

by TLC analysis. After 2 hours, the reaction was deemed complete (less than 1.0% of compound 10 remaining). The reaction mixture was cooled to 0 to -5 °C and quenched with aqueous NaOH solution (2.0 M, 176 mL) followed by aqueous 30% H₂O₂ (33 mL) while maintain internal temperature 0 to 10 °C. After stirring for 1 hour, the batch was warmed to room temperature and MTBE (70 mL) was added. The layer was separated and the aqueous layer was extracted with MTBE (70 mL). Combined organic layers were washed with water (40 mL) followed by saturated NaHCO₃ (40 mL) and brine (40 mL), dried over MgSO₄, and concentrated. The crude product was purified by column chromatography eluted with 0–25% EtOAc in heptane to afford compound **12** (6.1 g, 77.7% yield) as a colorless oil (see Attachment 9).

Step 12. Preparation of Compound 13 (Ref: 09GB024)

NaH (1.28 g, 32.0 mmol), BnBr (2.32 mL, 21.3 mmol) and nBu₄Ni (1.28 g) were added to a solution of compound **12** (6.0 g, 10.7 mmol) in a mixture of DMF (90 mL) and THF (90 mL) at 0 °C. The reaction mixture was stirred at room temperature and monitored by TLC analysis. After 16 hours, the reaction was deemed complete (less than 1.0% of compound 12 remaining). The reaction mixture was poured into ice cold water (60 mL) and extracted with EtOAc (2 × 60 mL). The organic layer was washed with water (30 mL), followed by brine (30 mL), dried over MgSO₄, and concentrated to give the crude product which was purified by column chromatography eluted with 0–10% EtOAc in heptane to afford compound **13** (6.5 g, 93% yield) as a colorless oil (see Attachment 10).

Step 13. Preparation of Compound A (Ref: 09GB025)

A solution of TBAF (1.0 M in THF, 19.6 mmol) was added to a solution of compound **13** (6.4 g, 9.8 mmol) in THF (64 mL) at room temperature. The reaction mixture was stirred at room temperature and monitored by ¹H NMR and HPLC analyses. After 40 hours, the reaction was deemed complete (less than 5.0% of compound 13 remaining). The reaction was worked up by quenching with water (64 mL) and extracting with EtOAc (2 × 96 mL). The organic layer was washed with water (32 mL), followed by brine (32 mL), dried over MgSO₄, and concentrated to give the crude product which was purified by column chromatography eluted with 0–25% EtOAc in heptane to afford compound **A** (4.35 g, 82% yield) as a colorless oil (see Attachment 11).

4.2 Preparation of Compound B

Step 1. Preparation of Compound 15 (Ref: 09DX014 and 14KL0187)

Compound **14** (400 g, 1.26 mol, Indofine lot # XX) and imidazole (257 g, 3.78 mol) were mixed in THF (1.6 L). TBDMSCl (284.8 g, 1.89 mol) was added. The reaction mixture was stirred at room temperature. The reaction was monitored by TLC analysis. After 16 hours, the reaction was deemed complete (less than 1.0% of compound 14 remaining). EtOAc (4 L) was added to the reaction mixture, and the solution was washed with water (3 × 2 L). The organic layer was dried over MgSO₄, filtered, and concentrated to dryness. The residue was loaded onto a silica column and eluted with 25% heptanes in EtOAc, EtOAc, and 5% MeOH

in EtOAc. The fractions of interest were concentrated to afford compound **15** (325 g, 60% yield) as white solids (see Attachment 12).

Step 2. Preparation of Compound 16 (Ref: 09DX017, 09DX018, and 14KL0210)

Compound **15** (215 g, 0.5 mol) and NaBH(OAc)₃ (264 g, 1.2 mol) were mixed in THF (1075 mL). Benzaldehyde (63.4 g, 0.6 mol) was added at room temperature. The reaction mixture was stirred at room temperature and monitored by ¹H NMR and TLC analyses. After 16 hours, the reaction was deemed complete (less than 1.0% of compound **15** remaining). EtOAc (1.35 L) and saturated NaHCO₃ (1.35 L) were added. The layers were separated and the organic layer was washed with saturated NaHCO₃ (1.35 L). Combined aqueous layers were extracted with EtOAc (700 mL). Combined organic layers were washed with brine (350 mL), dried over MgSO₄, filtered, and concentrated to dryness. The residue was combined with the crude from a 100 g run and purified by column chromatography eluted with 20% EtOAc in heptane to give compound **16** (208 g, 55% yield) as an oil (see Attachment 13).

Step 3. Preparation of Compound 17 (Ref: 24LL018)

To a solution of compound **16** (12 g, 22.99 mmol) in 2,2-dimethoxypropane (300 mL) was added *p*-TsOH•H₂O (4.50 g, 23.68 mmol). The reaction mixture was stirred at room temperature for 5 hours. TLC analysis on the sample quenched with saturated NaHCO₃ showed there was not starting material. Ethyl acetate (150 mL) and saturated NaHCO₃ (60 mL) were added. The layers were separated and the aqueous layer was extracted with ethyl acetate (30 mL). Combined organic layers were washed with brine (60 mL) and concentrated. The crude material was purified chromatographically eluted with 4–10% ethyl acetate in heptane to provide compound **17** (9.24 g, 71.5% yield) as a colorless oil (see Attachment 14).

Step 4. Preparation of Compound 18 (Ref: 24LL021)

To a solution of compound **17** (9.2 g, 16.37 mmol) in THF (30 mL) was added a solution of TBAF in THF (1 M, 32.7 mL). The reaction mixture was stirred at room temperature for 1.5 hours, at which time there was almost no starting material by TLC analysis. The reaction was quenched with water (30 mL) and ethyl acetate (30 mL) was added. The layers were separated and aqueous layer was extracted with ethyl acetate (30 mL). Combined organic layers was washed with brine (30 mL) and concentrated. The crude material was purified chromatographically eluted with 25–50% ethyl acetate in heptane to give compound **18** (7.03 g, 96% yield) as a colorless oil (see Attachment 15).

Step 5. Preparation of Compound 19 (Ref: 24LL024)

To a solution of compound **18** (7 g, 15.64 mmol) in dichloromethane (50 mL) was added triethylamine (6.54 mL, 46.91 mmol). The mixture was cooled to <−70 °C. Thionyl chloride (1.31 mL, 17.98 mmol) was added. After addition, the mixture was warmed to −40 °C and stirred at −35 °C to −45 °C for 1 hour, at which time there was no starting material by

TLC analysis. The reaction was quenched with water (21 mL) and the layers were separated. Organic layer was washed with water (21 mL) and brine (21 mL), dried over MgSO₄, and concentrated to give crude product **19** (9.5 g, quantitative). The material was taken to the next step without purification (see Attachment 16).

Step 6. Preparation of Compound **B** (Ref: 24LL025)

To a solution of compound **19** (9.5 g, crude, lot # 24LL024) in CH₃CN (112 mL) was added a solution of sodium periodate (6.7 g, 31.28 mmol) and ruthenium (III) chloride hydrate (0.16 g, 0.782 mmol) in water (56 mL) at 0–5 °C. After the addition was complete, the mixture was stirred at 0–5 °C for 15 minutes, at which time ¹H NMR analysis showed it was complete. The reaction was quenched with 20% sodium thiosulfate (40 mL). The mixture was concentrated to an aqueous residue. *tert*-Butyl methyl ether (MTBE, 50 mL) was added and the layers were separated. The aqueous layer was extracted with another MTBE (50 mL). The combined organic layers were washed with brine (21 mL), dried over MgSO₄, and concentrated. The crude material was purified by chromatography eluted with 5–10% ethyl acetate in heptane to provide compound **B** (6.79 g, 85% yield) as a colorless solid (see Attachment 17).

4.3 Preparation of Compound **RCAI-56**

Step 1. Preparation of Compound **25** (Ref: 09GB028)

NaH (0.117 g, 2.8 mmol) was added to a solution of compound **A** (0.5 g, 0.9 mmol) in a mixture of DMF (10 mL) and THF (5 mL) at 0 °C. After stirring at 0 °C for 1 hour, a solution of compound **B** (0.68 g, 1.3 mmol) in THF (5 mL) was added at 0 °C. The reaction mixture was slowly heated to 70 °C and stirred at that temperature for 4 hours. The reaction was monitored by TLC analysis. After 4 hours, the reaction was not completed (compound **B** was consumed but compound **A** remained ≈30% by TLC analysis). Additional NaH (0.035 g, 0.88 mmol) and a solution of compound **B** (0.23 g, 0.43 mmol) in THF (5 mL) were added at 70 °C and stirred for overnight. After overnight, the reaction was not completed (compound **B** was consumed but compound **A** remained ≈15% by TLC analysis). Additional NaH (0.035 g, 0.88 mmol) and a solution of compound **B** (0.23 g, 0.43 mmol) in THF (5 mL) were added at 70 °C and stirred for 4 hours. After 4 hours, the reaction was deemed complete (less than 5.0% of compound **A** remaining). The reaction mixture was concentrated to a residue. The residue was dissolved in MTBE (65 mL) and cooled to below 0 °C. 20% aqueous H₂SO₄ solution (60 mL) was added dropwise at 0 to 5 °C. After stirring at 0 °C for 30 minutes, the reaction mixture was neutralized with solid K₂CO₃ (pH ≈8 to 9) at 0 °C. After stirring at 0 °C for 40 minutes, MTBE (40 mL) and water (20 mL) were added to this mixture. The mixture was filtered through a pad of Celite and the pad was washed with MTBE (2 × 10 mL). Combined organic layers were washed with water (2 × 10 mL) followed by saturated NaHCO₃ solution (10 mL) and brine (10 mL), dried over solid K₂CO₃, filtered, and concentrated to give the crude. The crude material was purified by chromatography eluted with 0–25% ethyl acetate in heptane to afford compound **25** (0.6 g, 70% yield) as a colorless oil (see Attachment 18).

Step 2. Preparation of Compound 26 (Ref: 09GB030)

To a solution of compound **25** (0.5 g, 0.5 mmol) in MeOH (21 mL) and dichloromethane (10.5 mL) was added *p*-TsOH•H₂O (0.12 g, 1.20 equiv). The reaction mixture was stirred at 60 °C for 16 hours, at which time the reaction was complete by TLC analysis. The reaction mixture was cooled to room temperature and concentrated to a residue which was then dissolved in EtOAc (15 mL) and saturated NaHCO₃ (5 mL) was added. The layers were separated. The aqueous layer was extracted with EtOAc (5 mL). Combined organic layers were washed with saturated NaHCO₃ solution (2.5 mL) and brine (2.5 mL), dried over MgSO₄, filtered and concentrated to give crude material which was purified by column chromatography with 10–80% EtOAc in heptane to give compound **26** (0.37 g, 77% yield) as oil (see Attachment 19).

Step 3. Preparation of Compound 27 (Ref: 09GB031)

To a solution of compound **26** (0.35 g, 0.4 mmol) in MeOH (21.35 mL) and cyclohexene (3.5 mL) at room temperature was added 1 M HCl (0.38 mL, 0.38 mmol) and 10% Pd/C (62 mg). The reaction mixture was stirred at 65 °C for 16 hours, at which time the reaction was checked by TLC analysis which showed not complete. 6 M HCl (0.13 mL, 0.8 mmol) and Pd/C (62 mg) were and stirred for another 16 hours, at which time TLC analysis still showed starting material. The reaction mixture was filtered and concentrated. The residue was dissolved in MeOH (21.35 mL) and to the solution was charged with cyclohexene (3.5 mL), and 10% Pd/C (62 mg). The reaction mixture was currently heated at 65 °C.

5.0 Analytical (Temporary)

HPLC Method 1

Column: Water XBridge C18, 3.5 μm, 4.6 × 75 mm, P/N 186003034
Column Temperature: Ambient temperature
Flow Rate: 1.0 mL/min
Detection: 230 and 210 nm
Analysis Time: 36 min
Mobile Phase A: 0.05% TFA in water
Mobile Phase B: 0.05% TFA in acetonitrile

Table 29

Time (min)	% A	% B
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15.0	1	99
30.0	1	99
31.0	95	5
36.0	95	5

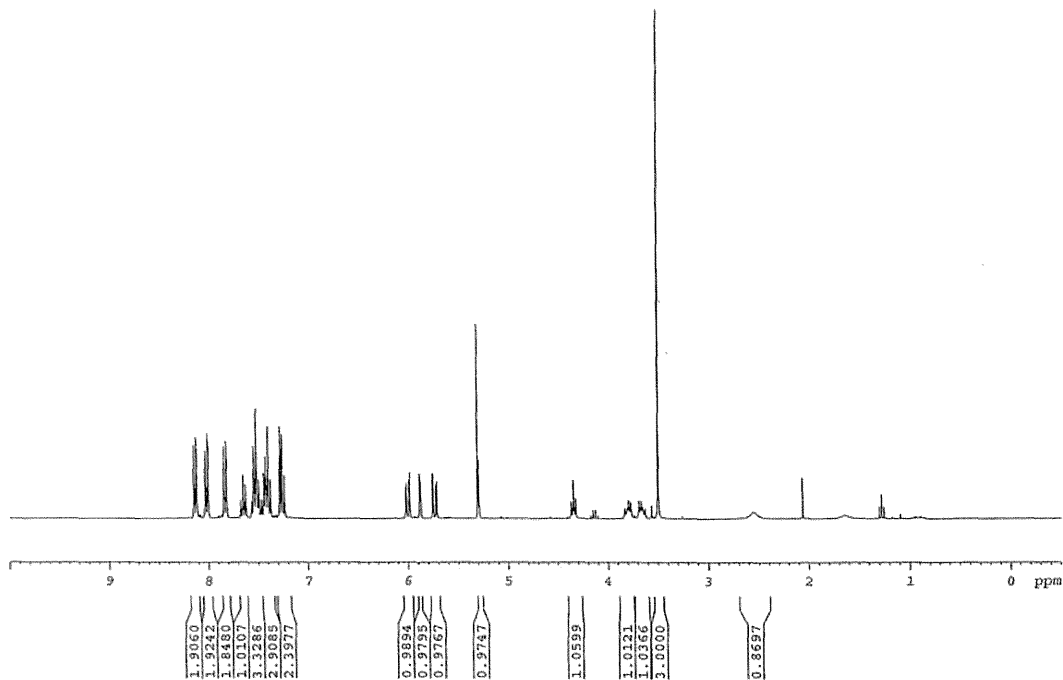
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Compound 4



Name ipella
Date 01/14/2014
NB # 09GB03H

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PROCNO 1
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Time_ 10.32
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SOLVENT CDCl3
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FIDRES 0.188380 Hz
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RG 256
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D1 1.00000000 sec
TDD 1

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SSB 0
LB 0.30 Hz
GB 0
PC 1.00



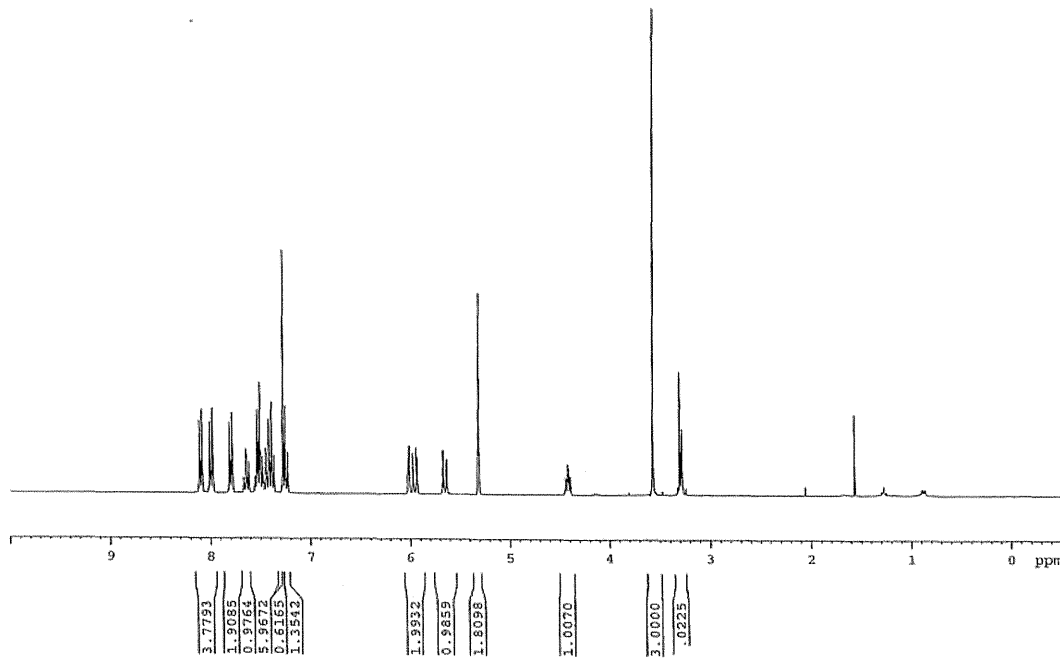
Attachment 1

Attachment 2

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location; 10
Compound 5



Name Geetha
Date 01/16/2014
NB# 09GB04A



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PROCNO 1
Date_ 20140116
Time 11.52
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PULPROG zg30
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SOLVENT CDCl3
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FIDRES 0.188380 Hz
AQ 2.6542580 sec
RG 362
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DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

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location; 10

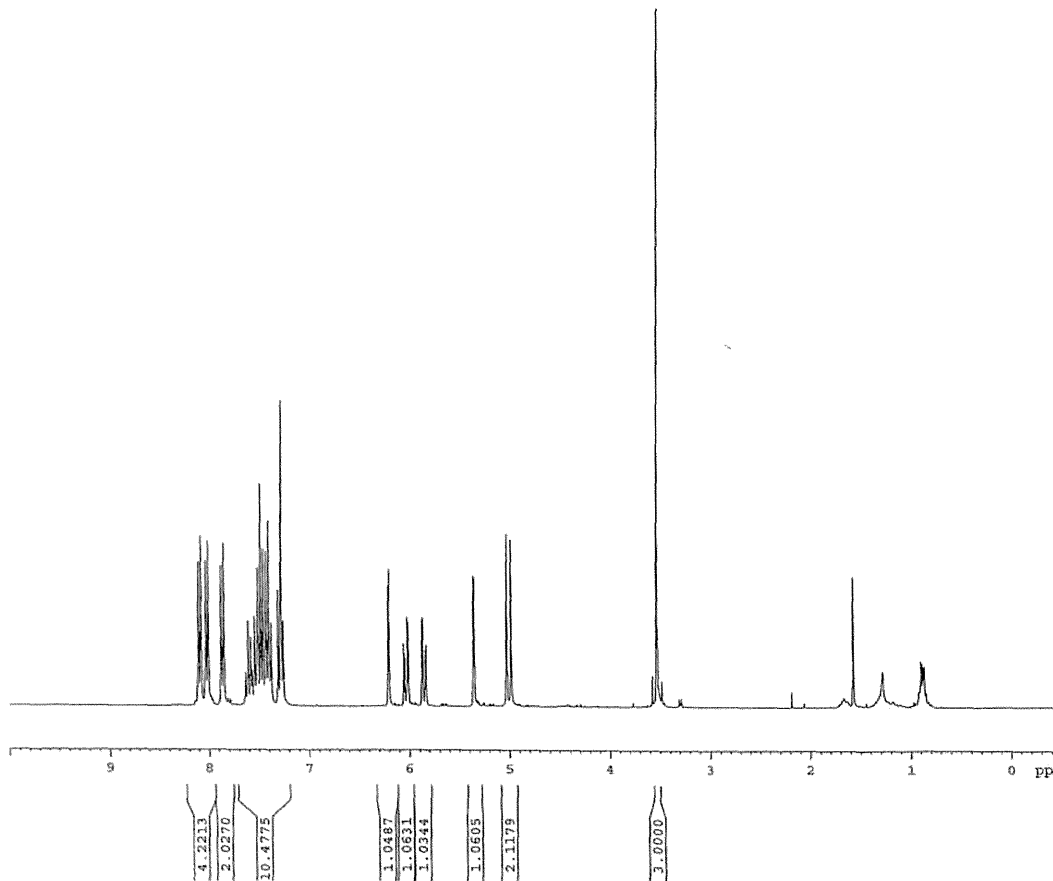
Compound 6



Name Geella
Date 02/07/2014
NB # 09GB012E

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PROCNO 1
Date_ 20140207
Time_ 10.32
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PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
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AQ 2.6542580 sec
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TD0 1

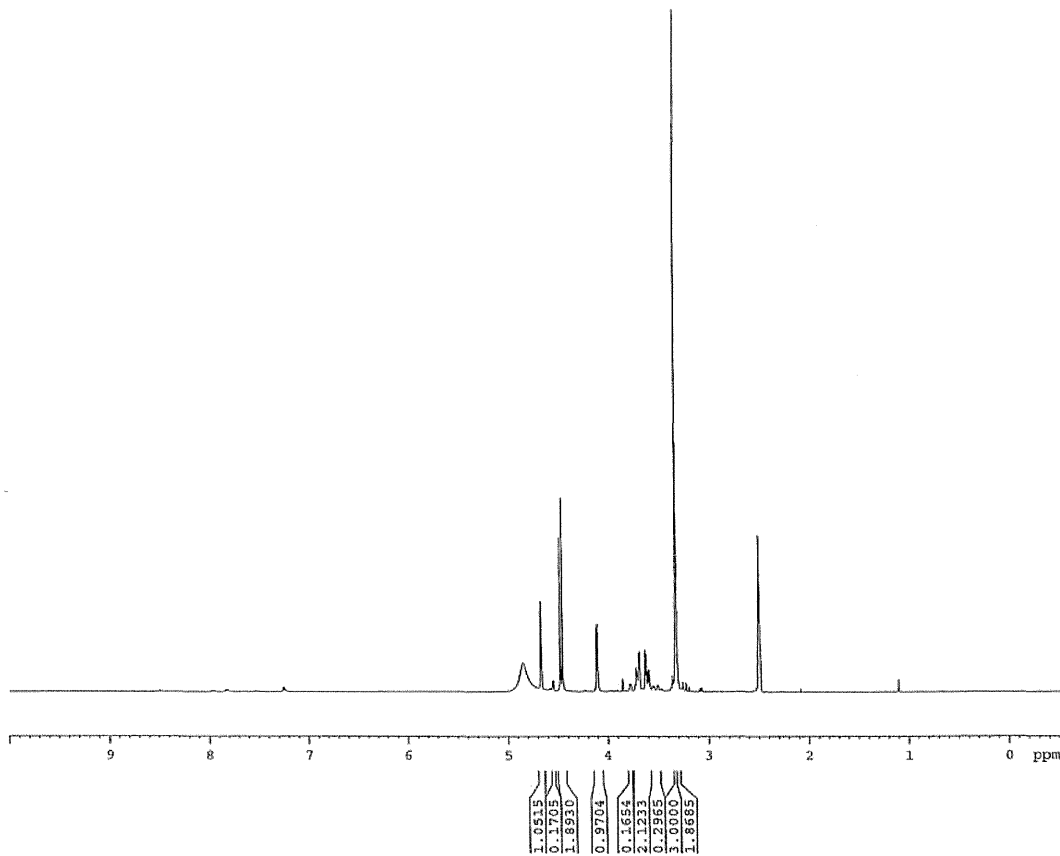
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PC 1.00



AMRI SRC AV-300
 location; 10
 Compound 7



Name Gpella
 Date 01/27/2014
 NB # 09GB06A



NAME 09GB06A
 EXPNO 10
 PROCNO 1
 Date_ 20140127
 Time_ 7.50
 INSTRUM spect
 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TD 32768
 SOLVENT DMSO
 NS 16
 DS 2
 SWH 6172.839 Hz
 FIDRES 0.188380 Hz
 AQ 2.6542580 sec
 RG 362
 DW 81.000 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.60000000 sec
 TD0 1

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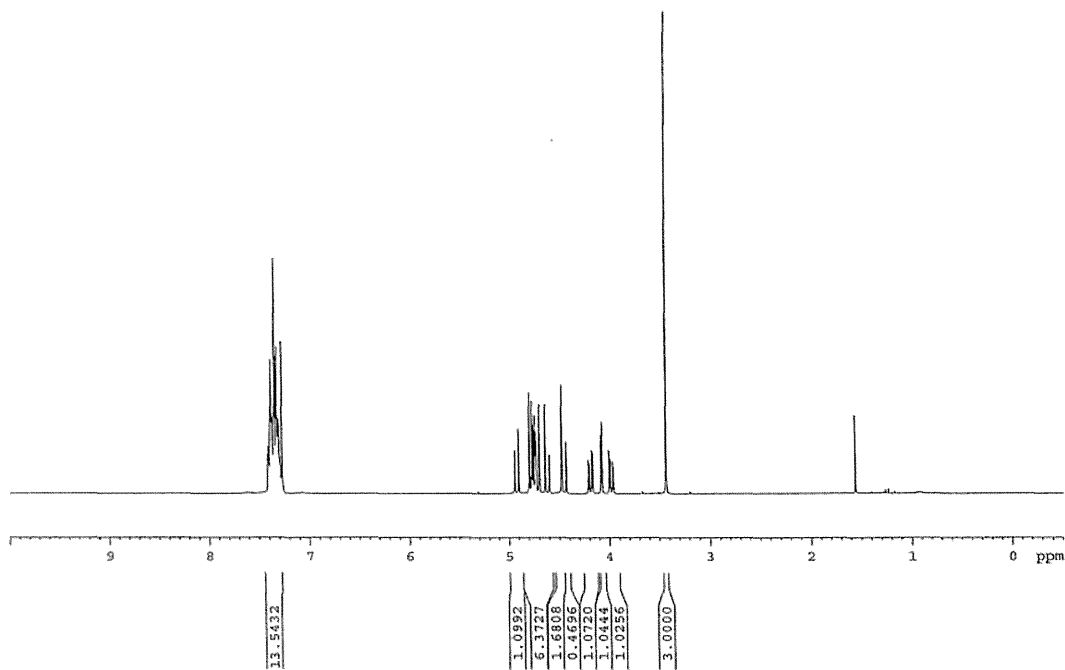
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Compound 8



Name Geelka
Date 01/31/2014
NB# 096B09B

NAME 09GB09B
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PROCNO 1
Date_ 20140131
Time 8.19
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PULPROG zg30
TD 32768
SOLVENT CDCl3
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DS 2
SWH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542580 sec
RG 362
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TE 300.0 K
D1 1.00000000 sec
TD0 1

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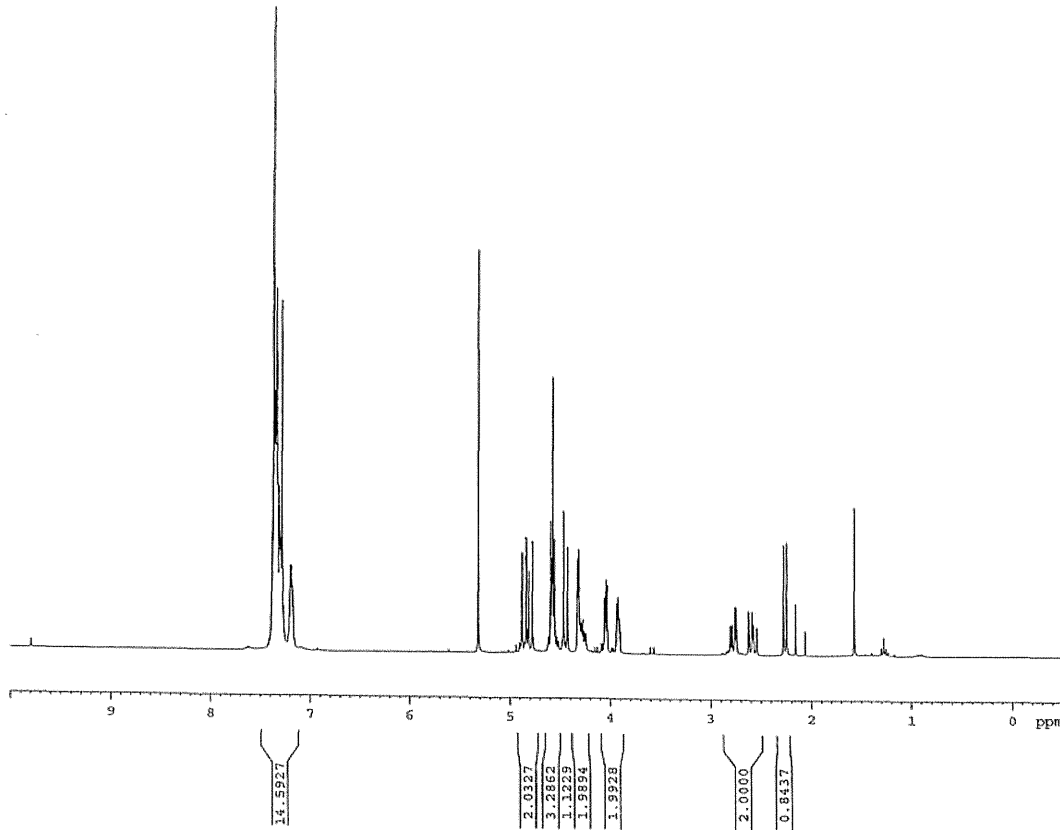
Attachment 5

Attachment 6

AMRI SRC AV-300
location; 10
Compound 9



Name Green
Date 02/06/2014
NB# 09GB011C



NAME 09GB011C
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PROCNO 1
Date_ 20140206
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PULPROG zg30
TD 32768
SOLVENT CDCl3
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DS 2
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AQ 2.6542580 sec
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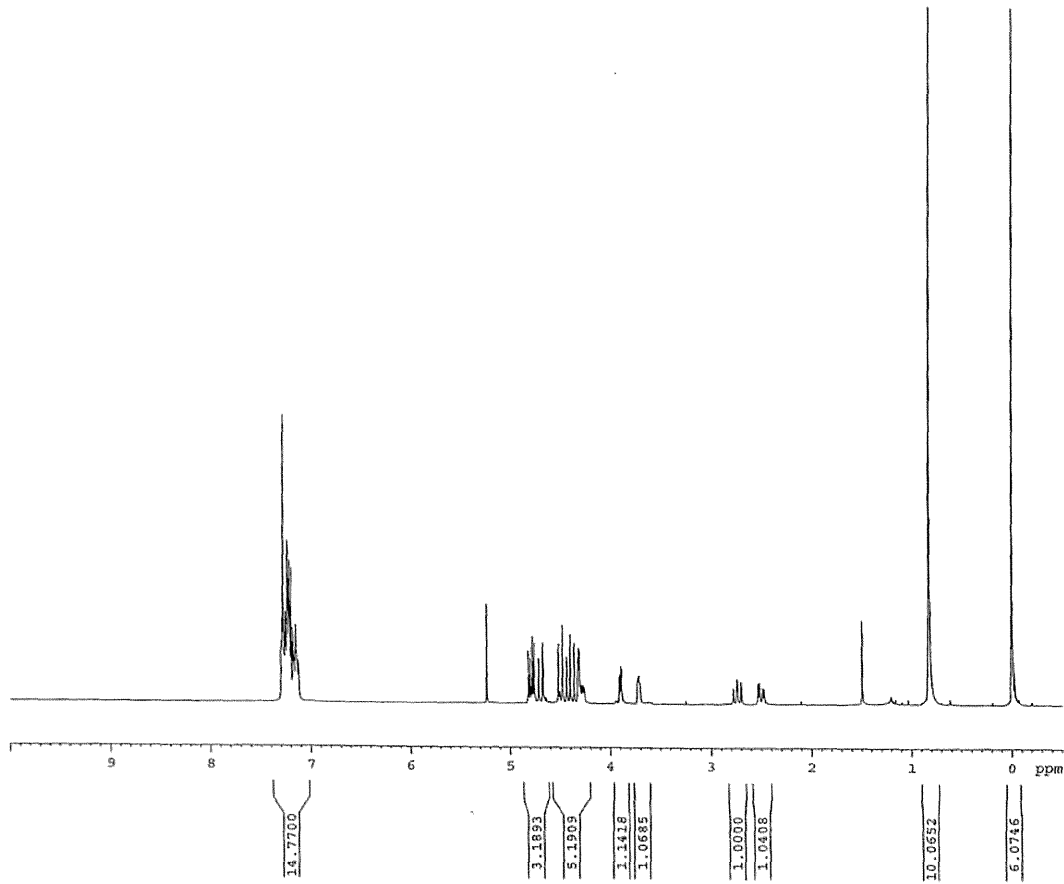
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LB 0.30 Hz
GB 0
PC 1.00

Attachment 7

AMRI SRC AV-300
location; 10
Compound 10



Name Gretha
Date 02/07/2014
NB # 09GB13B



NAME 09GB13B
EXPNO 10
PROCNO 1
Date_ 20140207
Time 13.50
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TD 32768
SOLVENT CDC13
NS 16
DS 2
SMH 6172.839 Hz
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AQ 2.6542580 sec
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TDO 1

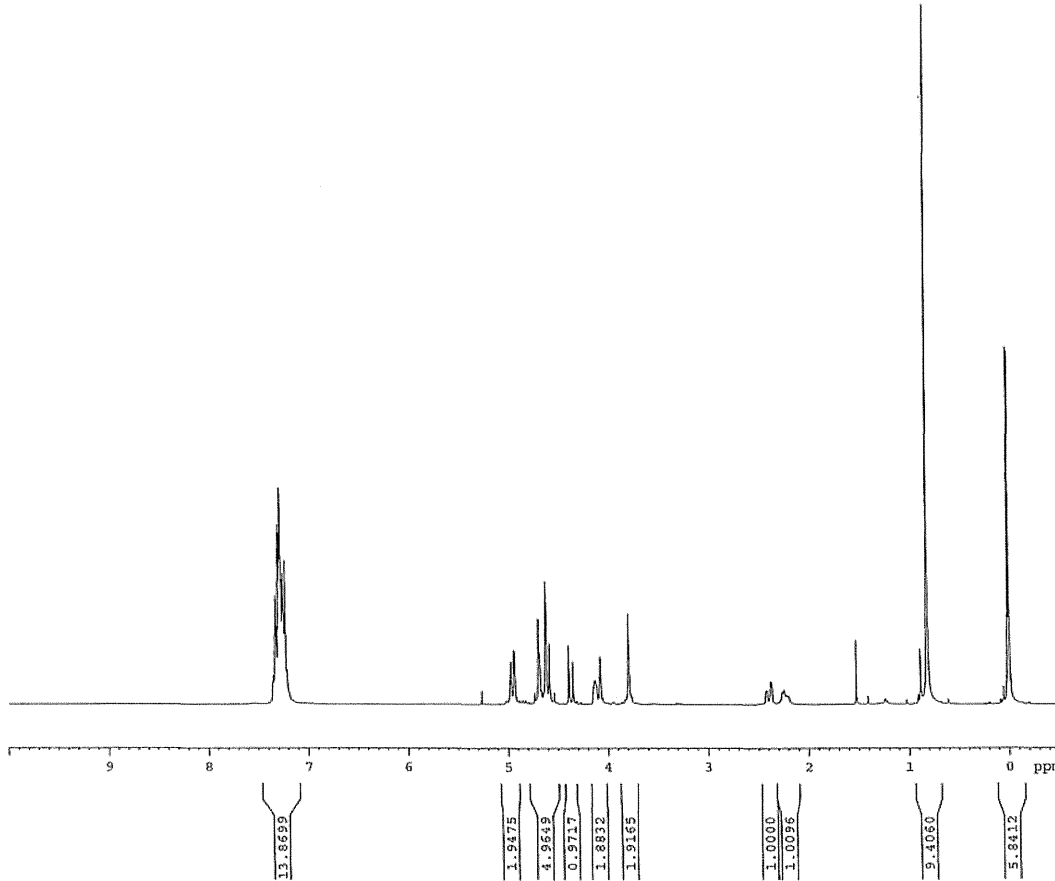
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Attachment 8

AMRI SRC AV-300
location; 10
Compound 11



Name Geelka
Date 02/11/2014
NB # 09GB015B



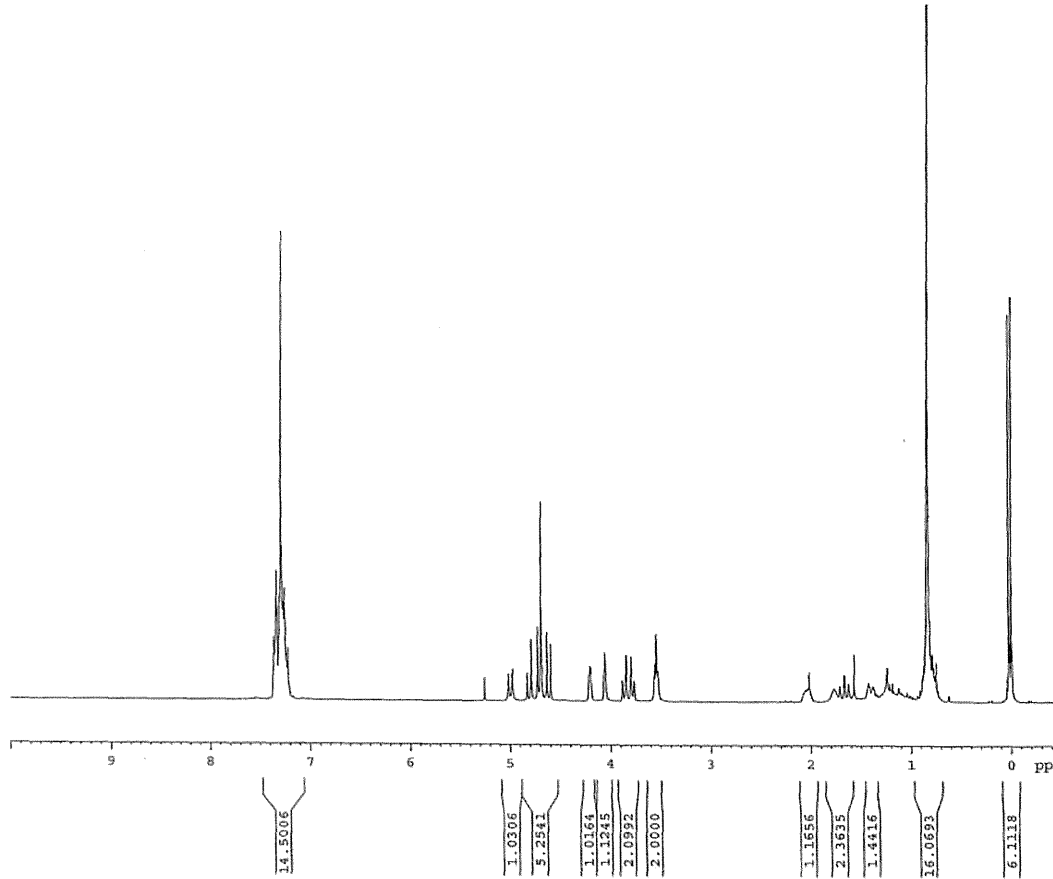
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RG 128
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TD0 1

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AMRI SRC AV-300
 location; 10
 Compound 12



Name hcelka
 Date 02/14/2014
 NB # 09GB17B



NAME 09GB17B
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 Date 20140214
 Time 13.54
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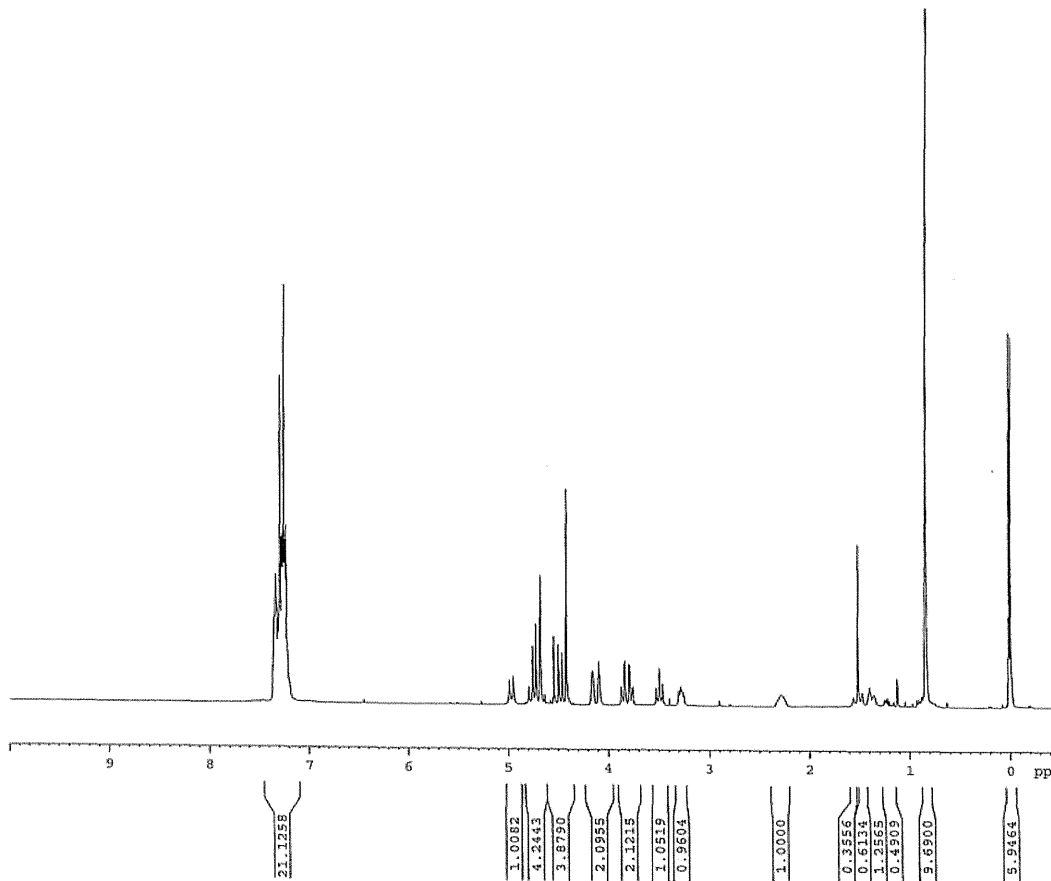
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Attachment 10

AMRI SRC AV-300
 location; 10
 Compound 13



Name Geeika
 Date 02/19/2014
 NB # 09GB18C



NAME 09GB18C
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 PROCNO 1
 Date_ 20140219
 Time 8.06
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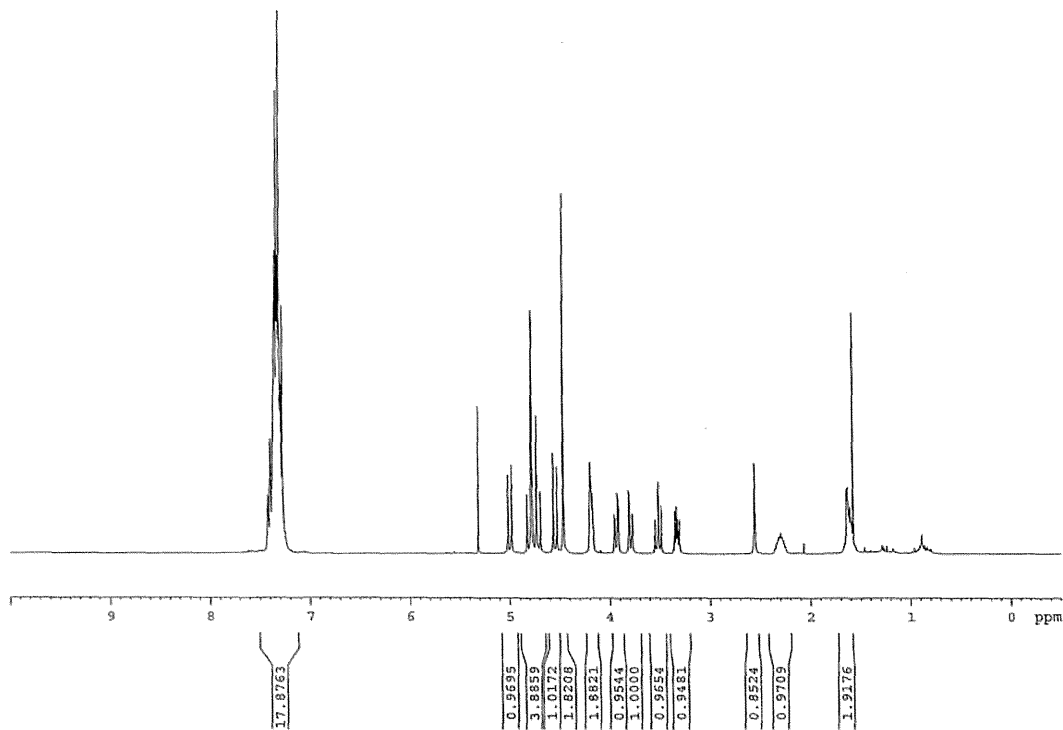
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Attachment 11

AMRI SRC AV-300
location; 10
Compound A



Name Geetha
Date 03/13/2014
NB# 09GB025C



NAME 09GB025C
EXPNO 10
PROCNO 1
Date 20140313
Time 9.45
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PULPROG zg30
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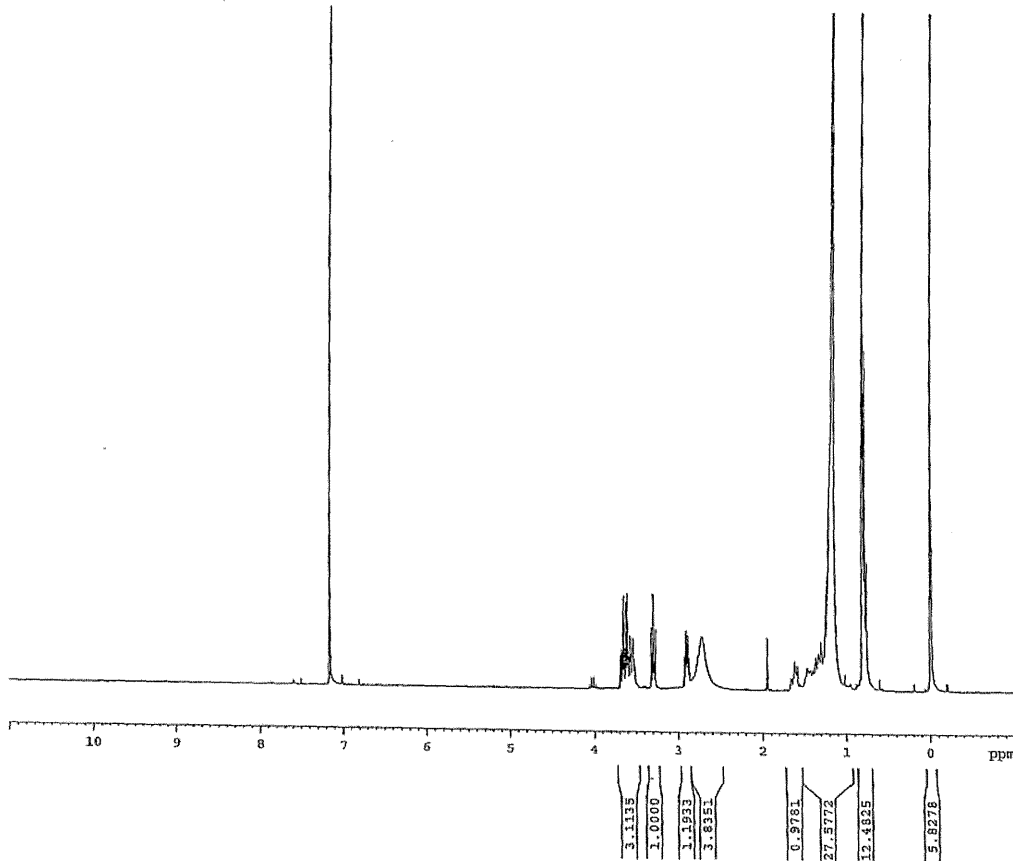
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LB 0.30 Hz
GB 0
PC 1.00

Attachment 12

AMRI SRC AV-300
location: 10
Compound 15



Name Di Xu
Date 3/6/2014
NB # 14KL0187



NAME 14KL0187
EXFNO 10
PROCNO 1
Date 20140306
Time 11.12
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PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
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TDO 1

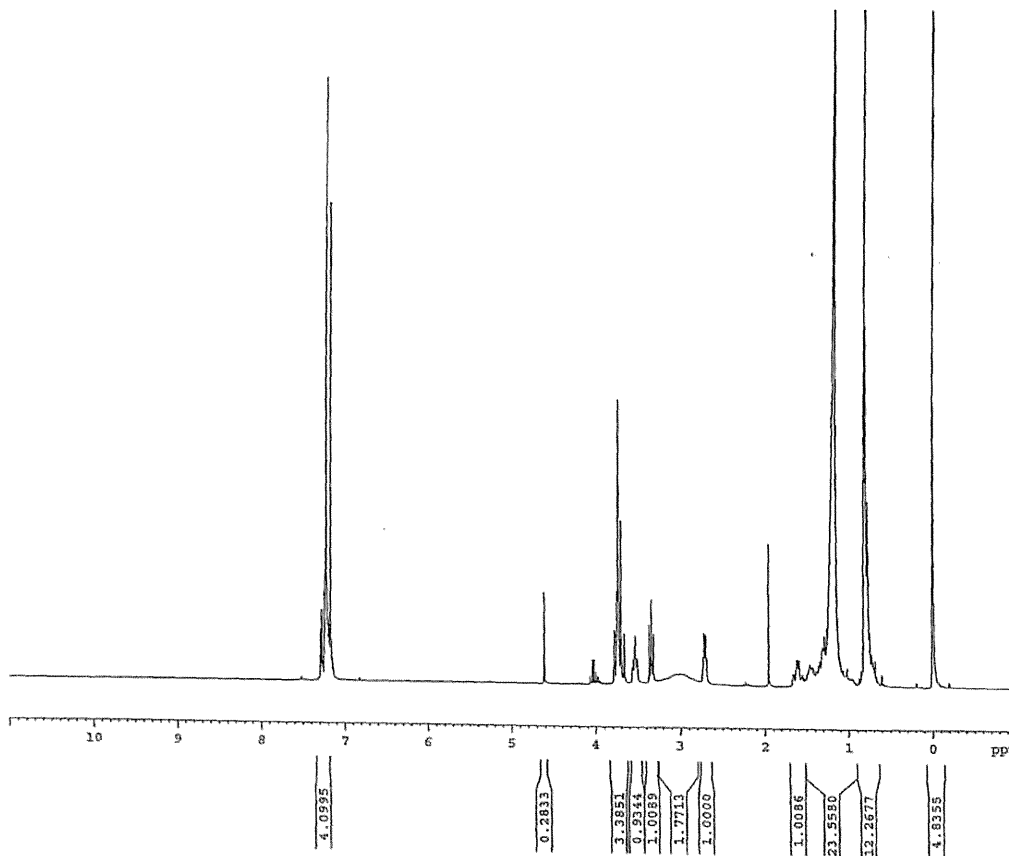
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SI 16384
SF 300.1400345 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Attachment 13

AMRI SRC AV-300
location; 10
Compound 16



Name Di Xu
Date 3/13/2014
NB# 14KLO210



NAME 14KL0210
EXPNO 10
PROCNO 1
Date_ 20140313
Time 16.29
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PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
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TD0 1

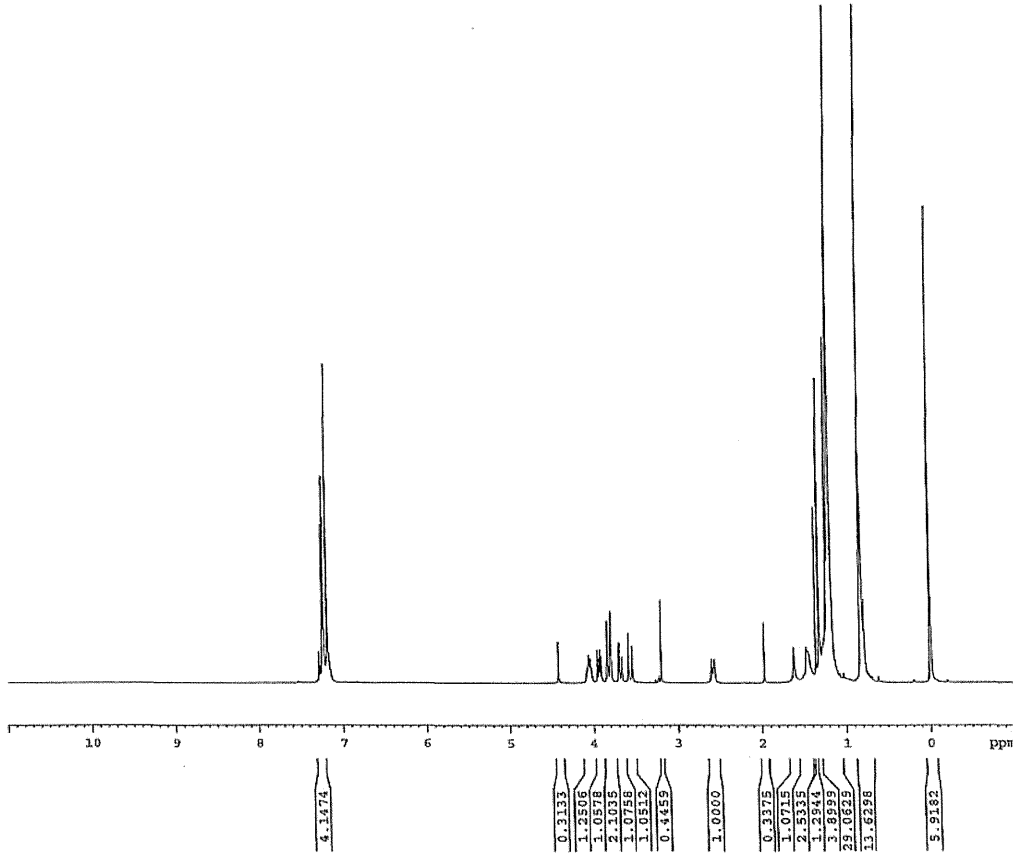
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GB 0
PC 1.00

Attachment 14

AMRI SRC AV-300
location; 10
Compound 17



Name Di Xu
Date 02/18/2014
NB # 09DX022B



NAME 09DX022B
EXPNO 10
PROCNO 1
Date_ 20140318
Time 9.33
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PULPROG zg30
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SOLVENT CDCl3
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SWH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542580 sec
RG 362
DN 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

***** CHANNEL f1 *****
NUC1 1H
P1 12.75 usec
PL1 2.00 dB
SFO1 300.1418534 MHz
SE 16384
SF 300.1400268 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

AMRI SRC AV-300
 location; 10
 Compound 18



Name Di Xu
 Date 3/20/2014
 NB # 09DX023

NAME 09DX023
 EXPNO 20
 PROCNO 1
 Date_ 20140320
 Time 12.19
 INSTRUM spect
 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 6172.839 Hz
 FIDRES 0.188380 Hz
 AQ 2.6542580 sec
 RG 256
 DW 81.000 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 12.75 usec
 PL1 2.00 dB
 SFO1 300.1418534 MHz
 SI 16384
 SF 300.1400268 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

