

Supporting Information

Brønsted acidic ionic liquid-catalyzed tandem reaction: an efficient approach towards regioselective synthesis of pyrano[3,2-c]coumarins under solvent-free conditions bearing lower E-factors

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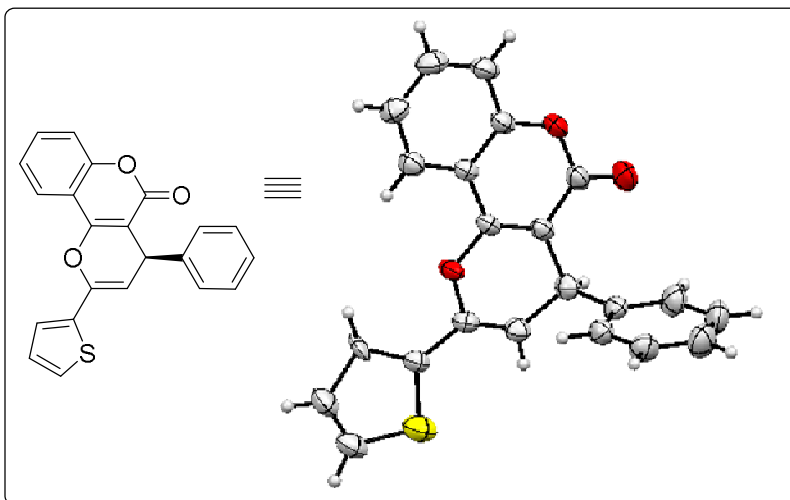
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Table of Contents

	Page No.
Structure Determination (X-ray crystallographic data for 3n)	2
E-factor Calculations	3
¹ H & ¹³ C NMR Spectra of all compounds	4

1. Structure Determination (X-ray crystallographic data for **3n**)

The white crystals of **3n** were obtained by crystallization from a solution in dichloromethane/ethylacetate/hexene. Chemical formula C₂₂H₁₄O₃S.



ORTEP (with 50% probability) diagram for the structure of 4-Phenyl-2-(thiophen-2-yl)-4H,5H-pyrano[3,2-c]chromen-5-one (**3n**).

Wavelength	0.71073 Å	
Formula	C ₂₂ H ₁₄ O ₃ S	
Crystal system	Monoclinic	
Space group	P 1 21/c 1	
Unit cell dimensions	a = 8.1926(11) Å	α = 90°
	b = 16.181(2) Å	β = 104.684(7)°
	c = 13.1852(18) Å	γ = 90°
Volume	1690.8(4) Å ³	
Z	2	
R-factor (%)	8.08	

The crystallographic data have been deposited with the Cambridge Crystallographic Data Centres as supplementary publication with a CCDC reference number CCDC **1539031**.

E-factor calculations:

For 3a: $E = [0.162 \text{ g(1a)} + 0.222 \text{ g(2a)} - 0.307 \text{ g (product} \times \text{yield)}] / 0.307 \text{ g} = 0.250$

For 3b: $E = [0.162 \text{ g(1a)} + 0.208 \text{ g(2b)} - 0.299 \text{ g (product} \times \text{yield)}] / 0.299 \text{ g} = 0.237$

For 3c: $E = [0.162 \text{ g (1a)} + 0.222 \text{ g(2c)} - 0.293 \text{ g (product} \times \text{yield)}] / 0.293 \text{ g} = 0.310$

For 3d: $E = [0.162 \text{ g(1a)} + 0.226 \text{ g(2d)} - 0.296 \text{ g (product} \times \text{yield)}] / 0.296 \text{ g} = 0.310$

For 3e: $E = [0.162 \text{ g(1a)} + 0.242 \text{ g(2e)} - 0.316 \text{ g (product} \times \text{yield)}] / 0.316 \text{ g} = 0.278$

For 3f: $E = [0.162 \text{ g(1a)} + 0.287 \text{ g(2f)} - 0.357 \text{ g (product} \times \text{yield)}] / 0.357 \text{ g} = 0.257$

For 3g: $E = [0.162 \text{ g(1a)} + 0.253 \text{ g(2g)} - 0.325 \text{ g (product} \times \text{yield)}] / 0.325 \text{ g} = 0.276$

For 3h: $E = [0.162 \text{ g(1a)} + 0.252 \text{ g(2h)} - 0.329 \text{ g (product} \times \text{yield)}] / 0.329 \text{ g} = 0.258$

For 3i: $E = [0.162 \text{ g(1a)} + 0.214 \text{ g(2i)} - 0.279 \text{ g (product} \times \text{yield)}] / 0.279 \text{ g} = 0.347$

For 3j: $E = [0.162 \text{ g(1a)} + 0.198 \text{ g(2j)} - 0.256 \text{ g (product} \times \text{yield)}] / 0.256 \text{ g} = 0.406$

For 3k: $E = [0.162 \text{ g(1a)} + 0.238 \text{ g(2k)} - 0.313 \text{ g (product} \times \text{yield)}] / 0.313 \text{ g} = 0.277$

For 3l: $E = [0.162 \text{ g(1a)} + 0.242 \text{ g(2l)} - 0.309 \text{ g (product} \times \text{yield)}] / 0.309 \text{ g} = 0.307$

For 3m: $E = [0.162 \text{ g(1a)} + 0.334 \text{ g(2m)} - 0.378 \text{ g (product} \times \text{yield)}] / 0.378 \text{ g} = 0.312$

For 3n : $E = [0.162 \text{ g(1a)} + 0.214 \text{ g(2n)} - 0.286 \text{ g (product} \times \text{yield)}] / 0.286 \text{ g} = 0.314$

For 3o: $E = [0.162 \text{ g(1a)} + 0.236 \text{ g(2o)} - 0.296 \text{ g (product} \times \text{yield)}] / 0.296 \text{ g} = 0.344$

For 3p: $E = [0.162 \text{ g(1a)} + 0.252 \text{ g(2p)} - 0.329 \text{ g (product} \times \text{yield)}] / 0.329 \text{ g} = 0.258$

For 3q: $E = [0.162 \text{ g(1a)} + 0.272 \text{ g(2q)} - 0.341 \text{ g (product} \times \text{yield)}] / 0.341 \text{ g} = 0.272$

For 3r: $E = [0.162 \text{ g(1a)} + 0.256 \text{ g(2r)} - 0.312 \text{ g (product} \times \text{yield)}] / 0.312 \text{ g} = 0.339$

For 3s: $E = [0.176 \text{ g(1b)} + 0.208 \text{ g(2b)} - 0.289 \text{ g (product} \times \text{yield)}] / 0.289 \text{ g} = 0.328$

For 3t: $E = [0.176 \text{ g(1b)} + 0.222 \text{ g(2a)} - 0.312 \text{ g (product} \times \text{yield)}] / 0.312 \text{ g} = 0.275$

For 3u: $E = [0.176 \text{ g(1b)} + 0.238 \text{ g(2k)} - 0.321 \text{ g (product} \times \text{yield)}] / 0.321 \text{ g} = 0.289$

For 3v: $E = [0.176 \text{ g(1b)} + 0.242 \text{ g(2e)} - 0.332 \text{ g (product} \times \text{yield)}] / 0.332 \text{ g} = 0.259$

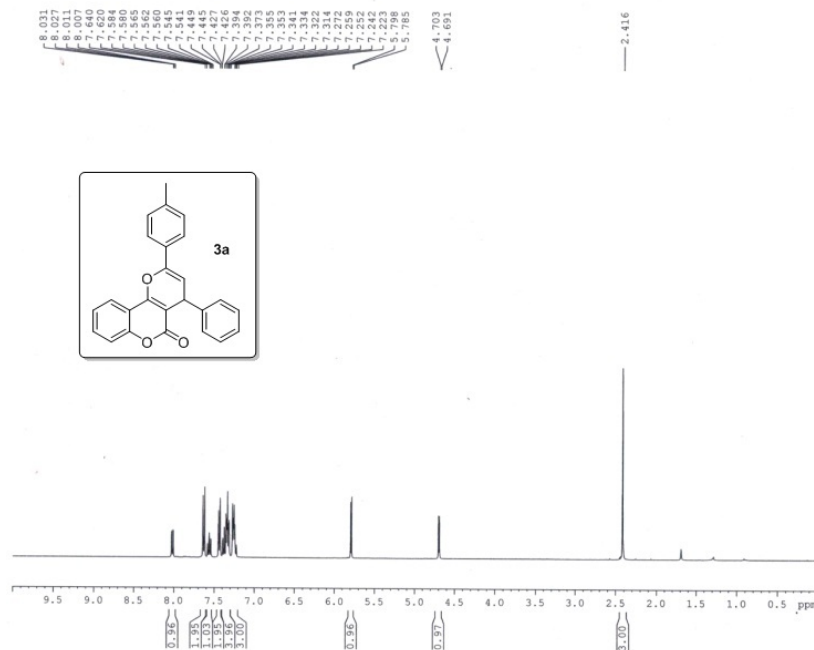
For 3w: $E = [0.176 \text{ g(1b)} + 0.252 \text{ g(2h)} - 0.348 \text{ g (product} \times \text{yield)}] / 0.348 \text{ g} = 0.229$

For 3x: $E = [0.178 \text{ g(1c)} + 0.208 \text{ g(2b)} - 0.287 \text{ g (product} \times \text{yield)}] / 0.287 \text{ g} = 0.344$

For 3y: $E = [0.178 \text{ g(1c)} + 0.222 \text{ g(2a)} - 0.305 \text{ g (product} \times \text{yield)}] / 0.305 \text{ g} = 0.311$

For 3z: $E = [0.178 \text{ g(1c)} + 0.252 \text{ g(2h)} - 0.338 \text{ g (product} \times \text{yield)}] / 0.338 \text{ g} = 0.272$

¹H of VBSSM 182



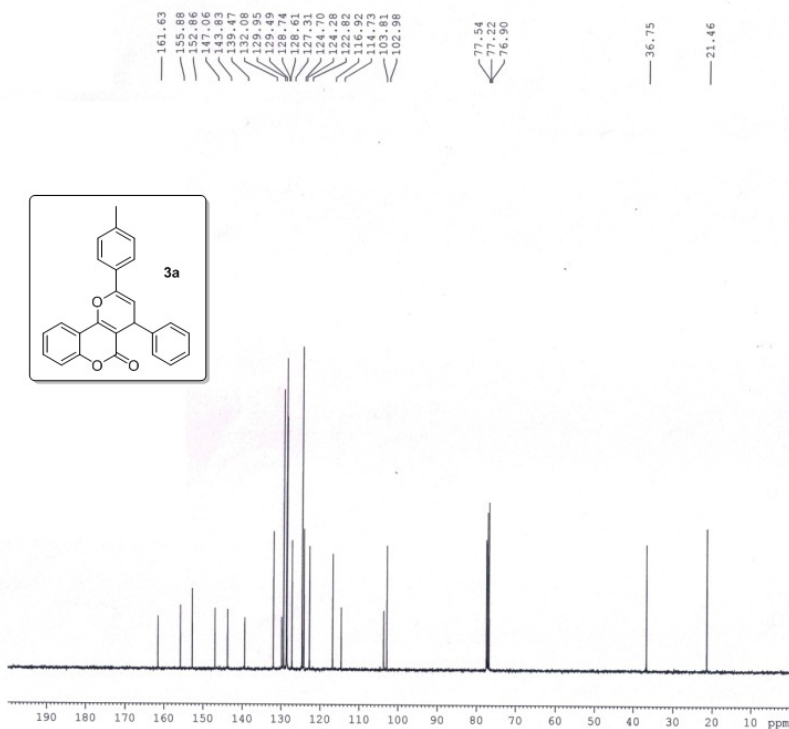
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SOLVENT CDCl3
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FIDRES 0.250967 Hz
AQ 1.9922944 sec
RG 47.25
DW 60.800 usec
DE 6.50 usec
TE 296.2 K
D1 1.00000000 sec
TDO 1

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NUC1 ¹H
P1 14.75 usec
PLW1 12.00000000 W

F2 - Processing parameters
SI 16384
SF 400.1500097 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C of VBSSM-182



Current Data Parameters
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EXPNO 355
PROCNO 1

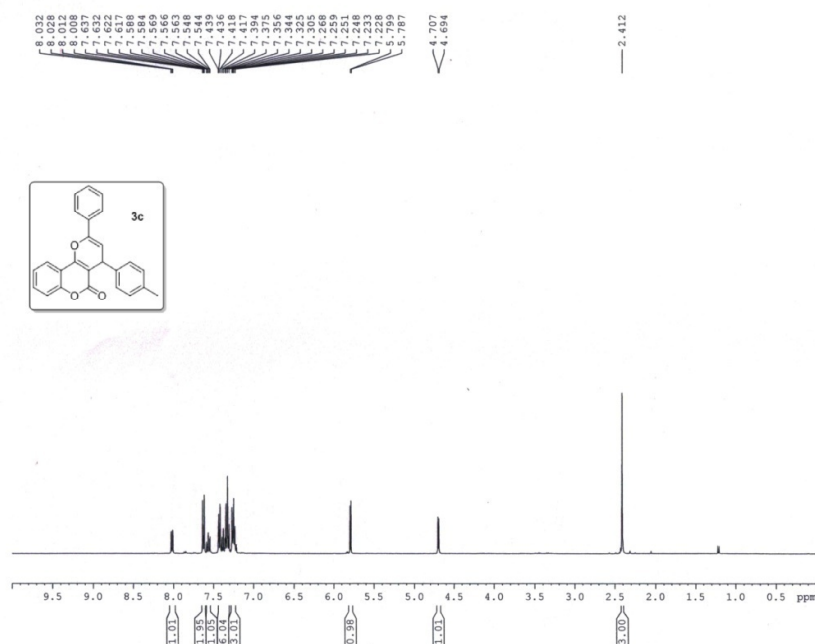
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PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 310
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SWH 24038.461 Hz
FIDRES 0.133596 Hz
AQ 0.6815744 sec
RG 47.25
DW 20.800 usec
DE 6.50 usec
TE 296.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TDO 1

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

===== CHANNEL f2 =====
SFO2 400.1516064 MHz
NUC2 ¹H
PCPDPRG2 waltz16
PCPD 90.00 usec
PLW2 12.00000000 W
PLW12 0.32231000 W
PLW13 0.16212000 W

F2 - Processing parameters
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SF 100.6177946 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1H of VBSSM-168



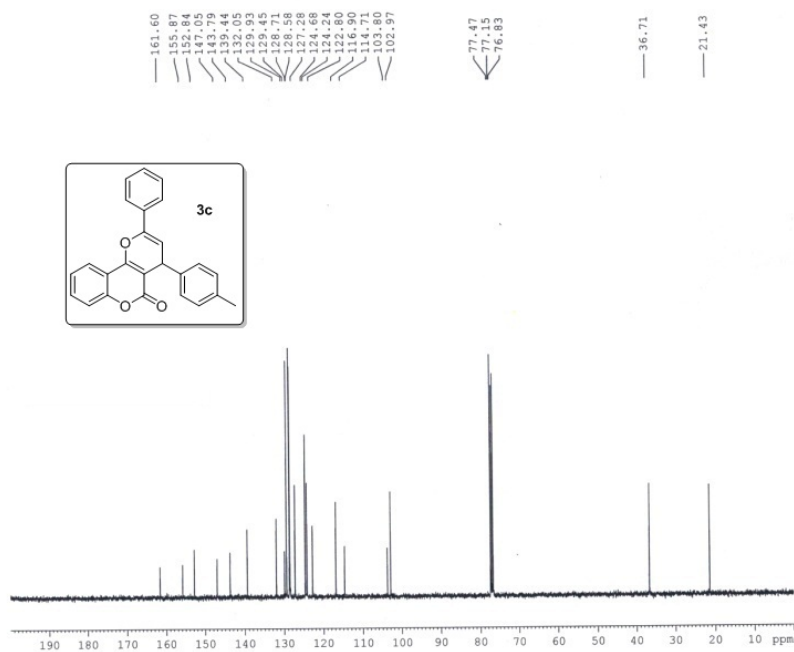
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NAME Dr. A MAJEE 2016
EXPNO 327
PROCNO 1

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Date 20161022
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PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 24
DS 1
SWH 8223.685 Hz
FIDRES 0.250967 Hz
AQ 1.9922944 sec
RG 62.69
DW 60.800 usec
DE 6.50 usec
TE 296.1 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 400.150097 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

13C of VBSSM-168



Current Data Parameters
NAME Dr. A MAJEE 2016
EXPNO 328
PROCNO 1

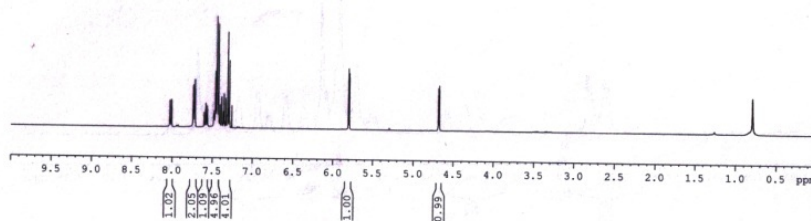
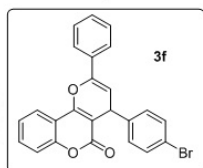
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Time 15.24
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TD 32768
SOLVENT CDCl3
NS 216
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SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 62.69
DW 20.800 usec
DE 6.50 usec
TE 296.7 K
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
CPCPD2 waltz16
PCPD2 90.00 usec
PLW2 12.00000000 W
PLW12 0.32210000 W
PLW13 0.16212000 W

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

1H of VBSSM 200



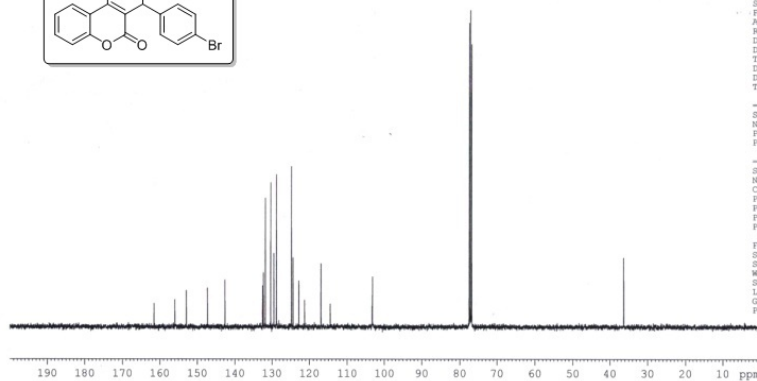
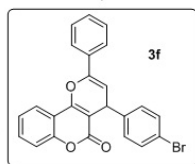
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NAME Dr. A MAJEE 2016
EXPNO 396
PROCNO 1

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PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 1
SWH 8223.685 Hz
FIDRES 0.250967 Hz
AQ 1.9922944 sec
RG 93.46
DM 60.800 usec
DE 6.50 usec
TE 295.9 K
D1 1.0000000 sec
TDO 1

CHANNEL f1
SFO1 400.1524711 MHz
NUC1 1H
P1 14.75 usec
PLW1 12.0000000 W

F2 - Processing parameters
SI 16384
SF 400.1500098 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

13C of VBSSM 200



Current Data Parameters
NAME Dr. A MAJEE 2016
EXPNO 397
PROCNO 1

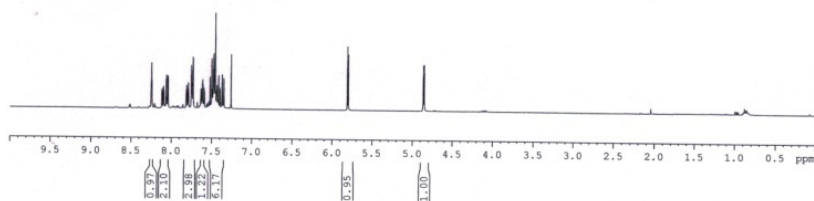
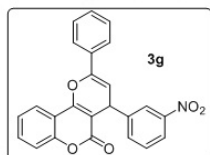
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INSTRUM spect
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PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 312
DS 2
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 93.46
DM 20.800 usec
DE 6.50 usec
TE 296.4 K
D1 2.0000000 sec
D11 0.0300000 sec
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
CPCPDW12 wait16
PCPD 90.00 usec
PLW2 12.0000000 W
PLW12 0.32231000 W
PLW13 0.16212000 W

F2 - Processing parameters
SI 16384
SF 100.6177872 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1H of VBSSM 199



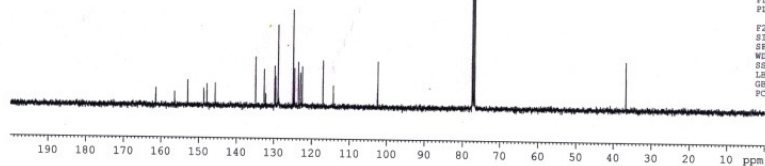
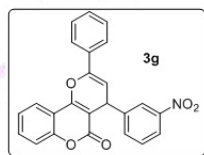
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NAME Dr. A MAJEE 2016
EXPNO 392
PROCNO 1

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PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 30
DS 1
SWH 8223.685 Hz
FIDRES 0.250967 Hz
AQ 1.9922944 sec
RG 120.16
DW 60.800 usec
DE 6.50 usec
TE 296.9 K
D1 1.00000000 sec
TDO 1

===== CHANNEL f1 =====
SFO1 400.1524711 MHz
NUC1 1H
P1 14.75 usec
PLM1 12.00000000 W

F2 - Processing parameters
SI 16384
SF 400.1500097 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

13C of VBSSM 199



Current Data Parameters
NAME Dr. A MAJEE 2016
EXPNO 393
PROCNO 1

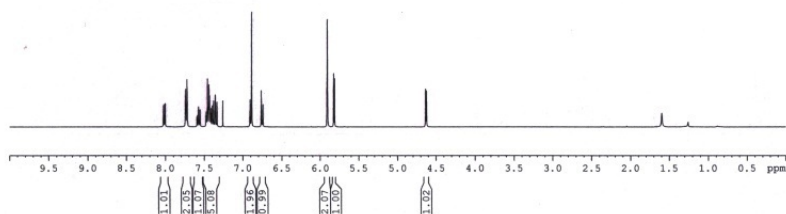
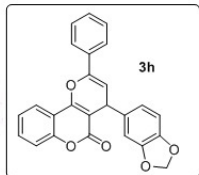
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Time 20.11
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 420
DS 2
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 120.16
DW 20.800 usec
DE 6.50 usec
TE 297.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TDO 1

===== CHANNEL f1 =====
SFO1 100.6278588 MHz
NUC1 13C
P1 8.50 usec
PLM1 54.00000000 W

===== CHANNEL f2 =====
SFO2 400.1516006 MHz
NUC2 1H
CHOPRG12 waltz16
PCPG2 90.00 usec
PLM2 12.00000000 W
PLM12 0.32231000 W
PLM13 0.16212000 W

F2 - Processing parameters
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SF 100.6177855 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1H of VBSSM 189

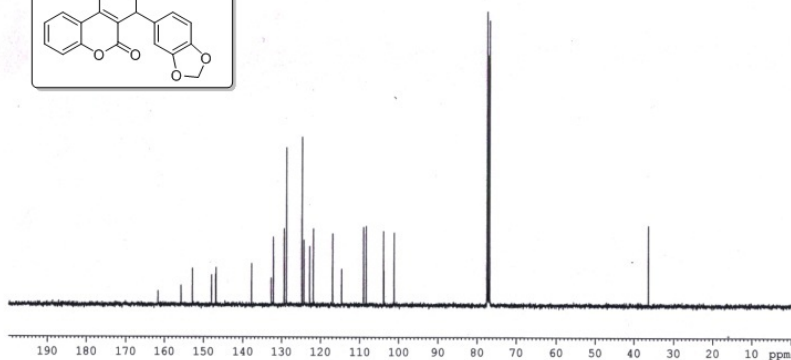
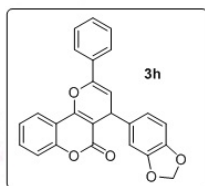
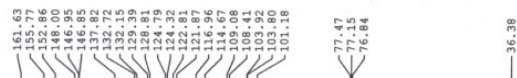


Current Data Parameters
NAME Dr. A. MAJEE 2016
EXPNO 372
PROCNO 1

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PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 1
SWH 8223.685 Hz
FIDRES 0.250967 Hz
AQ 1.9922944 sec
RG 120.16
DW 60.800 usec
DE 6.50 usec
TE 295.8 K
D1 1.00000000 sec
TDO 1

===== CHANNEL f1 =====
SFO1 400.1524711 MHz
NUC1 1H
P1 14.75 usec
PLM1 12.00000000 W
F2 - Processing parameters
SI 16384
SF 400.1500097 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

13C of VBSSM-189



Current Data Parameters
NAME Dr. A. MAJEE 2016
EXPNO 373
PROCNO 1

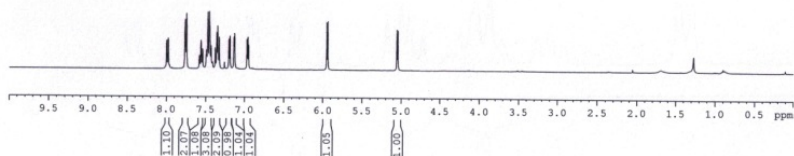
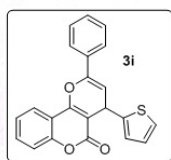
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Date_ 20161104
Time 13.10
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 480
DS 2
SWH 24038.461 Hz
FIDRES 0.733556 Hz
AQ 0.6815744 sec
RG 120.16
DW 20.800 usec
DE 6.50 usec
TE 296.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TDO 1

===== CHANNEL f1 =====
SFO1 100.6278588 MHz
NUC1 13C
P1 8.90 usec
PLM1 54.00000000 W

===== CHANNEL f2 =====
SFO2 400.1516006 MHz
NUC2 1H
CPDPRG2 waltz16
PCPD2 80.00 usec
PLM2 12.00000000 W
PLM12 0.32231000 W
PLM13 0.16212000 W

F2 - Processing parameters
SI 16384
SF 100.6177862 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

¹H of VBSSM 210



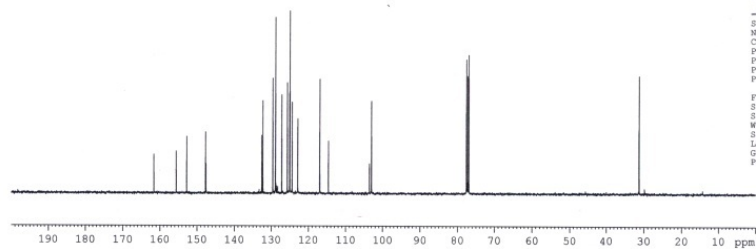
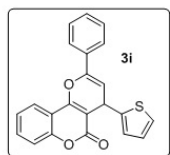
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NAME Dr. A MAJEE 2016
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PROCNO 1

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PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 1
SWH 8223.685 Hz
FIDRES 0.250967 Hz
AQ 1.9922944 sec
RG 57.28
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
WDM EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C of VBSSM 210



Current Data Parameters
NAME Dr. A MAJEE 2016
EXPNO 429
PROCNO 1

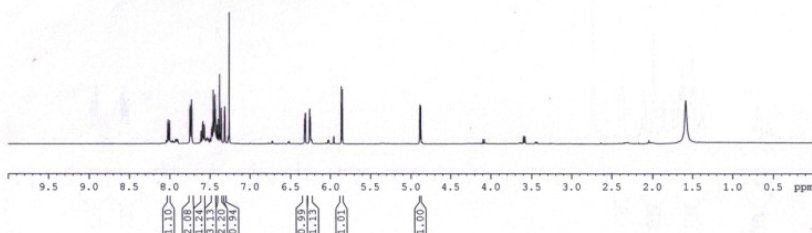
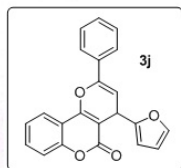
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PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 380
DS 2
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 57.28
DW 20.800 usec
DE 6.50 usec
TE 293.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TDO 1

===== CHANNEL f1 =====
SFO1 100.6270588 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
PLW13 0.16212000 W

F2 - Processing parameters
SI 16384
SF 100.6177839 MHz
WDM EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1H of VBSSM 212



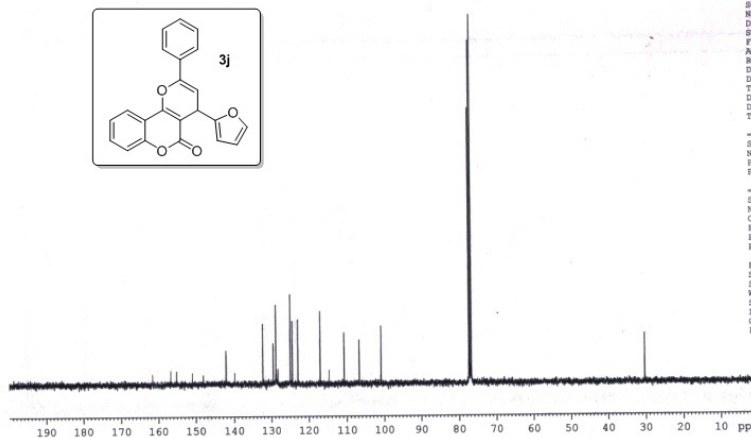
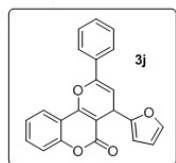
Current Data Parameters
NAME Dr. A MAJEE 2016
EXPNO 432
PROCNO 1

F2 - Acquisition Parameters
Date_ 20161202
Time 19.13
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 64
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 295.8 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 400.1524711 MHz
NUC1 1H
P1 14.75 usec
PLM1 12.00000000 W

F2 - Processing parameters
SI 16384
SF 400.1500097 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

13C of VBSSM 212



Current Data Parameters
NAME Dr. A MAJEE 2016
EXPNO 433
PROCNO 1

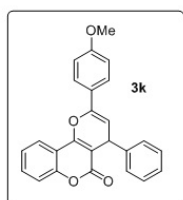
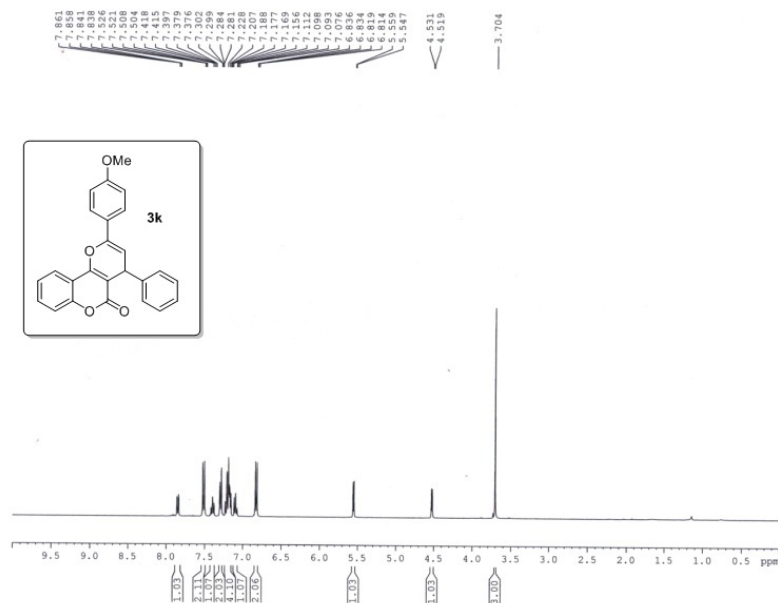
F2 - Acquisition Parameters
Date_ 20161207
Time 20.35
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 640
DS 2
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 186.42
DW 20.800 usec
DE 6.50 usec
TE 297.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 100.6278568 MHz
NUC1 13C
P1 8.90 usec
PLM1 54.00000000 W

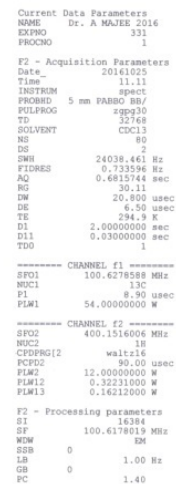
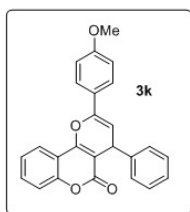
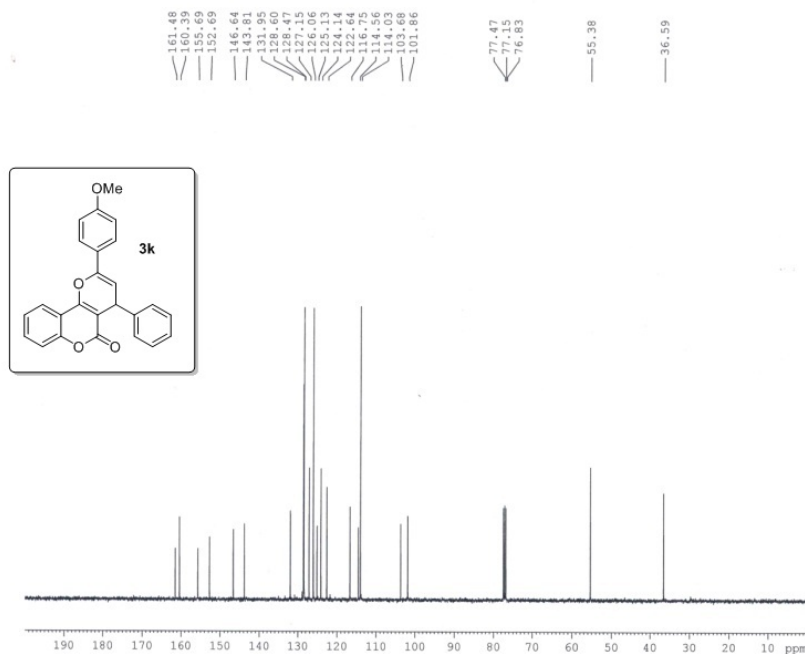
===== CHANNEL f2 =====
SFO2 400.1516006 MHz
NUC2 1H
CPDPRG2 waltz16
PCPD2 90.00 usec
PLM2 12.00000000 W
PLM12 0.32231000 W

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

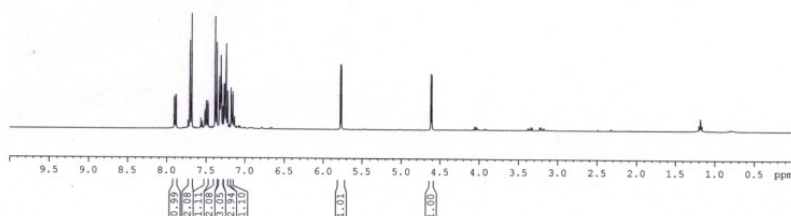
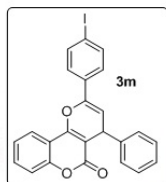
1H of VBSSM-171



13C of VBSSM-171



1H of VBSSM 180



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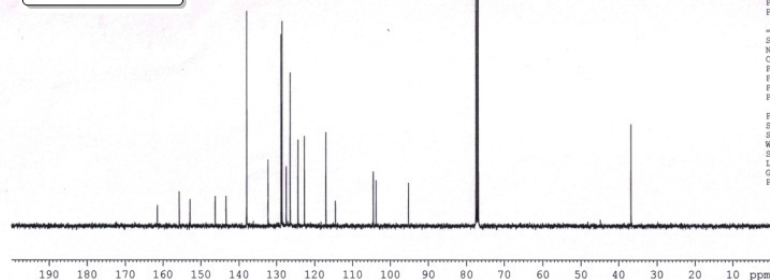
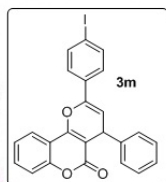
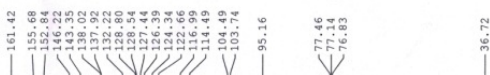
Current Data Parameters
NAME Dr. A MAJEE 2016
EXPNO 350
PROCNO 1

F2 - Acquisition Parameters
Date_ 20161029
Time 19.38
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 25
DS 1
SWH 8223.685 Hz
FIDRES 0.250967 Hz
AQ 1.9922944 sec
RG 87.66
DW 60.800 usec
DE 6.50 usec
TE 296.5 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 400.1500413 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

13C of VBSSM-180.



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Current Data Parameters
NAME Dr. A MAJEE 2016
EXPNO 351
PROCNO 1

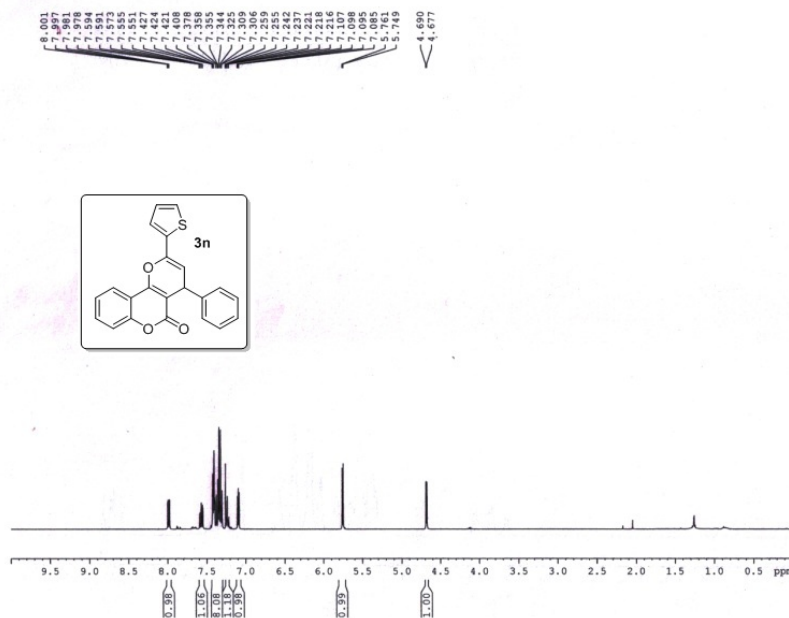
F2 - Acquisition Parameters
Date_ 20161029
Time 20.04
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 480
DS 2
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 87.66
DW 20.800 usec
DE 6.50 usec
TE 297.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
SF01 100.6278588 MHz
NUC1 13C
P1 9.90 usec
PLW1 54.00000000 W

===== CHANNEL f2 =====
SF02 400.1516006 MHz
NUC2 1H
CPOPRG12 waltz16
PCPD2 90.00 usec
PDW2 12.00000000 W
PLW12 0.32231000 W
PLW13 0.16212000 W

F2 - Processing parameters
SI 16384
SF 100.6177873 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

¹H of VBSSM-235



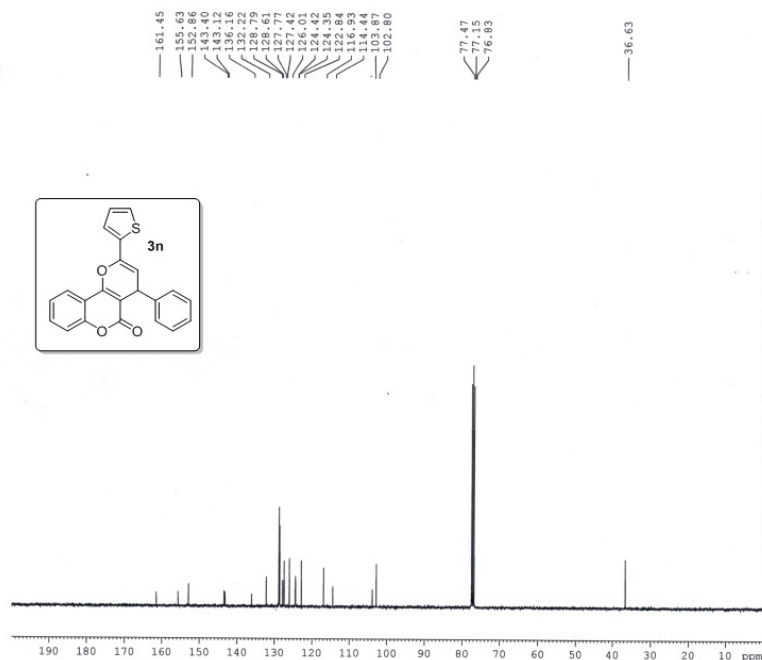
Current Data Parameters
NAME Dr. A MAJES 2017
EXPRO 42
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170119
Time 14.51
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 40
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 296.5 K
D1 1.00000000 sec
TDO 1

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

F2 - Processing parameters
SI 16384
SF 400.1500097 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C of VBssm-235



Current Data Parameters
NAME Dr. A MAJES 2017
EXPRO 43
PROCNO 1

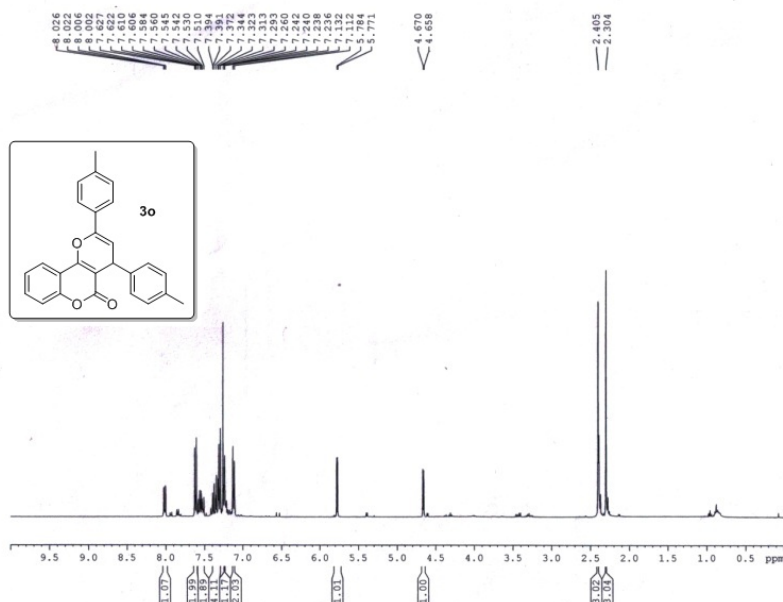
F2 - Acquisition Parameters
Date_ 20170119
Time 15.18
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 312
DS 2
SWH 24039.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 106.66
DW 20.800 usec
DE 6.50 usec
TE 297.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TDO 1

===== CHANNEL f1 =====
SF01 100.6278588 MHz
NUC1 ¹³C
P1 8.90 usec
PLW1 54.00000000 W

===== CHANNEL f2 =====
SF02 400.1516006 MHz
NUC2 ¹H
CPDPRG2 waltz16
PCPD2 90.00 usec
PLW2 12.00000000 W
PLW12 0.32231000 W
PLW13 0.16212000 W

F2 - Processing parameters
SI 16384
SF 100.6177859 MHz
WDW DM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1H of VBSSM 183



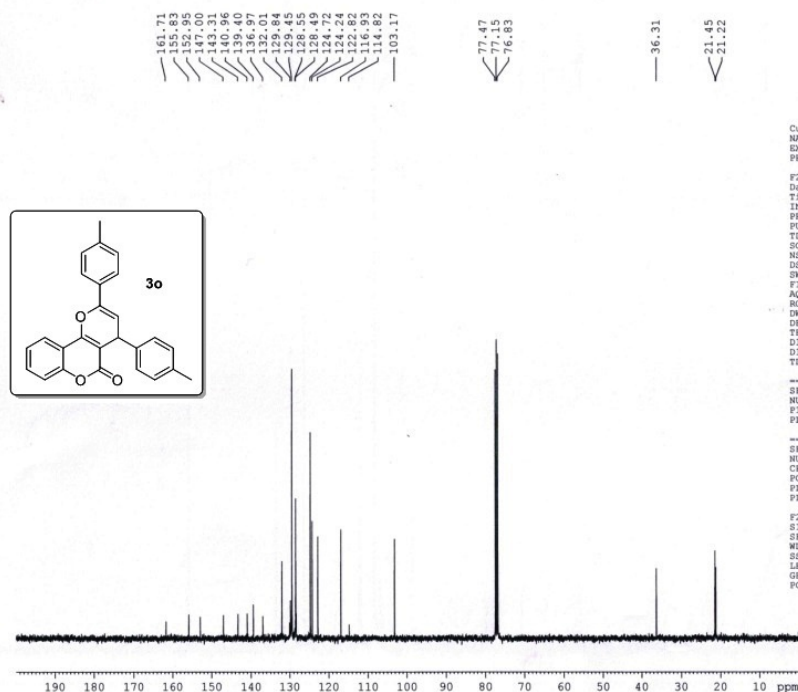
Current Data Parameters
NAME Dr. A MAJEE 2016
EXPNO 359
PROCNO 1

F2 - Acquisition Parameters
Date_ 20161031
Time 18.41
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 1
SWH 8223.685 Hz
FIDRES 0.250967 Hz
AQ 1.9922944 sec
RG 120.16
DW 60.800 usec
DE 6.50 usec
TE 298.9 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 VBSSM 183



Current Data Parameters
NAME Dr. A MAJEE 2017
EXPNO 133
PROCNO 1

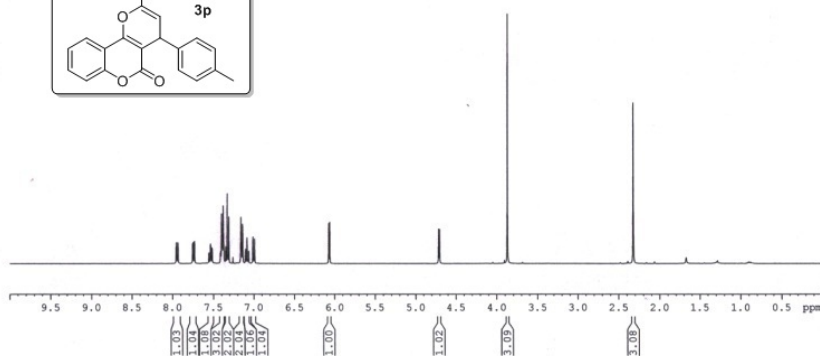
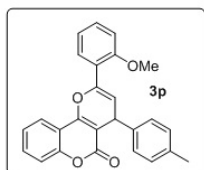
F2 - Acquisition Parameters
Date_ 20170212
Time 14.36
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 640
DS 2
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 186.42
DW 20.800 usec
DE 6.50 usec
TE 298.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TDO 1

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

CHANNEL f2
SFO2 400.1516006 MHz
NUC2 1H
CPCPRG12 waltz16
PCPD2 90.00 usec
PLW2 12.00000000 W
PLW12 0.32210000 W

F2 - Processing parameters
SI 16384
SF 100.6177843 MHz
WDW DM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

7.959	7.955	7.958	7.936	7.757	7.753	7.738	7.734	7.538	7.535	7.519	7.515	7.400	7.396	7.380	7.376	7.371	7.351	7.348	7.333	7.330	7.310	7.160	7.140	7.103	7.085	7.067	7.064	7.013	6.992	6.975	6.063
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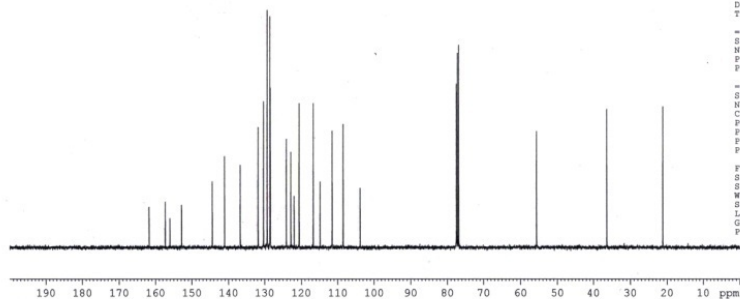
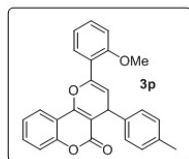
Current Data Parameters
NAME          Dr. A MAJEE 2016
EXPNO         401
PROCNO        1

F2 - Acquisition Parameters
=====
Time          2013.01.18
INSTRUM       spect
PROBHD        5 mm PABBO BBI
PULPROG       zgpg30
TD            32768
SOLVENT       CDCl3
NS            12
DS            1
SWH           823.65 Hz
FIDRES        0.250967 Hz
AQ            1.99943844 sec
RG            64.00
AW            50.87
DE            6.50 usec
TE            296.7 K
D1            1.00000000 sec
TD0           1
=====
===== CHANNEL f1 =====
FOF0          400.152471 MHz
NUC1          1H
NUC2          13C
F2L1         12.00000000 MHz
=====

F2 - Processing parameters
=====
SF            400.1500097 MHz
WDW           EM
SSB           0
GB            0.30 Hz
PC            1.00
=====

```

161.74	128.41	116.76	103.79
157.30	124.07	114.86	108.47
156.02	122.86	111.52	101.52
152.79	121.98	108.47	98.47
144.40	120.59	105.33	95.33
141.05	118.41	102.19	92.19
136.74	116.23	99.06	89.06
131.79	114.05	95.92	85.92
130.28	111.87	92.78	82.78
129.30	109.69	89.64	79.64
128.53	107.51	86.50	76.50
128.41	105.33	83.36	73.36
124.07	103.15	80.22	70.22
122.86	100.97	77.08	67.08
121.98	98.79	73.94	63.94
120.59	96.61	70.80	60.80
116.76	94.43	67.66	57.66
114.86	92.25	64.52	54.52
111.52	90.07	61.38	51.38
108.47	87.89	58.24	48.24
103.79	85.71	55.10	45.10

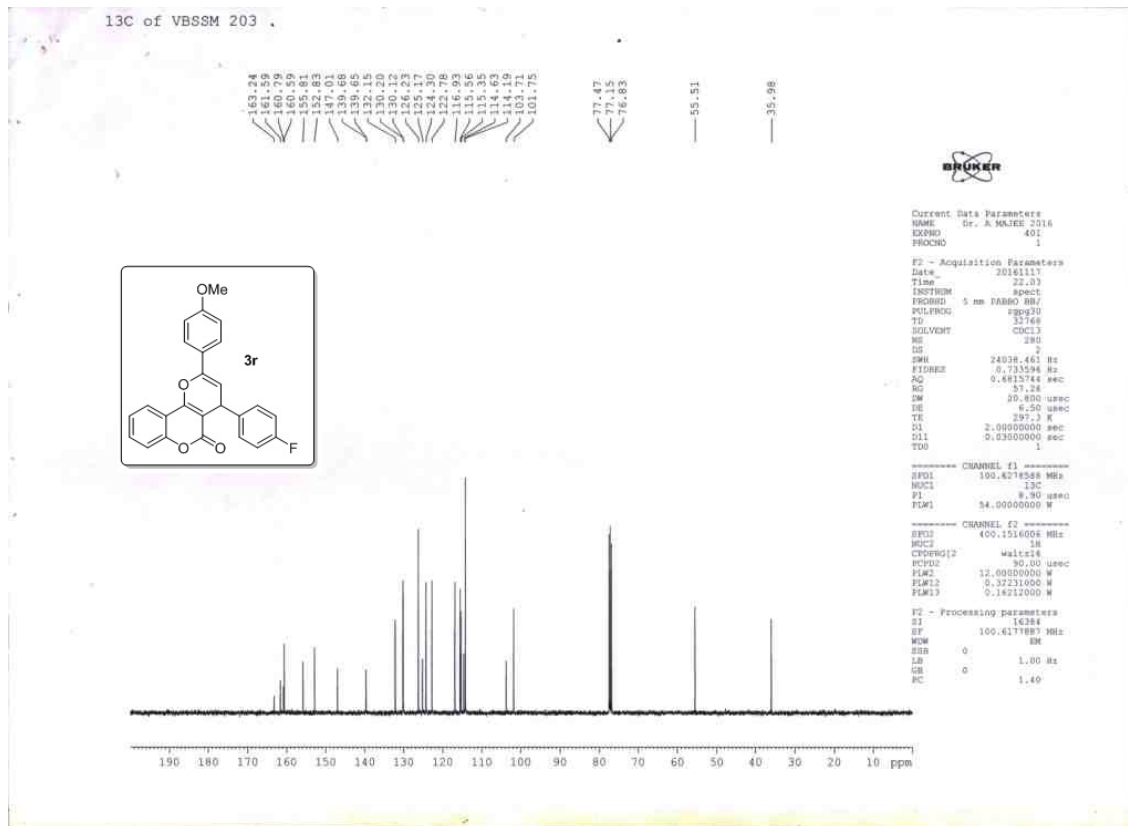
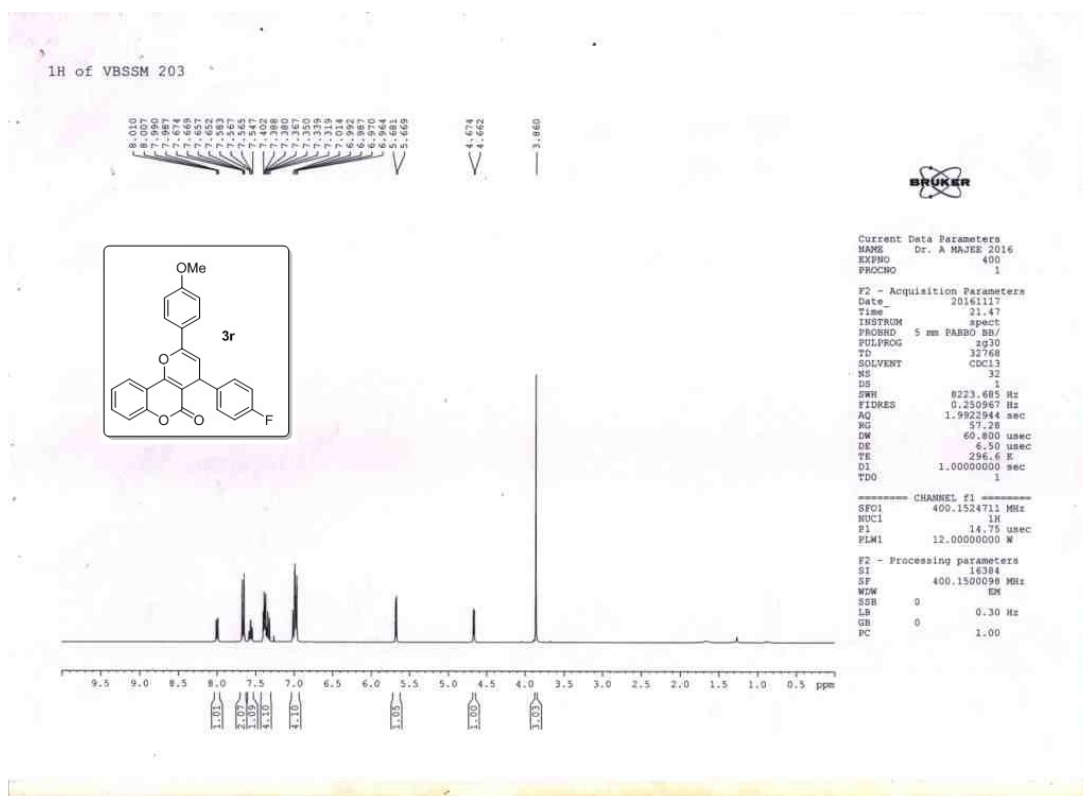


```

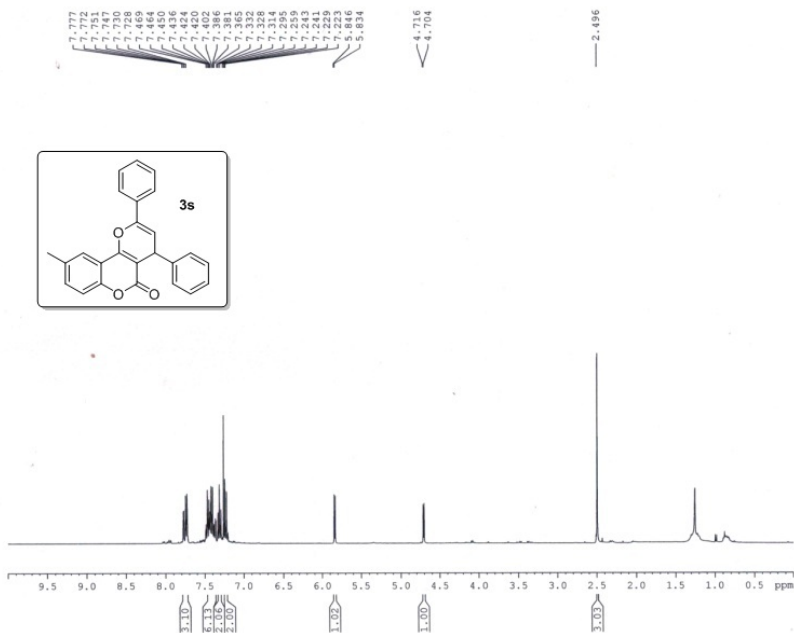
Current Data Parameters
NAME      Dr. A. MUEJER 1
ID        407
EXPID     1

F2 - Acquisition Parameters
Date      20161118
Time      12:00:00
INSTRUM    spect
FREQ      5 mm F4000
PULPROG    zgpg30
TD         32768
SFO        COC13
RG         SNR
NS         312
DS         2
SWH         24038.461 Hz
AQ          0.7333333 sec
AQ         0.6815742 sec
RG          25
DE          20.8000 usec
DI          6.50 usec
TE         278
D1          2.0000000 sec
D2          0.3300000 sec
TDO        1
===== CHANNEL 1 =====
NUC1       100.6271606 MHz
NUC1       13C
P1         8.90 usec
PL1        0.0000000 W
===== CHANNEL 2 =====
SF02       100.6151606 MHz
PCF02G12   waltz16
PCF02G12   100.6151606 sec
PCF02G12   12.0000000 MHz
PLW12       0.3223100 W
PLW12       0.3621921 W
SF03        13.683333 MHz
F2 - Processing parameters
NUC1       13C
SF01       100.6171606 MHz
SWH        EM
SFB        0
GB          0
PC          1.00 Hz
=====

```

1H of VBSSM 246



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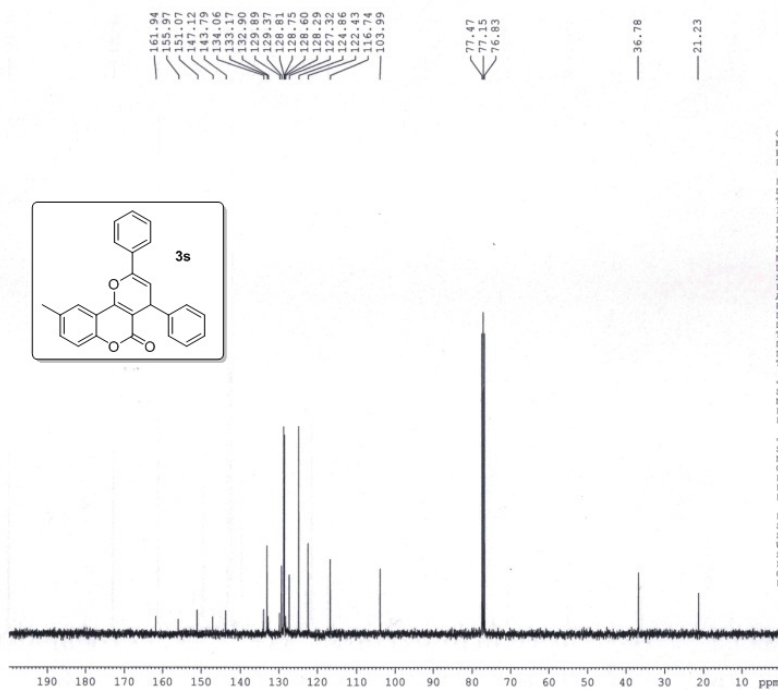
Current Data Parameters
NAME Dr. A MAJEE 2017
EXPNO 108
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170207
Time 20.30
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 48
DS 1
SWH 8223.685 Hz
FIDRES 0.250967 Hz
AQ 1.9922944 sec
RG 120.16
DW 60.800 usec
DE 6.50 usec
TE 295.3 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 400.1500098 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

13C of VBSSM 246



BRUKER

Current Data Parameters
NAME Dr. A MAJEE 2017
EXPNO 109
PROCNO 1

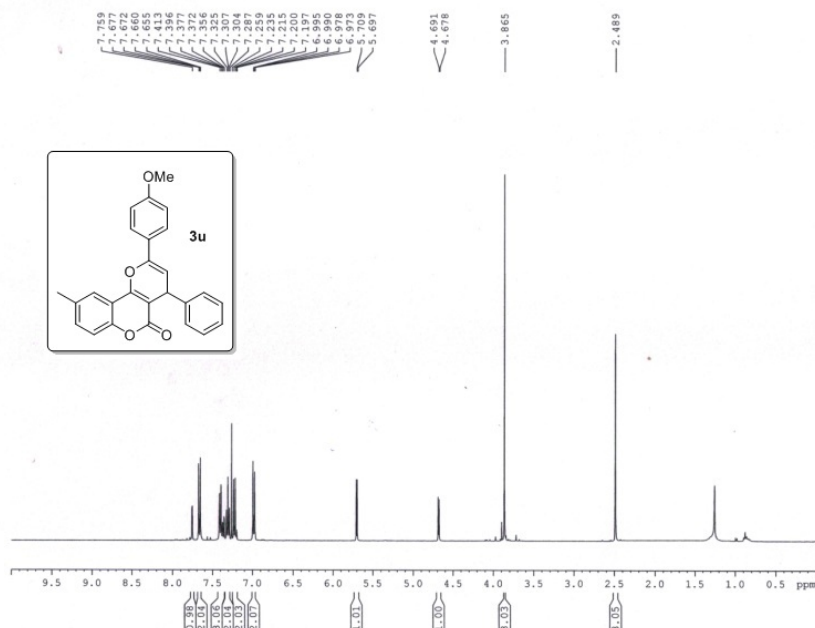
F2 - Acquisition Parameters
Date_ 20170207
Time 21.22
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg
TD 32768
SOLVENT CDCl3
NS 640
DS 2
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 120.16
DW 20.800 usec
DE 6.50 usec
TE 296.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TDO 1

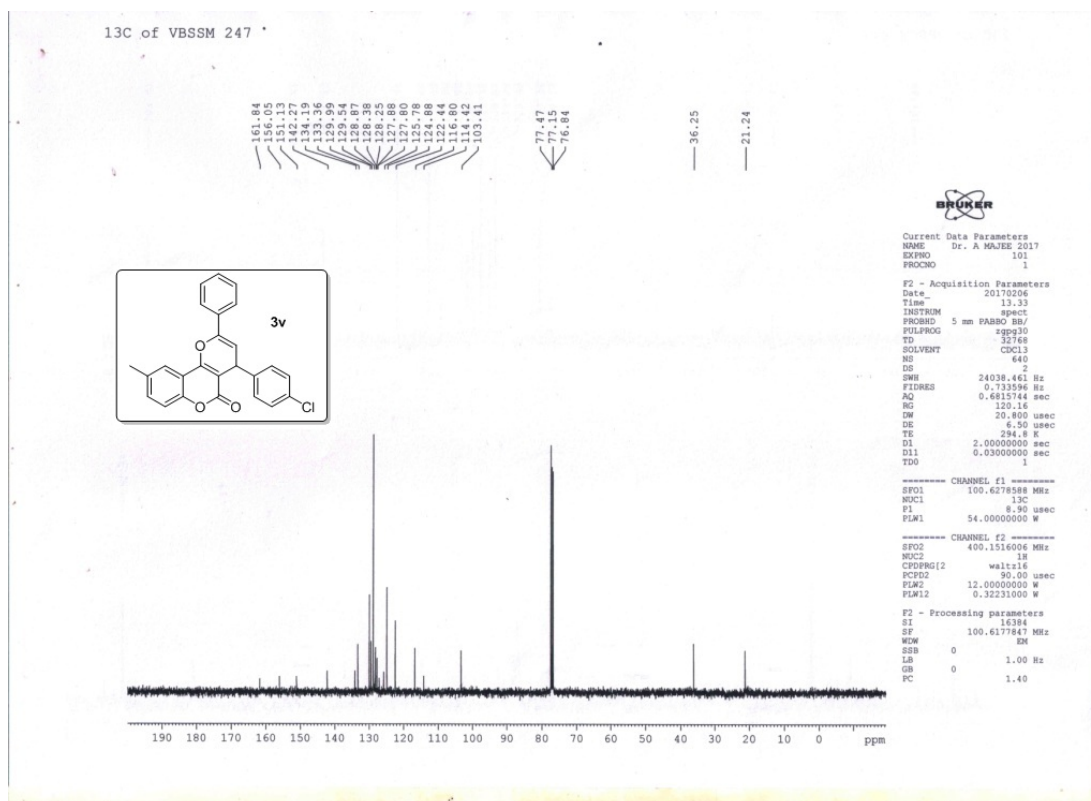
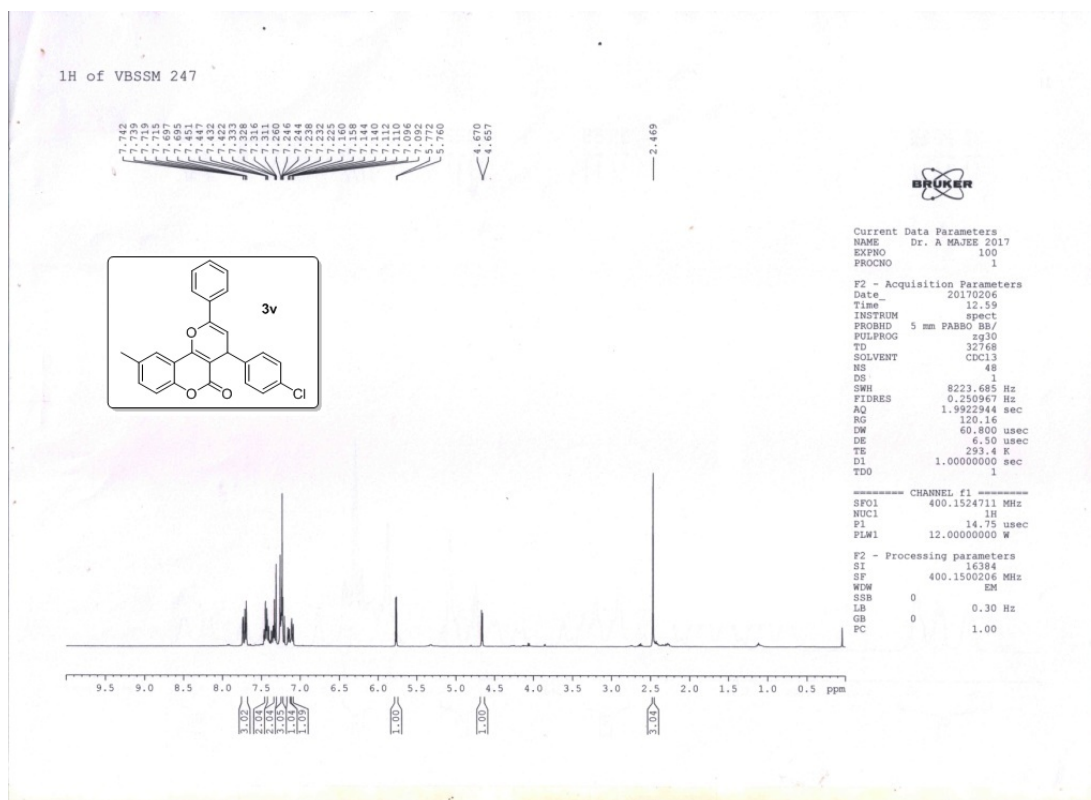
----- CHANNEL f1 -----
SFO1 100.6278568 MHz
NUC1 13C
P1 8.90 usec
PLW1 54.00000000 W

----- CHANNEL f2 -----
SFO2 400.1516006 MHz
NUC2 1H
CPOW2 waltz16
PCPO2 90.00 usec
PLW2 12.00000000 W
PLW2 0.32231000 W

F2 - Processing parameters
SI 16384
SF 100.6177845 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

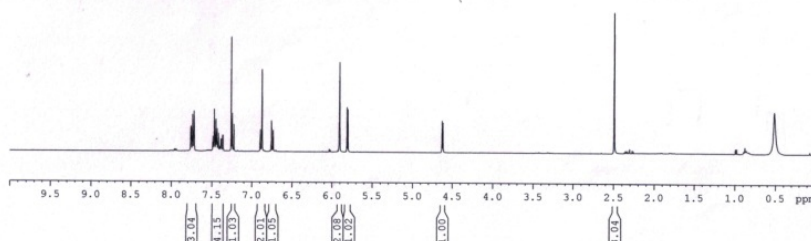
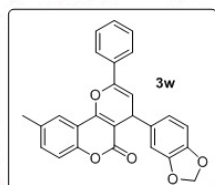
1H of VBSSM 209





7.764
7.761
7.745
7.741
7.723
7.721
7.490
7.486
7.469
7.450
7.436
7.433
7.419
7.388
7.383
7.367
7.362
7.259
7.248
7.227
6.900
6.899
6.894
6.875
6.757
6.756
6.738
6.737
5.907
5.814
5.802

1.630
1.618



```

F2 - Acquisition Parameters
Date      20161120
Time      10.58
INSTRUM    spect
PROBHD     5 mm PABBO BB/
PULPROG    zg30
TD         32768
SOLVENT     CDCl3
NS          64
DS          1
SWH         8223.685 Hz
FIDRES     0.509667 Hz
AQ         1.9922944 sec
RG          186.42
DW         60.800 usec
DE         6.50 usec
TE         293.5 K
D1         1.00000000 sec
TDO        1

```

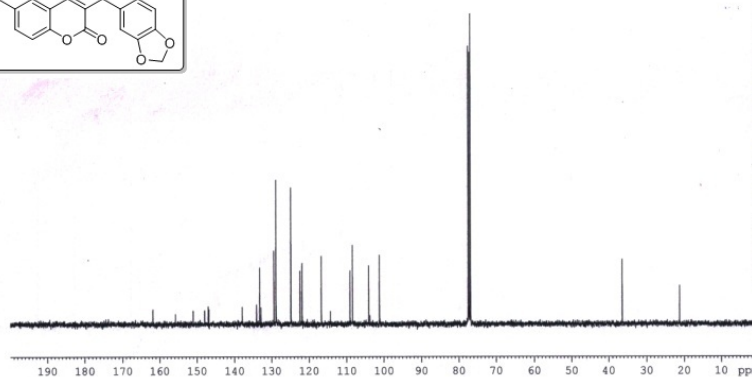
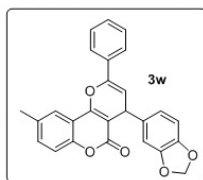
```

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

```

```
F2 - Processing parameters
SI                16384
SF                400.1500097 MHz
WDW              EM
SSB              0
LB                0.30 Hz
GB              0
PC                1.00
```

161.84
155.82
151.06
148.00
147.02
146.83
137.96
134.09
133.19
132.88
129.38
128.82
124.86
122.43
121.86
116.75
114.35
109.09
108.41
104.04
103.71
101.18



```

F2 - Acquisition Parameters
Date_      20161121
Time       23.08
INSTRUM    spect
PROBHD     5 mm PABBO 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          298.0 K
D1          2.00000000 sec
D11         0.03000000 sec
TDO         1

```

```

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

```

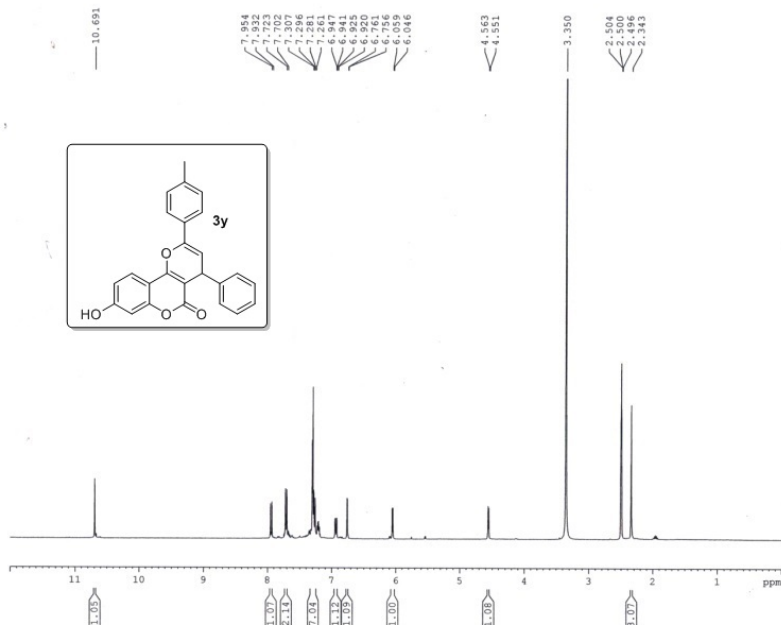
```

===== CHANNEL f2 =====
SFO2      400.1516006 MHz
NUC2              1H
CPDPRG[2      waltz16
PCPD2              90.00 use
PLW2      12.00000000 W
PLW12      0.32231000 W

```

```
F2 - Processing parameters
SI                16384
SF                100.6177036 MHz
WDW               EM
SSB               0
LB                1.00 Hz
GB               0
PC                1.40
```


¹H of VBSSM 214



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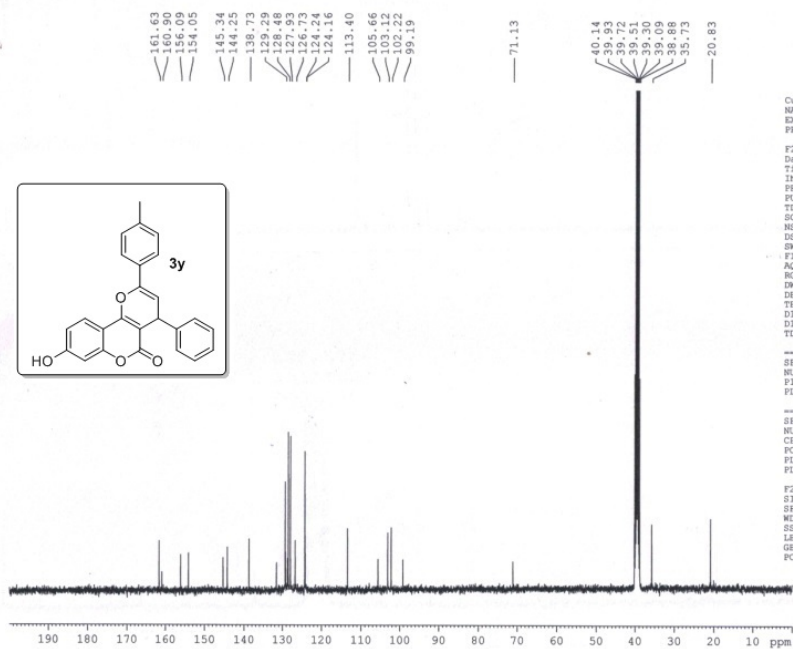
Current Data Parameters
NAME Dr. A MAJEE 2016
EXPNO 437
PROCNO 1

F2 - Acquisition Parameters
Date_ 20161215
Time 12.31
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 64
DS 2
SWH 8223.681 Hz
FIDRES 0.250967 Hz
AQ 1.9922944 sec
RG 106.66
DW 60.800 usec
DE 6.50 usec
TE 297.9 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 400.1500030 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C of VBSSM-214



BRUKER

Current Data Parameters
NAME Dr. A MAJEE 2016
EXPNO 444
PROCNO 1

F2 - Acquisition Parameters
Date_ 20161215
Time 15.30
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT DMSO
NS 640
DS 2
SWH 24030.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 106.66
DW 20.400 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TDO 1

===== CHANNEL f1 =====
SFO1 100.6278588 MHz
NUC1 ¹³C
P1 8.50 usec
PLW1 54.00000000 W

===== CHANNEL f2 =====
SFO2 400.1516006 MHz
NUC2 ¹H
CPDPRG2 waltz16
PCPD2 80.00 usec
PLW2 12.00000000 W
PLW12 0.32231000 W

F2 - Processing parameters
SI 16384
SF 100.6178458 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

