

Electronic Supplementary Information (ESI)

Nano-assembly of cytotoxic amides of moronic and morolic acid

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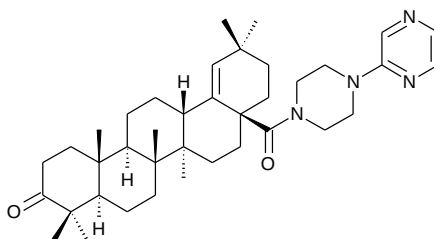
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Content

1. Experimental part – A List of the NMR Spectra
2. Additional Data on Cytotoxicity Screening Tests

1. Experimental Part – A List of the NMR Spectra

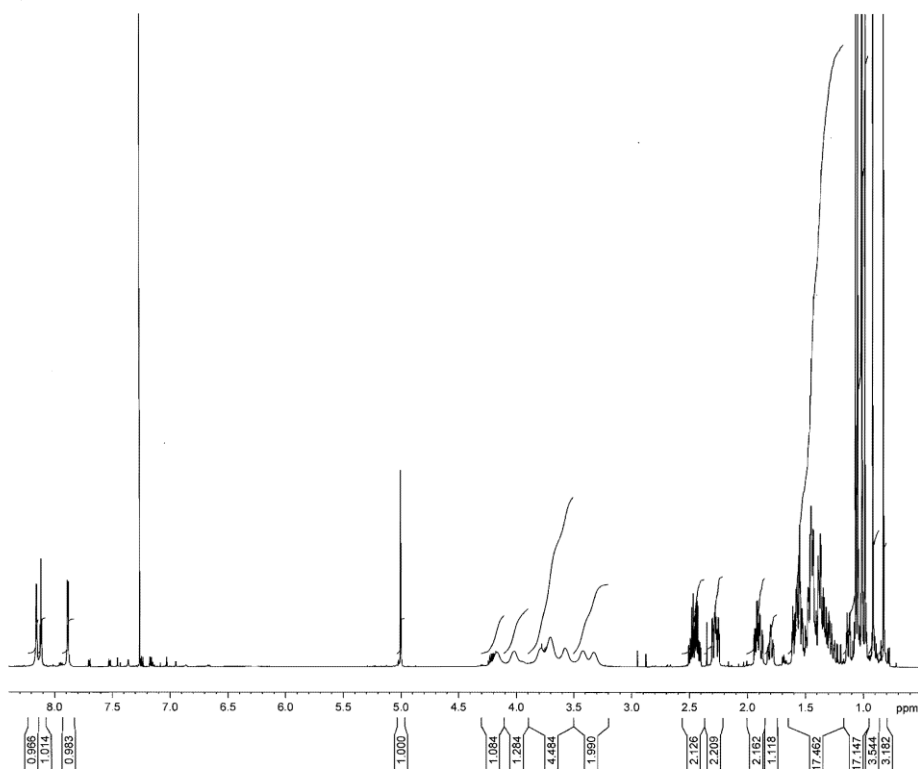
28-[4-(Pyrazin-2-yl)piperazin-1-yl]olean-18-en-3,28-dione (**3**)



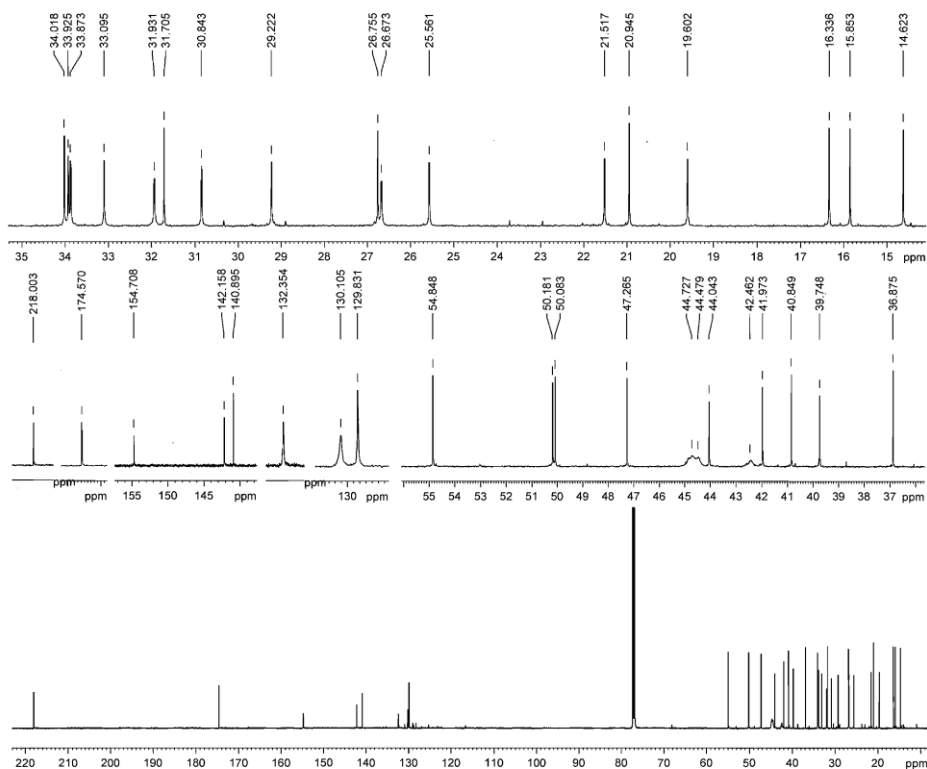
^1H NMR: δ 0.82 (3H, s, H27), 0.92 (3H, s, H26), 0.98 (3H, s, H25), 1.01 (3H, s, H24), 1.01 (3H, s, H30), 1.04 (3H, s, H29), 1.06 (3H, s, H23), 1.11 (1H, ddd, $J_1=2.9$ Hz, $J_2=3.6$ Hz, $J_3=13.6$ Hz, H15), 1.59 (1H, ddd, $J_1=3.8$ Hz, $J_2=4.4$ Hz, $J_3=12.9$ Hz, H16), 1.80 (1H, dt, $J_1=3.8$ Hz, $J_2=13.5$ Hz, $J_3=13.5$ Hz, H15), 1.89 (1H, ddd, $J_1=3.6$ Hz, $J_2=12.9$ Hz, $J_3=13.3$ Hz, H16), 1.92 (2H, ddd, $J_1=4.7$ Hz, $J_2=7.6$ Hz, $J_3=13.3$ Hz, H1), 2.29 (1H, ddd, $J_1=1.3$ Hz, $J_2=3.2$ Hz, $J_3=12.5$ Hz, H13), 2.42 (1H, ddd, $J_1=4.7$ Hz, $J_2=8.0$ Hz, $J_3=15.7$ Hz, H2), 2.48 (1H, ddd, $J_1=7.6$ Hz, $J_2=9.4$ Hz, $J_3=15.7$ Hz, H2), 3.30-4.21 (4H, m, H1'), 3.30-4.22 (4H, m, H2'), 5.00 (1H, t, $J=1.1$ Hz, H19), 7.89 (1H, bd, $J=2.5$ Hz, H5'), 8.12 (1H, dd, $J_1=1.5$ Hz, $J_2=2.5$ Hz, H6'), 8.16 (1H, bs, H4').

^{13}C NMR: δ 14.60 (q, C27), 15.90 (q, C25), 16.30 (q, C26), 19.60 (t, C6), 20.90 (q, C24), 21.50 (t, C11), 25.60 (t, C12), 26.70 (q, C30), 26.80 (q, C23), 29.20 (t, C15), 30.80 (t, C16), 31.70 (s, C20), 31.90 (q, C29), 33.10 (t, C7), 33.90 (t, C21), 33.90 (t, C22), 34.00 (t, C2), 36.90 (s, C10), 39.70 (t, C1), 40.80 (s, C8), 42.00 (d, C13), 42.50 (t, C2'), 44.00 (s, C4), 44.70 (t, C1'), 47.30 (s, C14), 50.10 (s, C17), 50.20 (d, C9), 54.80 (d, C5), 129.80 (d, C19), 130.10 (d, C4'), 132.40 (d, C5'), 140.90 (s, C18), 142.20 (d, C6'), 154.70 (s, C3'), 174.60 (s, C28), 218.00 (s, C3).

MS: $m/z = 601$ $[\text{M}+\text{H}]^+$. For $\text{C}_{38}\text{H}_{56}\text{N}_4\text{O}_2$ (600.88) calcd. C 75.96, H 9.39, N 9.32, found C 75.99, H 9.37, N 9.28.

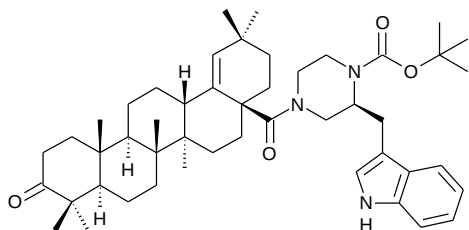


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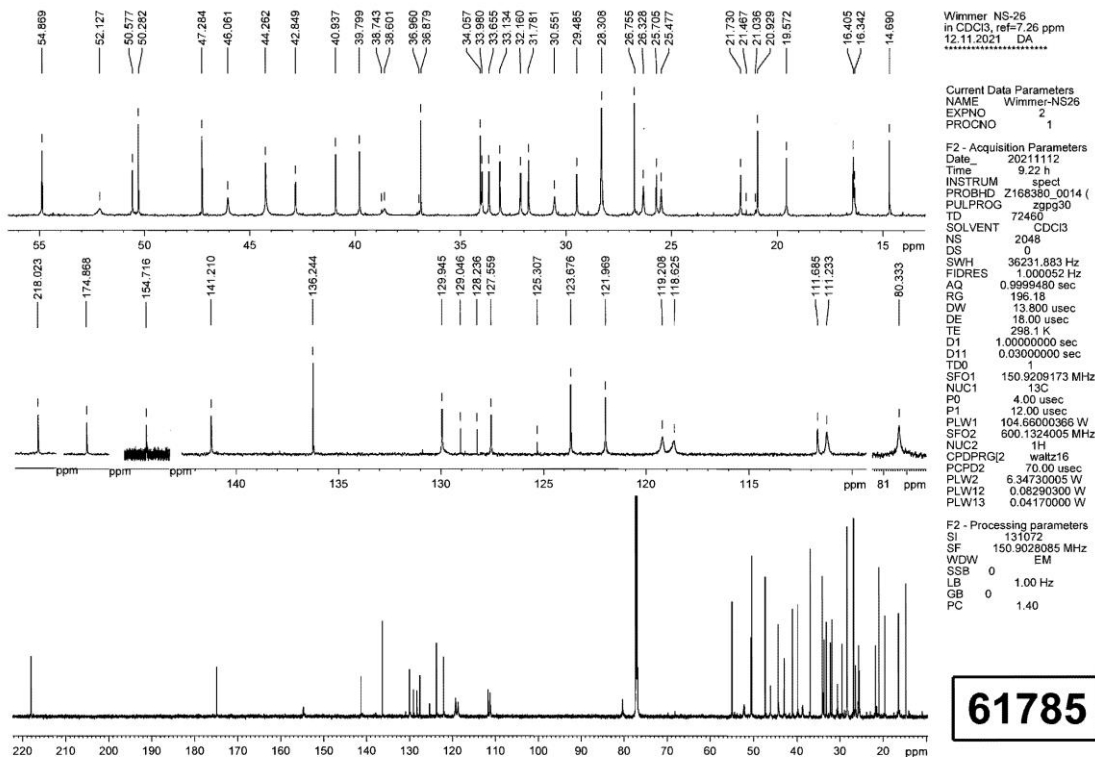
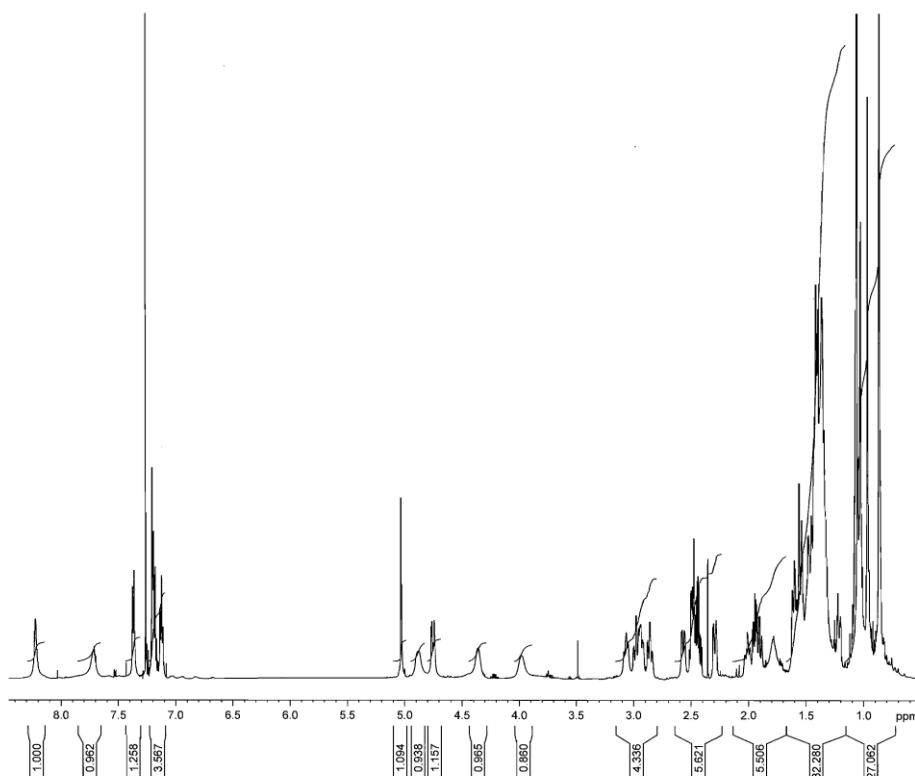
tert-Butyl-[(2*S*)-4-(3,28-dioxoolean-18-en-28-yl)-2-(1*H*-indol-3-ylmethyl)piperazine] 1-carboxylate (**4**)



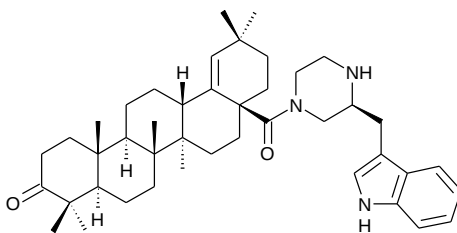
^1H NMR: δ 0.86 (3H, s, H25), 0.86 (3H, s, H27), 0.96 (3H, s, H24), 1.03 (3H, s, H30), 1.06 (3H, s, H23), 1.06 (3H, s, H26), 1.06 (3H, s, H29), 1.95 (2H, ddd, $J_1=4.6$ Hz, $J_2=7.6$ Hz, $J_3=12.4$ Hz, H1), 2.29 (2H, bdt, $J_1=3.2$ Hz, $J_2=3.2$ Hz, $J_3=13.0$ Hz, H22), 2.43 (1H, ddd, $J_1=4.6$ Hz, $J_2=7.9$ Hz, $J_3=15.7$ Hz, H2), 2.47-2.51 (1H, m, H2), 2.47-2.51 (1H, m, H13), 2.57 (1H, bdd, $J_1=3.2$ Hz, $J_2=13.5$ Hz, H1'), 2.86 (1H, bdt, $J_1=3.0$ Hz, $J_2=12.5$ Hz, $J_3=12.5$ Hz, H4'), 2.93 (1H, dd, $J_1=4.5$ Hz, $J_2=13.5$ Hz, H8'), 2.98 (1H, bdt, $J_1=3.0$ Hz, $J_2=12.9$ Hz, $J_3=12.9$ Hz, H3'), 3.07 (1H, bdd, $J_1=9.2$ Hz, $J_2=13.5$ Hz, H8'), 3.98 (1H, bd, $J=13.2$ Hz, H3'), 4.36 (1H, bs, H2'), 4.75 (1H, bd, $J=13.5$ Hz, H1'), 4.89 (1H, bd, $J=13.0$ Hz, H4'), 5.03 (2H, s, H19), 7.12 (1H, bt, $J=7.4$ Hz, H15'), 7.19 (1H, bt, $J=7.6$ Hz, H14'), 7.20 (1H, bs, H10'), 7.33 (1H, d, $J=8.1$ Hz, H13'), 7.72 (1H, bd, $J=7.2$ Hz, H16'), 8.22 (1H, bs, NH).

^{13}C NMR: δ 14.70 (q, C27), 16.30 (q, C25), 16.40 (q, C26), 19.60 (t, C6), 20.90 (q, C24), 21.70 (t, C11), 25.50 (t, C8'), 25.70 (t, C12), 26.30 (q, C30), 26.80 (q, C23), 28.30 (q, C7'), 29.50 (t, C15), 30.60 (t, C21), 31.80 (t, C16), 31.80 (s, C20), 32.20 (q, C29), 33.70 (t, C7), 34.00 (t, C22), 34.10 (t, C2), 36.90 (s, C10), 38.70 (t, C3'), 39.80 (t, C1), 40.90 (s, C8), 42.80 (d, C13), 44.30 (s, C14), 44.30 (t, C1'), 46.10 (t, C4'), 47.30 (s, C4), 50.30 (s, C17), 50.60 (d, C9), 52.10 (d, C2'), 54.90 (d, C5), 80.30 (s, C6'), 111.20 (d, C13'), 111.70 (s, C9'), 118.60 (d, C16'), 119.20 (d, C15'), 122.00 (d, C14'), 123.70 (d, C10'), 127.60 (s, C17'), 129.90 (d, C19), 136.20 (d, C12'), 141.20 (s, C18), 154.70 (s, C5'), 174.90 (s, C28), 218.00 (s, C3).

MS: $m/z = 753$ $[\text{M}+\text{H}]^+$. For $\text{C}_{48}\text{H}_{69}\text{N}_3\text{O}_4$ (752.08) calcd. C 76.66, H 9.25, N 5.59, found C 76.69, H 9.22, N 5.62.



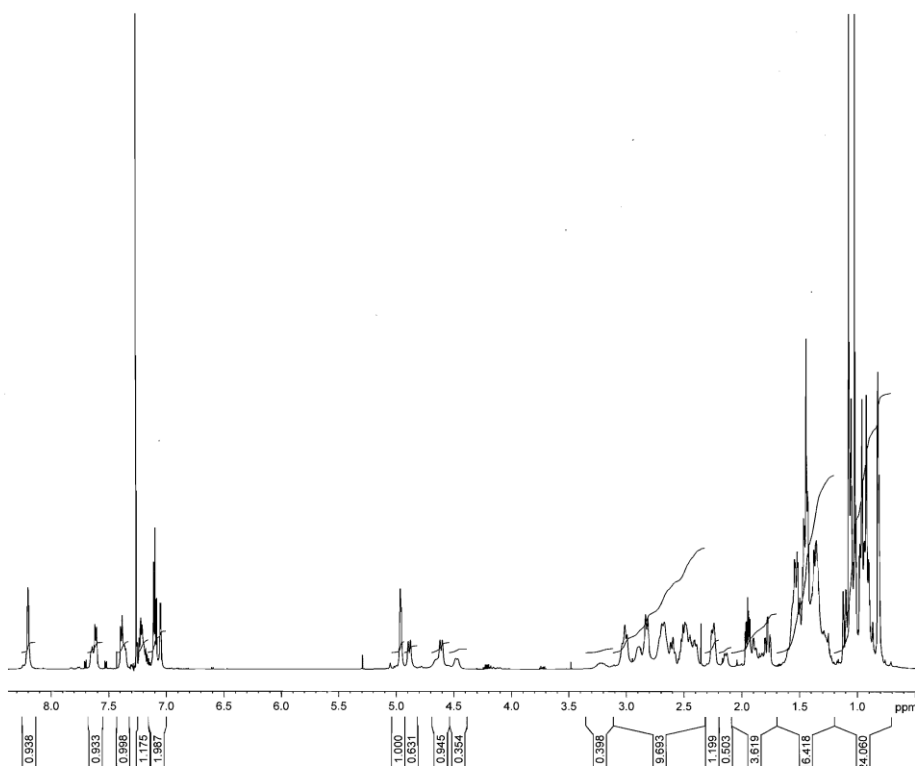
28-[(3S)-3-(1*H*-Indol-3-ylmethyl)piperazin-1-yl]olean-18-en-3,28-dione (5)



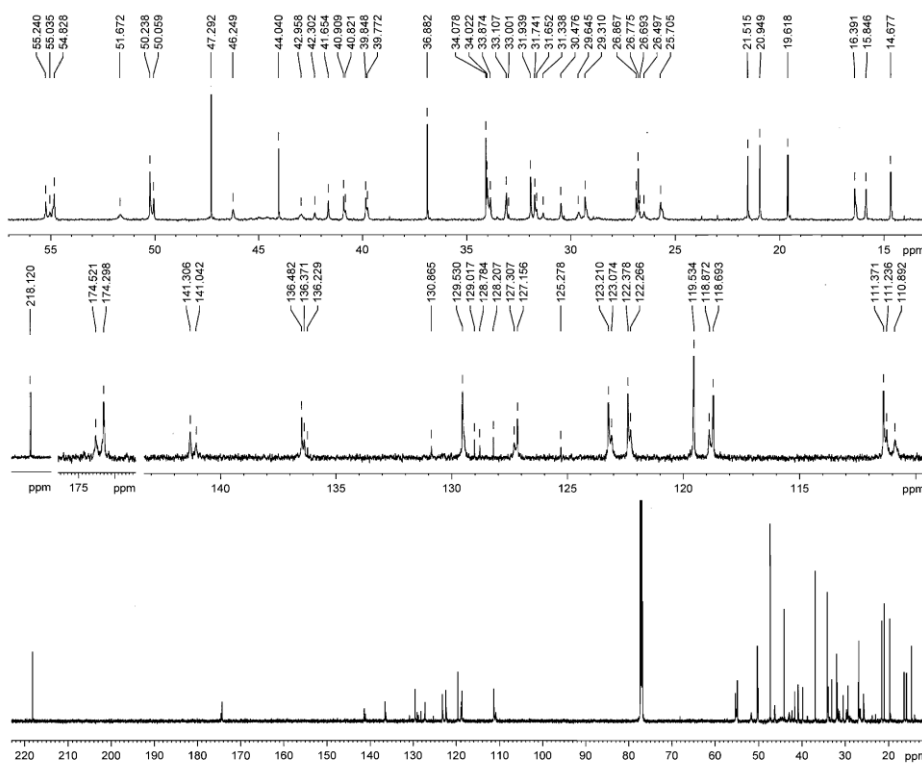
^1H NMR: δ 0.83 (3H, s, H27), 0.93 (3H, s, H26), 0.95 (3H, s, H25), 1.02 (3H, s, H24), 1.05 (3H, s, H29), 1.07 (3H, s, H23), 1.07 (3H, s, H30), 1.11 (1H, ddd, $J_1=2.6$ Hz, $J_2=3.6$ Hz, $J_3=13.5$ Hz, H15), 1.53 (2H, dt, $J_1=3.3$ Hz, $J_2=13.2$ Hz, $J_3=13.2$ Hz, H22), 1.78 (1H, dt, $J_1=4.5$ Hz, $J_2=13.2$ Hz, $J_3=13.2$ Hz, H15), 1.95 (2H, ddd, $J_1=4.7$ Hz, $J_2=7.7$ Hz, $J_3=12.5$ Hz, H1), 2.38-2.56 (2H, m, H2), 2.38-2.57 (1H, m, H13), 2.38-2.56 (2H, m, H3'), 2.60 (1H, bt, $J_1=13.0$ Hz, $J_2=13.0$ Hz, H5'), 3.04 (1H, bdd, $J_1=9.0$ Hz, $J_2=13.5$ Hz, H5'), 4.62 (2H, bd, $J=13.3$ Hz, H1'), 4.89 (2H, bd, $J=13.0$ Hz, H4'), 4.98 (1H, bs, H19), 7.05 (1H, s, H7'), 7.10 (1H, bt, $J=7.5$ Hz, H12'), 7.23 (1H, bt, $J=7.6$ Hz, H11'), 7.39 (1H, d, $J=7.7$ Hz, H10'), 7.62 (1H, bd, $J=7.5$ Hz, H13'), 8.20 (1H, s, H8'-NH).

^{13}C NMR: δ 14.70 (q, C27), 15.80 (q, C25), 16.40 (q, C26), 19.60 (t, C6), 20.90 (q, C24), 21.50 (t, C11), 25.70 (t, C12), 26.50 (q, C30), 26.80 (q, C23), 29.30 (t, C15), 30.50 (t, C21), 31.40 (q, C29), 31.70 (t, C16), 31.70 (s, C20), 33.90 (t, C7), 34.00 (t, C22), 34.10 (t, C2), 36.90 (s, C10), 39.80 (t, C1), 40.90 (s, C8), 41.70 (d, C13), 42.90 (t, C1'), 44.00 (s, C14), 44.60 (t, C3'), 46.20 (t, C5'), 47.30 (s, C4), 50.10 (s, C17), 50.20 (d, C9), 51.70 (t, C4'), 54.80 (d, C5), 55.20 (d, C2'), 111.20 (s, C6'), 111.40 (d, C10'), 119.50 (d, C12'), 122.40 (d, C11'), 123.20 (d, C7'), 129.50 (d, C19), 136.50 (s, C9'), 141.30 (s, C18), 174.30 (s, C28), 218.10 (s, C3).

MS: $m/z = 653$ $[\text{M}+\text{H}]^+$. For $\text{C}_{43}\text{H}_{61}\text{N}_3\text{O}_2$ (651.96) calcd. C 79.22, H 9.43, N 6.45, found C 79.25, H 9.40, N 6.43.

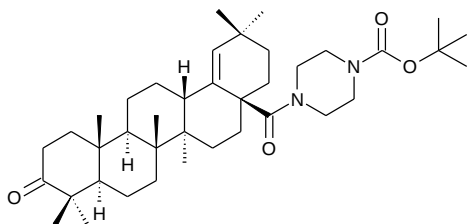


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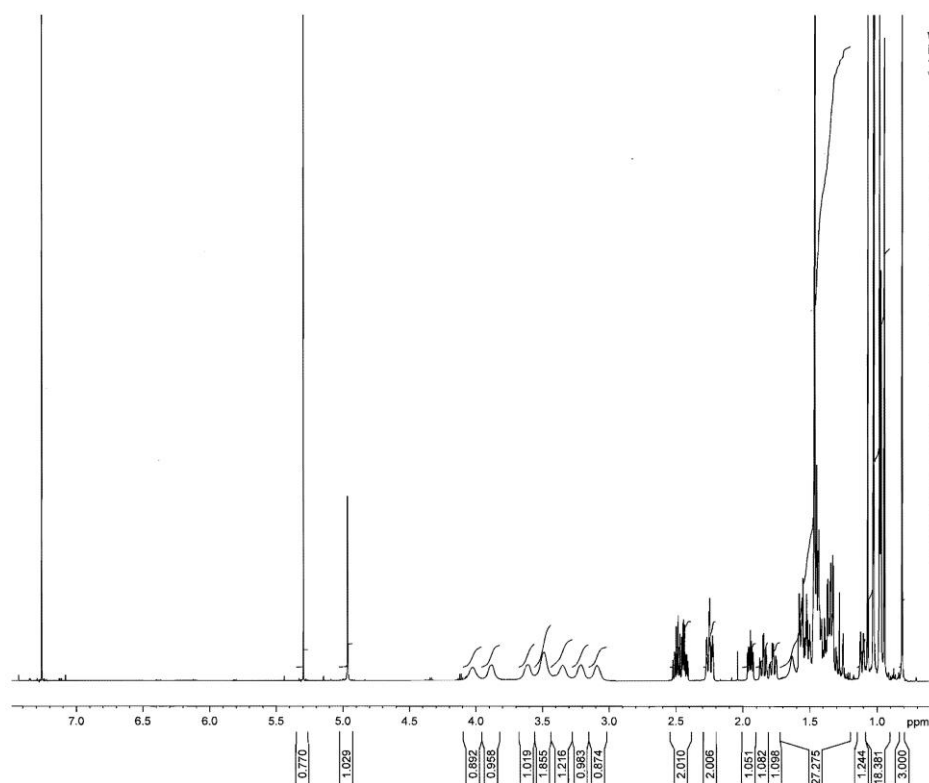
tert-Butyl-4-(3,28-dioxoolean-18-en-28-yl)piperazin-1-carboxylate (**6**)



^1H NMR: δ 0.81 (3H, d, $J=0.7$ Hz, H27), 0.95 (3H, s, H25), 0.97 (3H, s, H30), 0.98 (3H, d, $J=0.6$ Hz, H26), 1.02 (3H, s, H24), 1.03 (3H, s, H29), 1.07 (3H, s, H23), 1.11 (1H, ddd, $J_1=2.6$ Hz, $J_2=3.7$ Hz, $J_3=13.5$ Hz, H15), 1.47 (3H, s, H5'), 1.78 (1H, dt, $J_1=4.1$ Hz, $J_2=13.3$ Hz, $J_3=13.3$ Hz, H15), 1.85 (2H, ddd, $J_1=3.8$ Hz, $J_2=12.5$ Hz, $J_3=13.2$ Hz, H21), 1.94 (2H, ddd, $J_1=4.6$ Hz, $J_2=7.6$ Hz, $J_3=12.7$ Hz, H1), 2.24 (2H, ddd, $J_1=2.8$ Hz, $J_2=4.1$ Hz, $J_3=12.7$ Hz, H22), 2.26 (1H, ddd, $J_1=1.3$ Hz, $J_2=3.0$ Hz, $J_3=13.0$ Hz, H13), 2.43 (1H, ddd, $J_1=4.6$ Hz, $J_2=7.9$ Hz, $J_3=15.7$ Hz, H2), 2.50 (1H, ddd, $J_1=7.6$ Hz, $J_2=9.4$ Hz, $J_3=15.7$ Hz, H2), 3.32-3.36 (1H, m, H2'), 3.45-3.52 (1H, m, H1'), 3.87-3.92 (1H, m, H2'), 3.99-4.03 (1H, m, H1'), 4.97 (1H, t, $J=1.2$ Hz, H19).

^{13}C NMR: δ 14.62 (q, C27), 15.83 (q, C26), 16.37 (q, C25), 19.61 (t, C6), 20.97 (q, C24), 21.53 (t, C11), 25.50 (t, C12), 26.55 (q, C30), 26.75 (q, C23), 28.37 (q, C5'), 29.24 (t, C15), 30.84 (t, C21), 31.67 (t, C16), 31.67 (s, C20), 31.93 (q, C29), 33.10 (t, C7), 33.91 (t, C22), 34.05 (t, C2), 36.89 (s, C10), 39.76 (t, C1), 40.85 (s, C8), 41.87 (d, C13), 42.76 (t, C2'), 44.01 (s, C14), 45.26 (t, C1'), 47.28 (s, C4), 50.06 (s, C17), 50.19 (d, C9), 54.87 (d, C5), 80.23 (s, C4'), 129.67 (d, C19), 140.95 (s, C18), 154.52 (s, C3'), 174.52 (s, C28), 218.06 (s, C3).

IR 2931 (w), 2860 (w), 1697 (s), 1625 (m), 1410 (m), 1164 (s). MS: $m/z = 623$ $[\text{M}+\text{H}]^+$. For $\text{C}_{39}\text{H}_{62}\text{N}_2\text{O}_4$ (622.92) calcd. C 75.20, H 10.03, N 4.50, found C 75.18, H 10.05, N 4.52.



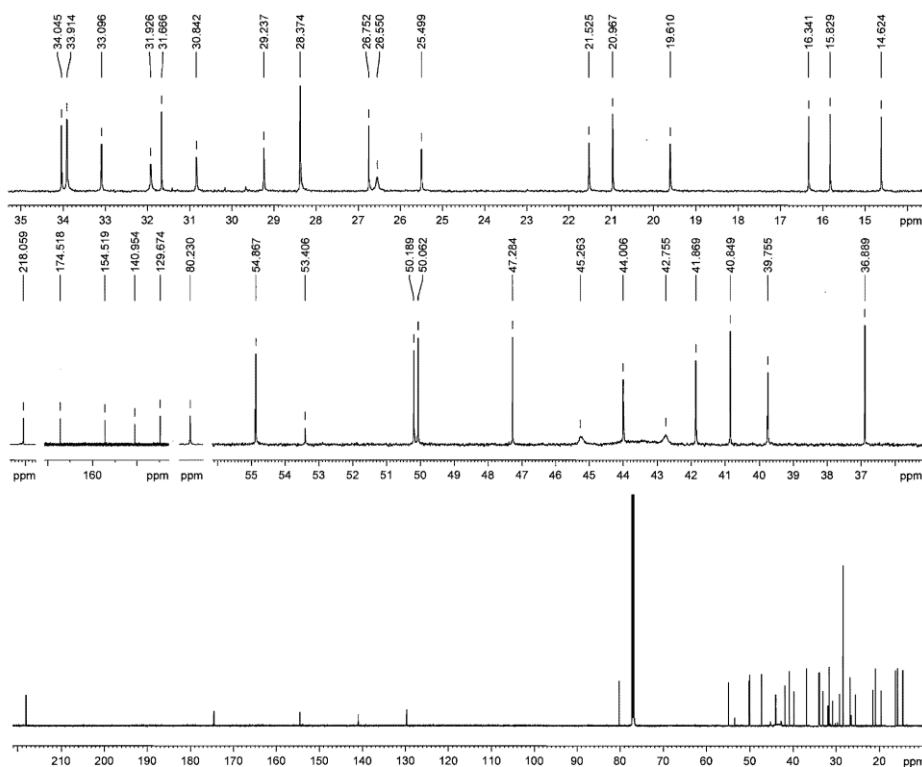
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56902



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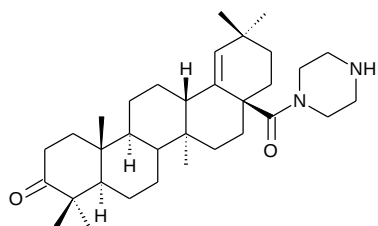
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56902

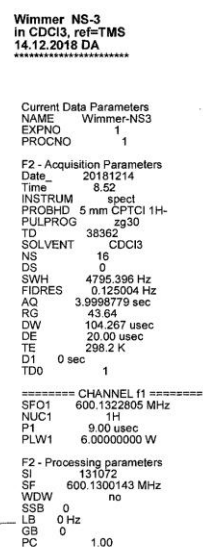
4-(3,28-Dioxoolean-18-en-28-yl)piperazine (7).



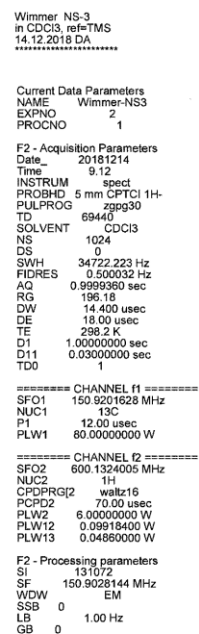
^1H NMR: δ 0.80 (3H, s, H27), 0.94 (3H, s, H30), 0.96 (3H, s, H25), 0.97 (3H, s, H26), 1.02 (3H, s, H24), 1.03 (3H, s, H29), 1.07 (3H, s, H23), 1.12 (1H, dt, $J_1=3.2$ Hz, $J_2=3.2$ Hz, $J_3=13.9$ Hz, H15), 1.73 (1H, dt, $J_1=3.7$ Hz, $J_2=13.4$ Hz, $J_3=13.4$ Hz, H15), 1.77 (2H, dt, $J_1=3.4$ Hz, $J_2=13.2$ Hz, $J_3=13.2$ Hz, H21), 1.95 (2H, ddd, $J_1=4.5$ Hz, $J_2=7.7$ Hz, $J_3=12.7$ Hz, H1), 2.13 (1H, bd, $J=13.0$ Hz, H13), 2.20 (2H, ddd, $J_1=3.5$ Hz, $J_2=4.5$ Hz, $J_3=12.6$ Hz, H22), 2.43 (1H, ddd, $J_1=4.3$ Hz, $J_2=7.7$ Hz, $J_3=15.6$ Hz, H2), 2.51 (1H, ddd, $J_1=4.6$ Hz, $J_2=9.5$ Hz, $J_3=15.6$ Hz, H2), 2.95-3.02 (1H, m, H2'), 3.19-3.25 (1H, m, H2'), 3.95-4.02 (1H, m, H1'), 4.11-4.20 (1H, m, H1'), 5.00 (1H, s, H19).

^{13}C NMR: δ 14.50 (q, C27), 15.92 (q, C26), 16.32 (q, C25), 19.55 (t, C6), 20.98 (q, C24), 21.49 (t, C11), 25.57 (t, C12), 26.62 (q, C30), 26.68 (q, C23), 29.03 (t, C15), 30.77 (t, C21), 31.68 (s, C20), 31.84 (q, C29), 32.91 (t, C16), 33.67 (t, C7), 33.95 (t, C22), 34.04 (t, C2), 36.89 (s, C10), 39.59 (t, C2'), 39.74 (t, C1), 40.82 (s, C8), 42.17 (d, C13), 42.31 (t, C2'), 43.43 (t, C1'), 43.81 (t, C1'), 44.11 (s, C14), 47.29 (s, C4), 50.13 (s, C17), 50.15 (d, C9), 54.91 (d, C5), 130.47 (d, C19), 140.41 (s, C18), 174.39 (s, C28), 217.91 (s, C3).

IR: 2930 (m), 2868 (w), 2700 (w), 1705 (s), 1627 (s), 1389 (s). MS: $m/z = 509$ $[\text{M}+\text{H}]^+$. For $\text{C}_{33}\text{H}_{52}\text{N}_2\text{O}_2$ (508.78) calcd. C 77.90, H 10.30, N 5.51, found C 77.93, H 10.28, N 5.48.

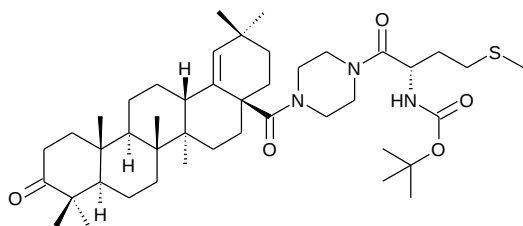


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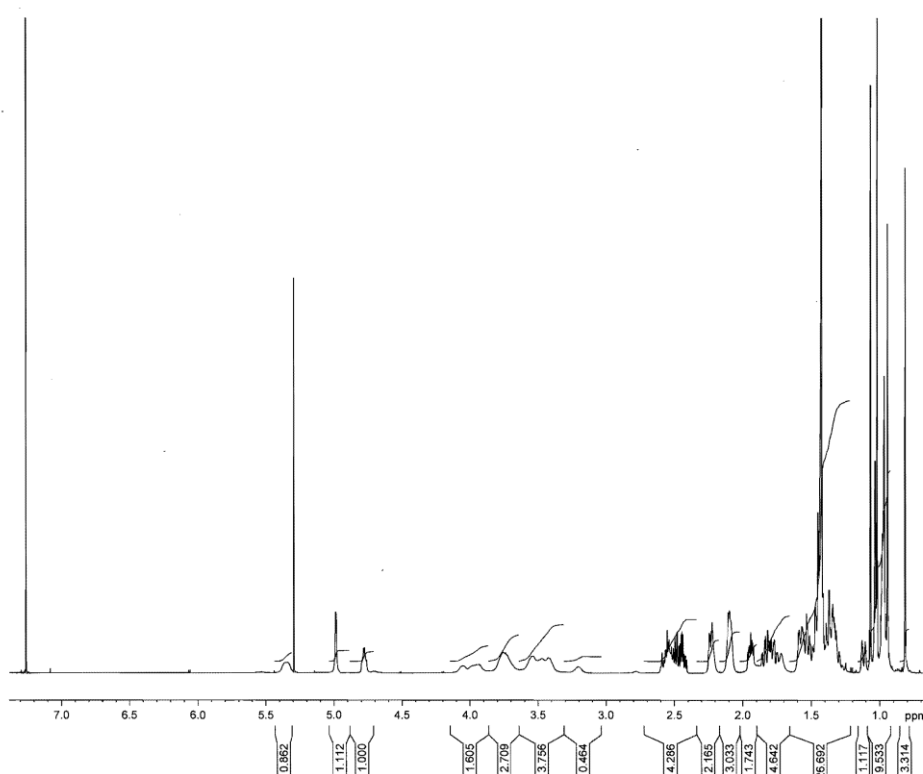
tert-Butyl-[(2*S*)-1-[4-(3,28-dioxoolean-18-en-28-yl)piperazin-1-yl]-4-(methylsulphonyl)-1-oxobutan-2-yl] carbamate (**8**)



^1H NMR: δ 0.82 (3H, s, H27), 0.94 (3H, s, H25), 0.97 (3H, s, H26), 0.98 (3H, s, H30), 1.02 (3H, s, H24), 1.04 (3H, s, H29), 1.07 (3H, s, H23), 1.12 (1H, ddd, $J_1=2.4$ Hz, $J_2=3.7$ Hz, $J_3=13.6$ Hz, H15), 1.43 (3H, s, H10'), 1.77 (1H, dt, $J_1=4.1$ Hz, $J_2=13.3$ Hz, $J_3=13.3$ Hz, H15), 1.83 (2H, dt, $J_1=4.0$ Hz, $J_2=12.7$ Hz, $J_3=13.7$ Hz, H21), 1.94 (2H, ddd, $J_1=4.6$ Hz, $J_2=7.6$ Hz, $J_3=13.0$ Hz, H1), 2.10 (3H, bs, H7'), 2.20-2.23 (1H, m, H13), 2.23 (2H, ddd, $J_1=2.8$ Hz, $J_2=4.0$ Hz, $J_3=12.7$ Hz, H22), 2.43 (1H, ddd, $J_1=4.6$ Hz, $J_2=7.9$ Hz, $J_3=15.6$ Hz, H2), 2.50 (1H, ddd, $J_1=7.6$ Hz, $J_2=9.3$ Hz, $J_3=15.6$ Hz, H2), 2.53-2.61 (2H, m, H5'), 3.35-3.60 (2H, m, H2'), 3.68-3.85 (2H, m, H1'), 4.78 (1H, dt, $J_1=4.8$ Hz, $J_2=8.2$ Hz, $J_3=8.2$ Hz, H4'), 4.99 (1H, bs, H19), 5.36 (1H, bd, $J=8.2$ Hz, H4'-NH).

^{13}C NMR: δ 14.61 (q, C27), 15.69 (q, C7'), 15.87 (q, C26), 16.35 (q, C25), 19.60 (t, C6), 20.96 (q, C24), 21.52 (t, C11), 25.51 (t, C12), 26.57 (q, C30), 26.75 (q, C23), 28.30 (q, C10'), 29.17 (t, C15), 30.19 (t, C6'), 30.90 (t, C21), 31.68 (s, C20), 31.68 (t, C16), 31.88 (q, C29), 33.05 (t, C7), 33.85 (t, C5'), 33.91 (t, C22), 34.03 (t, C2), 36.88 (s, C10), 39.75 (t, C1), 40.84 (s, C8), 42.04 (d, C13), 43.00 (t, C2'), 44.04 (s, C14), 45.45 (t, C1'), 47.23 (s, C4), 48.91 (d, C4'), 50.10 (s, C17), 50.17 (d, C9), 54.84 (d, C5), 79.95 (s, C9'), 129.91 (d, C19), 140.78 (s, C18), 155.44 (s, C8'), 170.51 (s, C3'), 174.49 (s, C28), 218.04 (s, C3).

MS: m/z = 755 $[\text{M}+\text{H}]^+$. For $\text{C}_{44}\text{H}_{71}\text{N}_3\text{O}_5\text{S}$ (754.12) C 70.08, H 9.49, N 5.57, S 4.25, found C 70.12, H 9.47, N 5.55, S 4.27.



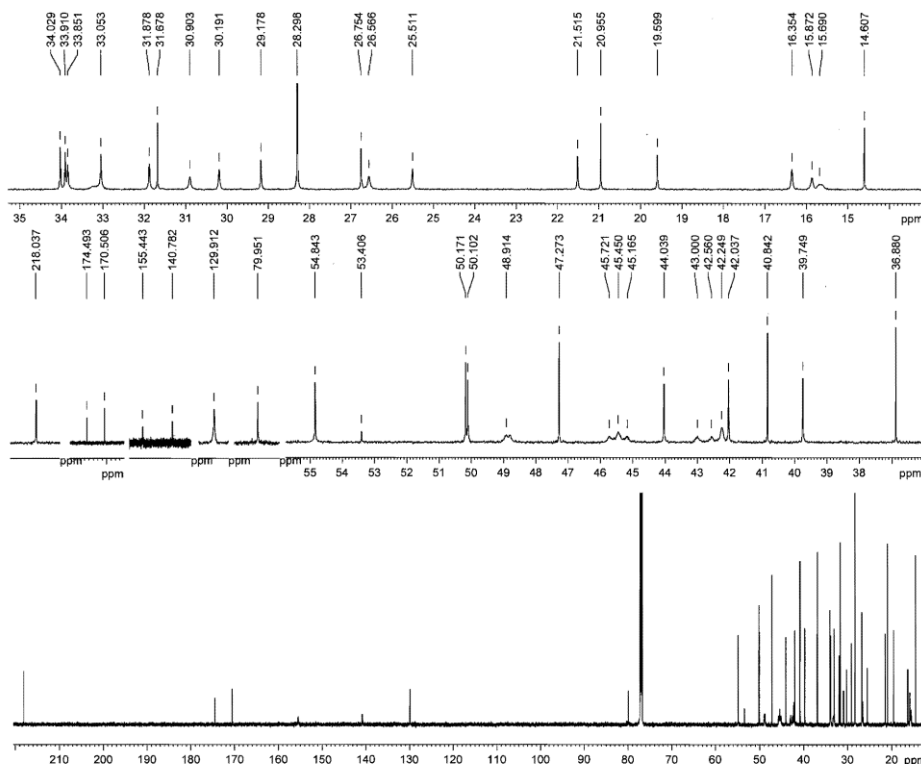
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in CDCl₃, ref=TMS
26.2.2019 DA

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PROCNO 1

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TE 298.1 K
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TD0 1

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PC 1.00

57132



Wimmer NS-4
in CDCl₃, ref=TMS
26.2.2019 DA

Current Data Parameters
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PROCNO 1

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TD0 1

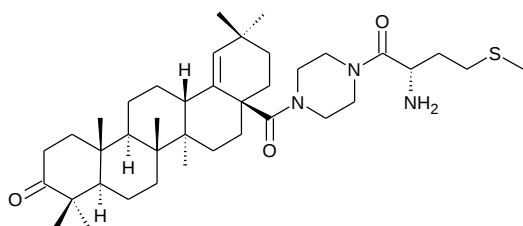
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57132

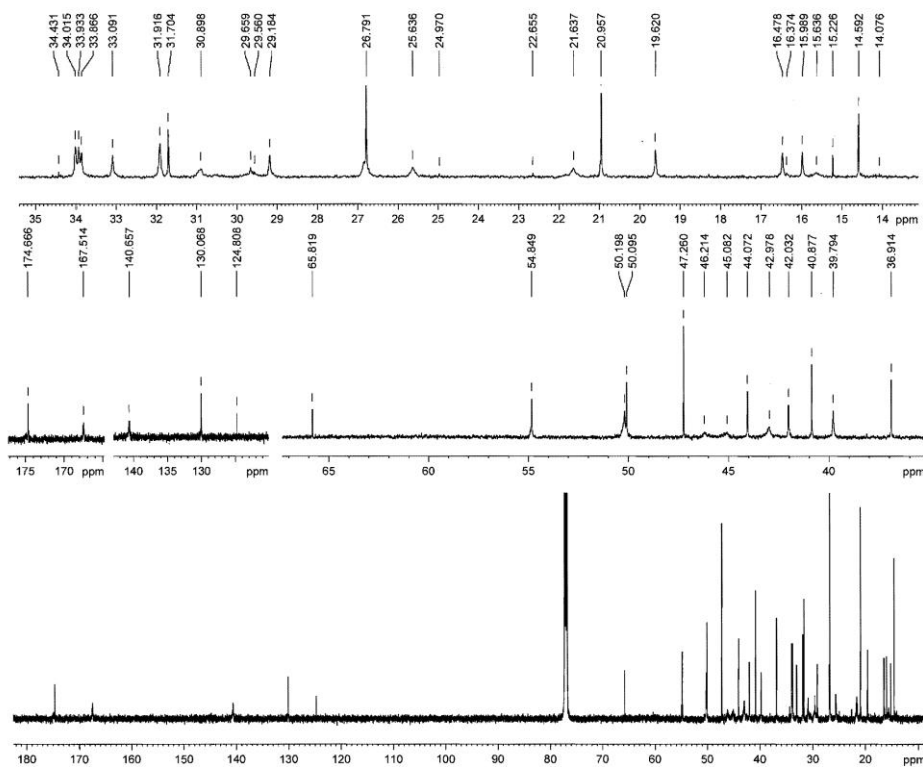
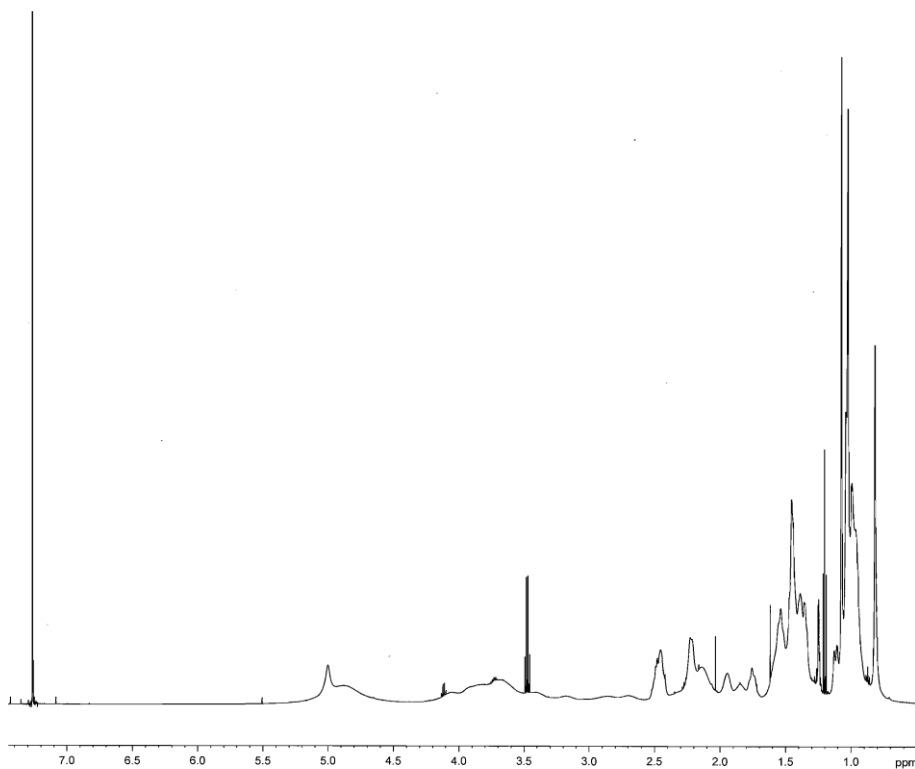
28-{4[(2S)-2-Amino-4-(methylsulfanyl)butanoyl]piperazin-1-yl}olean-18-en-3,28-dione (**9**)



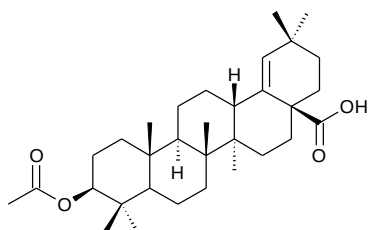
^1H NMR: δ 0.82 (3H, s, H27), 0.96 (3H, s, H25), 0.99 (3H, s, H26), 1.02 (3H, s, H24), 1.03 (3H, s, H23), 1.07 (3H, s, H29), 1.07 (3H, s, H30), 5.00 (1H, bs, H19).

^{13}C NMR: δ 14.59 (q, C27), 15.64 (q, C7'), 16.00 (q, C26), 16.48 (q, C25), 19.62 (t, C6), 20.96 (q, C24), 21.64 (t, C11), 25.64 (t, C12), 26.79 (q, C23), 27.79 (q, C30), 29.18 (t, C15), 29.66 (t, C5'), 30.90 (t, C21), 31.70 (s, C20), 31.92 (q, C29), 31.92 (t, C6'), 33.09 (t, C16), 33.87 (t, C7), 33.93 (t, C22), 34.02 (t, C2), 36.91 (s, C10), 39.79 (t, C1), 40.88 (s, C8), 42.03 (d, C13), 44.07 (s, C14), 45.08 (t, C2'), 46.21 (t, C1'), 47.26 (s, C4), 50.10 (s, C17), 50.10 (d, C4'), 50.20 (d, C9), 54.85 (d, C5), 130.07 (d, C19), 140.66 (s, C18), 167.51 (s, C3'), 174.67 (s, C28), 217.92 (s, C3).

MS: m/z = 655 $[\text{M}+\text{H}]^+$. For $\text{C}_{39}\text{H}_{63}\text{N}_3\text{O}_3\text{S}$ (654.00) calcd. C 71.62, H 9.71, N 6.43, S 4.90, found C 71.64, H 9.69, N 6.40, S 4.87.

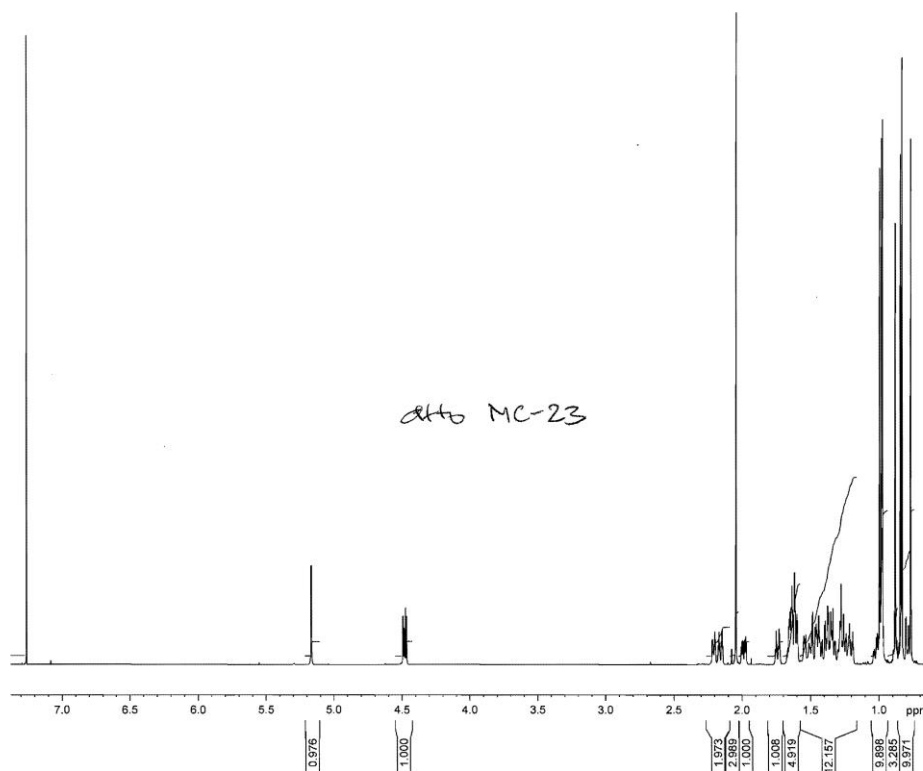


(3 β)-3-(Acetyloxy)olean-18-en-28-oic acid (**10**)



^1H NMR: δ 0.78 (3H, d, $J=0.5$ Hz, H27), 0.81 (1H, dd, $J_1=2.1$ Hz, $J_2=11.4$ Hz, H5), 0.84 (3H, s, H25), 0.85 (3H, s, H23), 0.90 (3H, d, $J=0.8$ Hz, H24), 0.98 (3H, s, H30), 1.00 (3H, d, $J=0.5$ Hz, H26), 1.00 (3H, s, H29), 1.03 (1H, bdt, $J_1=4.8$ Hz, $J_2=13.0$ Hz, $J_3=13.0$ Hz, H1), 1.21 (2H, ddd, $J_1=3.0$ Hz, $J_2=3.9$ Hz, $J_3=13.4$ Hz, H15), 1.75 (1H, dt, $J_1=3.6$ Hz, $J_2=3.6$ Hz, $J_3=13.2$ Hz, H1), 2.00 (2H, ddd, $J_1=3.4$ Hz, $J_2=6.2$ Hz, $J_3=13.9$ Hz, H7), 2.04 (3H, s, H2'), 2.17 (2H, ddd, $J_1=3.0$ Hz, $J_2=4.0$ Hz, $J_3=13.5$ Hz, H16), 2.23 (1H, ddd, $J_1=1.9$ Hz, $J_2=3.4$ Hz, $J_3=11.4$ Hz, H13), 4.49 (1H, dd, $J_1=5.3$ Hz, $J_2=11.3$ Hz, H3), 5.18 (1H, dd, $J_1=0.1$ Hz, $J_2=1.9$ Hz, H19).
 ^{13}C NMR: δ 14.85 (q, C27), 16.05 (q, C26), 16.51 (q, C25), 16.69 (q, C24), 18.19 (t, C6), 21.00 (t, C11), 21.23 (q, C2'), 23.75 (t, C2), 26.02 (t, C12), 27.96 (q, C23), 29.12 (q, C30), 29.41 (t, C15), 30.32 (q, C29), 32.08 (s, C20), 33.42 (t, C16), 33.55 (t, C7), 33.56 (t, C21), 34.58 (t, C22), 37.24 (s, C10), 37.89 (s, C8), 38.71 (t, C1), 40.79 (s, C14), 41.42 (d, C13), 42.67 (s, C4), 48.00 (s, C17), 51.17 (d, C9), 55.70 (d, C5), 81.00 (d, C3), 133.36 (d, C19), 136.77 (s, C18), 170.94 (s, C1'), 180.54 (s, C28).

MS: m/z = 439 $[\text{M}-\text{AcOH}+\text{H}]^+$, 516 $[\text{M}+\text{NH}_4]^+$ (ESI^+), 498 $[\text{M}-\text{H}]^+$ (ESI^-). For $\text{C}_{32}\text{H}_{50}\text{O}_4$ (498.74) calcd. C 77.06, H 10.10, found C 77.09, H 10.08.



Wimmer NS-6
in CDCl₃, ref=TMS
26.2.2019 DA

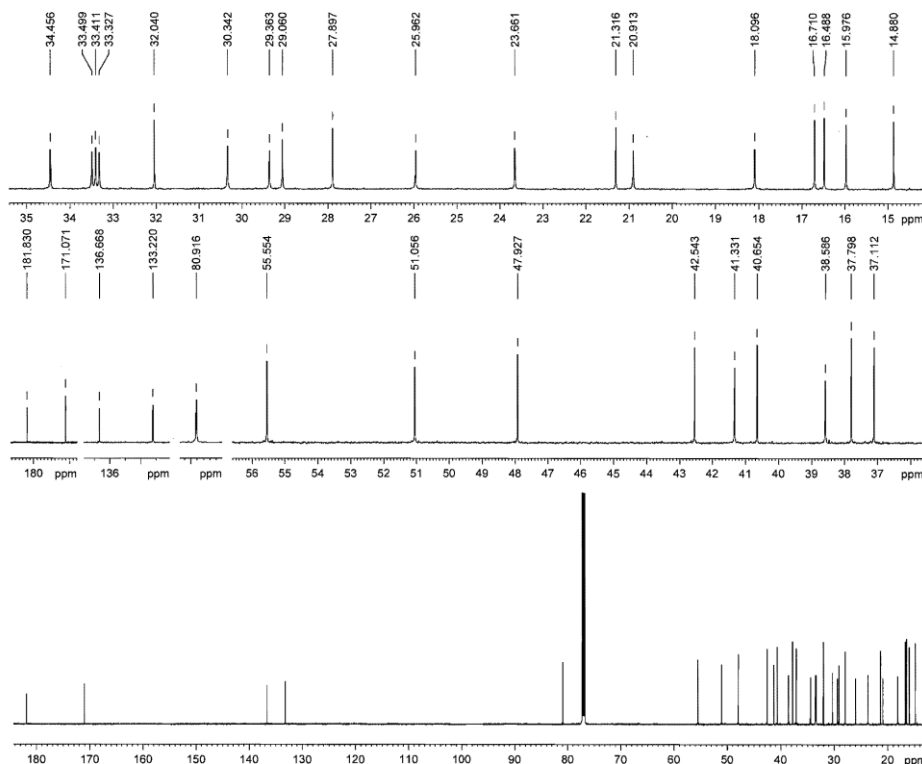
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FIDRES 0.125004 Hz
AQ 3.9998779 sec
RG 43.64
DW 104.267 usec
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TE 298.1 K
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TD0 1

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57131



Wimmer NS-6
in CDCl₃, ref=TMS
26.2.2019 DA

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PROCNO 1

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FIDRES 0.500032 Hz
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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

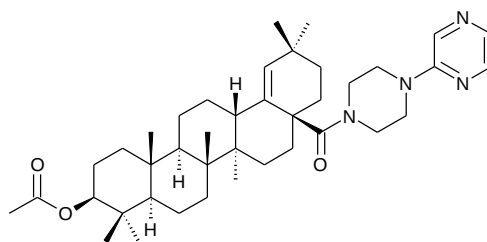
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F2 - Processing parameters
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WDW EM
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GB 0

57131

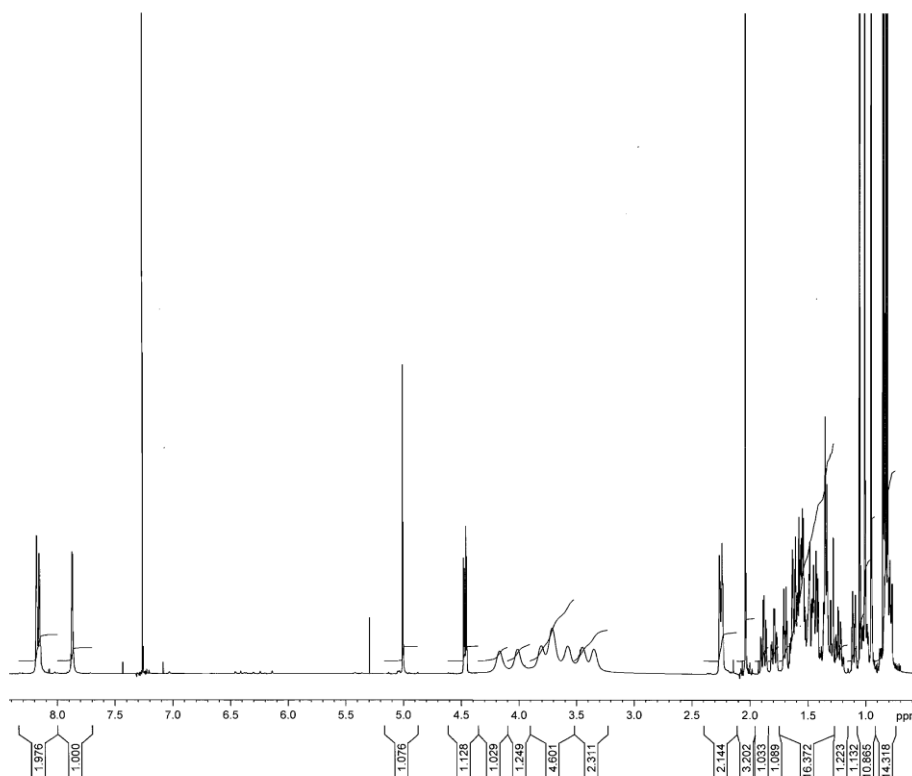
(3 β)-{28-Oxo-28-[4-(pyrazin-2-yl)piperazin-1-yl]olean-18-en-3-yl} acetate (**11**)



^1H NMR: δ 0.78 (2H, dd, $J_1=2.1$ Hz, $J_2=11.3$ Hz, H5), 0.81 (3H, d, $J=0.6$ Hz, H27), 0.82 (3H, s, H25), 0.84 (3H, bs, H24), 0.85 (3H, bs, H23), 0.95 (3H, bs, H26), 1.00 (3H, s, H30), 1.01 (1H, dt, $J_1=4.4$ Hz, $J_2=13.2$ Hz, $J_3=13.2$ Hz, H1), 1.05 (3H, s, H29), 1.10 (1H, ddd, $J_1=2.8$ Hz, $J_2=3.8$ Hz, $J_3=13.6$ Hz, H15), 1.24 (2H, dt, $J_1=4.0$ Hz, $J_2=12.5$ Hz, $J_3=12.5$ Hz, H11), 1.29 (1H, dd, $J_1=2.4$ Hz, $J_2=12.5$ Hz, H9), 1.70 (1H, dt, $J_1=3.6$ Hz, $J_2=3.6$ Hz, $J_3=13.2$ Hz, H1), 1.79 (1H, dt, $J_1=3.7$ Hz, $J_2=13.3$ Hz, $J_3=13.3$ Hz, H15), 1.88 (2H, dt, $J_1=3.2$ Hz, $J_2=12.7$ Hz, $J_3=12.7$ Hz, H16), 2.14 (3H, s, H2'), 2.23-2.27 (2H, m, H13), 2.23-2.27 (2H, m, H21), 3.34-4.21 (4H, m, H3'), 3.34-4.21 (4H, m, H4'), 4.47 (1H, dd, $J_1=5.2$ Hz, $J_2=11.3$ Hz, H3), 5.01 (1H, bt, $J=1.2$ Hz, H19), 7.87 (1H, bs, $J=3.3$ Hz, H7'), 8.16 (1H, bs, H6'), 8.18 (1H, bs, H8').

^{13}C NMR: δ 14.70 (q, C27), 16.00 (q, C26), 16.50 (q, C24), 16.50 (q, C25), 18.10 (t, C6), 20.90 (t, C11), 21.30 (q, C2'), 23.60 (t, C2), 25.50 (t, C12), 26.70 (q, C30), 27.90 (q, C23), 29.20 (t, C15), 30.90 (t, C16), 31.70 (q, C29), 31.90 (s, C20), 33.10 (t, C7), 33.90 (t, C21), 34.60 (t, C22), 37.10 (s, C10), 37.80 (s, C8), 38.50 (t, C1), 41.00 (s, C14), 41.90 (d, C13), 42.40 (t, C4'), 44.00 (s, C4), 44.60 (t, C3'), 50.10 (s, C17), 50.80 (d, C9), 55.50 (d, C5), 80.80 (d, C3), 127.90 (d, C6'), 129.40 (d, C7'), 129.80 (d, C19), 140.90 (s, C18), 143.30 (d, C8'), 154.90 (s, C5'), 171.00 (s, C1'), 174.70 (s, C28).

MS: m/z = 645 $[\text{M}+\text{H}]^+$. For $\text{C}_{40}\text{H}_{60}\text{N}_4\text{O}_3$ (644.93) calcd. C 74.49, H 9.38, N 8.69, found C 74.52, H 9.36, N 8.66.



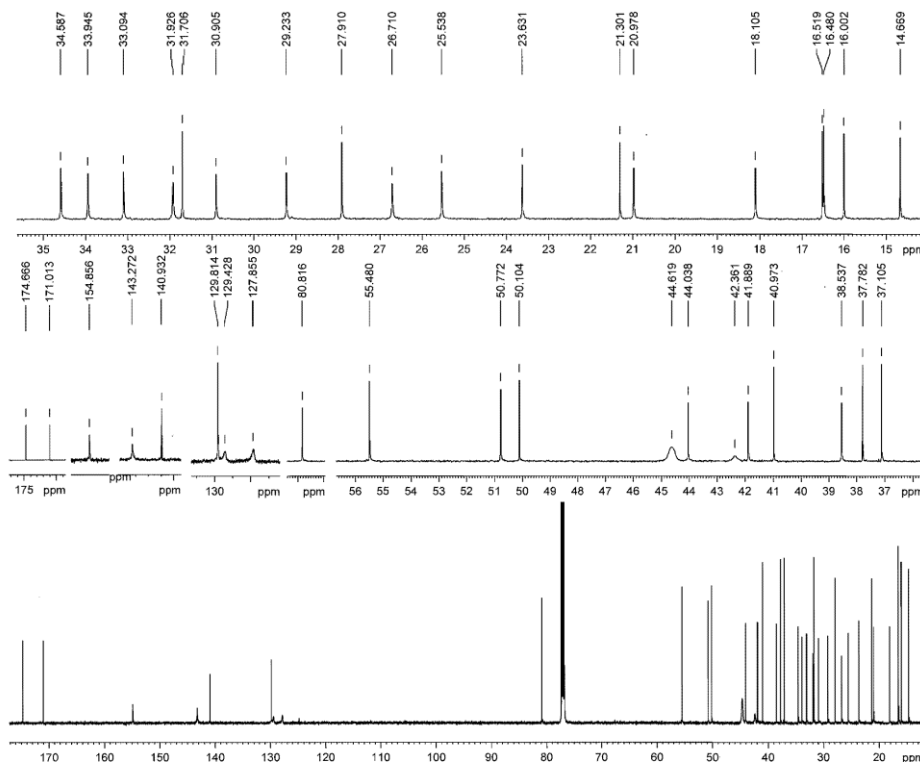
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10.5.2021 DA

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PROCNO 1

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F2 - Processing parameters
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60847



Wimmer-NS-19
in CDCl₃, ref=7.26 ppm
10.5.2021 DA

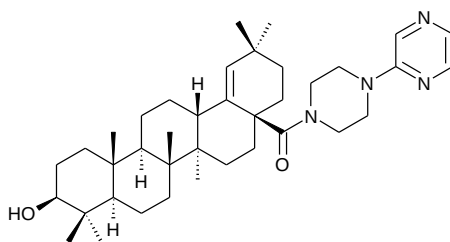
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D11 0.03000000 sec
TD0 1
SFO1 150.9178993 MHz
NUC1 13C
P0 4.00 usec
P1 12.00 usec
PLW1 104.66000366 W
SFO2 600.1324005 MHz
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60847

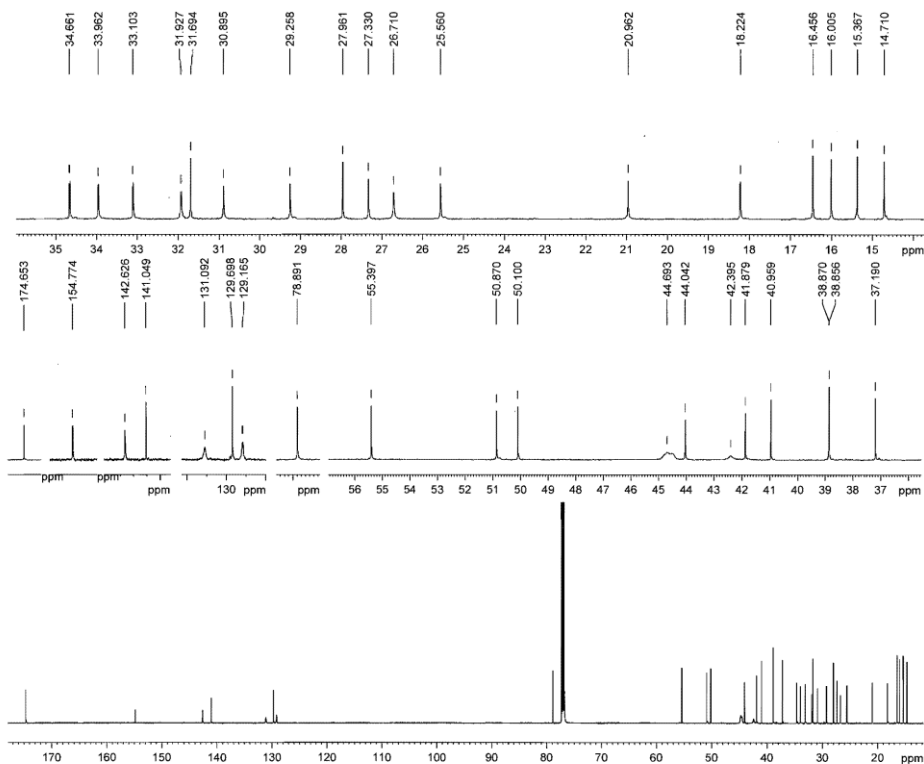
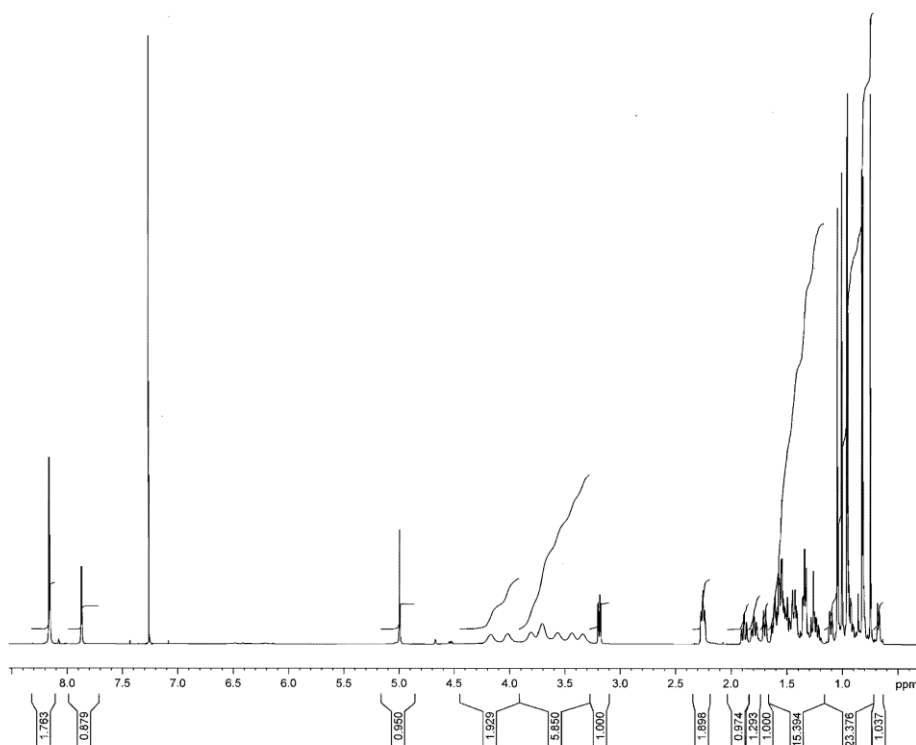
(3 β)-3-Hydroxy-28-[4-(pyrazin-2-yl)piperazin-1-yl]olean-18-en-28-one (**12**)



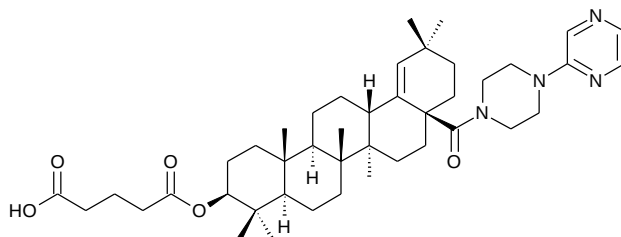
^1H NMR: δ 0.68 (1H, dd, $J_1=2.1$ Hz, $J_2=11.3$ Hz, H5), 0.75 (3H, s, H26), 0.81 (3H, d, $J=0.5$ Hz, H27), 0.82 (3H, s, H25), 0.92 (1H, dt, $J_1=4.4$ Hz, $J_2=13.2$ Hz, $J_3=13.2$ Hz, H1), 0.95 (3H, s, H24), 0.96 (3H, s, H23), 1.00 (3H, s, H30), 1.04 (3H, s, H29), 1.10 (1H, ddd, $J_1=2.8$ Hz, $J_2=3.9$ Hz, $J_3=13.6$ Hz, H15), 1.24 (2H, dt, $J_1=4.3$ Hz, $J_2=12.7$ Hz, $J_3=12.7$ Hz, H11), 1.27 (1H, dd, $J_1=2.3$ Hz, $J_2=12.7$ Hz, H9), 1.70 (1H, dt, $J_1=3.6$ Hz, $J_2=3.6$ Hz, $J_3=13.2$ Hz, H1), 1.80 (1H, dt, $J_1=3.9$ Hz, $J_2=13.4$ Hz, $J_3=13.4$ Hz, H15), 1.88 (2H, dt, $J_1=3.5$ Hz, $J_2=12.8$ Hz, $J_3=12.8$ Hz, H16), 2.26 (1H, ddd, $J_1=1.3$ Hz, $J_2=3.2$ Hz, $J_3=12.4$ Hz, H13), 3.19 (1H, dd, $J_1=4.7$ Hz, $J_2=11.6$ Hz, H3), 3.30-4.24 (4H, m, H1'), 3.30-4.24 (4H, m, H2'), 5.00 (1H, t, $J=1.3$ Hz, H19), 7.87 (1H, d, $J=2.2$ Hz, H5'), 8.15-8.17 (1H, m, H4'), 8.15-8.17 (1H, m, H6').

^{13}C NMR: δ 14.71 (q, C27), 15.36 (q, C26), 16.01 (q, C24), 16.46 (q, C25), 18.22 (t, C6), 20.96 (t, C11), 25.56 (t, C12), 26.71 (q, C30), 27.33 (t, C2), 27.96 (q, C23), 29.26 (t, C15), 30.90 (t, C16), 31.69 (s, C20), 31.93 (q, C29), 33.10 (t, C7), 33.66 (t, C22), 33.96 (t, C21), 37.19 (s, C10), 38.86 (s, C8), 38.87 (t, C1), 40.96 (d, C13), 41.88 (s, C4), 42.40 (t, C2'), 44.04 (s, C14), 44.69 (t, C1'), 50.10 (s, C17), 50.87 (d, C9), 55.40 (d, C5), 78.89 (d, C3), 129.17 (d, C4'), 129.70 (d, C19), 131.09 (d, C5'), 141.05 (s, C18), 142.63 (d, C6'), 154.77 (s, C3'), 174.65 (s, C28).

MS: m/z = 603 $[\text{M}+\text{H}]^+$. For $\text{C}_{38}\text{H}_{58}\text{N}_4\text{O}_2$ (602.89) calcd. C 75.70, H 9.70, N 9.29, found C 75.73, H 9.68, N 9.26.



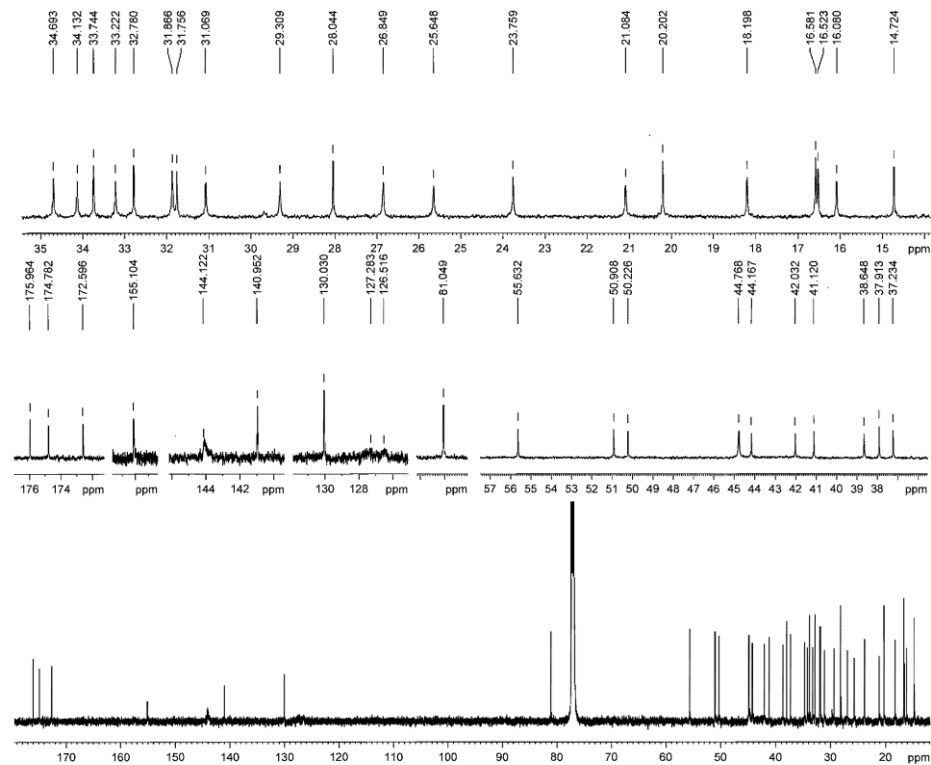
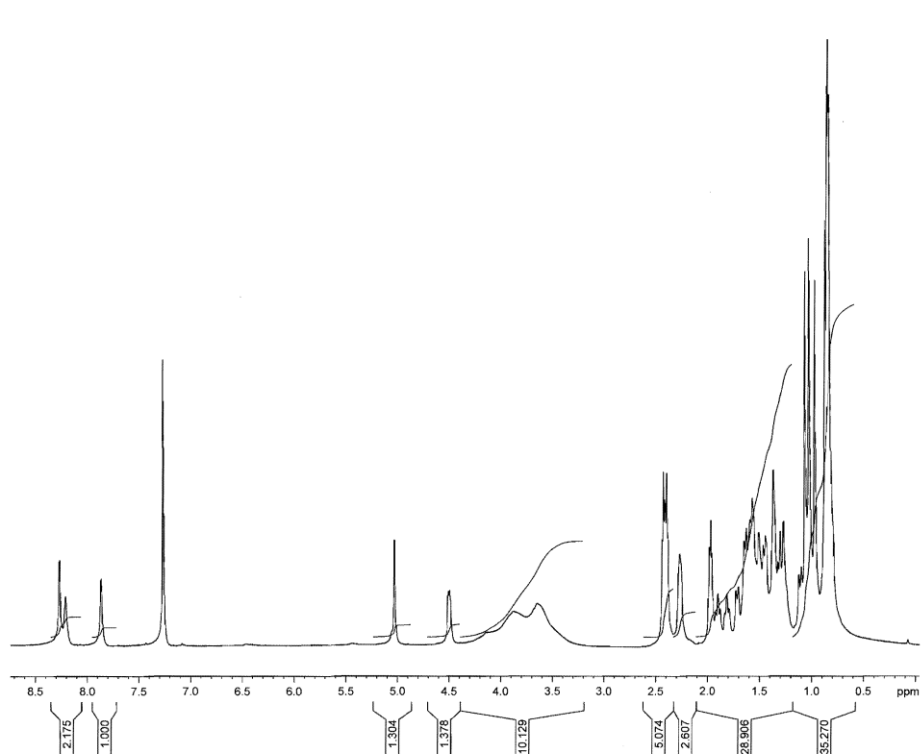
5-Oxo-5-((3 β)-28-oxo-28-[4-(pyrazin-2-yl)piperazin-1-yl]olean-18-en-3-yl oxy)pentanoic acid (**13**)



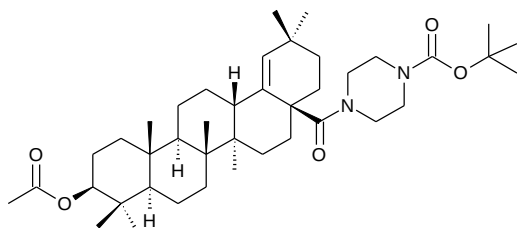
^1H NMR: δ 0.83 (3H, s, H25), 0.83 (3H, s, H27), 0.85 (3H, s, H24), 0.86 (3H, s, H23), 0.96 (3H, s, H26), 1.02 (3H, s, H30), 1.06 (3H, s, H29), 1.82 (2H, bt, $J=11.5$ Hz, H15), 1.89 (2H, bt, $J=12.1$ Hz, H16), 2.27 (1H, bs, H13), 2.27 (2H, bs, H21), 2.38 (4H, bt, $J=6.7$ Hz, H2'+H4'), 2.43 (4H, bs, $J=6.9$ Hz, H2'+H4'), 3.46-4.22 (8H, m, H6'+H7'), 4.50 (1H, dd, $J_1=4.0$ Hz, $J_2=10.3$ Hz, H3), 5.03 (1H, bs, H19), 7.87 (1H, bs, H10'), 8.21 (1H, bs, H9'), 8.26 (1H, bs, H11').

^{13}C NMR: δ 14.70 (q, C27), 16.10 (q, C26), 16.50 (q, C25), 16.60 (q, C24), 18.20 (t, C6), 20.20 (t, C11), 21.10 (t, C3'), 23.80 (t, C2), 25.60 (t, C12), 26.80 (q, C30), 28.00 (q, C23), 29.30 (t, C15), 31.10 (t, C16), 31.80 (q, C29), 31.90 (s, C20), 32.80 (t, C2'), 33.20 (t, C7), 33.70 (t, C4'), 34.10 (t, C21), 34.70 (t, C22), 37.20 (t, C10), 37.90 (s, C8), 38.60 (t, C1), 41.10 (s, C14), 42.00 (d, C13), 44.20 (s, C4), 44.80 (t, C6'+7'), 50.20 (s, C17), 50.90 (d, C9), 55.60 (d, C5), 81.00 (d, C3), 126.50 (d, C9'), 127.30 (s, C10'), 130.00 (d, C19), 141.00 (s, C18), 144.10 (d, C11'), 155.10 (s, C8'), 172.60 (s, C1'), 174.80 (s, C28), 176.00 (s, C5').

MS: $m/z = 717$ $[\text{M}+\text{H}]^+$. For $\text{C}_{43}\text{H}_{64}\text{N}_4\text{O}_5$ (716.99) calcd. C 72.03, H 9.00, N 7.81, found C 72.05, H 8.97, N 7.79.

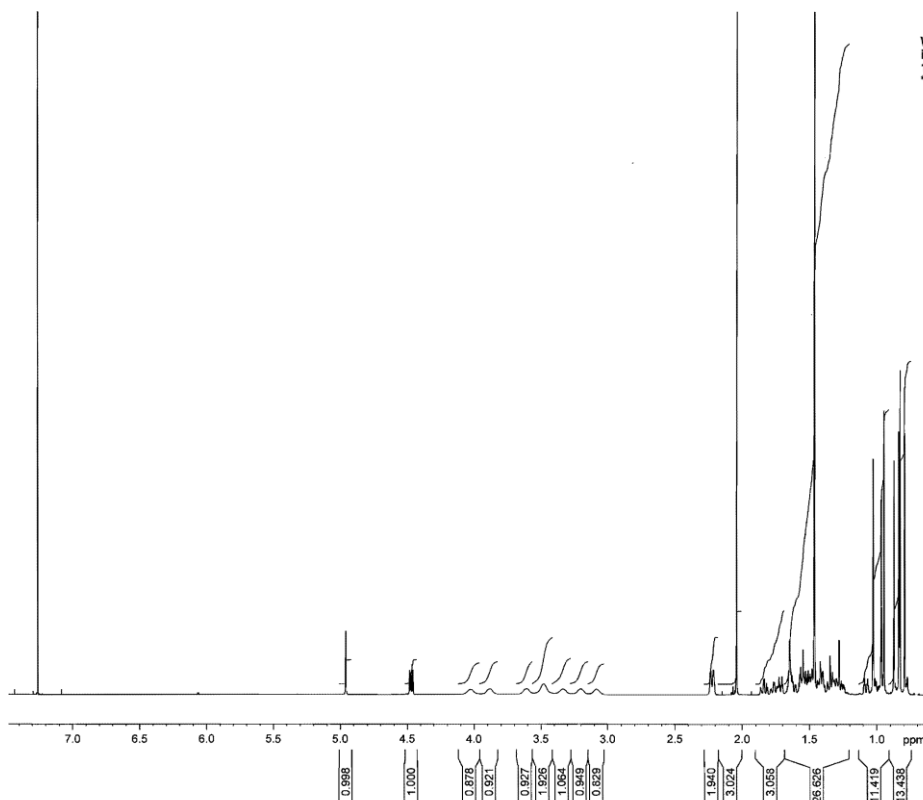


tert-Butyl-4-[(3 β)-3-(acetyloxy)-28-oxoolean-18-en-28-yl]piperazin-1-carboxylate (**14**):



^1H NMR: δ 0.78 (1H, dd, $J_1=1.9$ Hz, $J_2=11.1$ Hz, H5), 0.79 (3H, d, $J=0.7$ Hz, H27), 0.83 (3H, s, H25), 0.84 (3H, s, H23), 0.87 (3H, d, $J=0.5$ Hz, H24), 0.95 (3H, s, H26), 0.97 (3H, s, H30), 1.01 (1H, dt, $J_1=4.9$ Hz, $J_2=13.0$ Hz, $J_3=13.0$ Hz, H1), 1.03 (3H, s, H29), 1.08 (1H, ddd, $J_1=2.7$ Hz, $J_2=3.9$ Hz, $J_3=13.7$ Hz, H15), 1.46 (3H, bs, H7'), 1.71 (1H, dt, $J_1=3.5$ Hz, $J_2=3.5$ Hz, $J_3=13.1$ Hz, H1), 1.76 (1H, dt, $J_1=3.9$ Hz, $J_2=13.7$ Hz, $J_3=13.7$ Hz, H15), 1.84 (2H, dt, $J_1=4.2$ Hz, $J_2=13.6$ Hz, $J_3=13.6$ Hz, H21), 2.04 (3H, s, H2'), 2.22 (2H, ddd, $J_1=2.8$ Hz, $J_2=4.3$ Hz, $J_3=12.7$ Hz, H7), 2.23 (1H, ddd, $J_1=1.1$ Hz, $J_2=3.2$ Hz, $J_3=12.6$ Hz, H13), 3.08-3.48 (1H, bs, H4'), 3.20-3.61 (1H, bs, H3'), 3.34-3.89 (1H, bs, H4'), 3.48-4.03 (1H, bs, H3'), 4.47 (1H, dd, $J_1=5.4$ Hz, $J_2=11.1$ Hz, H3).

^{13}C NMR: δ 14.67 (q, C27), 15.96 (q, C26), 16.49 (q, C25), 16.52 (q, C24), 18.11 (t, C6), 20.97 (t, C11), 21.31 (q, C2'), 23.64 (t, C2), 25.45 (t, C12), 26.58 (q, C30), 27.91 (q, C23), 28.37 (q, C7'), 29.26 (t, C15), 30.88 (t, C21), 31.65 (s, C20), 31.93 (q, C29), 33.10 (t, C16), 33.99 (t, C7), 34.57 (t, C22), 37.11 (s, C10), 37.78 (s, C8), 38.54 (t, C1), 40.96 (s, C14), 41.73 (d, C13), 42.75 (t, C4'), 43.98 (s, C4), 45.23 (t, C3'), 50.06 (s, C17), 50.78 (d, C9), 55.48 (d, C5), 80.20 (s, C6'), 80.85 (d, C3), 129.54 (d, C19), 141.05 (s, C18), 154.52 (s, C5'), 171.03 (s, C1'), 174.60 (s, C28). MS: m/z = 667 $[\text{M}+\text{H}]^+$. For $\text{C}_{41}\text{H}_{66}\text{N}_2\text{O}_5$ (666.97) calcd. C 73.83, H 9.97, N 4.20, found C 73.85, H 9.95, N 4.22.



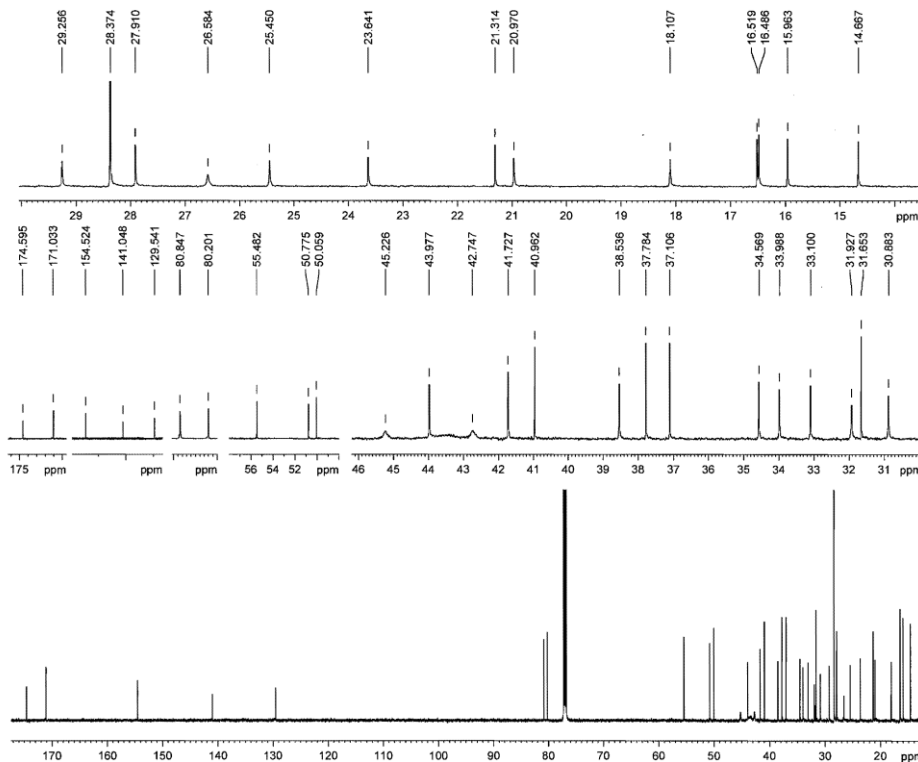
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in CDCl₃, ref=TMS
15.7.2019 DA

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PROCNO 1

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TD0 1

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57804



Wimmer NS-7
in CDCl₃, ref=TMS
15.7.2019 DA

Current Data Parameters
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PROCNO 1

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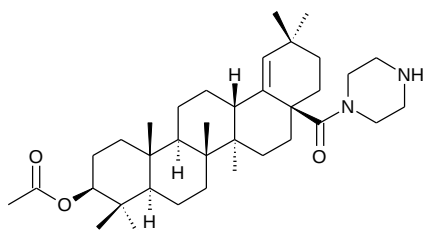
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PCPD2 70.00 usec
PLW2 6.00000000 W
PLW12 0.09918400 W
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GB 0

57804

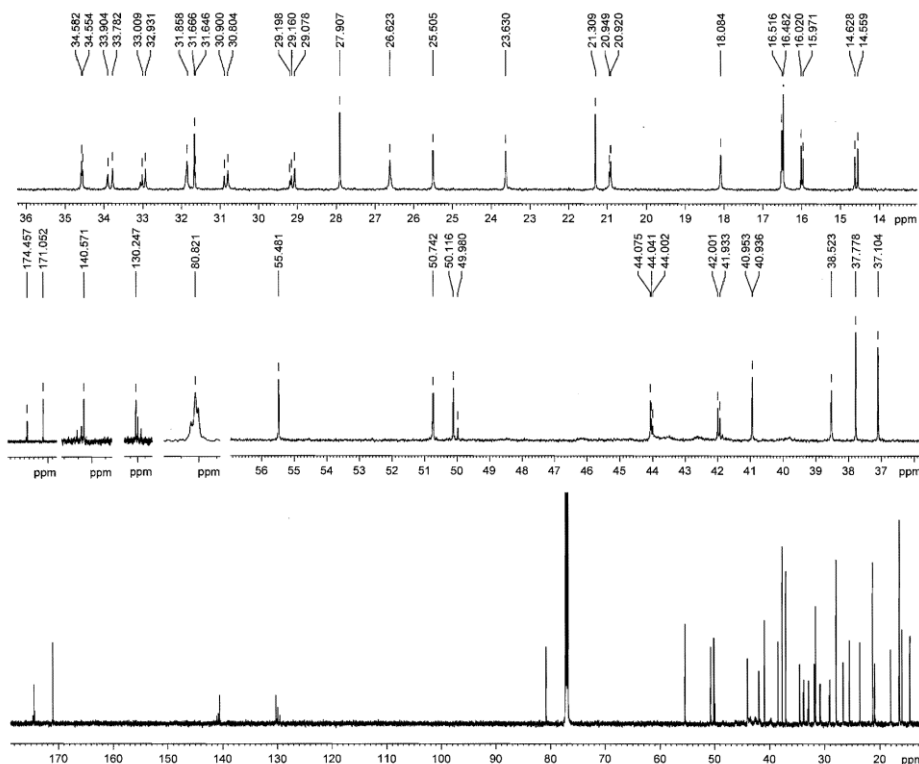
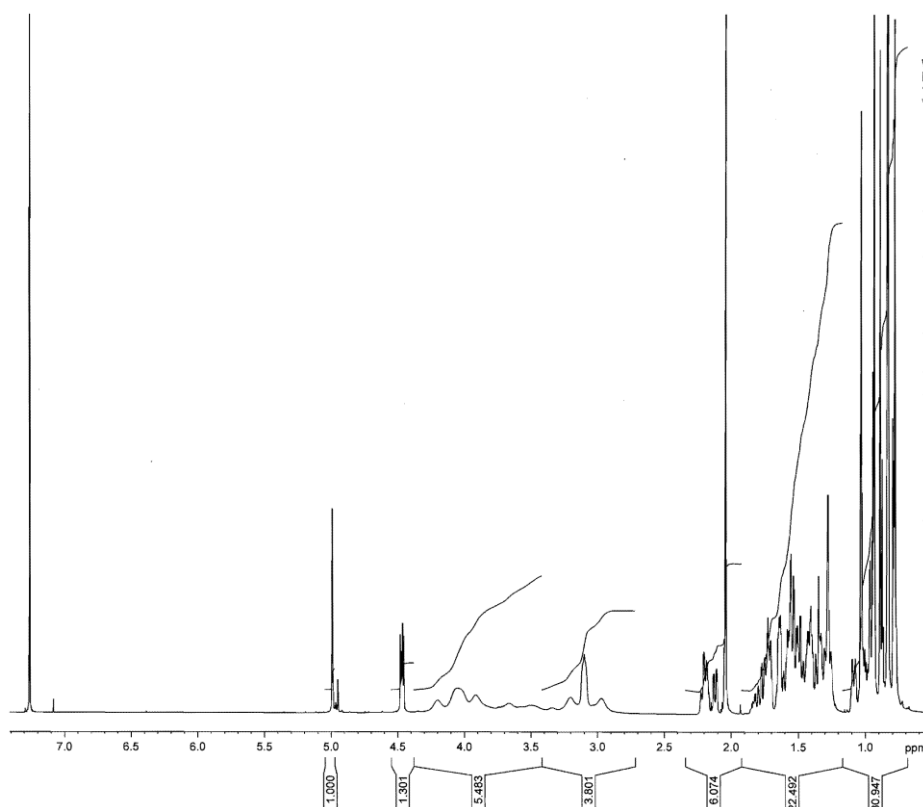
(3 β)-28-Oxo-28-(piperazin-1-yl)olean-18-en-3-yl acetate (**15**)



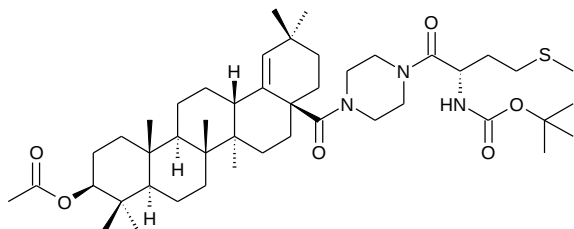
^1H NMR: δ 0.78 (1H, dd, $J_1=1.8$ Hz, $J_2=11.4$ Hz, H5), 0.78 (3H, s, H27), 0.83 (3H, s, H25), 0.84 (3H, s, H23), 0.89 (3H, s, H24), 0.93 (3H, s, H30), 0.93 (3H, s, H26), 1.03 (3H, s, H29), 1.06-1.11 (2H, m, H15), 1.77 (2H, ddd, $J_1=4.2$ Hz, $J_2=7.6$ Hz, $J_3=13.0$ Hz, H1), 2.04 (3H, s, H2'), 2.12 (1H, dd, $J_1=1.9$ Hz, $J_2=11.9$ Hz, H13), 2.21 (2H, ddd, $J_1=2.9$ Hz, $J_2=4.2$ Hz, $J_3=12.8$ Hz, H7), 2.93-3.27 (2H, m, H4'), 3.83-4.23 (2H, m, H3'), 4.47 (2H, dd, $J_1=5.8$ Hz, $J_2=11.0$ Hz, H3), 4.99 (1H, bs, H19).

^{13}C NMR: δ 14.63 (q, C27), 16.02 (q, C26), 16.48 (q, C25), 16.52 (q, C24), 18.08 (t, C6), 20.92 (t, C11), 21.31 (q, C2'), 23.63 (t, C2), 25.51 (t, C12), 26.62 (q, C30), 27.91 (q, C23), 29.08 (t, C15), 30.80 (t, C21), 31.67 (s, C20), 31.86 (q, C29), 32.93 (t, C16), 33.90 (t, C7), 34.58 (t, C22), 37.10 (s, C10), 37.78 (s, C8), 38.52 (t, C1), 40.94 (s, C14), 42.00 (d, C13), 42.64+39.86 (t, C4'), 43.75+43.45 (t, C3'), 44.08 (s, C4), 50.12 (s, C17), 50.74 (d, C9), 55.48 (d, C5), 80.82 (d, C3), 130.25 (d, C19), 140.57 (s, C18), 171.05 (s, C1'), 174.46 (s, C28).

MS: m/z = 567 $[\text{M}+\text{H}]^+$. For $\text{C}_{36}\text{H}_{58}\text{N}_2\text{O}_3$ (566.86) calcd. C 76.28, H 10.31 N 4.94, found C 76.30, H 10.29, N 4.92.

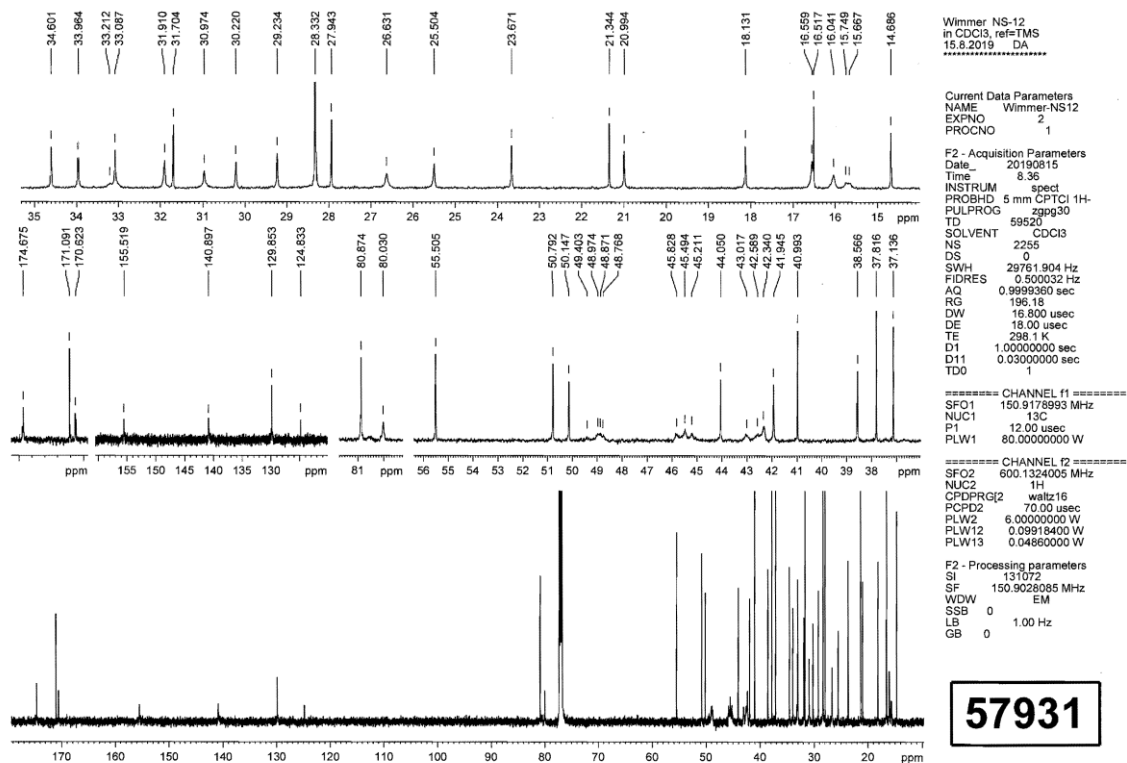
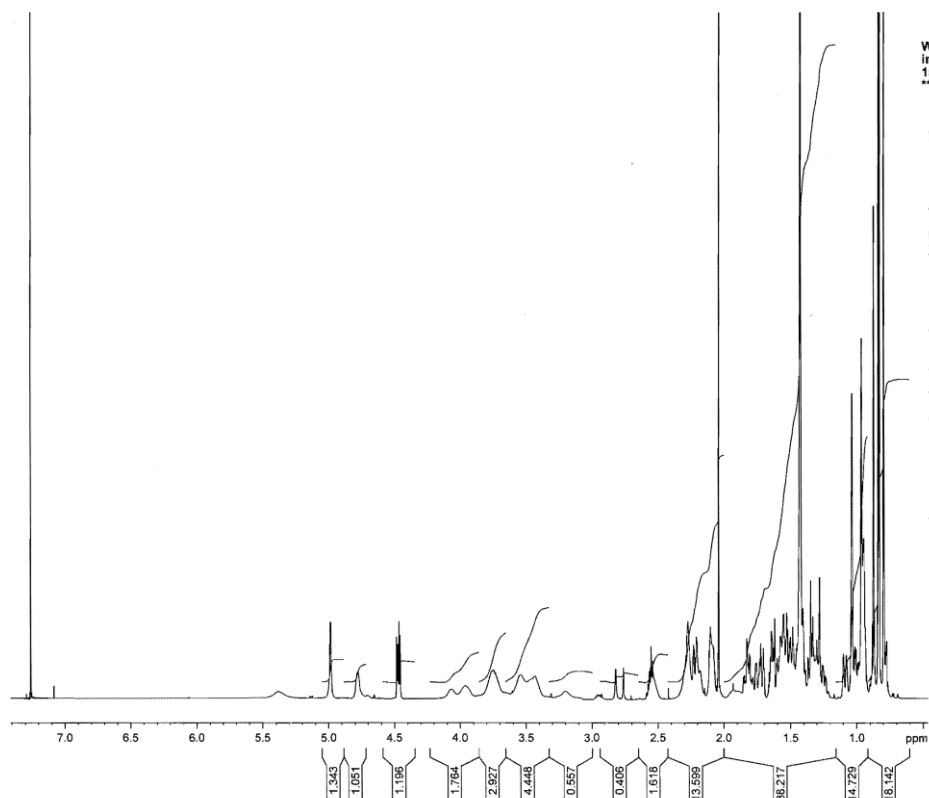


(3 β)-28-{4-[*N*-(*tert*-Butoxycarbonyl)-L-methionyl]piperazin-1-yl}-28-oxoolean-18-en-3-yl acetate (**16**)

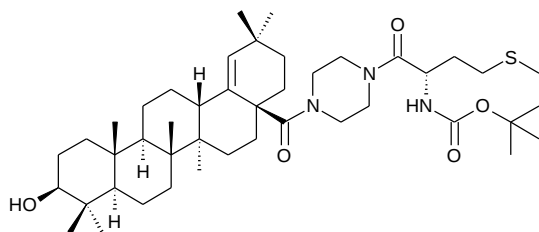


^1H NMR: δ 0.79 (1H, dd, $J_1=1.7$ Hz, $J_2=11.1$ Hz, H5), 0.80 (3H, s, H27), 0.83 (3H, s, H23), 0.83 (3H, s, H25), 0.87 (3H, s, H24), 0.95 (3H, s, H26), 0.97 (3H, s, H30), 1.01 (1H, dt, $J_1=4.8$ Hz, $J_2=13.0$ Hz, $J_3=13.0$ Hz, H1), 1.04 (3H, s, H29), 1.09 (1H, ddd, $J_1=2.9$ Hz, $J_2=3.6$ Hz, $J_3=13.5$ Hz, H15), 1.43 (3H, s, H12'), 1.72 (1H, dt, $J_1=3.6$ Hz, $J_2=3.6$ Hz, $J_3=13.2$ Hz, H1), 1.76 (1H, dt, $J_1=4.2$ Hz, $J_2=13.7$ Hz, $J_3=13.7$ Hz, H15), 1.83 (2H, dt, $J_1=3.7$ Hz, $J_2=13.5$ Hz, $J_3=13.5$ Hz, H21), 2.04 (3H, s, H2'), 2.10 (3H, s, H9'), 2.22 (2H, ddd, $J_1=2.7$ Hz, $J_2=4.0$ Hz, $J_3=12.7$ Hz, H7), 3.39-3.62 (2H, m, H4'), 3.67-3.72 (2H, m, H3'), 4.47 (1H, dd, $J_1=5.3$ Hz, $J_2=11.1$ Hz, H3), 4.76-4.78 (1H, m, H6').

^{13}C NMR: δ 14.69 (q, C27), 16.04 (q, C26), 16.52 (q, C25), 16.56 (q, C24), 18.13 (t, C6), 20.99 (t, C11), 23.67 (t, C2), 25.50 (t, C12), 26.63 (q, C30), 27.94 (q, C23), 29.23 (t, C15), 30.22 (t, C21), 31.70 (s, C20), 31.91 (q, C29), 33.09 (t, C16), 33.96 (t, C7), 34.60 (t, C22), 37.14 (s, C10), 37.82 (s, C8), 38.57 (t, C1), 40.99 (s, C14), 41.95 (d, C13), 44.05 (s, C4), 50.15 (s, C17), 50.79 (d, C9), 55.51 (d, C5), 80.87 (d, C3), 129.85 (d, C19), 140.90 (s, C18), 174.68 (s, C28).
MS: $m/z = 799$ $[\text{M}+\text{H}]^+$. For $\text{C}_{46}\text{H}_{75}\text{N}_3\text{O}_6\text{S}$ (798.17) calcd. C 69.22, H 9.47, N 5.26, S 4.02, found C 69.25, H 9.46, N 5.24, S 4.04.



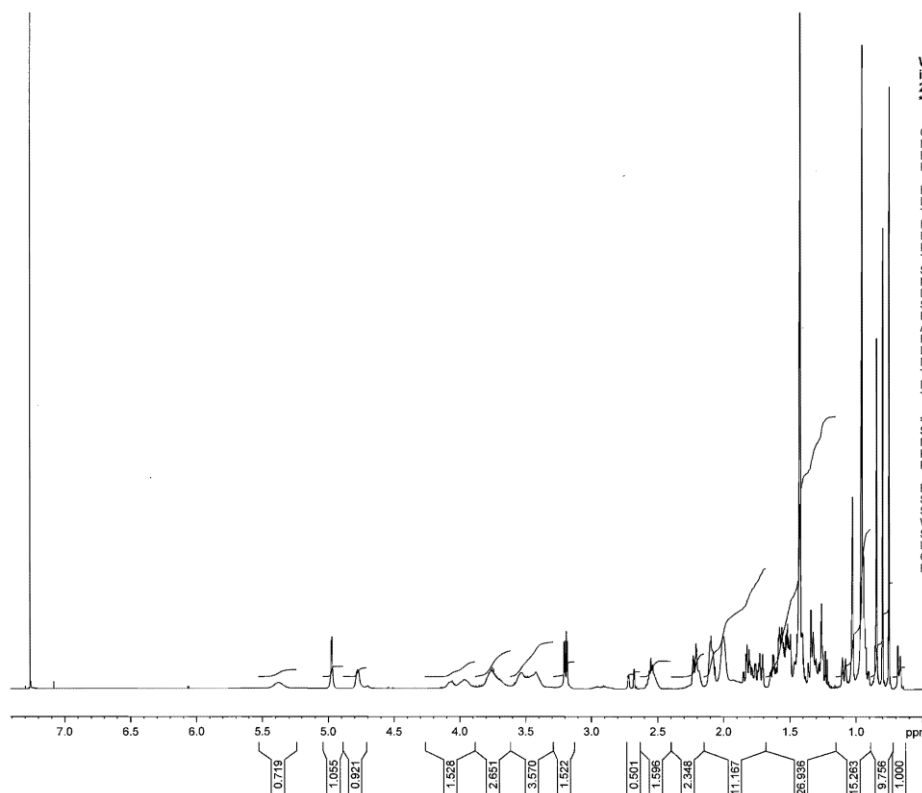
tert-Butyl-[(2*S*)-1-{4-[(3 β)-3-hydroxy-28-oxoolean-18-en-28-yl]piperazin-1-yl}-4-(methylsulphanyl)-1-oxobutan-2-yl] carbamate (**17**)



¹H NMR: δ 0.68 (1H, dd, $J_1=1.9$ Hz, $J_2=11.5$ Hz, H5), 0.75 (3H, s, H26), 0.80 (3H, s, H27), 0.84 (3H, s, H24), 0.95 (3H, s, H25), 0.96 (3H, s, H23), 0.96 (3H, s, H30), 1.03 (3H, s, H29), 1.09 (1H, ddd, $J_1=2.9$ Hz, $J_2=3.8$ Hz, $J_3=13.7$ Hz, H15), 1.43 (3H, s, H10'), 1.72 (2H, dt, $J_1=3.5$ Hz, $J_2=3.5$ Hz, $J_3=13.1$ Hz, H1), 1.76 (1H, dt, $J_1=3.9$ Hz, $J_2=13.7$ Hz, $J_3=13.7$ Hz, H15), 1.83 (2H, dt, $J_1=4.1$ Hz, $J_2=13.6$ Hz, $J_3=13.6$ Hz, H21), 2.09 (3H, bs, H7'), 2.22 (2H, ddd, $J_1=2.7$ Hz, $J_2=4.0$ Hz, $J_3=12.6$ Hz, H7), 3.20 (1H, dd, $J_1=4.8$ Hz, $J_2=11.5$ Hz, H3), 3.38-3.59 (2H, m, H2'), 3.67-3.81 (2H, m, H1'), 4.76-4.79 (1H, m, H4').

¹³C NMR: δ 14.69 (q, C27), 15.38 (q, C26), 15.71 (q, C7'), 16.01 (q, C25), 16.46 (q, C24), 18.21 (t, C6), 20.95 (t, C11), 25.49 (t, C12), 26.60 (q, C30), 27.28 (t, C2), 27.95 (q, C23), 28.30 (q, C10'), 29.21 (t, C15), 30.18 (t, C21), 30.93 (t, C6'), 31.66 (s, C20), 31.87 (q, C29), 33.05 (t, C16), 33.11 (t, C5'), 33.63 (t, C22), 33.93 (t, C7), 37.18 (s, C10), 38.84 (s, C8), 38.84 (t, C1), 40.94 (s, C14), 41.92 (d, C13), 42.54 (t, C2'), 44.02 (s, C4), 45.42 (t, C1'), 48.79 (d, C4'), 50.11 (s, C17), 50.85 (d, C9), 55.38 (d, C5), 78.97 (d, C3), 79.97 (s, C9'), 129.76 (d, C19), 140.93 (s, C18), 155.48 (s, C8'), 170.57 (s, C3'), 174.61 (s, C28).

MS: m/z = 756 [M+H]⁺, 778 [M+Na]⁺. For C₄₄H₇₃N₃O₅S (756.13) calcd. C 69.89, H 9.73, N 5.56, S 4.24, found C 69.92, H 9.71, N 5.57, S 4.22.



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in CDCl₃, ref=TMS
22.10.2019 DA

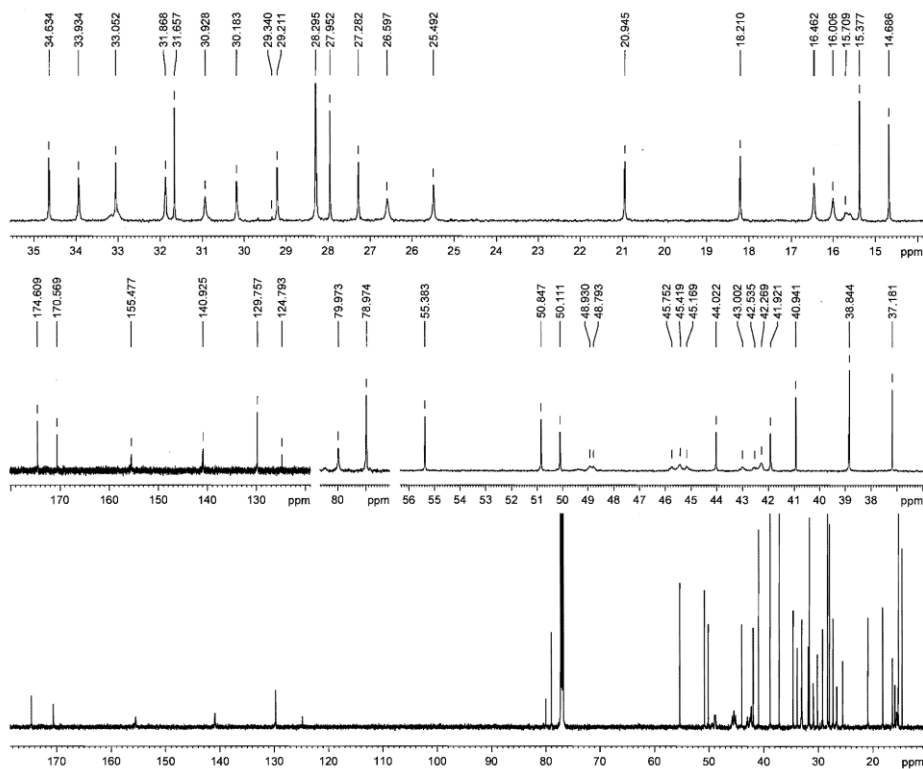
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PROCNO 1

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TD0 1

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58324



Wimmer NS-13
in CDCl₃, ref=TMS
22.10.2019 DA

Current Data Parameters
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PROCNO 1

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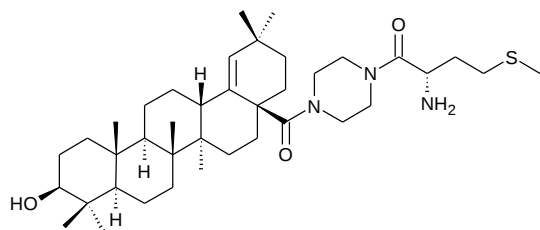
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PLW12 0.09918400 W
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GB 0

58324

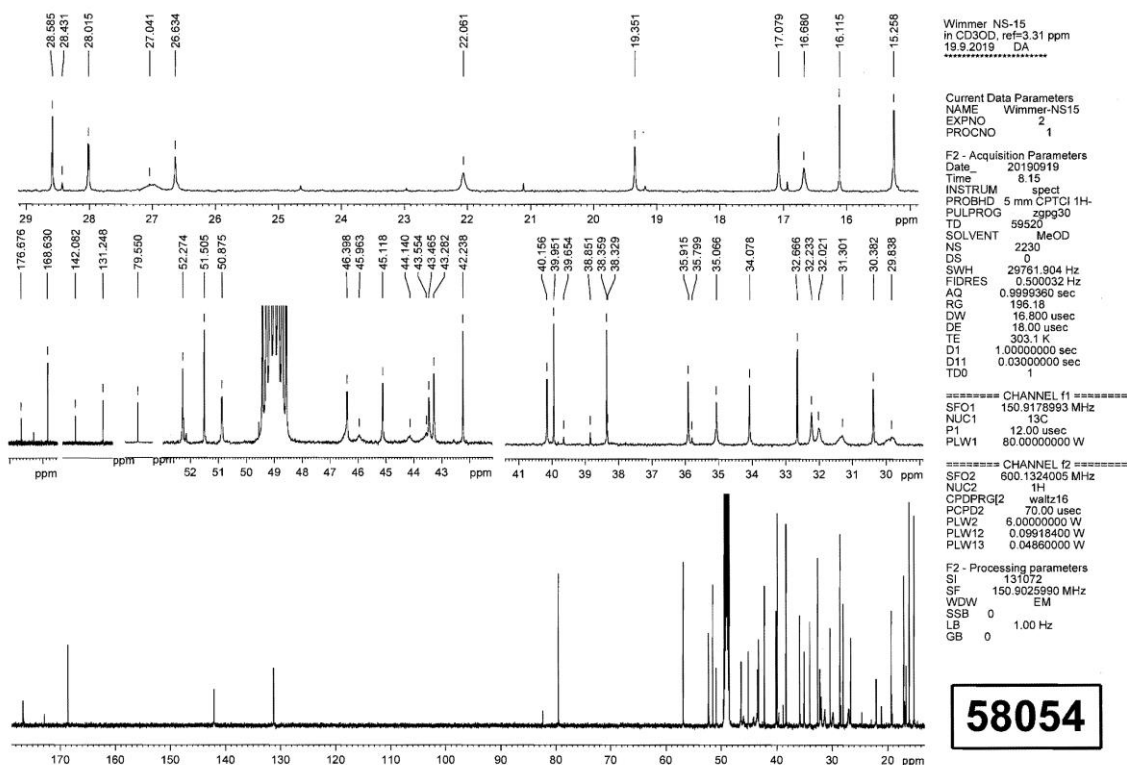
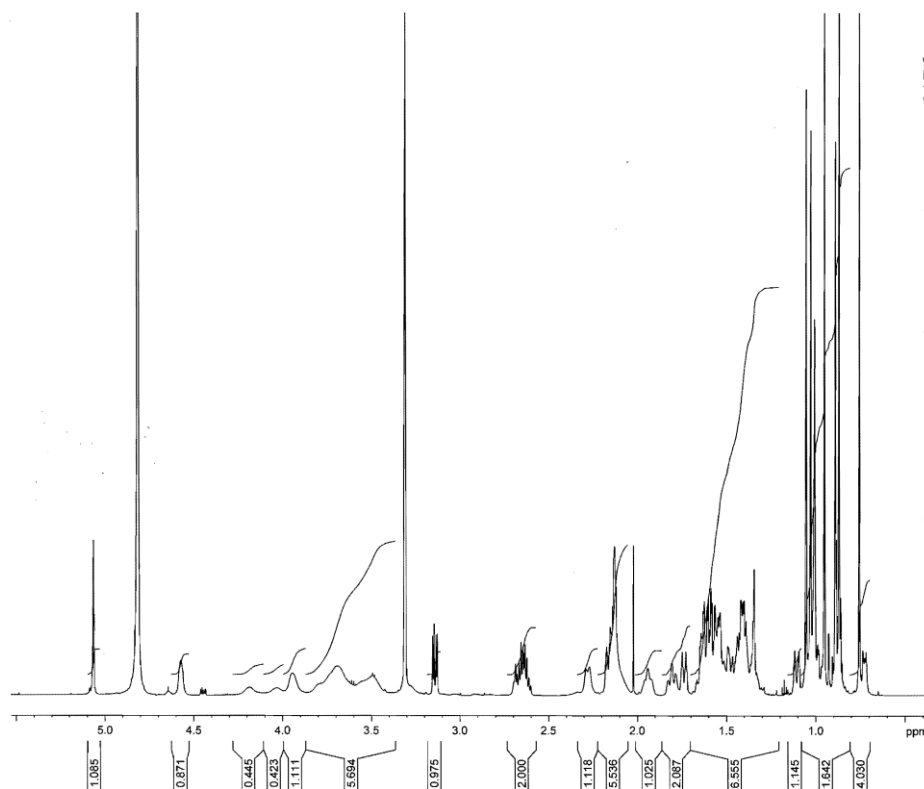
(3 β)-28-{4-[(2S)-2-Amino-4-(methylsulphanyl)butanoyl]piperazin-1-yl}-3-hydroxyolean-18-en-28-one (**18**)



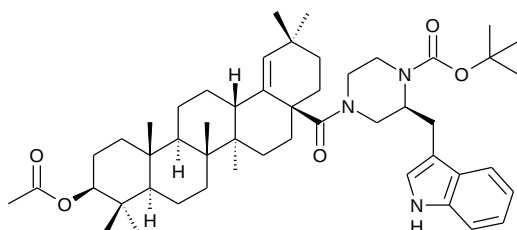
^1H NMR: δ 0.73 (1H, dd, $J_1=1.9$ Hz, $J_2=11.2$ Hz, H5), 0.76 (3H, s, H25), 0.87 (3H, s, H27), 0.89 (3H, s, H23), 0.95 (3H, s, H24), 1.01 (3H, s, H26), 1.03 (3H, s, H30), 1.05 (3H, s, H29), 1.11 (1H, ddd, $J_1=3.0$ Hz, $J_2=3.5$ Hz, $J_3=13.5$ Hz, H15), 1.74 (2H, dt, $J_1=3.5$ Hz, $J_2=3.5$ Hz, $J_3=13.0$ Hz, H1), 1.80 (1H, dt, $J_1=3.5$ Hz, $J_2=13.7$ Hz, $J_3=13.7$ Hz, H15), 1.95 (2H, bt, $J=12.8$ Hz, H21), 2.10 (3H, bs, H7'), 2.16 (2H, ddd, $J_1=2.7$ Hz, $J_2=3.6$ Hz, $J_3=12.6$ Hz, H16), 2.28 (1H, bd, $J=11.4$ Hz, H13), 2.60-2.70 (2H, m, H5'), 3.14 (1H, dd, $J_1=4.8$ Hz, $J_2=11.5$ Hz, H3), 3.38-3.58 (2H, m, H2'), 3.60-3.75 (2H, m, H1'), 4.56-4.58 (1H, m, H4'), 5.07 (1H, bs, H19).

^{13}C NMR: δ 15.26 (q, C7'), 15.26 (q, C27), 16.12 (q, C26), 16.68 (q, C25), 17.08 (q, C24), 19.35 (t, C6), 22.06 (t, C11), 26.63 (t, C2), 27.05 (q, C30), 28.02 (t, C12), 28.59 (q, C23), 29.84 (t, C5'), 30.38 (t, C15), 31.30 (t, C6'), 32.02 (t, C21), 32.23 (q, C29), 32.67 (s, C20), 34.08 (t, C7), 35.07 (t, C16), 35.92 (t, C22), 38.36 (s, C10), 39.95 (s, C14), 39.95 (s, C8), 40.16 (t, C1), 42.24 (s, C4), 43.28 (d, C13), 45.12 (t, C2'), 46.40 (t, C1'), 50.88 (d, C4'), 52.27 (d, C9), 56.90 (d, C5), 57.51 (s, C17), 79.55 (d, C3), 131.25 (d, C19), 142.08 (s, C18), 168.63 (s, C3'), 176.68 (s, C28).

MS: m/z = 656 $[\text{M}+\text{H}]^+$. For $\text{C}_{39}\text{H}_{65}\text{N}_3\text{O}_3\text{S}$ (656.02) calcd. C 71.40, H 9.99, N 6.41, S 4.89, found C 71.43, H 9.98, N 6.39, S 4.90.



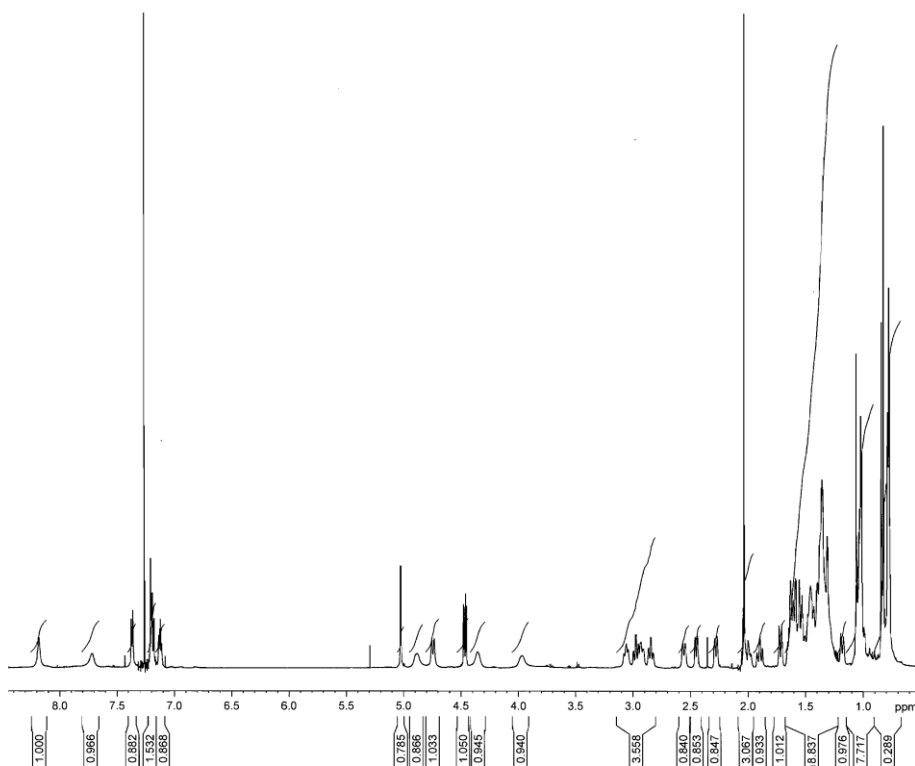
tert-Butyl-[(2*S*)-4-[(3 β)-3-(acetyloxy)-28-oxoolean-18-en-28-yl]-2-(1*H*-indol-3-ylmethyl)piperazine] 1-carboxylate (**19**)



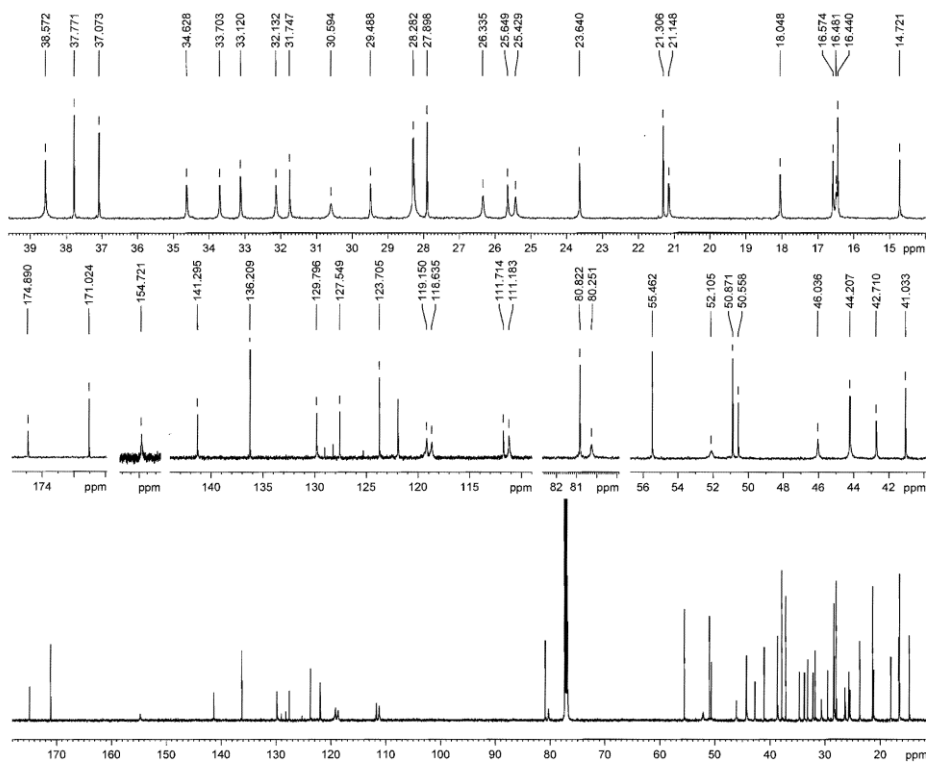
^1H NMR: δ 0.77 (3H, s, H26), 0.79 (3H, s, H25), 0.82 (3H, s, H23), 0.84 (3H, s, H27), 1.01 (1H, dt, $J_1=4.2$ Hz, $J_2=13.5$ Hz, $J_3=13.5$ Hz, H1), 1.02 (3H, s, H30), 1.03 (3H, s, H24), 1.06 (3H, s, H29), 1.18 (1H, dt, $J_1=3.4$ Hz, $J_2=3.4$ Hz, $J_3=13.0$ Hz, H15), 1.37 (3H, s, H7'), 1.72 (1H, dt, $J_1=3.6$ Hz, $J_2=3.6$ Hz, $J_3=13.2$ Hz, H1), 1.90 (2H, dt, $J_1=3.6$ Hz, $J_2=13.5$ Hz, $J_3=13.5$ Hz, H16), 2.00 (1H, bdt, $J_1=3.5$ Hz, $J_2=13.2$ Hz, $J_3=13.2$ Hz, H15), 2.03 (3H, s, H19'), 2.45 (1H, dd, $J_1=2.2$ Hz, $J_2=12.6$ Hz, H13), 2.56 (1H, dd, $J_1=3.3$ Hz, $J_2=13.4$ Hz, H1'), 2.85 (1H, dt, $J_1=2.7$ Hz, $J_2=12.5$ Hz, $J_3=12.5$ Hz, H4'), 2.93 (1H, bdd, $J_1=4.6$ Hz, $J_2=13.5$ Hz, H8'), 2.98 (1H, dt, $J_1=3.0$ Hz, $J_2=13.2$ Hz, $J_3=13.2$ Hz, H3'), 3.06 (1H, bdd, $J_1=9.8$ Hz, $J_2=13.5$ Hz, H8'), 3.97 (1H, bs, H3'), 4.36 (1H, bs, H2'), 4.46 (1H, dd, $J_1=5.0$ Hz, $J_2=11.5$ Hz, H3), 4.74 (1H, d, $J=13.4$ Hz, H1'), 4.89 (1H, bd, $J=13.2$ Hz, H4'), 5.03 (1H, bs, H19), 7.13 (1H, t, $J=7.5$ Hz, H15'), 7.19 (1H, t, $J=7.8$ Hz, H14'), 7.20 (1H, bs, H10'), 7.37 (1H, bd, $J=8.0$ Hz, H13'), 7.72 (1H, bs, H16'), 8.18 (1H, bs, H11'-NH).

^{13}C NMR: δ 14.70 (q, C27), 16.40 (q, C26), 16.50 (q, C25), 16.60 (q, C24), 18.00 (t, C6), 21.10 (t, C11), 21.30 (q, C19'), 23.60 (t, C2), 25.40 (t, C8'), 25.60 (t, C12), 26.30 (q, C30), 27.90 (q, C23), 28.30 (q, C7'), 29.50 (t, C15), 30.60 (t, C16), 31.70 (s, C20), 32.10 (q, C29), 33.10 (t, C7), 33.70 (t, C21), 34.60 (t, C22), 37.10 (s, C10), 37.80 (s, C8), 38.60 (t, C1), 38.60 (t, C3'), 41.00 (s, C14), 42.70 (d, C13), 44.20 (s, C4), 44.20 (t, C1'), 46.00 (t, C4'), 50.60 (s, C17), 50.90 (d, C9), 52.10 (d, C2'), 55.50 (d, C5), 80.30 (s, C6'), 80.80 (d, C3), 111.20 (d, C13'), 111.70 (s, C9'), 118.60 (d, C16'), 119.20 (d, C15'), 121.90 (d, C14'), 123.70 (d, C10'), 127.50 (s, C17'), 129.80 (d, C19), 136.20 (s, C12'), 141.30 (s, C18), 154.70 (s, C5'), 171.00 (s, C18'), 174.90 (s, C28).

MS: m/z = 796 $[\text{M}+\text{H}]^+$. For $\text{C}_{50}\text{H}_{73}\text{N}_3\text{O}_5$ (796.13) calcd. C 75.43, H 9.24, N 5.28, found C 75.45, H 9.22, N 5.25.

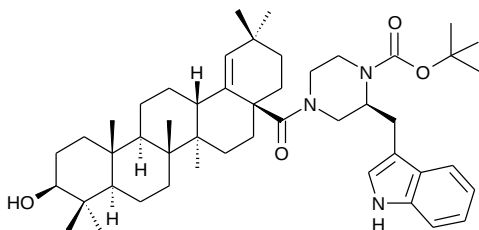


62232



62232

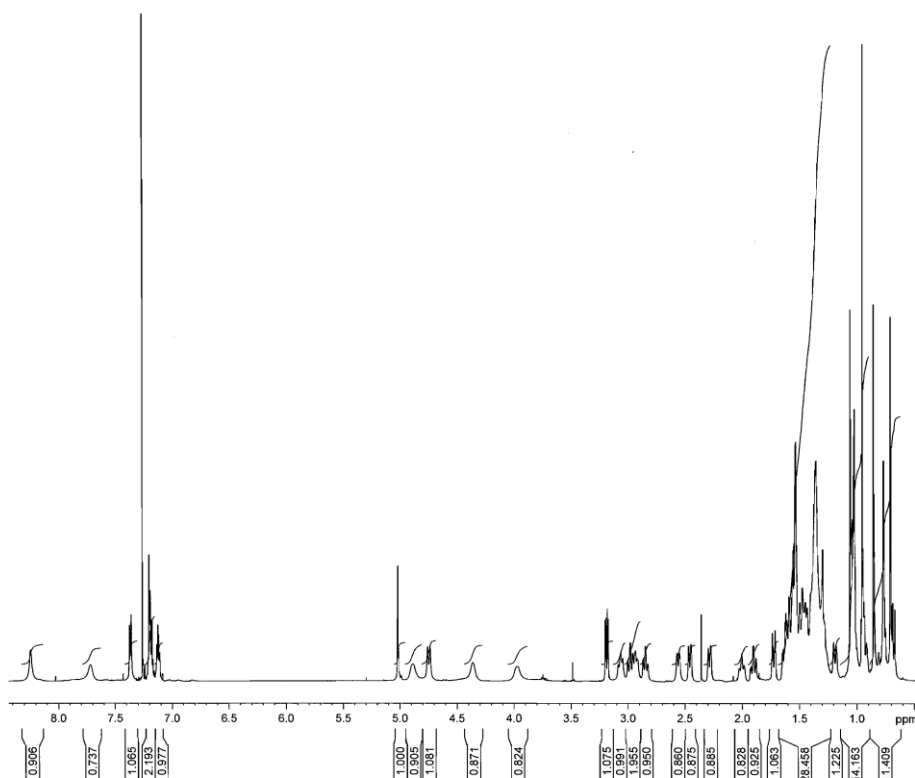
tert-Butyl-[(2*S*)-4-[(3 β)-3-hydroxy-28-oxoolean-18-en-28-yl]-2-(1*H*-indol-3-ylmethyl)piperazine] 1-carboxylate (**20**)



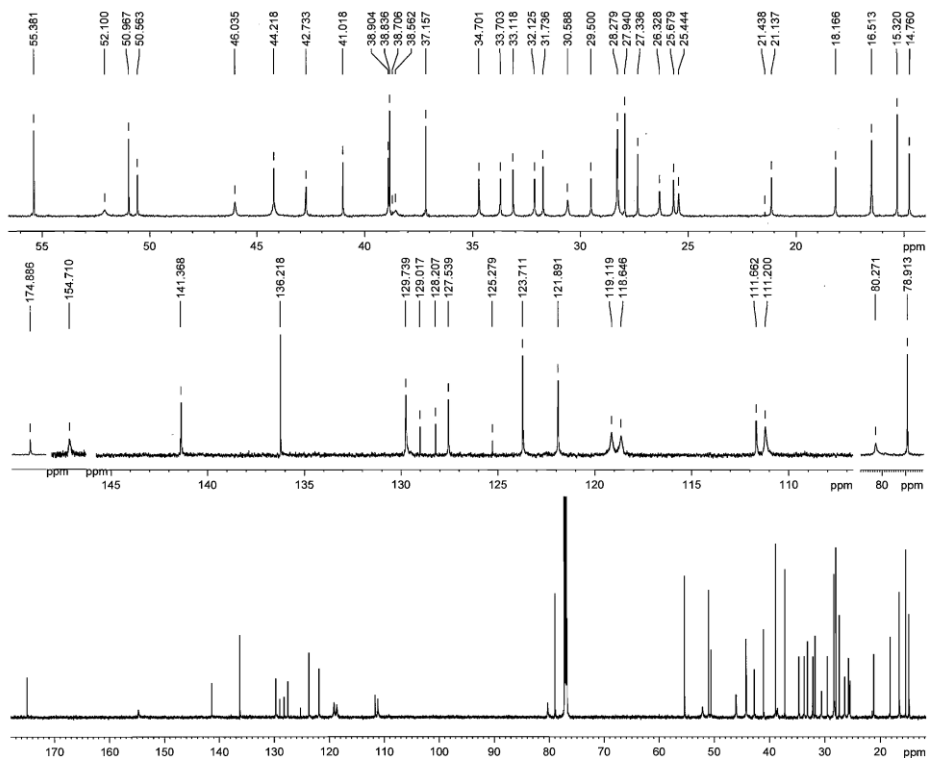
¹H NMR: δ 0.68 (1H, dd, $J_1=1.8$ Hz, $J_2=11.7$ Hz, H5), 0.71 (3H, s, H25), 0.76 (3H, s, H26), 0.85 (3H, s, H27), 0.93 (1H, dt, $J_1=4.5$ Hz, $J_2=13.5$ Hz, $J_3=13.5$ Hz, H1), 0.95 (3H, s, H23), 1.02 (3H, s, H30), 1.04 (3H, s, H24), 1.06 (3H, s, H29), 1.36 (3H, s, H7'), 1.73 (1H, dt, $J_1=3.6$ Hz, $J_2=13.7$ Hz, $J_3=13.7$ Hz, H1), 1.90 (2H, dt, $J_1=3.5$ Hz, $J_2=13.7$ Hz, $J_3=13.7$ Hz, H16), 2.00 (2H, dt, $J_1=3.5$ Hz, $J_2=13.4$ Hz, $J_3=13.4$ Hz, H15), 2.46 (1H, dd, $J_1=2.0$ Hz, $J_2=12.7$ Hz, H13), 2.56 (1H, dd, $J=3.4$ Hz, H1'), 2.85 (1H, bdt, $J_1=3.1$ Hz, $J_2=12.5$ Hz, $J_3=12.5$ Hz, H4'), 2.93 (1H, dd, $J_1=4.6$ Hz, $J_2=13.7$ Hz, H8'), 2.98 (1H, dt, $J_1=2.9$ Hz, $J_2=12.8$ Hz, $J_3=12.8$ Hz, H3'), 3.07 (1H, dd, $J_1=9.0$ Hz, $J_2=13.7$ Hz, H8'), 3.19 (1H, dd, $J_1=4.6$ Hz, $J_2=11.6$ Hz, H3), 3.96 (1H, dt, $J_1=2.9$ Hz, $J_2=12.8$ Hz, $J_3=12.8$ Hz, H3'), 4.37 (1H, bs, H2'), 4.74 (1H, bd, $J=13.5$ Hz, H1'), 4.88 (1H, bs, H4'), 5.02 (1H, s, H19), 7.12 (1H, bt, $J=7.6$ Hz, H15'), 7.18 (1H, bt, $J=7.8$ Hz, H14'), 7.20 (1H, bs, H10'), 7.37 (1H, d, $J=8.0$ Hz, H13'), 7.92 (1H, bd, $J=7.4$ Hz, H16'), 8.24 (1H, bs, H11'-NH).

¹³C NMR: δ 14.80 (q, C27), 15.30 (q, C26), 16.50 (q, C24), 16.50 (q, C25), 18.20 (t, C6), 21.10 (t, C11), 25.40 (t, C8'), 25.70 (t, C12), 26.30 (q, C30), 27.30 (t, C2), 27.90 (q, C23), 28.30 (q, C7'), 29.50 (t, C15), 30.60 (t, C16), 31.70 (s, C20), 32.10 (q, C29), 33.10 (t, C7), 33.70 (t, C22), 34.70 (t, C21), 37.20 (s, C10), 38.70 (t, C3'), 38.80 (s, C8), 38.90 (t, C1), 41.00 (s, C4), 42.70 (d, C13), 44.20 (s, C14), 44.20 (t, C1'), 46.00 (t, C4'), 50.00 (s, C17), 51.00 (d, C9), 52.10 (d, C2'), 55.40 (d, C5), 78.90 (d, C3), 80.30 (s, C6'), 111.20 (d, C13'), 111.70 (s, C9'), 118.60 (d, C16'), 119.10 (d, C15'), 121.90 (d, C14'), 123.70 (d, C10'), 127.50 (s, C17'), 129.70 (d, C19), 136.20 (s, C12'), 141.40 (s, C18), 154.70 (s, C5'), 174.90 (s, C28).

MS: $m/z = 754$ $[M+H]^+$. For $C_{48}H_{71}N_3O_4$ (754.10) calcd. C 76.45, H 9.49, N 5.57, found C 76.48, H 9.48, N 5.54.

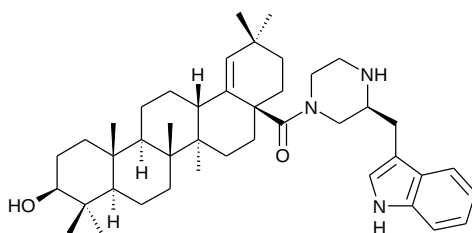


61804



61804

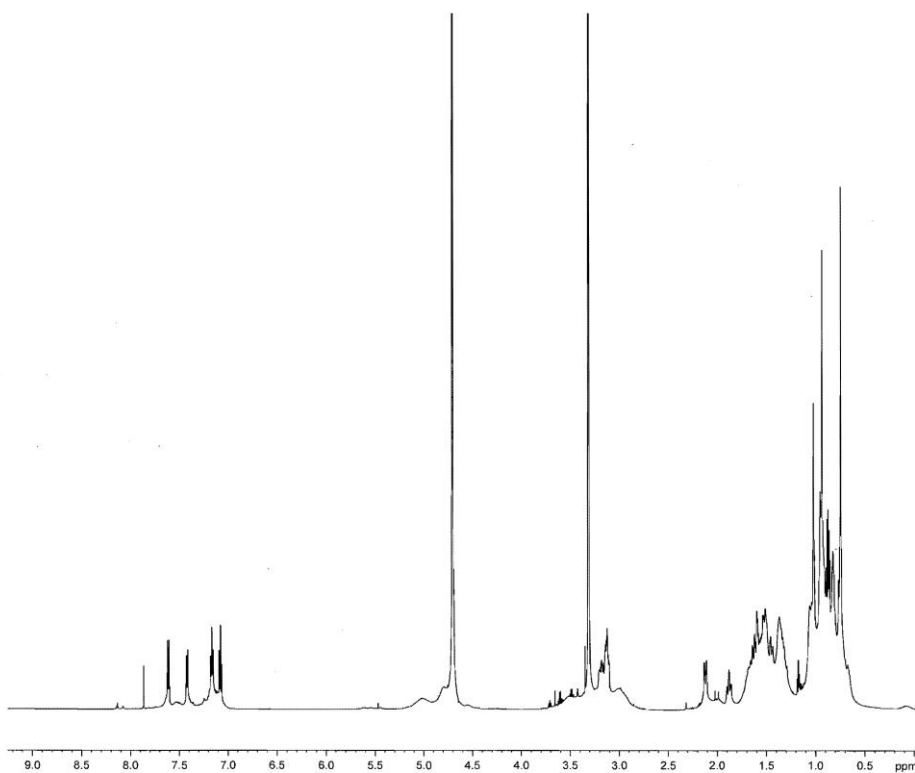
(3 β)-3-Hydroxy-28-[(3*S*)-3-(1*H*-indol-3-ylmethyl)piperazin-1-yl]olean-18-en-28-one (**21**)



^1H NMR: δ 0.74 (3H, s, H25), 0.74 (3H, s, H26), 0.76 (1H, dd, $J_1=2.1$ Hz, $J_2=11.5$ Hz, H5), 0.82 (3H, s, H27), 0.95 (3H, s, H23), 1.02 (3H, s, H24), 1.02 (3H, s, H30), 1.06 (3H, s, H29), 1.88 (2H, dt, $J_1=3.5$ Hz, $J_2=13.0$ Hz, $J_3=13.0$ Hz, H16), 2.13 (2H, ddd, $J_1=3.2$ Hz, $J_2=3.5$ Hz, $J_3=12.8$ Hz, H22), 3.12 (2H, dd, $J_1=9.0$ Hz, $J_2=13.0$ Hz, H5'), 3.19 (1H, dd, $J_1=4.7$ Hz, $J_2=11.3$ Hz, H3), 5.03 (1H, bs, H19), 7.02 (1H, bd, $J=7.9$ Hz, H13'), 7.07 (1H, ddd, $J_1=1.0$ Hz, $J_2=7.0$ Hz, $J_3=7.9$ Hz, H12'), 7.18 (1H, bs, H7'), 7.18 (1H, bt, $J=7.5$ Hz, H11'), 7.72 (1H, bd, $J=7.8$ Hz, H10').

^{13}C NMR: δ 15.20 (q, C27), 16.00 (q, C26), 16.70 (q, C24), 17.00 (q, C25), 19.30 (t, C6), 21.90 (t, C11), 25.70 (t, C12), 26.80 (t, C5'), 28.10 (q, C30), 28.60 (q, C23), 30.30 (t, C15), 32.00 (t, C16), 32.00 (q, C29), 32.80 (s, C20), 34.20 (t, C2), 34.20 (t, C7), 35.10 (t, C22), 35.80 (t, C21), 38.40 (s, C10), 39.90 (s, C8), 40.20 (t, C1), 42.20 (s, C4), 42.20 (d, C13), 43.40 (t, C1'), 45.10 (s, C14), 45.80 (t, C3'), 49.60 (t, C4'), 51.70 (s, C17), 52.30 (d, C9), 56.90 (d, C5), 57.20 (d, C2'), 79.60 (d, C3), 107.80 (s, C6'), 112.90 (d, C10'), 118.90 (d, C13'), 120.50 (d, C12'), 123.10 (d, C11'), 125.60 (d, C7'), 128.30 (d, C14'), 131.90 (d, C19), 138.50 (s, C9'), 141.10 (s, C18), 176.90 (s, C28).

MS: m/z = 654 $[\text{M}+\text{H}]^+$. For $\text{C}_{43}\text{H}_{63}\text{N}_3\text{O}_2$ (653.98) calcd. C 78.97, H 9.71, N 6.43, found C 79.00, H 9.69, N 6.42.



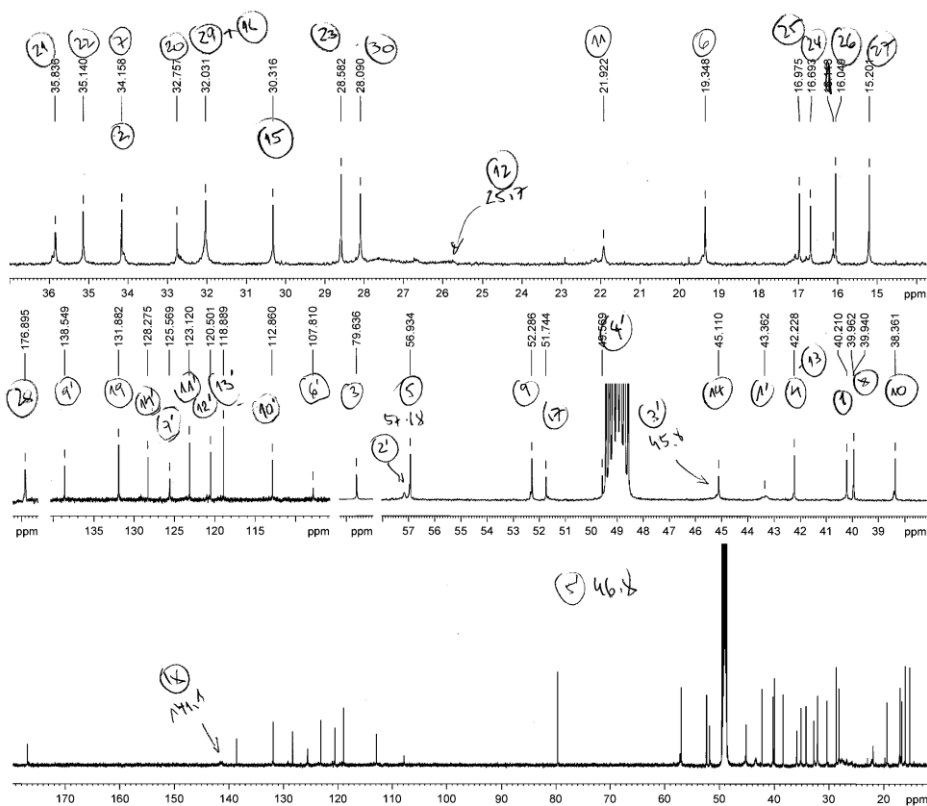
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62386



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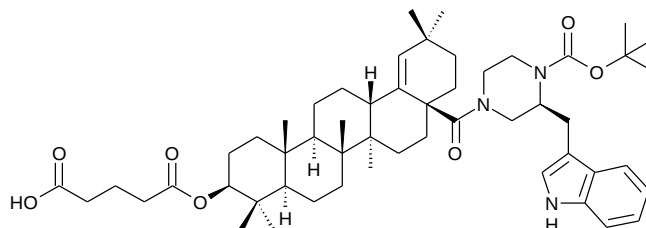
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62386

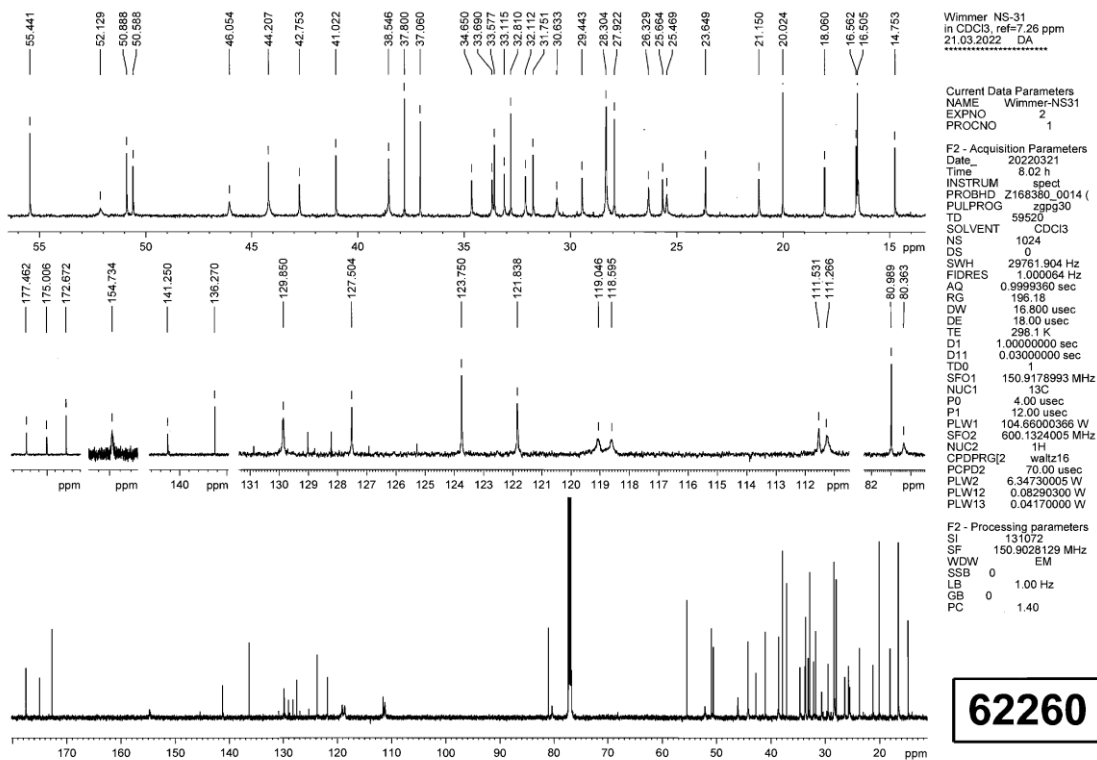
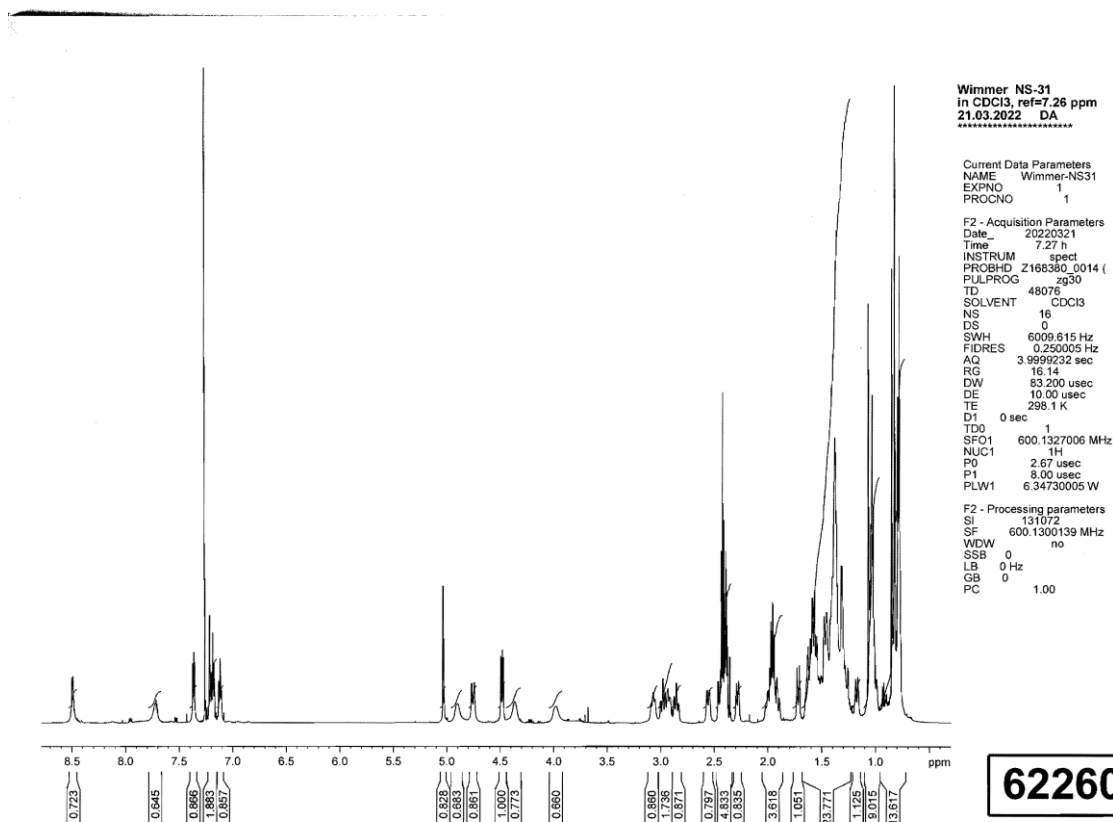
5-((3 β)-28-[(3*S*)-4-(*tert*-Butoxycarbonyl)-3-(1*H*-indol-3-ylmethyl)piperazin-1-yl]-28-oxooolean-18-en-3-yl)oxy)-5-oxopentanoic acid (**22**)



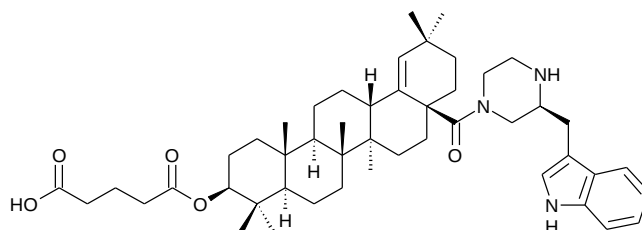
^1H NMR: δ 0.77 (3H, s, H26), 0.79 (3H, s, H25), 0.82 (3H, s, H23), 0.85 (3H, s, H27), 1.01 (1H, dt, $J_1=4.4$ Hz, $J_2=13.2$ Hz, $J_3=13.2$ Hz, H1), 1.03 (3H, s, H30), 1.04 (3H, s, H24), 1.06 (3H, s, H29), 1.18 (1H, dt, $J_1=3.0$ Hz, $J_2=3.0$ Hz, $J_3=13.0$ Hz, H15), 1.38 (3H, s, H12'), 1.72 (1H, dt, $J_1=3.5$ Hz, $J_2=3.5$ Hz, $J_3=13.4$ Hz, H1), 1.92 (2H, dt, $J_1=3.5$ Hz, $J_2=13.5$ Hz, $J_3=13.5$ Hz, H16), 1.93-1.99 (2H, m, H11), 2.00 (1H, dt, $J_1=3.7$ Hz, $J_2=13.3$ Hz, $J_3=13.3$ Hz, H15), 2.28 (2H, dt, $J_1=3.2$ Hz, $J_2=3.2$ Hz, $J_3=12.8$ Hz, H7), 2.37-2.41 (2H, m, H4'), 2.42 (2H, t, $J=7.3$ Hz, H2'), 2.45 (1H, dd, $J_1=3.3$ Hz, $J_2=12.5$ Hz, H13), 2.56 (1H, dd, $J_1=3.4$ Hz, $J_2=13.4$ Hz, H6'), 2.86 (1H, bt, $J_1=3.2$ Hz, $J_2=12.8$ Hz, $J_3=12.8$ Hz, H9'), 2.92 (1H, bdd, $J_1=4.5$ Hz, $J_2=13.5$ Hz, H13'), 2.98 (1H, dt, $J_1=2.6$ Hz, $J_2=12.8$ Hz, $J_3=12.8$ Hz, H8'), 3.07 (1H, bdd, $J_1=9.1$ Hz, $J_2=13.5$ Hz, H13'), 3.98 (1H, bs, H8'), 4.36 (1H, bs, H7'), 4.48 (1H, dd, $J_1=4.9$ Hz, $J_2=11.4$ Hz, H3), 4.76 (1H, d, $J=13.4$ Hz, H6'), 4.91 (1H, bd, $J=13.2$ Hz, H9'), 5.03 (1H, s, H19), 7.12 (1H, t, $J=7.5$ Hz, H20'), 7.19 (1H, t, $J=7.7$ Hz, H19'), 7.22 (1H, bs, H15'), 7.37 (1H, bd, $J=7.9$ Hz, H18'), 7.72 (1H, bs, H21'), 8.49 (1H, bs, H11'-NH).

^{13}C NMR: δ 14.80 (q, C27), 16.50 (q, C25), 16.50 (q, C26), 16.60 (q, C24), 18.10 (t, C6), 20.00 (t, C11), 21.20 (t, C3'), 23.60 (t, C2), 25.50 (t, C13'), 25.70 (t, C12), 26.30 (q, C30), 27.90 (q, C23), 28.30 (q, C12'), 29.40 (t, C15), 30.60 (t, C16), 31.80 (s, C20), 32.10 (q, C29), 32.80 (t, C2'), 33.10 (t, C7), 33.60 (t, C21), 33.70 (t, C4'), 34.70 (t, C22), 37.10 (s, C10), 37.80 (s, C8), 38.50 (t, C1), 38.50 (t, C8'), 41.00 (s, C14), 42.80 (d, C13), 44.20 (s, C4), 44.20 (t, C6'), 46.10 (t, C9'), 50.60 (s, C17), 50.90 (d, C9), 52.10 (d, C7'), 55.40 (d, C5), 80.40 (s, C11'), 81.00 (d, C3), 111.30 (d, C18'), 111.50 (s, C14'), 118.60 (d, C21'), 119.00 (d, C20'), 121.80 (d, C19'), 123.80 (d, C15'), 127.50 (s, C22'), 129.90 (d, C19), 136.20 (s, C17'), 141.30 (s, C18), 154.70 (s, C10'), 172.70 (s, C1'), 175.00 (s, C28), 177.50 (s, C5').

MS: m/z = 868 $[\text{M}+\text{H}]^+$. For $\text{C}_{53}\text{H}_{77}\text{N}_3\text{O}_7$ (868.19) calcd. C 73.32, H 8.94, N 4.84, found C 73.30, H 8.95, N 4.82.



5-((3 β)-28-[(3*S*)-3-(1*H*-Indol-3-ylmethyl)piperazin-1-yl]-28-oxoolean-18-en-3-yl)oxy)-5-oxopentanoic acid (**23**)



^1H NMR: δ 0.77 (3H, s, H26), 0.79 (3H, s, H25), 0.84 (3H, s, H23), 0.86 (3H, s, H27), 0.93 (3H, s, H24), 1.02 (3H, s, H30), 1.05 (3H, s, H29), 1.88 (2H, dt, $J_1=3.5$ Hz, $J_2=13.3$ Hz, $J_3=13.3$ Hz, H16), 2.35 (2H, dt, $J_1=3.2$ Hz, $J_2=3.2$ Hz, $J_3=13.0$ Hz, H7), 2.37 (2H, t, $J=7.3$ Hz, H2'), 2.38 (1H, dd, $J_1=3.5$ Hz, $J_2=13.0$ Hz, H13), 3.15 (2H, dd, $J_1=9.0$ Hz, $J_2=13.5$ Hz, H10'), 4.46 (1H, dd, $J_1=5.6$ Hz, $J_2=10.9$ Hz, H3), 5.02 (1H, bs, H19), 7.07 (1H, ddd, $J_1=1.0$ Hz, $J_2=7.0$ Hz, $J_3=7.0$ Hz, H17'), 7.17 (1H, bs, H12'), 7.17 (1H, ddd, $J_1=1.0$ Hz, $J_2=7.0$ Hz, $J_3=8.0$ Hz, H16'), 7.42 (1H, bd, $J=8.0$ Hz, H15'), 7.62 (1H, dt, $J_1=1.0$ Hz, $J_2=1.0$ Hz, $J_3=7.9$ Hz, H18').

^{13}C NMR: δ 15.20 (q, C27), 16.70 (q, C26), 17.00 (q, C24), 17.00 (q, C25), 19.20 (t, C6), 21.60 (t, C11), 21.90 (t, C3'), 25.80 (q, C30), 27.70 (t, C2), 27.70 (t, C12), 27.80 (t, C10'), 28.20 (q, C23), 28.50 (t, C15), 30.30 (t, C16), 32.00 (q, C29), 32.80 (t, C2'), 34.00 (s, C20), 34.20 (t, C4'), 34.60 (t, C7), 35.10 (t, C21), 35.70 (t, C22), 38.30 (s, C8), 38.30 (s, C10), 38.90 (t, C1), 39.70 (s, C14), 41.90 (t, C6'), 42.20 (d, C13), 43.30 (t, C8'), 45.10 (s, C4), 49.60 (t, C9'), 51.70 (s, C17), 52.20 (d, C9), 56.90 (d, C5), 57.20 (d, C7'), 82.50 (d, C3), 107.80 (s, C11'), 112.90 (d, C15'), 118.90 (d, C18'), 120.50 (d, C17'), 123.10 (d, C16'), 125.60 (d, C12'), 128.30 (s, C19'), 131.90 (d, C19), 138.50 (s, C14'), 141.20 (s, C18), 174.70 (s, C1'), 176.50 (s, C28), 176.90 (s, C5').

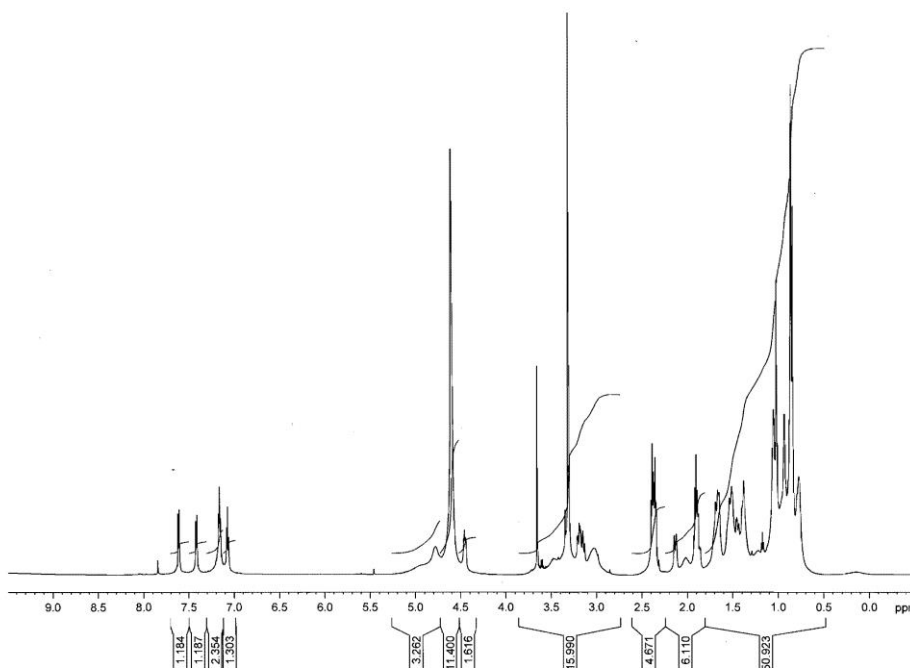
MS: m/z = 768 $[\text{M}+\text{H}]^+$. For $\text{C}_{48}\text{H}_{69}\text{N}_3\text{O}_5$ (768.08) calcd. C 75.06, H 9.05, N 5.47, found C 75.09, H 9.04, N 5.45.

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07.04.2022 DA

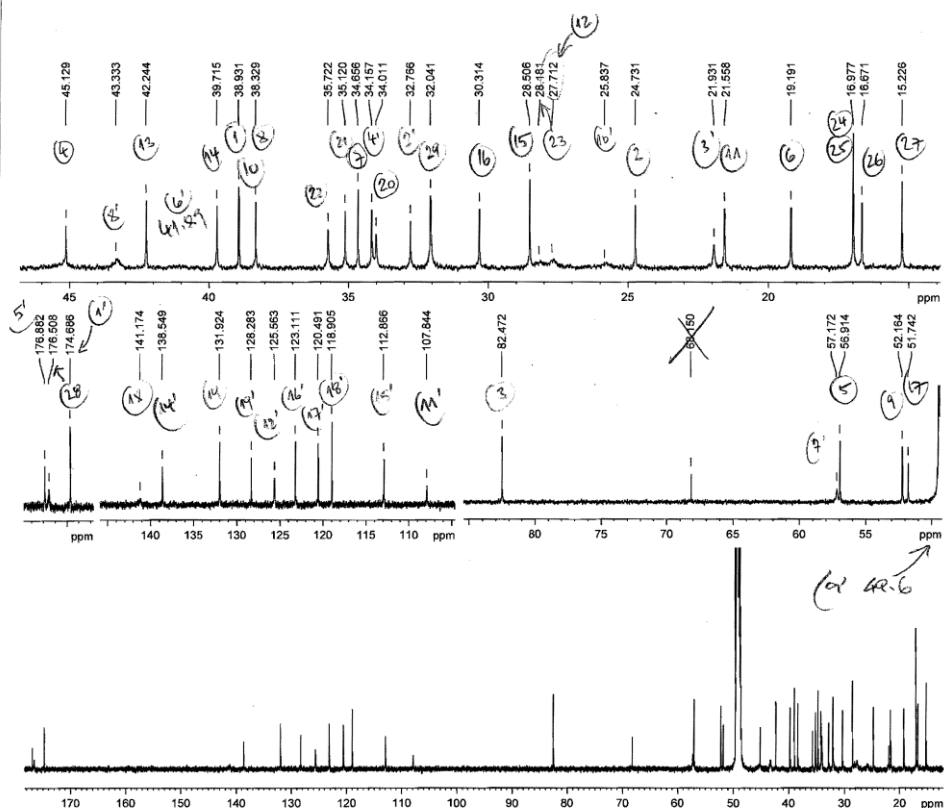
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62388



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07.04.2022 DA

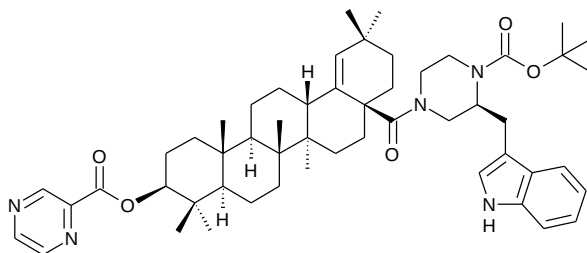
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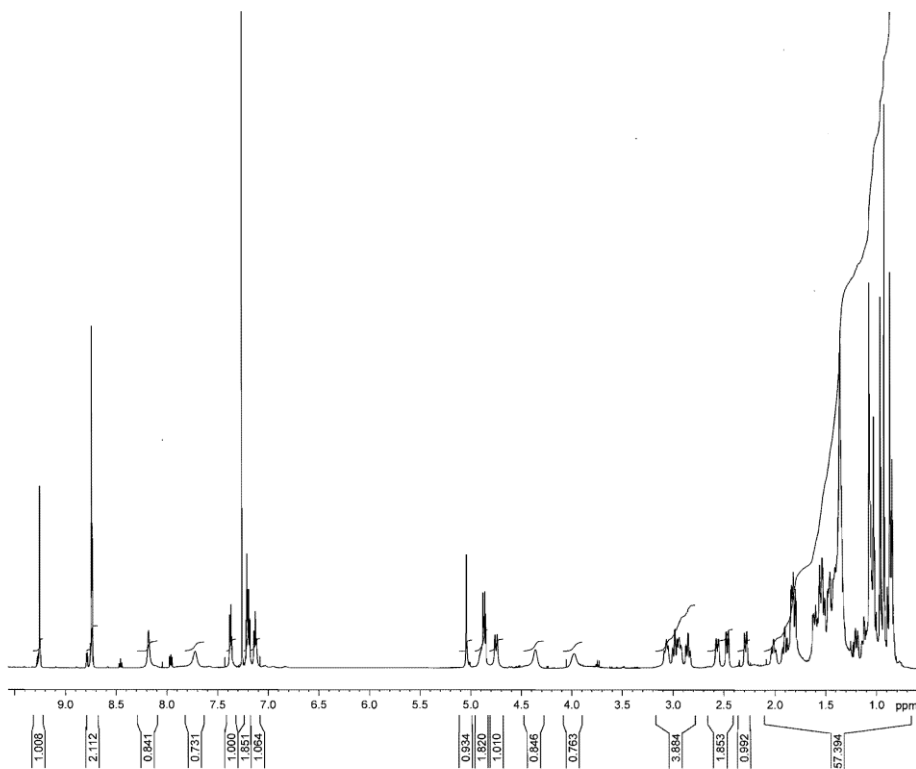
{(3 β)-28-[(3*S*)-4-(*tert*-Butoxycarbonyl)-3-(1*H*-indol-3-ylmethyl)piperazin-1-yl]-28-oxoolean-18-en-3-yl}pyrazine-2-carboxylate (**24**)



^1H NMR: δ 0.85 (3H, s, H26), 0.87 (3H, s, H27), 0.89 (1H, dd, $J_1=1.5$ Hz, $J_2=11.7$ Hz, H5), 0.92 (3H, s, H23), 0.96 (3H, s, H25), 1.03 (3H, s, H30), 1.06 (3H, s, H24), 1.07 (3H, s, H29), 1.13 (2H, dt, $J_1=4.7$ Hz, $J_2=13.2$ Hz, $J_3=13.2$ Hz, H1), 1.20 (1H, dt, $J_1=3.0$ Hz, $J_2=3.0$ Hz, $J_3=13.5$ Hz, H15), 1.36 (3H, bs, H12'), 1.91 (2H, dt, $J_1=3.5$ Hz, $J_2=13.7$ Hz, $J_3=13.7$ Hz, H16), 2.02 (1H, dt, $J_1=3.5$ Hz, $J_2=13.5$ Hz, $J_3=13.5$ Hz, H15), 2.47 (1H, bdd, $J_1=3.2$ Hz, $J_2=12.3$ Hz, H13), 2.57 (1H, dd, $J_1=3.3$ Hz, $J_2=13.4$ Hz, H6'), 2.86 (1H, dt, $J_1=3.0$ Hz, $J_2=12.9$ Hz, $J_3=12.9$ Hz, H9'), 2.93 (1H, bdd, $J_1=4.5$ Hz, $J_2=13.5$ Hz, H13'), 2.98 (1H, dt, $J_1=2.8$ Hz, $J_2=12.8$ Hz, $J_3=12.8$ Hz, H8'), 3.07 (1H, bdd, $J_1=9.3$ Hz, $J_2=13.5$ Hz, H13'), 3.98 (1H, bs, H8'), 4.37 (1H, bs, H7'), 4.75 (1H, d, $J=13.4$ Hz, H6'), 4.87 (1H, dd, $J_1=5.6$ Hz, $J_2=10.7$ Hz, H3), 4.88 (1H, bd, $J=13.2$ Hz, H9'), 5.04 (1H, bs, H19), 7.13 (1H, bt, $J=7.5$ Hz, H20'), 7.20 (1H, bt, $J=7.7$ Hz, H19'), 7.22 (1H, bs, H15'), 7.37 (1H, bd, $J=7.9$ Hz, H18'), 7.72 (1H, bs, H21'), 8.18 (1H, bs, H16'-NH), 8.74 (1H, dd, $J_1=1.3$ Hz, $J_2=2.4$ Hz, H4'), 8.74 (1H, dd, $J_1=1.3$ Hz, $J_2=2.4$ Hz, H5'), 9.26 (1H, d, $J=1.3$ Hz, H3').

^{13}C NMR: δ 14.70 (q, C27), 16.50 (q, C26), 16.60 (q, C25), 16.70 (q, C24), 18.10 (t, C6), 21.20 (t, C11), 23.70 (t, C2), 25.40 (t, C13'), 25.60 (t, C12), 26.30 (q, C30), 28.10 (q, C23), 28.30 (q, C12'), 29.50 (t, C15), 30.60 (t, C16), 31.80 (s, C20), 32.10 (q, C29), 33.10 (t, C7), 33.70 (t, C21), 34.60 (t, C22), 37.10 (s, C10), 38.20 (s, C8), 38.60 (t, C1), 38.60 (t, C8'), 41.10 (s, C14), 42.70 (d, C13), 44.20 (s, C4), 44.20 (t, C6'), 46.00 (t, C9'), 50.60 (s, C17), 50.90 (d, C9), 52.10 (d, C7'), 55.50 (d, C5), 80.30 (s, C11'), 83.20 (d, C3), 111.20 (d, C18'), 111.70 (s, C14'), 118.60 (d, C21'), 119.20 (d, C20'), 121.90 (d, C19'), 123.70 (d, C15'), 127.60 (s, C22'), 129.80 (d, C19), 136.20 (s, C17'), 141.30 (s, C18), 144.10 (s, C2'), 144.70 (d, C4'), 146.00 (d, C3'), 147.30 (d, C5'), 154.70 (s, C10'), 163.50 (s, C1'), 174.90 (s, C28).

MS: m/z = 860 $[\text{M}+\text{H}]^+$. For $\text{C}_{53}\text{H}_{73}\text{N}_5\text{O}_5$ (860.18) calcd. C 74.00, H 8.55, N 8.14, found C 74.03, H 8.53, N 8.13.

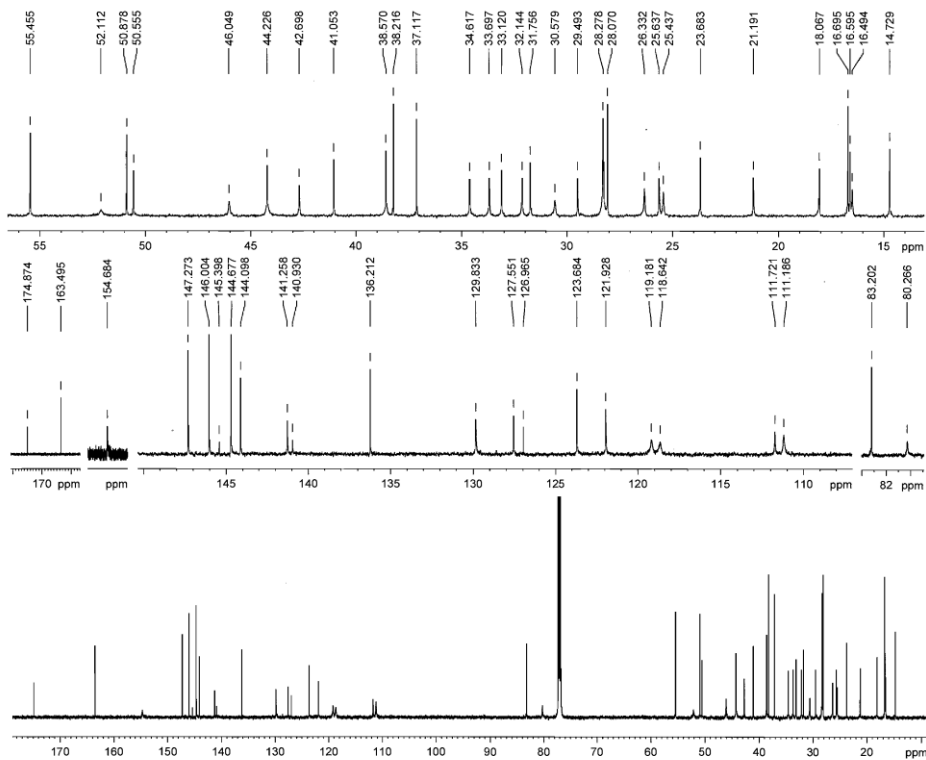


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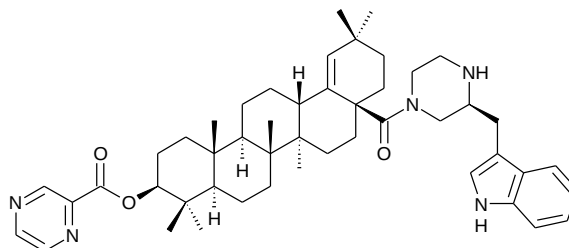
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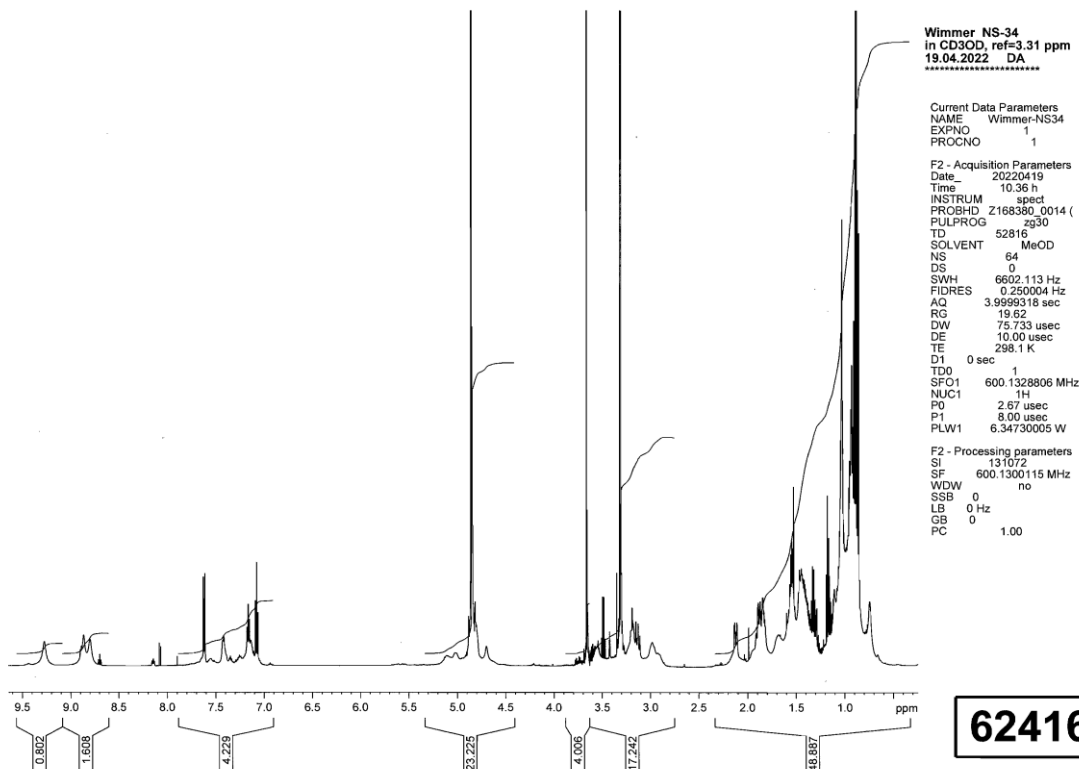
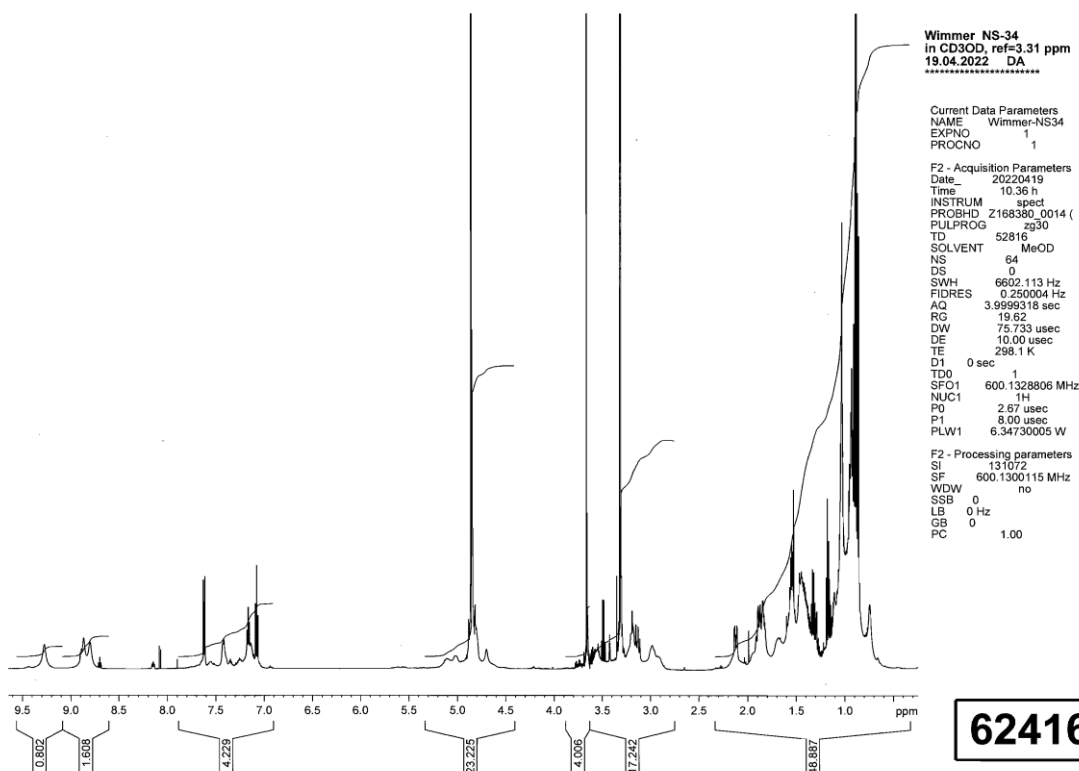
[(3 β)-28-[(3S)-3-(1*H*-Indol-3-ylmethyl)piperazin-1-yl]-28-oxoolean-18-en-3-yl]pyrazine-2-carboxylate (**25**)



^1H NMR: δ 0.85 (3H, s, H26), 0.87 (3H, s, H27), 0.89 (1H, dd, $J_1=1.5$ Hz, $J_2=11.7$ Hz, H5), 0.92 (3H, s, H23), 0.96 (3H, s, H25), 1.03 (3H, s, H30), 1.06 (3H, s, H24), 1.07 (3H, s, H29), 1.13 (2H, dt, $J_1=4.7$ Hz, $J_2=13.2$ Hz, $J_3=13.2$ Hz, H1), 1.20 (1H, dt, $J_1=3.0$ Hz, $J_2=3.0$ Hz, $J_3=13.5$ Hz, H15), 1.91 (2H, dt, $J_1=3.5$ Hz, $J_2=13.7$ Hz, $J_3=13.7$ Hz, H16), 2.02 (1H, dt, $J_1=3.5$ Hz, $J_2=13.5$ Hz, $J_3=13.5$ Hz, H15), 2.47 (1H, bdd, $J_1=3.2$ Hz, $J_2=12.3$ Hz, H13), 2.57 (1H, dd, $J_1=3.3$ Hz, $J_2=13.4$ Hz, H6'), 2.86 (1H, dt, $J_1=3.0$ Hz, $J_2=12.9$ Hz, $J_3=12.9$ Hz, H9'), 2.93 (1H, bdd, $J_1=4.5$ Hz, $J_2=13.5$ Hz, H13'), 2.98 (1H, dt, $J_1=2.8$ Hz, $J_2=12.8$ Hz, $J_3=12.8$ Hz, H8'), 3.07 (1H, bdd, $J_1=9.3$ Hz, $J_2=13.5$ Hz, H13'), 3.98 (1H, bs, H8'), 4.37 (1H, bs, H7'), 4.75 (1H, d, $J=13.4$ Hz, H6'), 4.87 (1H, dd, $J_1=5.6$ Hz, $J_2=10.7$ Hz, H3), 4.88 (1H, bd, $J=13.2$ Hz, H9'), 5.04 (1H, bs, H19), 7.13 (1H, bt, $J=7.5$ Hz, H20'), 7.20 (1H, bt, $J=7.7$ Hz, H19'), 7.22 (1H, bs, H15'), 7.37 (1H, bd, $J=7.9$ Hz, H18'), 7.72 (1H, bs, H21'), 8.18 (1H, bs, H16'-NH), 8.74 (1H, dd, $J_1=1.3$ Hz, $J_2=2.4$ Hz, H4'), 8.74 (1H, dd, $J_1=1.3$ Hz, $J_2=2.4$ Hz, H5'), 9.26 (1H, d, $J=1.3$ Hz, H3').

^{13}C NMR: δ 14.70 (q, C27), 16.50 (q, C26), 16.60 (q, C25), 16.70 (q, C24), 18.10 (t, C6), 21.20 (t, C11), 23.70 (t, C2), 25.40 (t, C13'), 25.60 (t, C12), 26.30 (q, C30), 28.10 (q, C23), 29.50 (t, C15), 30.60 (t, C16), 31.80 (s, C20), 32.10 (q, C29), 33.10 (t, C7), 33.70 (t, C21), 34.60 (t, C22), 37.10 (s, C10), 38.20 (s, C8), 38.60 (t, C1), 38.60 (t, C8'), 41.10 (s, C14), 42.70 (d, C13), 44.20 (s, C4), 44.20 (t, C6'), 46.00 (t, C9'), 50.60 (s, C17), 50.90 (d, C9), 52.10 (d, C7'), 55.50 (d, C5), 83.20 (d, C3), 111.20 (d, C18'), 111.70 (s, C14'), 118.60 (d, C21'), 119.20 (d, C20'), 121.90 (d, C19'), 123.70 (d, C15'), 127.60 (s, C22'), 129.80 (d, C19), 136.20 (s, C17'), 141.30 (s, C18), 144.10 (s, C2'), 144.70 (d, C4'), 146.00 (d, C3'), 147.30 (d, C5'), 163.50 (s, C1'), 174.90 (s, C28).

MS: $m/z = 760$ $[\text{M}+\text{H}]^+$. For $\text{C}_{48}\text{H}_{65}\text{N}_5\text{O}_3$ (760.06) calcd. C 75.85, H 8.62, N 9.21, found C 75.88, H 8.60, N 9.23.



2. Additional Data on Cytotoxicity Screening Tests

The cytotoxicity screening tests were performed according to an experimental procedure published in our earlier papers.^{49,54} The cell viability was measured after 72 h using resazurin (Sigma, MO, USA).

The whole series of the target compounds was subjected to a rapid screening first to determine cell viability (ESI, Table S1). This screening of the introductory selection of the compounds resulted in identification of compounds with a potential to show acceptable cytotoxicity data. The introductory screening tests were performed in cervical carcinoma cell line (HeLa), and the cell viability was compared with that found in normal human fibroblasts (BJ). The tests were made with the compounds concentrations $c = 50 \mu\text{M}$ and $c = 16.7 \mu\text{M}$ (ESI, Table S1).

Table S1. Introductory Cytotoxicity Screening Tests Evaluated as Cell Viability [%] after 72 h of Exposition

Compd.	MW	Conc. [μM]	Viability [%] – 72 h			
			HeLa, run 1	HeLa, run 2	BJ, run 1	BJ, run 2
1	454.70	50.0	30	17	110	113
		16.7	86	81	98	97
2	456.71	50.0	86	44	126	135
		16.7	108	106	93	99
3	600.88	50.0	9	11	29	30
		16.7	78	86	44	31
5	651.98	50.0	0	0	0	0
		16.7	3	0	64	26
7	508.78	50.0	0	0	0	0
		16.7	1	1	64	1
9	654.00	50.0	0	0	0	0
		16.7	0	0	59	9
10	498.74	50.0	75	2	129	109
		16.7	89	61	114	102
11	644.93	50.0	0	0	0	0
		16.7	2	4	6	0

12	602.89	50.0	111	96	95	93
		16.7	108	100	99	90
13	716.99	50.0	110	64	138	99
		16.7	110	97	117	107
15	566.86	50.0	0	0	0	0
		16.7	0	0	1	0
18	656.02	50.0	0	0	0	0
		16.7	1	1	3	2
23	768.08	50.0	95	89	90	87
		16.7	104	98	99	88
25	760.06	50.0	0	0	0	0
		16.7	34	20	36	30