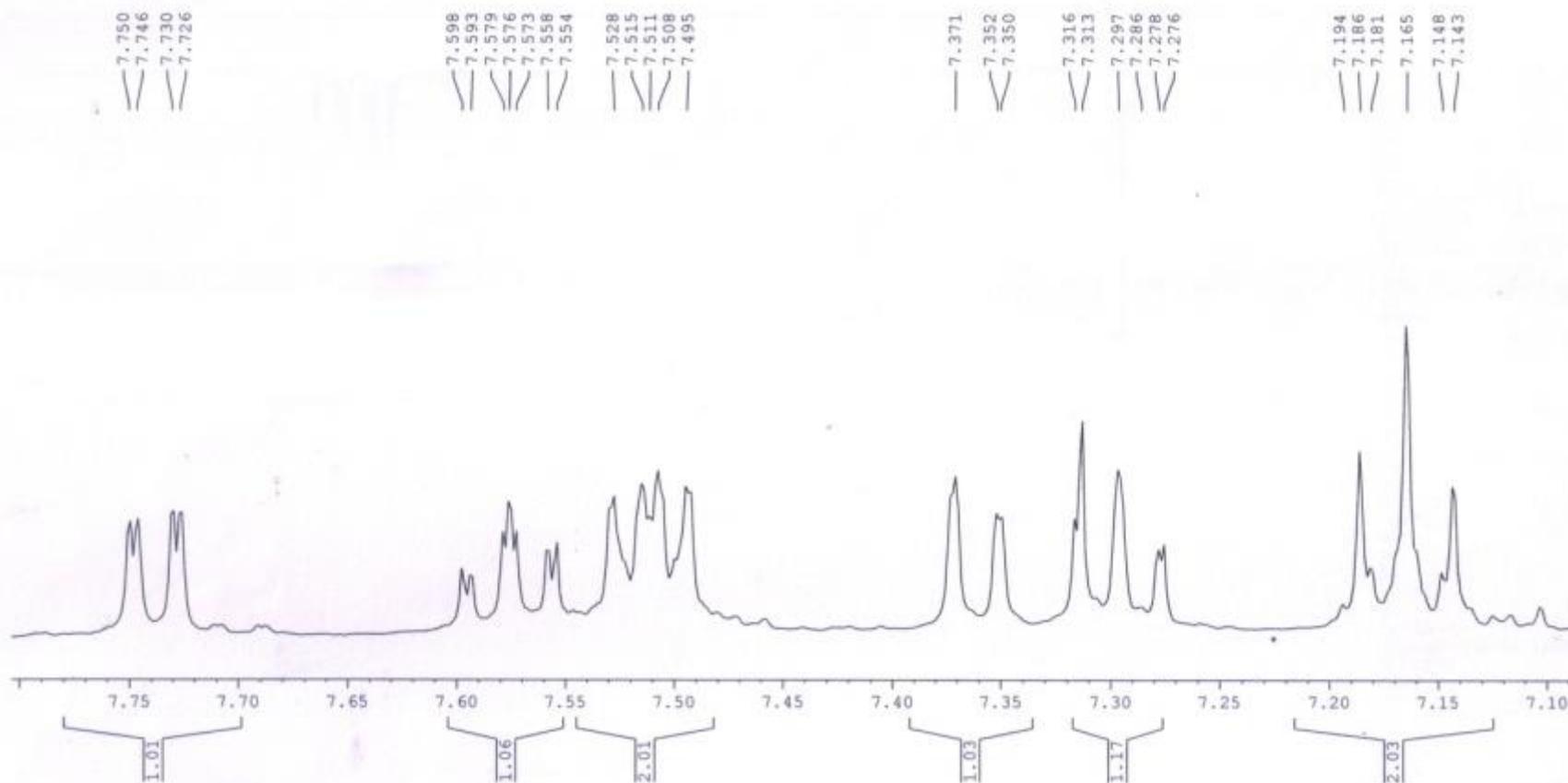
Current Data Parameters  
NAME Dr. A MAJEE 2020  
EXPNO 434  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20201011  
Time 13.23  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zg30  
TD 32768  
SOLVENT CDCl3  
NS 8  
DS 1  
SWH 8223.685 Hz  
FIDRES 0.250967 Hz  
AQ 1.9922944 sec  
RG 148.91  
DW 60.800 usec  
DE 6.50 usec  
TE 299.9 K  
D1 1.00000000 sec  
TD0 1

----- CHANNEL f1 -----  
SF01 400.1524711 MHz  
NUC1 1H  
P1 14.75 usec  
PLW1 12.00000000 W

F2 - Processing parameters  
SI 16384  
SF 400.1499877 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 2.00





13C of VBRC-4-F

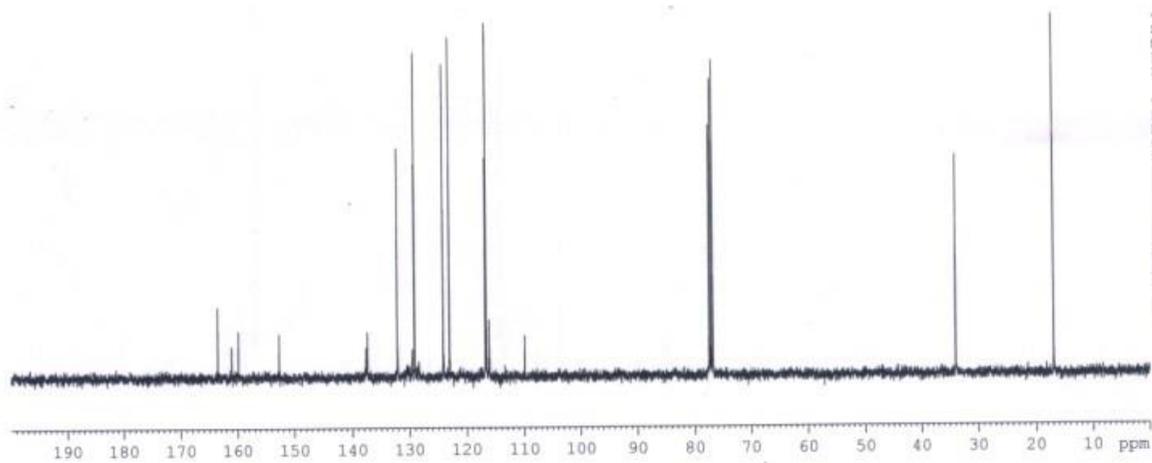
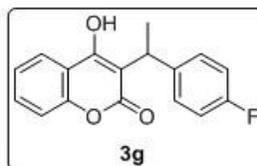
163.51  
161.04  
159.82  
152.65

137.38  
137.36  
132.14  
129.14  
129.07  
124.09  
122.96  
116.77  
116.58  
116.56  
116.04  
109.89

77.47  
77.15  
76.83

33.97

16.93



Current Data Parameters  
NAME Dr. A MAJEE 2020  
EXPCNO 442  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20201014  
Time 11.30  
INSTRUM spect  
PROBHD 5 mm FAPBO BB/  
PULPROG zgpg30  
TD 32768  
SOLVENT CDCl3  
NS 680  
DS 2  
SWH 24038.461 Hz  
FIDRES 0.733596 Hz  
AQ 0.6815744 sec  
RG 186.42  
DW 20.800 usec  
DE 6.50 usec  
TE 298.6 K  
D1 2.00000000 sec  
D11 0.03000000 sec  
TDO 1

----- CHANNEL f1 -----  
SFO1 100.6278588 MHz  
NUC1 13C  
P1 8.90 usec  
PLW1 54.00000000 W  
  
----- CHANNEL f2 -----  
SFO2 400.1516006 MHz  
NUC2 1H  
CPDPRG12 waltz16  
PCPD2 90.00 usec  
PLW2 12.00000000 W  
PLW12 0.32231000 W

F2 - Processing parameters  
SI 16384  
SF 100.6177842 MHz  
WOW SM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.00

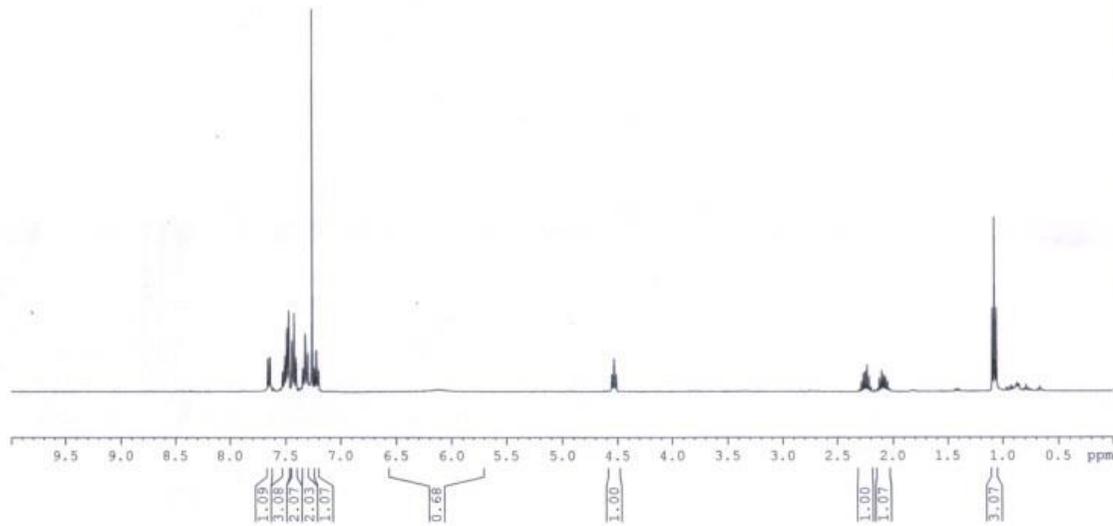
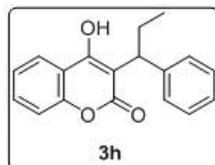
1H of VBRC-B

7.663  
7.660  
7.643  
7.640  
7.530  
7.527  
7.513  
7.509  
7.491  
7.488  
7.472  
7.443  
7.438  
7.425  
7.405  
7.344  
7.223  
7.221  
7.210  
7.201  
7.200  
7.259  
7.242  
7.191  
7.184  
7.122  
7.104  
7.102  
0.123

4.553  
4.533  
4.515

2.288  
2.269  
2.252  
2.234  
2.217  
2.222  
2.103  
2.085  
2.068  
2.051

1.095  
1.077  
1.059

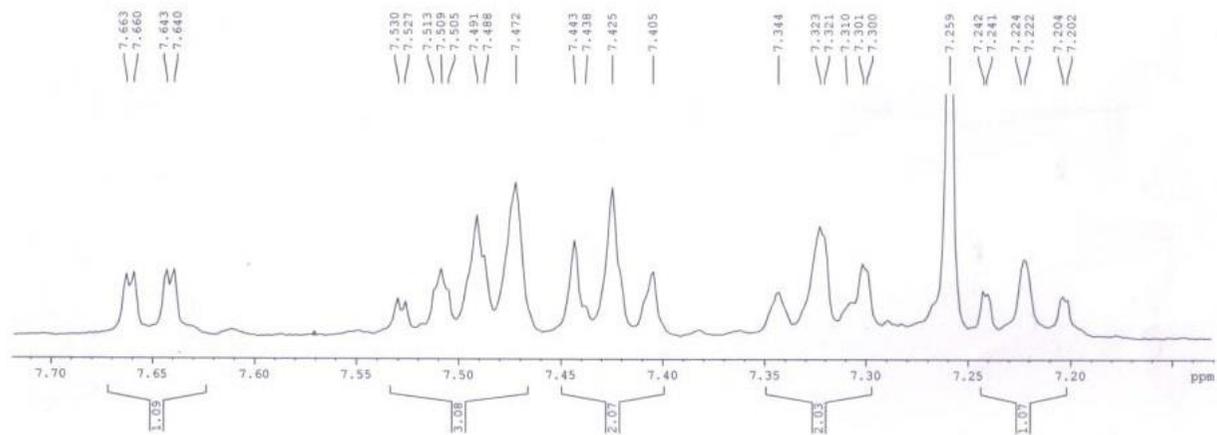


Current Data Parameters  
NAME Dr. A MAJEE 2020  
EXPNO 384  
PROCNO 1

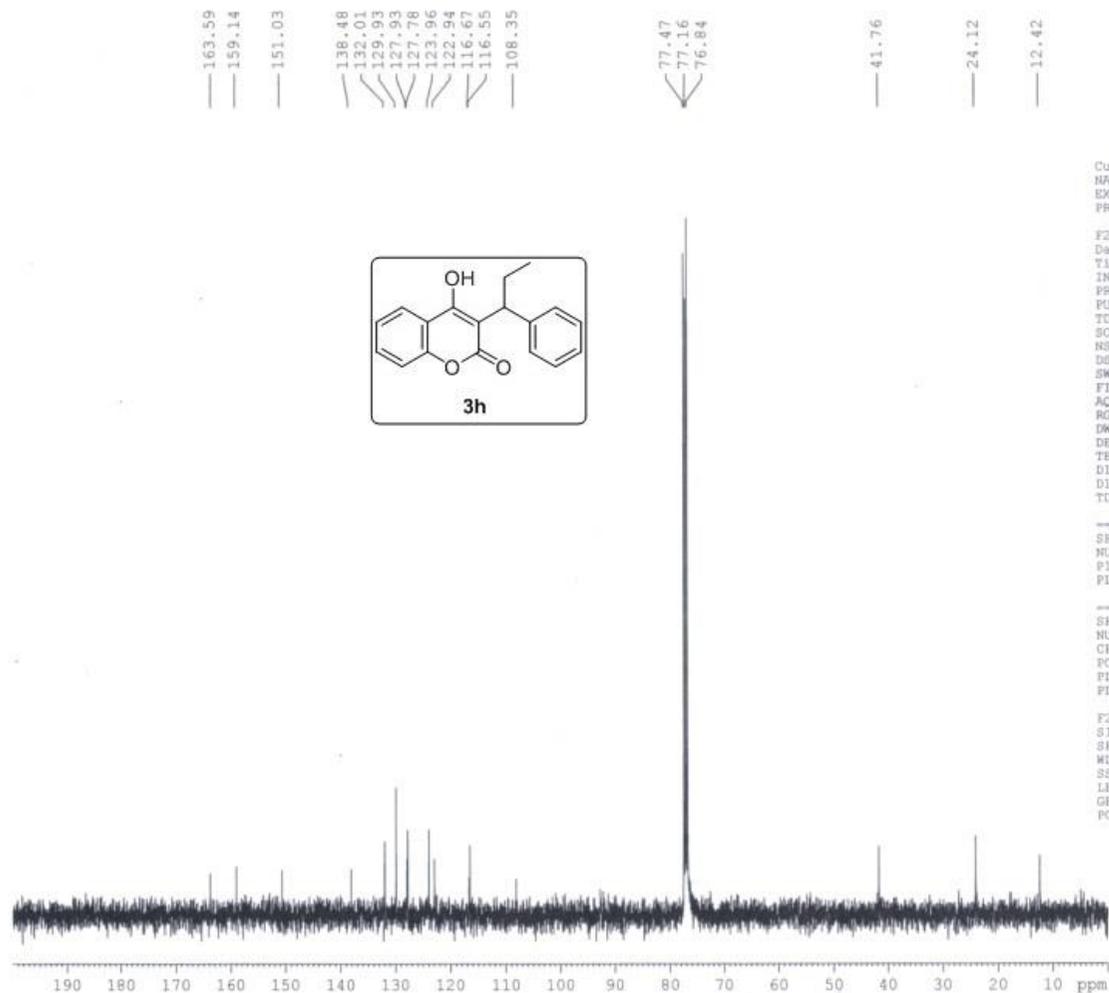
F2 - Acquisition Parameters  
Date\_ 20200929  
Time 12.39  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zg30  
TD 32768  
SOLVENT CDCl3  
NS 16  
DS 1  
SWH 8223.685 Hz  
FIDRES 0.250967 Hz  
AQ 1.9922944 sec  
RG 186.42  
DW 60.800 usec  
DE 6.50 usec  
TE 295.4 K  
D1 1.00000000 sec  
TDO 1

===== CHANNEL f1 =====  
SFO1 400.1524711 MHz  
NUC1 1H  
P1 14.75 usec  
PLW1 12.00000000 W

F2 - Processing parameters  
SI 16384  
SF 400.1500093 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 2.00



13C of VBrc-b



Current Data Parameters  
NAME Dr. A MAJEE 2020  
EXPNO 385  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20200929  
Time 14.35  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zgdc  
TD 32768  
SOLVENT CDCl3  
NS 1024  
DS 2  
SWH 24038.461 Hz  
FIDRES 0.733596 Hz  
AQ 0.6815744 sec  
RG 186.42  
DW 20.800 usec  
DE 6.50 usec  
TE 297.5 K  
D1 2.00000000 sec  
D11 0.03000000 sec  
TD0 1

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

----- CHANNEL f2 -----  
SFO2 400.1516006 MHz  
NUC2 1H  
CPDPRG2 waltz16  
PCPD2 90.00 usec  
PLW2 12.00000000 W  
PLW12 0.32231000 W

F2 - Processing parameters:  
SI 16384  
SF 100.6177830 MHz  
MW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.00