

SUPPLEMENTARY MATERIAL

Spermine amides of selected triterpenoid acids: Dynamic supramolecular systems formation influences cytotoxicity of the drugs

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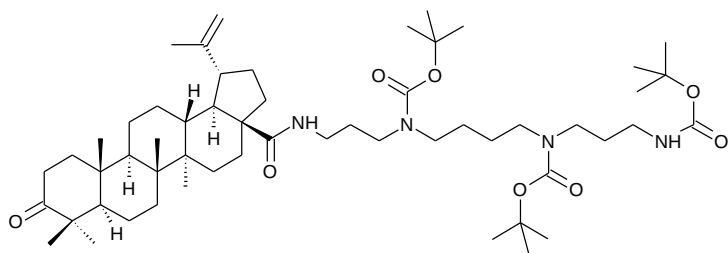
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^h Aalto University, School of Science, Department of Applied Physics, P.O. Box 15100, FI-00076 Espoo, Finland

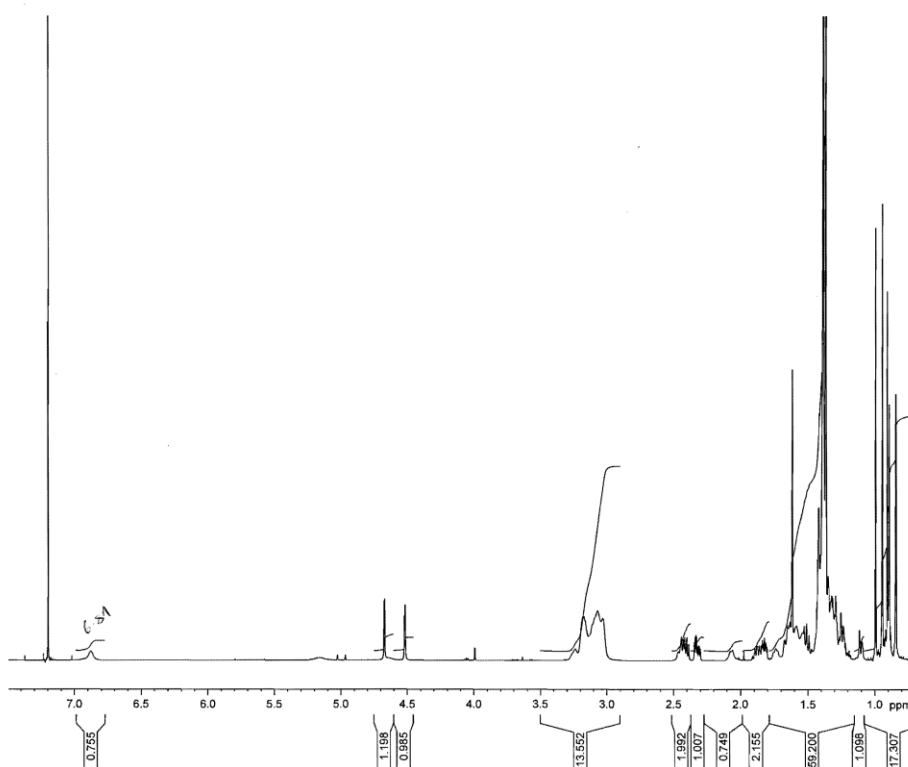
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Analytical data of the prepared compounds

Analytical data of 2a



2a: $^1\text{H-NMR}$ (600.13 MHz, CDCl_3): δ [ppm] 0.85 (s, 3H, 25- CH_3), 0.90 (s, 3H, 26- CH_3), 0.91 (s, 3H, 27- CH_3), 0.95 (s, 3H, 24- CH_3), 1.00 (s, 3H, 23- CH_3), 1.37 (s, Boc), 1.39 (s, Boc), 1.39 (s, Boc), 1.62 (dd, 3H, $J=0.7$; 1.4 Hz, 29- CH_3), 1.83 (ddd, 2H, $J=4.5$; 7.7; 13.3 Hz, 1- CH_2), 1.88 (ddt, 2H, $J=7.4$; 10.7; 12.5; 12.5 Hz, 21- CH_2), 2.32 (ddd, 1H, $J=4.5$; 7.6; 15.7 Hz, 2- CH_2), 2.41 (ddd, 1H, $J=7.7$; 9.8; 12.5 Hz, 2- CH_2), 2.46 (dt, 1H, $J=3.3$; 12.5; 12.5 Hz, 13-CH), 3.03 (bq, 2H, $J=6.3$ Hz, 1'- CH_2), 3.06-3.21 (m, 10H, 3', 4', 7', 8', 10'- CH_2), 4.52 (dq, 1H, $J=1.4$; 1.4; 1.4; 2.4 Hz, 30- CH_2), 4.67 (dq, 1H, $J=0.7$; 0.7; 0.7; 2.4 Hz, 30- CH_2); $^{13}\text{C-NMR}$ (150.92 MHz, CDCl_3): δ [ppm] 14.58 (q, 27-C), 15.94 (q, 25-C), 16.01 (q, 26-C), 19.54 (q, 29-C), 19.71 (t, 11-C), 21.03 (q, 24-C), 21.56 (t, 6-C), 25.75 (t, 12-C), 26.05 (t, 5'-C), 26.05 (t, 6'-C), 26.68 (q, 23-C), 27.96 (t, 2'-C), 29.52 (t, 15-C), 31.00 (t, 21-C), 33.50 (t, 22-C), 33.79 (t, 16-C), 34.16 (t, 2-C), 34.96 (t, 10'-C), 36.99 (s, 4-C), 36.99 (s, 10-C), 37.74 (t, 1'-C), 37.74 (t, 9'-C), 37.89 (d, 13-C), 38.45 (t, 7-C), 39.68 (t, 1-C), 40.80 (s, 8-C), 42.59 (s, 14-C), 43.37 (t, 7'-C), 44.08 (t, 8'-C), 44.24 (t, 4'-C), 46.55 (t, 3'-C), 47.33 (d, 18-C), 50.10 (d, 19-C), 50.12 (d, 9-C), 55.11 (d, 5-C), 55.79 (s, 17-C), 109.23 (t, 30-C), 151.17 (s, 20-C), 176.18 (s, 28-C), 217.96 (s, 3-C). IR (cm^{-1}): 3383, 2943, 2868, 1697, 1522, 1419, 1366, 1249, 1168, 882, 755. MS (ESI, 20 V): m/z = 937.5 $[\text{M-H}]^+$, 939.6 $[\text{M+H}]^+$. For $\text{C}_{55}\text{H}_{94}\text{N}_4\text{O}_8$ (939.36) calcd. (%) C (70.32), H (10.09), N (5.96), found (%) C (70.30), H (10.08), N (5.98).



Z.Wimmer MV-5
in CDCl₃, rfp=TMS
21.8.2017 DA

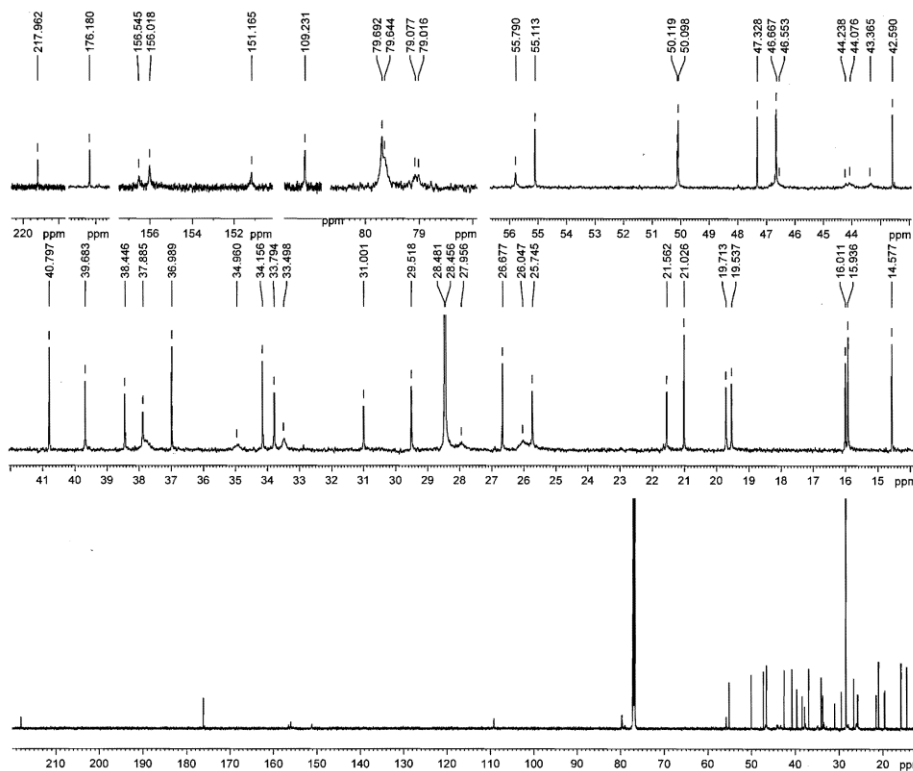
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PROCNO 1

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PULPROG zg30
TD 43298
SOLVENT CDCl₃
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SWH 5411.255 Hz
FIDRES 0.125006 Hz
AQ 3.9995112 sec
RG 35
DW 92.400 usec
DE 20.00 usec
TE 298.2 K
D1 0 sec
TD0 1

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NUC1 1H
P1 9.00 usec
PLW1 6.00000000 W

F2 - Processing parameters
SI 131072
SF 600.1300511 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00

54776



Z.Wimmer MV-5
in CDCl₃, rfp=TMS
21.8.2017 DA

Current Data Parameters
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EXPNO 102
PROCNO 1

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PROBHD 5 mm CPTCI 1H-
PULPROG zgpg30
TD 72459
SOLVENT CDCl₃
NS 1768
DS 0
SWH 36231.883 Hz
FIDRES 0.500026 Hz
AQ 0.9999480 sec
RG 195.16
DW 13.800 usec
DE 18.00 usec
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D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

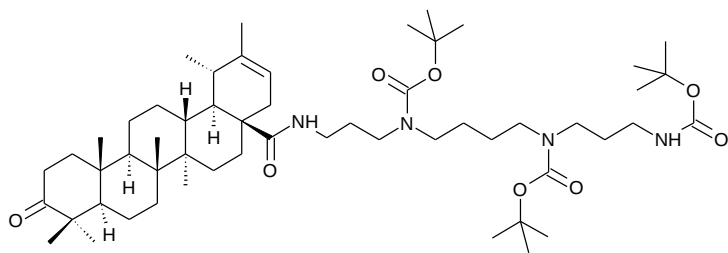
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NUC2 1H
CPDPRG2 waltz16
PCPD2 70.00 usec
PLW2 6.00000000 W
PLW12 0.09919400 W
PLW13 0.04860000 W

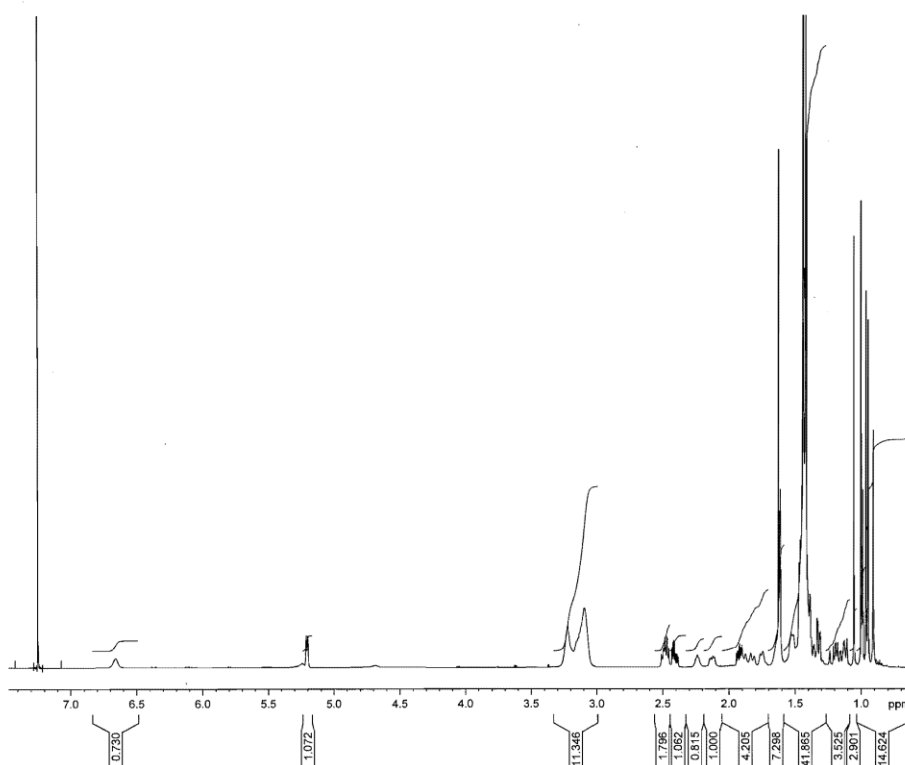
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SF 150.9028052 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0

54776

Analytical data of 2b



2b: $^1\text{H-NMR}$ (600.13 MHz, CDCl_3): δ [ppm] 0.91 (s, 3H, 25- CH_3), 0.95 (s, 3H, 26- CH_3), 0.96 (s, 3H, 27- CH_3), 0.99 (d, 3H, $J=6.6$ Hz, 29- CH_3), 1.00 (s, 3H, 24- CH_3), 1.05 (s, 3H, 23- CH_3), 1.20 (dd, 1H, $J=7.9$; 11.1 Hz, 18-CH), 1.33 (dd, 1H, $J=3.4$; 11.0 Hz, 5-CH), 1.42 (s, Boc), 1.44 (s, Boc), 1.44 (s, Boc), 1.62 (t, 3H, $J=1.9$ Hz, 30- CH_3), 1.93 (ddd, 2H, $J=4.4$; 7.6; 13.2 Hz, 1- CH_2), 2.13 (bdd, 2H, $J=6.4$; 15.4 Hz, 22- CH_2), 2.24 (bdq, 1H, $J=6.6$; 6.6; 6.6; 7.9 Hz, 19-CH), 2.41 (ddd, 1H, $J=4.4$; 7.7; 15.6 Hz, 2- CH_2), 2.45-2.50 (m, 1H, 13-CH), 2.48 (ddd, 1H, $J=7.6$; 9.6; 15.6 Hz, 2- CH_2), 3.04-3.26 (m, 12H, 1', 3', 4', 7', 8', 10'- CH_2), 6.66 (bs, NH); $^{13}\text{C-NMR}$ (150.92 MHz, CDCl_3): δ [ppm] 14.84 (q, 27-C), 15.93 (q, 26-C), 16.18 (q, 25-C), 19.68 (t, 6-C), 21.00 (q, 24-C), 21.80 (q, 30-C), 22.13 (t, 11-C), 23.62 (q, 29-C), 25.89 (t, 6'-C), 26.11 (t, 5'-C), 26.72 (q, 23-C), 27.41 (t, 15-C), 27.81 (t, 2'-C), 28.33 (q, Boc), 28.46 (q, Boc), 28.46 (q, Boc), 28.82 (t, 12-C), 33.57 (t, 16-C), 34.15 (t, 7-C), 34.24 (t, 2-C), 34.96 (t, 10'-C), 36.87 (s, 10-C), 37.10 (d, 19-C), 37.37 (t, 9'-C), 37.72 (t, 1'-C), 39.05 (t, 22-C), 39.10 (d, 13-C), 39.70 (t, 1-C), 40.87 (s, 8-C), 42.05 (s, 14-C), 43.21 (t, 7'-C), 43.76 (t, 8'-C), 44.19 (t, 4'-C), 46.64 (t, 3'-C), 47.33 (s, 4-C), 48.22 (s, 17-C), 49.51 (d, 18-C), 50.08 (d, 9-C), 54.94 (d, 5-C), 79.57 (s, Boc), 79.57 (s, Boc), 79.76 (s, Boc), 116.94 (d, 21-C), 142.84 (s, 20-C), 155.92 (s, Boc), 156.10 (s, Boc), 156.46 (s, Boc), 175.70 (s, 28-C), 218.32 (s, 3-C). IR (cm^{-1}): 3386, 2937, 2868, 1697, 1520, 1419, 1366, 1250, 1169. MS (ESI, 15 V): m/z = 937.1 $[\text{M-H}]^+$, 939.0 $[\text{M+H}]^+$, 960.6 $[\text{M+Na}]^+$, 973.5 $[\text{M+Cl}]^-$. For $\text{C}_{55}\text{H}_{94}\text{N}_4\text{O}_8$ (939.36) calcd. (%) C (70.32), H (10.09), N (5.96), found (%) C (70.30), H (10.10), N (5.95).



Z.Wimmer MV-3
in CDCl₃, rf=TMS
2.5.2017 DA

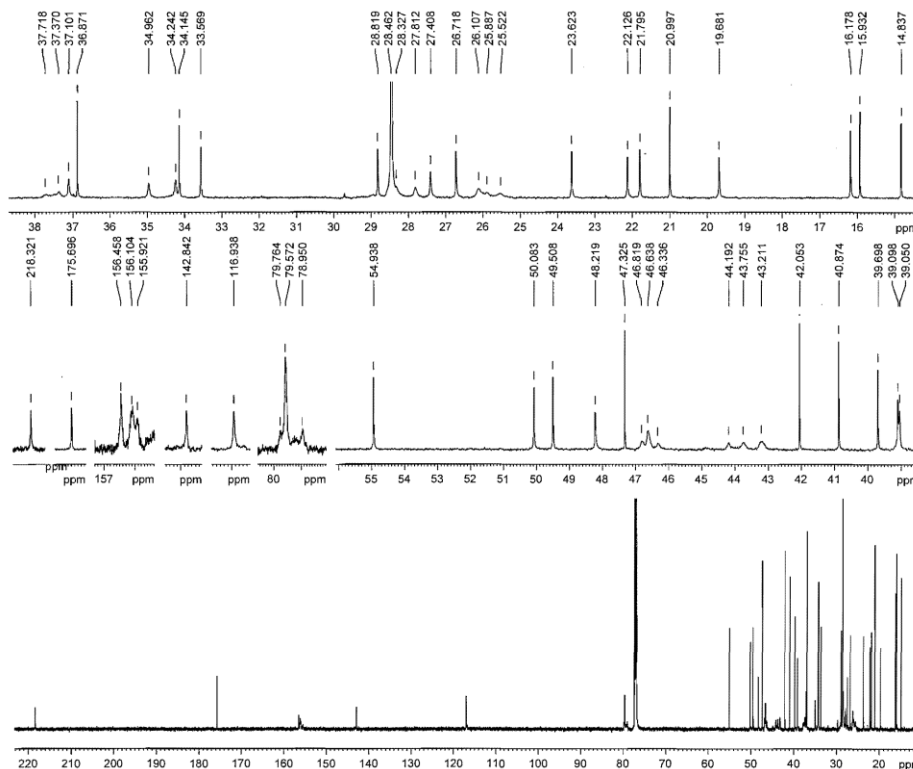
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PROCNO 1

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PULPROG zg30
TD 43288
SOLVENT CDCl₃
NS 32
DS 0
SWH 5411.255 Hz
FIDRES 0.125006 Hz
AQ 3.9998112 sec
RG 40.16
DW 92.400 usec
DE 20.00 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1

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NUC1 1H
P1 9.00 usec
PLW1 6.00000000 W

F2 - Processing parameters
SI 131072
SF 600.1300212 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00

55009



Z.Wimmer MV-3
in CDCl₃, rf=TMS
2.5.2017 DA

Current Data Parameters
NAME Wimmer-MV_3
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170502
Time 12.30
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zgpg30
TD 72460
SOLVENT CDCl₃
NS 4894
DS 0
SWH 36231.883 Hz
FIDRES 0.500026 Hz
AQ 0.9999480 sec
RG 195.18
DW 13.800 usec
DE 18.00 usec
TE 298.1 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

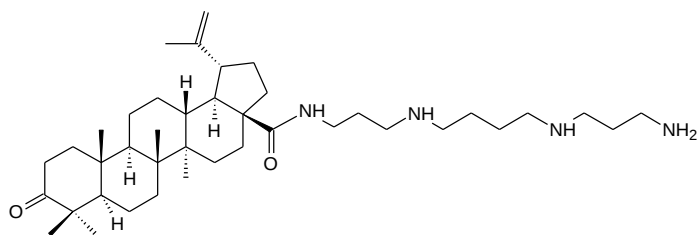
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PLW1 80.00000000 W

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PCPD2 70.00 usec
PLW2 6.00000000 W
PLW12 0.09918400 W
PLW13 0.04860000 W

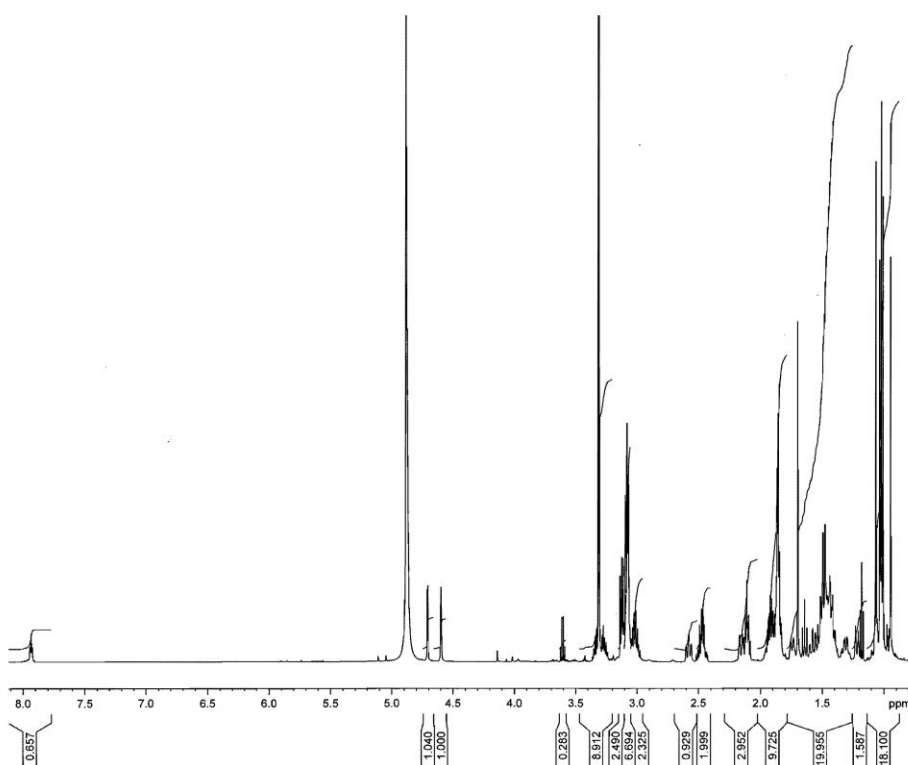
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SF 150.9028085 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0

55009

Analytical data of 3a



3a: ¹H-NMR (600.13 MHz, CD₃OD): δ [ppm] 0.94 (s, 3H, 25-CH₃), 1.00 (s, 3H, 26-CH₃), 1.02 (s, 3H, 24-CH₃), 1.03 (s, 3H, 27-CH₃), 1.06 (s, 3H, 23-CH₃), 1.18 (dt, 2H, *J*=3.2; 3.2; 13.5 Hz, 21-CH₂), 1.58 (dt, 1H, *J*=3.4; 13.6; 13.6 Hz, 16-CH₂), 1.64 (t, 1H, *J*=11.3 Hz, 9-CH), 1.70 (dd, 3H, *J*=0.7; 1.3 Hz, 29-CH₃), 2.15 (ddt, 1H, *J*=1.1; 3.4; 3.4; 13.7 Hz, 16-CH₂), 2.45 (ddd, 1H, *J*=4.9; 8.1; 15.7 Hz, 2-CH₂), 2.49 (ddd, 1H, *J*=7.6; 9.2; 15.7 Hz, 2-CH₂), 2.58 (ddd, 1H, *J*=3.7; 11.6; 13.0 Hz, 13-CH), 2.97-3.04 (m, 2H, 7'-CH₂), 3.06-3.10 (m, 6H, 3', 8', 10'-CH₂), 3.11-3.14 (m, 2H, 4'-CH₂), 3.23-3.29 (t, 2H, 1'-CH₂), 3.32-3.37 (m, 2H, 1'-CH₂), 4.58 (dq, 1H, *J*=1.3; 1.3; 1.3; 2.4 Hz, 30-CH₂), 4.71 (dq, 1H, *J*=0.7; 0.7; 0.7; 2.4 Hz, 30-CH₂), 7.94 (t, 1H, *J*=6.1 Hz, 1'-NH); ¹³C-NMR (150.92 MHz, CD₃OD): δ [ppm] 14.97 (q, 27-C), 16.61 (q, 25-C), 16.65 (q, 26-C), 19.54 (q, 29-C), 20.77 (t, 6-C), 21.41 (q, 24-C), 22.73 (t, 11-C), 24.49 (t, 6'-C), 24.57 (t, 5'-C), 25.61 (t, 2'-C), 26.96 (t, 12-C), 27.21 (q, 23-C), 27.86 (t, 9'-C), 30.62 (t, 21-C), 31.98 (t, 15-C), 33.95 (t, 16-C), 34.77 (t, 22-C), 35.01 (t, 2-C), 36.77 (t, 7-C), 36.89 (t, 1'-C), 38.06 (s, 4-C), 38.06 (t, 10'-C), 39.16 (s, 10-C), 39.41 (d, 13-C), 40.71 (t, 1-C), 41.92 (s, 8-C), 43.61 (s, 14-C), 46.10 (t, 7'-C), 46.58 (t, 4'-C), 48.17 (t, 8'-C), 48.20 (t, 3'-C), 48.37 (d, 18-C), 51.22 (d, 9-C), 51.27 (d, 19-C), 56.00 (d, 5-C), 57.05 (s, 17-C), 110.14 (t, 30-C), 152.19 (s, 20-C), 180.34 (s, 28-C), 220.91 (s, 3-C). IR (cm⁻¹): 3391, 2929, 2866, 1701, 1635, 1541, 1458, 1382, 1245, 1114, 879. MS (ESI, 30 V): *m/z* = 639.3 [M+H]⁺. For C₄₀H₇₀N₄O₂ (639.01) calcd. (%) C (75.18), H (11.04), N (8.77), found C (75.15), H (11.03), N (8.75).



Z.Wimmer MV-6
in CD3OD, rfp=3.31 ppm
2.6.2017 DA

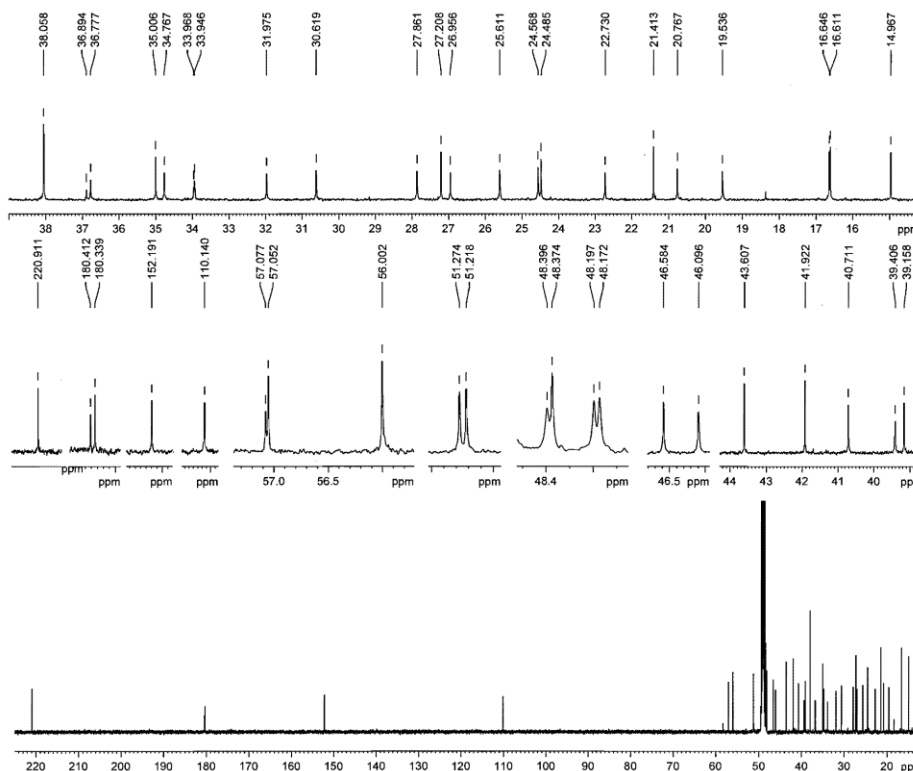
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PROCNO 1

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PULPROG zg30
TD 43288
SOLVENT MeOD
NS 16
DS 0
SWH 5411.255 Hz
FIDRES 0.125006 Hz
AQ 3.9998112 sec
RG 50.38
DW 92.400 usec
DE 20.00 usec
TE 298.1 K
D1 0 sec
TD0 1

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NUC1 1H
P1 9.00 usec
PLW1 6.00000000 W

F2 - Processing parameters
SI 131072
SF 600.1300115 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00

54488



Z.Wimmer MV-6
in CD3OD, rfp=3.31 ppm
2.6.2017 DA

Current Data Parameters
NAME Wimmer-MV_6
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170602
Time 8.09
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zgpg30
TD 72460
SOLVENT MeOD
NS 782
DS 0
SWH 36231.883 Hz
FIDRES 0.500026 Hz
AQ 0.9999480 sec
RG 196.18
DW 13.800 usec
DE 18.00 usec
TE 298.1 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

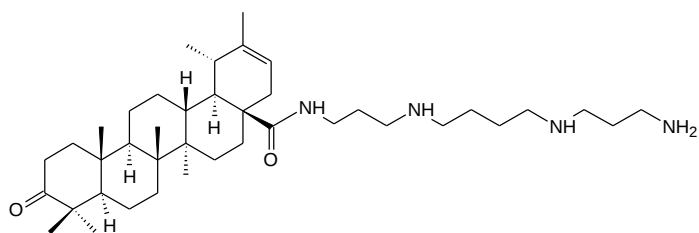
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PLW1 80.00000000 W

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CPDPRG2 waltz16
PCPD2 70.00 usec
PLW2 6.00000000 W
PLW12 0.09918400 W
PLW13 0.04860000 W

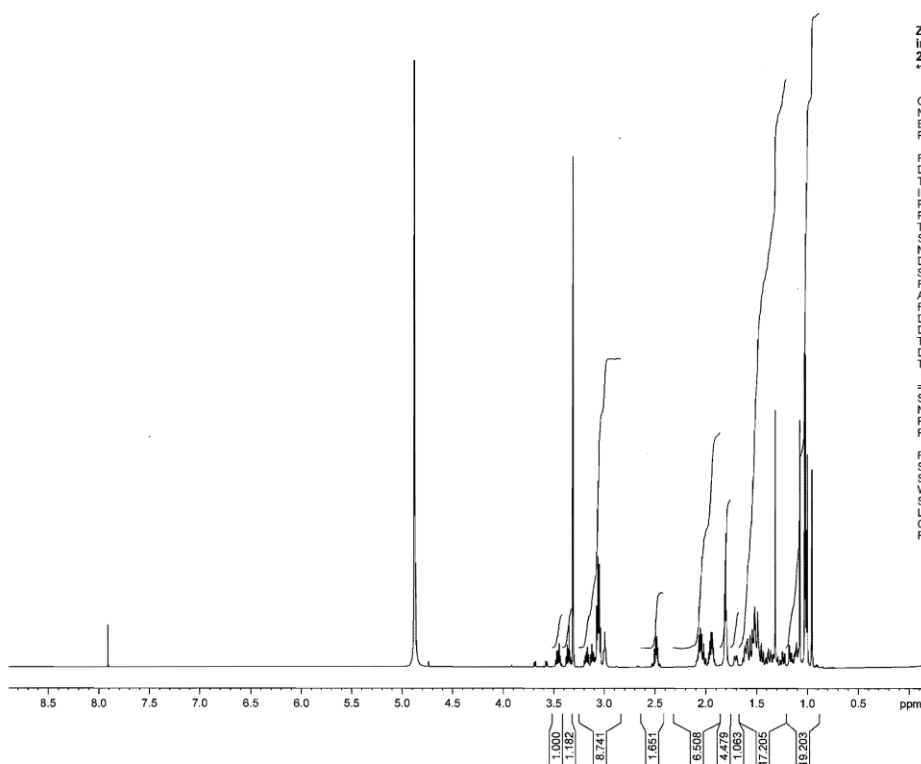
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WDW EM
SSB 0
LB 1.00 Hz
GB 0

54488

Analytical data of 3b



3b: ¹H-NMR (600.13 MHz, CD₃OD): δ [ppm] 0.91 (s, 3H, 25-CH₃), 0.95 (s, 3H, 26-CH₃), 0.96 (s, 3H, 27-CH₃), 0.99 (d, 3H, *J*=6.6 Hz, 29-CH₃), 1.00 (s, 3H, 24-CH₃), 1.05 (s, 3H, 23-CH₃), 1.20 (dd, 1H, *J*=7.9; 11.0 Hz, 18-CH), 1.62 (t, 3H, *J*=1.6 Hz, 30-CH₃), 2.13 (bdd, 2H, *J*=6.4; 15.4 Hz, 22-CH₂), 2.24 (bdq, 1H, *J*=3 x 6.6; 7.9 Hz, 19-CH), 2.41 (ddd, 1H, *J*=4.4; 7.7; 15.6 Hz, 2-CH₂), 2.45-2.58 (m, 13-CH), 2.48 (ddd, 1H, *J*=7.6; 9.6; 15.6 Hz, 2-CH₂), 2.97-3.04 (m, 2H, 7'-CH₂), 3.06-3.10 (m, 6H, 3', 8', 10'-CH₂), 3.11-3.14 (m, 2H, 4'-CH₂), 3.23-3.29 (t, 2H, 1'-CH₂), 3.32-3.37 (m, 2H, 1'-CH₂), 7.94 (t, 1H, *J*=6.1 Hz, 1'-NH); ¹³C-NMR (150.92 MHz, CD₃OD): δ [ppm] 14.84 (q, 27-C), 15.93 (q, 26-C), 16.18 (q, 25-C), 19.68 (t, 6-C), 21.00 (q, 24-C), 21.80 (q, 30-C), 22.13 (t, 11-C), 23.62 (q, 29-C), 24.49 (t, 6'-C), 24.57 (t, 5'-C), 25.61 (t, 2'-C), 26.72 (q, 23-C), 27.41 (t, 15-C), 27.86 (t, 9'-C), 28.82 (t, 12-C), 33.57 (t, 16-C), 34.15 (t, 7-C), 34.24 (t, 2-C), 36.87 (s, 10-C), 36.89 (t, 1'-C), 37.10 (d, 19-C), 38.06 (t, 10'-C), 39.05 (t, 22-C), 39.10 (d, 13-C), 39.70 (t, 1-C), 40.87 (s, 8-C), 42.05 (s, 14-C), 46.10 (t, 7'-C), 46.58 (t, 4'-C), 47.33 (s, 4-C), 48.17 (t, 8'-C), 48.20 (t, 3'-C), 48.22 (s, 17-C), 49.51 (d, 18-C), 50.08 (d, 9-C), 54.94 (d, 5-C), 116.94 (d, 21-C), 142.84 (s, 20-C), 175.70 (s, 28-C), 218.32 (s, 3-C). IR (cm⁻¹): 3390, 2941, 2869, 1679, 1457, 1382, 1230, 1075, 972, 753. MS (ESI, 50 V): *m/z* = 639.2 [M+H]⁺. For C₄₀H₇₀N₄O₂ (639.01) calcd. (%) C (75.18), H (11.04), N (8.77), found (%) C (75.15), H (11.05), N (8.75).



Z.Wimmer MV-4
In CD3OD, rfp=3.31 ppm
28.8.2017 DA

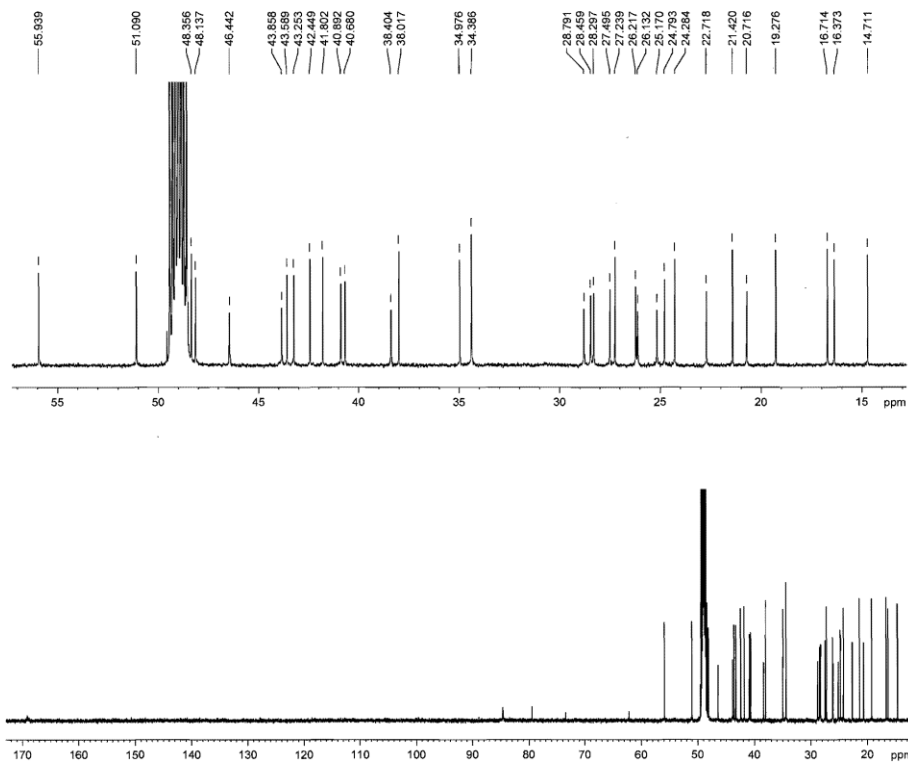
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PROCNO 1

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TD 43288
SOLVENT MeOD
NS 16
DS 0
SWH 5411.255 Hz
FIDRES 0.125006 Hz
AQ 3.9598112 sec
RG 50.38
DW 92.400 usec
DE 20.00 usec
TE 298.2 K
D1 0 sec
TD0 1

===== CHANNEL f1 =====
SFO1 600.1326406 MHz
NUC1 1H
P1 9.00 usec
PLW1 6.00000000 W

F2 - Processing parameters
SI 131072
SF 600.1300113 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00

54823



Z.Wimmer MV-4
In CD3OD, rfp=3.31 ppm
28.8.2017 DA

Current Data Parameters
NAME Wimmer-MV_4
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170828
Time 8.21
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zgpg30
TD 59520
SOLVENT MeOD
NS 2048
DS 0
SWH 29761.904 Hz
FIDRES 0.500032 Hz
AQ 0.9999360 sec
RG 196.16
DW 16.800 usec
DE 18.00 usec
TE 298.2 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

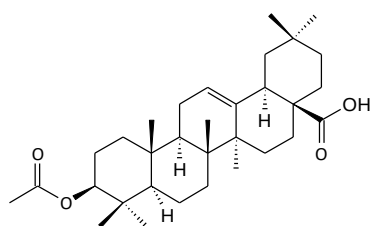
===== CHANNEL f1 =====
SFO1 150.9178993 MHz
NUC1 13C
P1 12.00 usec
PLW1 80.00000000 W

===== CHANNEL f2 =====
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG2 waltz16
PCPD2 70.00 usec
PLW2 6.00000000 W
PLW12 0.09918400 W
PLW13 0.04860000 W

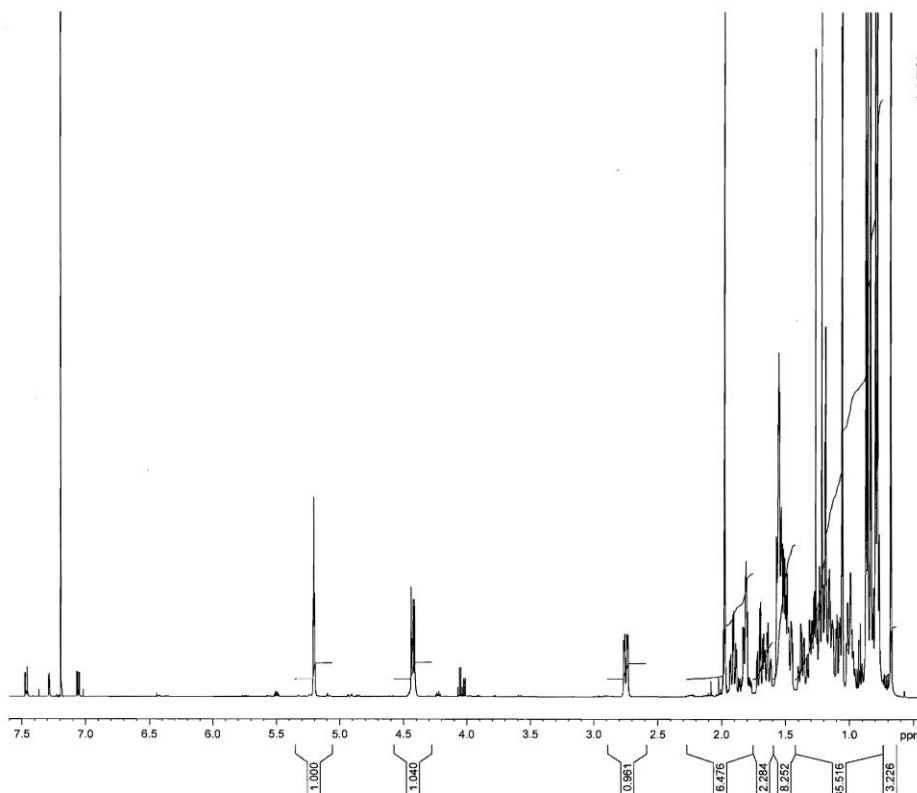
F2 - Processing parameters
SI 131072
SF 150.9025979 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0

54823

Analytical data of 5a



5a: $^1\text{H-NMR}$ (600.13 MHz, CDCl_3): δ [ppm] 0.68 (s, 3H, 26- CH_3), 0.78 (dd, 1H, $J=1.8$; 11.3 Hz, 5-CH), 0.78 (s, 3H, 24- CH_3), 0.79 (s, 3H, 23- CH_3), 0.83 (s, 3H, 29- CH_3), 0.86 (s, 3H, 30- CH_3), 0.87 (d, 3H, $J=0.5$ Hz, 25- CH_3), 1.00 (ddd, 1H, $J=2.8$; 4.0; 13.9 Hz, 15- CH_2), 1.06 (d, 3H, $J=0.6$ Hz, 27- CH_3), 1.09 (ddd, 2H, $J=2.3$; 4.7; 13.6 Hz, 19- CH_2), 1.14 (dt, 1H, $J=2.8$; 2.8; 13.6 Hz, 21- CH_2), 1.22 (dt, 2H, $J=3.2$; 3.2; 12.0 Hz, 7- CH_2), 1.27 (dt, 1H, $J=4.1$; 13.9; 13.9 Hz, 21- CH_2), 1.50 (ddd, 1H, $J=2.9$; 3.2; 13.6 Hz, 22- CH_2), 1.64 (dt, 1H, $J=4.3$; 14.1; 14.1 Hz, 15- CH_2), 1.70 (dt, 1H, $J=4.2$; 13.6; 13.6 Hz, 22- CH_2), 1.79 (ddd, 1H, $J=3.6$; 7.4; 18.5 Hz, 2- CH_2), 1.84 (ddd, 1H, $J=3.5$; 10.8; 18.5 Hz, 2- CH_2), 1.91 (dt, 2H, $J=4.2$; 13.6; 13.6 Hz, 16- CH_2), 1.98 (s, 3H, CH_3CO), 2.75 (dd, 1H, $J=4.5$; 14.3 Hz, 18-CH), 4.42 (dd, 1H, $J=5.6$; 10.6 Hz, 3-CH); $^{13}\text{C-NMR}$ (150.92 MHz, CDCl_3): δ [ppm] 15.37 (q, 25-C), 16.64 (q, 24-C), 17.14 (q, 26-C), 18.15 (t, 6-C), 21.30 (q, CH_3CO), 22.85 (t, 16-C), 23.37 (t, 11-C), 23.50 (t, 2-C), 23.56 (q, 30-C), 25.89 (q, 27-C), 27.64 (t, 15-C), 28.02 (q, 23-C), 30.65 (s, 20-C), 32.41 (t, 22-C), 32.49 (t, 7-C), 33.04 (q, 29-C), 33.76 (t, 21-C), 36.96 (s, 4-C), 37.67 (s, 10-C), 38.03 (t, 1-C), 39.25 (s, 8-C), 40.89 (d, 18-C), 41.52 (s, 14-C), 45.81 (t, 19-C), 46.52 (s, 17-C), 47.52 (d, 9-C), 55.26 (d, 5-C), 80.90 (d, 3-C), 122.54 (d, 12-C), 143.58 (s, 13-C), 171.04 (s, CH_3CO), 183.90 (s, 28-C). IR (cm^{-1}): 3219, 2926, 2855, 1728, 1369, 1252, 1180. MS (ESI, 20 V): m/z = 497.35 $[\text{M-H}]^+$, 521.3 $[\text{M}+\text{Na}]^+$. For $\text{C}_{32}\text{H}_{50}\text{O}_4$ (498.74) calcd. (%) C (77.06), H (10.10), found (%) C (77.08), H (10.08).



Z.Wimmer MV-7
in CDCl₃, rfp=TMS
14.8.2017 DA

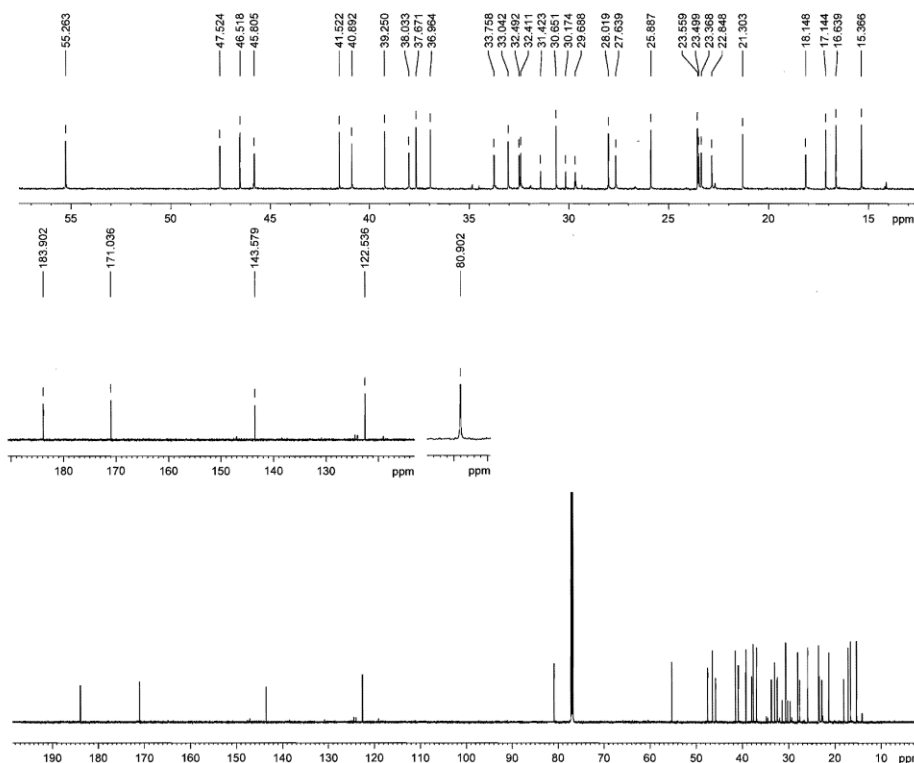
Current Data Parameters
NAME Wimmer-MV_7
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170814
Time 7.49
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 43288
SOLVENT CDCl₃
NS 16
DS 0
SWH 5411.255 Hz
FIDRES 0.125006 Hz
AQ 3.9998112 sec
RG 35
DW 92.400 usec
DE 20.00 usec
TE 298.1 K
D1 0 sec
TD0 1

===== CHANNEL f1 =====
SFO1 600.1325806 MHz
NUC1 1H
P1 9.00 usec
PLW1 6.00000000 W

F2 - Processing parameters
SI 131072
SF 600.1300537 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00

54734



Z.Wimmer MV-7
in CDCl₃, rfp=TMS
14.8.2017 DA

Current Data Parameters
NAME Wimmer-MV_7
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170814
Time 8.04
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zgpg30
TD 59520
SOLVENT CDCl₃
NS 458
DS 0
SWH 29761.904 Hz
FIDRES 0.500032 Hz
AQ 0.9999360 sec
RG 196.18
DW 16.800 usec
DE 18.00 usec
TE 298.2 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

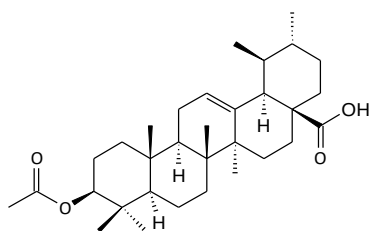
===== CHANNEL f1 =====
SFO1 150.9178993 MHz
NUC1 13C
P1 12.00 usec
PLW1 80.00000000 W

===== CHANNEL f2 =====
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG2 waltz16
PCPD2 70.00 usec
PLW2 6.00000000 W
PLW12 0.09918400 W
PLW13 0.04860000 W

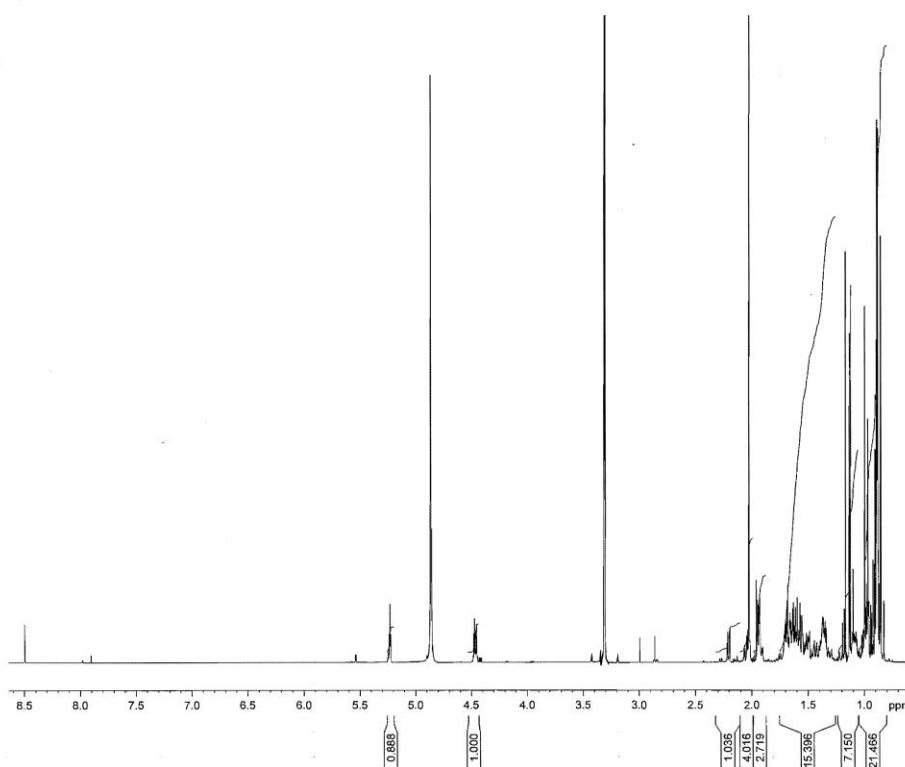
F2 - Processing parameters
SI 131072
SF 150.9028124 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0

54734

Analytical data of 5b



5b: $^1\text{H-NMR}$ (600.13 MHz, CD_3OD): δ [ppm] 0.86 (s, 3H, 26- CH_3), 0.89 (s, 3H, 23- CH_3), 0.89 (s, 3H, 29- CH_3), 0.90 (s, 3H, 25- CH_3), 0.97 (s, 3H, 30- CH_3), 1.00 (d, 3H, $J=0.8$ Hz, 24- CH_3), 1.14 (d, 3H, $J=0.8$ Hz, 27- CH_3), 2.03 (s, 3H, CH_3CO), 2.04 (dt, 2H, $J=4.5$; 13.4; 13.4 Hz, 16- CH_2), 2.21 (dd, 1H, $J=1.8$; 11.3 Hz, 18-CH), 4.46 (dd, 1H, $J=4.9$; 11.4 Hz, 3-CH), 5.23 (t, 1H, $J=3.7$ Hz, 12-CH); $^{13}\text{C-NMR}$ (150.92 MHz, CD_3OD): δ [ppm] 16.03 (q, 24-C), 17.21 (q, 25-C), 17.66 (q, 29-C), 17.79 (q, 26-C), 19.32 (t, 6-C), 21.13 (q, CH_3CO), 21.57 (q, 30-C), 24.10 (q, 27-C), 24.35 (t, 2-C), 24.55 (t, 11-C), 25.32 (t, 16-C), 28.61 (q, 23-C), 29.21 (t, 15-C), 31.78 (t, 21-C), 34.21 (t, 7-C), 38.06 (t, 22-C), 38.12 (s, 10-C), 38.74 (s, 4-C), 39.46 (t, 1-C), 40.43 (d, 19-C), 40.43 (d, 20-C), 40.79 (s, 8-C), 43.26 (s, 14-C), 47.45 (s, 17-C), 48.96 (d, 9-C), 54.36 (d, 18-C), 56.74 (d, 5-C), 82.50 (d, 3-C), 126.75 (d, 12-C), 139.70 (s, 13-C), 172.87 (s, CH_3CO), 181.68 (s, 28-C). IR (cm^{-1}): 2930, 2874, 1735, 1694, 1459, 1371, 1246. MS (ESI, 20 V): $m/z = 497.0$ $[\text{M-H}]^+$. For $\text{C}_{32}\text{H}_{50}\text{O}_4$ (498.74) calcd. (%) C (77.06), H (10.10), found (%) C (77.07), H (10.11).



Z.Wimmer MV-11
in CD3OD, rfp=3.31 ppm
30.10.2017 DA

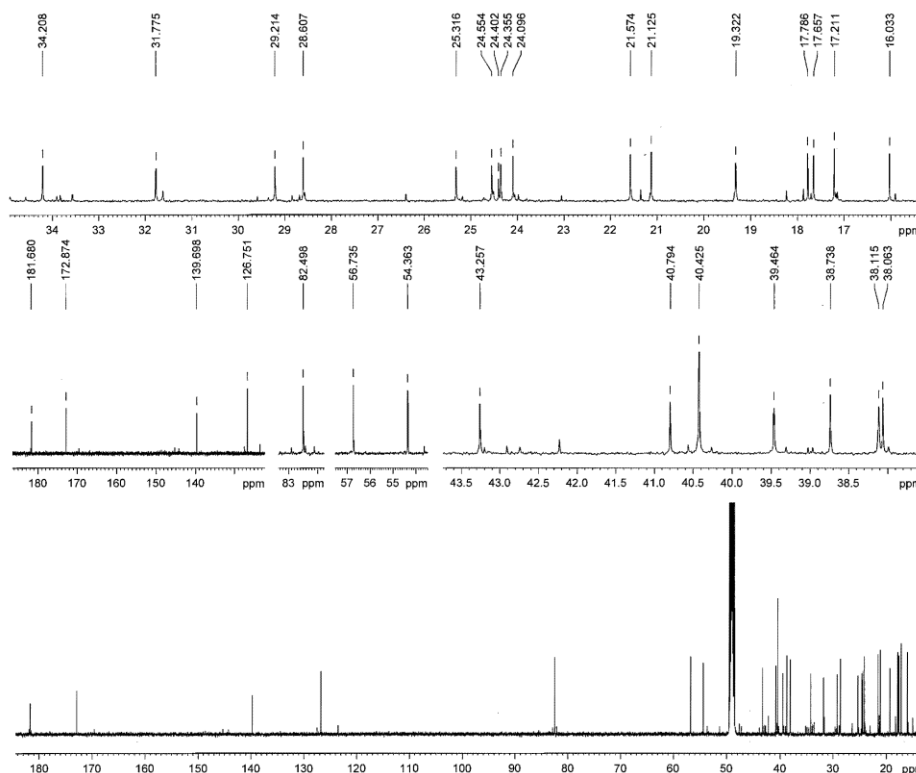
Current Data Parameters
NAME Wimmer-MV_11
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20171030
Time 8.10
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 43288
SOLVENT MeOD
NS 8
DS 0
SWH 5411.255 Hz
FIDRES 0.125006 Hz
AQ 3.9998112 sec
RG 55.4
DW 92.400 usec
DE 20.00 usec
TE 298.1 K
D1 0 sec
TD0 1

===== CHANNEL f1 =====
SFO1 600.1327006 MHz
NUC1 1H
P1 9.00 usec
PLW1 6.00000000 W

F2 - Processing parameters
SI 131072
SF 600.1300114 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00

55051



Z.Wimmer MV-11
in CD3OD, rfp=3.31 ppm
30.10.2017 DA

Current Data Parameters
NAME Wimmer-MV_11
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20171030
Time 9.21
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zgpg30
TD 59520
SOLVENT MeOD
NS 2048
DS 0
SWH 29761.904 Hz
FIDRES 0.500032 Hz
AQ 0.9999360 sec
RG 196.16
DW 16.800 usec
DE 18.00 usec
TE 298.1 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

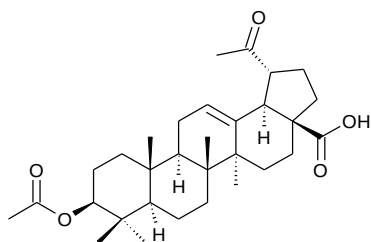
===== CHANNEL f1 =====
SFO1 150.9178993 MHz
NUC1 13C
P1 12.00 usec
PLW1 80.00000000 W

===== CHANNEL f2 =====
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG2 waltz16
PCPD2 70.00 usec
PLW2 6.00000000 W
PLW12 0.09918400 W
PLW13 0.04850000 W

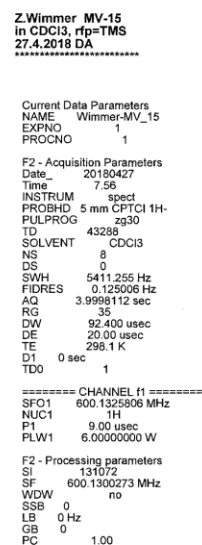
F2 - Processing parameters
SI 131072
SF 150.9025963 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0

55051

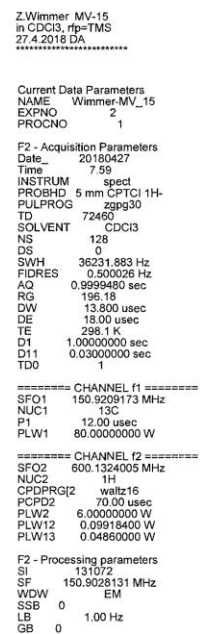
Analytical data of 5c



5c: ¹H-NMR (600.13 MHz, CDCl₃): δ [ppm] 0.77 (dd, 1H, *J*=2.1; 11.5 Hz, 5-CH), 0.80 (s, 3H, 23-CH₃), 0.82 (s, 3H, 25-CH₃), 0.82 (s, 3H, 26-CH₃), 0.89 (s, 3H, 24-CH₃), 0.98 (s, 3H, 27-CH₃), 2.02 (s, 3H, CH₃CO), 2.13 (t, 1H, *J*=11.3 Hz, 18-CH), 2.16 (s, 3H, 29-CH₃), 3.22 (dt, 1H, *J*=5.0; 11.3; 11.3 Hz, 19-CH₂), 4.45 (dd, 1H, *J*=4.8; 11.4 Hz, 3-CH); ¹³C-NMR (150.92 MHz, CDCl₃): δ [ppm] 14.67 (q, 27-C), 15.94 (q, 24-C), 16.14 (q, 26-C), 16.45 (q, 25-C), 18.11 (t, 6-C), 20.83 (t, 11-C), 21.30 (q, CH₃CO), 23.63 (t, 2-C), 27.13 (t, 12-C), 27.92 (q, 23-C), 28.24 (t, 21-C), 29.67 (t, 15-C), 30.08 (q, 29-C), 31.41 (t, 16-C), 34.10 (t, 7-C), 36.68 (t, 22-C), 37.09 (s, 10-C), 37.46 (s, 4-C), 37.77 (d, 13-C), 38.31 (t, 1-C), 40.56 (s, 8-C), 42.20 (s, 14-C), 49.18 (d, 18-C), 50.21 (d, 9-C), 51.17 (d, 19-C), 55.32 (s, 17-C), 56.20 (d, 5-C), 80.84 (d, 3-C), 171.01 (s, CH₃CO), 181.51 (s, 28-C), 212.10 (s, 20-C). IR (cm⁻¹): 3366, 2945, 2874, 1728, 1449, 1366, 1252, 1092, 977. MS (ESI, 20 V): *m/z* = 499.2 [M-H]⁺, 999.4 [2M-H]⁺. For C₃₁H₄₆O₅ (498.69) calcd. (%) C (74.66), H (9.30), found (%) C (74.68), H (9.29).

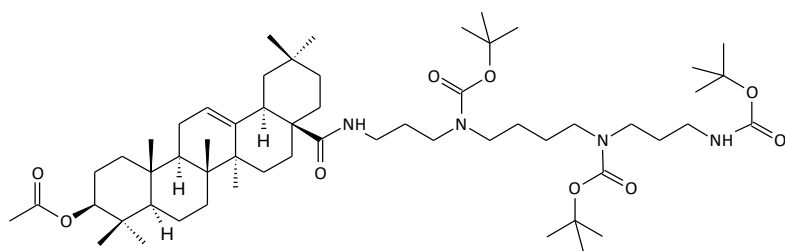


55839

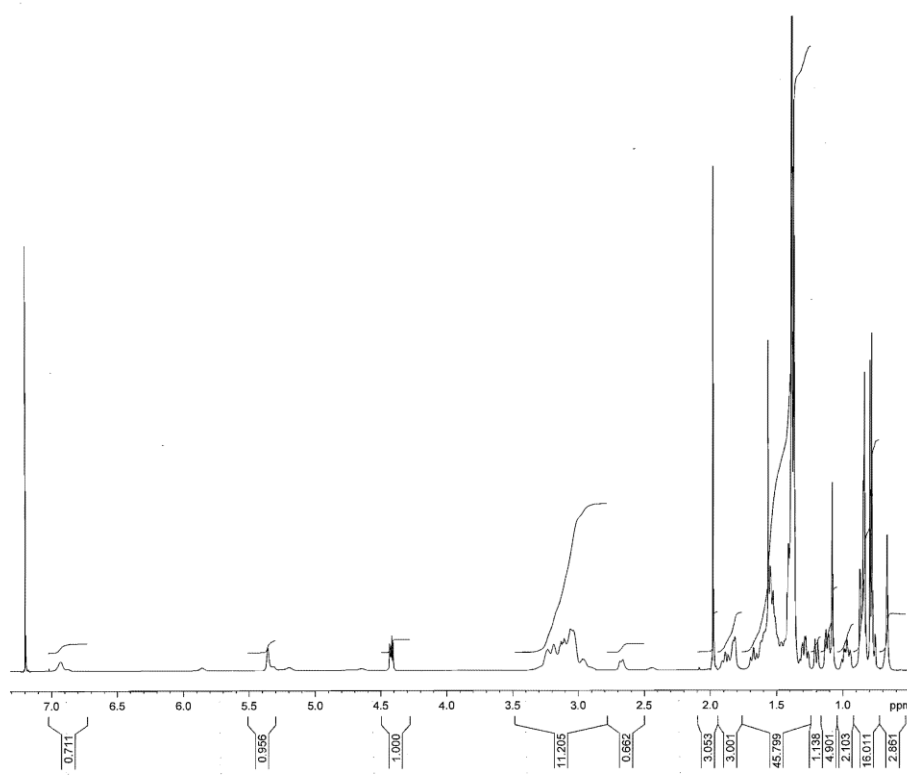


55839

Analytical data of 6a



6a: $^1\text{H-NMR}$ (600.13 MHz, CDCl_3): δ [ppm] 0.67 (s, 3H, 26- CH_3), 0.76 (dd, 1H, $J=1.9$; 11.2 Hz, 5-CH), 0.78 (s, 3H, 24- CH_3), 0.80 (s, 3H, 23- CH_3), 0.84 (s, 3H, 29- CH_3), 0.86 (s, 3H, 30- CH_3), 0.87 (s, 3H, 25- CH_3), 0.97 (dt, 2H, $J=4.8$; 13.5; 13.5 Hz, 1- CH_2), 0.98 (dt, 2H, $J=4.0$; 13.8; 13.8 Hz, 15- CH_2), 1.08 (s, 3H, 27- CH_3), 1.20 (dt, 2H, $J=3.2$; 3.2; 12.7 Hz, 7- CH_2), 1.29 (dt, 2H, $J=4.5$; 13.8; 13.8 Hz, 21- CH_2), 1.37 (s, Boc), 1.39 (s, Boc), 1.40 (s, Boc), 1.89 (dt, 2H, $J=4.0$; 13.6; 13.6 Hz, 16- CH_2), 1.98 (s, 3H, CH_3CO), 2.67 (bd, 1H, $J=11.2$ Hz, 18-CH), 2.92-3.30 (m, 12H, 2', 3', 5', 6', 9', 10', 12'- CH_2), 4.42 (dd, 1H, $J=5.7$; 10.4 Hz, 3-CH); $^{13}\text{C-NMR}$ (150.92 MHz, CDCl_3): δ [ppm] 15.39 (q, 25-C), 16.67 (q, 24-C), 17.01 (q, 26-C), 18.27 (t, 6-C), 21.22 (q, CH_3CO), 23.53 (t, 16-C), 23.58 (t, 11-C), 23.63 (t, 2-C), 23.65 (q, 30-C), 25.82 (q, 27-C), 26.08 (t, 4'-C), 26.08 (t, 8'-C), 27.49 (t, 7'-C), 28.07 (t, 11'-C), 28.08 (t, 15-C), 28.41 (q, 23-C), 28.47 (q, Boc), 28.47 (q, Boc), 28.49 (q, Boc), 30.79 (s, 20-C), 33.05 (t, 22-C), 32.52 (t, 7-C), 33.05 (q, 29-C), 34.30 (t, 21-C), 35.32 (t, 12'-C), 36.99 (s, 4-C), 37.75 (s, 10-C), 37.86 (t, 3'-C), 38.25 (t, 1-C), 39.52 (s, 8-C), 41.79 (d, 18-C), 41.79 (t, 9'-C), 42.01 (s, 14-C), 43.50 (t, 6'-C), 44.16 (t, 5'-C), 46.34 (t, 19-C), 46.78 (t, 10'-C), 46.80 (s, 17-C), 47.62 (d, 9-C), 55.35 (d, 5-C), 79.52 (s, Boc), 79.60 (s, Boc), 79.60 (s, Boc), 80.95 (d, 3-C), 122.56 (d, 12-C), 144.60 (s, 13-C), 156.03 (s, Boc), 156.24 (s, Boc), 156.24 (s, Boc), 170.88 (s, CH_3CO), 177.75 (s, 28-C). IR (cm^{-1}): 3378, 2945, 2876, 1678, 1525, 1476, 1419, 1366, 1247, 1168, 1027, 755. MS (ESI, 20 V): m/z = 983.5 $[\text{M}+\text{H}]^+$, 1005.5 $[\text{M}+\text{Na}]^+$. For $\text{C}_{57}\text{H}_{98}\text{N}_4\text{O}_9$ (983.41) calcd. (%) C (69.62), H (10.04), N (5.70), found (%) C (69.65), H (10.03), N (5.72).



Z.Wimmer MV-8
in CDCl₃, rfp=TMS
18.10.2017 DA

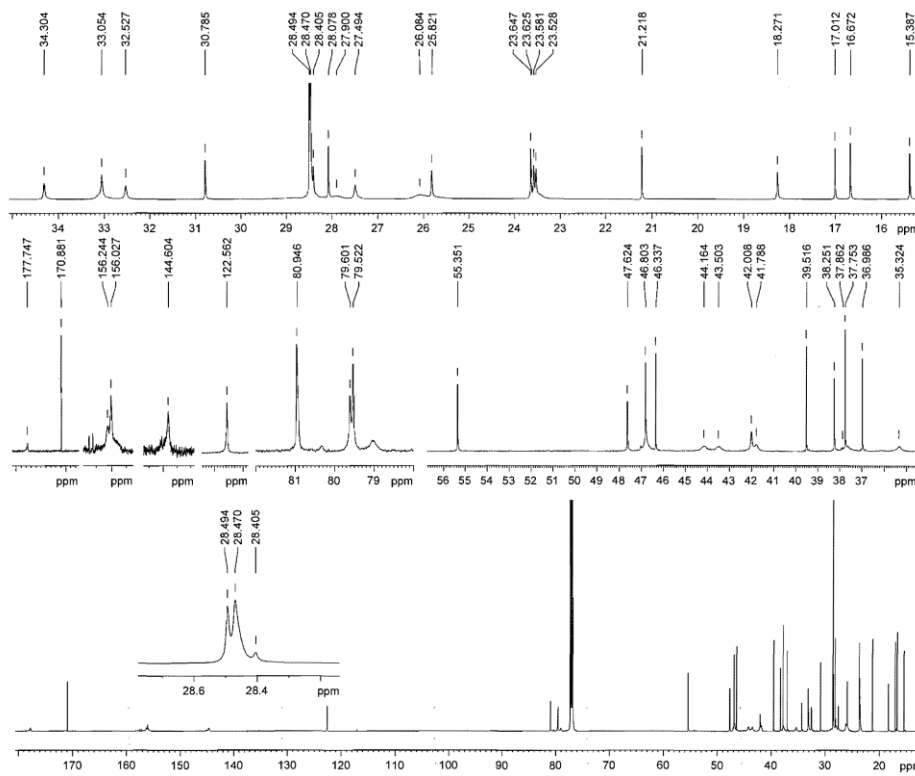
Current Data Parameters
NAME Wimmer-MV_8
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20171018
Time 7.08
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 43288
SOLVENT CDCl₃
NS 16
DS 0
SWH 5411.255 Hz
FIDRES 0.125006 Hz
AQ 3.9998112 sec
RG 35
DW 92.400 usec
DE 20.00 usec
TE 298.1 K
D1 0 sec
TD0 1

===== CHANNEL f1 =====
SFO1 600.1325806 MHz
NUC1 1H
P1 9.00 usec
PLW1 6.00000000 W

F2 - Processing parameters
SI 131072
SF 600.1300537 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00

55025



Z.Wimmer MV-8
in CDCl₃, rfp=TMS
18.10.2017 DA

Current Data Parameters
NAME Wimmer-MV_8
EXPNO 22
PROCNO 1

F2 - Acquisition Parameters
Date_ 20171018
Time 16.53
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zgpg30
TD 58520
SOLVENT CDCl₃
NS 28679
DS 0
SWH 29761.904 Hz
FIDRES 0.500032 Hz
AQ 0.9999360 sec
RG 156.16
DW 16.800 usec
DE 18.00 usec
TE 318.2 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

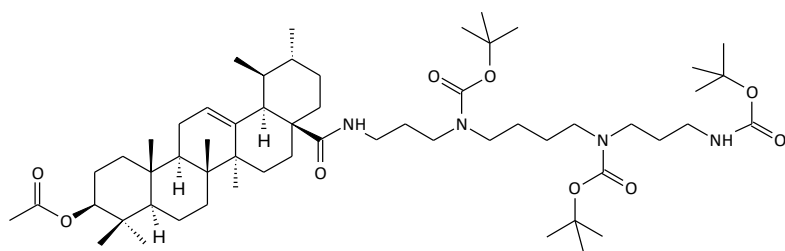
===== CHANNEL f1 =====
SFO1 150.9178993 MHz
NUC1 13C
P1 12.00 usec
PLW1 80.00000000 W

===== CHANNEL f2 =====
SFO2 600.1324005 MHz
NUC2 1H
GPDPRG2 waitz16
PCPD2 70.00 usec
PLW2 6.00000000 W
PLW12 0.99918400 W
PLW13 0.94888000 W

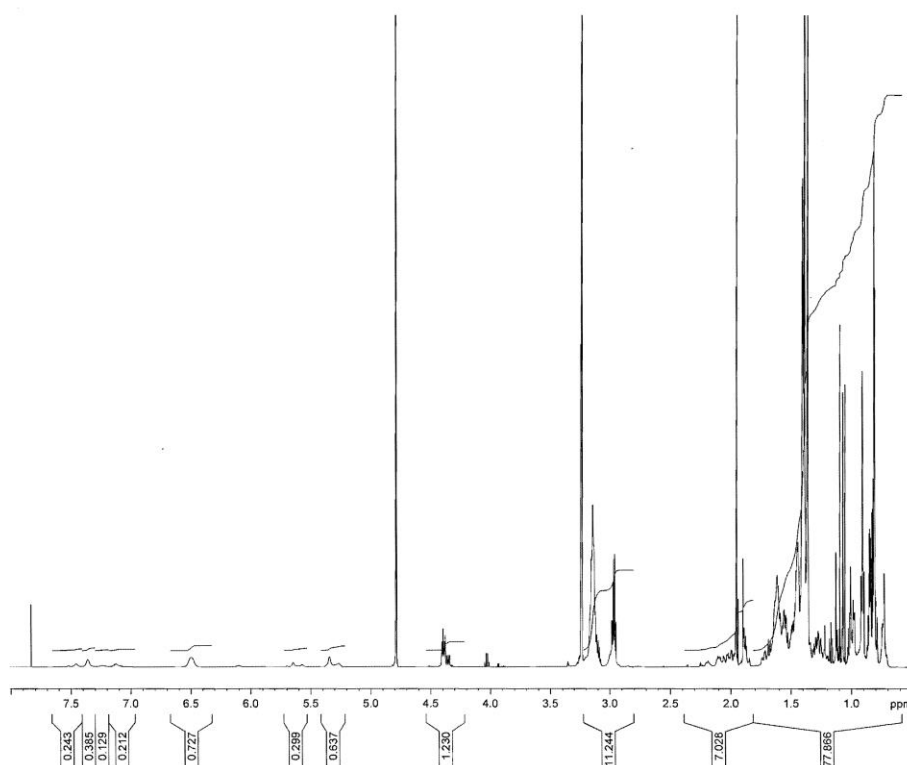
F2 - Processing parameters
SI 131072
SF 150.9028026 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0

55025

Analytical data of 6b



6b: $^1\text{H-NMR}$ (600.13 MHz, CDCl_3): δ [ppm] 0.69 (bd, 1H, $J=11.2$ Hz, 5-CH), 0.70 (s, 3H, 24- CH_3), 0.73 (s, 3H, 26- CH_3), 0.84 (d, 3H, $J=6.4$ Hz, 29- CH_3), 0.88 (s, 3H, 25- CH_3), 0.90 (s, 3H, 23- CH_3), 0.90 (d, 3H, $J=6.5$ Hz, 30- CH_3), 1.06 (s, 3H, 27- CH_3), 1.37 (s, Boc), 1.38 (s, Boc), 1.39 (s, Boc), 1.91 (dt, 2H, $J=3.5$; 13.7; 13.7 Hz, 16- CH_2), 1.98 (s, 3H, CH_3CO), 2.87-3.30 (m, 12H, 1', 3', 4', 7', 8', 10', 12'- CH_2), 4.42 (dd, 1H, $J=5.7$; 10.4 Hz, 3-CH); $^{13}\text{C-NMR}$ (150.92 MHz, CDCl_3): δ [ppm] 16.03 (q, 25-C), 16.34 (q, 24-C), 17.68 (q, 29-C), 18.01 (q, 26-C), 19.44 (t, 6-C), 21.22 (q, CH_3CO), 21.50 (q, 30-C), 24.37 (t, 11-C), 24.40 (t, 16-C), 24.08 (q, 27-C), 25.88 (t, 6'-C), 26.06 (t, 2'-C), 27.63 (t, 2-C), 27.72 (t, 5'-C), 28.47 (q, Boc), 28.47 (q, Boc), 28.49 (q, Boc), 28.66 (t, 9'-C), 28.75 (q, 23-C), 29.00 (t, 15-C), 31.89 (t, 21-C), 34.15 (t, 7-C), 35.29 (t, 10'-C), 37.54 (t, 1'-C), 38.13 (s, 10-C), 38.25 (t, 1-C), 39.20 (t, 22-C), 39.82 (s, 4-C), 40.32 (t, 19-C), 40.80 (s, 20-C), 40.94 (s, 8-C), 43.31 (s, 14-C), 43.37 (t, 7'-C), 43.69 (t, 4'-C), 44.14 (t, 3'-C), 46.64 (s, 17-C), 46.70 (t, 8'-C), 49.02 (d, 9-C), 54.09 (d, 18-C), 56.68 (d, 5-C), 79.52 (s, Boc), 79.60 (s, Boc), 79.60 (s, Boc), 79.65 (d, 3-C), 127.32 (d, 12-C), 139.84 (s, 13-C), 156.03 (s, Boc), 156.24 (s, Boc), 156.24 (s, Boc), 170.89 (s, CH_3CO), 181.68 (s, 28-C). IR (cm^{-1}): 2975, 2873, 1682, 1523, 1456, 1420, 1367, 1248, 1168, 1029. MS (ESI, 20 V): m/z = 983.7 $[\text{M}+\text{H}]^+$, 1005.6 $[\text{M}+\text{Na}]^+$. For $\text{C}_{57}\text{H}_{98}\text{N}_4\text{O}_9$ (983.41) calcd. (%) C (69.62), H (10.04), N (5.70), found (%) C (69.64), H (10.03), N (5.72).



Z.Wimmer MV-12
in CD3OD, $r_f=3.31$ ppm
15.1.2018 DA

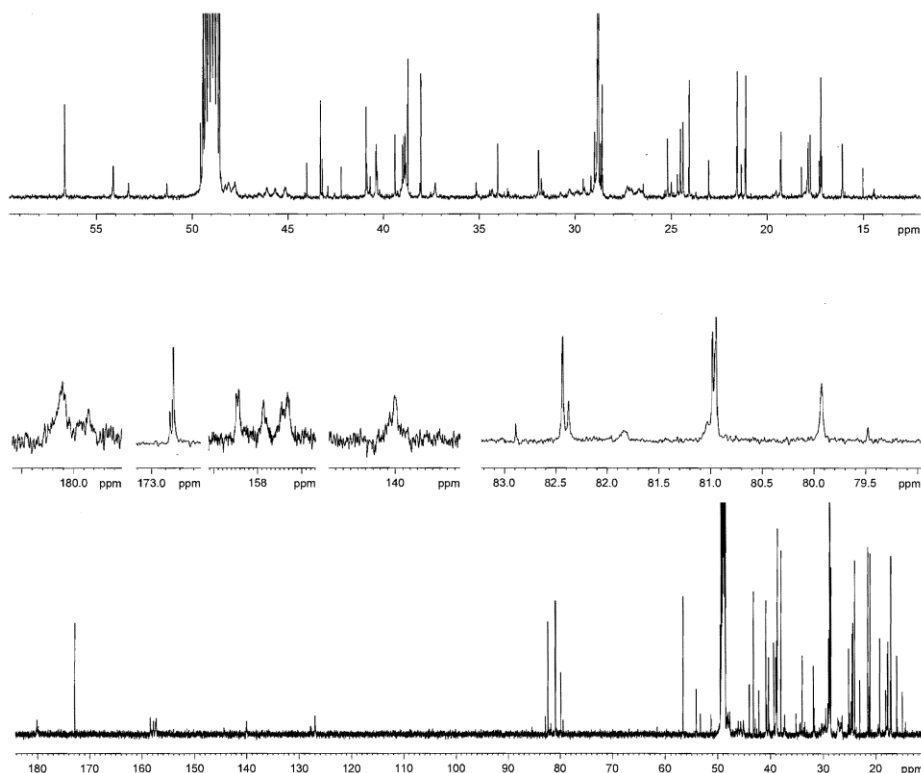
Current Data Parameters
NAME Wimmer-MV_12
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180115
Time 7.13
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 43288
SOLVENT MeOD
NS 16
DS 0
SWH 5411.255 Hz
FIDRES 0.125006 Hz
AQ 3.9988112 sec
RG 61.26
DW 92.400 usec
DE 20.00 usec
TE 298.1 K
D1 0 sec
TD0 1

===== CHANNEL f1 =====
SFO1 600.1325806 MHz
NUC1 1H
P1 9.00 usec
PLW1 6.00000000 W

F2 - Processing parameters
SI 131072
SF 600.1300537 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00

55353



Z.Wimmer MV-12
in CD3OD, $r_f=3.31$ ppm
15.1.2018 DA

Current Data Parameters
NAME Wimmer-MV_12
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180115
Time 7.43
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zgpg30
TD 59520
SOLVENT MeOD
NS 4096
DS 0
SWH 29761.904 Hz
FIDRES 0.500032 Hz
AQ 0.9999360 sec
RG 196.18
DW 16.800 usec
DE 18.00 usec
TE 298.1 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

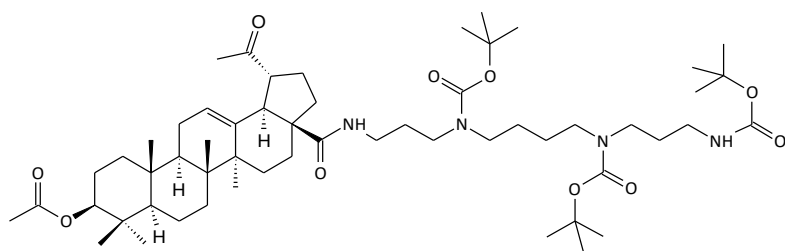
===== CHANNEL f1 =====
SFO1 150.9178993 MHz
NUC1 13C
P1 12.00 usec
PLW1 80.00000000 W

===== CHANNEL f2 =====
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG2 waltz16
PCPD2 70.00 usec
PLW2 6.00000000 W
PLW12 0.09918400 W
PLW13 0.04860000 W

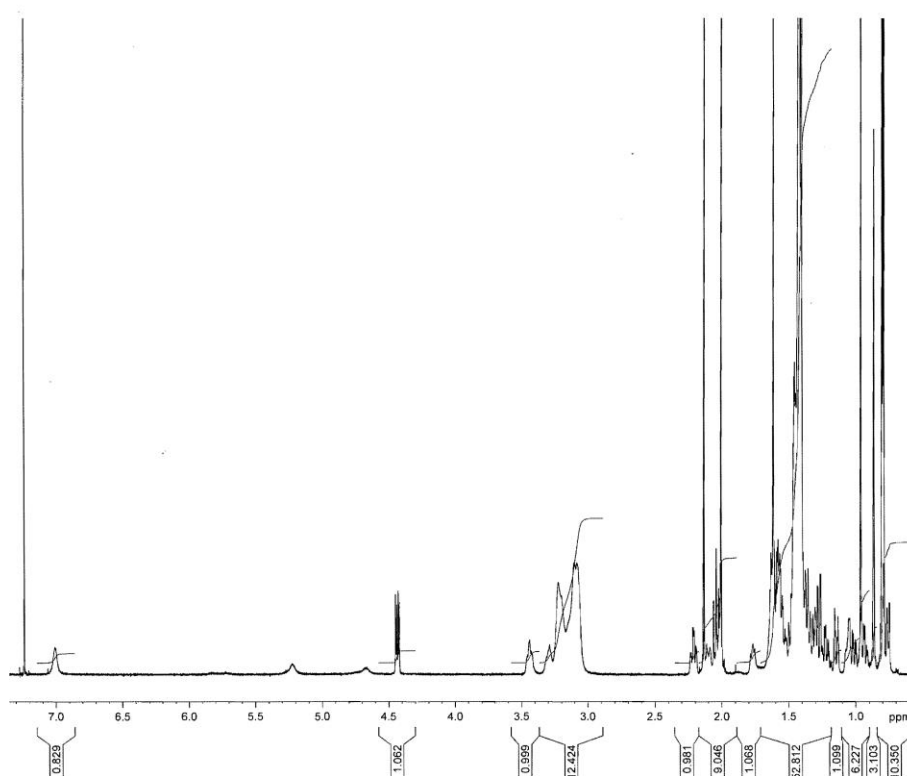
F2 - Processing parameters
SI 131072
SF 150.9025972 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0

55353

Analytical data of 6c



6c: $^1\text{H-NMR}$ (600.13 MHz, CDCl_3): δ [ppm] 0.76 (dd, 1H, $J=2.1$; 11.4 Hz, 5-CH), 0.79 (s, 3H, 25- CH_3), 0.80 (s, 3H, 23- CH_3), 0.81 (s, 3H, 26- CH_3), 0.87 (s, 3H, 24- CH_3), 0.94 (dt, 2H, $J=4.5$; 13.7; 13.7 Hz, 1- CH_2), 0.97 (s, 3H, 27- CH_3), 1.15 (dt, 2H, $J=3.3$; 3.3; 13.6 Hz, 15- CH_2), 1.41 (s, Boc), 1.43 (s, Boc), 1.43 (s, Boc), 2.01 (s, 3H, CH_3CO), 2.04 (t, 1H, $J=11.3$ Hz, 18-CH), 2.14 (s, 3H, 29- CH_3), 2.22 (bdt, H, $J=4.1$; 11.8; 11.8 Hz, 13-CH), 3.04-3.35 (m, 12H, 3', 5', 6', 9', 10', 12'- CH_2), 3.45 (dt, 2H, $J=4.2$; 11.1; 11.1 Hz, 19- CH_2), 4.44 (dd, 1H, $J=5.6$; 11.3 Hz, 3-CH), 7.01 (bs, 1H, 1'-NH); $^{13}\text{C-NMR}$ (150.92 MHz, CDCl_3): δ [ppm] 14.65 (q, 27-C), 16.06 (q, 24-C), 16.16 (q, 26-C), 16.47 (q, 25-C), 18.14 (t, 6-C), 20.94 (t, 11-C), 21.30 (q, CH_3CO), 23.65 (t, 2-C), 25.48 (t, 8'-C), 26.03 (t, 7'-C), 27.21 (t, 12-C), 27.78 (t, 11'-C), 27.92 (t, 21-C), 27.92 (q, 23-C), 28.93 (t, 4'-C), 29.48 (t, 15-C), 30.29 (q, 29-C), 32.59 (t, 16-C), 34.20 (t, 7-C), 34.65 (t, 12'-C), 36.80 (t, 22-C), 37.09 (s, 10-C), 37.68 (s, 4-C), 37.77 (t, 3'-C), 38.01 (d, 13-C), 38.32 (t, 1-C), 40.65 (s, 8-C), 42.21 (s, 14-C), 43.10 (t, 9'-C), 43.74 (t, 10'-C), 44.18 (t, 6'-C), 46.76 (t, 5'-C), 49.95 (d, 18-C), 50.38 (d, 9-C), 51.12 (d, 19-C), 55.36 (d, 5-C), 55.58 (s, 17-C), 80.86 (d, 3-C), 170.94 (s, CH_3CO), 176.00 (s, 28-C), 213.13 (s, 20-C). IR (cm^{-1}): 3365, 2937, 1689, 1417, 1364, 1244, 1025, 979, 771. MS (ESI, 20 V): m/z = 983.5 $[\text{M-H}]^+$, 985.5 $[\text{M+H}]^+$, 1007.5 $[\text{M+Na}]^+$. For $\text{C}_{56}\text{H}_{94}\text{N}_4\text{O}_{10}$ (983.37) calcd. (%) C (68.40), H (9.63), N (5.70), found (%) C (68.37), H (9.64), N (5.71).



Z.Wimmer MV-16
in CDCl₃, rfp=TMS
27.4.2018 DA

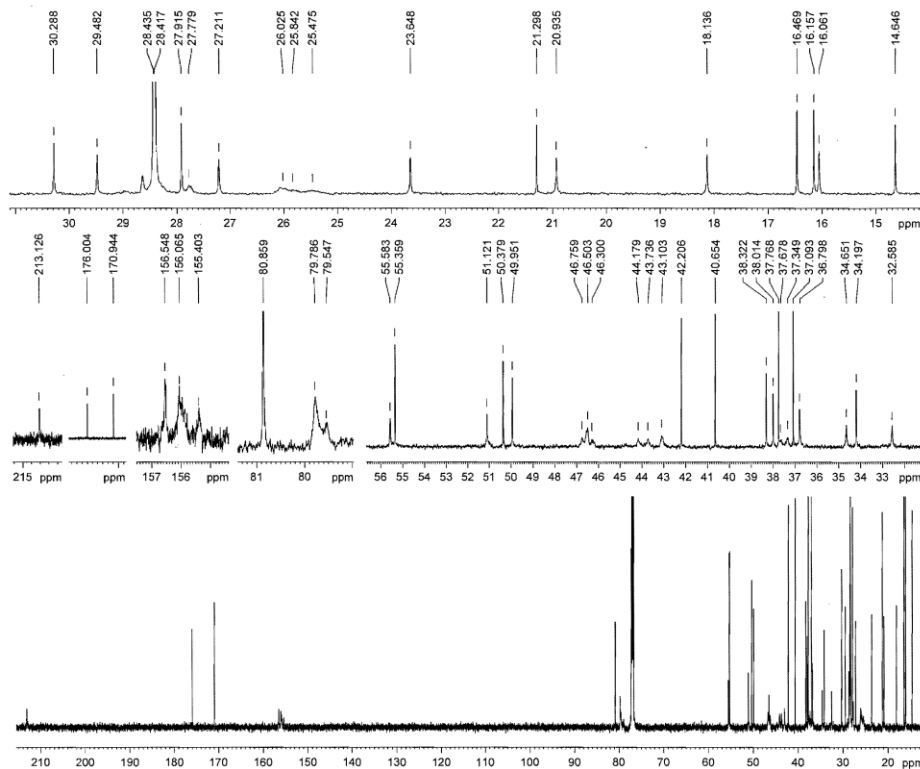
Current Data Parameters
NAME Wimmer-MV_16
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180427
Time 8.44
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 43288
SOLVENT CDCl₃
NS 16
DS 0
SWH 5411.255 Hz
FIDRES 0.125006 Hz
AQ 3.9998112 sec
RG 3
DW 92.400 usec
DE 20.00 usec
TE 298.2 K
D1 0 sec
TD0 1

===== CHANNEL f1 =====
SFO1 600.1325806 MHz
NUC1 1H
P1 9.00 usec
PLW1 6.00000000 W

F2 - Processing parameters
SI 131072
SF 600.1300274 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00

55840



Z.Wimmer MV-16
in CDCl₃, rfp=TMS
27.4.2018 DA

Current Data Parameters
NAME Wimmer-MV_16
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180427
Time 9.10
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zgpg30
TD 72468
SOLVENT CDCl₃
NS 1442
DS 0
SWH 36231.883 Hz
FIDRES 0.500026 Hz
AQ 0.9999480 sec
RG 195.18
DW 13.800 usec
DE 18.00 usec
TE 298.2 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

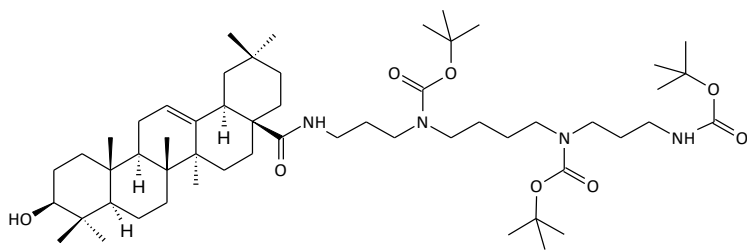
===== CHANNEL f1 =====
SFO1 150.9209173 MHz
NUC1 13C
P1 12.00 usec
PLW1 80.00000000 W

===== CHANNEL f2 =====
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG2 waltz16
PCPD2 70.00 usec
PLW2 6.00000000 W
PLW12 0.09918400 W
PLW13 0.04860000 W

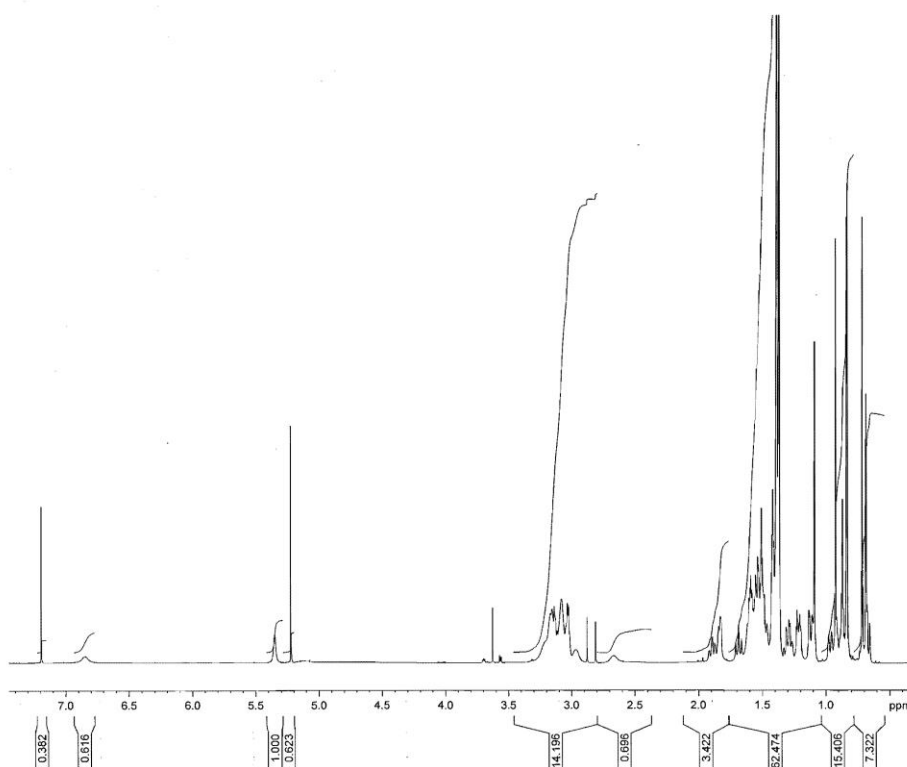
F2 - Processing parameters
SI 131072
SF 150.9209133 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0

55840

Analytical data of 7a



7a: $^1\text{H-NMR}$ (600.13 MHz, CDCl_3): δ [ppm] 0.67 (dd, 1H, $J=1.9$; 11.7 Hz, 5-CH), 0.68 (s, 3H, 26- CH_3), 0.71 (s, 3H, 24- CH_3), 0.83 (s, 3H, 25- CH_3), 0.83 (s, 3H, 29- CH_3), 0.87 (s, 3H, 30- CH_3), 0.92 (s, 3H, 23- CH_3), 0.97 (dt, 2H, $J=3.4$; 3.4; 12.8 Hz, 15- CH_2), 1.09 (s, 3H, 27- CH_3), 1.22 (dt, 2H, $J=3.4$; 3.4; 12.5 Hz, 7- CH_2), 1.29 (dt, 2H, $J=4.2$; 13.5; 13.5 Hz, 21- CH_2), 1.37 (s, Boc), 1.39 (s, Boc), 1.39 (s, Boc), 1.69 (t, 2H, $J=13.4$ Hz, 19- CH_2), 1.90 (dt, 2H, $J=4.0$; 13.6; 13.6 Hz, 2- CH_2), 2.68 (bs, 1H, 18-CH), 2.93-3.26 (m, 12H, 1', 3', 4', 7', 8', 10'- CH_2), 3.18 (dd, 1H, $J=4.0$; 10.7 Hz, 3-CH), 5.35 (bs, 1H, 12-CH); $^{13}\text{C-NMR}$ (150.92 MHz, CDCl_3): δ [ppm] 15.33 (q, 25-C), 15.56 (q, 24-C), 17.01 (q, 26-C), 18.38 (t, 6-C), 23.53 (t, 2-C), 23.53 (t, 11-C), 23.53 (t, 16-C), 23.64 (q, 30-C), 25.86 (q, 27-C), 26.08 (t, 2'-C), 26.08 (t, 6'-C), 27.26 (t, 15-C), 27.51 (t, 5'-C), 27.87 (t, 9'-C), 28.14 (q, 23-C), 30.78 (s, 20-C), 33.06 (t, 22-C), 32.59 (t, 7-C), 33.06 (q, 29-C), 34.30 (t, 21-C), 35.31 (t, 10'-C), 37.09 (s, 4-C), 37.71 (t, 1'-C), 38.57 (s, 10-C), 38.81 (t, 1-C), 39.50 (s, 8-C), 41.78 (t, 7'-C), 42.00 (s, 14-C), 42.00 (d, 18-C), 43.51 (t, 4'-C), 44.14 (t, 3'-C), 46.33 (t, 19-C), 46.80 (t, 8'-C), 46.84 (s, 17-C), 47.70 (d, 9-C), 55.27 (d, 5-C), 79.02 (d, 3-C), 122.95 (d, 12-C), 144.59 (s, 13-C), 177.79 (s, 28-C). IR (cm^{-1}): 3388, 2942, 2870, 1678, 1528, 1477, 1419, 1366, 1250, 1168. MS (ESI, 20 V): m/z = 941.6 $[\text{M}+\text{H}]^+$, 963.6 $[\text{M}+\text{Na}]^+$. For $\text{C}_{55}\text{H}_{96}\text{N}_4\text{O}_8$ (941.37) calcd. (%) C (70.17), H (10.28), N (5.95), found (%) C (70.15), H (10.29), N (5.94).



Z.Wimmer MV-9
in CDCl₃, rf=TMS
27.10.2017 DA

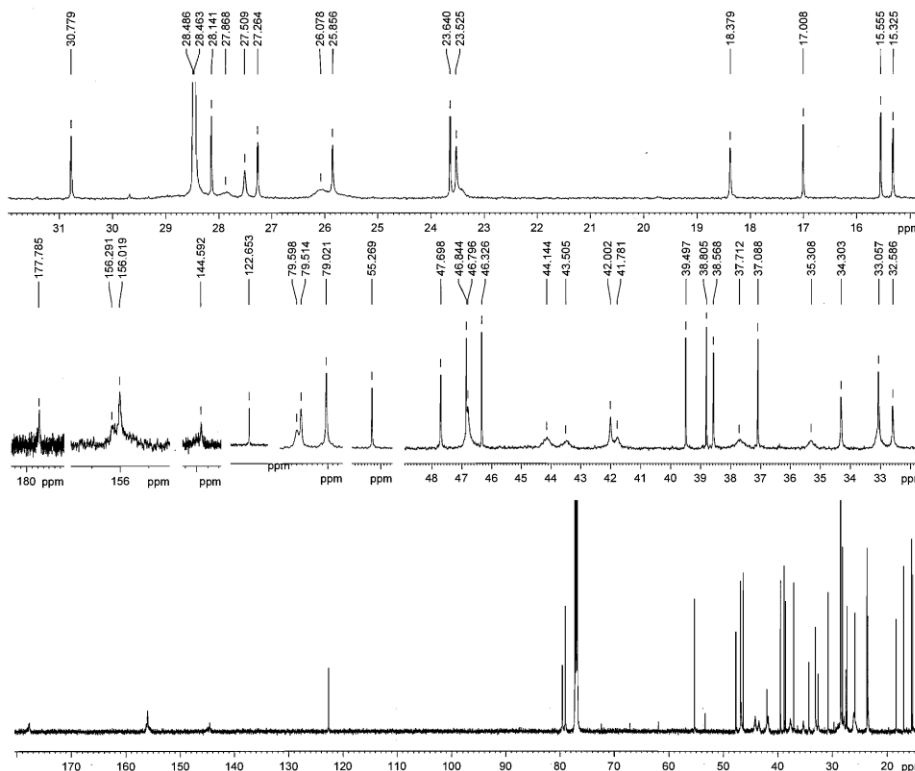
Current Data Parameters
NAME Wimmer-MV_9
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20171027
Time 7.40
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 43288
SOLVENT CDCl₃
NS 16
DS 0
SWH 5411.255 Hz
FIDRES 0.125006 Hz
AQ 3.9995112 sec
RG 35
DW 92.400 usec
DE 20.00 usec
TE 318.1 K
D1 0 sec
TDO 1

===== CHANNEL f1 =====
SFO1 600.1325806 MHz
NUC1 1H
P1 9.00 usec
PLW1 6.00000000 W

F2 - Processing parameters
SI 131072
SF 600.1300537 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00

55050



Z.Wimmer MV-9
in CDCl₃, rf=TMS
27.10.2017 DA

Current Data Parameters
NAME Wimmer-MV_9
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20171027
Time 9.55
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zgpg30
TD 59520
SOLVENT CDCl₃
NS 3899
DS 0
SWH 29761.904 Hz
FIDRES 0.500032 Hz
AQ 0.9999360 sec
RG 195.18
DW 16.800 usec
DE 18.00 usec
TE 318.2 K
D1 1.00000000 sec
D11 0.03000000 sec
TDO 1

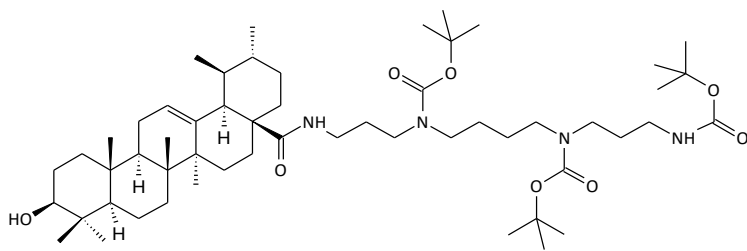
===== CHANNEL f1 =====
SFO1 150.9178993 MHz
NUC1 13C
P1 12.00 usec
PLW1 80.00000000 W

===== CHANNEL f2 =====
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG2 waltz16
PCPD2 70.00 usec
PLW2 6.00000000 W
PLW12 0.09918400 W
PLW13 0.04860000 W

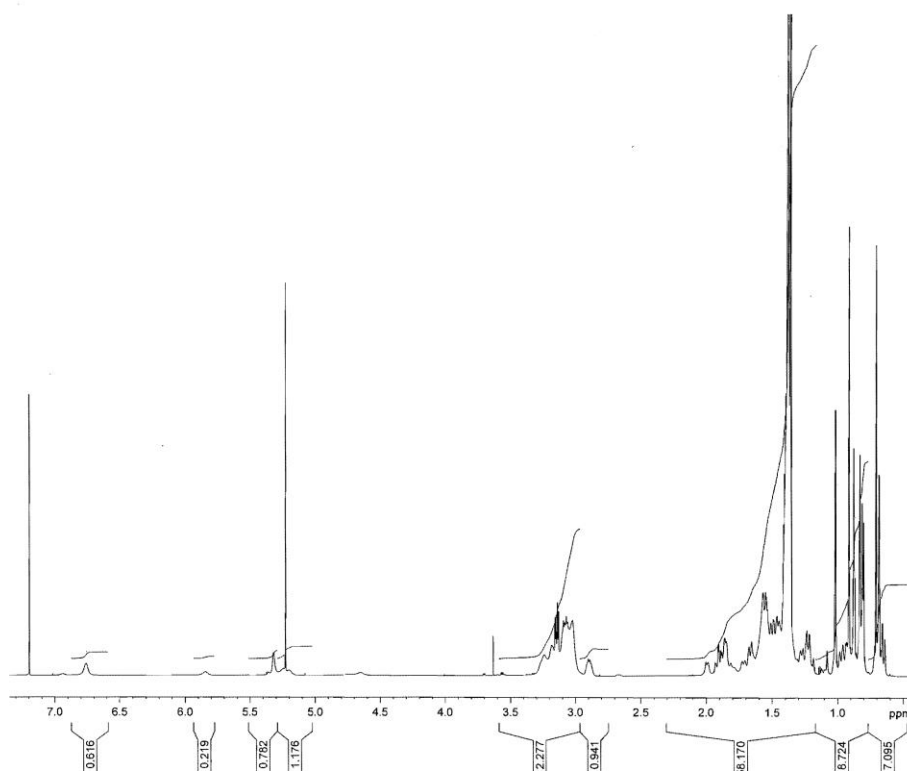
F2 - Processing parameters
SI 131072
SF 150.9028033 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0

55050

Analytical data of 7b



7b: $^1\text{H-NMR}$ (600.13 MHz, CDCl_3): δ [ppm] 0.65 (dd, 1H, $J=1.7$; 11.8 Hz, 5-CH), 0.71 (s, 3H, 26- CH_3), 0.81 (d, 3H, $J=6.4$ Hz, 29- CH_3), 0.83 (s, 3H, 24- CH_3), 0.87 (d, 3H, $J=6.4$ Hz, 30- CH_3), 0.92 (s, 3H, 23- CH_3), 0.92 (s, 3H, 25- CH_3), 1.02 (s, 3H, 27- CH_3), 1.37 (s, Boc), 1.38 (s, Boc), 1.39 (s, Boc), 1.91 (dt, 2H, $J=3.5$; 13.7; 13.7 Hz, 16- CH_2), 2.87-3.30 (m, 12H, 1', 3', 4', 7', 8', 10'- CH_2), 3.15 (dd, 1H, $J=4.5$; 11.4 Hz, 3-CH), 5.32 (bs, 1H, 12-CH), 6.75 (bs, 1H, 1'-NH); $^{13}\text{C-NMR}$ (150.92 MHz, CDCl_3): δ [ppm] 15.42 (q, 25-C), 15.59 (q, 24-C), 16.92 (q, 29-C), 17.23 (q, 26-C), 21.25 (q, 30-C), 23.28 (t, 11-C), 23.36 (q, 27-C), 24.54 (t, 16-C), 25.88 (t, 6'-C), 26.06 (t, 2'-C), 27.17 (t, 2-C), 27.72 (t, 5'-C), 27.87 (t, 15-C), 28.11 (q, 23-C), 28.66 (t, 9'-C), 30.96 (t, 21-C), 32.80 (t, 7-C), 35.29 (t, 10'-C), 36.93 (s, 10-C), 37.33 (t, 22-C), 37.54 (t, 1'-C), 38.58 (t, 1-C), 38.73 (d, 20-C), 39.01 (s, 4-C), 39.52 (s, 8-C), 39.68 (d, 19-C), 42.24 (s, 14-C), 43.37 (t, 7'-C), 43.69 (t, 4'-C), 44.14 (t, 3'-C), 46.70 (t, 8'-C), 47.50 (s, 17-C), 47.52 (d, 9-C), 53.11 (d, 18-C), 55.12 (d, 5-C), 78.98 (d, 3-C), 125.55 (d, 12-C), 139.00 (s, 13-C), 177.70 (s, 28-C). IR (cm^{-1}): 2975, 2871, 1684, 1525, 1476, 1420, 1366, 1250, 1168, 1045. MS (ESI, 20 V): m/z = 941.5 $[\text{M}+\text{H}]^+$, 963.5 $[\text{M}+\text{Na}]^+$, 985.4 $[\text{M}+\text{HCOO}]^-$. For $\text{C}_{55}\text{H}_{96}\text{N}_4\text{O}_8$ (941.37) calcd. (%) C (70.17), H (10.28), N (5.95), found (%) C (70.16), H (10.29), N (5.93).



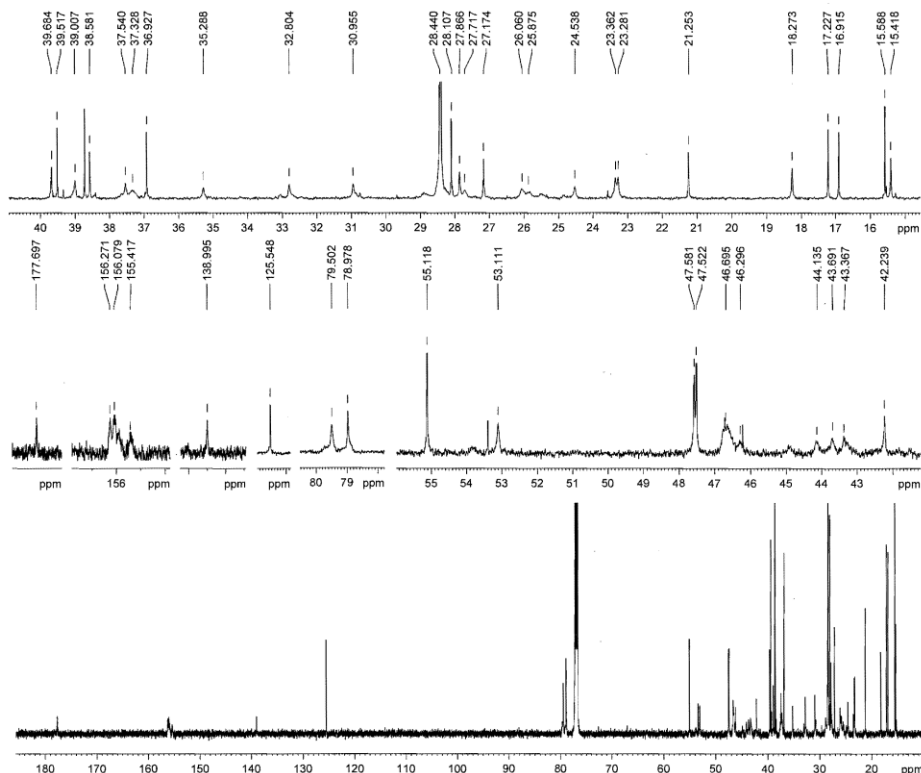
Z.Wimmer MV-13
in CDCl₃, rfp=TMS
22.1.2018 DA

Current Data Parameters
NAME Wimmer-MV_13
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180122
Time 7.33 h
INSTRUM spect
PROBHD Z44896_0113 (C
PULPROG zg30
TD 43288
SOLVENT CDCl₃
NS 16
DS 0
SWH 5411.255 Hz
FIDRES 0.250012 Hz
AQ 3.9998112 sec
RG 61.28
DW 92.400 usec
DE 20.00 usec
TE 298.2 K
D1 0 sec
TD0 1
SFO1 600.1325806 MHz
NUC1 1H
P1 9.00 usec
PLW1 6.0000000 W

F2 - Processing parameters
SI 131072
SF 600.1300537 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00

55376



Z.Wimmer MV-13
in CDCl₃, rfp=TMS
22.1.2018 DA

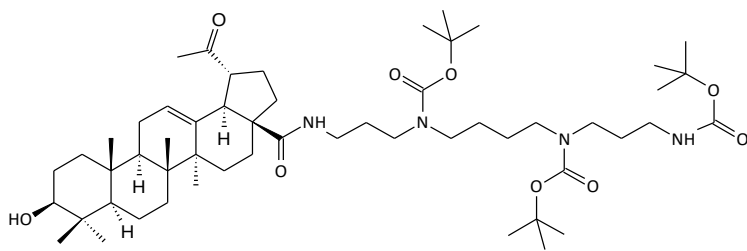
Current Data Parameters
NAME Wimmer-MV_13
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180122
Time 9.05 h
INSTRUM spect
PROBHD Z44896_0113 (C
PULPROG zgpg30
TD 59528
SOLVENT CDCl₃
NS 2671
DS 0
SWH 29781.904 Hz
FIDRES 1.000064 Hz
AQ 0.9999360 sec
RG 195.18
DW 16.800 usec
DE 18.00 usec
TE 298.2 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 150.9178993 MHz
NUC1 13C
P1 12.00 usec
PLW1 80.00000000 W
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG2 waltz16
PCPD2 70.00 usec
PLW2 6.00000000 W
PLW12 0.09918400 W
PLW13 0.04986900 W

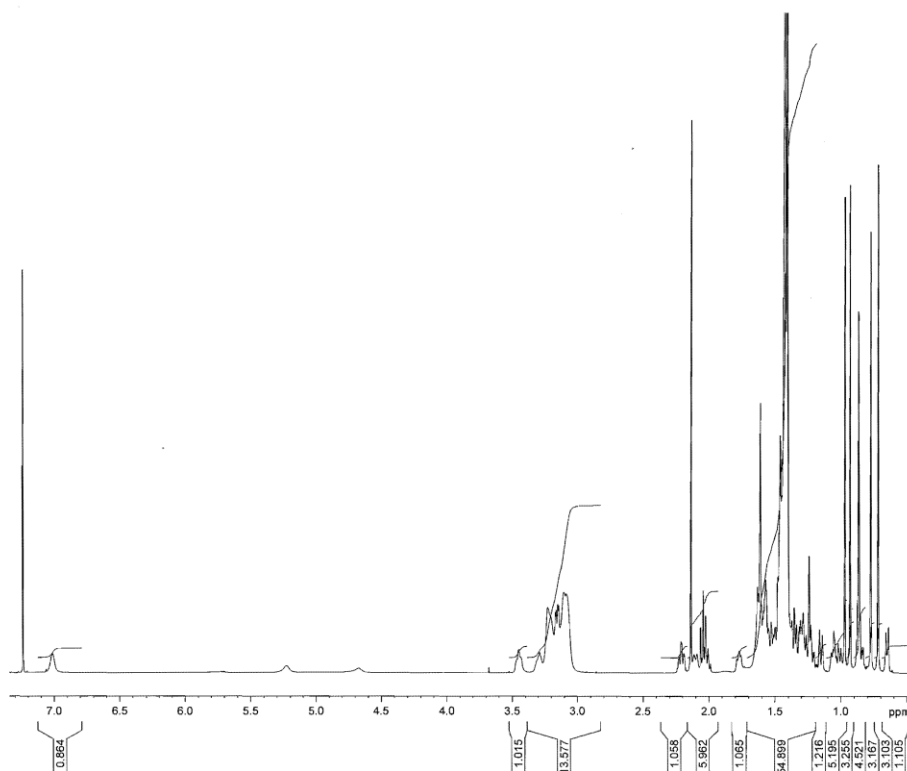
F2 - Processing parameters
SI 131072
SF 150.9028136 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

55353

Analytical data of 7c



7c: $^1\text{H-NMR}$ (600.13 MHz, CDCl_3): δ [ppm] 0.65 (dd, 1H, $J=2.2$; 11.3 Hz, 5-CH), 0.72 (d, 3H, $J=1.1$ Hz, 26- CH_3), 0.78 (s, 3H, 25- CH_3), 0.87 (s, 3H, 24- CH_3), 0.93 (d, 3H, $J=0.9$ Hz, 23- CH_3), 0.97 (s, 3H, 27- CH_3), 1.15 (dt, 2H, $J=3.1$; 3.1; 13.4 Hz, 15- CH_2), 1.41 (s, Boc), 1.43 (s, Boc), 1.43 (s, Boc), 2.05 (t, 1H, $J=11.3$ Hz, 18-CH), 2.14 (s, 3H, 29- CH_3), 2.21 (dt, H, $J=3.6$; 12.2; 12.2 Hz, 13-CH), 3.04-3.33 (m, 12H, 1', 3', 4', 7', 8', 10'- CH_2), 3.45 (bdt, 2H, $J=4.3$; 11.2; 11.2 Hz, 19- CH_2), 3.17 (dd, 1H, $J=5.6$; 11.3 Hz, 3-CH), 7.02 (bs, 1H, 1'-NH); $^{13}\text{C-NMR}$ (150.92 MHz, CDCl_3): δ [ppm] 14.61 (q, 27-C), 15.35 (q, 26-C), 16.06 (q, 24-C), 16.10 (q, 25-C), 18.25 (t, 6-C), 20.92 (t, 11-C), 25.45 (t, 6'-C), 26.05 (t, 5'-C), 27.25 (t, 2-C), 27.25 (t, 12-C), 27.37 (t, 21-C), 27.77 (t, 9'-C), 27.96 (q, 23-C), 28.42 (q, Boc), 28.66 (t, 2'-C), 29.50 (t, 15-C), 30.33 (q, 29-C), 32.58 (t, 16-C), 34.27 (t, 7-C), 34.64 (t, 10'-C), 36.83 (t, 22-C), 37.13 (s, 10-C), 37.70 (s, 4-C), 37.70 (t, 1'-C), 38.64 (d, 13-C), 38.83 (t, 1-C), 40.64 (s, 8-C), 42.21 (s, 14-C), 43.08 (t, 7'-C), 43.74 (t, 8'-C), 44.17 (t, 4'-C), 46.77 (t, 3'-C), 49.97 (d, 18-C), 50.47 (d, 9-C), 51.12 (d, 19-C), 55.28 (d, 5-C), 55.59 (s, 17-C), 78.87 (d, 3-C), 79.55 (s, Boc), 79.78 (s, Boc), 79.78 (s, Boc), 156.05 (s, Boc), 156.05 (s, Boc), 156.56 (s, Boc), 176.00 (s, 28-C), 213.19 (s, 20-C). IR (cm^{-1}): 3367, 2934, 2866, 1671, 1418, 1364, 1246, 1169, 750. MS (ESI, 20 V): m/z = 943.3 $[\text{M}+\text{H}]^+$, 965.6 $[\text{M}+\text{Na}]^+$, 987.5 $[\text{M}+\text{HCOO}]^-$. For $\text{C}_{54}\text{H}_{92}\text{N}_4\text{O}_9$ (941.33) calcd. (%) C (68.90), H (9.85), N (5.95), found (%) C (68.92), H (9.84), N (5.97).



Z.Wimmer MV-17
in CDCl₃, rfp=TMS
27.4.2018 DA

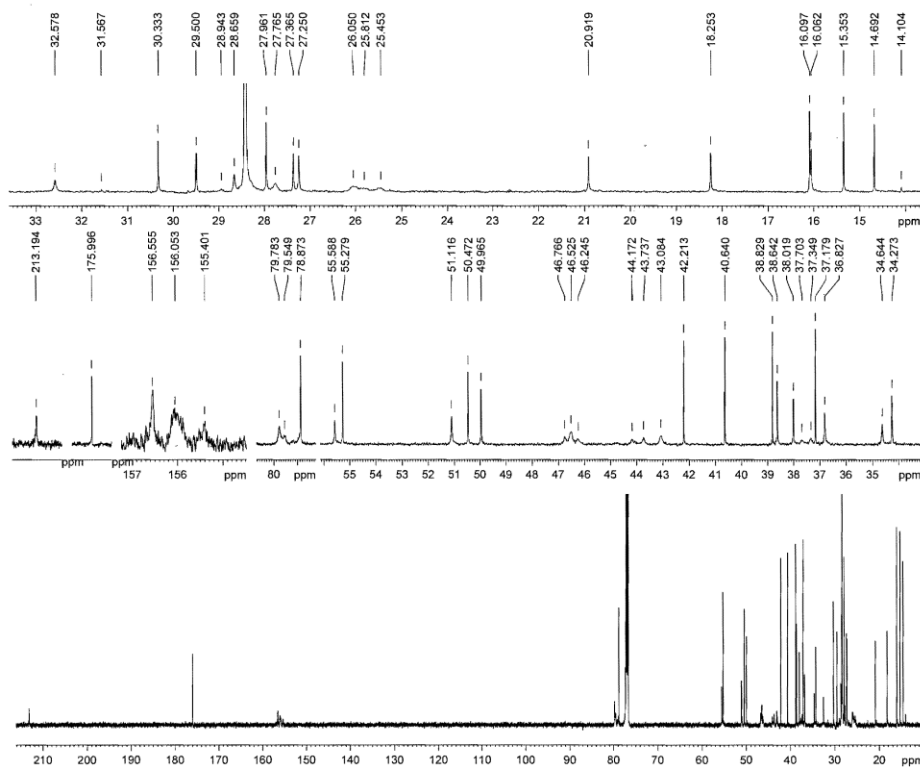
Current Data Parameters
NAME Wimmer-MV_17
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180427
Time 11.41
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 43288
SOLVENT CDCl₃
NS 16
DS 0
SWH 5411.255 Hz
FIDRES 0.125006 Hz
AQ 3.9998112 sec
RG 198.18
DW 92.400 usec
DE 20.00 usec
TE 298.1 K
D1 0 sec
TD0 1

===== CHANNEL f1 =====
SFO1 600.1325806 MHz
NUC1 1H
P1 9.00 usec
PLW1 6.00000000 W

F2 - Processing parameters
SI 131072
SF 600.1300271 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00

55841



Z.Wimmer MV-17
in CDCl₃, rfp=TMS
27.4.2018 DA

Current Data Parameters
NAME Wimmer-MV_17
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180427
Time 13.03
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zgpg30
TD 72460
SOLVENT CDCl₃
NS 2382
DS 0
SWH 36231.883 Hz
FIDRES 0.500026 Hz
AQ 0.9999480 sec
RG 198.18
DW 13.800 usec
DE 18.00 usec
TE 298.1 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

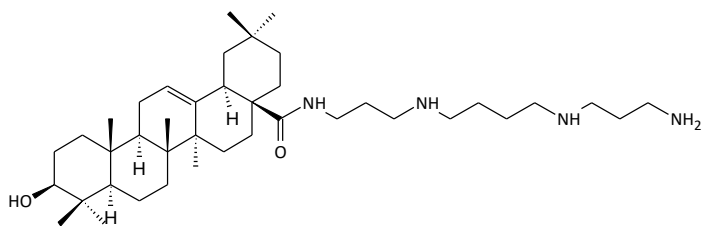
===== CHANNEL f1 =====
SFO1 150.9209173 MHz
NUC1 13C
P1 12.00 usec
PLW1 80.00000000 W

===== CHANNEL f2 =====
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG2 waltz16
PCPD2 70.00 usec
PLW2 4.59999990 W
PLW12 0.08008200 W
PLW13 0.02944000 W

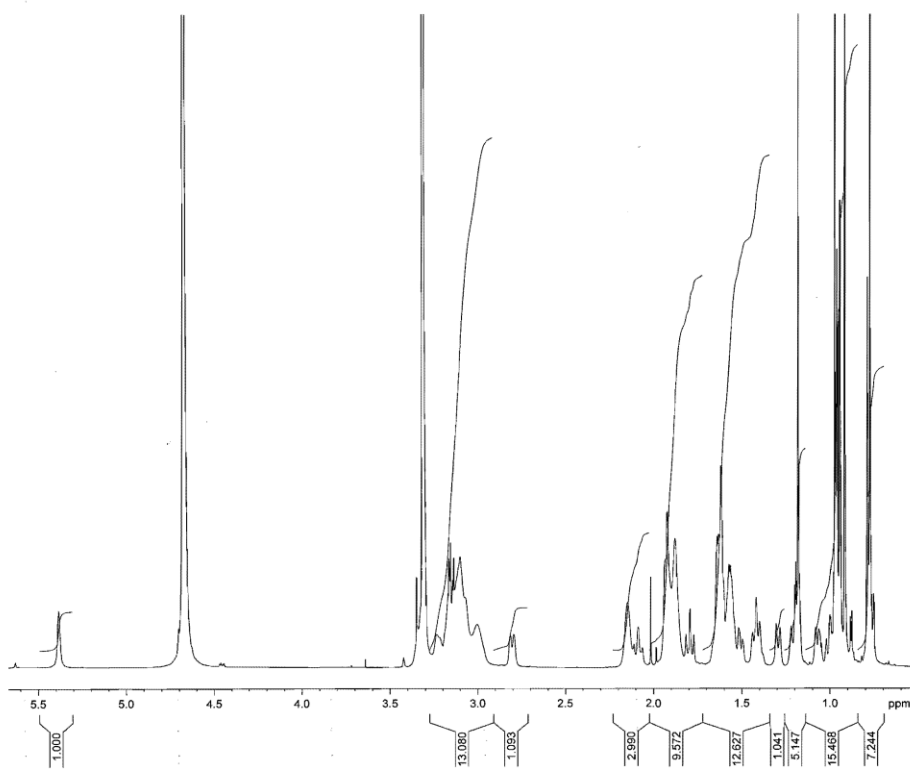
F2 - Processing parameters
SI 131072
SF 150.9028132 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0

55841

Analytical data of 8a



8a: $^1\text{H-NMR}$ (600.13 MHz, CDCl_3): δ [ppm] 0.76 (dd, 1H, $J=1.8$; 11.2 Hz, 5-CH), 0.78 (s, 3H, 24- CH_3), 0.79 (s, 3H, 26- CH_3), 0.92 (s, 3H, 29- CH_3), 0.94 (s, 3H, 25- CH_3), 0.96 (s, 3H, 30- CH_3), 0.97 (s, 3H, 23- CH_3), 1.18 (s, 3H, 27- CH_3), 1.79 (bt, 2H, $J=13.6$ Hz, 19- CH_2), 2.80 (bd, 1H, $J=12.8$ Hz, 18-CH), 2.96-3.19 (m, 10H, 3',4', 7',8',10'- CH_2), 3.15 (dd, 1H, $J=4.8$; 11.4 Hz, 3-CH), 3.21-3.26 (m, 1H, 1'- CH_2), 3.31-3.37 (m, 1H, 1'- CH_2), 5.38 (bs, 1H, 12-CH); $^{13}\text{C-NMR}$ (150.92 MHz, CDCl_3): δ [ppm] 15.93 (q, 25-C), 16.28 (q, 24-C), 18.08 (q, 26-C), 19.49 (t, 6-C), 24.04 (t, 11-C), 24.16 (q, 30-C), 24.51 (t, 2-C), 24.51 (t, 6'-C), 24.59 (t, 5'-C), 25.49 (t, 2'-C), 26.44 (q, 27-C), 27.74 (t, 16-C), 27.88 (t, 15-C), 28.61 (t, 9'-C), 28.73 (q, 23-C), 31.59 (s, 20-C), 33.45 (q, 29-C), 33.88 (t, 22-C), 34.83 (t, 7-C), 35.13 (t, 21-C), 37.28 (t, 1'-C), 38.19 (s, 4-C), 38.24 (t, 10'-C), 39.83 (t, 1-C), 39.83 (s, 10-C), 40.77 (s, 8-C), 42.57 (d, 18-C), 42.97 (s, 14-C), 46.28 (t, 7'-C), 46.74 (t, 4'-C), 47.63 (s, 17-C), 47.77 (t, 19-C), 48.43 (t, 8'-C), 48.50 (t, 3'-C), 48.89 (d, 9-C), 56.72 (d, 5-C), 79.68 (d, 3-C), 124.31 (d, 12-C), 145.07 (s, 13-C), 181.86 (s, 28-C). IR (cm^{-1}): 2943, 2874, 1735, 1694, 1450, 1371, 1246, 1030. MS (ESI, 20 V): m/z = 641.5 $[\text{M}+\text{H}]^+$. For $\text{C}_{40}\text{H}_{72}\text{N}_4\text{O}_2$ (641.03) calcd. (%) C (74.95), H (11.32), N (8.74), found (%) C (74.94), H (11.33), N (8.72).



Z.Wimmer MV-10
in CD3OD, rfp=3.31 ppm
26.10.2017 DA

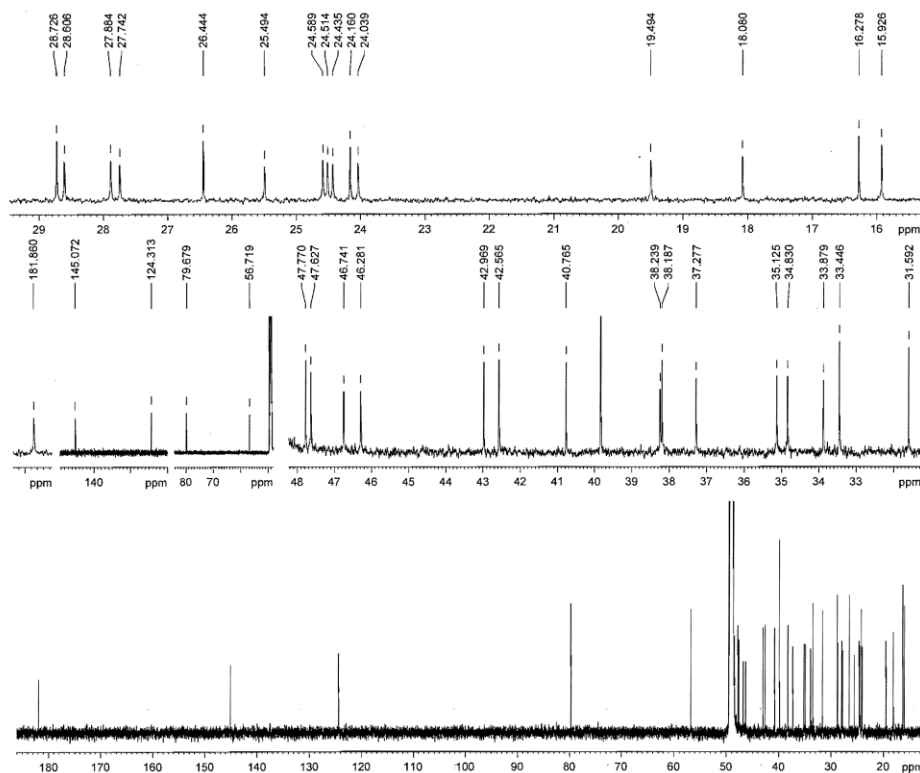
Current Data Parameters
NAME Wimmer-MV_10
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20171026
Time 7.55
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 43288
SOLVENT MeOD
NS 16
DS 0
SWH 5411.255 Hz
FIDRES 0.125006 Hz
AQ 3.9998112 sec
RG 55.4
DW 92.400 usec
DE 20.00 usec
TE 318.1 K
D1 0 sec
TD0 1

===== CHANNEL f1 =====
SFO1 600.1325806 MHz
NUC1 1H
P1 9.00 usec
PLW1 6.00000000 W

F2 - Processing parameters
SI 131072
SF 600.1300099 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00

55048



Z.Wimmer MV-10
in CD3OD, rfp=3.31 ppm
26.10.2017 DA

Current Data Parameters
NAME Wimmer-MV_10
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20171026
Time 7.57
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zgpg30
TD 59520
SOLVENT MeOD
NS 1003
DS 0
SWH 29761.904 Hz
FIDRES 0.500032 Hz
AQ 0.9999360 sec
RG 196.18
DW 16.800 usec
DE 18.00 usec
TE 318.2 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

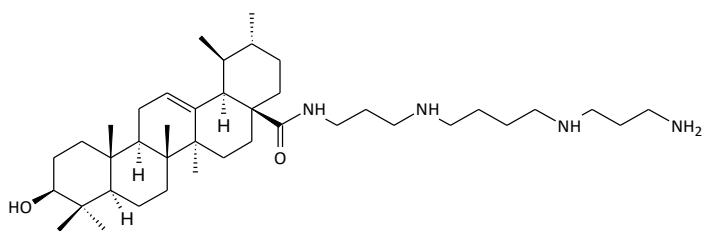
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NUC1 13C
P1 12.00 usec
PLW1 80.00000000 W

===== CHANNEL f2 =====
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG2 waltz16
PCPD2 70.00 usec
PLW2 6.00000000 W
PLW12 0.09918400 W
PLW13 0.04850000 W

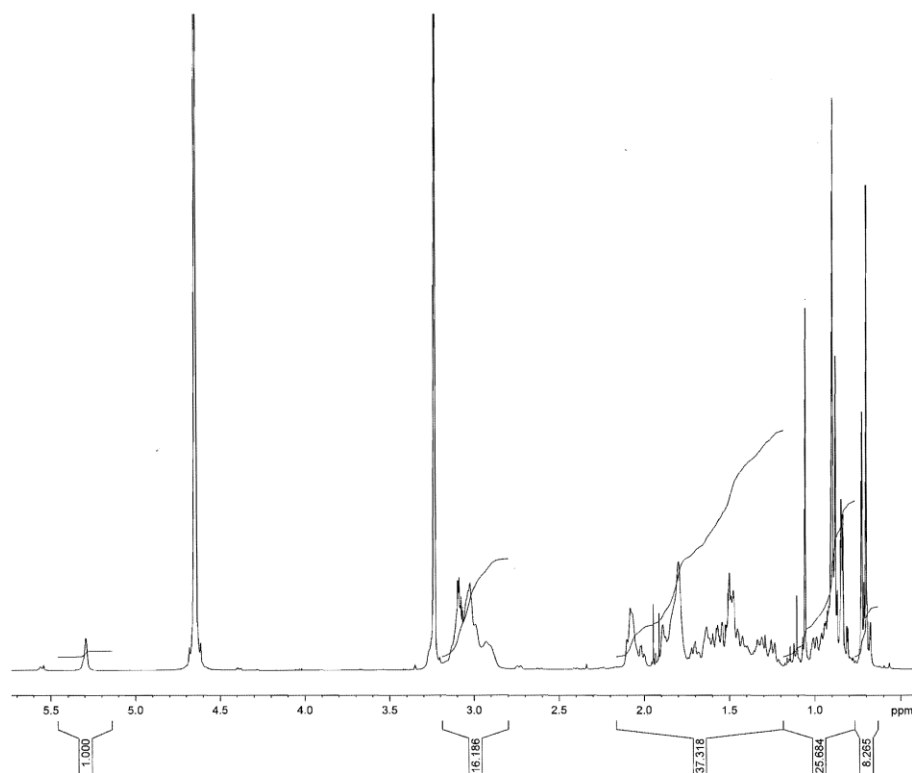
F2 - Processing parameters
SI 131072
SF 150.9025976 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0

55048

Analytical data of 8b



8b: ¹H-NMR (600.13 MHz, CD₃OD): δ [ppm] 0.69 (bd, 1H, *J*=11.2 Hz, 5-CH), 0.70 (s, 3H, 24-CH₃), 0.73 (s, 3H, 26-CH₃), 0.84 (d, 3H, *J*=6.4 Hz, 29-CH₃), 0.88 (s, 3H, 25-CH₃), 0.90 (s, 3H, 23-CH₃), 0.90 (d, 3H, *J*=6.5 Hz, 30-CH₃), 1.06 (s, 3H, 27-CH₃), 2.87-3.15 (m, 12H, 1', 3', 4', 7', 8', 10'-CH₂), 3.08 (dd, 1H, *J*=4.5; 11.4 Hz, 3-CH); ¹³C-NMR (150.92 MHz, CD₃OD): δ [ppm] 16.03 (q, 25-C), 16.34 (q, 24-C), 17.68 (q, 29-C), 18.01 (q, 26-C), 21.50 (q, 30-C), 24.08 (q, 27-C), 24.37 (t, 11-C), 24.40 (t, 16-C), 24.45 (t, 6'-C), 25.18 (t, 5'-C), 25.44 (t, 2'-C), 27.63 (t, 2-C), 27.89 (t, 9'-C), 28.75 (q, 23-C), 29.00 (t, 15-C), 31.89 (t, 21-C), 34.15 (t, 7-C), 37.15 (t, 1'-C), 37.15 (t, 10'-C), 38.13 (s, 10-C), 39.20 (t, 22-C), 39.82 (s, 4-C), 39.96 (t, 1-C), 40.32 (d, 19-C), 40.80 (d, 20-C), 40.94 (s, 8-C), 43.31 (s, 14-C), 46.15 (t, 4'-C), 46.15 (t, 7'-C), 46.64 (s, 17-C), 48.35 (t, 3'-C), 48.35 (t, 8'-C), 49.02 (d, 9-C), 54.09 (d, 18-C), 56.68 (d, 5-C), 79.65 (d, 3-C), 127.32 (d, 12-C), 139.84 (s, 13-C), 181.68 (s, 28-C). IR (cm⁻¹): 2927, 2868, 1624, 1525, 1457, 1383, 1245, 1030, 996. MS (ESI, 20 V): *m/z* = 641.4 [M+H]⁺. For C₄₀H₇₂N₄O₂ (641.03) calcd. (%) C (74.95), H (11.32), N (8.74), found (%) C (74.97), H (11.31), N (8.75).



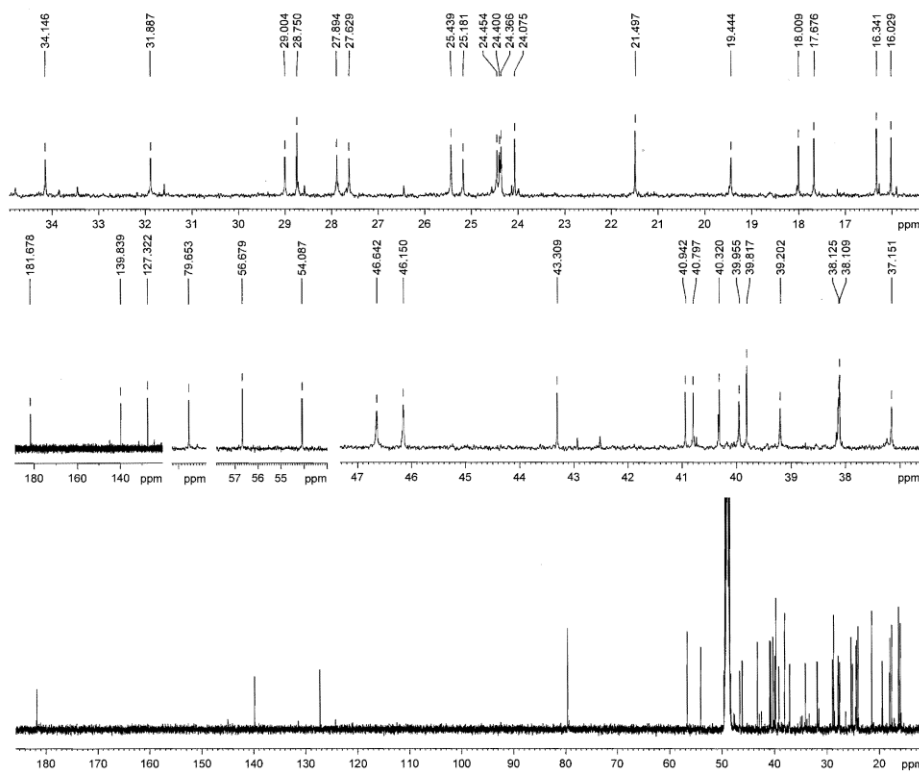
ZWimmer MV-14
in CD3OD, rfp=TMS
22.2.2018 DA

Current Data Parameters
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EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180222
Time 8.25 h
INSTRUM spect
PROBHD Z44896_0113 (C
PULPROG zg30
TD 43288
SOLVENT MeOD
NS 16
DS 0
SWH 5411.255 Hz
FIDRES 0.250012 Hz
AQ 3.9998112 sec
RG 61.25
DW 92.400 usec
DE 10.00 usec
TE 313.2 K
D1 0 sec
TD0 1
SFO1 600.1325806 MHz
NUC1 1H
P1 9.00 usec
PLW1 6.00000000 W

F2 - Processing parameters
SI 131072
SF 600.1300537 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00

55545



ZWimmer MV-14
in CD3OD, rfp=TMS
22.2.2018 DA

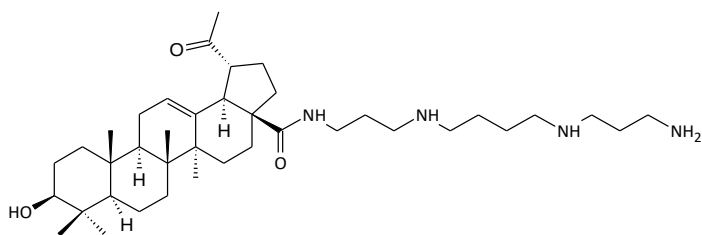
Current Data Parameters
NAME Wimmer-MV_14
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180222
Time 9.01 h
INSTRUM spect
PROBHD Z44896_0113 (C
PULPROG zgpg30
TD 59520
SOLVENT MeOD
NS 1060
DS 0
SWH 29761.904 Hz
FIDRES 1.000064 Hz
AQ 0.9999360 sec
RG 196.18
DW 16.800 usec
DE 18.00 usec
TE 313.2 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 150.9178993 MHz
NUC1 13C
P1 12.00 usec
PLW1 6.00000000 W
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG2 waltz16
PCPD2 70.00 usec
PLW2 6.00000000 W
PLW12 0.09918400 W
PLW13 0.04988900 W

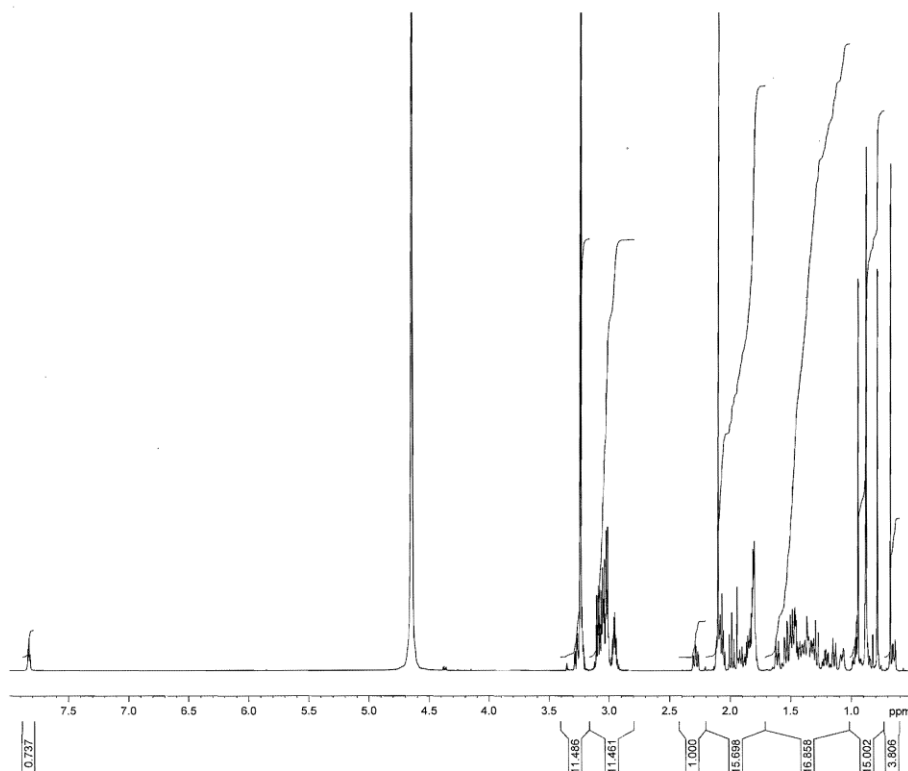
F2 - Processing parameters
SI 131072
SF 150.9025990 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

55545

Analytical data of 8c



8c: $^1\text{H-NMR}$ (600.13 MHz, CD_3OD): δ [ppm] 0.72 (dd, 1H, $J=1.8$; 11.1 Hz, 5-CH), 0.75 (s, 3H, 26- CH_3), 0.86 (s, 3H, 25- CH_3), 0.95 (s, 3H, 23- CH_3), 0.95 (s, 3H, 24- CH_3), 1.02 (s, 3H, 27- CH_3), 1.21 (dt, 2H, $J=3.4$; 3.4; 13.7 Hz, 15- CH_2), 2.06 (t, 1H, $J=11.3$ Hz, 18-CH), 2.15 (s, 3H, 29- CH_3), 2.35 (ddd, H, $J=3.8$; 11.8; 13.0 Hz, 13-CH), 3.01-3.18 (m, 12H, 1', 3', 4', 7', 8', 10'- CH_2), 3.34 (dt, 2H, $J=4.4$; 11.7; 11.7 Hz, 19- CH_2), 3.08-3.18 (m, 1H, 3-CH), 7.90 (bt, 1H, $J=5.9$ Hz, 1'-NH); $^{13}\text{C-NMR}$ (150.92 MHz, CD_3OD): δ [ppm] 15.01 (q, 27-C), 16.09 (q, 26-C), 16.75 (q, 24-C), 16.91 (q, 25-C), 19.44 (t, 6-C), 22.23 (t, 11-C), 24.32 (t, 6'-C), 24.38 (t, 5'-C), 25.39 (t, 12-C), 27.82 (t, 2-C), 28.05 (t, 21-C), 28.38 (t, 9'-C), 28.60 (q, 23-C), 29.47 (t, 2'-C), 29.54 (t, 15-C), 30.67 (q, 29-C), 33.36 (t, 16-C), 35.54 (t, 7-C), 36.87 (t, 10'-C), 37.97 (t, 22-C), 38.26 (s, 10-C), 38.39 (s, 4-C), 39.12 (t, 1'-C), 39.95 (t, 1-C), 40.07 (d, 13-C), 42.00 (s, 8-C), 43.36 (s, 14-C), 45.99 (t, 7'-C), 46.66 (t, 8'-C), 48.15 (t, 4'-C), 48.32 (t, 3'-C), 51.67 (d, 18-C), 52.00 (d, 9-C), 52.88 (d, 19-C), 56.88 (d, 5-C), 57.00 (s, 17-C), 79.60 (d, 3-C), 179.93 (s, 28-C), 215.90 (s, 20-C). IR (cm^{-1}): 3355, 2938, 2865, 1629, 1522, 1458, 1358, 1247, 1196, 1032. MS (ESI, 20 V): $m/z = 643.4$ $[\text{M}+\text{H}]^+$. For $\text{C}_{39}\text{H}_{68}\text{N}_4\text{O}_3$ (640.98) calcd. (%) C (73.08), H (10.69), N (8.74), found (%) C (73.05), H (10.70), N (8.73).



Z.Wimmer MV-18
in CD3OD, rfp=3.31
2.5.2018

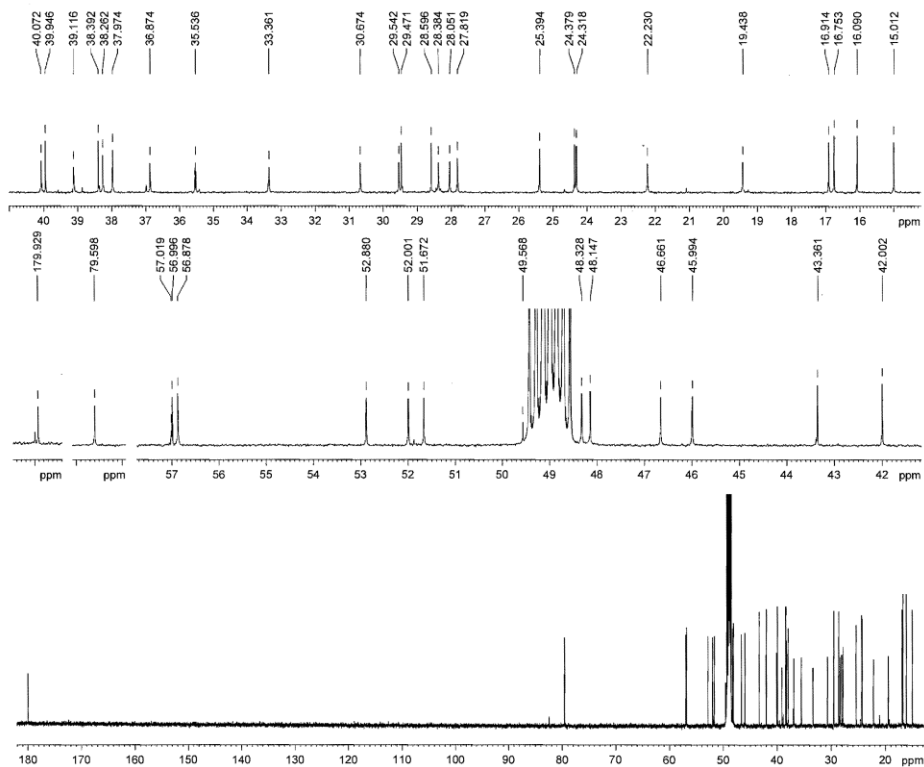
Current Data Parameters
NAME Wimmer-MV_18
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180502
Time 9.31
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 43286
SOLVENT MeOD
NS 16
DS 0
SWH 5411.255 Hz
FIDRES 0.125006 Hz
AQ 3.9988112 sec
RG 117.6
DW 92.400 usec
DE 20.00 usec
TE 313.1 K
D1 0 sec
TD0 1

===== CHANNEL f1 =====
SFO1 600.1325806 MHz
NUC1 1H
P1 9.00 usec
PLW1 6.00000000 W

F2 - Processing parameters
SI 131072
SF 600.1300537 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00

55850



Z.Wimmer MV-18
in CD3OD, rfp=3.31
2.5.2018

Current Data Parameters
NAME Wimmer-MV_18
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180502
Time 10.22
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zgpg30
TD 59520
SOLVENT MeOD
NS 1449
DS 0
SWH 29761.904 Hz
FIDRES 0.500032 Hz
AQ 0.9999360 sec
RG 196.16
DW 16.800 usec
DE 18.00 usec
TE 313.1 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

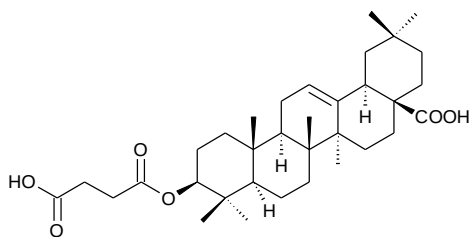
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SFO1 150.9178993 MHz
NUC1 13C
P1 12.00 usec
PLW1 80.00000000 W

===== CHANNEL f2 =====
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG2 waitz16
PCPD2 70.00 usec
PLW2 6.00000000 W
PLW12 0.09918400 W
PLW13 0.04860000 W

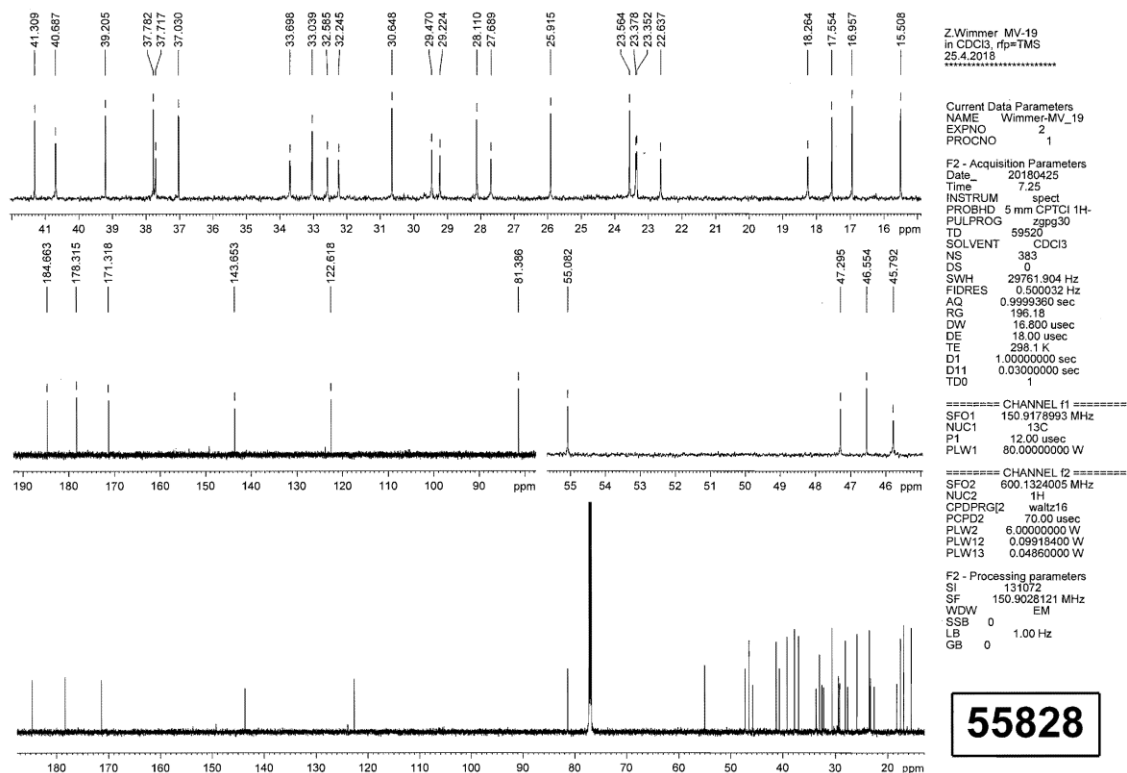
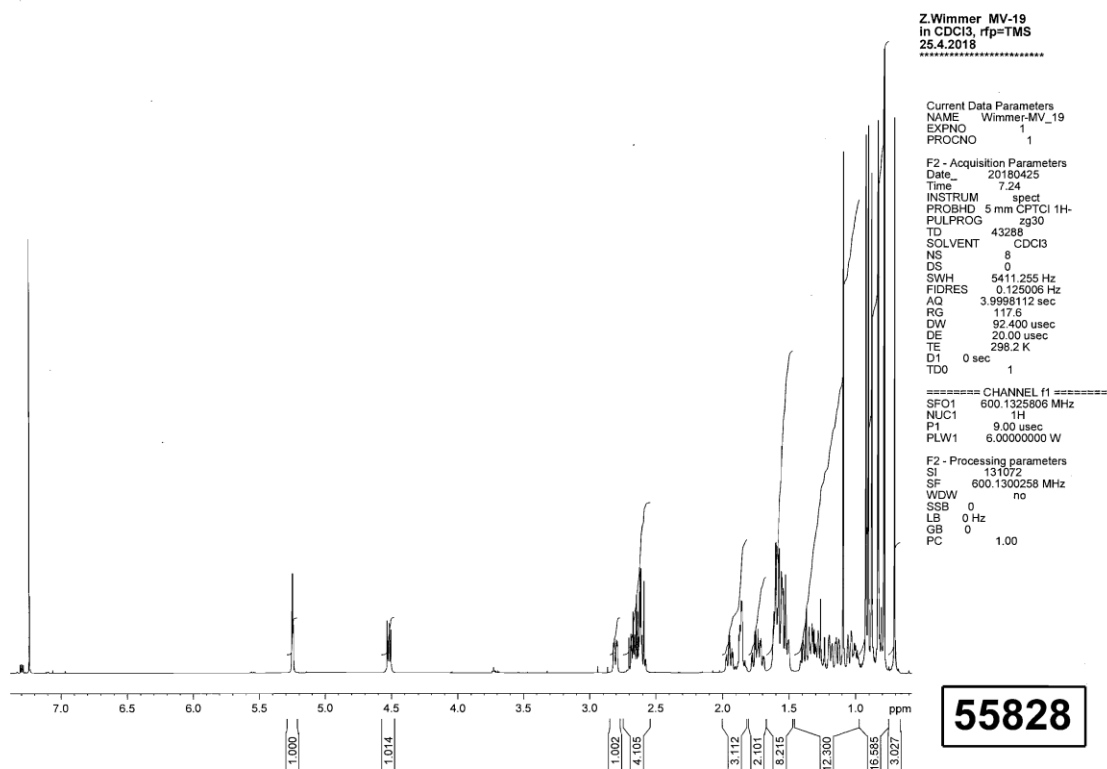
F2 - Processing parameters
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SF 150.9025972 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0

55850

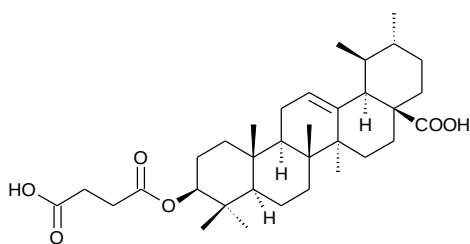
Analytical data of 9a



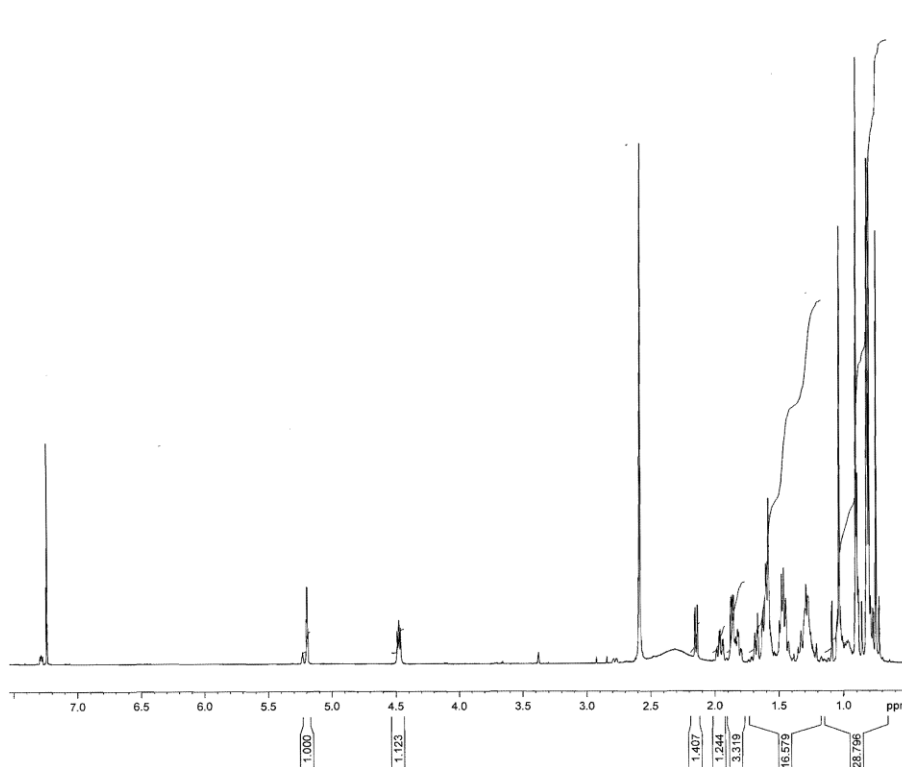
9a: ¹H-NMR (600.13 MHz, CDCl₃): δ [ppm] 0.71 (s, 3H, 26-CH₃), 0.79 (s, 3H, 25-CH₃), 0.82 (dd, 1H, *J*=1.8; 11.7 Hz, 5-CH), 0.83 (s, 3H, 24-CH₃), 0.88 (s, 3H, 29-CH₃), 0.91 (s, 3H, 23-CH₃), 0.92 (s, 3H, 30-CH₃), 1.05 (ddd, 2H, *J*=2.7; 3.8; 14.0 Hz, 16-CH₂), 1.09 (s, 3H, 27-CH₃), 1.14 (ddd, 2H, *J*=2.4; 3.7; 13.6 Hz, 19-CH₂), 1.82-1.91 (m, 2H, 2-CH₂), 1.95 (dt, 2H, *J*=4.1; 13.5; 13.5 Hz, 11-CH₂), 2.57-2.71 (m, 2H, 2', 3'-CH₂), 2.81 (bdd, 1H, *J*=4.5; 13.6 Hz, 18-CH), 4.52 (dd, 1H, *J*=5.9; 10.4 Hz, 3-CH), 5.25 (t, 1H, *J*=3.7 Hz, 12-CH); ¹³C-NMR (150.92 MHz, CDCl₃): δ [ppm] 15.51 (q, 24-C), 16.96 (q, 25-C), 17.55 (q, 26-C), 18.26 (t, 6-C), 22.64 (t, 11-C), 23.35 (t, 2-C), 23.38 (t, 2'-C), 23.56 (q, 30-C), 25.92 (q, 27-C), 27.69 (t, 16-C), 28.11 (t, 15-C), 29.22 (q, 23-C), 29.47 (t, 3'-C), 30.65 (s, 20-C), 32.25 (t, 22-C), 32.59 (t, 7-C), 33.04 (q, 29-C), 33.70 (t, 21-C), 37.03 (t, 1-C), 37.72 (s, 4-C), 37.78 (s, 10-C), 39.21 (s, 8-C), 40.69 (d, 18-C), 41.31 (s, 14-C), 45.79 (t, 19-C), 46.55 (s, 17-C), 47.30 (d, 9-C), 55.08 (d, 5-C), 81.39 (d, 3-C), 122.62 (d, 12-C), 143.65 (s, 13-C), 171.32 (s, 1'-C), 178.32 (s, 4'-C), 184.66 (s, 28-C). IR (cm⁻¹): 3392, 2941, 1697, 1165, 753. MS (ESI, 20 V): *m/z* = 579.2 [M+Na]⁺, 555.25 [M-H]⁺. For C₃₄H₅₂O₆ (556.77) calcd. (%) C (73.34), H (9.41), found (%) C (73.36), H (9.39).



Analytical data of 9b



9b: ¹H-NMR (600.13 MHz, CDCl₃): δ [ppm] 0.80 (s, 3H, 25-CH₃), 0.82 (s, 3H, 23-CH₃), 0.82 (s, 3H, 24-CH₃), 0.82 (d, 3H, *J*=6.5 Hz, 29-CH₃), 0.90 (d, 3H, *J*=6.5 Hz, 30-CH₃), 0.91 (s, 3H, 26-CH₃), 1.03 (s, 3H, 27-CH₃), 1.82 (dt, 2H, *J*=5.2; 14.4; 14.4 Hz, 15-CH₂), 1.83-1.93 (m, 2H, 2-CH₂), 1.96 (dt, 2H, *J*=4.5; 13.8; 13.8 Hz, 16-CH₂), 2.15 (bd, 1H, *J*=11.4 Hz, 18-CH), 2.21-2.58 (m, 2H, 2', 3'-CH₂), 4.48 (dd, 1H, *J*=5.8; 11.2 Hz, 3-CH), 5.20 (t, 1H, *J*=3.6 Hz, 12-CH); ¹³C-NMR (150.92 MHz, CDCl₃): δ [ppm] 15.46 (q, 25-C), 16.69 (q, 24-C), 16.92 (q, 29-C), 16.97 (q, 26-C), 18.13 (t, 6-C), 21.12 (q, 30-C), 23.22 (t, 11-C), 23.40 (q, 27-C), 23.49 (t, 2-C), 24.07 (t, 16-C), 27.94 (t, 15-C), 27.98 (q, 23-C), 28.95 (t, 2'-C), 29.56 (t, 3'-C), 30.57 (t, 21-C), 32.80 (t, 7-C), 36.82 (t, 22-C), 36.71 (s, 10-C), 37.68 (d, 20-C), 38.14 (t, 1-C), 38.79 (s, 4-C), 38.99 (s, 8-C), 39.42 (d, 19-C), 41.92 (s, 14-C), 47.35 (d, 9-C), 47.78 (s, 17-C), 52.61 (d, 18-C), 55.22 (d, 5-C), 81.41 (d, 3-C), 125.47 (d, 12-C), 138.06 (s, 13-C), 172.12 (s, 1'-C), 175.21 (s, 4'-C), 181.44 (s, 28-C). IR (cm⁻¹): 3385, 2923, 1686, 1169, 966. MS (ESI, 20 V): *m/z* = 554.9 [M-H]⁺. For C₃₄H₅₂O₆ (556.77) calcd. (%) C (73.34), H (9.41), found (%) C (73.36), H (9.39).



Z.Wimmer MV-20
in CDCl₃, rfp=TMS
2.5.2018

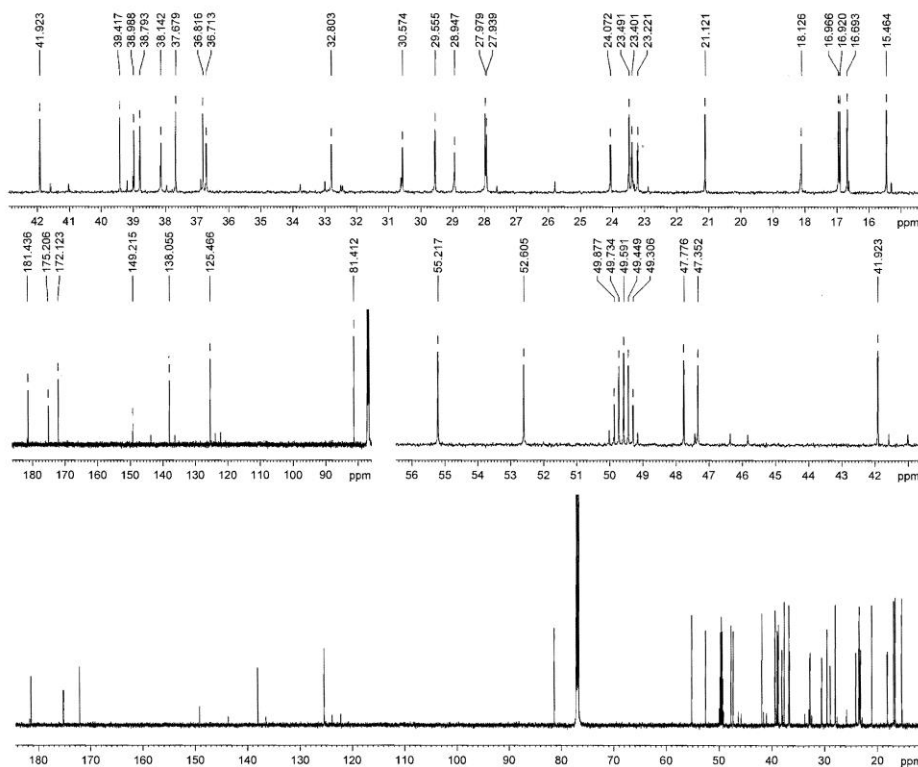
Current Data Parameters
NAME Wimmer-MV_20
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180502
Time 10.38
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 43288
SOLVENT CDCl₃
NS 16
DS 0
SWH 5411.255 Hz
FIDRES 0.125006 Hz
AQ 3.9998112 sec
RG 117.6
DW 92.400 usec
DE 20.00 usec
TE 298.1 K
D1 0 sec
TD0 1

===== CHANNEL f1 =====
SFO1 600.1325806 MHz
NUC1 1H
P1 9.00 usec
PLW1 6.00000000 W

F2 - Processing parameters
SI 131072
SF 600.1300269 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00

55853



Z.Wimmer MV-20
in CDCl₃, rfp=TMS
2.5.2018

Current Data Parameters
NAME Wimmer-MV_20
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180502
Time 11.27
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zgpg30
TD 59520
SOLVENT CDCl₃
NS 1427
DS 0
SWH 29761.904 Hz
FIDRES 0.500032 Hz
AQ 0.9999360 sec
RG 196.18
DW 16.800 usec
DE 18.00 usec
TE 298.1 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

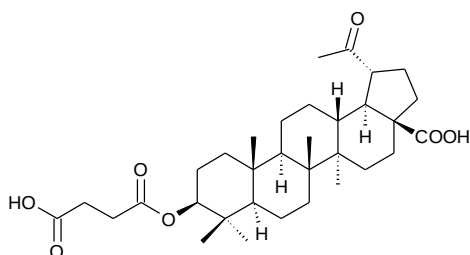
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SFO1 150.9178993 MHz
NUC1 13C
P1 12.00 usec
PLW1 80.00000000 W

===== CHANNEL f2 =====
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG2 waltz16
PCPD2 70.00 usec
PLW2 6.00000000 W
PLW12 0.09918400 W
PLW13 0.04860000 W

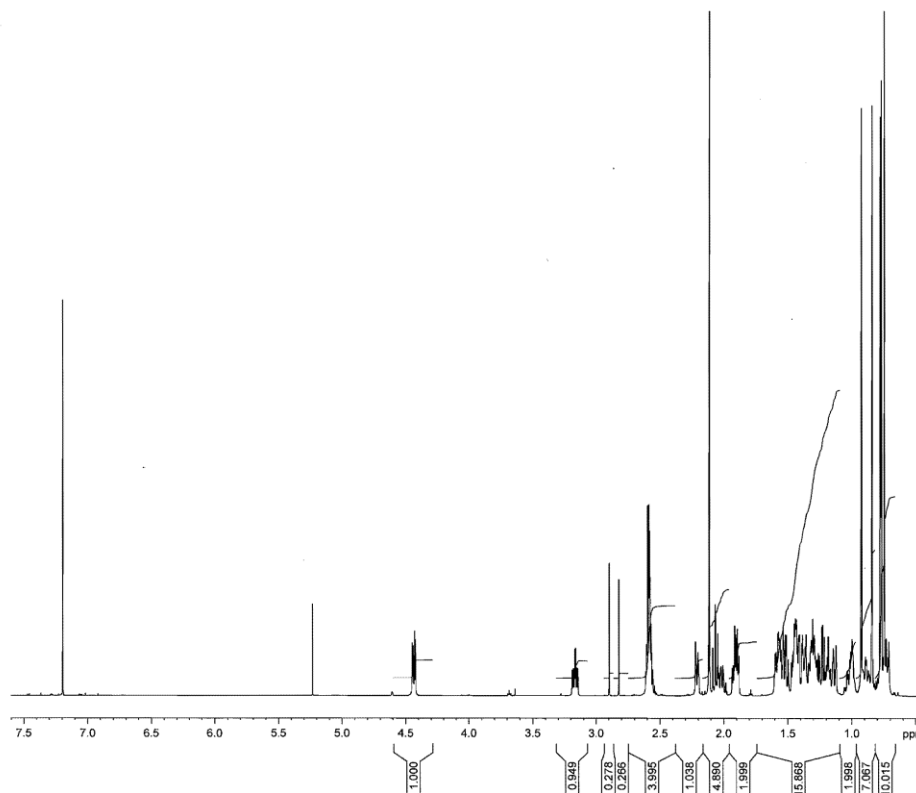
F2 - Processing parameters
SI 131072
SF 150.9028162 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0

55853

Analytical data of 9c



9c: ¹H-NMR (600.13 MHz, CDCl₃): δ [ppm] 0.72 (dd, 1H, *J*=1.8; 11.2 Hz, 5-CH), 0.74 (s, 3H, 23-CH₃), 0.77 (s, 3H, 25-CH₃), 0.78 (s, 3H, 24-CH₃), 0.84 (s, 3H, 26-CH₃), 0.93 (s, 3H, 27-CH₃), 2.07 (t, 1H, *J*=11.2 Hz, 18-CH₂), 2.11 (s, 3H, 29-CH₃), 2.52-2.61 (m, 2H, 2', 3'-CH₂), 3.17 (dt, 1H, *J*=5.1; 11.4; 11.4 Hz, 19-CH), 4.44 (dd, 1H, *J*=4.8; 11.4 Hz, 3-CH); ¹³C-NMR (150.92 MHz, CDCl₃): δ [ppm] 14.64 (q, 27-C), 16.19 (q, 24-C), 16.23 (q, 26-C), 16.55 (q, 25-C), 18.15 (t, 6-C), 20.87 (t, 11-C), 23.53 (t, 2-C), 27.07 (t, 12-C), 27.98 (q, 23-C), 28.25 (t, 21-C), 29.14 (t, 15-C), 29.44 (t, 2'-C), 29.68 (t, 3'-C), 30.10 (q, 29-C), 31.40 (t, 16-C), 34.00 (t, 7-C), 36.68 (t, 22-C), 37.09 (s, 10-C), 37.85 (s, 4-C), 37.85 (d, 13-C), 38.16 (t, 1-C), 40.53 (s, 8-C), 42.16 (s, 14-C), 49.17 (d, 18-C), 50.03 (d, 9-C), 51.20 (s, 19-C), 55.25 (s, 17-C), 56.26 (d, 5-C), 81.41 (d, 3-C), 171.58 (s, 1'-C), 177.98 (s, 4'-C), 181.95 (s, 28-C), 212.42 (s, 20-C). IR (cm⁻¹): 2936, 1686, 1163, 983. MS (ESI, 20 V): *m/z* = 556.9 [M-H]⁺. For C₃₃H₅₀O₇ (558.75) calcd. (%) C (70.94), H (9.02), found (%) C (70.95), H (9.00).



Z.Wimmer MV-21
in CDCl₃, rfp=TMS
3.5.2018

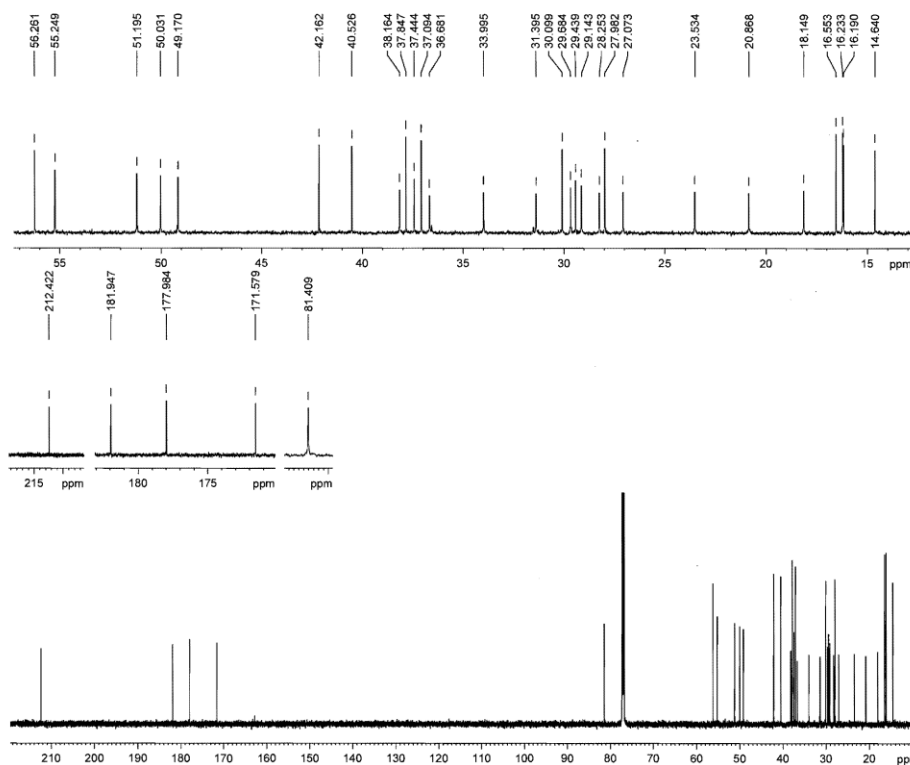
Current Data Parameters
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EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
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Time 9.32
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 43288
SOLVENT CDCl₃
NS 16
DS 0
SWH 5411.255 Hz
FIDRES 0.125006 Hz
AQ 3.9998112 sec
RG 117.5
DW 92.400 usec
DE 20.00 usec
TE 298.1 K
D1 0 sec
TD0 1

===== CHANNEL f1 =====
SFO1 600.1325806 MHz
NUC1 1H
P1 9.00 usec
PLW1 6.00000000 W

F2 - Processing parameters
SI 131072
SF 600.1300537 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00

55852



Z.Wimmer MV-21
in CDCl₃, rfp=TMS
3.5.2018

Current Data Parameters
NAME Wimmer-MV_21
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180503
Time 10.04
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zgpg30
TD 72468
SOLVENT CDCl₃
NS 610
DS 0
SWH 36231.883 Hz
FIDRES 0.500026 Hz
AQ 0.9999480 sec
RG 196.16
DW 13.800 usec
DE 18.00 usec
TE 298.1 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

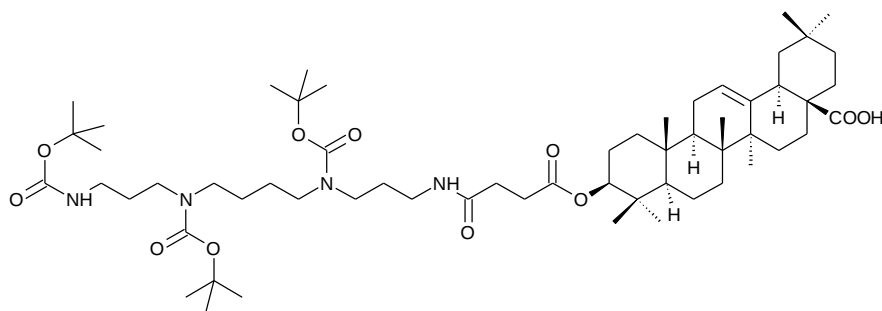
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NUC1 13C
P1 12.00 usec
PLW1 80.00000000 W

===== CHANNEL f2 =====
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG2 waltz16
PCPD2 70.00 usec
PLW2 6.00000000 W
PLW12 0.09918400 W
PLW13 0.04660000 W

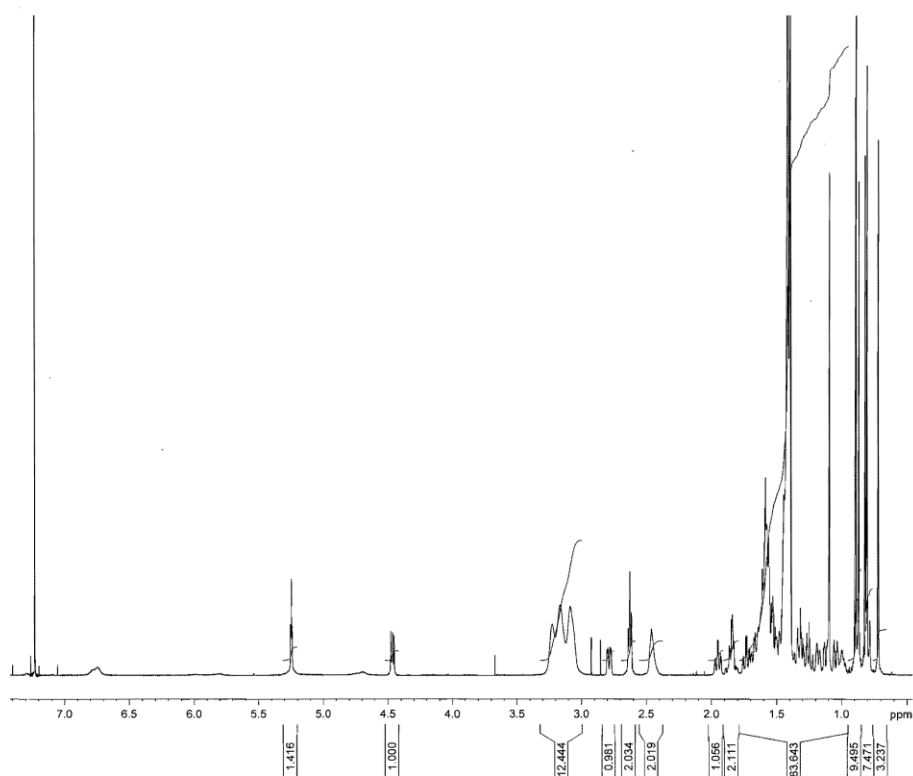
F2 - Processing parameters
SI 131072
SF 150.9209173 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0

54852

Analytical data of 10a



10a: $^1\text{H-NMR}$ (600.13 MHz, CDCl_3): δ [ppm] 0.72 (s, 3H, 26- CH_3), 0.79 (dd, 1H, $J=1.9$; 11.6 Hz, 5-CH), 0.81 (s, 3H, 24- CH_3), 0.82 (s, 3H, 25- CH_3), 0.87 (s, 3H, 29- CH_3), 0.90 (s, 3H, 23- CH_3), 0.90 (s, 3H, 30- CH_3), 1.10 (s, 3H, 27- CH_3), 1.41 (s, Boc), 1.42 (s, Boc), 1.42 (s, Boc), 1.83 (ddd, 1H, $J=3.7$; 7.3; 18.5 Hz, 2- CH_2), 1.87 (ddd, 1H, $J=3.7$; 10.8; 18.5 Hz, 2- CH_2), 1.95 (dt, 2H, $J=4.1$; 13.7; 13.7 Hz, 11- CH_2), 2.42-2.49 (m, 2H, 2'- CH_2), 2.60-2.66 (m, 2H, 3'- CH_2), 2.79 (bdd, 1H, $J=4.3$; 14.0 Hz, 18-CH), 3.03-3.27 (m, 12H, 5', 7', 8', 11', 12', 14'- CH_2), 4.47 (dd, 1H, $J=6.8$; 8.9 Hz, 3-CH), 5.25 (t, 1H, $J=3.8$; 3.8 Hz, 12-CH); $^{13}\text{C-NMR}$ (150.92 MHz, CDCl_3): δ [ppm] 15.36 (q, 24-C), 16.71 (q, 25-C), 17.06 (q, 26-C), 18.16 (t, 6-C), 22.95 (t, 11-C), 23.38 (t, 2'-C), 23.48 (t, 2-C), 23.56 (q, 30-C), 25.45 (t, 10'-C), 25.86 (q, 27-C), 26.08 (t, 9'-C), 27.33 (t, 13'-C), 27.65 (t, 16-C), 28.05 (t, 15-C), 28.45 (q, 23-C), 28.92 (t, 6'-C), 30.03 (t, 3'-C), 30.66 (s, 20-C), 32.39 (t, 22-C), 32.54 (t, 7-C), 33.04 (q, 29-C), 33.79 (t, 21-C), 35.71 (t, 14'-C), 36.94 (t, 1-C), 37.73 (s, 10-C), 37.73 (t, 5'-C), 38.04 (s, 4-C), 39.26 (s, 8-C), 41.02 (d, 18-C), 41.61 (s, 14-C), 43.31 (t, 11'-C), 43.71 (t, 12'-C), 44.18 (t, 8'-C), 45.84 (t, 19-C), 46.56 (s, 17-C), 46.73 (t, 7'-C), 47.51 (d, 9-C), 55.29 (d, 5-C), 81.05 (d, 3-C), 122.51 (d, 12-C), 143.60 (s, 13-C), 171.37 (s, 1'-C), 172.65 (s, 4'-C), 182.19 (s, 28-C). IR (cm^{-1}): 2932, 1684, 1418, 1364, 1248, 1160, 751. MS (ESI, 20 V): m/z = 1039.8 $[\text{M-H}]^+$, 1041.7 $[\text{M+H}]^+$, 1058.7 $[\text{M+NH}_4]^+$, 1063.7 $[\text{M+Na}]^+$. For $\text{C}_{59}\text{H}_{100}\text{N}_4\text{O}_{11}$ (1041.45) calcd. (%) C (68.04), H (9.68), N (5.38), found (%) C (68.05), H (9.66), N (5.40).



Z.Wimmer MV-22
in CDCl₃, rfp=TMS
25.4.2018

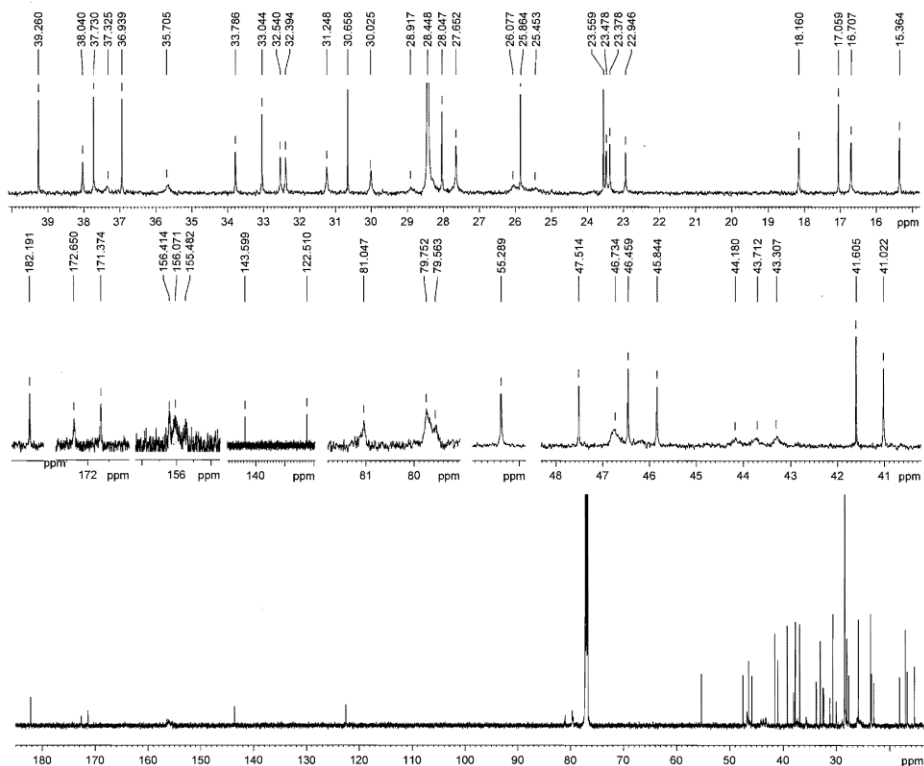
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EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
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INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 43288
SOLVENT CDCl₃
NS 16
DS 0
SWH 5411.255 Hz
FIDRES 0.125006 Hz
AQ 3.9998112 sec
RG 117.6
DW 92.400 usec
DE 20.00 usec
TE 298.1 K
D1 0 sec
TD0 1

===== CHANNEL f1 =====
SFO1 600.1325806 MHz
NUC1 1H
P1 9.00 usec
PLW1 6.00000000 W

F2 - Processing parameters
SI 131072
SF 600.1300319 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00

55836



Z.Wimmer MV-22
in CDCl₃, rfp=TMS
25.4.2018

Current Data Parameters
NAME Wimmer-MV_22
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180425
Time 15.12
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zgpg30
TD 59520
SOLVENT CDCl₃
NS 3625
DS 0
SWH 29761.904 Hz
FIDRES 0.500032 Hz
AQ 0.9999360 sec
RG 196.18
DW 16.800 usec
DE 18.00 usec
TE 298.2 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

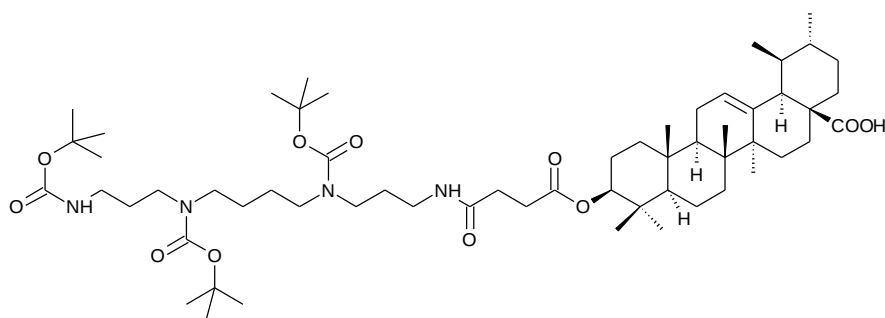
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NUC1 13C
P1 12.00 usec
PLW1 80.00000000 W

===== CHANNEL f2 =====
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG2 waltz16
PCPD2 70.00 usec
PLW2 6.00000000 W
PLW12 0.09918400 W
PLW13 0.04860000 W

F2 - Processing parameters
SI 131072
SF 150.9028121 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0

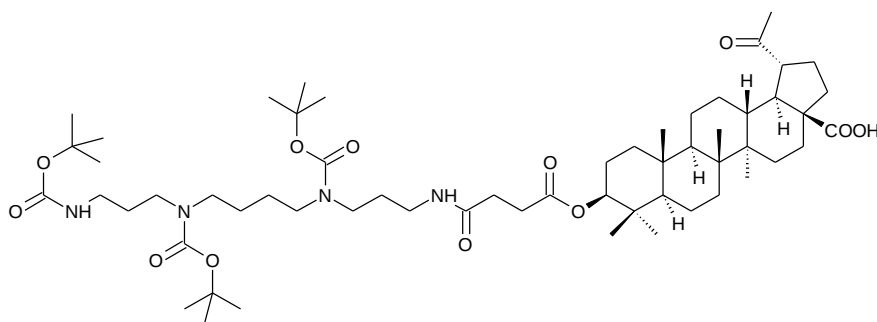
55836

Analytical data of 10b

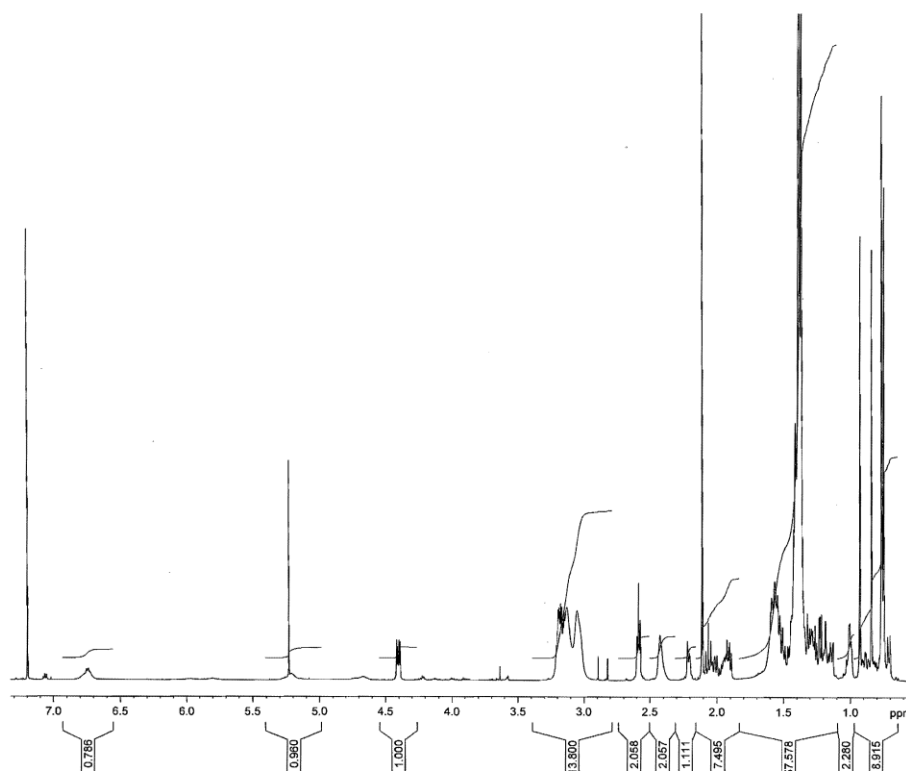


10b: $^1\text{H-NMR}$ (600.13 MHz, CDCl_3): δ [ppm] 0.75 (s, 3H, 26- CH_3), 0.79 (dd, 1H, $J=1.7$; 11.7 Hz, 5-CH), 0.82 (s, 3H, 24- CH_3), 0.83 (s, 3H, 25- CH_3), 0.84 (d, 3H, $J=6.4$ Hz, 29- CH_3), 0.92 (d, 3H, $J=6.4$ Hz, 30- CH_3), 0.92 (s, 3H, 23- CH_3), 1.05 (s, 3H, 27- CH_3), 1.41 (s, Boc), 1.53 (s, 2xBoc), 1.70 (dt, 1H, $J=3.5$; 3.5; 13.6 Hz, 16- CH_2), 1.83 (dt, 2H, $J=4.7$; 13.8; 13.8 Hz, 15- CH_2), 1.98 (dt, 1H, $J=4.7$; 13.4; 13.4 Hz, 16- CH_2), 2.16 (dd, 1H, $J=1.6$; 11.5 Hz, 18-CH), 2.43-2.51 (m, 2H, 2'- CH_2), 2.62-2.67 (m, 2H, 3'- CH_2), 3.05-3.30 (m, 12H, 5'-, 7'-, 8'-, 11'-, 12'-, 14'- CH_2), 4.48 (dd, 1H, $J=5.8$; 10.9 Hz, 3-CH), 5.21 (t, 1H, $J=3.8$ Hz, 12-CH); $^{13}\text{C-NMR}$ (150.92 MHz, CDCl_3): δ [ppm] 15.51 (q, 24-C), 16.75 (q, 25-C), 17.00 (q, 26-C), 17.03 (q, 29-C), 18.14 (t, 6-C), 21.16 (q, 30-C), 23.26 (t, 2-C), 23.51 (t, 11-C), 23.51 (q, 27-C), 24.69 (t, 16-C), 25.84 (t, 10'-C), 26.03 (t, 6'-C), 27.67 (t, 9'-C), 27.97 (t, 15-C), 28.08 (q, 23-C), 28.44 (t, 13'-C), 30.00 (t, 2'-C), 30.59 (t, 21-C), 31.24 (t, 3'-C), 32.83 (t, 7-C), 35.69 (t, 14'-C), 36.68 (s, 10-C), 36.87 (t, 22-C), 37.36 (t, 5'-C), 37.72 (s, 4-C), 38.22 (t, 1-C), 38.80 (t, 8-C), 39.01 (d, 20-C), 39.47 (d, 19-C), 41.92 (s, 14-C), 43.31 (t, 11'-C), 43.75 (t, 8'-C), 44.17 (t, 7'-C), 46.76 (t, 12'-C), 47.42 (d, 9-C), 47.88 (s, 17-C), 52.58 (d, 18-C), 55.28 (d, 6-C), 81.06 (t, 3-C), 125.68 (d, 12-C), 137.96 (s, 13-C), 171.38 (s, 1'-C), 172.64 (s, 4'-C), 182.38 (s, 28-C), Boc: 28.44 (q), 79.57 (s), 79.73 (t), 155.43 (s), 156.10 (s), 156.40 (s). IR (cm^{-1}): 3326, 2927, 1685, 1454, 1417, 1364, 1152, 879, 771. MS (ESI, 20 V): m/z = 1039.8 $[\text{M-H}]^+$, 1041.9 $[\text{M+H}]^+$, 1063.6 $[\text{M+Na}]^+$. For $\text{C}_{59}\text{H}_{100}\text{N}_4\text{O}_{11}$ (1041.45) calcd. (%) C (68.04), H (9.68), N (5.38), found (%) C (68.06), H (9.67), N (5.35).

Analytical data of 10c



10c: $^1\text{H-NMR}$ (600.13 MHz, CDCl_3): δ [ppm] 0.71 (dd, 1H, $J=1.8$; 11.2 Hz, 5-CH), 0.75 (s, 3H, 26- CH_3), 0.76 (s, 3H, 24- CH_3), 0.76 (s, 3H, 25- CH_3), 0.84 (s, 3H, 23- CH_3), 0.93 (s, 3H, 27- CH_3), 2.06 (t, 1H, $J=11.4$ Hz, 18-CH), 2.11 (s, 3H, 29- CH_3), 2.40-2.46 (m, 2H, 2'- CH_2), 2.55-2.63 (m, 2H, 3'- CH_2), 3.00-3.23 (m, 12H, 5'-, 7'-, 8'-, 11'-, 12'-, 14'- CH_2), 3.18 (dt, 1H, $J= 5.0$; 11.5; 11.5 Hz, 19-CH), 4.40 (dd, 1H, $J=5.1$; 11.2 Hz); $^{13}\text{C-NMR}$ (150.92 MHz, CDCl_3): δ [ppm] 14.67 (q, 27-C), 15.91 (q, 26-C), 16.14 (q, 24-C), 16.51 (q, 25-C), 18.11 (t, 6-C), 20.85 (t, 11-C), 23.61 (t, 2-C), 25.78 (t, 10'-C), 26.04 (t, 9'-C), 27.17 (t, 12-C), 27.66 (t, 13'-C), 27.94 (t, 21-C), 28.23 (q, 23-C), 28.91 (t, 6'-C), 29.68 (t, 15-C), 30.00 (t, 2'-C), 30.09 (q, 29-C), 31.24 (t, 3'-C), 31.44 (t, 16-C), 34.10 (t, 7-C), 35.70 (t, 14'-C), 36.68 (t, 22-C), 37.08 (s, 10-C), 37.40 (s, 4-C), 37.40 (t, 5'-C), 37.83 (d, 13-C), 38.29 (t, 1-C), 40.58 (s, 8-C), 42.21 (s, 14-C), 43.30 (t, 11'-C), 43.76 (t, 12'-C), 44.18 (t, 8'-C), 46.76 (t, 7'-C), 49.17 (d, 18-C), 50.23 (d, 9-C), 51.14 (d, 19-C), 55.34 (d, 5-C), 56.11 (s, 17-C), 80.99 (d, 3-C), 171.47 (s, 1'-C), 172.60 (s, 4'-C), 180.12 (s, 28-C), 212.18 (s, 20-C). IR (cm^{-1}): 2938, 1686, 1418, 1364, 1247, 1160, 978, 866, 771, 735. MS (ESI, 20 V): $m/z = 1041.6$ $[\text{M-H}]^+$, 1043.7 $[\text{M+H}]^+$, 1065.7 $[\text{M+Na}]^+$. For $\text{C}_{58}\text{H}_{98}\text{N}_4\text{O}_{12}$ (1043.42) calcd. (%) C (66.76), H (9.47), N (5.37), found (%) C (66.74), H (9.48), N (5.35).



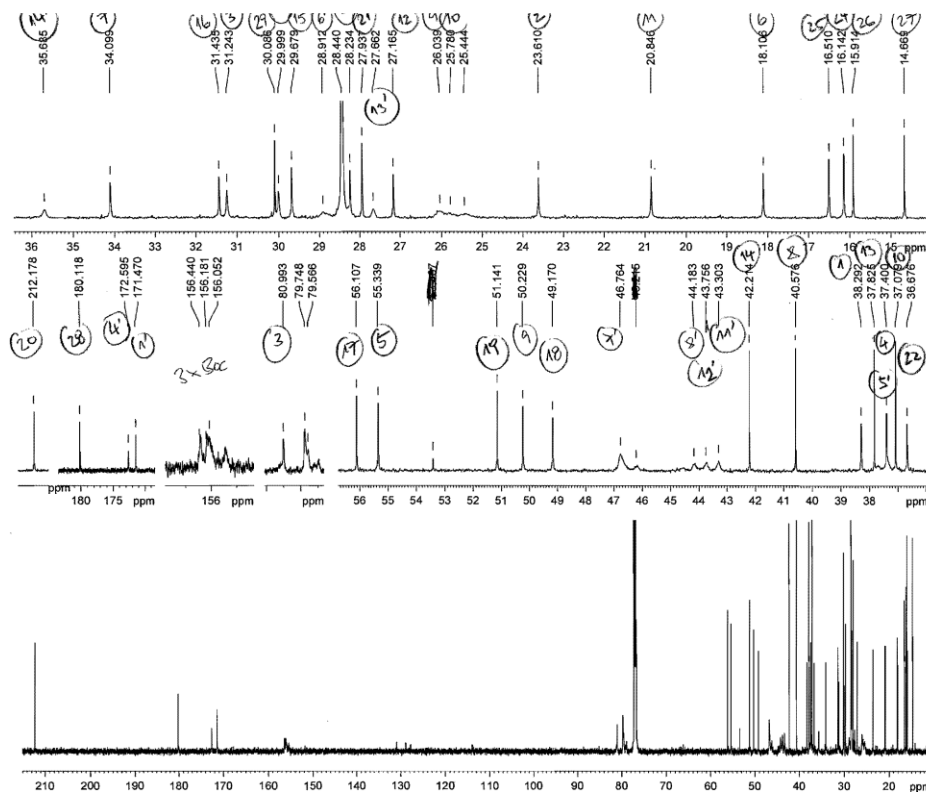
Z.Wimmer MV-24
in CDCl₃, rfp=TMS
9.5.2018

Current Data Parameters
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EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180509
Time 9.52
INSTRUM spect
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PULPROG zg30
TD 43288
SOLVENT CDCl₃
NS 16
DS 0
SWH 5411.255 Hz
FIDRES 0.125006 Hz
AQ 3.9998112 sec
RG 132.82
DW 92.400 usec
DE 20.00 usec
TE 298.1 K
D1 0 sec
TD0 1

===== CHANNEL f1 =====
SFO1 600.1325806 MHz
NUC1 1H
P1 9.00 usec
PLW1 6.00000000 W
F2 - Processing parameters
SI 131072
SF 600.1300337 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00

55882



Z.Wimmer MV-24
in CDCl₃, rfp=TMS
9.5.2018

Current Data Parameters
NAME Wimmer-MV_24
EXPNO 1
PROCNO 1

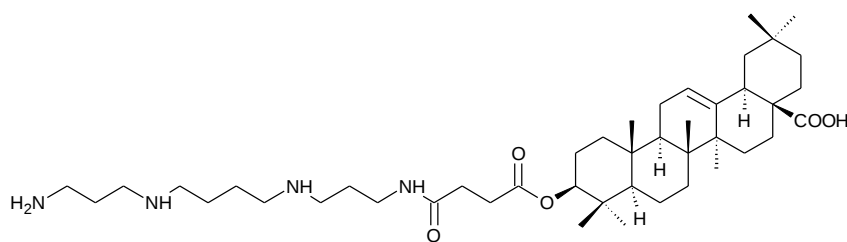
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PULPROG zgpg30
TD 72460
SOLVENT CDCl₃
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DS 0
SWH 36231.883 Hz
FIDRES 0.500026 Hz
AQ 0.9999480 sec
RG 196.16
DW 13.800 usec
DE 18.00 usec
TE 298.2 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 150.9209173 MHz
NUC1 13C
P1 12.00 usec
PLW1 80.00000000 W
===== CHANNEL f2 =====
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG2 waltz16
PCPD2 70.00 usec
PLW2 6.00000000 W
PLW12 0.09918400 W
PLW13 0.04860000 W

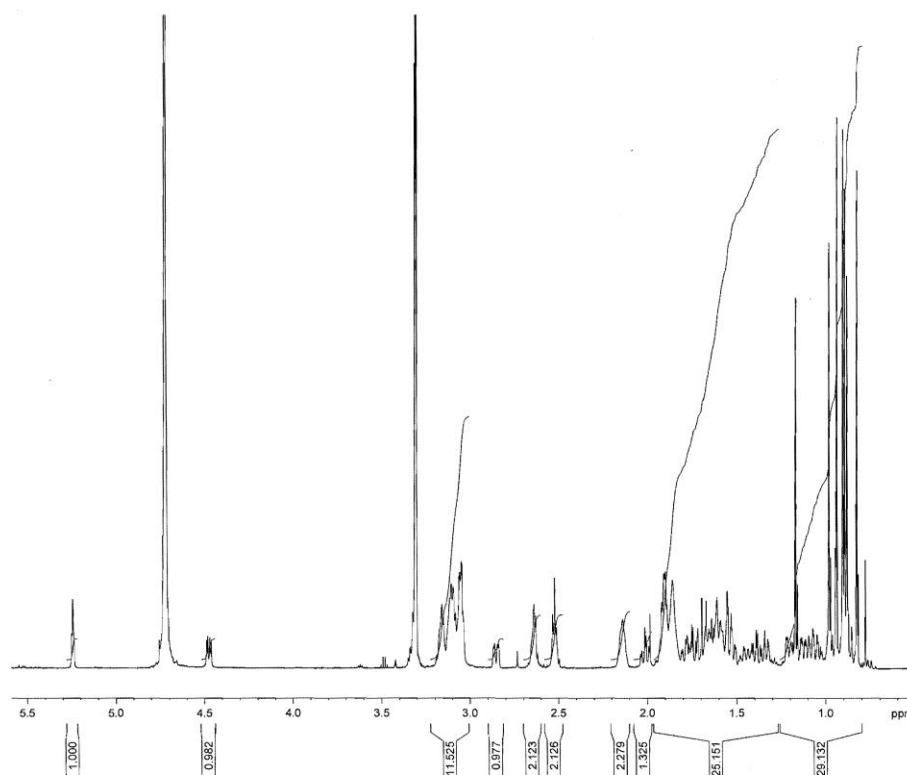
F2 - Processing parameters
SI 131072
SF 150.9028132 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0

55832

Analytical data of 11a



11a: ^1H -NMR (600.13 MHz, CD_3OD): δ [ppm] 0.83 (s, 3H, 25- CH_3), 0.87 (dd, 1H, $J=1.7$; 11.5 Hz, 5-CH), 0.88 (s, 3H, 23- CH_3), 0.90 (s, 3H, 26- CH_3), 0.91 (s, 3H, 29- CH_3), 0.94 (s, 3H, 30- CH_3), 0.98 (s, 3H, 24- CH_3), 1.13 (ddd, 2H, $J=2.4$; 4.7; 13.6 Hz, 18- CH_2), 1.17 (s, 3H, 27- CH_3), 2.02 (bdt, 2H, $J=3.8$; 13.8; 13.8 Hz, 11- CH_2), 2.51-2.55 (m, 2H, 2'- CH_2), 2.61-2.66 (m, 2H, 3'- CH_2), 2.86 (bdd, 1H, $J=4.3$; 13.8 Hz, 18-CH), 3.03-3.18 (m, 12H, 5', 7', 8', 11', 12', 14'- CH_2), 4.48 (dd, 1H, $J=4.8$; 11.5 Hz, 3-CH), 5.24 (t, 1H, $J=3.6$ Hz, 12-CH); ^{13}C -NMR (150.92 MHz, CD_3OD): δ [ppm] 15.91 (q, 24-C), 17.23 (q, 25-C), 17.73 (q, 26-C), 19.36 (t, 6-C), 23.97 (t, 11-C), 24.08 (q, 30-C), 24.30 (t, 2-C), 24.40 (t, 10'-C), 24.53 (t, 9'-C), 24.61 (t, 13'-C), 25.41 (t, 6'-C), 26.40 (q, 27-C), 27.72 (t, 16-C), 28.72 (q, 23-C), 28.86 (t, 15-C), 30.69 (t, 2'-C), 31.41 (t, 3'-C), 31.59 (s, 20-C), 33.53 (q, 29-C), 33.81 (t, 22-C), 33.95 (t, 7-C), 34.92 (t, 21-C), 36.86 (t, 14'-C), 38.04 (s, 10-C), 38.16 (t, 5'-C), 38.86 (s, 4-C), 39.38 (t, 1-C), 40.62 (s, 8-C), 42.77 (d, 18-C), 42.95 (s, 14-C), 46.07 (t, 11'-C), 46.40 (t, 12'-C), 47.30 (t, 19-C), 47.65 (s, 17-C), 48.22 (t, 8'-C), 48.41 (t, 7'-C), 49.05 (d, 9-C), 56.85 (d, 5-C), 82.75 (d, 3-C), 123.51 (d, 12-C), 145.28 (s, 13-C), 174.23 (s, 1'-C), 175.60 (s, 4'-C), 181.72 (s, 28-C). IR (cm^{-1}): 3362, 2941, 1691, 1638, 1559, 1459, 1364, 1240, 1176, 993, 755. MS (ESI, 25 V): m/z = 739.4 $[\text{M}-\text{H}]^+$, 741.4 $[\text{M}+\text{H}]^+$. For $\text{C}_{44}\text{H}_{76}\text{N}_4\text{O}_5$ (741.10) calcd. (%) C (71.31), H (10.34), N (7.56), found (%) C (71.33), H (10.33), N (7.58).



Z.Wimmer MV-25
in CD3OD, rfp=3.31
2.5.2018

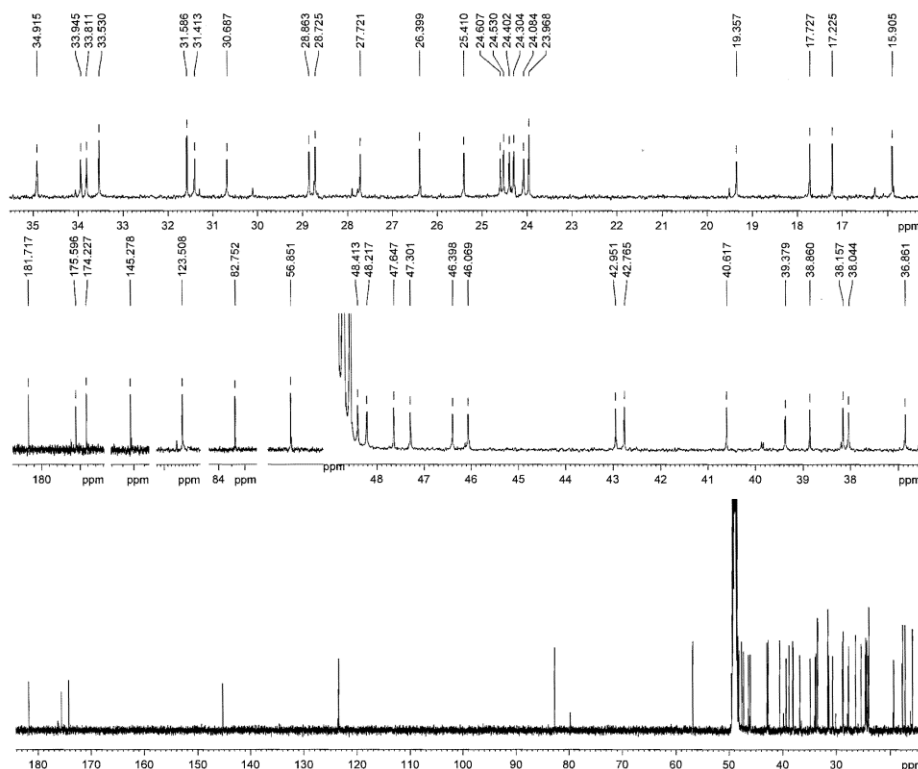
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EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
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Time 7.39
INSTRUM spect
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PULPROG zg30
TD 43288
SOLVENT MeOD
NS 16
DS 0
SWH 5411.255 Hz
FIDRES 0.125006 Hz
AQ 3.9998112 sec
RG 117.6
DW 92.400 usec
DE 20.00 usec
TE 313.2 K
D1 0 sec
TD0 1

===== CHANNEL f1 =====
SFO1 600.1325806 MHz
NUC1 1H
P1 9.00 usec
PLW1 6.00000000 W

F2 - Processing parameters
SI 131072
SF 600.1300111 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00

55851



Z.Wimmer MV-25
in CD3OD, rfp=3.31
2.5.2018

Current Data Parameters
NAME Wimmer-MV_25
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180502
Time 8.16
INSTRUM spect
PROBHD 5 mm CPTCl 1H-
PULPROG zgpg30
TD 59520
SOLVENT MeOD
NS 1063
DS 0
SWH 29761.904 Hz
FIDRES 0.500032 Hz
AQ 0.9999360 sec
RG 196.16
DW 16.800 usec
DE 18.00 usec
TE 313.1 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

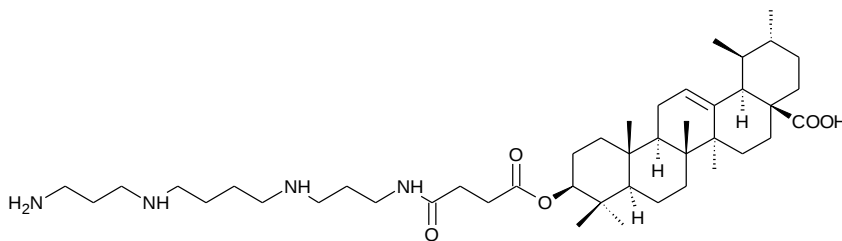
===== CHANNEL f1 =====
SFO1 150.9178993 MHz
NUC1 13C
P1 12.00 usec
PLW1 80.00000000 W

===== CHANNEL f2 =====
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG2 waitz16
PCPD2 70.00 usec
PLW2 6.00000000 W
PLW12 0.09918400 W
PLW13 0.04860000 W

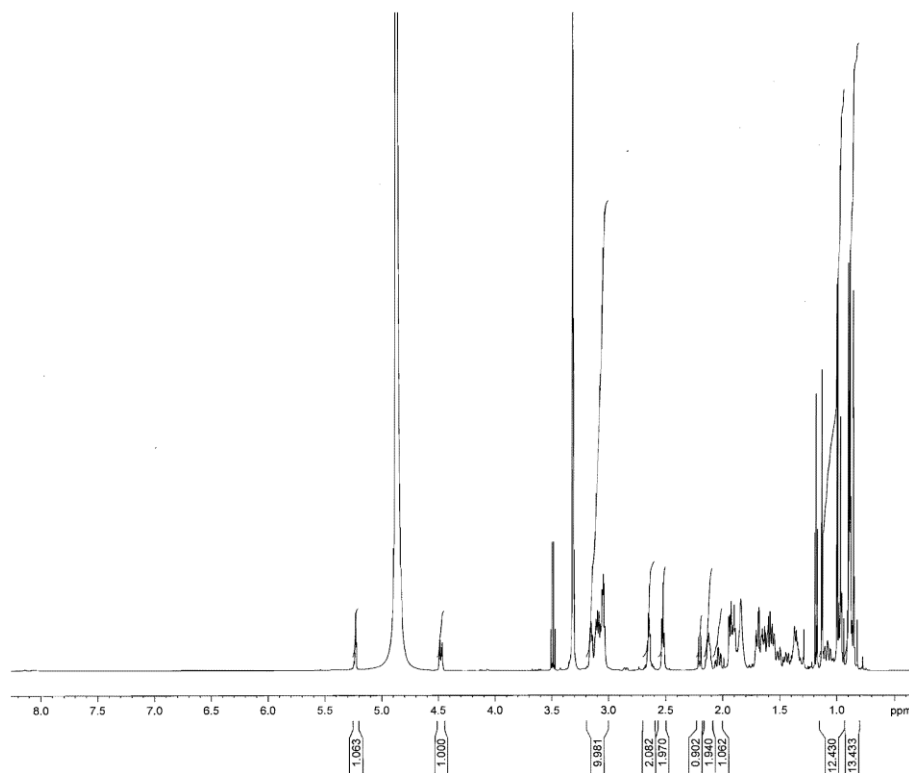
F2 - Processing parameters
SI 131072
SF 150.9025976 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0

55851

Analytical data of 11b



11b: $^1\text{H-NMR}$ (600.13 MHz, CD_3OD): δ [ppm] 0.86 (dd, 1H, $J=1.8$; 11.6 Hz, 5-CH), 0.86 (s, 3H, 26- CH_3), 0.88 (s, 3H, 24- CH_3), 0.89 (d, 3H, $J=6.5$ Hz, 29- CH_3), 0.90 (d, 3H, $J=6.5$ Hz, 30- CH_3), 0.97 (s, 3H, 23- CH_3), 1.00 (s, 3H, 25- CH_3), 1.13 (s, 3H, 27- CH_3), 2.04 (dt, 2H, $J=4.4$; 13.6; 13.6 Hz, 16- CH_2), 2.20 (dd, 1H, $J=1.7$; 11.3 Hz, 18-CH), 2.51-2.55 (m, 2H, 2'- CH_2), 2.60-2.68 (m, 2H, 3'- CH_2), 3.02-3.18 (m, 18H, spermine CH_2) 3.30-3.33 (m, 2H, 5'- CH_2), 4.48 (dd, 1H, $J=4.6$; 11.5 Hz, 3-CH), 5.23 (t, 1H, $J=3.7$ Hz, 12-C); $^{13}\text{C-NMR}$ (150.92 MHz, CD_3OD): δ [ppm] 16.03 (q, 25-C), 17.29 (q, 24-C), 17.65 (q, 29-C), 17.76 (q, 26-C), 19.30 (t, 6-C), 21.56 (q, 30-C), 24.10 (q, 27-C), 24.30 (t, 11-C), 24.35 (t, 9'-C), 24.40 (t, 10'-C), 24.60 (t, 13'-C), 25.28 (t, 6'-C), 25.41 (t, 16-C), 27.72 (t, 2-C), 28.72 (q, 23-C), 29.19 (t, 15-C), 30.60 (t, 2'-C), 31.31 (t, 3'-C), 31.73 (t, 21-C), 34.19 (t, 7-C), 36.78 (t, 5'-C), 37.93 (t, 14'-C), 38.05 (s, 10-C), 38.08 (t, 22-C), 38.83 (s, 8-C), 39.47 (t, 1-C), 40.39 (s, 20-C), 40.40 (t, 19-C), 40.78 (s, 4-C), 43.24 (s, 14-C), 46.00 (t, 11'-C), 46.00 (s, 17-C), 46.29 (t, 8'-C), 48.16 (t, 12'-C), 48.35 (t, 7'-C), 49.21 (d, 9-C), 54.32 (d, 18-C), 56.77 (d, 5-C), 82.67 (d, 3-C), 126.72 (d, 12-C), 139.69 (s, 13-C), 174.26 (s, 1'-C), 175.64 (s, 4'-C), 181.59 (s, 28-C); IR (cm^{-1}): 3362, 2925, 1691, 1646, 1558, 1456, 1377, 1238, 1171, 966, 660; MS (ESI, 20 V): m/z = 739.1 $[\text{M-H}]^+$, 741.4 $[\text{M+H}]^+$, 775.4 $[\text{M+Cl}]^-$. For $\text{C}_{44}\text{H}_{76}\text{N}_4\text{O}_5$ (741.10) calcd. (%) C (71.31), H (10.34), N (7.56), found (%) C (71.32), H (10.36), N (7.55).



Z.Wimmer MV-26
in CD3OD, rfp=3.31
4.5.2018

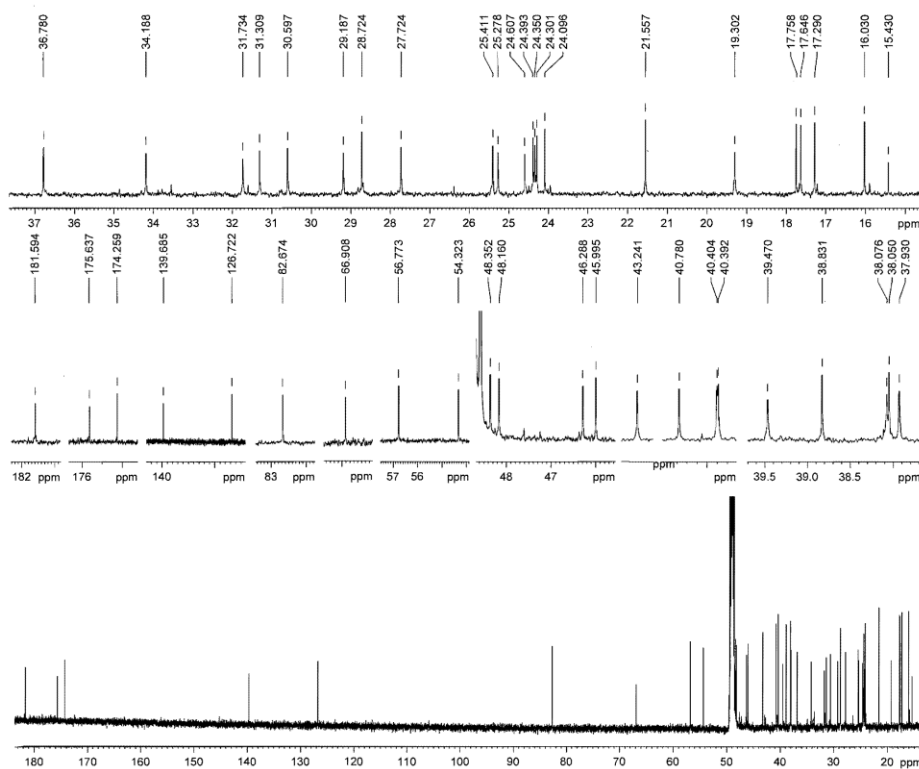
Current Data Parameters
NAME Wimmer-MV_26
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180504
Time 7.53
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 43288
SOLVENT MeOD
NS 16
DS 0
SWH 5411.255 Hz
FIDRES 0.125006 Hz
AQ 3.9998112 sec
RG 195.18
DW 92.400 usec
DE 20.00 usec
TE 298.2 K
D1 0 sec
TD0 1

===== CHANNEL f1 =====
SFO1 600.1325806 MHz
NUC1 1H
P1 9.00 usec
PLW1 6.00000000 W

F2 - Processing parameters
SI 131072
SF 600.1300109 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00

55873



Z.Wimmer MV-26
in CD3OD, rfp=3.31
4.5.2018

Current Data Parameters
NAME Wimmer-MV_26
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180504
Time 9.58
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zgpg30
TD 59520
SOLVENT MeOD
NS 2832
DS 0
SWH 29761.904 Hz
FIDRES 0.500032 Hz
AQ 0.9998360 sec
RG 195.18
DW 16.800 usec
DE 18.00 usec
TE 298.2 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

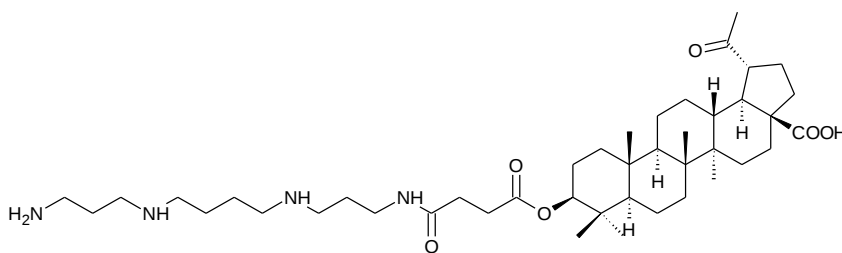
===== CHANNEL f1 =====
SFO1 150.9178993 MHz
NUC1 13C
P1 12.00 usec
PLW1 80.00000000 W

===== CHANNEL f2 =====
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG2 waltz16
PCPD2 70.00 usec
PLW2 6.00000000 W
PLW12 0.09918400 W
PLW13 0.04860000 W

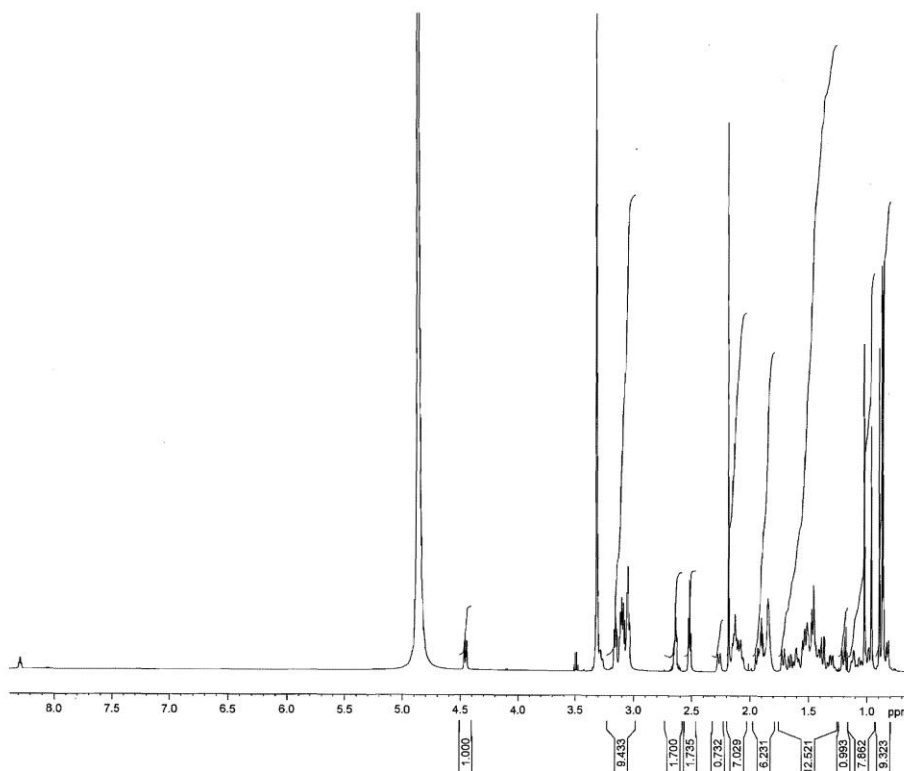
F2 - Processing parameters
SI 131072
SF 150.9025983 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0

55873

Analytical data of 11c



11c: $^1\text{H-NMR}$ (600.13 MHz, CD_3OD): δ [ppm] 0.82 (dd, 1H, $J=2.1$; 11.1 Hz, 5-CH), 0.86 (s, 3H, 23- CH_3), 0.87 (s, 3H, 25- CH_3), 0.89 (s, 3H, 26- CH_3), 0.96 (s, 3H, 24- CH_3), 1.02 (s, 3H, 27- CH_3), 1.72 (dt, 2H, $J=3.4$; 3.4; 13.4 Hz, 1- CH_2), 2.18 (s, 3H, 29- CH_3), 2.50-2.54 (m, 2H, 2'- CH_2), 2.60-2.67 (m, 2H, 3'- CH_2), 3.02-3.18 (m, 12H, 5'-, 7'-, 8'-, 11'-, 12'-, 14'- CH_2), 3.28 (bd t, 1H, $J=3.5$; 11.4; 11.4 Hz), 4.46 (dd, 1H, $J=4.8$; 11.6 Hz, 3-CH); $^{13}\text{C-NMR}$ (150.92 MHz, CD_3OD): δ [ppm] 15.07 (q, 27-C), 16.52 (q, 24-C), 16.70 (q, 26-C), 17.01 (q, 25-C), 19.23 (t, 6-C), 22.15 (t, 11-C), 24.29 (t, 2-C), 24.38 (t, 10'-C), 24.71 (t, 9'-C), 25.41 (t, 13'-C), 27.72 (t, 12-C), 28.39 (t, 21-C), 28.55 (t, 6'-C), 28.56 (q, 23-C), 29.37 (t, 15-C), 29.83 (q, 29-C), 30.58 (t, 2'-C), 30.81 (t, 16-C), 31.30 (3'-C), 32.70 (t, 7-C), 35.36 (t, 14'-C), 36.77 (t, 22-C), 37.92 (t, 5'-C), 38.30 (s, 10-C), 38.70 (d, 13-C), 38.94 (s, 4-C), 39.57 (t, 1-C), 41.85 (s, 8-C), 43.38 (s, 14-C), 45.98 (t, 11'-C), 46.28 (t, 12'-C), 48.15 (t, 8'-C), 48.33 (t, 7'-C), 50.51 (d, 18-C), 51.82 (d, 9-C), 52.80 (d, 19-C), 56.88 (d, 5-C), 56.88 (s, 17-C), 82.64 (d, 3-C), 174.27 (s, 1'-C), 175.63 (s, 4'-C), 179.54 (s, 28-C), 215.49 (s, 20-C). IR (cm^{-1}): 3348, 2941, 1701, 1647, 1541, 1457, 1362, 1245, 1170, 978, 743. MS (ESI, 20 V): m/z = 740.9 $[\text{M-H}]^+$, 743.3 $[\text{M+H}]^+$, 777.5 $[\text{M+Cl}]^-$. For $\text{C}_{43}\text{H}_{74}\text{N}_4\text{O}_6$ (743.07) calcd. (%) C (69.50), H (10.04), N (7.54), found (%) C (69.47), H (10.03), N (7.56).



Z.Wimmer MV-27
in CD3OD, rfp=3.31
11.5.2018

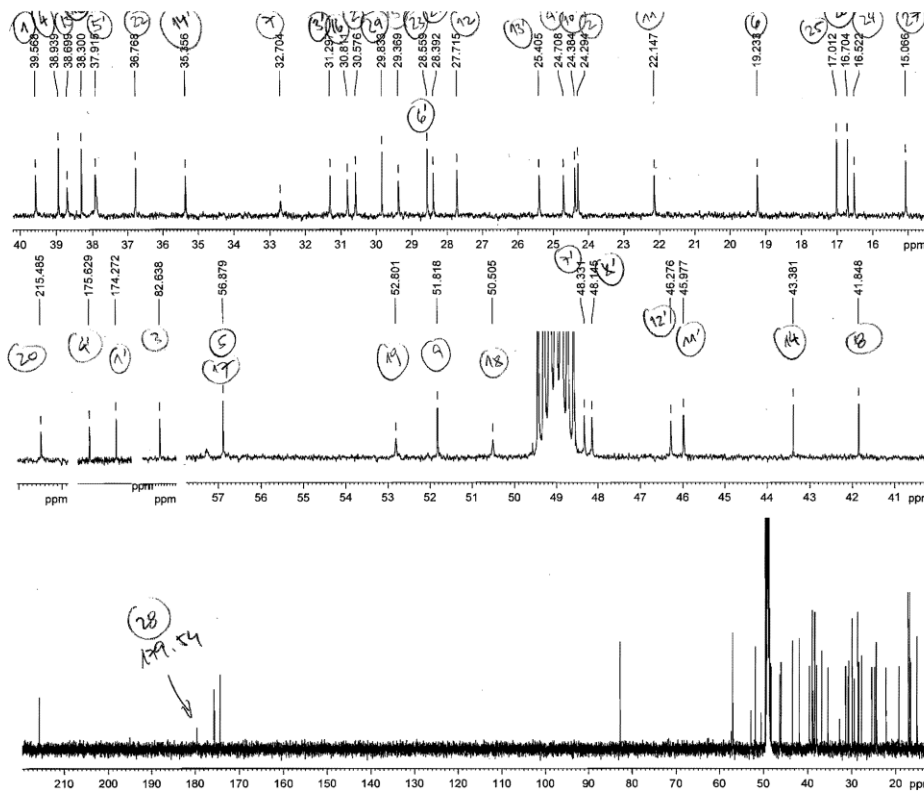
Current Data Parameters
NAME Wimmer-MV_27
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180511
Time 7.44
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 43288
SOLVENT MeOD
NS 8
DS 0
SWH 5411.255 Hz
FIDRES 0.125006 Hz
AQ 3.9998112 sec
RG 68.99
DW 92.400 usec
DE 20.00 usec
TE 298.2 K
D1 0 sec
TD0 1

===== CHANNEL f1 =====
SFO1 600.132805 MHz
NUC1 1H
P1 9.00 usec
PLW1 6.00000000 W

F2 - Processing parameters
SI 131072
SF 600.1300114 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00

55883



Z.Wimmer MV-27
in CD3OD, rfp=3.31
11.5.2018

Current Data Parameters
NAME Wimmer-MV_27
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180511
Time 8.58
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zgpg30
TD 72460
SOLVENT MeOD
NS 512
DS 0
SWH 36231.883 Hz
FIDRES 0.500026 Hz
AQ 0.9999480 sec
RG 198.18
DW 13.800 usec
DE 18.00 usec
TE 298.1 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

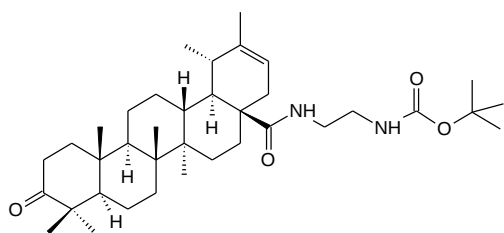
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SFO1 150.9209173 MHz
NUC1 13C
P1 12.00 usec
PLW1 80.00000000 W

===== CHANNEL f2 =====
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG2 waltz16
LPCPD2 70.00 usec
PLW2 6.00000000 W
PLW12 0.09918400 W
PLW13 0.04860000 W

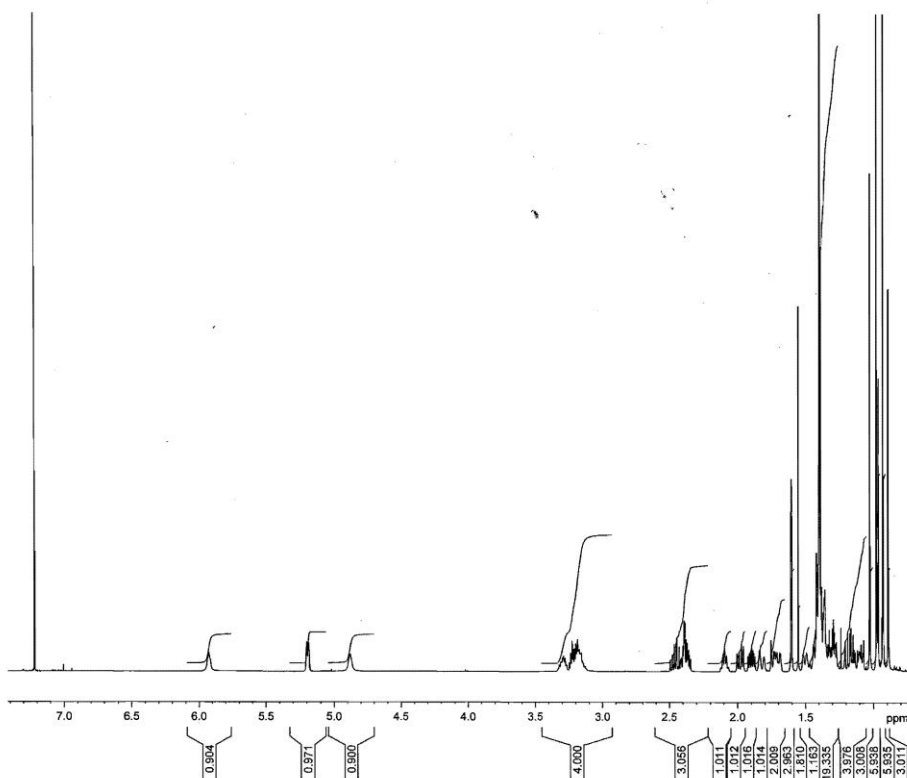
F2 - Processing parameters
SI 131072
SF 150.9025987 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0

55883

Analytical data of 12b



12b: $^1\text{H-NMR}$ (600.13 MHz, CDCl_3): δ [ppm] 0.89 (d, 3H, $J=0.7$ Hz, 25- CH_3), 0.93 (s, 3H, 26- CH_3), 0.93 (s, 3H, 27- CH_3), 0.97 (d, 3H, $J=6.5$ Hz, 29- CH_3), 0.98 (s, 3H, 24- CH_3), 1.03 (s, 3H, 23- CH_3), 1.35-1.39 (m, 1H, 1- CH_2), 1.40 (s, 3H, 5'- CH_3), 1.61 (t, 3H, $J=1.9$ Hz, 30- CH_3), 1.71 (bdq, 2H, $J=3.2$; 3.2; 3.2; 12.6 Hz, 15- CH_2), 1.82 (bdq, 1H, $J=2.1$; 2.1; 2.1; 15.9 Hz, 22- CH_2), 1.90 (ddd, 1H, $J=4.4$; 7.6; 13.2 Hz, 1- CH_2), 1.98 (dd, 1H, $J=7.2$; 15.9 Hz, 22- CH_2), 2.06-2.13 (m, 1H, 19- CH_2), 2.38 (ddd, 1H, $J=4.4$; 7.6; 15.7 Hz, 2- CH_2), 2.40 (dt, 1H, $J=4.4$; 11.5; 11.5 Hz, 13-CH), 2.46 (ddd, 1H, $J=7.6$; 9.7; 15.7 Hz, 2- CH_2), 3.12-3.34 (m, 4H, 1', 2'- CH_2), 5.19 (m, 1H, $J=1.5$; 1.5; 1.5; 1.5; 7.2 Hz, 21-CH); $^{13}\text{C-NMR}$ (125.74 MHz, CDCl_3): δ [ppm] 14.83 (q, 27-C), 15.91 (q, 26-C), 16.12 (q, 25-C), 19.61 (t, 6-C), 20.99 (q, 24-C), 21.80 (t, 11-C), 22.01 (q, 30-C), 23.56 (q, 29-C), 26.63 (q, 23-C), 27.30 (t, 15-C), 28.39 (q, 5'-C), 28.77 (t, 12-C), 33.50 (t, 16-C), 34.12 (t, 7-C), 34.24 (t, 2-C), 36.84 (s, 10-C), 37.38 (d, 19-C), 39.07 (t, 22-C), 39.12 (d, 13-C), 39.67 (t, 1-C), 40.10 (t, 2'-C), 40.81 (s, 8-C), 40.84 (t, 1'-C), 42.03 (s, 14-C), 47.32 (s, 4-C), 48.20 (s, 17-C), 49.36 (d, 18-C), 50.01 (d, 9-C), 54.95 (d, 5-C), 79.46 (s, 4'-C), 116.78 (d, 21-C), 143.10 (s, 20-C), 156.65 (s, 3'-C), 176.66 (s, 28-C), 218.21 (s, 3-C). IR (cm^{-1}): 3357, 2939, 2864, 1686, 1635, 1534, 1403, 1178. MS (ESI, 15 V): m/z = 597.2 $[\text{M}+\text{H}]^+$; 619.2 $[\text{M}+\text{Na}]^+$. For $\text{C}_{37}\text{H}_{60}\text{N}_2\text{O}_4$ (596.88) calcd. (%) C (74.45), H (10.13), N (4.69), found (%) C (74.47), H (10.12), N (4.70).



Z.Wimmer MV-1
in CDCl₃, ref=TMS
11.1.2017 DA

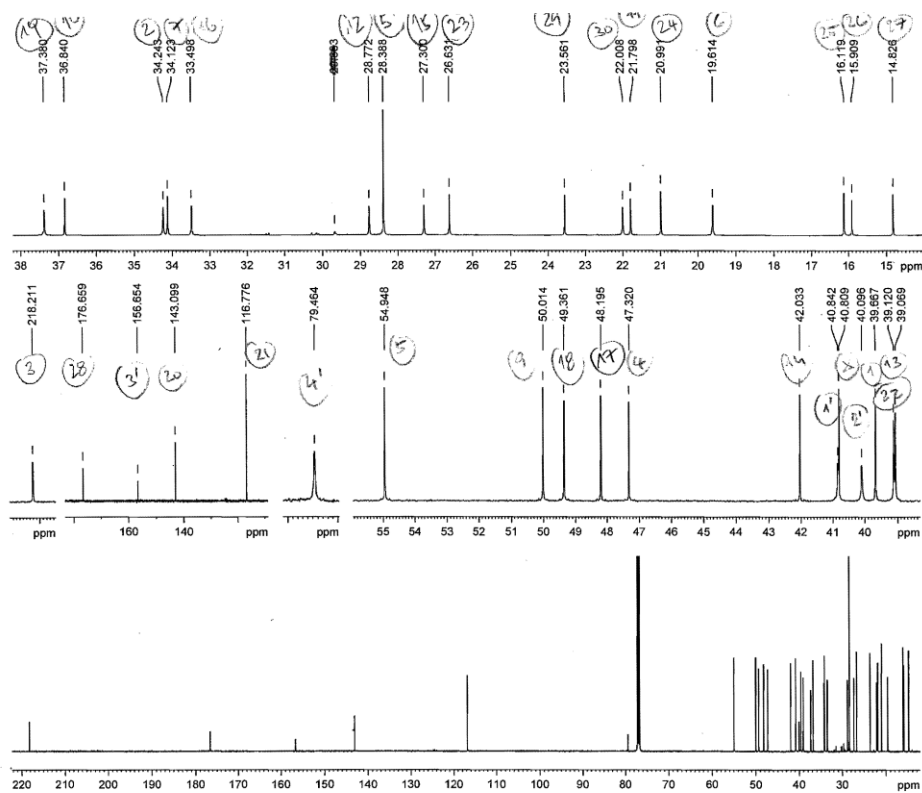
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EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170111
Time 9.36
INSTRUM spect
PROBHD 5 mm CPBBO BB-
PULPROG zg30
TD 31998
SOLVENT CDCl₃
NS 16
DS 0
SWH 4000.000 Hz
FIDRES 0.125008 Hz
AQ 3.997499 sec
RG 49.82
DW 125.000 usec
DE 18.00 usec
TE 298.0 K
D1 0 sec
TD0 1

===== CHANNEL f1 =====
SFO1 499.9820999 MHz
NUC1 1H
P1 10.00 usec
PLW1 2.79999995 W

F2 - Processing parameters
SI 131072
SF 499.9800326 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00

53903



Z.Wimmer MV-1
in CDCl₃, ref=TMS
11.1.2017 DA

Current Data Parameters
NAME Wimmer-MV_1
EXPNO 22
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170116
Time 13.09
INSTRUM spect
PROBHD 5 mm CPBBO BB-
PULPROG zgpg30
TD 59520
SOLVENT CDCl₃
NS 1024
DS 0
SWH 29761.904 Hz
FIDRES 0.500032 Hz
AQ 0.9999360 sec
RG 199.71
DW 16.800 usec
DE 18.00 usec
TE 298.0 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

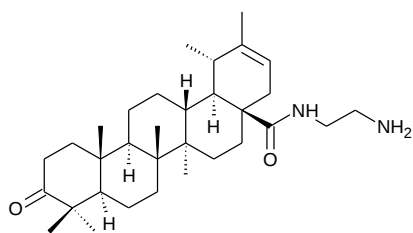
===== CHANNEL f1 =====
SFO1 125.7351574 MHz
NUC1 13C
P1 12.00 usec
PLW1 35.00000000 W

===== CHANNEL f2 =====
SFO2 499.9819999 MHz
NUC2 1H
CPDPRG2 waltz16
PCPD2 80.00 usec
PLW2 8.60000038 W
PLW12 0.19350000 W
PLW13 0.09732900 W

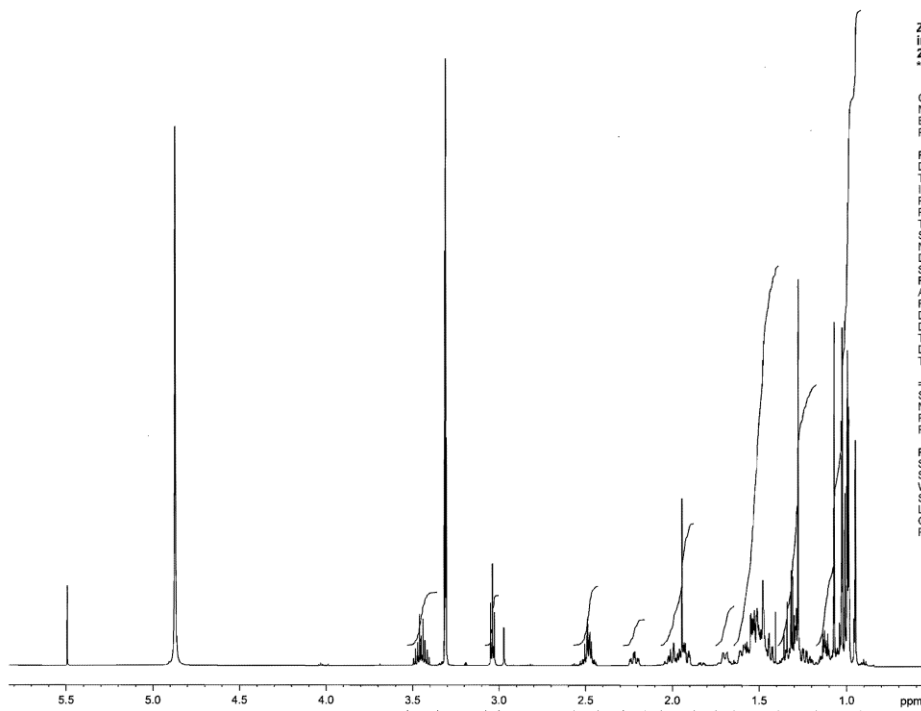
F2 - Processing parameters
SI 131072
SF 125.7200748 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0

53903

Analytical data of 13b



13b: ¹H-NMR (600.13 MHz, CD₃OD): δ [ppm] 0.90 (d, 3H, *J*=0.7 Hz, 25-CH₃), 0.93 (s, 3H, 26-CH₃), 0.94 (s, 3H, 27-CH₃), 0.97 (d, 3H, *J*=6.5 Hz, 29-CH₃), 0.98 (s, 3H, 24-CH₃), 1.03 (s, 3H, 23-CH₃), 1.35-1.39 (m, 1H, 1-CH₂), 1.61 (t, 3H, *J*=1.9 Hz, 30-CH₃), 1.71 (bdq, 2H, *J*=3.2; 3.2; 3.2; 12.6 Hz, 15-CH₂), 1.82 (bdq, 1H, *J*=2.1; 2.1; 2.1; 15.9 Hz, 22-CH₂), 1.90 (ddd, 1H, *J*=4.4; 7.6; 13.2 Hz, 1-CH₂), 1.98 (dd, 1H, *J*=7.2; 15.9 Hz, 22-CH₂), 2.06-2.13 (m, 1H, 19-CH₂), 2.38 (ddd, 1H, *J*=4.4; 7.6; 15.7 Hz, 2-CH₂), 2.40 (dt, 1H, *J*=4.4; 11.5; 11.5 Hz, 13-CH), 2.46 (ddd, 1H, *J*=7.6; 9.7; 15.7 Hz, 2-CH₂), 3.12-3.34 (m, 4H, 1', 2'-CH₂), 5.19 (m, 1H, *J*=1.5; 1.5; 1.5; 1.5; 7.2 Hz, 21-CH); ¹³C-NMR (125.74 MHz, CDCl₃): δ [ppm] 14.83 (q, 27-C), 15.91 (q, 26-C), 16.12 (q, 25-C), 19.61 (t, 6-C), 20.99 (q, 24-C), 21.80 (t, 11-C), 22.01 (q, 30-C), 23.56 (q, 29-C), 26.63 (q, 23-C), 27.30 (t, 15-C), 28.77 (t, 12-C), 33.50 (t, 16-C), 34.12 (t, 7-C), 34.24 (t, 2-C), 36.84 (s, 10-C), 37.38 (d, 19-C), 39.07 (t, 22-C), 39.12 (d, 13-C), 39.67 (t, 1-C), 40.10 (t, 2'-C), 40.81 (s, 8-C), 40.84 (t, 1'-C), 42.03 (s, 14-C), 47.32 (s, 4-C), 48.20 (s, 17-C), 49.36 (d, 18-C), 50.01 (d, 9-C), 54.95 (d, 5-C), 116.78 (d, 21-C), 143.10 (s, 20-C), 176.66 (s, 28-C), 218.21 (s, 3-C). IR (cm⁻¹): 2938, 2868, 1704, 1681, 1457, 1382, 1075. MS (ESI, 20 V): *m/z* = 497.3 [M+H]⁺. For C₃₂H₅₂N₂O₂ (496.77) calcd. (%) C (77.37), H (10.55), N (6.44), found (%) C (77.35), H (10.53), N (6.43).



Z.Wimmer MV-2
in CD3OD, rfp=3.31 ppm
23.10.2017 DA

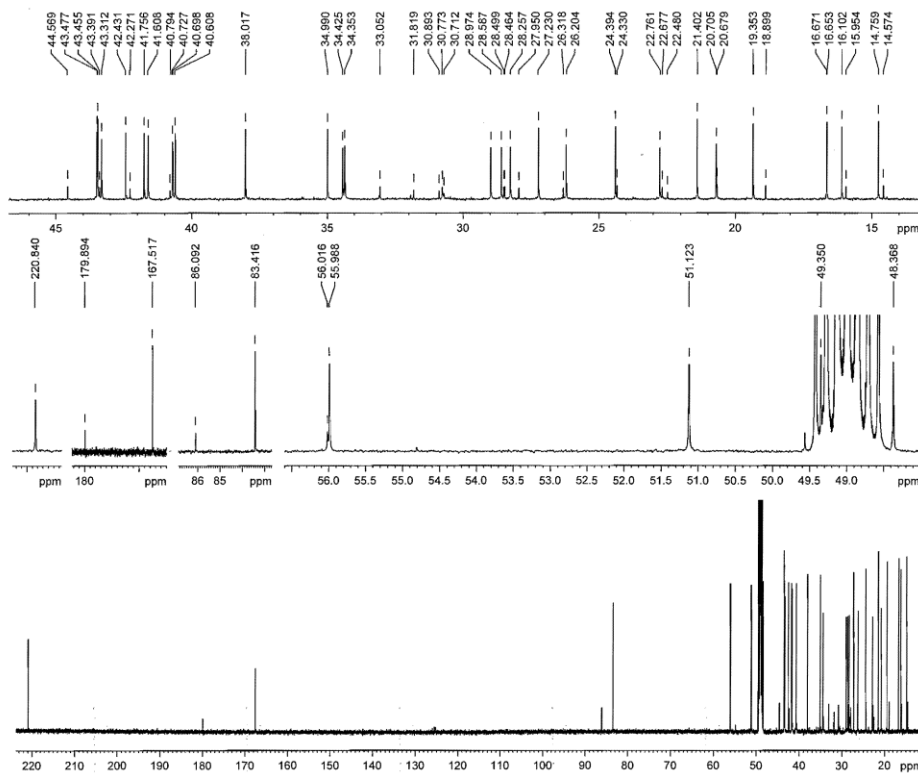
Current Data Parameters
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EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20171023
Time 6.59
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 43288
SOLVENT MeOD
NS 8
DS 0
SWH 5411.255 Hz
FIDRES 0.125006 Hz
AQ 3.9998112 sec
RG 43.64
DW 92.400 usec
DE 20.00 usec
TE 298.1 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 600.1324005 MHz
NUC1 1H
P1 9.00 usec
PLW1 6.00000000 W

F2 - Processing parameters
SI 131072
SF 600.1300116 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00

55027



Z.Wimmer MV-2
in CD3OD, rfp=3.31 ppm
23.10.2017 DA

Current Data Parameters
NAME Wimmer-MV_2
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20171023
Time 7.00
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zgpg30
TD 72460
SOLVENT MeOD
NS 1024
DS 0
SWH 36231.883 Hz
FIDRES 0.500026 Hz
AQ 0.9999480 sec
RG 196.18
DW 13.800 usec
DE 18.00 usec
TE 298.1 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 150.9194083 MHz
NUC1 13C
P1 12.00 usec
PLW1 80.00000000 W

===== CHANNEL f2 =====
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG2 waltz16
PCPD2 70.00 usec
PLW2 6.00000000 W
PLW12 0.09918400 W
PLW13 0.04860000 W

F2 - Processing parameters
SI 131072
SF 150.9025976 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0

55027

Self-assembly detected in the UV measurement

The stock solutions of the studied compounds were prepared at a concentration of 1 mg·mL⁻¹, in water. A series of water/acetonitrile mixtures were then prepared starting from water/acetonitrile ratio 0/100 up to 100/0 in 10% steps. The stock solutions of the studied compounds (0.15 mL) were added separately to each vial containing water/acetonitrile mixtures (3 mL), and the UV spectra were recorded in the wavelength range of 200–400 nm. Spectra were recorded every 24 h for 8 days. As an example of the obtained data, UV spectra of **8a** are shown in **Figure S1**. The dynamic behaviour of the spectra with changing ratios of water/acetonitrile mixture in the respective days of measurement is clearly visible. The most pronounced changes in intensity of the maxima were detected in acetonitrile/water = 60 : 40 ratio. Comparing absorbance maxima in all three presented ratios of acetonitrile/water (50 : 50, 60 : 40 and 70 : 30), the maxima changed day by day, showing increasing maxima intensity from day 1 to day 8. However, irregularity was observed in days 7 and 8 in acetonitrile/water 60 : 40 ratio. This indicates dynamic behaviour of the supramolecular networks formed, i.e., the formed supramolecular networks are subjects of further changes in their nature, depending on the time. This behaviour also assisted in explaining irregularities observed in the biological replicates of the cytotoxicity assays *in vitro*. Other studied compounds (**8b**, **8c** and **11a–11c**) showed smaller changes in the absorbance maxima intensity. Amides **3a** and **3b**, derived from **1a** and **1b**, showed no self-assembly under the described conditions, and no such a behaviour described above was observed with these compounds.

Figure S2 shows examples of the UV spectra measured for **3a**, **8b**, **8c** and **11a**, which demonstrate that either only slight self-assembly was observed (**3a**) or no effect was

detected (**8b**, **8c** and **11c**). Other target compounds showed no self-assembly under the given conditions.

Figure S1. Irregularity in the absorbance maxima demonstrating a formation of supramolecular networks in the studied systems of **8a**.

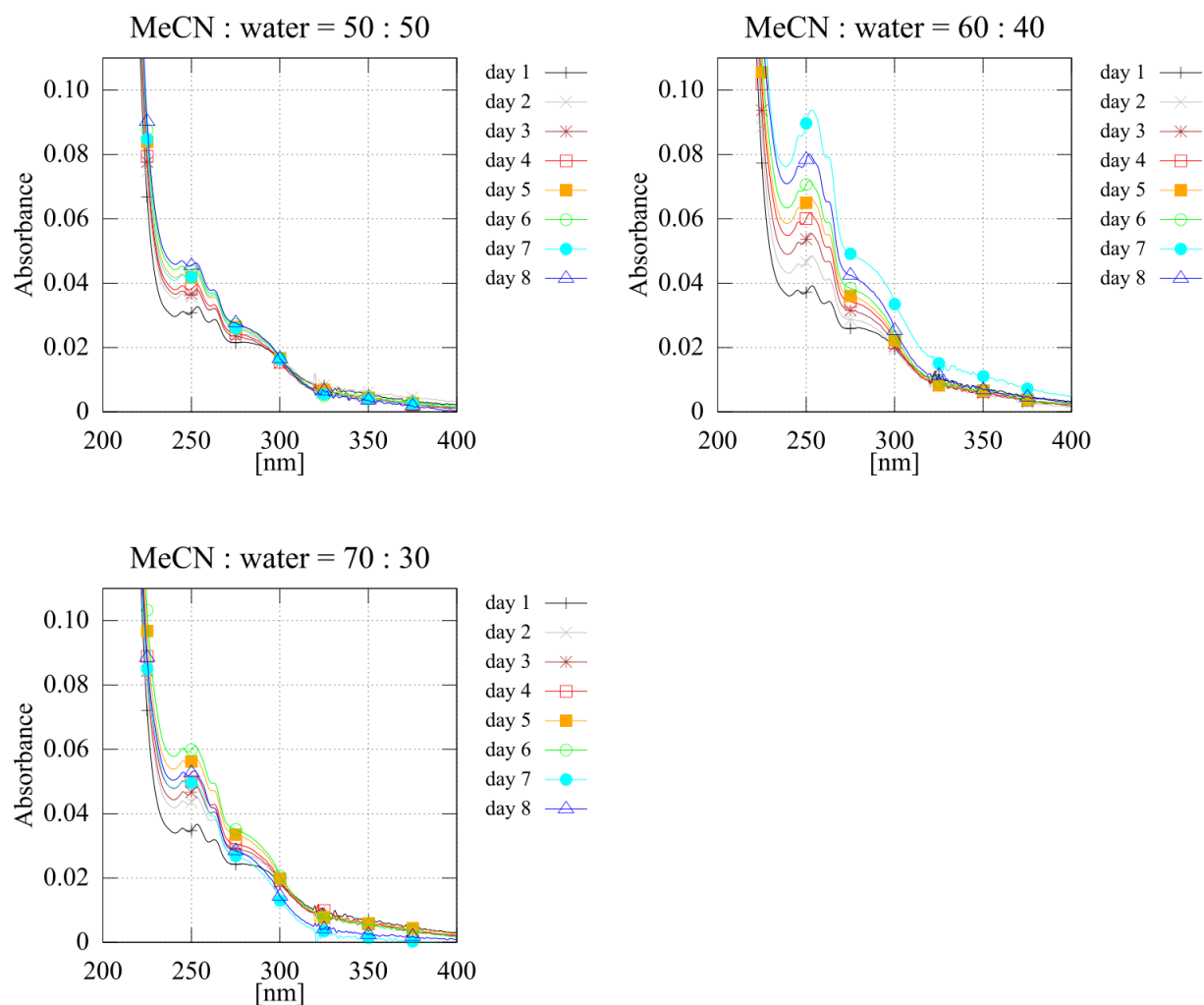


Figure S2. UV spectra of **3a**, **8b**, **8c** and **11a** demonstrating no visible self-assembly of these compounds under the given experimental conditions.

Figure S2a. UV spectra of **3a**.

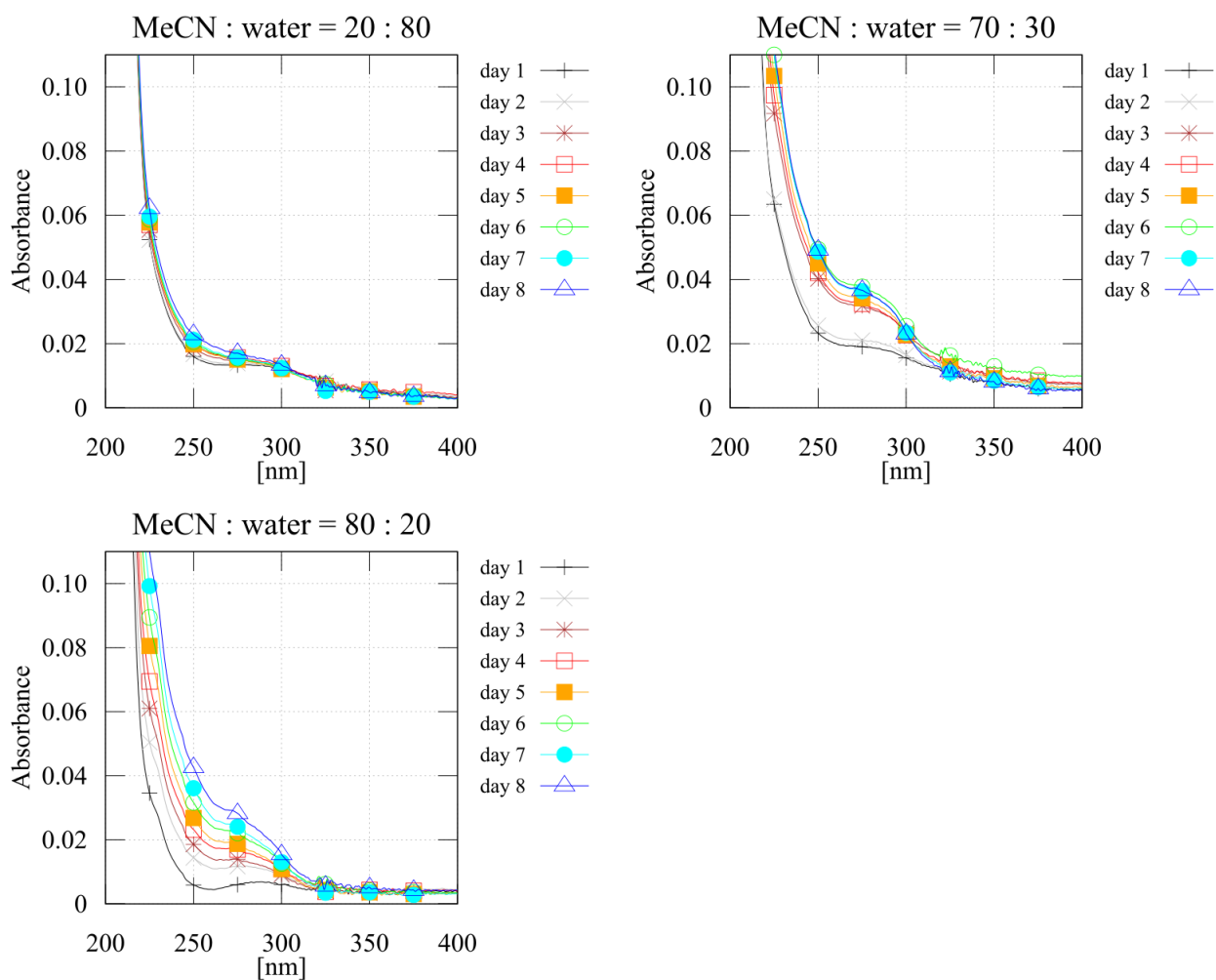


Figure S2b. UV spectra of **8b**.

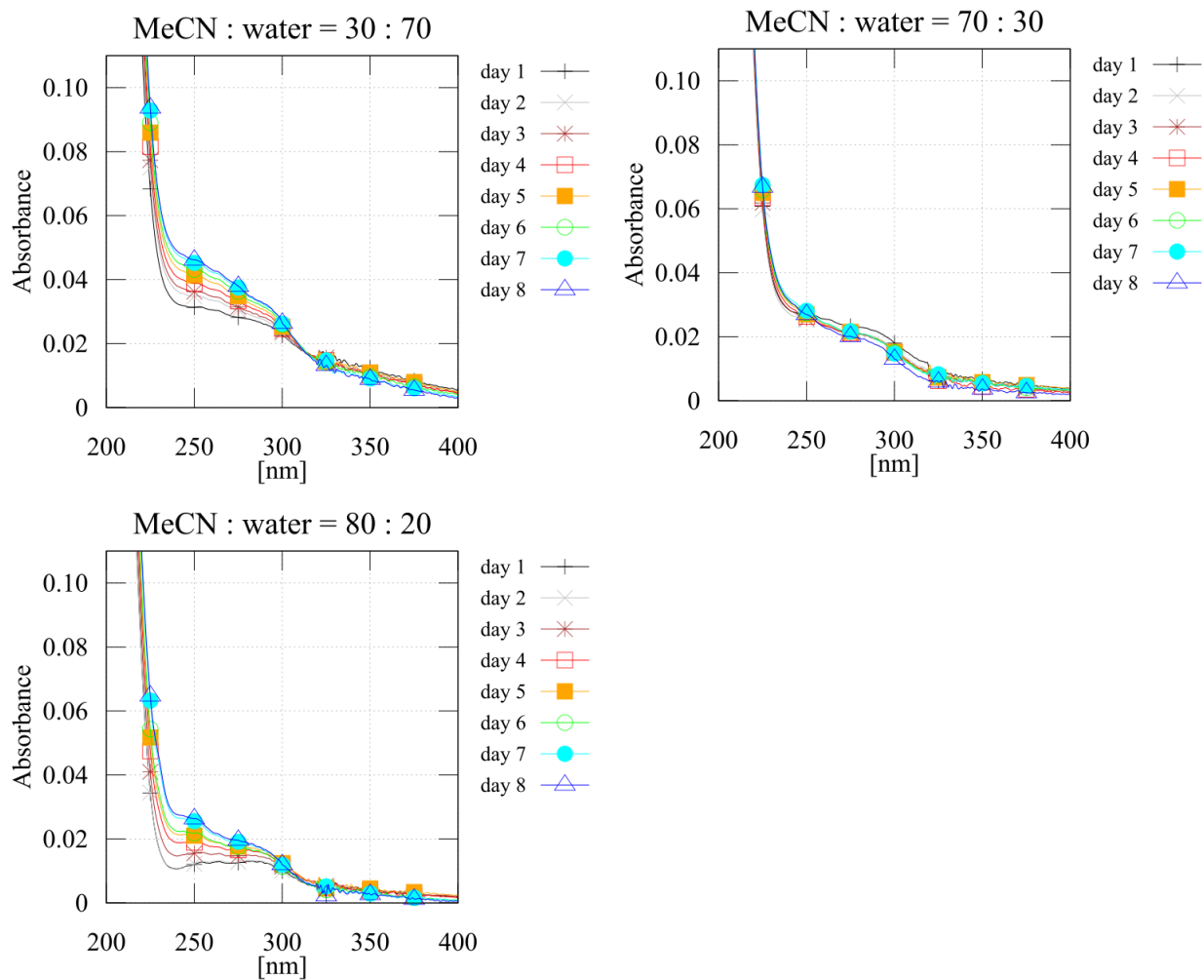


Figure S2c. UV spectra of **8c**.

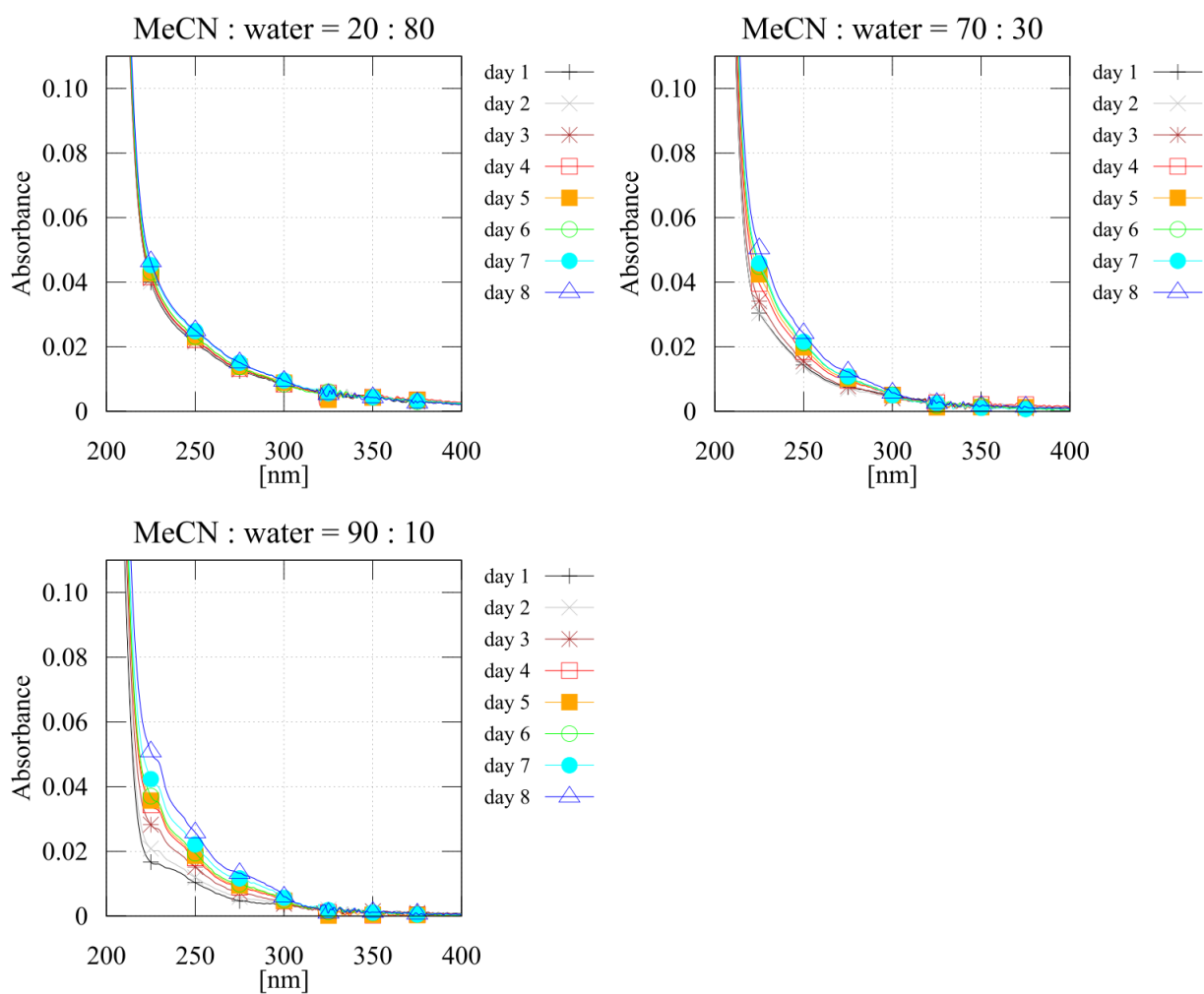


Figure S2d. UV spectra of **11a**.

