**Domino reaction for the synthesis of highly functionalized triazatricyclo [6.2.2.0^{1,6}]
dodecane**

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1. Experimental Section

1.1. General methods

Chemicals were purchased from Aldrich and they were used without further purification. TLC -Thin layer chromatography (Merck, Silica gel 60 F254) - was performed on pre-coated silica gel on alumina plates for monitoring the reaction. FT-IR spectra were recorded in the range of 4000-400 cm^{-1} on JASCO-4100 spectrometer instrument using KBr pellets. ^1H , ^{13}C NMR spectra, DEPT-90 and DEPT-135 were recorded using a BRUKER AMX 400 FT. HPLC was performed on a water's liquid chromatography equipped with a dual wavelength UV detector (Deuterium lamp, 192–600 nm), using a symmetry C18 column (10 cm X 4.6mm) 5 μ HPLC grade. Mass Spectroscopy – ESI, Triple quad (Waters Quattro premier XE) was used to analyse the ESI mass. HRMS analysis was obtained from JEOL GC Mate.

1.2. General procedure for the preparation of Synthesis of products 4a-j

A dry 100ml Erlenmeyer flask was charged with N-benzyl-4-piperidone (10 mmol); aromatic aldehydes (20 mmol); cyanoacetamide (20 mmol); sodium hydroxide (0.5 mol %) and methanol (15 ml). The reaction mixture was stirred at room temperature for 30-60 min. The reaction was monitored by TLC and after the completion of reaction, the mixture was neutralised using 0.1 N HCl and extracted with DCM (3 X 20 ml). The crude reaction mixture was purified by column chromatography on silica gel using ethyl acetate/hexane as the eluents.

Spectral data

1.2.1. 2-benzylidene-4-benzyl-7-phenyl-4,10,11-

triazatricyclo[6.2.2.0^{1,6}]dodecane-9,12-dione (4a): Light creamy powder; Yield 70%; mp > 300°C; R_f 0.67 (60% ethyl acetate : hexane); FTIR (KBr) v: 3541, 3309, 3045, 2870, 1687 cm⁻¹; ¹H NMR (400 MHz, DMSO-d₆): 1.63 (s, CH₂, 2H), 1.92 (s, CH, 1H), 1.99 (s, CH, 1H), 2.41 (t, CH₂, 1H), 2.76 (q, CH₂, 1H), 3.94 (s, CH, 1H), 4.04 (s, CH₂, 2H), 6.05 (s, vinylic-H, 1H), 6.98-7.49 (ArH, 15H), 9.43 (s, amide NH, 2H) ppm; ¹³C NMR (100MHz, DMSO-d₆): 26.36, 28.90, 49.68, 58.39, 70.91, 126.48, 127.68, 128.04, 128.95, 129.08, 129.66, 131.50, 133.35, 137.32 169.60, 169.73 ppm; HRMS: calculated for C₂₉H₂₇N₃O₂ ([M]⁺) 449.2103, Found 449.2102.

1.2.2. 2-(2-fluorobenzylidene)-4-benzyl-7-(2-fluorophenyl)-4,10,11-

triazatricyclo [6.2.2.0^{1,6}]dodecane-9,12-dione (4b): Light yellow powder; Yield 72%; mp > 300°C; R_f 0.47 (60% ethyl acetate : hexane); FTIR (KBr) v: 3582, 3297, 3089, 2830, 1698 cm⁻¹; ¹H NMR (400 MHz, DMSO-d₆): 1.92 (s, CH₂, 2H), 2.41 (s, CH₂, 2H), 3.06-3.08 (t, CH, 1H), 3.19-3.02 (q, CH, 1H), 3.78 (s, CH₂, 2H), 4.02 (s, CH, 1H), 6.15 (s, vinylic-H, 1H), 6.79-7.62 (ArH, 13H), 9.31 (s, amide NH, 2H) ppm; ¹³C NMR (100MHz, DMSO-d₆): 25.91, 28.89, 56.19, 59.87, 73.67, 118.21, 119.83, 126.25, 126.29, 127.92, 130.76, 131.93, 133.13, 134.32, 135.01, 135.88, 139.28, 140.03, 140.05, 159.91, 169.11 ppm; HRMS: calculated for C₂₉H₂₅F₂N₃O₂ ([M]⁺) 485.1915, Found 485.1914.

1.2.3. 2-(3-fluorobenzylidene)-4-benzyl-7-(3-fluorophenyl)-4,10,11-

triazatricyclo [6.2.2.0^{1,6}]dodecane-9,12-dione (4c): Yellow powder; Yield 60%; mp > 300°C; R_f 0.49 (60% ethyl acetate : hexane); FTIR (KBr) v: 3501, 3287, 2992, 2804, 1681 cm⁻¹; ¹H NMR (400 MHz, DMSO-d₆): 1.72 (s, CH₂, 2H), 2.31-2.35 (t, CH, 1H), 2.55-2.59 (q, CH, 1H), 3.49 (s, CH₂, 2H), 3.78 (s, CH, 1H), 3.94 (s, CH₂,

2H), 6.89 (s, vinylic-H, 1H), 7.32-8.08 (ArH, 13H), 9.12 (s, amide NH, 2H) ppm; ^{13}C NMR (100MHz, DMSO- d_6): 29.02, 29.95, 39.11, 56.75, 60.04, 70.98, 64.52, 121.71, 122.01, 126.85, 126.89, 127.01, 129.81, 130.01, 130.04, 130.09, 131.12, 134.35, 134.63, 137.76, 137.79, 141.98, 142.02, 159.98, 160.00, 169.12 ppm; HRMS: calculated for $\text{C}_{29}\text{H}_{25}\text{F}_2\text{N}_3\text{O}_2$ ($[\text{M}]^+$) 485.1915, Found 485.1915.

1.2.4. 2-(4-fluorobenzylidene)-4-benzyl-7-(4-fluorophenyl)-4,10,11-

triazatricyclo [6.2.2.0^{1,6}]dodecane-9,12-dione (4d): Yellow powder; Yield 68%; mp > 300°C; R_f 0.57 (60% ethyl acetate : hexane); FTIR (KBr) ν : 3492, 3313, 3033, 2863, 1648 cm^{-1} ; ^1H NMR (400 MHz, DMSO- d_6): 1.98 (s, CH_2 , 2H), 2.38-2.41 (t, CH, 1H), 2.58-2.62 (q, CH, 1H), 2.85 (s, CH_2 , 2H), 3.19 (s, CH, 1H), 3.84 (s, CH_2 , 2H), 6.17 (s, vinylic-H, 1H), 7.21-8.20 (ArH, 13H), 9.50 (s, amide NH, 2H) ppm; ^{13}C NMR (100MHz, DMSO- d_6): 29.91, 30.05, 38.89, 46.84, 53.21, 67.27, 117.82, 117.87, 121.01, 121.04, 124.93, 125.04, 129.01, 129.90, 130.04, 130.07, 132.07, 133.46, 136.89, 140.04, 140.94, 159.96, 168.82 ppm; HRMS: calculated for $\text{C}_{29}\text{H}_{25}\text{F}_2\text{N}_3\text{O}_2$ ($[\text{M}]^+$) 485.1915, Found 485.1913.

1.2.5. 2-(2-methylbenzylidene)-4-benzyl-7-(2-methylphenyl)-4,10,11-

triazatricyclo [6.2.2.0^{1,6}]dodecane-9,12-dione (4e): Yellowish brown powder; Yield 66%; mp > 300°C; R_f 0.52 (60% ethyl acetate : hexane); FTIR (KBr) ν : 3548, 3301, 3096, 2878, 1699 cm^{-1} ; ^1H NMR (400 MHz, DMSO- d_6): 1.57 (s, CH_2 , 2H), 1.99 (s, CH_3 , 6H), 2.28-2.31 (t, CH, 1H), 2.46 (s, CH_2 , 2H), 2.64-2.68 (q, CH, 1H), 2.96 (s, CH, 1H), 4.08 (s, CH_2 , 2H), 6.52 (s, vinylic-H, 1H), 7.09-8.09 (ArH, 13H), 9.20 (s, amide NH, 2H) ppm; ^{13}C NMR (100MHz, DMSO- d_6): 22.01, 28.82, 32.28, 39.18, 48.37, 50.00, 69.01, 118.97, 119.02, 125.04, 126.63, 126.67, 128.94, 129.85, 130.00, 130.04, 130.88, 130.91, 134.89, 137.85, 144.89, 145.01, 169.58 ppm; HRMS: calculated for $\text{C}_{31}\text{H}_{31}\text{N}_3\text{O}_2$ ($[\text{M}]^+$) 477.2416, Found 477.2416.

1.2.6. 2-(3-methylbenzylidene)-4-benzyl-7-(3-methylphenyl)-4,10,11-

triazatricyclo [6.2.2.0^{1,6}]dodecane-9,12-dione (4f): Brownish yellow powder; Yield 58%; mp > 300°C; R_f 0.83 (60% ethyl acetate : hexane); FTIR (KBr) v: 3591, 3284, 3002, 2812, 1674 cm⁻¹; ¹H NMR (400 MHz, DMSO-d₆): 1.56 (s, CH₂, 2H), 1.86 (s, CH₃, 6H), 2.25-2.28 (t, CH, 1H), 2.50-2.55 (q, CH, 1H), 3.21 (s, CH₂, 2H), 3.85 (s, CH₂, 2H), 3.91 (s, CH, 1H), 6.40 (s, vinylic-H, 1H), 7.15-8.28 (ArH, 13H), 9.01 (s, amide NH, 2H) ppm; ¹³C NMR (100MHz, DMSO-d₆): 20.09, 28.98, 30.01, 36.63, 49.99, 53.24, 67.76, 118.92, 119.98, 120.01, 122.41, 125.86, 126.71, 128.93, 129.96, 130.01, 130.06, 137.76, 138.59, 142.42, 142.65, 168.27 ppm; HRMS: calculated for C₃₁H₃₁N₃O₂ ([M]⁺) 477.2416, Found 477.2415.

1.2.7. 2-(4-methylbenzylidene)-4-benzyl-7-(4-methylphenyl)-4,10,11-

triazatricyclo [6.2.2.0^{1,6}]dodecane-9,12-dione (4g): Light yellow powder; Yield 62%; mp > 300°C; R_f 0.78 (60% ethyl acetate : hexane); FTIR (KBr) v: 3556, 3067, 2889, 1697 cm⁻¹; ¹H NMR (400 MHz, DMSO-d₆): 2.05 (s, CH₂, 2H), 2.27 (s, CH₃, 6H), 2.62-2.66 (t, CH, 1H), 2.82-2.87 (q, CH, 1H), 3.19 (s, CH₂, 2H), 3.80 (s, CH, 1H), 3.82 (s, CH₂, 2H), 6.31 (s, vinylic-H, 1H), 7.12-7.77 (ArH, 13H), 8.98 (s, amide NH, 2H) ppm; ¹³C NMR (100MHz, DMSO-d₆): 20.07, 28.91, 29.98, 37.03, 46.74, 49.58, 68.74, 119.58, 120.01, 120.05, 125.65, 128.89, 129.21, 129.24, 130.00, 130.06, 131.03, 131.55, 132.86, 134.94, 135.06, 137.89, 141.56, 141.95, 168.99 ppm; HRMS: calculated for C₃₁H₃₁N₃O₂ ([M]⁺) 477.2416, Found 477.2413.

1.2.8. 2-(2-chlorobenzylidene)-4-benzyl-7-(2-chlorophenyl)-4,10,11-

triazatricyclo [6.2.2.0^{1,6}]dodecane-9,12-dione (4h): Light creamy powder; Yield 71%; mp > 300°C; R_f 0.38 (60% ethyl acetate : hexane); FTIR (KBr) v: 3562, 3372, 3064, 2841, 1672 cm⁻¹; ¹H NMR (400 MHz, DMSO-d₆): 1.90 (s, CH₂, 2H), 2.26-2.29 (t, CH, 1H), 2.68-2.73 (q, CH, 1H), 3.24 (s, CH₂, 2H), 3.39 (s, CH, 1H), 3.82

(s, CH₂, 2H), 6.27 (s, vinylic-H, 1H), 7.00-8.37 (ArH, 13H), 9.23 (s, amide NH, 2H) ppm; ¹³C NMR (100MHz, DMSO-d₆): 24.03, 26.94, 40.05, 50.00, 52.17, 67.96, 127.86, 128.83, 129.01, 129.89, 130.05, 132.87, 138.76, 138.78, 140.08, 143.87, 170.00 ppm; HRMS: calculated for C₂₉H₂₅Cl₂N₃O₂ ([M]⁺) 517.1324, Found 517.1324.

1.2.9. 2-(4-chlorobenzylidene)-4-benzyl-7-(4-chlorophenyl)-4,10,11-

triazatricyclo [6.2.2.0^{1,6}]dodecane-9,12-dione (4i): Light yellow powder Solid; Yield 70%; mp > 300°C; R_f 0.42 (60% ethyl acetate : hexane); FTIR (KBr) v: 3593, 3284, 3094, 2915, 1679 cm⁻¹; ¹H NMR (400 MHz, DMSO-d₆): 2.22 (s, CH₂, 2H), 2.31-2.35 (t, CH, 1H), 2.52-2.57 (q, CH, 1H), 3.15 (s, CH₂, 2H), 3.75 (s, CH, 1H), 4.56 (s, CH₂, 2H), 6.59 (s, vinylic-H, 1H), 6.90-7.98 (ArH, 13H), 9.31 (s, amide NH, 2H) ppm; ¹³C NMR (100MHz, DMSO-d₆): 27.29, 29.97, 37.71, 45.89, 49.98, 68.02, 124.82, 129.87, 129.86, 129.96, 130.04, 130.86, 130.95, 131.11, 132.05, 135.01, 136.98, 137.08, 141.88, 141.94, 168.94 ppm; HRMS: calculated for C₂₉H₂₅Cl₂N₃O₂ ([M]⁺) 517.1324, Found 517.1325.

1.2.10. 2-((naphthalen-1-yl)methylene)-4-benzyl-7-(1-naphthyl)-4,10,11-

triazatricyclo [6.2.2.0^{1,6}]dodecane-9,12-dione (4j): Light yellowish powder Solid; Yield 61%; mp > 300°C; R_f 0.64 (60% ethyl acetate : hexane); FTIR (KBr) v: 3545, 3313, 3056, 2810, 1653 cm⁻¹; ¹H NMR (400 MHz, DMSO-d₆): 1.99 (s, CH₂, 2H), 2.24-2.27 (t, CH, 1H), 2.52-2.58 (q, CH, 1H), 3.18 (s, CH, 1H), 3.25 (s, CH₂, 2H), 3.69 (s, CH₂, 2H), 6.38 (s, vinylic-H, 1H), 6.85-7.54 (ArH, 19H), 8.61 (s, amide NH, 2H) ppm; ¹³C NMR (100MHz, DMSO-d₆): 28.99, 29.93, 37.81, 46.69, 47.83, 67.84, 119.15, 123.54, 125.02, 125.74, 126.58, 128.13, 128.21, 129.28, 129.97, 129.99, 130.02, 130.11, 132.03, 133.42, 135.09, 135.56, 136.26, 137.47, 138.36,

138.76, 138.83, 167.58 ppm; HRMS: calculated for $C_{37}H_{31}N_3O_2$ ($[M]^+$) 549.2416,
Found 549.2418.

^1H & ^{13}C – NMR of 2-benzylidene-4-benzyl-7-phenyl-4, 10, 11-triazatricyclo [6.2.2.0^{1,6}] dodecane-9, 12-dione (4a)

NBPParCN

Current Data Parameters
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EXPNO 27
PROCNO 1

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SWH 8223.685 Hz
FIDRES 0.125483 Hz
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RG 143.73
DW 60.800 usec
DE 6.50 usec
TE 295.7 K
D1 1.00000000 sec
TD0 1

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PLW1 14.00000000 W
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F2 - Processing parameters
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NBPPar C13

Current Data Parameters
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PROCNO 1

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SFO1 100.6550182 MHz

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LB 1.00 Hz
GB 0
PC 1.40

DEPT-135 & 90 spectra of 2-benzylidene-4-benzyl-7-phenyl-4, 10, 11-triazatricyclo [6.2.2.0^{1,6}] dodecane-9, 12-dione (4a)

HPLC data for compound (4a) in acetonitrile

Default Individual Report

Reported by User: System

Project Name: Defaults

SAMPLE INFORMATION

Sample Name: NBPPaCN
Sample Type: Unknown
Vial: 1
Injection #: 4
Injection Volume: 10.00 ul
Run Time: 20.0 Minutes
Sample Set Name:

Acquired By: System
Date Acquired: 2/19/2013 12:45:46 PM
Acq. Method Set: vit1
Date Processed: 2/19/2013 12:59:09 PM
Processing Method: VIT4
Channel Name: 2487Channel 2
Proc. Chnl. Descr.: 263

	Peak Name	RT	Area	% Area	Height
1	NBPPaCN	5.612	38138011	100.00	2763067

Mass spectra of 2-benzylidene-4-benzyl-7-phenyl-4, 10, 11-triazatricyclo [6.2.2.0^{1,6}] dodecane-9, 12-dione (4a)

HRMS spectra of 2-benzylidene-4-benzyl-7-phenyl-4, 10, 11-triazatricyclo [6.2.2.0^{1,6}] dodecane-9, 12-dione (4a)

¹H & ¹³C – NMR of 2-(2-fluorobenzylidene)-4-benzyl-7-(2-fluorophenyl)-4,10,11-triazatricyclo
[6.2.2.0^{1,6}] dodecane-9,12-dione (4b)

NBP2F

Current Data Parameters
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PROCNO 1

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RG 175.97
DW 60.800 usec
DE 6.50 usec
TE 294.8 K
D1 1.00000000 sec
TD0 1

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PLW1 14.00000000 W
SFO1 400.2604718 MHz

F2 - Processing parameters
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PC 1.00

NBP2F

Current Data Parameters
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EXPNO 21
PROCNO 1

F2 - Acquisition Parameters
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FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 175.97
DW 20.800 usec
DE 6.50 usec
TE 295.2 K
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DELTA 1.89999998 sec
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SFO1 100.6550182 MHz
NUC1 13C
P1 9.80 usec
PLW1 58.00000000 W
SFO2 400.2596010 MHz
NUC2 1H
CPDPRG2 waltz16
PCPD2 90.00 usec
PLW2 14.00000000 W
PLW12 0.35097000 W
PLW13 0.28428999 W

F2 - Processing parameters
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SF 100.6450043 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

¹H & ¹³C – NMR of 2-(3-fluorobenzylidene)-4-benzyl-7-(2-fluorophenyl)-4,10,11-triazatricyclo
[6.2.2.0^{1,6}] dodecane-9,12-dione (4c)

NBP3F

BRUKER

Current Data Parameters
NAME NBP3F
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130311
Time 22.12
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9845889 sec
RG 127.79
DW 60.800 usec
DE 6.50 usec
TE 294.1 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 14.25 usec
PLW1 14.00000000 W
SFO1 400.2604718 MHz

F2 - Processing parameters
SI 65536
SF 400.2579875 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

NBP3F C13

BRUKER

Current Data Parameters
NAME NBP3F C13
EXPNO 12
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130403
Time 16.27
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 512
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 175.97
DW 20.800 usec
DE 6.50 usec
TE 295.6 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999999 sec
TD0 1
SFO1 100.6550182 MHz
NUC1 13C
P1 9.80 usec
PLW1 58.00000000 W
SFO2 400.2596010 MHz
NUC2 1H
CPDPRG[2] waltz16
PCPD2 90.00 usec
PLW2 14.00000000 W
PLW12 0.35097000 W
PLW13 0.28428999 W

F2 - Processing parameters
SI 32768
SF 100.6449913 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

¹H & ¹³C – NMR of 2-(4-fluorobenzylidene)-4-benzyl-7-(2-fluorophenyl)-4,10,11-triazatricyclo
[6.2.2.0^{1,6}] dodecane-9,12-dione (4d)

NBP4F

Current Data Parameters
NAME NBP4F
EXPNO 32
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130124
Time 15.22
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8223.686 Hz
FIDRES 0.126483 Hz
AQ 3.9845889 sec
RG 127.79
DW 60.800 usec
DE 6.50 usec
TE 297.2 K
D1 1.00000000 sec
TD0 1

CHANNEL f1
NUC1 1H
P1 14.25 usec
PLW1 14.00000000 W
SFO1 400.2604718 MHz

F2 - Processing parameters
SI 65536
SF 400.2579543 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

NBP4F C13

Current Data Parameters
NAME NBP4F C13
EXPNO 17
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130404
Time 4.39
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 512
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 156.91
DW 20.800 usec
DE 6.50 usec
TE 295.1 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.899999998 sec
TD0 1
SFO1 100.6550182 MHz
NUC1 13C
P1 9.80 usec
PLW1 58.00000000 W
SFO2 400.2596010 MHz
NUC2 1H
CPDPRG[2] waltz16
PCPD2 90.00 usec
PLW2 14.00000000 W
PLW12 0.35097000 W
PLW13 0.28428999 W

F2 - Processing parameters
SI 32768
SF 100.6450043 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

**¹H & ¹³C – NMR of 2-(2-methylbenzylidene)-4-benzyl-7-(2-methylphenyl)-4,10,11-triazatricyclo
[6.2.2.0^{1,6}] dodecane-9,12-dione (4e)**

NBP2Me

```
Current Data Parameters
NAME      NBP2Me
EXPNO     26
PROCNO    1

F2 - Acquisition Parameters
Date_     20130311
Time      21.97
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zg30
TD        65536
SOLVENT   DMSO
NS         16
DS         2
SWH        8223.685 Hz
FIDRES     0.125483 Hz
AQ         3.9845889 sec
RG         143.73
DW         60.800 usec
DE         6.50 usec
TE         293.8 K
D1         1.00000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       1H
P1         14.25 usec
PLW1       14.00000000 W
SFO1       400.2604718 MHz

F2 - Processing parameters
SI         65536
SF         400.2579932 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
```

NBP2M

```
Current Data Parameters
NAME      NBP2M
EXPNO     12
PROCNO    1

F2 - Acquisition Parameters
Date_     20130403
Time      5.23
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zgpg30
TD        65536
SOLVENT   DMSO
NS         512
DS         4
SWH        24038.461 Hz
FIDRES     0.366798 Hz
AQ         1.3631488 sec
RG         175.97
DW         20.800 usec
DE         6.50 usec
TE         295.6 K
D1         2.00000000 sec
d11        0.03000000 sec
DELTA      1.89999998 sec
TD0        1
SFO1       100.6550182 MHz
NUC1       13C
P1         9.80 usec
PLW1       58.00000000 W
SFO2       400.2596010 MHz
NUC2       1H
CPDPRG2   waltz16
PCPD2      90.00 usec
PLW2       14.00000000 W
PLW12      0.35097000 W
PLW13      0.28428999 W

F2 - Processing parameters
SI         32768
SF         100.6449913 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
```

¹H & ¹³C – NMR of 2-(3-methylbenzylidene)-4-benzyl-7-(2-methylphenyl)-4,10,11-triazatricyclo
[6.2.2.0^{1,6}] dodecane-9,12-dione (4f)

NBP3Me

Current Data Parameters
NAME NBP3Me
EXPNO 19
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130206
Time 18.21
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9845889 sec
RG 112.69
DW 60.800 usec
DE 6.50 usec
TE 295.2 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 14.25 usec
PLW1 14.00000000 W
SFO1 400.2604718 MHz

F2 - Processing parameters
SI 65536
SF 400.2580401 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

NBP3M C13

Current Data Parameters
NAME NBP3M C13
EXPNO 23
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130404
Time 5.01
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 512
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 143.73
DW 20.800 usec
DE 6.50 usec
TE 295.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
SFO1 100.6550182 MHz
NUC1 13C
P1 9.80 usec
PLW1 58.00000000 W
SFO2 400.2596010 MHz
NUC2 1H
CPDPRG2 waltz16
PCPD2 90.00 usec
PLW2 14.00000000 W
PLW12 0.35097000 W
PLW13 0.28428899 W

F2 - Processing parameters
SI 32768
SF 100.6450043 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

¹H & ¹³C – NMR of 2-(4-methylbenzylidene)-4-benzyl-7-(2-methylphenyl)-4,10,11-triazatricyclo
[6.2.2.0^{1,6}] dodecane-9,12-dione (4g)

NBP4Me

BRUKER

Current Data Parameters
NAME NBP4Me
EXPNO 13
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130206
Time 16.15
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9845889 sec
RG 156.91
DW 60.800 usec
DE 6.50 usec
TE 295.4 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 14.25 usec
PLW1 14.00000000 W
SF01 400.2604718 MHz

F2 - Processing parameters
SI 65536
SF 400.2579895 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

NB4M C13

BRUKER

Current Data Parameters
NAME NB4M C13
EXPNO 14
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130403
Time 6.24
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 512
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 156.91
DW 20.800 usec
DE 6.50 usec
TE 295.5 K
D1 2.00000000 sec
S11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
SF01 100.6550182 MHz
NUC1 13C
P1 9.80 usec
PLW1 58.00000000 W
SF02 400.2596019 MHz
NUC2 1H
CPDPRG2 waltz16
PCPD2 90.00 usec
PLW2 14.00000000 W
PLW12 0.35097000 W
PLW13 0.28428999 W

F2 - Processing parameters
SI 32768
SF 100.6444930 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

¹H & ¹³C – NMR of 2-(2-chlorobenzylidene)-4-benzyl-7-(2-chlorophenyl)-4,10,11-triazatricyclo
[6.2.2.0^{1,6}] dodecane-9,12-dione (4h)

NBP2C1

Current Data Parameters
NAME NBP2C1
EXPNO 39
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130315
Time 17.16
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9845889 sec
RG 71.13
DW 60.800 usec
DE 6.50 usec
TE 296.2 K
D1 1.00000000 sec
TD0 1

----- CHANNEL f1 -----
NUC1 1H
P1 14.25 usec
PLW1 14.00000000 W
SFO1 400.2604719 MHz

F2 - Processing parameters
SI 65536
SF 400.2579917 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

NBP2C1 C13

Current Data Parameters
NAME NBP2C1 C13
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130206
Time 5.21
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 1024
DS 4
SWH 24038.461 Hz
FIDRES 0.166798 Hz
AQ 1.3431488 sec
RG 143.73
DW 20.800 usec
DE 6.50 usec
TE 295.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

----- CHANNEL f1 -----
NUC1 13C
P1 9.80 usec
PLW1 58.00000000 W
SFO1 100.6550182 MHz

----- CHANNEL f2 -----
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 14.00000000 W
PLW12 0.3597000 W
PLW13 0.28428999 W
SFO2 400.2596010 MHz

F2 - Processing parameters
SI 32768
SF 100.6450063 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

¹H & ¹³C – NMR of 2-(4-chlorobenzylidene)-4-benzyl-7-(2-chlorophenyl)-4,10,11-triazatricyclo
[6.2.2.0^{1,6}] dodecane-9,12-dione (4i)

NBP4C1

Current Data Parameters
NAME NBP4C1
EXPNO 21
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130309
Time 16.48
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9845889 sec
RG 127.79
DW 60.800 usec
DE 6.50 usec
TE 292.7 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 14.25 usec
PLW1 14.00000000 W
SFO1 400.2604718 MHz

F2 - Processing parameters
SI 65536
SF 400.2580000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

NBP4C C13

Current Data Parameters
NAME NBP4C C13
EXPNO 28
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130403
Time 10.22
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 512
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 156.91
DW 20.800 usec
DE 6.50 usec
TE 297.7 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
SFO1 100.6550182 MHz
NUC1 13C
P1 9.80 usec
PLW1 58.00000000 W
SFO2 400.2596010 MHz
NUC2 1H
CPDPRG2 waltz16
PCPD2 90.00 usec
PLW2 14.00000000 W
PLW12 0.35097000 W
PLW13 0.28428999 W

F2 - Processing parameters
SI 32768
SF 100.6450043 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

¹H & ¹³C – NMR of 2-((naphthalen-1-yl)methylene)-4-benzyl-7-(1-naphthyl)-4,10,11-triazatricyclo [6.2.2.0^{1,6}] dodecane-9,12-dione (4j)