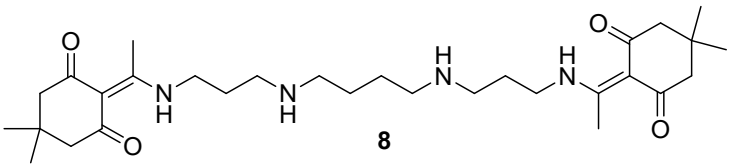
Supporting Information

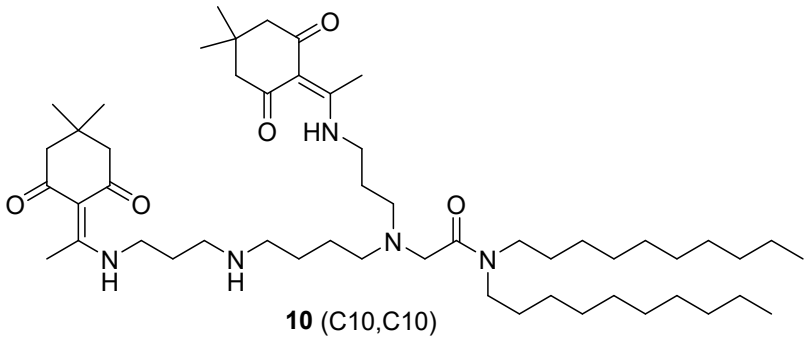
Compound 8



8

Yield: 80%; IR: ν_{max}, cm^{-1} : 3438, 2924, 2854, 1636, 1572, 1461, 1369, 1335, 1305, 1141, 1096, 1032, 890, 723; ^1H NMR (400 MHz, $CDCl_3$): δ 1.00 (s, 12H, 2 × C(CH_3)$_2$), 1.51 (m, 4H, NHCH$_2$CH_2CH_2CH$_2$NH), 1.83 (quint, J = 6.9 Hz, 4H, 2 × DdeNHCH$_2$CH_2CH$_2$NH), 2.33 (s, 8H, 4 × COCH_2), 2.53 (s, 6H, 2 × CH_3C=C), 2.60 (t, J = 5.9 Hz, 4H, 2 × NHCH_2CH$_2$CH$_2$CH_2NH), 2.71 (t, J = 6.9 Hz, 4H, 2 × NHCH_2CH$_2$CH$_2$NHDde), 3.46 (m, 4H, 2 × DdeNHCH_2CH$_2$CH$_2$NH), 13.41 (br, s, 2H, 2 × NHDde); ^{13}C NMR (100 MHz, $CDCl_3$): δ 27.6, 29.3, 41.3, 46.6, 49.6, 17.9, 28.2, 30.1, 52.8, 107.8, 173.6, 197.4; HRMS (ESI-TOF), m/z calcd for $C_{52}H_{94}N_5O_5$ $[M+H]^+$ 531.3905; found 531.3913.

Compound 10 (C10,C10)

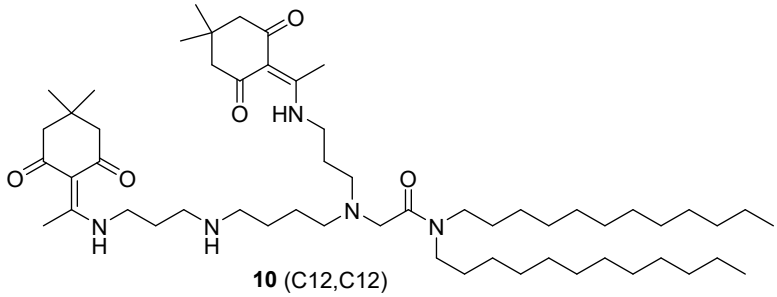


10 (C10,C10)

Yield: 80%; IR: ν_{max}, cm^{-1} : 3438, 2924, 2854, 1636, 1572, 1461, 1369, 1335, 1305, 1141, 1096, 1032, 890, 723; ^1H NMR (400 MHz, $CDCl_3$): δ 0.85 (t, J = 6.7 Hz, 6H, 2 × CH_3), 1.00 (s, 12H, 2 × C(CH_3)$_2$), 1.23 (s, 28H, 2 × (CH_2)$_7$), 1.46 (m, 2H, CONCH$_2$CH_2), 1.50 (m, 2H, CONCH$_2$CH_2), 1.64 (br s, 2H, NHCH$_2$CH$_2$CH_2CH$_2$N), 1.89 (m, 2H, NCH$_2$CH_2CH$_2$NHDde), 2.13 (br s, 2H, NHCH$_2$CH_2CH$_2$CH$_2$N), 2.32 (s, 10H, 4 × COCH_2, DdeNHCH$_2$CH_2CH$_2$NH), 2.53 and 2.55 (s, 6H, 2 × CH_3C=C), 2.68 (m, 2H, NHCH$_2$CH$_2$CH$_2$CH_2N), 2.85 (m, 2H, NCH_2CH$_2$CH$_2$NHDde), 2.96 (t, J = 5.1 Hz, 2H, NHCH_2CH$_2$CH$_2$CH$_2$N), 3.10 (t, J = 7.4 Hz, 2H, DdeNHCH$_2$CH$_2$CH_2NH), 3.16 (t, J = 7.5 Hz, 2H, NCH_2CH$_2$), 3.26 (m, 2H, NCH_2CH$_2$), 3.30 (s, 2H, NCH_2CO), 3.44 (q, J = 5.3 Hz, 2H, NCH$_2$CH$_2$CH_2NHDde), 3.56 (q, J = 5.5 Hz,

2H, DdeNHCH_2CH$_2$CH$_2$NH), 13.45 (br s, 1H, N*H*Dde), 13.50 (br s, 1H, N*H*Dde); ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.1, 18.0, 18.1, 22.6, 25.4, 25.8, 26.9, 27.1, 27.7, 28.2, 29.3, 29.4, 29.5, 30.0, 31.8, 41.2, 41.5, 46.4, 46.6, 47.5, 48.5, 52.8, 53.6, 55.3, 55.9, 107.9, 108.1, 170.0, 173.8, 174.0, 197.8; HRMS (ESI-TOF), *m*/*z* calcd for $C_{52}H_{94}N_5O_5$ $[M+H]^+$ 868.7249; found 868.7258.

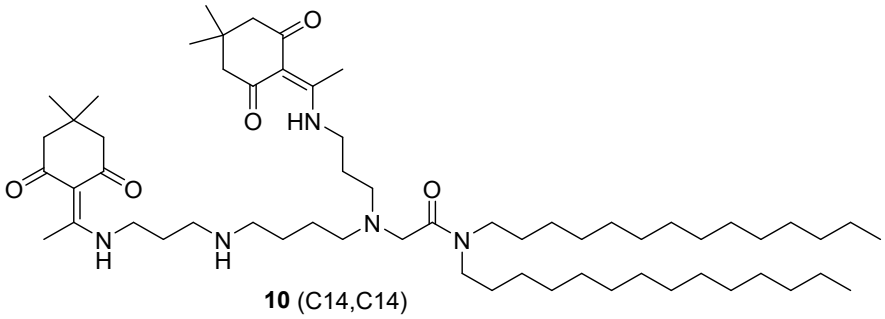
Compound 10 (C12,C12)



10 (C12,C12)

Yield: 72%; IR: ν_{max}, cm^{-1} : 3437, 2923, 2854, 1638, 1572, 1460, 1369, 1334, 1295, 1141, 1089, 1029, 885, 722; ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.85 (t, *J* = 6.0 Hz, 6H, 2 × CH_3), 1.00 (s, 12H, 2 × C(CH_3)$_2$), 1.23 (s, 36H, 2 × (CH_2)$_9$), 1.47 (m, 2H, CONCH$_2$CH_2), 1.51 (m, 2H, CONCH$_2$CH_2), 1.64 (br s, 2H, NHCH$_2$CH$_2$CH_2CH$_2$N), 1.89 (m, 2H, NCH$_2$CH_2CH$_2$NHDde), 2.12 (br s, 2H, NHCH$_2$CH_2CH$_2$CH$_2$N), 2.33 (s, 10H, 4 × COCH_2, DdeNHCH$_2$CH_2CH$_2$NH), 2.54 and 2.55 (s, 6H, 2 × CH_3C=C), 2.68 (m, 2H, NHCH$_2$CH$_2$CH$_2$CH_2N), 2.85 (m, 2H, NCH_2CH$_2$CH$_2$NHDde), 2.96 (t, *J* = 5.2 Hz, 2H, NHCH_2CH$_2$CH$_2$CH$_2$N), 3.09 (t, *J* = 7.4 Hz, 2H, DdeNHCH$_2$CH$_2$CH_2NH), 3.16 (t, *J* = 7.7 Hz, 2H, NCH_2CH$_2$), 3.27 (t, *J* = 7.7 Hz, 2H, NCH_2CH$_2$), 3.30 (s, 2H, NCH_2CO), 3.45 (q, *J* = 5.3 Hz, 2H, NCH$_2$CH$_2$CH_2NHDde), 3.55 (q, *J* = 5.5 Hz, 2H, DdeNHCH_2CH$_2$CH$_2$NH), 13.46 (br s, 1H, N*H*Dde), 13.50 (br s, 1H, N*H*Dde); ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.1, 18.0, 18.1, 22.7, 25.4, 25.8, 26.9, 27.1, 27.7, 28.2, 29.3, 29.4, 29.6, 30.0, 31.9, 41.2, 41.5, 46.4, 46.6, 47.5, 48.5, 52.8, 53.5, 55.4, 55.8, 107.9, 108.1, 170.0, 173.8, 174.0, 197.8; HRMS (ESI-TOF), *m*/*z* calcd for $C_{56}H_{102}N_5O_5$ $[M+H]^+$ 924.7875; found 924.7878.

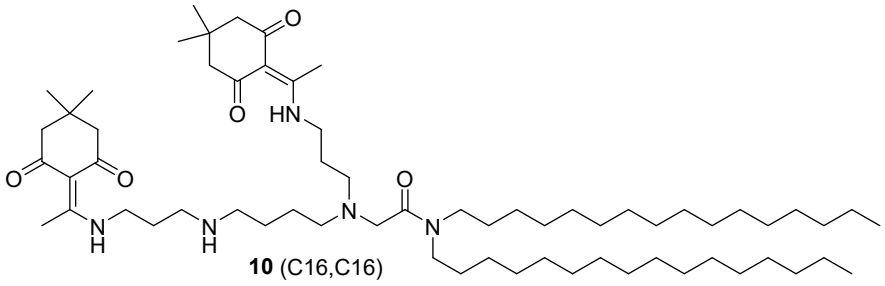
Compound 10 (C14,C14)



10 (C14,C14)

Yield: 66%; IR: ν_{max}, cm^{-1} : 3434, 2922, 2853, 1639, 1573, 1461, 1369, 1334, 1295, 1140, 1092, 1030, 855, 721; ^1H NMR (400 MHz, $CDCl_3$): δ 0.85 (t, J = 6.6 Hz, 6H, 2 × CH_3), 1.00 (s, 12H, 2 × C(CH_3)$_2$), 1.23 (s, 44H, 2 × (CH_2)$_{11}$), 1.47 (m, 2H, CONCH$_2$CH_2), 1.51 (m, 2H, CONCH$_2$CH_2), 1.60 (br s, 2H, NHCH$_2$CH$_2$CH_2CH$_2$N), 1.87 (m, 2H, NCH$_2$CH_2CH$_2$NHDde), 2.01 (br s, 2H, NHCH$_2$CH_2CH$_2$CH$_2$N), 2.25 (br s, 2H, DdeNHCH$_2$CH_2CH$_2$NH), 2.33 (s, 8H, 4 × COCH_2), 2.54 and 2.55 (s, 6H, 2 × CH_3C=C), 2.66 (m, 2H, NHCH$_2$CH$_2$CH$_2$CH_2N), 2.83 (m, 2H, NCH_2CH$_2$CH$_2$NHDde), 2.90 (m, 2H, NHCH_2CH$_2$CH$_2$CH$_2$N), 3.03 (m, 2H, DdeNHCH$_2$CH$_2$CH_2NH), 3.17 (t, J = 7.7 Hz, 2H, NCH_2CH$_2$), 3.26 (t, J = 7.6 Hz, 2H, NCH_2CH$_2$), 3.29 (s, 2H, NCH_2CO), 3.44 (q, J = 5.3 Hz, 2H, NCH$_2$CH$_2$CH_2NHDde), 3.53 (q, J = 5.4 Hz, 2H, DdeNHCH_2CH$_2$CH$_2$NH), 13.45 (br s, 1H, NHDde), 13.49 (br s, 1H, NHDde); ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.1, 18.0, 18.1, 22.7, 25.5, 26.1, 27.0, 27.1, 27.7, 28.3, 29.3, 29.4, 29.7, 30.1, 31.9, 41.2, 41.5, 46.5, 47.4, 48.7, 52.8, 53.2, 55.5, 55.6, 107.9, 108.1, 170.0, 173.8, 173.9, 197.8; HRMS (ESI-TOF), m/z calcd for $C_{60}H_{110}N_5O_5$ $[M+H]^+$ 980.8501; found 980.8503.

Compound 10 (C16,C16)

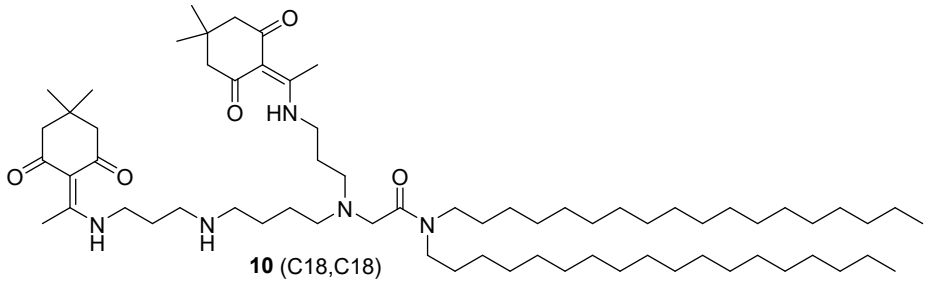


10 (C16,C16)

Yield: 84%; IR: ν_{max}, cm^{-1} : 3419, 2923, 2853, 1638, 1573, 1462, 1369, 1335, 1302, 1142, 1096, 1032, 855, 721; ^1H NMR (400 MHz, $CDCl_3$): δ 0.85 (t, J = 6.7 Hz, 6H, 2 × CH_3), 1.00 (s, 12H, 2 × C(CH_3)$_2$), 1.23 (s, 52H, 2 × (CH_2)$_{13}$), 1.44 (m, 2H, CONCH$_2$CH_2), 1.52 (m, 2H,

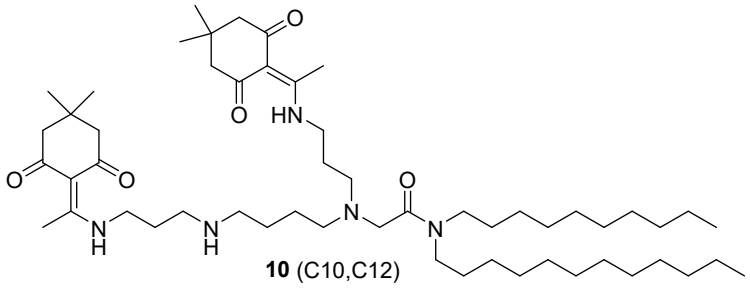
$CONCH_2CH_2$), 1.60 (br s, 2H, $NHCH_2CH_2CH_2CH_2N$), 1.87 (m, 2H, $NCH_2CH_2CH_2NHDde$), 2.02 (br s, 2H, $NHCH_2CH_2CH_2CH_2N$), 2.26 (m, 2H, $DdeNHCH_2CH_2CH_2NH$), 2.32 (s, 8H, 4 × $COCH_2$), 2.53 and 2.54 (s, 6H, 2 × $CH_3C{=}C$), 2.65 (m, 2H, $NHCH_2CH_2CH_2CH_2N$), 2.80 (m, 2H, $NCH_2CH_2CH_2NHDde$), 2.90 (t, J = 5.2 Hz, 2H, $NHCH_2CH_2CH_2CH_2N$), 3.04 (t, J = 7.3 Hz, 2H, $DdeNHCH_2CH_2CH_2NH$), 3.16 (t, J = 7.4 Hz, 2H, NCH_2CH_2), 3.26 (t, J = 7.8 Hz, 2H, NCH_2CH_2), 3.28 (s, 2H, NCH_2CO), 3.43 (q, J = 5.3 Hz, 2H, $NCH_2CH_2CH_2NHDde$), 3.55 (q, J = 5.2 Hz, 2H, $DdeNHCH_2CH_2CH_2NH$), 13.45 (br s, 1H, N*H*Dde), 13.48 (br s, 1H, N*H*Dde); ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.1, 18.0, 18.1, 22.6, 25.4, 25.9, 26.9, 27.1, 27.7, 28.2, 29.3, 29.6, 29.7, 30.1, 31.9, 41.2, 41.6, 46.4, 46.5, 47.4, 48.6, 52.8, 53.2, 55.3, 55.6, 107.9, 108.0, 169.9, 173.8, 173.9, 197.8; HRMS (ESI-TOF), m/z calcd for $C_{64}H_{118}N_5O_5$ $[M+H]^+$ 1036.9127; found 1036.9132.

Compound 10 (C18,C18)



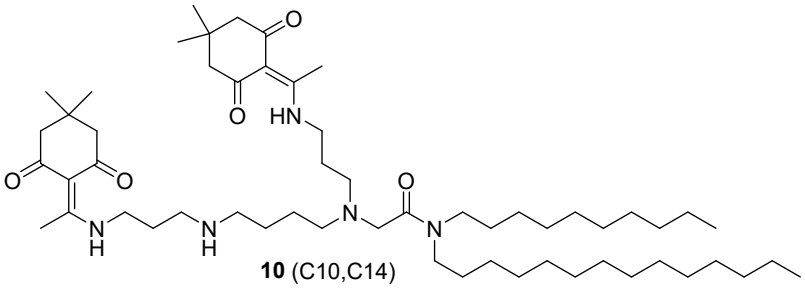
Yield: estimated 63% (<95% purity); IR: ν_{max}, cm^{-1} : 3418, 2918, 2851, 1634, 1570, 1463, 1369, 1337, 1299, 1144, 1092, 1026, 1026, 886, 720; ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.85 (t, J = 6.7 Hz, 6H, 2 × CH_3), 1.00 (s, 12H, 2 × $C(CH_3)_2$), 1.23 (s, 60H, 2 × $(CH_2)_{15}$), 1.47 (m, 2H, $CONCH_2CH_2$), 1.53 (m, 2H, $CONCH_2CH_2$), 1.65 (br s, 2H, $NHCH_2CH_2CH_2CH_2N$), 1.88 (m, 2H, $NCH_2CH_2CH_2NHDde$), 2.14 (br s, 2H, $NHCH_2CH_2CH_2CH_2N$), 2.32 (s, 10H, 4 × $COCH_2$, $DdeNHCH_2CH_2CH_2NH$), 2.53 and 2.54 (s, 6H, 2 × $CH_3C{=}C$), 2.70 (m, 2H, $NHCH_2CH_2CH_2CH_2N$), 2.86 (m, 2H, $NCH_2CH_2CH_2NHDde$), 3.00 (m, 2H, $NHCH_2CH_2CH_2CH_2N$), 3.12 (m, 2H, $DdeNHCH_2CH_2CH_2NH$), 3.16 (m, 2H, NCH_2CH_2), 3.26 (m, 2H, NCH_2CH_2), 3.31 (s, 2H, NCH_2CO), 3.46 (m, 2H, $NCH_2CH_2CH_2NHDde$), 3.57 (m, 2H, $DdeNHCH_2CH_2CH_2NH$), 13.45 (br s, 1H, N*H*Dde), 13.50 (br s, 1H, N*H*Dde); ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.1, 18.0, 18.1, 22.7, 25.4, 25.8, 26.9, 27.1, 27.7, 28.2, 29.3, 29.4, 29.7, 30.1, 31.9, 41.2, 41.5, 46.4, 46.6, 47.5, 48.5, 52.8, 53.6, 55.3, 55.9, 108.0, 108.1, 170.0, 173.9, 174.0, 198.0; HRMS (ESI-TOF), m/z calcd for $C_{68}H_{126}N_5O_5$ $[M+H]^+$ 1092.9753; found 1092.9744.

Compound 10 (C10,C12)



Yield: 76%; IR: ν_{max}, cm^{-1} : 3426, 2924, 2854, 1636, 1571, 1461, 1369, 1333, 1298, 1141, 1092, 1030, 883, 722; ^1H NMR (400 MHz, $CDCl_3$): δ 0.85 (t, J = 6.3 Hz, 6H, 2 × CH_3), 1.00 (s, 12H, 2 × C(CH_3)$_2$), 1.23 (s, 32H, 2 × (CH_2)$_7$ and (CH_2)$_9$), 1.45 (m, 2H, CONCH$_2$CH_2), 1.52 (m, 2H, CONCH$_2$CH_2), 1.60 (br s, 2H, NHCH$_2$CH$_2$CH_2CH$_2$N), 1.88 (m, 2H, NCH$_2$CH_2CH$_2$NHDde), 1.99 (br s, 2H, NHCH$_2$CH_2CH$_2$CH$_2$N), 2.23 (m, 2H, DdeNHCH$_2$CH_2CH$_2$NH), 2.32 (s, 8H, 4 × COCH_2), 2.53 and 2.54 (s, 6H, 2 × CH_3C=C), 2.65 (m, 2H, NHCH$_2$CH$_2$CH$_2$CH_2N), 2.79 (m, 2H, NCH_2CH$_2$CH$_2$NHDde), 2.88 (t, J = 5.5 Hz, 2H, NHCH_2CH$_2$CH$_2$CH$_2$N), 3.02 (t, J = 7.3 Hz, 2H, DdeNHCH$_2$CH$_2$CH_2NH), 3.16 (t, J = 7.7 Hz, 2H, NCH_2CH$_2$), 3.26 (t, J = 7.8 Hz, 2H, NCH_2CH$_2$), 3.28 (s, 2H, NCH_2CO), 3.43 (q, J = 6.7 Hz, 2H, NCH$_2$CH$_2$CH_2NHDde), 3.55 (q, J = 6.5 Hz, 2H, DdeNHCH_2CH$_2$CH$_2$NH), 13.44 (br s, 1H, N*H*Dde), 13.48 (br s, 1H, N*H*Dde); ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.1, 17.9, 18.0, 22.6, 25.5, 26.4, 26.9, 27.1, 27.7, 28.2, 29.2, 29.3, 29.4, 29.5, 29.6, 30.1, 31.9, 41.2, 41.6, 46.4, 47.4, 48.6, 52.8, 53.1, 55.4, 55.6, 107.9, 108.0, 169.9, 173.7, 173.9, 197.8; HRMS (ESI-TOF), m/z calcd for $C_{54}H_{98}N_5O_5$ $[M+H]^+$ 896.7562; found 896.7583.

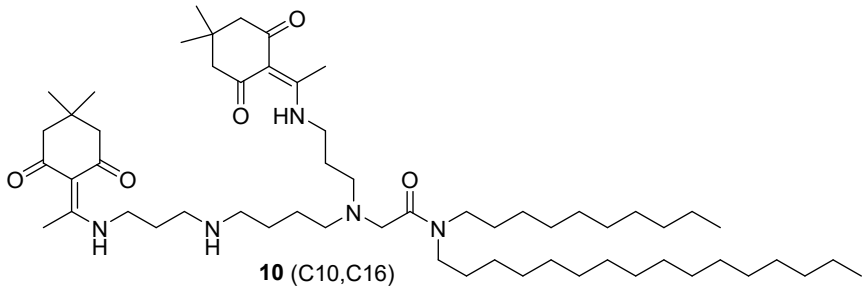
Compound 10 (C10,C14)



Yield: estimated 64% (<95% purity); IR: ν_{max}, cm^{-1} : 3424, 2923, 2854, 1638, 1570, 1461, 1368, 1333, 1297, 1142, 1095, 1030, 884, 722; ^1H NMR (400 MHz, $CDCl_3$): δ 0.85 (t, J = 6.5 Hz, 6H, 2 × CH_3), 1.00 (s, 12H, 2 × C(CH_3)$_2$), 1.23 (s, 36H, (CH_2)$_7$ and (CH_2)$_{11}$), 1.45 (m, 2H, CONCH$_2$CH_2), 1.52 (m, 2H, CONCH$_2$CH_2), 1.63 (br s, 2H, NHCH$_2$CH$_2$CH_2CH$_2$N), 1.88

(m, 2H, $NCH_2CH_2CH_2NHDde$), 2.09 (br s, 2H, $NHCH_2CH_2CH_2CH_2N$), 2.32 (s, 10H, 4 × $COCH_2$, $DdeNHCH_2CH_2CH_2NH$), 2.53 and 2.55 (s, 6H, 2 × $CH_3C{=}C$), 2.68 (m, 2H, $NHCH_2CH_2CH_2CH_2N$), 2.83 (m, 2H, $NCH_2CH_2CH_2NHDde$), 2.94 (t, J = 5.8 Hz, 2H, $NHCH_2CH_2CH_2CH_2N$), 3.08 (t, J = 7.2 Hz, 2H, $DdeNHCH_2CH_2CH_2NH$), 3.16 (t, J = 7.0 Hz, 2H, NCH_2CH_2), 3.26 (t, J = 8.2 Hz, 2H, NCH_2CH_2), 3.29 (s, 2H, NCH_2CO), 3.45 (q, J = 5.4 Hz, 2H, $NCH_2CH_2CH_2NHDde$), 3.55 (q, J = 5.4 Hz, 2H, $DdeNHCH_2CH_2CH_2NH$), 13.45 (br s, 1H, N*H*Dde), 13.49 (br s, 1H, N*H*Dde); ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.1, 18.0, 18.1, 22.6, 25.4, 25.8, 26.9, 27.1, 27.7, 28.2, 29.3, 29.4, 29.6, 30.1, 31.9, 41.2, 41.5, 46.4, 46.5, 47.5, 48.5, 52.8, 53.4, 55.4, 55.8, 107.9, 108.1, 170.0, 173.8, 174.0, 197.9; HRMS (ESI-TOF), m/z calcd for $C_{56}H_{102}N_5O_5$ $[M+H]^+$ 924.7875; found 924.7869.

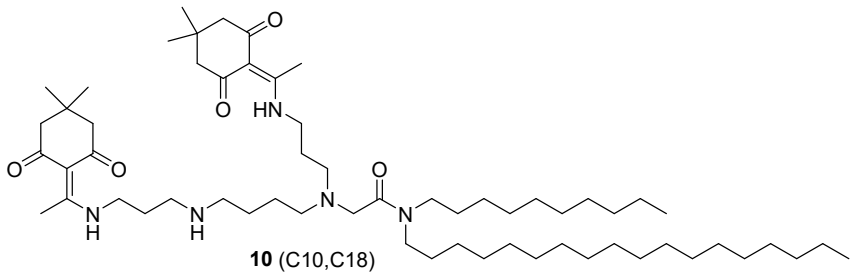
Compound 10 (C10,C16)



10 (C10,C16)

Yield: 67%; IR: ν_{max}, cm^{-1} : 3429, 2924, 2854, 1634, 1573, 1462, 1371, 1335, 1302, 1146, 1092, 1032, 889, 754; ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.85 (t, J = 6.6 Hz, 6H, 2 × CH_3), 1.00 (s, 12H, 2 × $C(CH_3)_2$), 1.23 (s, 40H, $(CH_2)_7$ and $(CH_2)_{13}$), 1.45 (m, 2H, $CONCH_2CH_2$), 1.53 (m, 2H, $CONCH_2CH_2$), 1.63 (br s, 2H, $NHCH_2CH_2CH_2CH_2N$), 1.87 (m, 2H, $NCH_2CH_2CH_2NHDde$), 2.01 (m, 2H, $NHCH_2CH_2CH_2CH_2N$), 2.26 (m, 2H, $DdeNHCH_2CH_2CH_2NH$), 2.33 (s, 8H, 4 × $COCH_2$), 2.54 and 2.56 (s, 6H, 2 × $CH_3C{=}C$), 2.70 (m, 2H, $NHCH_2CH_2CH_2CH_2N$), 2.84 (m, 2H, $NCH_2CH_2CH_2NHDde$), 2.94 (m, 2H, $NHCH_2CH_2CH_2CH_2N$), 3.08 (m, 2H, $DdeNHCH_2CH_2CH_2NH$), 3.16 (m, 2H, NCH_2CH_2), 3.27 (m, 2H, NCH_2CH_2), 3.30 (s, 2H, NCH_2CO), 3.46 (q, J = 6.0 Hz, 2H, $NCH_2CH_2CH_2NHDde$), 3.56 (q, J = 5.3 Hz, 2H, $DdeNHCH_2CH_2CH_2NH$), 13.40 (br s, 1H, N*H*Dde), 13.46 (br s, 1H, N*H*Dde); ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.1, 18.0, 18.1, 22.6, 25.4, 25.8, 26.9, 27.1, 27.7, 28.2, 29.2, 29.3, 29.7, 30.1, 31.9, 41.0, 41.2, 46.4, 46.6, 47.5, 48.7, 52.8, 53.6, 55.4, 55.9, 108.0, 108.1, 170.0, 173.8, 174.0, 197.9; HRMS (ESI-TOF), m/z calcd for $C_{58}H_{106}N_5O_5$ $[M+H]^+$ 952.8188; found 952.8183.

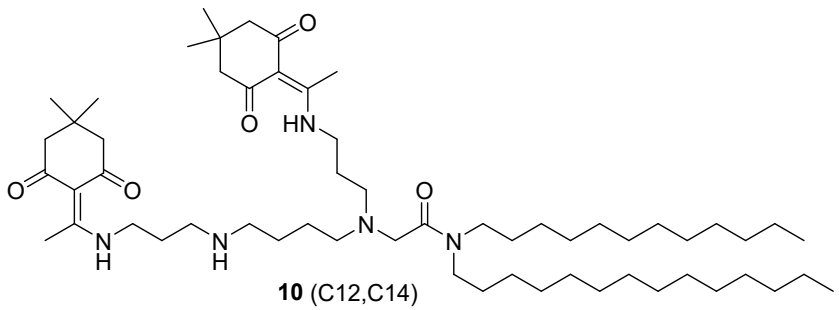
Compound 10 (C10,C18)



10 (C10,C18)

Yield: 72%; IR: ν_{max}, cm^{-1} : 3419, 2923, 2854, 1637, 1572, 1461, 1369, 1334, 1298, 1141, 1096, 1031, 883, 721; ^1H NMR (400 MHz, $CDCl_3$): δ 0.85 (t, J = 6.6 Hz, 6H, 2 × CH_3), 1.00 (s, 12H, 2 × $C(CH_3)_2$), 1.23 (s, 44H, $(CH_2)_7$ and $(CH_2)_{15}$), 1.45 (m, 2H, $CONCH_2CH_2$), 1.52 (m, 2H, $CONCH_2CH_2$), 1.58 (m, 2H, $NHCH_2CH_2CH_2CH_2N$), 1.86 (m, 2H, $NCH_2CH_2CH_2NHDde$), 1.94 (m, 2H, $NHCH_2CH_2CH_2CH_2N$), 2.20 (m, 2H, $DdeNHCH_2CH_2CH_2NH$), 2.32 (s, 8H, 4 × $COCH_2$), 2.53 and 2.54 (s, 6H, 2 × $CH_3C{=}C$), 2.63 (t, J = 5.5 Hz, 2H, $NHCH_2CH_2CH_2CH_2N$), 2.78 (m, 2H, $NCH_2CH_2CH_2NHDde$), 2.86 (m, 2H, $NHCH_2CH_2CH_2CH_2N$), 3.00 (m, 2H, $DdeNHCH_2CH_2CH_2NH$), 3.17 (m, 2H, NCH_2CH_2), 3.26 (m, 2H, NCH_2CH_2), 3.28 (s, 2H, NCH_2CO), 3.43 (q, J = 5.3 Hz, 2H, $NCH_2CH_2CH_2NHDde$), 3.55 (q, J = 6.5 Hz, 2H, $DdeNHCH_2CH_2CH_2NH$), 13.43 (br s, 1H, N*H*Dde), 13.47 (br s, 1H, N*H*Dde); ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.1, 17.9, 18.0, 22.7, 25.5, 26.1, 26.9, 27.1, 27.7, 28.2, 29.3, 29.6, 29.7, 30.1, 31.9, 41.2, 41.6, 46.4, 47.4, 48.7, 52.8, 53.0, 55.4, 107.9, 108.0, 169.9, 173.7, 173.9, 197.8; HRMS (ESI-TOF), *m*/*z* calcd for $C_{60}H_{110}N_5O_5$ $[M+H]^+$ 980.8501; found 980.8500.

Compound 10 (C12,C14)

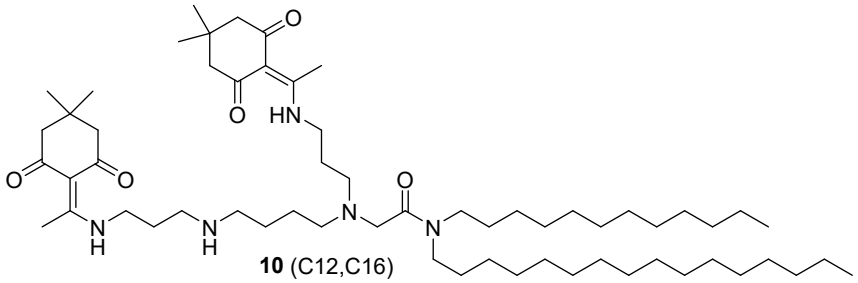


10 (C12,C14)

Yield: 67%; IR: ν_{max}, cm^{-1} : 3433, 2923, 2854, 1636, 1571, 1461, 1368, 1334, 1298, 1141, 1096, 1030, 884, 721; ^1H NMR (400 MHz, $CDCl_3$): δ 0.85 (t, J = 6.4 Hz, 6H, 2 × CH_3), 1.00 (s, 12H, 2 × $C(CH_3)_2$), 1.23 (s, 40H, $(CH_2)_9$ and $(CH_2)_{11}$), 1.45 (m, 2H, $CONCH_2CH_2$), 1.51

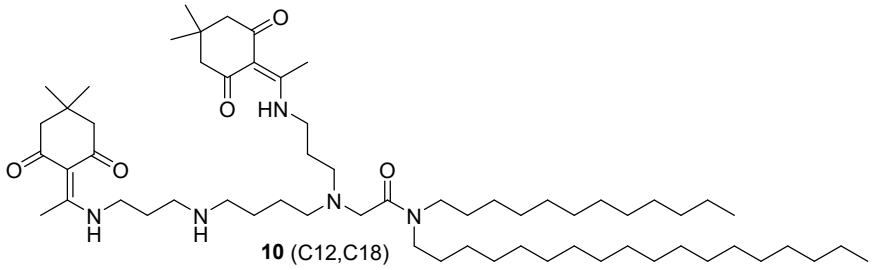
(m, 2H, $CONCH_2CH_2$), 1.62 (m, 2H, $NHCH_2CH_2CH_2CH_2N$), 1.88 (m, 2H, $NCH_2CH_2CH_2NHDde$), 2.06 (m, 2H, $NHCH_2CH_2CH_2CH_2N$), 2.29 (m, 2H, $DdeNHCH_2CH_2CH_2NH$), 2.33 (s, 8H, 4 × $COCH_2$), 2.54 and 2.55 (s, 6H, 2 × $CH_3C{=}C$), 2.67 (t, J = 5.3 Hz, 2H, $NHCH_2CH_2CH_2CH_2N$), 2.82 (m, 2H, $NCH_2CH_2CH_2NHDde$), 2.93 (t, J = 5.2 Hz, 2H, $NHCH_2CH_2CH_2CH_2N$), 3.07 (t, J = 7.3 Hz, 2H, $DdeNHCH_2CH_2CH_2NH$), 3.16 (t, J = 7.7 Hz, 2H, NCH_2CH_2), 3.26 (m, 2H, NCH_2CH_2), 3.29 (s, 2H, NCH_2CO), 3.45 (q, J = 5.3 Hz, 2H, $NCH_2CH_2CH_2NHDde$), 3.54 (q, J = 5.5 Hz, 2H, $DdeNHCH_2CH_2CH_2NH$), 13.45 (br s, 1H, N*H*Dde), 13.49 (br s, 1H, N*H*Dde); ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.1, 18.0, 18.1, 22.7, 25.4, 25.9, 26.9, 27.1, 27.7, 28.2, 29.3, 29.4, 29.6, 30.1, 31.9, 41.2, 41.5, 46.4, 46.5, 47.5, 48.6, 52.8, 53.4, 55.4, 55.8 107.9, 108.1, 170.0, 173.8, 173.9, 197.8; HRMS (ESI-TOF), m/z calcd for $C_{58}H_{106}N_5O_5$ $[M+H]^+$ 952.8188; found 952.8193.

Compound 10 (C12,C16)



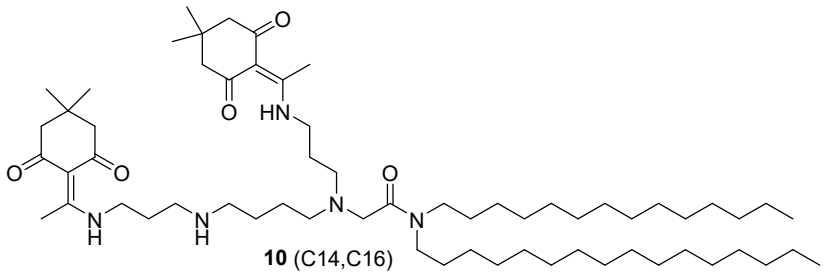
Yield: 72%; IR: ν_{max}, cm^{-1} : 3438, 2923, 2853, 1637, 1571, 1461, 1369, 1334, 1298, 1142, 1095, 1030, 884, 722; ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.85 (t, J = 6.5 Hz, 6H, 2 × CH_3), 1.00 (s, 12H, 2 × $C(CH_3)_2$), 1.23 (s, 44H, $(CH_2)_9$ and $(CH_2)_{13}$), 1.45 (m, 2H, $CONCH_2CH_2$), 1.52 (m, 2H, $CONCH_2CH_2$), 1.64 (br s, 2H, $NHCH_2CH_2CH_2CH_2N$), 1.89 (m, 2H, $NCH_2CH_2CH_2NHDde$), 2.11 (br s, 2H, $NHCH_2CH_2CH_2CH_2N$), 2.32 (s, 10H, 4 × $COCH_2$, $DdeNHCH_2CH_2CH_2NH$), 2.54 and 2.55 (s, 6H, 2 × $CH_3C{=}C$), 2.68 (m, 2H, $NHCH_2CH_2CH_2CH_2N$), 2.85 (m, 2H, $NCH_2CH_2CH_2NHDde$), 2.95 (t, J = 5.2 Hz, 2H, $NHCH_2CH_2CH_2CH_2N$), 3.09 (t, J = 7.3 Hz, 2H, $DdeNHCH_2CH_2CH_2NH$), 3.16 (t, J = 7.4 Hz, 2H, NCH_2CH_2), 3.26 (t, J = 7.7 Hz, 2H, NCH_2CH_2), 3.30 (s, 2H, NCH_2CO), 3.45 (q, J = 5.3 Hz, 2H, $NCH_2CH_2CH_2NHDde$), 3.56 (q, J = 6.6 Hz, 2H, $DdeNHCH_2CH_2CH_2NH$), 13.45 (br s, 1H, N*H*Dde), 13.50 (br s, 1H, N*H*Dde); ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.1, 18.0, 18.1, 22.6, 25.4, 25.6, 26.9, 27.1, 27.7, 28.2, 29.3, 29.6, 29.7, 30.1, 31.9, 41.2, 41.5, 46.4, 46.6, 47.5, 48.5, 52.8, 53.5, 55.4, 55.9, 108.0, 108.1, 170.0, 173.8, 174.0, 197.8; HRMS (ESI-TOF), m/z calcd for $C_{60}H_{110}N_5O_5$ $[M+H]^+$ 980.8501; found 980.8503.

Compound 10 (C12,C18)



Yield: 67%; IR: ν_{max}, cm^{-1} : 3434, 2922, 2853, 1638, 1572, 1461, 1369, 1334, 1298, 1142, 1094, 1030, 884, 721; ^1H NMR (400 MHz, $CDCl_3$): δ 0.85 (t, J = 6.6 Hz, 6H, 2 × CH_3), 1.00 (s, 12H, 2 × C(CH_3)$_2$), 1.23 (s, 48H, (CH_2)$_9$ and (CH_2)$_{15}$), 1.45 (m, 2H, CONCH$_2$CH_2), 1.52 (m, 2H, CONCH$_2$CH_2), 1.64 (br s, 2H, NHCH$_2$CH$_2$CH_2CH$_2$N), 1.89 (m, 2H, NCH$_2$CH_2CH$_2$NHDde), 2.13 (br s, 2H, NHCH$_2$CH_2CH$_2$CH$_2$N), 2.33 (s, 10H, 4 × COCH_2, DdeNHCH$_2$CH_2CH$_2$NH), 2.54 and 2.55 (s, 6H, 2 × CH_3C=C), 2.69 (t, J = 4.8 Hz, 2H, NHCH$_2$CH$_2$CH$_2$CH_2N), 2.85 (m, 2H, NCH_2CH$_2$CH$_2$NHDde), 2.94 (t, J = 5.2 Hz, 2H, NHCH_2CH$_2$CH$_2$CH$_2$N), 3.09 (t, J = 7.4 Hz, 2H, DdeNHCH$_2$CH$_2$CH_2NH), 3.16 (t, J = 7.7 Hz, 2H, NCH_2CH$_2$), 3.27 (t, J = 7.9 Hz, 2H, NCH_2CH$_2$), 3.30 (s, 2H, NCH_2CO), 3.45 (q, J = 5.3 Hz, 2H, NCH$_2$CH$_2$CH_2NHDde), 3.55 (q, J = 5.5 Hz, 2H, DdeNHCH_2CH$_2$CH$_2$NH), 13.46 (br s, 1H, NHDde), 13.50 (br s, 1H, NHDde); ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.1, 18.0, 18.1, 22.7, 25.4, 25.8, 26.9, 27.1, 27.7, 28.2, 29.3, 29.4, 29.7, 30.1, 31.9, 41.2, 41.5, 46.4, 46.6, 47.5, 48.5, 52.8, 53.6, 55.4, 55.9, 108.0, 108.1, 170.0, 173.8, 174.0, 197.8; HRMS (ESI-TOF), m/z calcd for $C_{62}H_{114}N_5O_5$ $[M+H]^+$ 1008.8814; found 1008.8808.

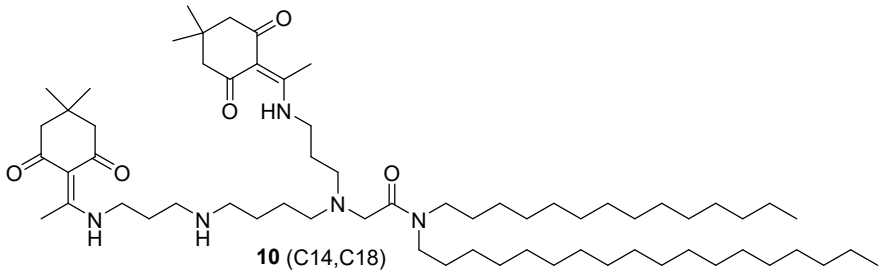
Compound 10 (C14,C16)



Yield: 68%; IR: ν_{max}, cm^{-1} : 3435, 2922, 2853, 1638, 1572, 1461, 1369, 1335, 1298, 1142, 1092, 1030, 884, 721; ^1H NMR (400 MHz, $CDCl_3$): δ 0.85 (t, J = 6.6 Hz, 6H, 2 × CH_3), 1.00 (s, 12H, 2 × C(CH_3)$_2$), 1.23 (s, 48H, (CH_2)$_{11}$ and (CH_2)$_{13}$), 1.45 (m, 2H, CONCH$_2$CH_2), 1.52 (m, 2H, CONCH$_2$CH_2), 1.64 (br s, 2H, NHCH$_2$CH$_2$CH_2CH$_2$N), 1.89 (m, 2H,

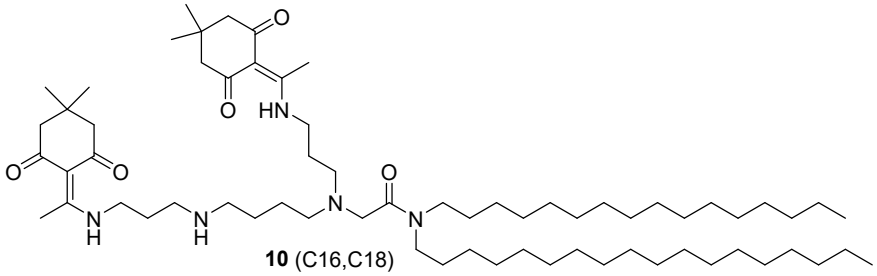
$NCH_2CH_2CH_2NHDde$), 2.13 (br s, 2H, $NHCH_2CH_2CH_2CH_2N$), 2.32 (s, 10H, 4 × $COCH_2$, $DdeNHCH_2CH_2CH_2NH$), 2.53 and 2.55 (s, 6H, 2 × $CH_3C{=}C$), 2.69 (t, J = 4.7 Hz, 2H, $NHCH_2CH_2CH_2CH_2N$), 2.86 (m, 2H, $NCH_2CH_2CH_2NHDde$), 2.96 (t, J = 5.1 Hz, 2H, $NHCH_2CH_2CH_2CH_2N$), 3.10 (t, J = 7.4 Hz, 2H, $DdeNHCH_2CH_2CH_2NH$), 3.16 (t, J = 7.6 Hz, 2H, NCH_2CH_2), 3.26 (t, J = 7.6 Hz, 2H, NCH_2CH_2), 3.30 (s, 2H, NCH_2CO), 3.45 (q, J = 5.4 Hz, 2H, $NCH_2CH_2CH_2NHDde$), 3.56 (q, J = 5.5 Hz, 2H, $DdeNHCH_2CH_2CH_2NH$), 13.46 (br s, 1H, N*H*Dde), 13.50 (br s, 1H, N*H*Dde); ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.1, 18.0, 18.1, 22.7, 25.4, 25.8, 26.9, 27.1, 27.7, 28.2, 29.3, 29.6, 29.7, 30.0, 31.9, 41.2, 41.5, 46.4, 46.6, 47.5, 48.5, 52.8, 53.6, 55.4, 55.9, 108.0, 108.1, 170.0, 173.8, 174.0, 197.8; HRMS (ESI-TOF), m/z calcd for $C_{62}H_{114}N_5O_5$ $[M+H]^+$ 1008.8814; found 1008.8818.

Compound 10 (C14,C18)



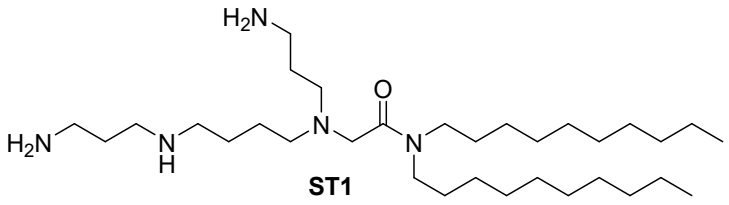
Yield: 69%; IR: ν_{max}, cm^{-1} : 3443, 2922, 2853, 1637, 1572, 1461, 1368, 1335, 1298, 1142, 1094, 1030, 884, 721; ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.85 (t, J = 6.6 Hz, 6H, 2 × CH_3), 1.00 (s, 12H, 2 × $C(CH_3)_2$), 1.23 (s, 52H, $(CH_2)_{11}$ and $(CH_2)_{15}$), 1.45 (m, 2H, $CONCH_2CH_2$), 1.52 (m, 2H, $CONCH_2CH_2$), 1.64 (br s, 2H, $NHCH_2CH_2CH_2CH_2N$), 1.89 (m, 2H, $NCH_2CH_2CH_2NHDde$), 2.13 (br s, 2H, $NHCH_2CH_2CH_2CH_2N$), 2.32 (s, 10H, 4 × $COCH_2$, $DdeNHCH_2CH_2CH_2NH$), 2.54 and 2.55 (s, 6H, 2 × $CH_3C{=}C$), 2.69 (t, J = 4.5 Hz, 2H, $NHCH_2CH_2CH_2CH_2N$), 2.86 (m, 2H, $NCH_2CH_2CH_2NHDde$), 2.96 (t, J = 4.7 Hz, 2H, $NHCH_2CH_2CH_2CH_2N$), 3.09 (t, J = 7.3 Hz, 2H, $DdeNHCH_2CH_2CH_2NH$), 3.16 (t, J = 7.5 Hz, 2H, NCH_2CH_2), 3.27 (t, J = 7.4 Hz, 2H, NCH_2CH_2), 3.30 (s, 2H, NCH_2CO), 3.45 (q, J = 5.5 Hz, 2H, $NCH_2CH_2CH_2NHDde$), 3.56 (q, J = 5.5 Hz, 2H, $DdeNHCH_2CH_2CH_2NH$), 13.46 (br s, 1H, N*H*Dde), 13.50 (br s, 1H, N*H*Dde); ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.1, 18.0, 18.1, 22.7, 25.4, 25.8, 26.9, 27.1, 27.7, 28.2, 29.3, 29.4, 29.7, 30.0, 31.9, 41.2, 41.5, 46.4, 46.6, 47.5, 48.5, 52.8, 53.6, 55.4, 55.9, 108.0, 108.1, 170.0, 173.8, 174.0, 197.8; HRMS (ESI-TOF), m/z calcd for $C_{64}H_{118}N_5O_5$ $[M+H]^+$ 1036.9127; found 1036.9129.

Compound 10 (C16,C18)



Yield: 65%; IR: ν_{max}, cm^{-1} : 3443, 2921, 2852, 1636, 1572, 1463, 1368, 1337, 1305, 1143, 1092, 1029, 884, 721; ^1H NMR (400 MHz, $CDCl_3$): δ 0.85 (t, J = 6.6 Hz, 6H, 2 × CH_3), 1.00 (s, 12H, 2 × C(CH_3)$_2$), 1.23 (s, 56H, (CH_2)$_{13}$ and (CH_2)$_{15}$), 1.45 (m, 2H, CONCH$_2$CH_2), 1.53 (m, 2H, CONCH$_2$CH_2), 1.65 (br s, 2H, NHCH$_2$CH$_2$CH_2CH$_2$N), 1.90 (m, 2H, NCH$_2$CH_2CH$_2$NHDde), 2.14 (br s, 2H, NHCH$_2$CH_2CH$_2$CH$_2$N), 2.33 (s, 10H, 4 × COCH_2, DdeNHCH$_2$CH_2CH$_2$NH), 2.54 and 2.55 (s, 6H, 2 × CH_3C=C), 2.69 (t, J = 4.2 Hz, 2H, NHCH$_2$CH$_2$CH$_2$CH_2N), 2.86 (m, 2H, NCH_2CH$_2$CH$_2$NHDde), 2.96 (t, J = 5.0 Hz, 2H, NHCH_2CH$_2$CH$_2$CH$_2$N), 3.10 (t, J = 7.4 Hz, 2H, DdeNHCH$_2$CH$_2$CH_2NH), 3.16 (t, J = 7.6 Hz, 2H, NCH_2CH$_2$), 3.26 (t, J = 7.6 Hz, 2H, NCH_2CH$_2$), 3.30 (s, 2H, NCH_2CO), 3.45 (q, J = 5.3 Hz, 2H, NCH$_2$CH$_2$CH_2NHDde), 3.57 (q, J = 5.5 Hz, 2H, DdeNHCH_2CH$_2$CH$_2$NH), 13.46 (br s, 1H, N*H*Dde), 13.50 (br s, 1H, N*H*Dde); ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.1, 18.0, 18.1, 22.7, 25.4, 25.8, 26.9, 27.1, 27.7, 28.3, 29.3, 29.6, 29.7, 30.1, 31.9, 41.2, 41.5, 46.4, 46.6, 47.5, 48.5, 52.8, 53.6, 55.4, 56.0, 108.0, 108.1, 170.0, 173.8, 174.0, 197.8; HRMS (ESI-TOF), m/z calcd for $C_{66}H_{112}N_5O_5$ $[M+H]^+$ 1064.9440; found 1064.9419.

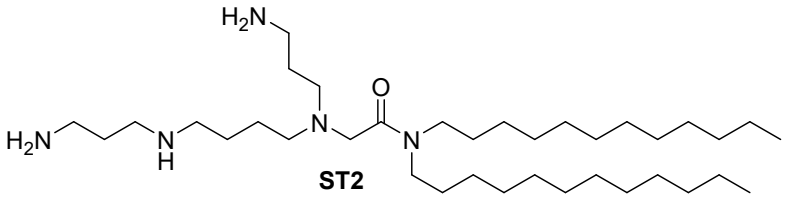
Lipid ST1



Yield: 75%; IR: ν_{max}, cm^{-1} : 3402, 3265, 2922, 2853, 1635, 1548, 1460, 1374, 1276, 1110, 1074, 751, 722; ^1H NMR (400 MHz, $CDCl_3$): δ 0.86 (t, J = 6.7 Hz, 6H, 2 × CH_3), 1.23 (s, 28H, 2 × (CH_2)$_7$), 1.47−1.53 (m, 6H, 2 × NCH$_2$CH_2, NHCH$_2$CH$_2$CH_2CH$_2$N), 1.65 (m, 2H, NHCH$_2$CH_2CH$_2$CH$_2$N), 1.82 (m, 2H, NCH$_2$CH_2CH$_2$NH$_2$), 1.89−1.91 (m, 2H, NH$_2$CH$_2$CH_2CH$_2$NH), 2.46 (t, J = 6.6 Hz, 2H, NHCH$_2$CH$_2$CH$_2$CH_2N), 2.53 (t, J = 5.3 Hz,

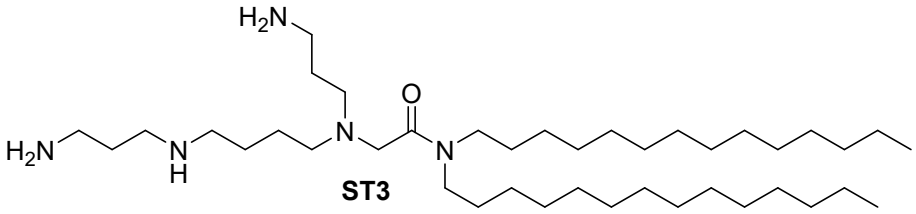
2H, NCH_2CH$_2$CH$_2$NH$_2$), 2.74 (t, J = 6.4 Hz, 2H, NHCH_2CH$_2$CH$_2$CH$_2$N), 2.90 (t, J = 6.1 Hz, 2H, NH$_2$CH_2CH$_2$CH$_2$NH), 2.97 (t, J = 5.9 Hz, 2H, NH$_2$CH$_2$CH$_2$CH_2NH), 3.07 (br s, 2H, NCH$_2$CH$_2$CH_2NH$_2$), 3.20 (m, 2H, NCH_2CO), 3.13 (t, J = 7.8 Hz, 2H, NCH_2CH$_2$), 3.23 (m, 2H, NCH_2CH$_2$); ^{13}C NMR (100 MHz, CDCl$_3$): δ 14.2, 22.7, 24.5, 25.1, 26.0, 26.9, 27.1, 27.0, 29.0, 29.3, 29.4, 29.5, 29.6, 29.7, 31.9, 39.8, 40.5, 46.5, 47.3, 47.7, 48.5, 53.8, 54.7, 55.8, 170.2; HRMS (ESI-TOF), m/z calcd for $C_{32}H_{70}N_5O$ [M+H]$^+$ 540.5575; found 540.5585.

Lipid ST2



Yield: 72%; IR: ν_{max}, cm^{-1} : 3430, 2921, 2852, 1628, 1460, 1374, 1312, 1266, 1123, 1077, 751, 722; ^{1}H NMR (400 MHz, CDCl$_3$): δ 0.86 (t, J = 5.9 Hz, 6H, 2 × CH_3), 1.23 (s, 36H, 2 × (CH_2)$_9$), 1.48 (m, 6H, 2 × NCH$_2$CH_2, NHCH$_2$CH$_2$CH_2CH$_2$N), 1.67 (br s, 2H, NHCH$_2$CH_2CH$_2$CH$_2$N), 1.81 (br s, 2H, NCH$_2$CH_2CH$_2$NH$_2$), 2.06 (m, 2H, NH$_2$CH$_2$CH_2CH$_2$NH), 2.42 (br s, 2H, NHCH$_2$CH$_2$CH$_2$CH_2N), 2.51 (br s, 2H, NCH_2CH$_2$CH$_2$NH$_2$), 2.89 (br s, 2H, NHCH_2CH$_2$CH$_2$CH$_2$N), 3.05−3.10 (m, 12H, NH$_2$CH_2CH$_2$CH$_2$NH, NH$_2$CH$_2$CH$_2$CH_2NH, NCH$_2$CH$_2$CH_2NH$_2$, NCH_2CO, 2 × NCH_2CH$_2$); ^{13}C NMR (100 MHz, CDCl$_3$): δ 14.1, 22.7, 24.5, 24.9, 26.7, 27.1, 27.6, 28.9, 29.3, 29.5, 29.6, 31.9, 38.2, 40.5, 46.2, 46.7, 47.4, 48.1, 53.7, 54.9, 55.5, 170.2; HRMS (ESI-TOF), m/z calcd for $C_{36}H_{78}N_5O$ [M+H]$^+$ 596.6201; found 596.6220.

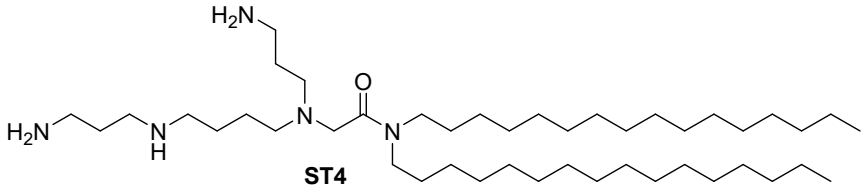
Lipid ST3



Yield: 79%; IR: ν_{max}, cm^{-1} : 3423, 2922, 2853, 1636, 1551, 1462, 1375, 1314, 1271, 1092, 1013, 753; ^{1}H NMR (400 MHz, CDCl$_3$): δ 0.86 (t, J = 6.6 Hz, 6H, 2 × CH_3), 1.21 (s, 44H, 2 × (CH_2)$_{11}$), 1.47−1.57 (m, 8H, 2 × NCH$_2$CH_2, NHCH$_2$CH$_2$CH_2CH$_2$N, NHCH$_2$CH_2CH$_2$CH$_2$N), 1.71−1.77 (m, 4H, NCH$_2$CH_2CH$_2$NH$_2$, NH$_2$CH$_2$CH_2CH$_2$NH), 2.45 (t, J = 6.6 Hz, 2H,

NHCH$_2$CH$_2$CH$_2$C*H*$_2$N), 2.51 (t, *J* = 6.3 Hz, 2H, NC*H*$_2$CH$_2$CH$_2$NH$_2$), 2.65 (t, *J* = 6.6 Hz, 2H, NHC*H*$_2$CH$_2$CH$_2$CH$_2$N), 2.77 (t, *J* = 6.6 Hz, 2H, NH$_2$C*H*$_2$CH$_2$CH$_2$NH), 2.85 (t, *J* = 6.5 Hz, 2H, NH$_2$CH$_2$CH$_2$C*H*$_2$NH), 2.94 (t, *J* = 5.8 Hz, 2H, NCH$_2$CH$_2$C*H*$_2$NH$_2$), 3.13−3.22 (m, 6H, NC*H*$_2$CO, 2 × NC*H*$_2$CH$_2$); ^{13}C NMR (100 MHz, CDCl$_3$): *δ* 14.2, 22.6, 24.5, 24.9, 26.0, 26.6, 26.9, 27.0, 27.5, 28.9, 29.3, 29.4, 29.5, 29.6, 31.8, 39.9, 40.2, 46.2, 47.2, 47.6, 48.9, 53.7, 53.8, 170.1; HRMS (ESI-TOF), *m*/*z* calcd for $C_{40}H_{86}N_5O$ $[M+H]^+$ 652.6826; found 652.6833.

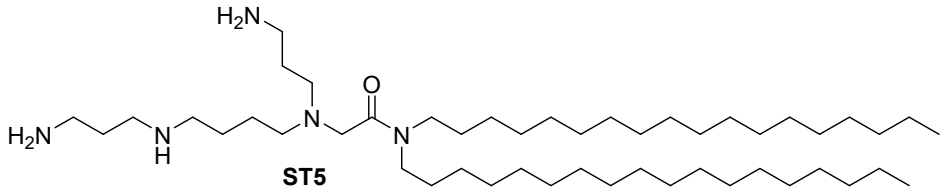
Lipid ST4



ST4

Yield: 77%; IR: ν_{max}, cm^{-1} : 3438, 2919, 2851, 1681, 1633, 1465, 1431, 1378, 1273, 1197, 1129, 1021, 831, 800, 753, 720; ^{1}H NMR (400 MHz, CDCl$_3$): *δ* 0.86 (t, *J* = 6.6 Hz, 6H, 2 × C*H*$_3$), 1.23 (s, 52H, 2 × (C*H*$_2$)$_{13}$), 1.50 (m, 6H, 2 × NCH$_2$C*H*$_2$, NHCH$_2$CH$_2$C*H*$_2$CH$_2$N), 1.61 (m, 2H, NHCH$_2$C*H*$_2$CH$_2$CH$_2$N), 1.80 (m, 2H, NCH$_2$C*H*$_2$CH$_2$NH$_2$), 1.95 (m, 2H, NH$_2$CH$_2$C*H*$_2$CH$_2$NH), 2.41 (br s, 2H, NHCH$_2$CH$_2$CH$_2$C*H*$_2$N), 2.52 (br s, 2H, NC*H*$_2$CH$_2$CH$_2$NH$_2$), 2.80 (t, *J* = 6.3 Hz, 2H, NHC*H*$_2$CH$_2$CH$_2$CH$_2$N), 2.99 (t, *J* = 6.3 Hz, 2H, NH$_2$C*H*$_2$CH$_2$CH$_2$NH), 3.04 (t, *J* = 6.0 Hz, 2H, NH$_2$CH$_2$CH$_2$C*H*$_2$NH), 3.10 (br s, 4H, NCH$_2$CH$_2$C*H*$_2$NH$_2$, NC*H*$_2$CH$_2$) 33.17−3.24 (m, 4H, NC*H*$_2$CO, NC*H*$_2$CH$_2$); ^{13}C NMR (100 MHz, CDCl$_3$): *δ* 14.1, 22.7, 24.9, 25.3, 25.6, 26.9, 27.1, 27.6, 28.9, 29.3, 29.4, 29.6, 29.7, 31.9, 39.2, 40.6, 46.7, 47.3, 48.2, 53.8, 55.0, 55.4, 170.2; HRMS (ESI-TOF), *m*/*z* calcd for $C_{44}H_{94}N_5O$ $[M+H]^+$ 708.7453; found 708.7451.

Lipid ST5

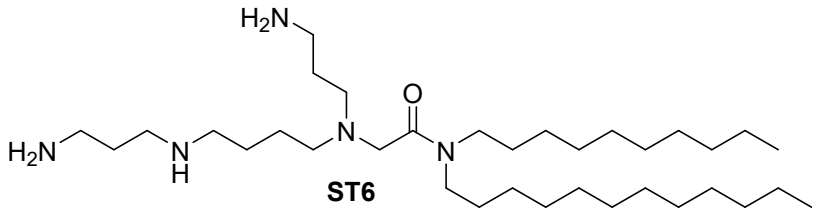


ST5

Yield: 86%; IR: ν_{max}, cm^{-1} : 3366, 2918, 2850, 1689, 1635, 1561, 1464, 1434, 1375, 1199, 1176, 1128, 1018, 751, 720; ^{1}H NMR (400 MHz, CDCl$_3$): *δ* 0.83 (t, *J* = 6.7 Hz, 6H, 2 ×

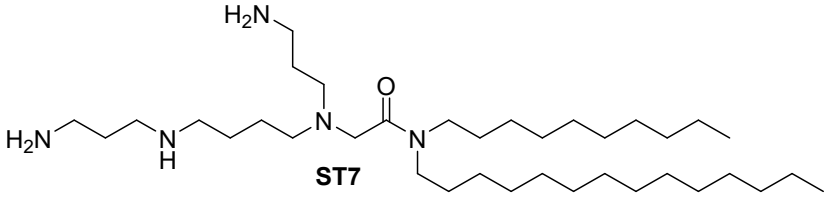
CH_3), 1.20 (s, 60H, 2 × (CH_2)$_{15}$), 1.44−1.58 (m, 8H, 2 × NCH$_2$CH_2, NHCH$_2$CH$_2$CH_2CH$_2$N, NHCH$_2$CH_2CH$_2$CH$_2$N), 1.72−1.78 (m, 4H, NCH$_2$CH_2CH$_2$NH$_2$, NH$_2$CH$_2$CH_2CH$_2$NH), 2.44 (t, J = 6.4 Hz, 2H, NHCH$_2$CH$_2$CH$_2$CH_2N), 2.51 (t, J = 5.8 Hz, 2H, NCH_2CH$_2$CH$_2$NH$_2$), 2.66 (t, J = 6.8 Hz, 2H, NHCH_2CH$_2$CH$_2$CH$_2$N), 2.79 (t, J = 6.8 Hz, 2H, NH$_2$CH_2CH$_2$CH$_2$NH), 2.86 (t, J = 6.4 Hz, 2H, NH$_2$CH$_2$CH$_2$CH_2NH), 2.96 (t, J = 5.9 Hz, 2H, NCH$_2$CH$_2$CH_2NH$_2$), 3.12−3.22 (m, 6H, NCH_2CO, 2 × NCH_2CH$_2$); ^{13}C NMR (100 MHz, CDCl$_3$): δ 14.1, 22.6, 24.9, 25.6, 26.4, 26.9, 27.0, 27.6, 28.9, 29.3, 29.4, 29.5, 29.6, 31.8, 40.0, 40.2, 46.3, 47.2, 47.7, 48.7, 53.7, 53.8, 55.8, 170.1; HRMS (ESI-TOF), m/z calcd for C$_{48}$H$_{102}$N$_5$O [M+H]$^+$ 764.8079; found 764.8094.

Lipid ST6



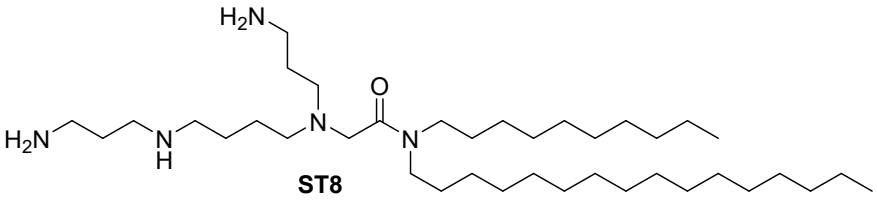
Yield: 81%; IR: ν_{max}, cm^{-1} : 3383, 2922, 2853, 1634, 1561, 1461, 1377, 1312, 1271, 1111, 1082, 1013, 752, 720; ^{1}H NMR (400 MHz, CDCl$_3$): δ 0.83 (t, J = 6.6 Hz, 6H, 2 × CH_3), 1.21 (s, 32H, (CH_2)$_7$ and (CH_2)$_9$), 1.45−1.53 (m, 8H, 2 × NCH$_2$CH_2, NHCH$_2$CH$_2$CH_2CH$_2$N, NHCH$_2$CH_2CH$_2$CH$_2$N), 1.67−1.77 (m, 4H, NCH$_2$CH_2CH$_2$NH$_2$, NH$_2$CH$_2$CH_2CH$_2$NH), 2.45 (t, J = 6.6 Hz, 2H, NHCH$_2$CH$_2$CH$_2$CH_2N), 2.51 (t, J = 6.1 Hz, 2H, NCH_2CH$_2$CH$_2$NH$_2$), 2.63 (t, J = 6.6 Hz, 2H, NHCH_2CH$_2$CH$_2$CH$_2$N), 2.75 (t, J = 6.7 Hz, 2H, NH$_2$CH_2CH$_2$CH$_2$NH), 2.85 (t, J = 6.5 Hz, 2H, NH$_2$CH$_2$CH$_2$CH_2NH), 2.91 (t, J = 6.0 Hz, 2H, NCH$_2$CH$_2$CH_2NH$_2$), 3.14−3.23 (m, 6H, NCH_2CO, 2 × NCH_2CH$_2$); ^{13}C NMR (100 MHz, CDCl$_3$): δ 14.1, 22.6, 24.7, 24.9, 26.5, 26.8, 26.9, 27.1, 27.6, 28.9, 29.2, 29.3, 29.4, 29.5, 29.6, 30.0, 31.8, 40.0, 40.2, 46.2, 47.2, 47.7, 49.0, 53.5, 53.8, 56.1, 170.0; HRMS (ESI-TOF), m/z calcd for C$_{34}$H$_{74}$N$_5$O [M+H]$^+$ 568.5888; found 568.5899.

Lipid ST7



Yield: 78%; IR: ν_{max}, cm^{-1} : 3408, 2919, 2851, 1677, 1634, 1465, 1429, 1376, 1197, 1129, 831, 800, 719; ^1H NMR (400 MHz, $CDCl_3$): δ 0.85 (t, J = 6.6 Hz, 6H, 2 × CH_3), 1.23 (s, 36H, (CH_2)$_7$ and (CH_2)$_{11}$), 1.51 (m, 4H, 2 × NCH$_2$CH_2), 1.66 (br s, 2H, NHCH$_2$CH$_2$CH_2CH$_2$N), 1.85 (br s, 2H, NHCH$_2$CH_2CH$_2$CH$_2$N), 1.97 (br s, 2H, NCH$_2$CH_2CH$_2$NH$_2$), 2.25 (br s, 2H, NH$_2$CH$_2$CH_2CH$_2$NH), 2.50 (br s, 2H, NHCH$_2$CH$_2$CH$_2$CH_2N), 2.56 (br s, 2H, NCH_2CH$_2$CH$_2$NH$_2$), 3.07 (br s, 2H, NHCH_2CH$_2$CH$_2$CH$_2$N), 3.15 (br s, 2H, NH$_2$CH_2CH$_2$CH$_2$NH), 3.22−3.28 (m, 10H, NH$_2$CH$_2$CH$_2$CH_2NH, NCH$_2$CH$_2$CH_2NH$_2$, NCH_2CO, 2 × NCH_2CH$_2$); ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.2, 22.6, 24.6, 25.0, 25.3, 26.9, 27.2, 27.8, 29.0, 29.3, 29.4, 29.5, 29.6, 31.9, 38.8, 41.2, 46.7, 46.9, 47.7, 48.2, 53.8, 55.0, 56.0, 170.6; HRMS (ESI-TOF), m/z calcd for $C_{44}H_{78}N_5O$ $[M+H]^+$ 596.6201; found 596.6219.

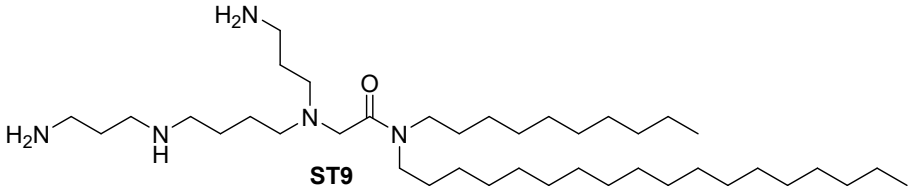
Lipid ST8



ST8

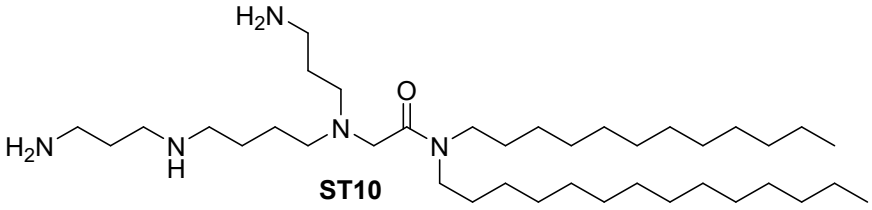
Yield: 77%; IR: ν_{max}, cm^{-1} : 3401, 2922, 2853, 1679, 1638, 1461, 1374, 1198, 1171, 1126, 1014, 799, 719; ^1H NMR (400 MHz, $CDCl_3$): δ 0.85 (t, J = 6.6 Hz, 6H, 2 × CH_3), 1.22 (s, 40H, (CH_2)$_7$ and (CH_2)$_{13}$), 1.51 (m, 6H, 2 × NCH$_2$CH_2, NHCH$_2$CH$_2$CH_2CH$_2$N), 1.65 (m, 2H, NHCH$_2$CH_2CH$_2$CH$_2$N), 1.76−1.784 (m, 4H, NCH$_2$CH_2CH$_2$NH$_2$, NH$_2$CH$_2$CH_2CH$_2$NH), 2.48 (t, J = 7.3 Hz, 2H, NHCH$_2$CH$_2$CH$_2$CH_2N), 2.55 (t, J = 6.0 Hz, 2H, NCH_2CH$_2$CH$_2$NH$_2$), 2.74 (t, J = 6.6 Hz, 2H, NHCH_2CH$_2$CH$_2$CH$_2$N), 2.88 (m, 4H, NH$_2$CH_2CH$_2$CH$_2$NH, NH$_2$CH$_2$CH$_2$CH_2NH), 2.90 (t, J = 5.7 Hz, 2H, NCH$_2$CH$_2$CH_2NH$_2$), 3.15−3.25 (m, 6H, NCH_2CO, 2 × NCH_2CH$_2$); ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.1, 22.6, 25.0, 26.1, 26.5, 26.9, 27.1, 27.9, 29.0, 29.3, 29.4, 29.5, 29.6, 31.9, 40.3, 40.5, 46.4, 47.4, 48.0, 48.7, 50.6, 53.9, 54.0, 56.0, 170.2; HRMS (ESI-TOF), m/z calcd for $C_{38}H_{82}N_5O$ $[M+H]^+$ 624.6514; found 624.6538.

Lipid ST9



Yield: 76%; IR: ν_{max}, cm^{-1} : 3395, 2921, 2852, 1637, 1554, 1461, 1375, 1314, 1108, 1018, 720; ^1H NMR (400 MHz, $CDCl_3$): δ 0.83 (t, J = 6.6 Hz, 6H, 2 × CH_3), 1.21 (s, 44H, (CH_2)$_7$ and (CH_2)$_{15}$), 1.45−1.56 (m, 8H, 2 × NCH$_2$CH_2, NHCH$_2$CH$_2$CH_2CH$_2$N, NHCH$_2$CH_2CH$_2$CH$_2$N), 1.74 (m, 4H, NCH$_2$CH_2CH$_2$NH$_2$, NH$_2$CH$_2$CH_2CH$_2$NH), 2.45 (t, J = 6.7 Hz, 2H, NHCH$_2$CH$_2$CH$_2$CH_2N), 2.52 (t, J = 6.0 Hz, 2H, NCH_2CH$_2$CH$_2$NH$_2$), 2.66 (t, J = 6.5 Hz, 2H, NHCH_2CH$_2$CH$_2$CH$_2$N), 2.78 (t, J = 6.6 Hz, 2H, NH$_2$CH_2CH$_2$CH$_2$NH), 2.85 (t, J = 6.3 Hz, 2H, NH$_2$CH$_2$CH$_2$CH_2NH), 2.92 (t, J = 5.8 Hz, 2H, NCH$_2$CH$_2$CH_2NH$_2$), 3.13−3.23 (m, 6H, NCH_2CO, 2 × NCH_2CH$_2$); ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.0, 22.6, 24.9, 26.4, 26.7, 26.9, 27.1, 27.6, 28.9, 29.3, 29.4, 29.5, 29.6, 29.9, 31.8, 40.1, 40.2, 46.2, 47.2, 47.8, 48.9, 53.5, 53.9, 56.1, 170.1; HRMS (ESI-TOF), m/z calcd for $C_{40}H_{86}N_5O$ $[M+H]^+$ 652.6826; found 652.6835.

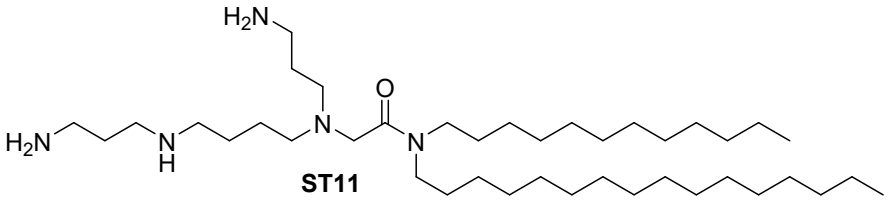
Lipid ST10



Yield: 72%; IR: ν_{max}, cm^{-1} : 3419, 2921, 2852, 1637, 1551, 1461, 1374, 1311, 1086, 720; ^1H NMR (400 MHz, $CDCl_3$): δ 0.85 (t, J = 7.0 Hz, 6H, 2 × CH_3), 1.21 (s, 40H, (CH_2)$_9$ and (CH_2)$_{11}$), 1.48 (m, 6H, 2 × NCH$_2$CH_2, NHCH$_2$CH$_2$CH_2CH$_2$N), 1.63 (m, 2H, NHCH$_2$CH_2CH$_2$CH$_2$N), 1.76−1.82 (m, 4H, NCH$_2$CH_2CH$_2$NH$_2$, NH$_2$CH$_2$CH_2CH$_2$NH), 2.47 (t, J = 6.7 Hz, 2H, NHCH$_2$CH$_2$CH$_2$CH_2N), 2.54 (t, J = 6.0 Hz, 2H, NCH_2CH$_2$CH$_2$NH$_2$), 2.72 (t, J = 6.5 Hz, 2H, NHCH_2CH$_2$CH$_2$CH$_2$N), 2.85 (t, J = 6.6 Hz, 2H, NH$_2$CH_2CH$_2$CH$_2$NH), 2.90 (t, J = 6.3 Hz, 2H, NH$_2$CH$_2$CH$_2$CH_2NH), 2.99 (t, J = 5.7 Hz, 2H, NCH$_2$CH$_2$CH_2NH$_2$) 3.14−3.24 (m, 6H, NCH_2CO, 2 × NCH_2CH$_2$); ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.1, 22.6, 25.0, 25.9, 26.4, 26.9, 27.1, 27.7, 29.0, 29.3, 29.4, 29.5, 29.6, 31.9, 40.3, 40.5, 46.4, 47.3,

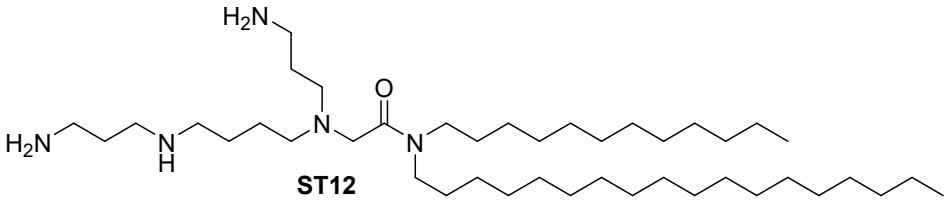
48.0, 48.7, 53.9, 56.0, 170.2; HRMS (ESI-TOF), *m/z* calcd for $C_{38}H_{82}N_5O$ $[M+H]^+$ 624.6514; found 624.6525.

Lipid ST11



Yield: 80%; IR: ν_{max}, cm^{-1} : 3419, 2921, 2852, 1638, 1551, 1461, 1374, 1311, 1248, 1086, 1011, 721; ^1H NMR (400 MHz, $CDCl_3$): δ 0.85 (t, J = 6.6 Hz, 6H, 2 × CH_3), 1.21 (s, 44H, (CH_2)$_9$ and (CH_2)$_{13}$), 1.51 (m, 6H, 2 × NCH$_2$CH_2, NHCH$_2$CH$_2$CH_2CH$_2$N), 1.59 (m, 2H, NHCH$_2$CH_2CH$_2$CH$_2$N), 1.70−1.77 (m, 4H, NCH$_2$CH_2CH$_2$NH$_2$, NH$_2$CH$_2$CH_2CH$_2$NH), 2.48 (t, J = 7.1 Hz, 2H, NHCH$_2$CH$_2$CH$_2$CH_2N), 2.54 (t, J = 6.3 Hz, 2H, NCH_2CH$_2$CH$_2$NH$_2$), 2.68 (t, J = 6.6 Hz, 2H, NHCH_2CH$_2$CH$_2$CH$_2$N), 2.79 (t, J = 6.7 Hz, 2H, NH$_2$CH_2CH$_2$CH$_2$NH), 2.85 (t, J = 6.5 Hz, 2H, NH$_2$CH$_2$CH$_2$CH_2NH), 2.91 (t, J = 6.1 Hz, 2H, NCH$_2$CH$_2$CH_2NH$_2$), 3.17−3.25 (m, 6H, NCH_2CO, 2 × NCH_2CH$_2$); ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.1, 22.6, 24.9, 26.8, 26.9, 27.1, 27.2, 27.6, 29.0, 29.3, 29.4, 29.5, 29.6, 29.7, 31.9, 40.4, 40.5, 46.2, 47.3, 48.0, 49.0, 53.5, 54.0, 56.3, 170.2; HRMS (ESI-TOF), *m/z* calcd for $C_{40}H_{86}N_5O$ $[M+H]^+$ 652.6827; found 652.6830.

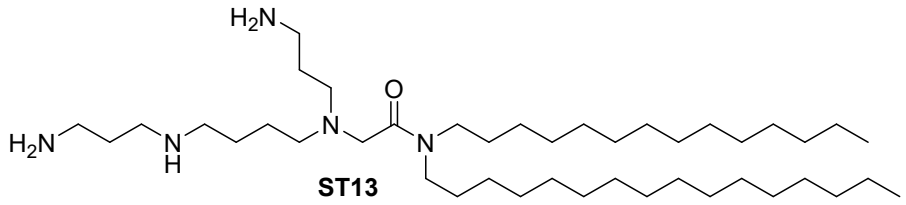
Lipid ST12



Yield: 77%; IR: ν_{max}, cm^{-1} : 3393, 2922, 2853, 1637, 1554, 1460, 1372, 1294, 1113, 784, 719; ^1H NMR (400 MHz, $CDCl_3$): δ 0.85 (t, J = 6.7 Hz, 6H, 2 × CH_3), 1.23 (s, 48H, (CH_2)$_9$ and (CH_2)$_{15}$), 1.50 (m, 6H, 2 × NCH$_2$CH_2, NHCH$_2$CH$_2$CH_2CH$_2$N), 1.60 (m, 2H, NHCH$_2$CH_2CH$_2$CH$_2$N), 1.74−1.81 (m, 4H, NCH$_2$CH_2CH$_2$NH$_2$, NH$_2$CH$_2$CH_2CH$_2$NH), 2.48 (t, J = 6.8 Hz, 2H, NHCH$_2$CH$_2$CH$_2$CH_2N), 2.54 (t, J = 6.1 Hz, 2H, NCH_2CH$_2$CH$_2$NH$_2$), 2.70 (t, J = 6.6 Hz, 2H, NHCH_2CH$_2$CH$_2$CH$_2$N), 2.82 (t, J = 6.7 Hz, 2H, NH$_2$CH_2CH$_2$CH$_2$NH),

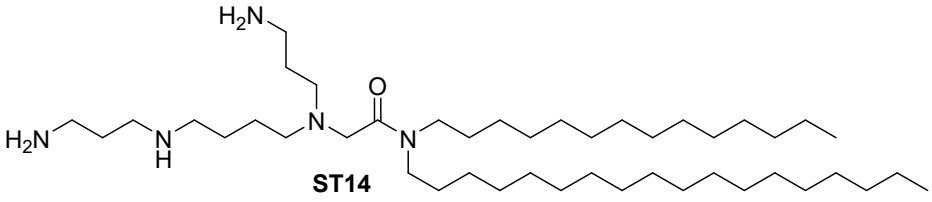
2.89 (t, J = 6.4 Hz, 2H, $NH_2CH_2CH_2CH_2NH$), 2.96 (t, J = 5.9 Hz, 2H, $NCH_2CH_2CH_2NH_2$), 3.16−3.25 (m, 6H, NCH_2CO, 2 × NCH_2CH_2); ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.1, 22.7, 25.0, 26.6, 26.7, 26.9, 27.1, 27.7, 29.0, 29.3, 29.4, 29.5, 29.6, 29.7, 31.9, 40.3, 40.5, 46.3, 47.3, 48.0, 48.9, 53.6, 54.0, 56.2, 170.2; HRMS (ESI-TOF), m/z calcd for $C_{42}H_{90}N_5O$ $[M+H]^+$ 680.7140; found 680.7153.

Lipid ST13



Yield: 89%; IR: ν_{max}, cm^{-1} : 3393, 2921, 2852, 1645, 1461, 1374, 1269, 1117, 753, 722; ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.85 (t, J = 6.6 Hz, 6H, 2 × CH_3), 1.22 (s, 48H, $(CH_2)_{11}$ and $(CH_2)_{13}$), 1.51 (m, 6H, 2 × NCH_2CH_2, $NHCH_2CH_2CH_2CH_2N$), 1.63 (m, 2H, $NHCH_2CH_2CH_2CH_2N$), 1.75−1.84 (m, 4H, $NCH_2CH_2CH_2NH_2$, $NH_2CH_2CH_2CH_2NH$), 2.48 (t, J = 6.7 Hz, 2H, $NHCH_2CH_2CH_2CH_2N$), 2.54 (t, J = 6.1 Hz, 2H, $NCH_2CH_2CH_2NH_2$), 2.72 (t, J = 6.7 Hz, 2H, $NHCH_2CH_2CH_2CH_2N$), 2.85 (t, J = 6.7 Hz, 2H, $NH_2CH_2CH_2CH_2NH$), 2.90 (t, J = 6.4 Hz, 2H, $NH_2CH_2CH_2CH_2NH$), 2.98 (t, J = 5.8 Hz, 2H, $NCH_2CH_2CH_2NH_2$), 3.15−3.24 (m, 6H, NCH_2CO, 2 × NCH_2CH_2); ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.1, 22.6, 25.0, 26.2, 26.5, 26.9, 27.1, 27.7, 29.0, 29.3, 29.4, 29.5, 29.6, 29.7, 31.9, 40.2, 40.5, 46.4, 47.3, 47.9, 48.8, 53.9, 54.0, 56.1, 170.2; HRMS (ESI-TOF), m/z calcd for $C_{42}H_{90}N_5O$ $[M+H]^+$ 680.7140; found 680.7133.

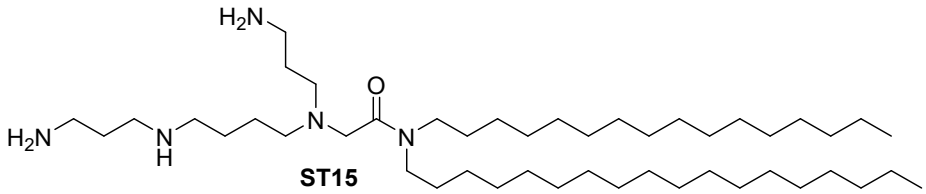
Lipid ST14



Yield: 79%; IR: ν_{max}, cm^{-1} : 3416, 2922, 2853, 1633, 1556, 1460, 1373, 1274, 1112, 1074, 1016, 751, 725; ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.84 (t, J = 6.7 Hz, 6H, 2 × CH_3), 1.22 (s, 52H, $(CH_2)_{11}$ and $(CH_2)_{15}$), 1.50 (m, 6H, 2 × NCH_2CH_2, $NHCH_2CH_2CH_2CH_2N$), 1.61 (m, 2H,

NHCH$_2$C*H*$_2$CH$_2$CH$_2$N), 1.71−1.78 (m, 4H, NCH$_2$C*H*$_2$CH$_2$NH$_2$, NH$_2$CH$_2$C*H*$_2$CH$_2$NH), 2.49 (t, *J* = 5.5 Hz, 2H, NHCH$_2$CH$_2$CH$_2$C*H*$_2$N), 2.55 (t, *J* = 6.4 Hz, 2H, NC*H*$_2$CH$_2$CH$_2$NH$_2$), 2.72 (t, *J* = 6.6 Hz, 2H, NHC*H*$_2$CH$_2$CH$_2$CH$_2$N), 2.83 (m, 4H, NH$_2$C*H*$_2$CH$_2$CH$_2$NH, NH$_2$CH$_2$CH$_2$C*H*$_2$NH), 2.91 (t, *J* = 5.8 Hz, 2H, NCH$_2$CH$_2$C*H*$_2$NH$_2$), 3.17−3.24 (m, 6H, NC*H*$_2$CO, 2 × NC*H*$_2$CH$_2$); ^{13}C NMR (100 MHz, CDCl$_3$): *δ* 14.1, 22.6, 25.0, 26.8, 26.9, 27.1, 27.2, 27.7, 29.0, 29.3, 29.4, 29.5, 29.7, 31.9, 40.4, 46.3, 47.3, 48.1, 48.8, 53.5, 54.0, 56.2, 170.2; HRMS (ESI-TOF), *m*/*z* calcd for $C_{44}H_{94}N_5O$ $[M+H]^+$ 708.7453; found 708.7451.

Lipid ST15



Yield: 79%; IR: ν_{max}, cm^{-1} : 3382, 2920, 2852, 1636, 1541, 1559, 1516, 1461, 1374, 1317, 1272, 1105, 752, 720; ^{1}H NMR (400 MHz, CDCl$_3$): *δ* 0.85 (t, *J* = 6.7 Hz, 6H, 2 × C*H*$_3$), 1.22 (s, 56H, (C*H*$_2$)$_{13}$ and (C*H*$_2$)$_{15}$), 1.51 (m, 6H, 2 × NCH$_2$C*H*$_2$, NHCH$_2$CH$_2$C*H*$_2$CH$_2$N), 1.62 (m, 2H, NHCH$_2$C*H*$_2$CH$_2$CH$_2$N), 1.75−1.82 (m, 4H, NCH$_2$C*H*$_2$CH$_2$NH$_2$, NH$_2$CH$_2$C*H*$_2$CH$_2$NH), 2.48 (t, *J* = 6.8 Hz, 2H, NHCH$_2$CH$_2$CH$_2$C*H*$_2$N), 2.54 (t, *J* = 6.1 Hz, 2H, NC*H*$_2$CH$_2$CH$_2$NH$_2$), 2.72 (t, *J* = 6.6 Hz, 2H, NHC*H*$_2$CH$_2$CH$_2$CH$_2$N), 2.84 (t, *J* = 6.6 Hz, 2H, NH$_2$C*H*$_2$CH$_2$CH$_2$NH), 2.90 (t, *J* = 6.4 Hz, 2H, NH$_2$CH$_2$CH$_2$C*H*$_2$NH), 2.98 (t, *J* = 5.8 Hz, 2H, NCH$_2$CH$_2$C*H*$_2$NH$_2$), 3.15−3.23 (m, 6H, NC*H*$_2$CO, 2 × NC*H*$_2$CH$_2$); ^{13}C NMR (100 MHz, CDCl$_3$): *δ* 14.1, 22.6, 25.0, 26.2, 26.5, 26.9, 27.1, 27.7, 29.0, 29.3, 29.4, 29.6, 29.7, 31.9, 40.3, 40.5, 46.4, 47.3, 48.0, 48.8, 53.9, 54.0, 56.1, 170.2; HRMS (ESI-TOF), *m*/*z* calcd for $C_{46}H_{98}N_5O$ $[M+H]^+$ 736.7766; found 736.7780.

Fig. S1 Gel electrophoresis Gel electrophoresis of cationic liposome/DNA complexes at N/P ratios of 0.5, 1, 2.5, 5, 10, 15, 20, and 25 with the final DNA concentration of 0.1 μg/well. The liposomes were formulated using cationic lipid alone and with the combination of DOPE (1:1 weight ratio).

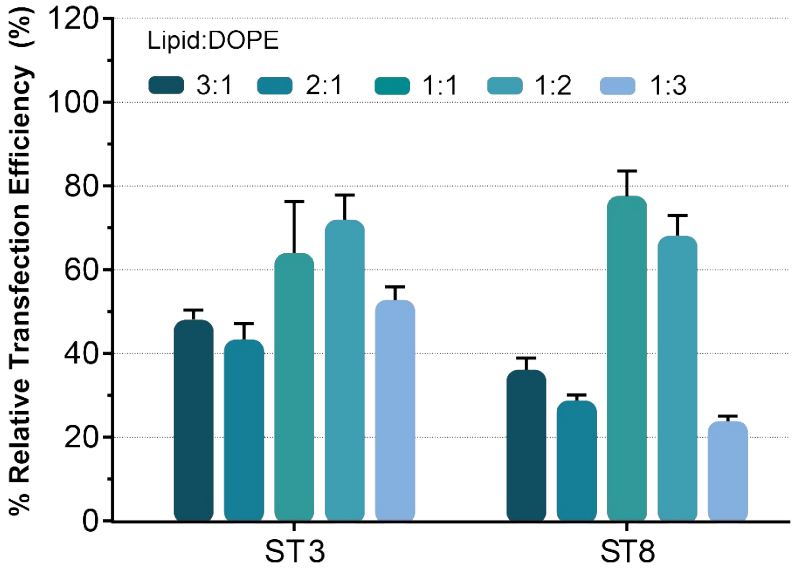


Fig. S2 Transfection efficiency of cationic lipids **ST3** and **ST8** with DOPE at various weight ratios against HEK293T. The relative transfection efficiency of the lipids was compared with that of L3K, which was calculated as 100% (data not shown). Each value represents the mean ± S.D. of three measurements.

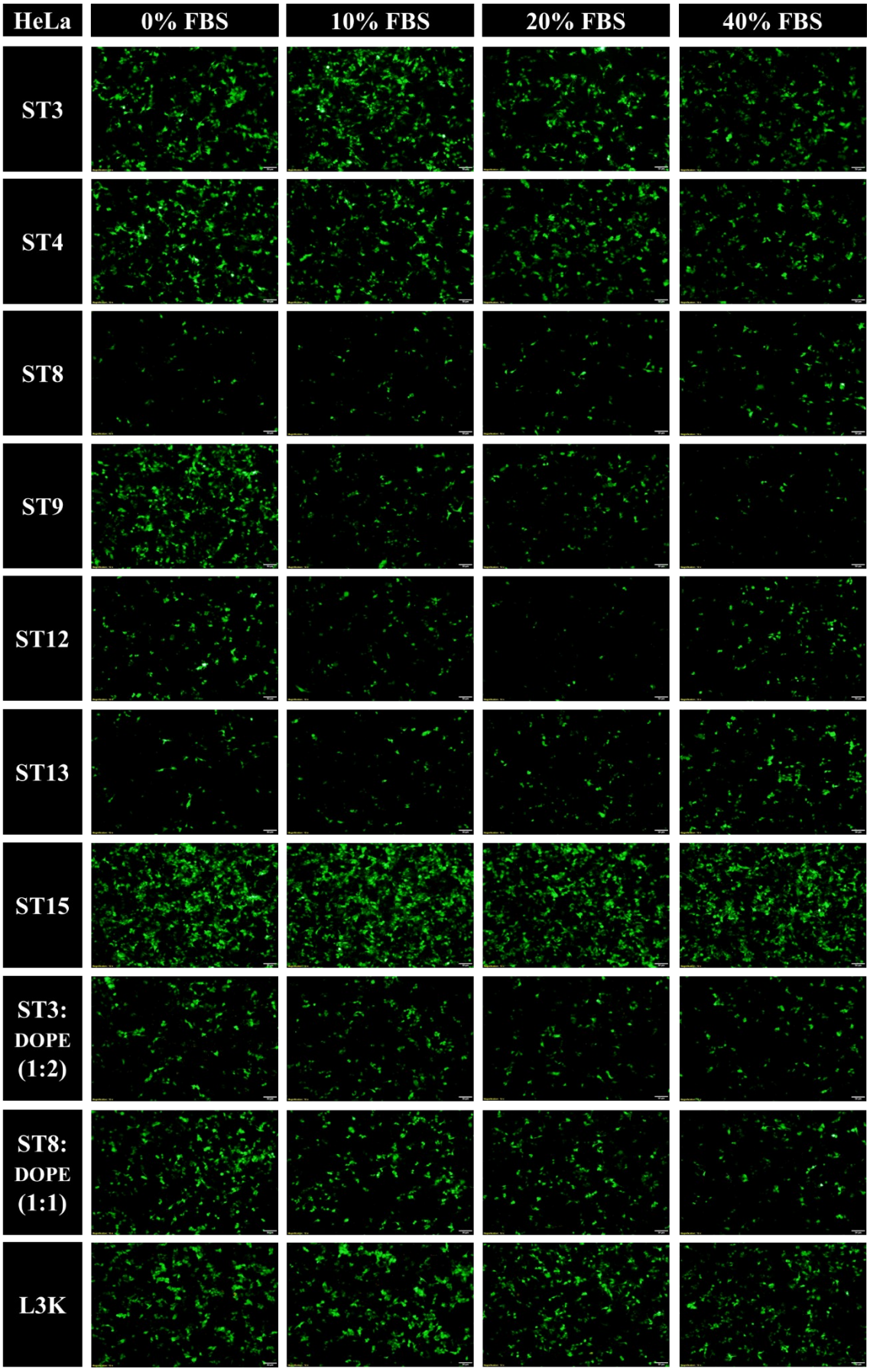


Fig. S3 Green fluorescent protein expression in HeLa cells transfected with lipoplexes of selected lipids under serum-free and in the presence of 10%, 20% and 40% serum medium.

The fluorescence microscope images were captured 48 h post-transfection under 10X magnification. Lipofectamine3000 (L3K) served as positive control. The scale bar is 50 μm.

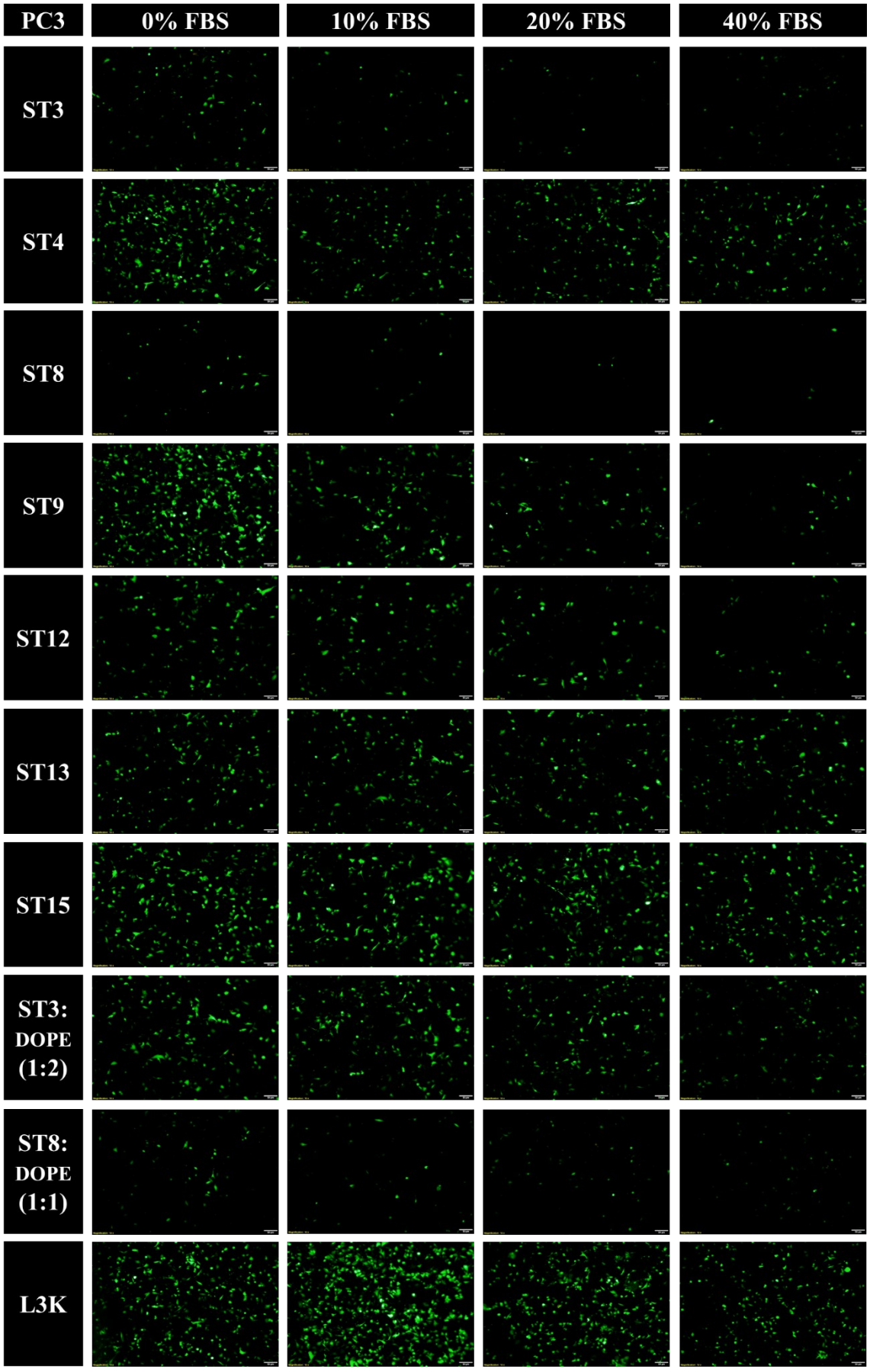


Fig. S4 Green fluorescent protein expression in PC3cells transfected with lipoplexes of selected lipids under serum-free and in the presence of 10%, 20% and 40% serum medium. The fluorescence microscope images were captured 48 h post-transfection under 10X magnification. Lipofectamine3000 (L3K) served as positive control. The scale bar is 50 μm.

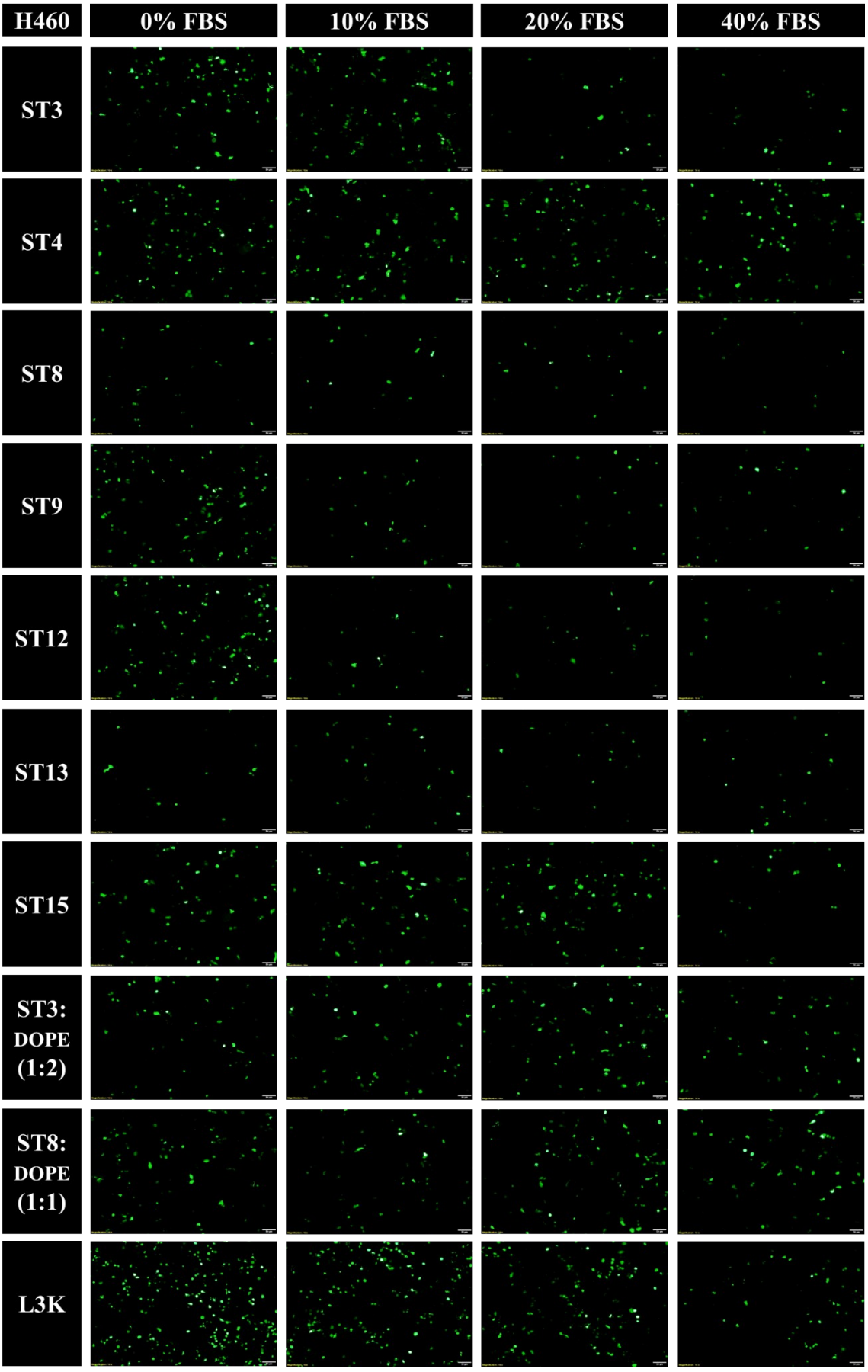


Fig. S5 Green fluorescent protein expression in H460 cells transfected with lipoplexes of selected lipids under serum-free and in the presence of 10%, 20% and 40% serum medium.

The fluorescence microscope images were captured 48 h post-transfection under 10X magnification. Lipofectamine3000 (L3K) served as positive control. The scale bar is 50 μm.

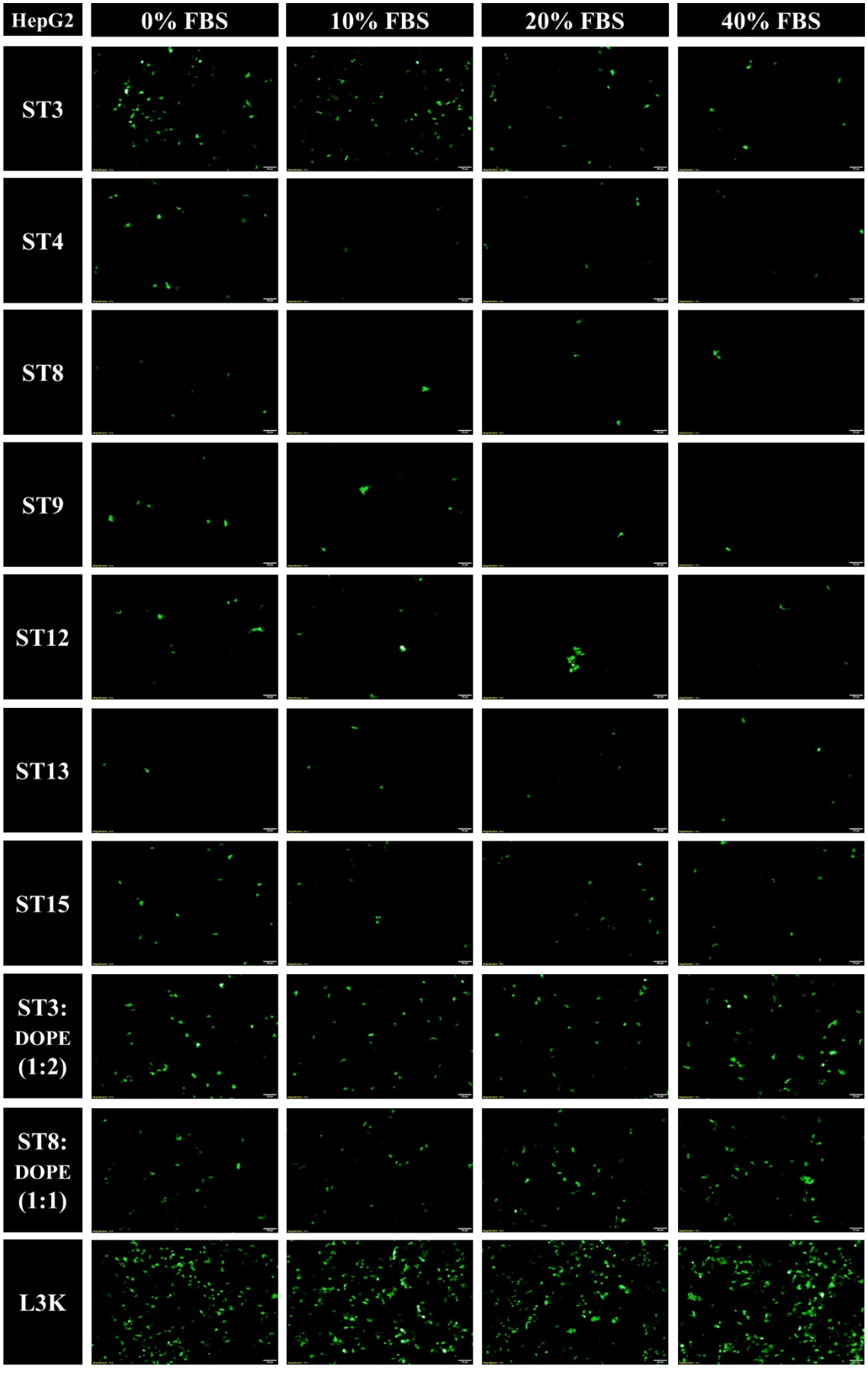


Fig. S6 Green fluorescent protein expression in HepG2cells transfected with lipoplexes of selected lipids under serum-free and in the presence of 10%, 20% and 40% serum medium. The fluorescence microscope images were captured 48 h post-transfection under 10X magnification. Lipofectamine3000 (L3K) served as positive control. The scale bar is 50 µm.

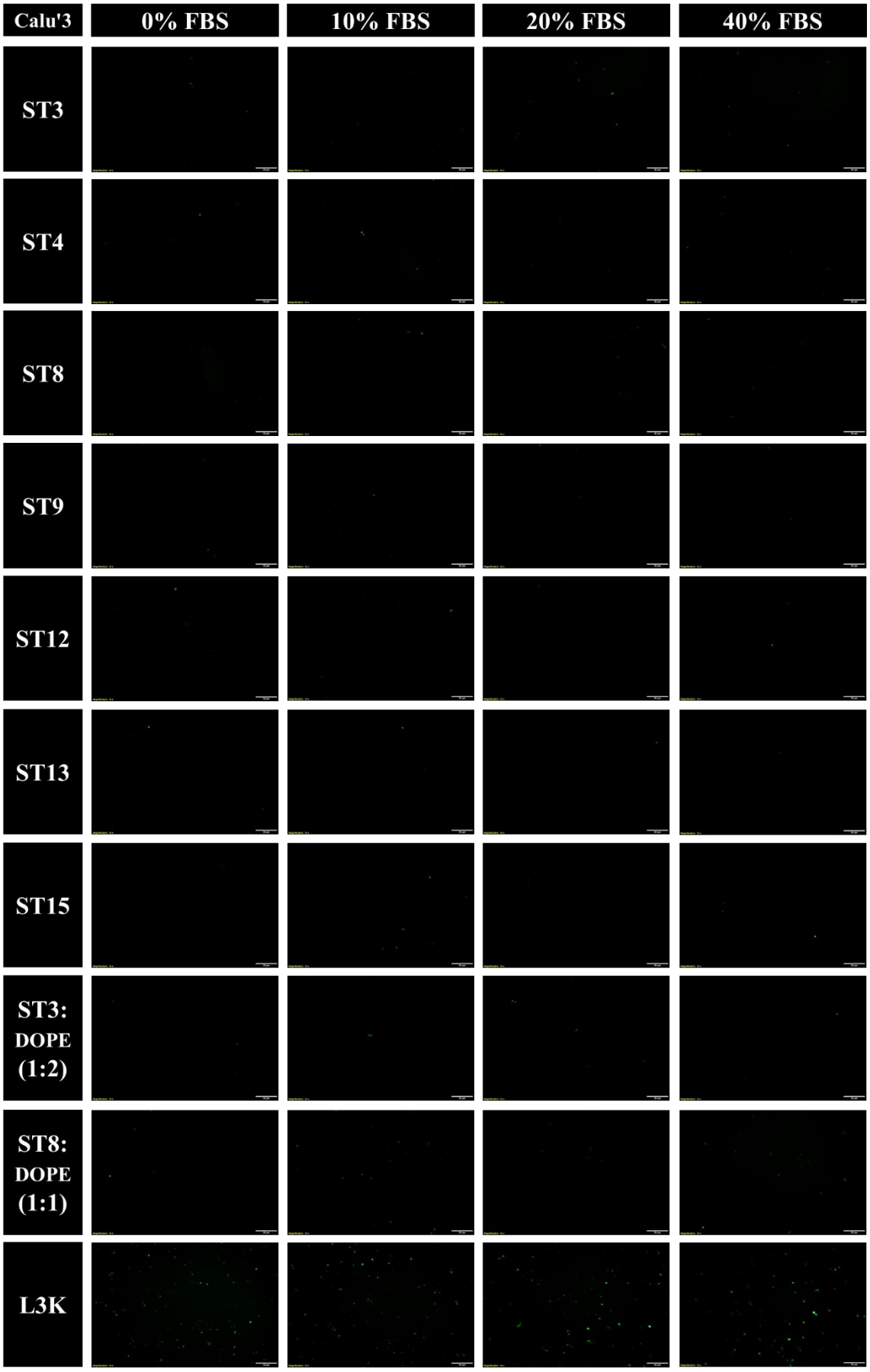


Fig. S7 Green fluorescent protein expression in Calu'3 cells transfected with lipoplexes of selected lipids under serum-free and in the presence of 10%, 20% and 40% serum medium.

The fluorescence microscope images were captured 48 h post-transfection under 16X magnification. Lipofectamine3000 (L3K) served as positive control. The scale bar is 50 μm.

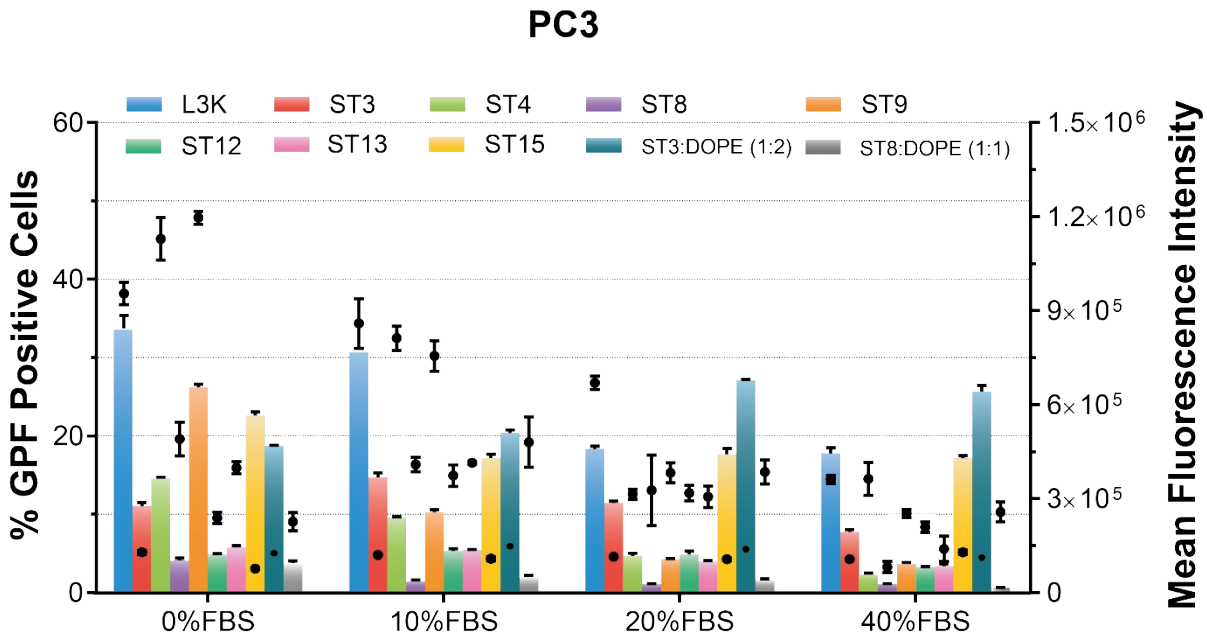


Fig. S8 The percentage of GFP positive cells (bar graph) and mean fluorescence intensity (■) of PC3 cells transfected with lipoplexes encapsulating pEGFP-C2 in the absence and presence of 10–40% serum. The expression of green fluorescent protein was measured by flow cytometer. Each value represents the mean ± S.D. of three measurements.

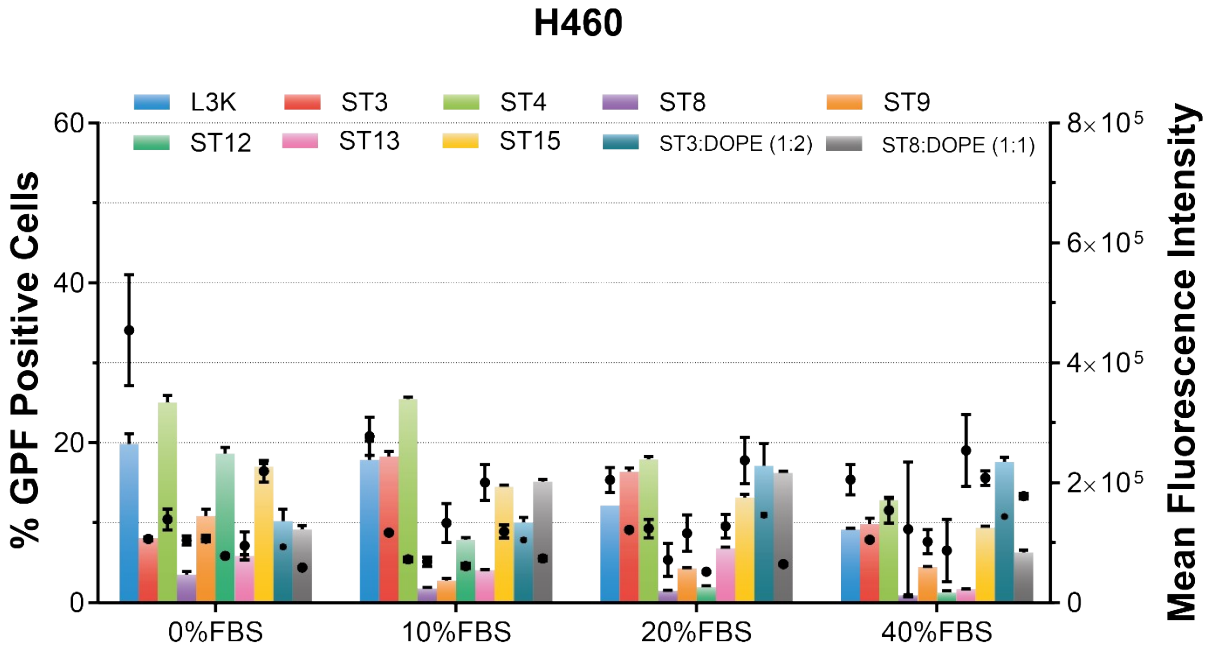


Fig. S9 The percentage of GFP positive cells (bar graph) and mean fluorescence intensity (■) of H460 cells transfected with lipoplexes encapsulating pEGFP-C2 in the absence and presence of 10–40% serum. The expression of green fluorescent protein was measured by flow cytometer. Each value represents the mean ± S.D. of three measurements.

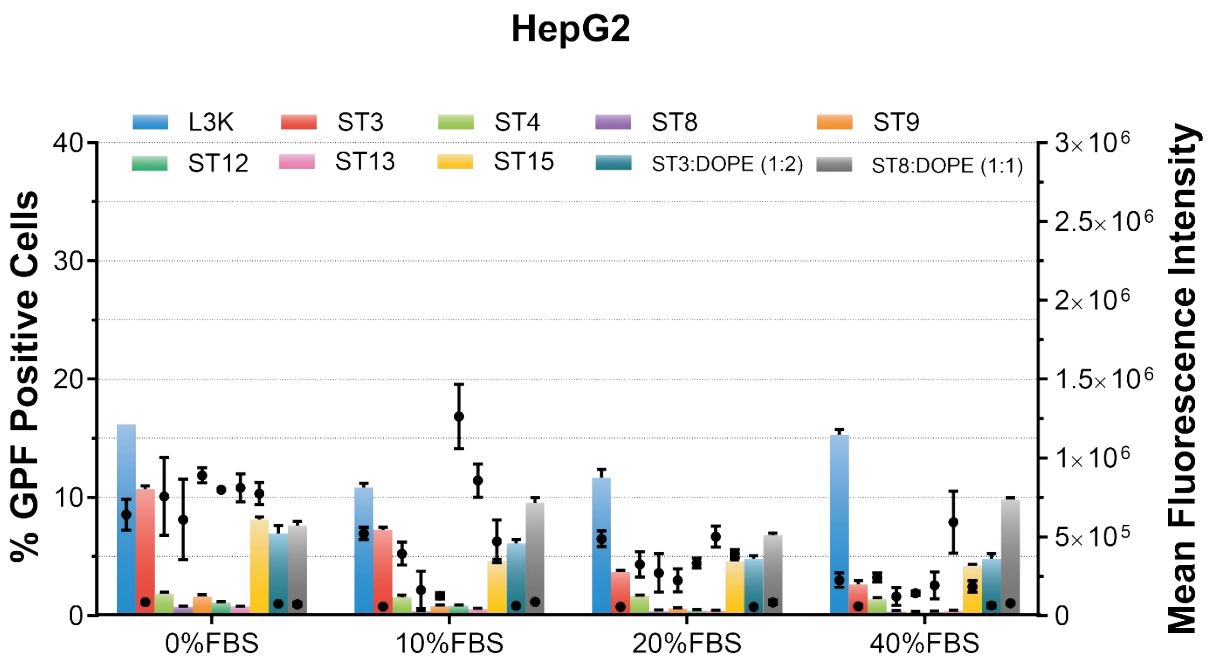


Fig. S10 The percentage of GFP positive cells (bar graph) and mean fluorescence intensity (■) of HepG2 cells transfected with lipoplexes encapsulating pEGFP-C2 in the absence and presence of 10–40% serum. The expression of green fluorescent protein was measured by flow cytometer. Each value represents the mean ± S.D. of three measurements.

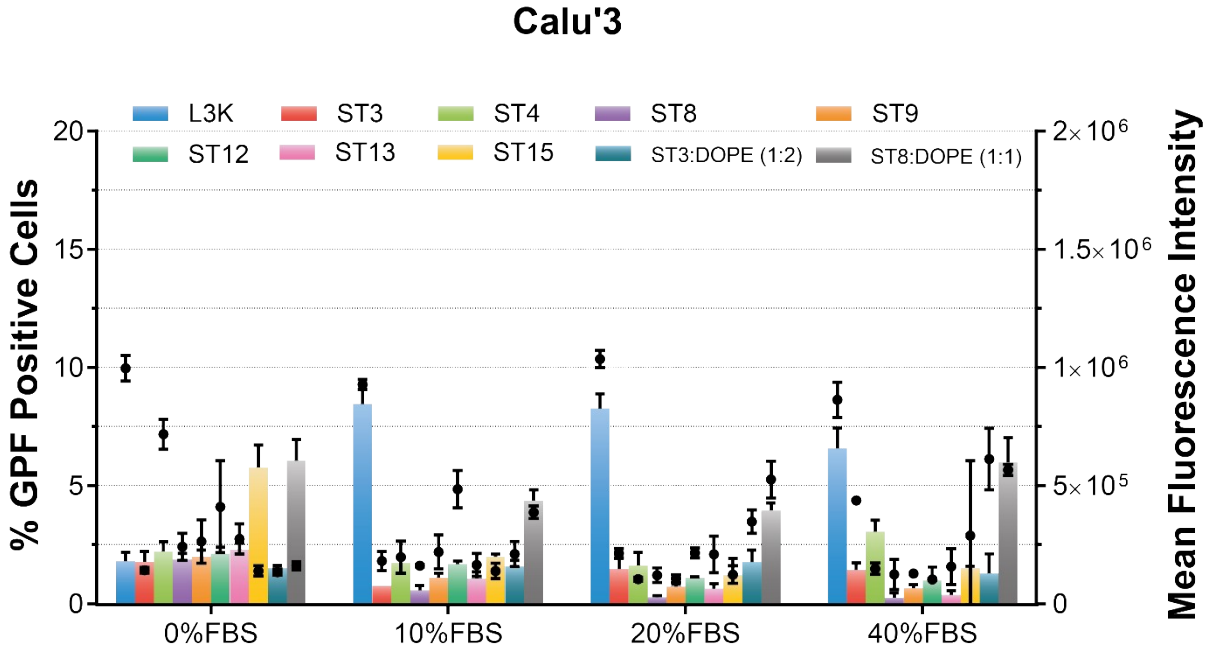


Fig. S11 The percentage of GFP positive cells (bar graph) and mean fluorescence intensity (■) of Calu'3 cells transfected with lipoplexes encapsulating pEGFP-C2 in the absence and presence of 10–40% serum. The expression of green fluorescent protein was measured by flow cytometer. Each value represents the mean ± S.D. of three measurements.

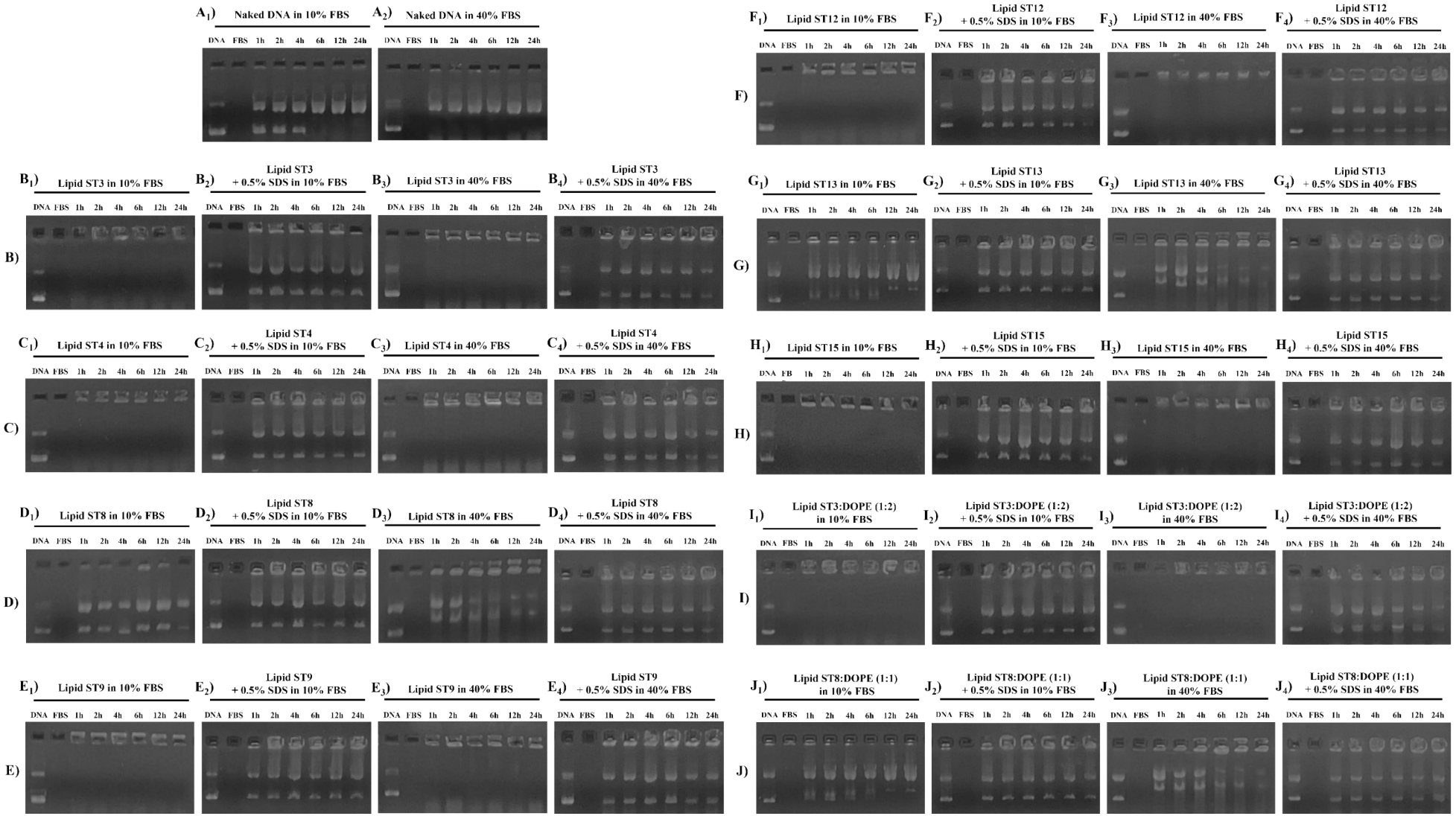


Fig. S12 Serum stability of plasmid DNA in liposome formulations under 10% and 40% serum concentrations at various times. Stability was analyzed by 1% agarose gel electrophoresis, 100 V for 45 min.

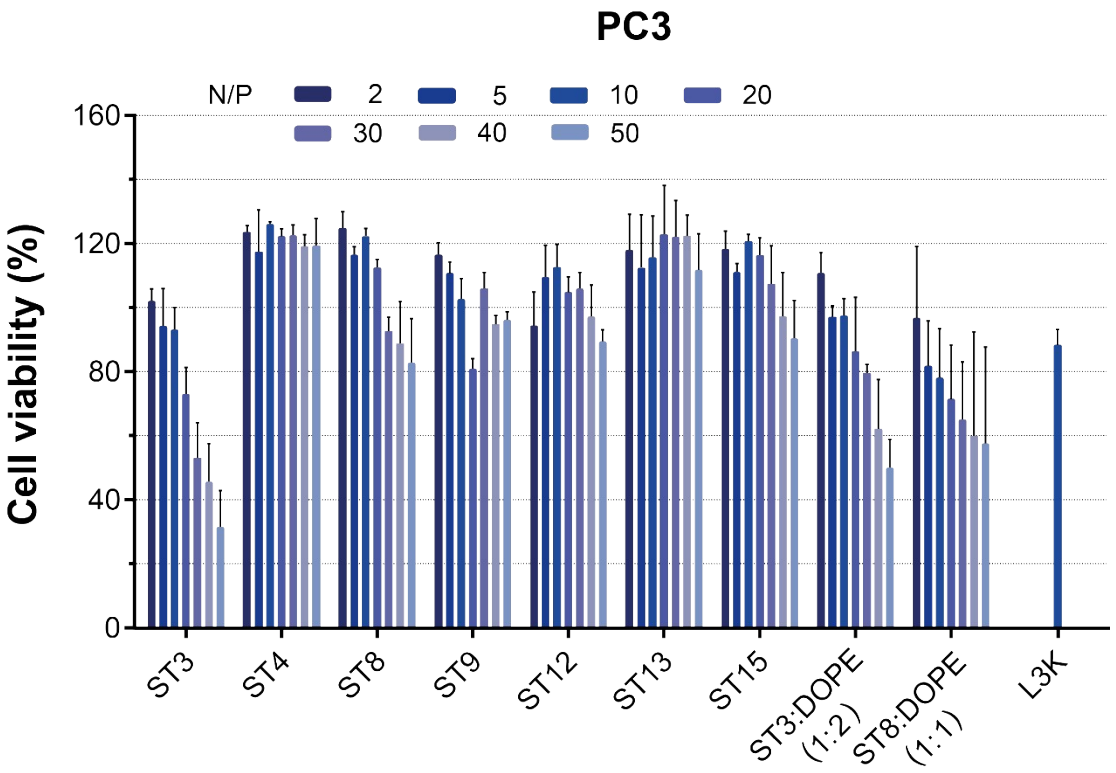


Fig. S13 Cytotoxicity of T-shape cationic liposome/pEGFP-C2 complexes at different N/P ratios in PC3 cells. The cytotoxicity was determined by MTT assay after 48 h. Each value represents the mean ± S.D. of three measurements.

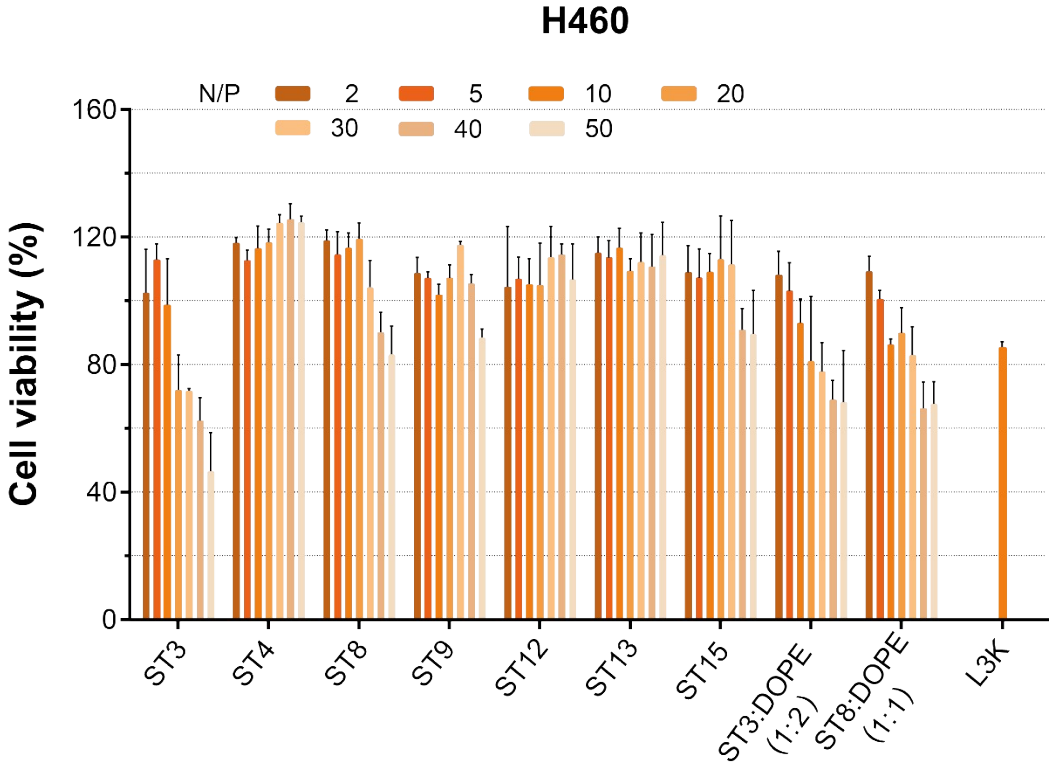


Fig. S14 Cytotoxicity of T-shape cationic liposome/pEGFP-C2 complexes at different N/P ratios in H460 cells. The cytotoxicity was determined by MTT assay after 48 h. Each value represents the mean ± S.D. of three measurements.

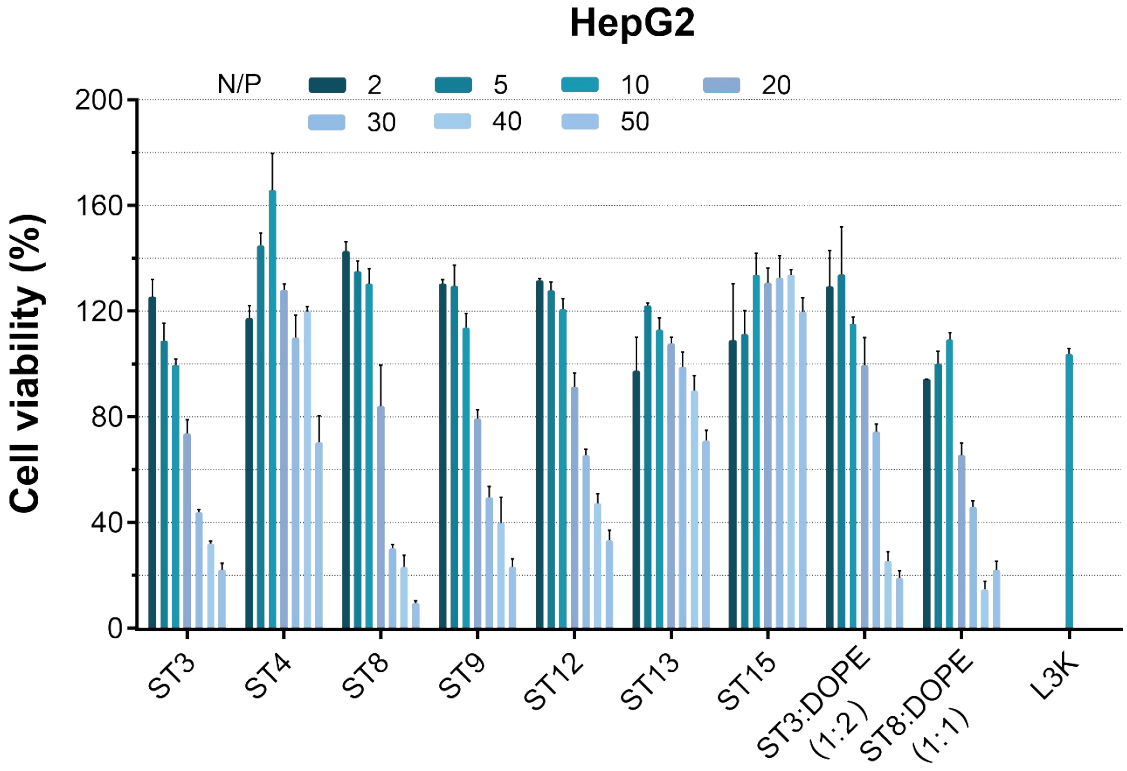


Fig. S15 Cytotoxicity of T-shape cationic liposome/pEGFP-C2 complexes at different N/P ratios in HepG2 cells. The cytotoxicity was determined by MTT assay after 48 h. Each value represents the mean ± S.D. of three measurements.

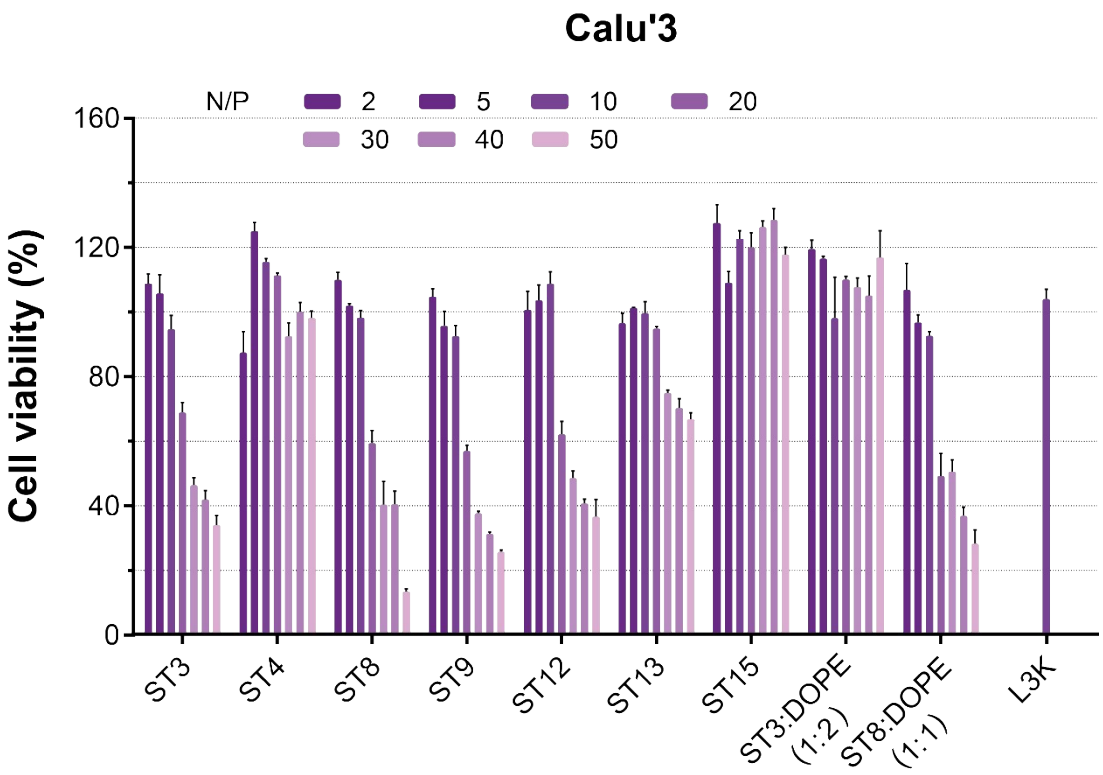


Fig. S16 Cytotoxicity of T-shape cationic liposome/pEGFP-C2 complexes at different N/P ratios in Calu′3 cells. The cytotoxicity was determined by MTT assay after 48 h. Each value represents the mean ± S.D. of three measurements.

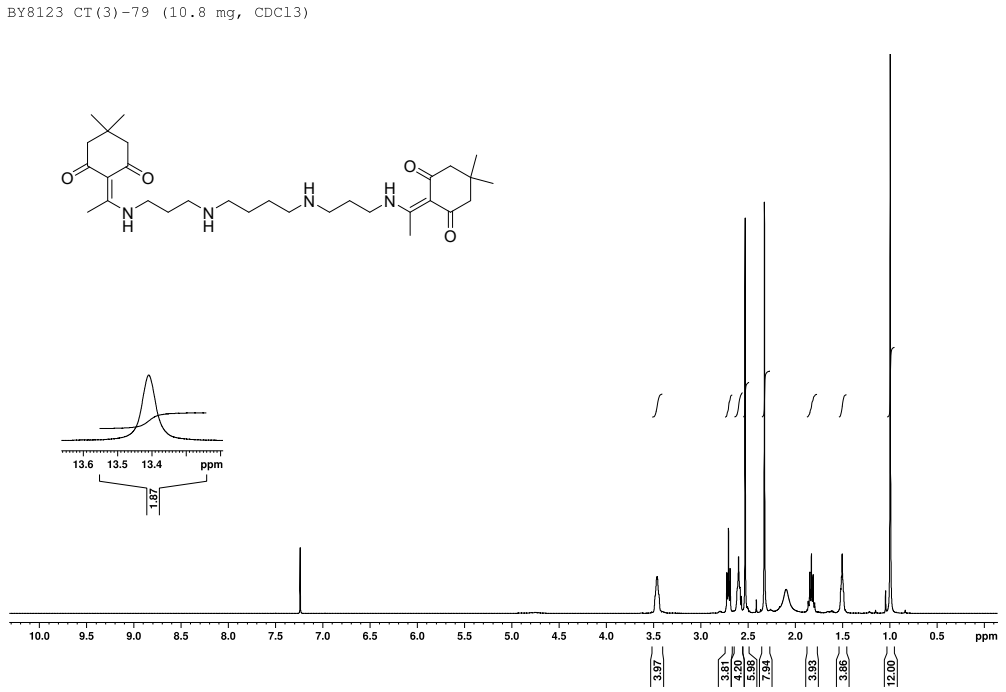


Fig. S17 ^{1}H NMR Spectrum (400 MHz, $CDCl_3$) of compound **8**

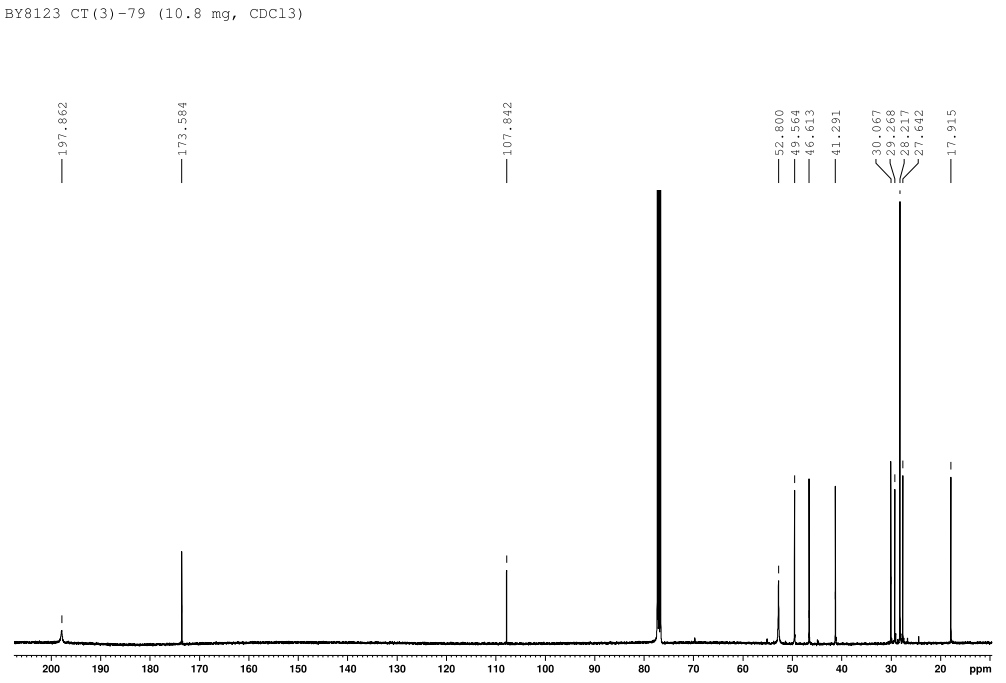


Fig. S18 ^{13}C NMR Spectrum (100 MHz, $CDCl_3$) of compound **8**

Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\Data\Boon-ek\ESI\BY-HRMS 1328 (pos).d	Acquisition Date	8/5/2020 1:30:33 PM
Method	tune_low_pos1-m.m	Operator	RU
Sample Name	CT(3)-79	Instrument	micrOTOF 8213750.10411
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	2.0 Bar
Focus	Not active			Set Dry Heater	180 °C
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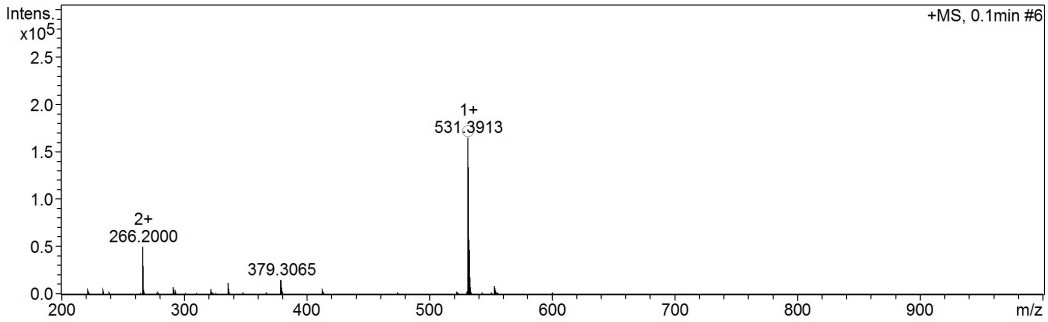
Scan End	2000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



Meas. m/z	#	Ion Formula	m/z	|err| [mDa]	err [ppm]	Mean err [ppm]	rdb	N-Rule	e⁻ Conf	mSigma
531.391333	1	C30H51N4O4	531.390483	0.9	1.6	-1.5	7.5	ok	even	4.3

Fig. S19 HRMS Spectrum of compound **8**

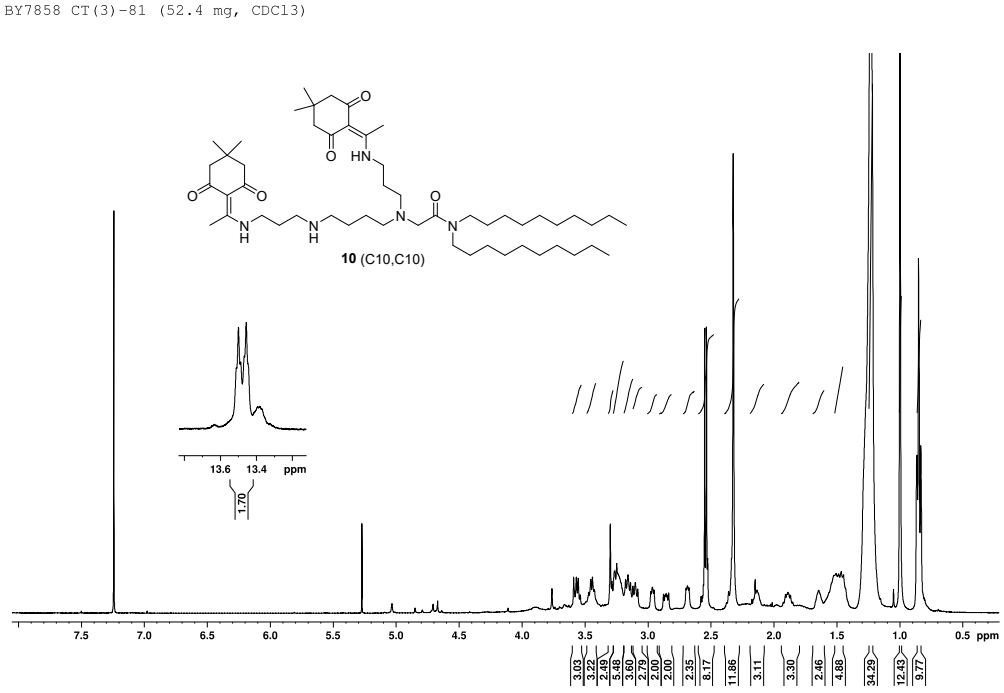


Fig. S20 ^{1}H NMR Spectrum (400 MHz, $CDCl_3$) of compound **10 (C10,C10)**

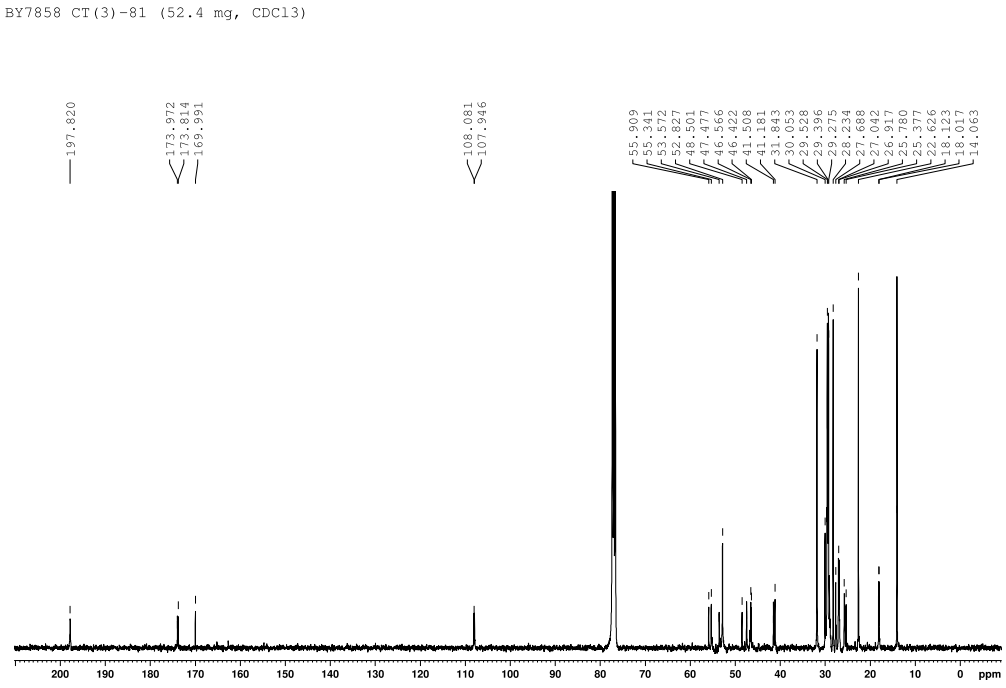


Fig. S21 ^{13}C NMR Spectrum (100 MHz, $CDCl_3$) of compound **10 (C10,C10)**

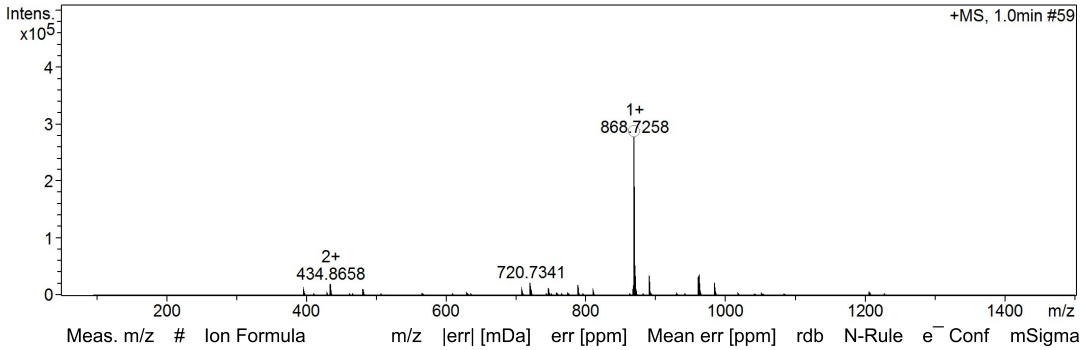
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\Data\Boon-ek\ESI\BY-HRMS 1501 (pos).d	Acquisition Date	12/15/2020 3:33:08 PM
Method	tune_wide(pos).m	Operator	RU
Sample Name	CT(3)-81	Instrument	micrOTOF 8213750.10411
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.3 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	100 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	4000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



Meas. m/z	#	Ion Formula	m/z	|err| [mDa]	err [ppm]	Mean err [ppm]	rdb	N-Rule	e⁻ Conf	mSigma
868.725846	1	C52H94N5O5	868.724948	0.9	-1.0	-0.8	8.5	ok	even	8.8

Fig. S22 HRMS Spectrum of compound **10 (C10,C10)**

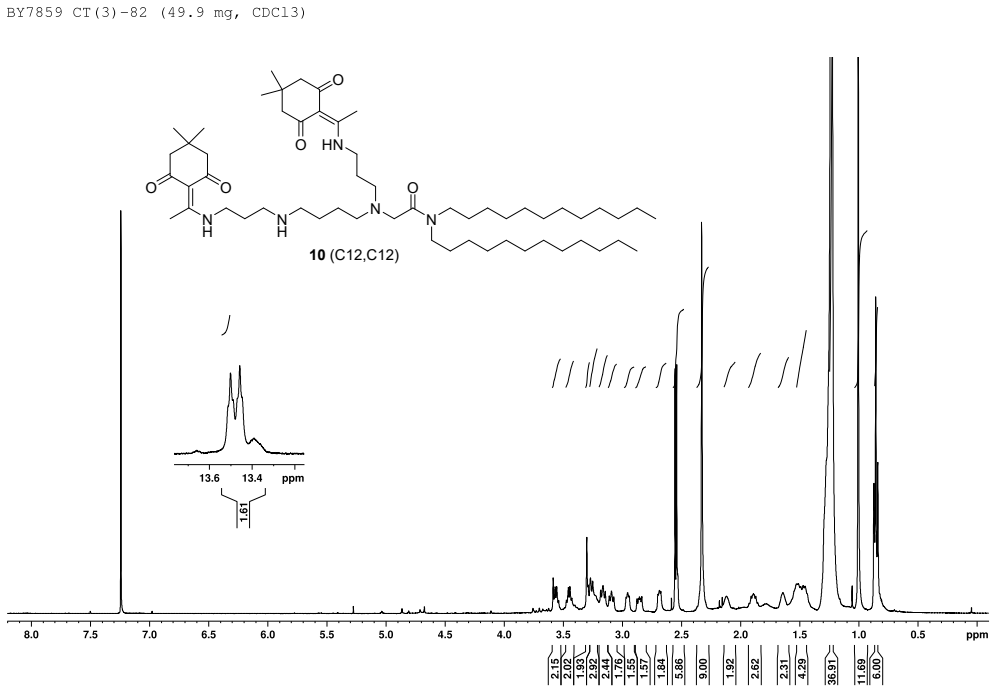


Fig. S23 ^{1}H NMR Spectrum (400 MHz, $CDCl_3$) of compound **10 (C12,C12)**

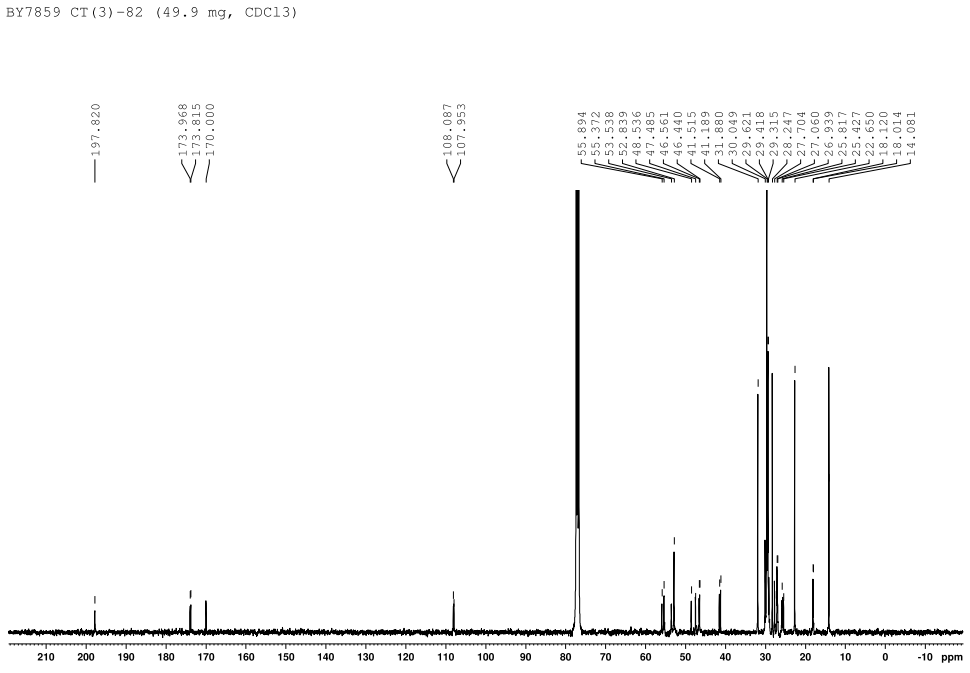


Fig. S24 ^{13}C NMR Spectrum (100 MHz, $CDCl_3$) of compound **10 (C12,C12)**

Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\Data\Boon-ek\ESI\BY-HRMS 1502 (pos).d	Acquisition Date	12/15/2020 3:35:29 PM
Method	tune_wide(pos).m	Operator	RU
Sample Name	CT(3)-82	Instrument	micrOTOF 8213750.10411
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.3 Bar
Focus	Not active			Set Dry Heater	180 °C
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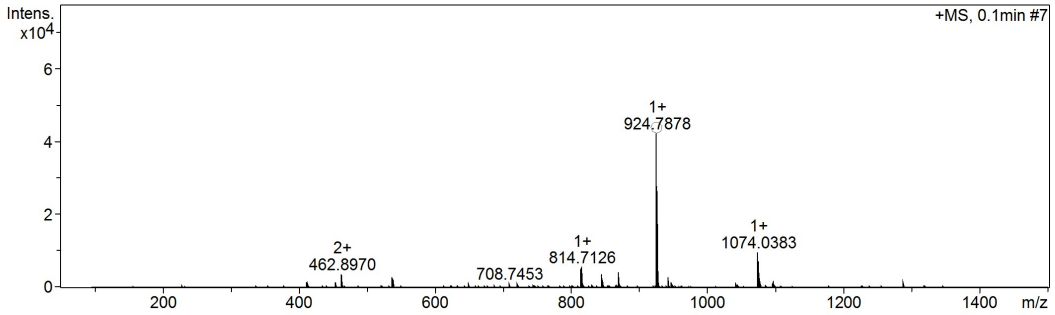
Scan End	4000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



Meas. m/z	#	Ion Formula	m/z	|err| [mDa]	err [ppm]	Mean err [ppm]	rdb	N-Rule	e⁻ Conf	mSigma
924.787752	1	C56H102N5O5	924.787548	0.2	0.2	-0.4	8.5	ok	even	5.1

Fig. S25 HRMS Spectrum of compound **10 (C12,C12)**

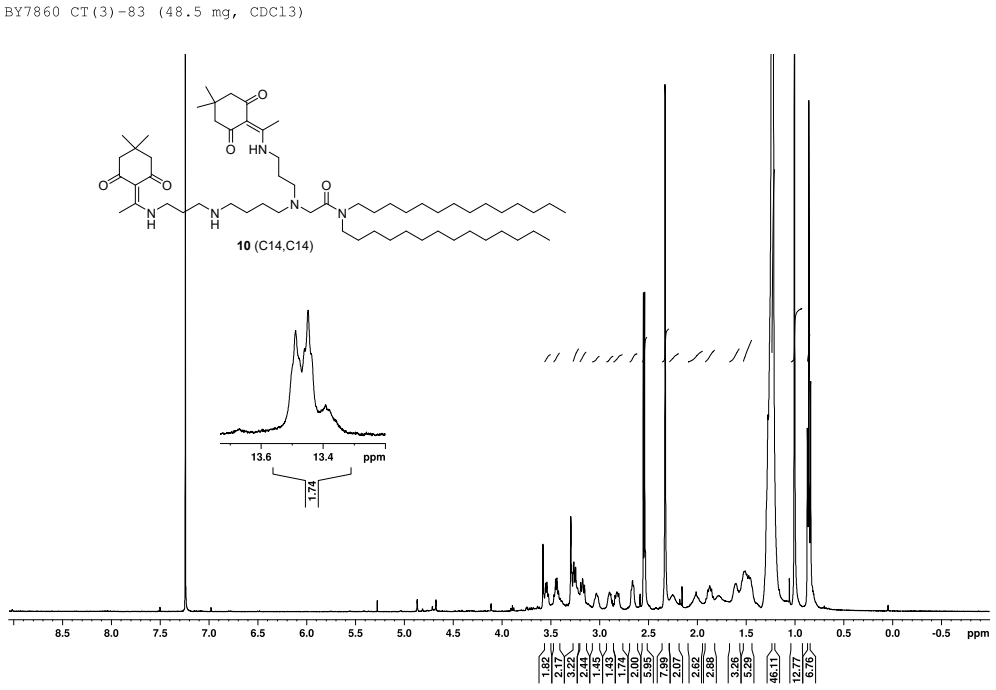


Fig. S26 ^{1}H NMR Spectrum (400 MHz, $CDCl_3$) of compound **10 (C14,C14)**

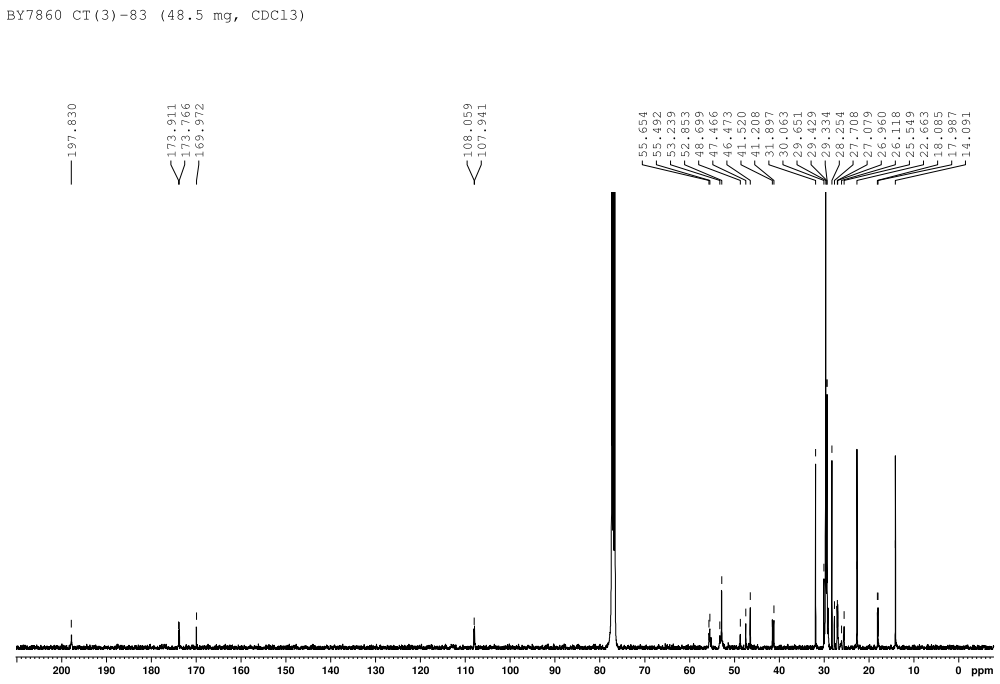


Fig. S27 ^{13}C NMR Spectrum (100 MHz, $CDCl_3$) of compound **10 (C14,C14)**

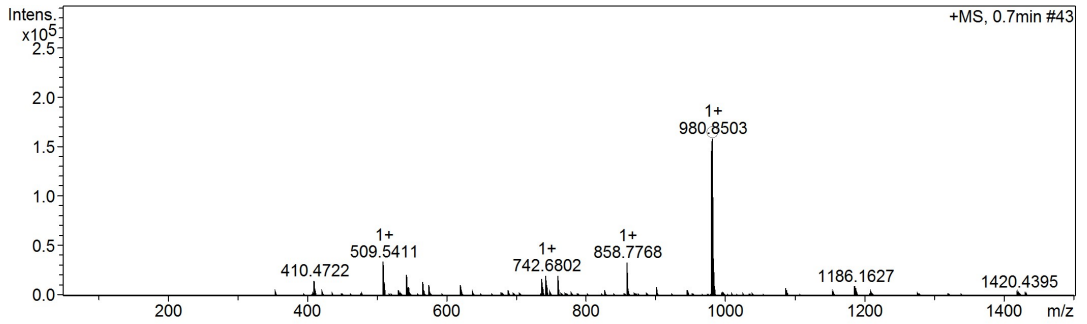
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\Data\Boon-ek\ESI\BY-HRMS 1521 (pos).d	Acquisition Date	12/22/2020 3:07:41 PM
Method	tune_wide-RU.m	Operator	RU
Sample Name	CT(3)-83	Instrument	micrOTOF 8213750.10411
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.3 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	4000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



Meas. m/z	#	Ion Formula	m/z	|err| [mDa]	err [ppm]	Mean err [ppm]	rdb	N-Rule	e⁻ Conf	mSigma
980.850258	1	C60H110N5O5	980.850148	0.1	-0.1	-0.2	8.5	ok	even	11.9

Fig. S28 HRMS Spectrum of compound **10 (C14,C14)**

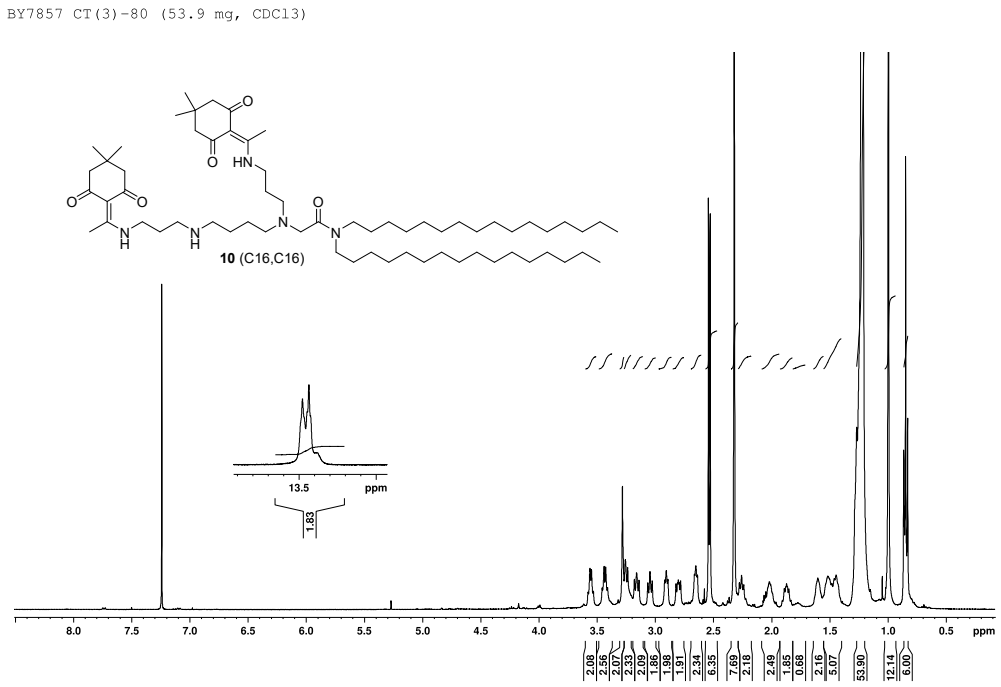


Fig. S29 ^{1}H NMR Spectrum (400 MHz, $CDCl_3$) of compound **10 (C16,C16)**

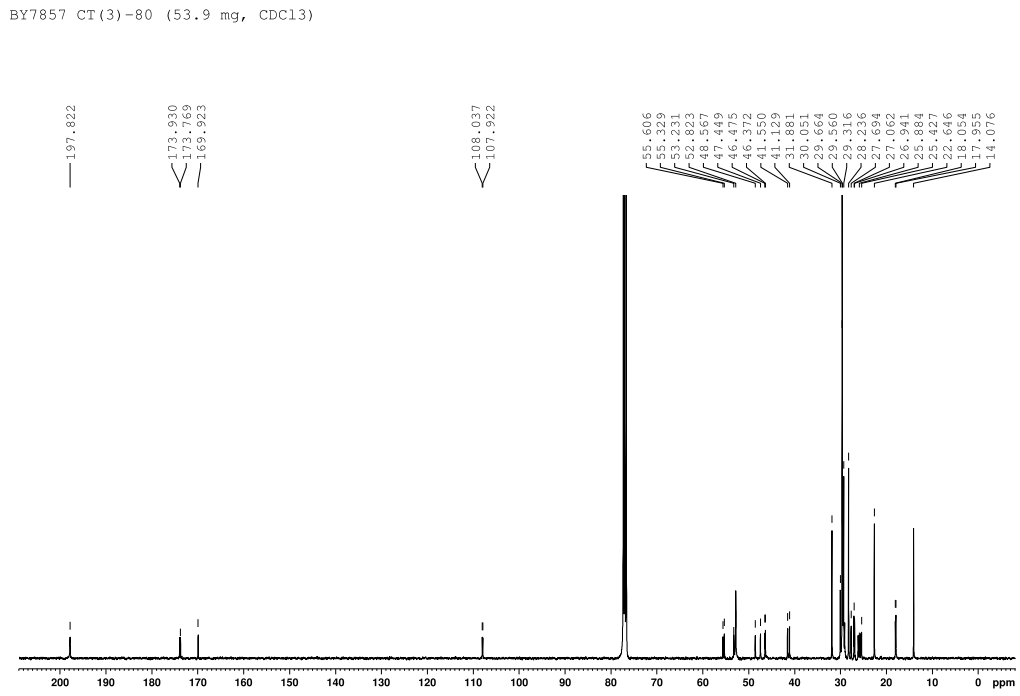


Fig. S30 ^{13}C NMR Spectrum (100 MHz, $CDCl_3$) of compound **10 (C16,C16)**

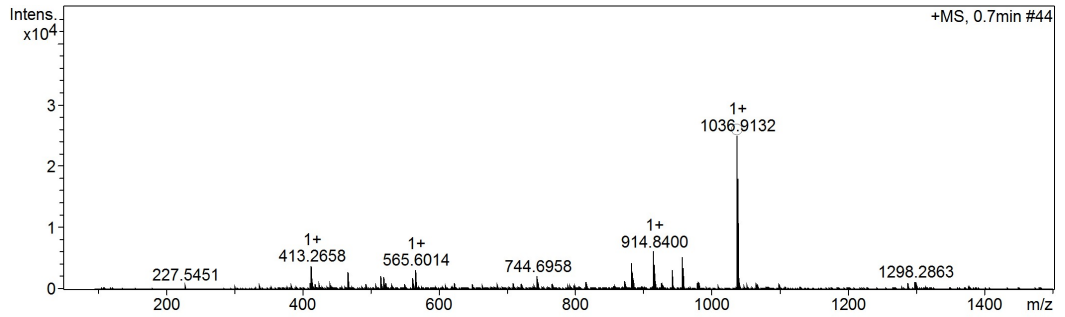
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\Data\Boon-ek\ESI\BY-HRMS 1506 (pos).d	Acquisition Date	12/15/2020 3:49:35 PM
Method	tune_wide(pos).m	Operator	RU
Sample Name	CT(3)-80	Instrument	micrOTOF 8213750.10411
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.3 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	100 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	4000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



Meas. m/z	#	Ion Formula	m/z	|err| [mDa]	err [ppm]	Mean err [ppm]	rdb	N-Rule	e⁻ Conf	mSigma
1036.913249	1	C64H118N5O5	1036.912748	0.5	-0.5	-0.4	8.5	ok	even	4.3

Fig. S31 HRMS Spectrum of compound **10 (C16,C16)**

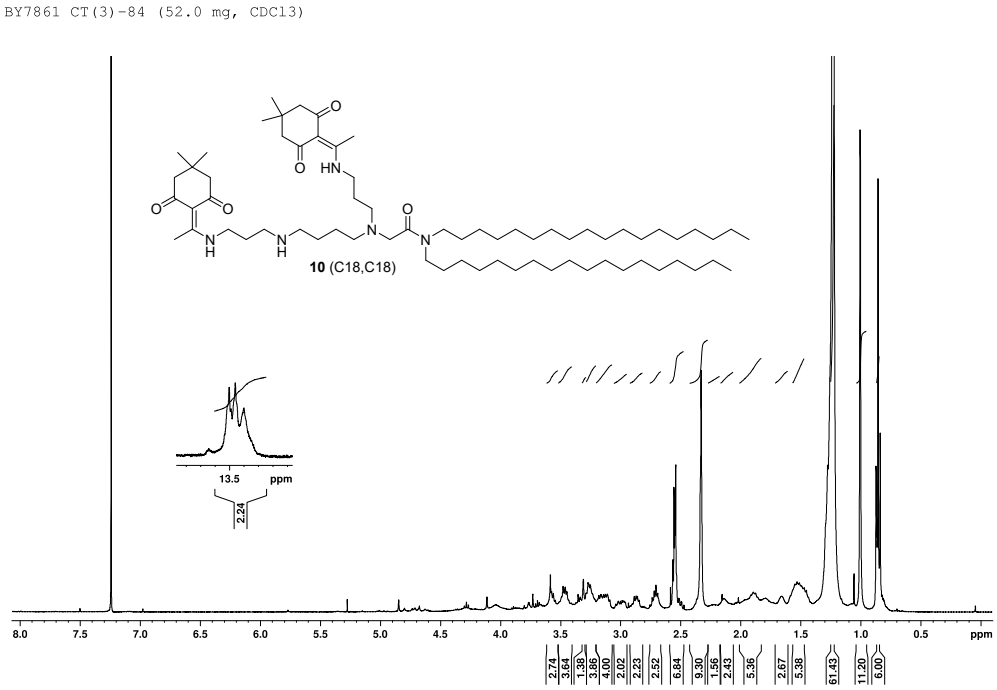


Fig. S32 ^{1}H NMR Spectrum (400 MHz, $CDCl_3$) of compound **10 (C18,C18)**

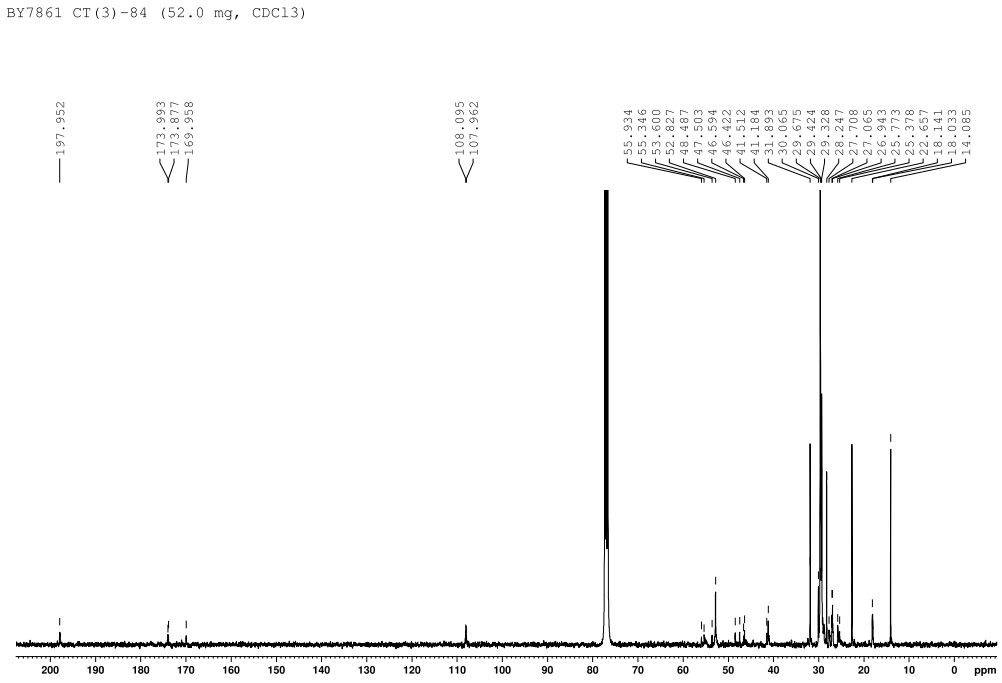


Fig. S33 ^{13}C NMR Spectrum (100 MHz, $CDCl_3$) of compound **10 (C18,C18)**

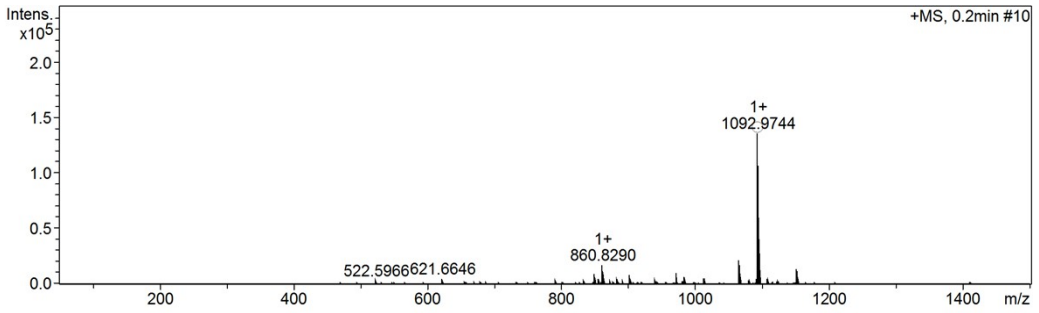
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\Data\Boon-ek\ESI\BY-HRMS 1522 (pos).d	Acquisition Date	12/22/2020 3:09:08 PM
Method	tune_wide-RU.m	Operator	RU
Sample Name	CT(3)-84	Instrument	micrOTOF 8213750.10411
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.3 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	4000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



Meas. m/z	#	Ion Formula	m/z	|err| [mDa]	err [ppm]	Mean err [ppm]	rdb	N-Rule	e⁻ Conf	mSigma
1092.974388	1	C68H126N5O5	1092.975349	1.0	-0.9	1.3	8.5	ok	even	7.6

Fig. S34 HRMS Spectrum of compound **10 (C18,C18)**

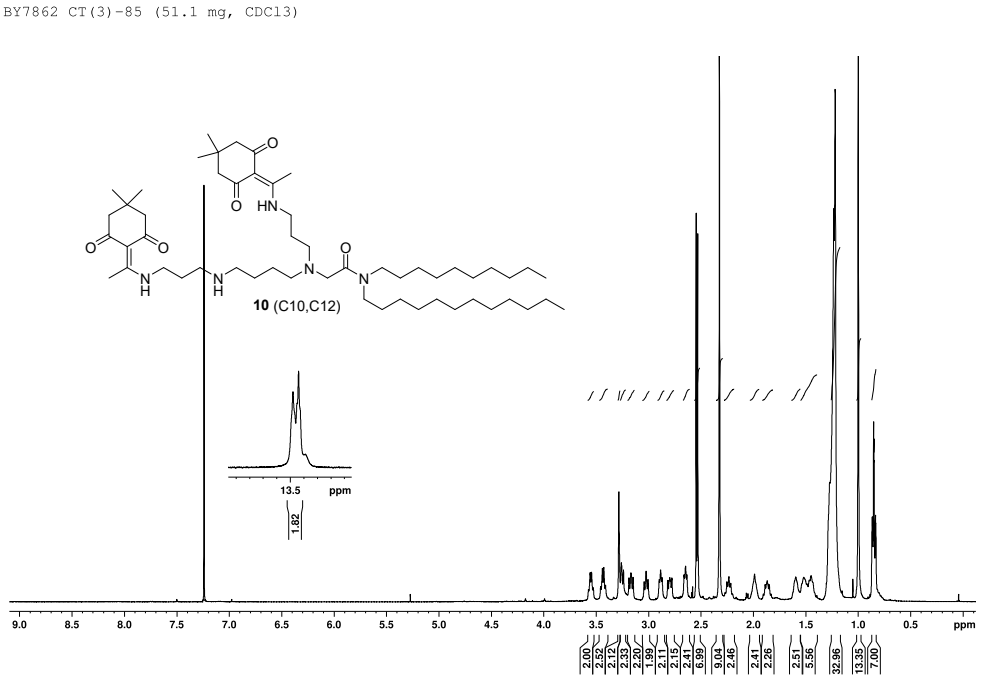


Fig. S35 ^{1}H NMR Spectrum (400 MHz, $CDCl_3$) of compound **10 (C10,C12)**

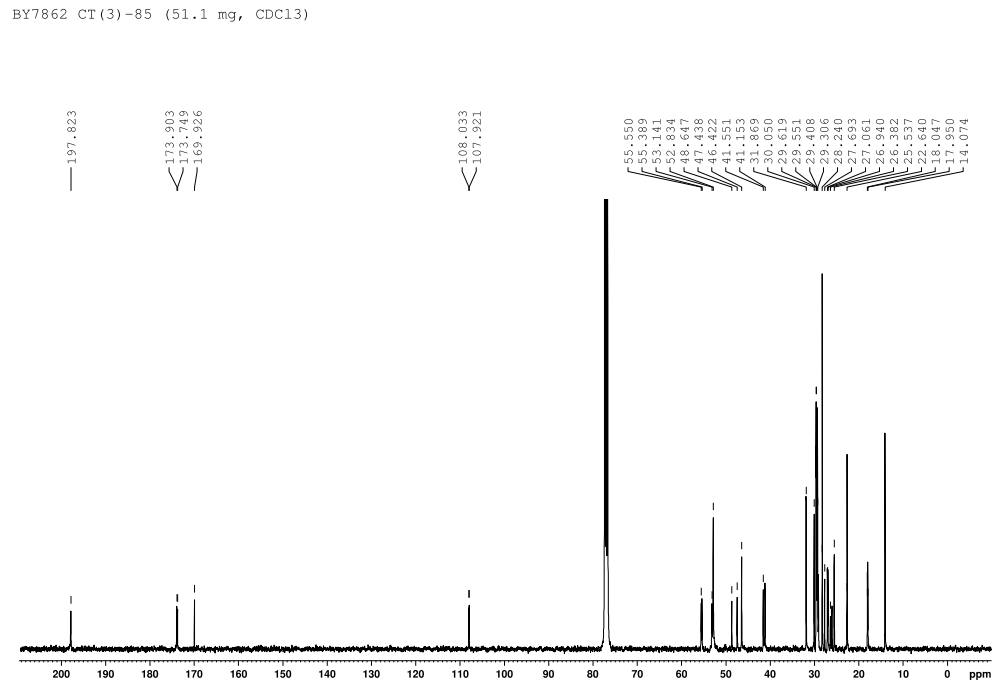


Fig. S36 ^{13}C NMR Spectrum (100 MHz, $CDCl_3$) of compound **10 (C10,C12)**

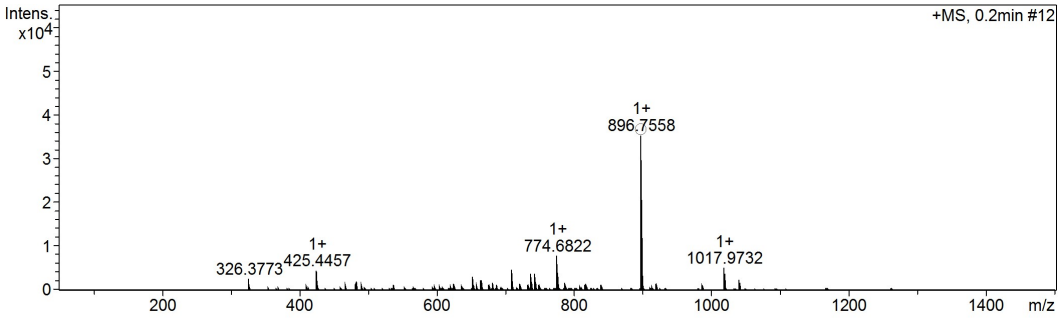
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\Data\Boon-ek\ESI\BY-HRMS 1523 (pos).d	Acquisition Date	12/22/2020 3:10:35 PM
Method	tune_wide-RU.m	Operator	RU
Sample Name	CT(3)-85	Instrument	micrOTOF 8213750.10411
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.3 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	4000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



Meas. m/z	#	Ion Formula	m/z	|err| [mDa]	err [ppm]	Mean err [ppm]	rdb	N-Rule	e⁻ Conf	mSigma
896.755827	1	C54H98N5O5	896.756248	0.4	-0.5	0.4	8.5	ok	even	3.6

Fig. S37 HRMS Spectrum of compound **10 (C10,C12)**

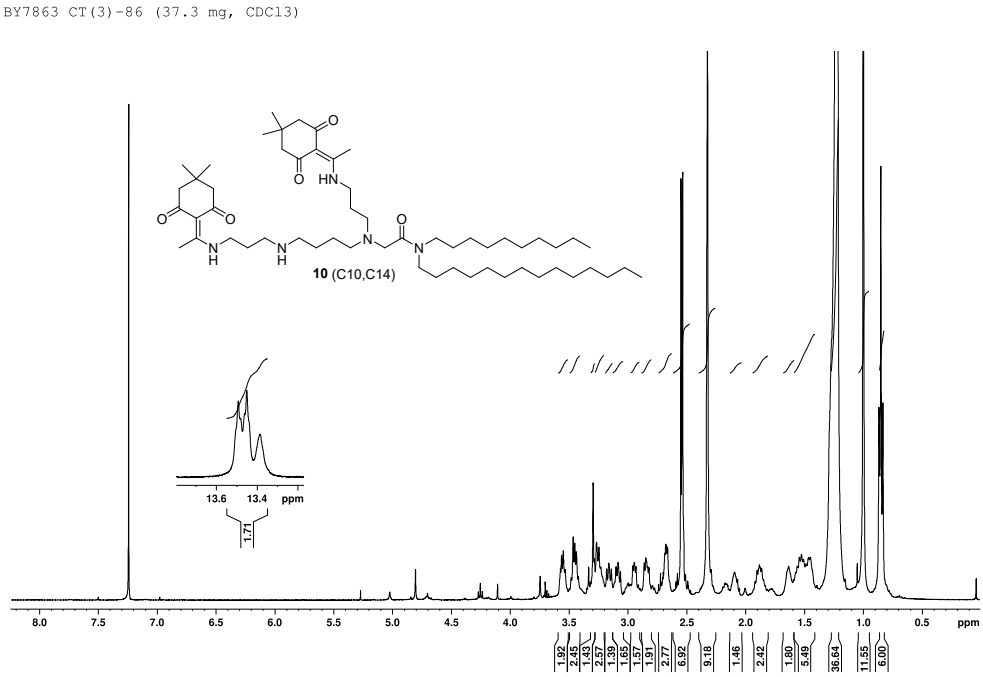


Fig. S38 ^{1}H NMR Spectrum (400 MHz, $CDCl_3$) of compound **10 (C10,C14)**

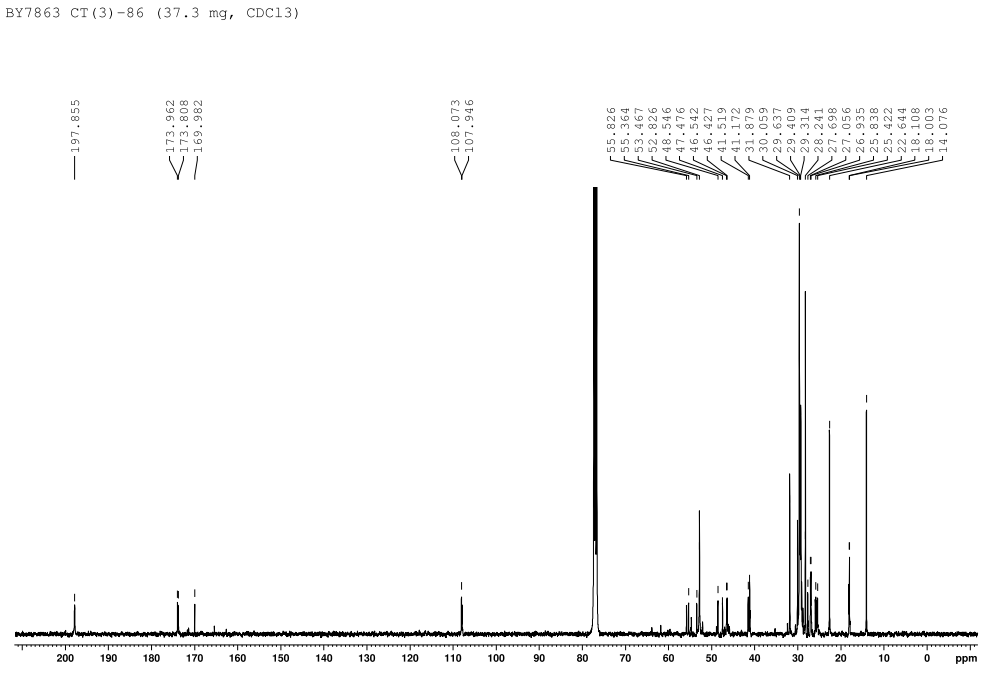


Fig. S39 ^{13}C NMR Spectrum (100 MHz, $CDCl_3$) of compound **10 (C10,C14)**

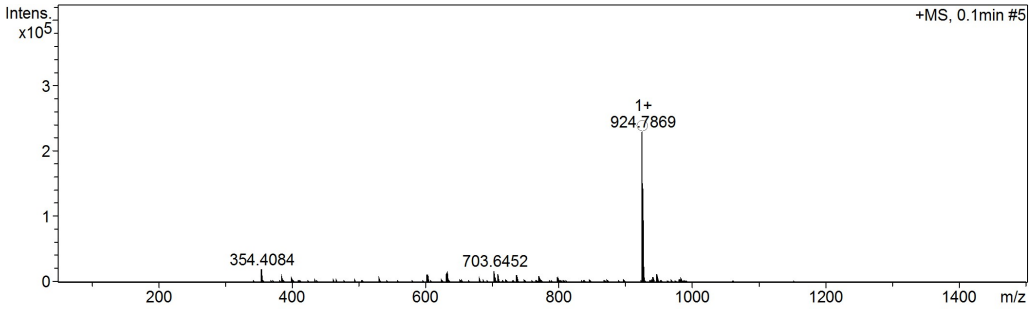
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\Data\Boon-ek\ESI\BY-HRMS 1524 (pos).d	Acquisition Date	12/22/2020 3:11:47 PM
Method	tune_wide-RU.m	Operator	RU
Sample Name	CT(3)-86	Instrument	micrOTOF 8213750.10411
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.3 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	4000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



Meas. m/z	#	Ion Formula	m/z	|err| [mDa]	err [ppm]	Mean err [ppm]	rdb	N-Rule	e⁻ Conf	mSigma
924.786901	1	C56H102N5O5	924.787548	0.6	0.7	1.0	8.5	ok	even	8.3

Fig. S40 HRMS Spectrum of compound **10 (C10,C14)**

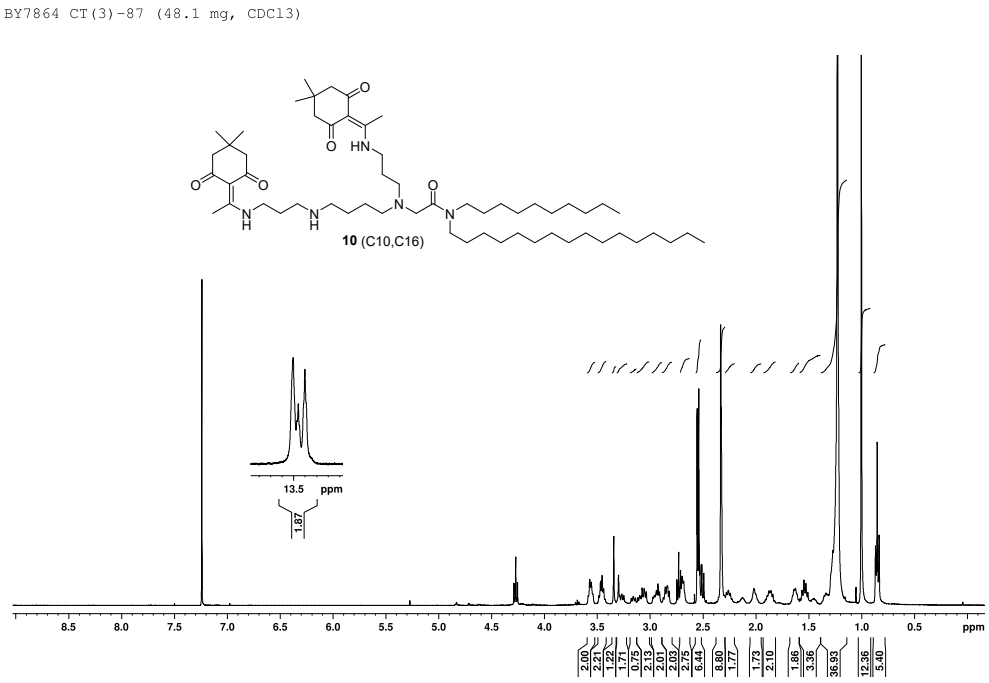


Fig. S41 ^{1}H NMR Spectrum (400 MHz, $CDCl_3$) of compound **10 (C10,C16)**

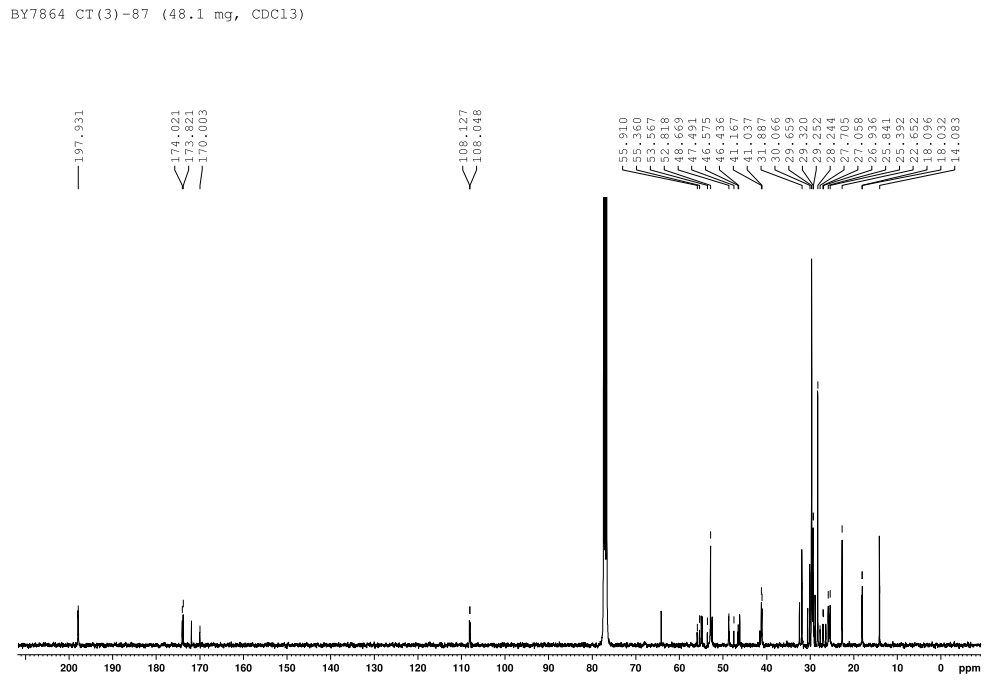


Fig. S42 ^{13}C NMR Spectrum (100 MHz, $CDCl_3$) of compound **10 (C10,C16)**

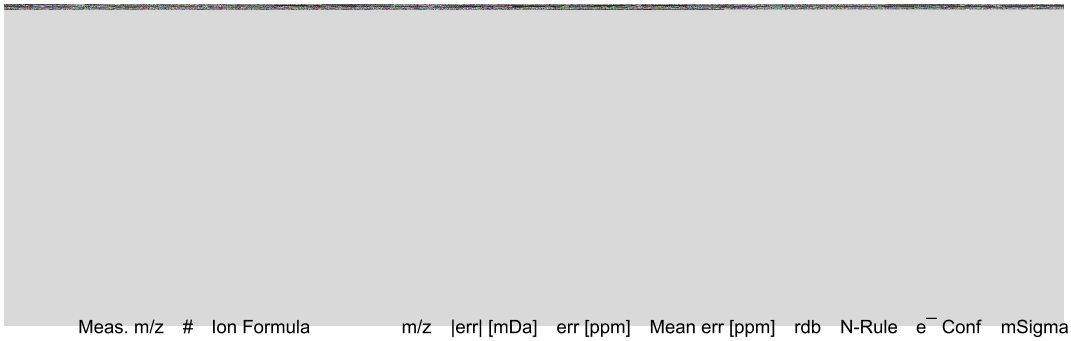
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\Data\Boon-ek\ESI\BY-HRMS 1524 (pos).d	Acquisition Date	12/22/2020 3:11:47 PM
Method	tune_wide-RU.m	Operator	RU
Sample Name	CT(3)-86	Instrument	micrOTOF 8213750.10411
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.3 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	4000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



Meas. m/z	#	Ion Formula	m/z	|err| [mDa]	err [ppm]	Mean err [ppm]	rdb	N-Rule	e⁻ Conf	mSigma
924.786901	1	C56H102N5O5	924.787548	0.6	0.7	1.0	8.5	ok	even	8.3

Fig. S43 HRMS Spectrum of compound **10 (C10,C16)**

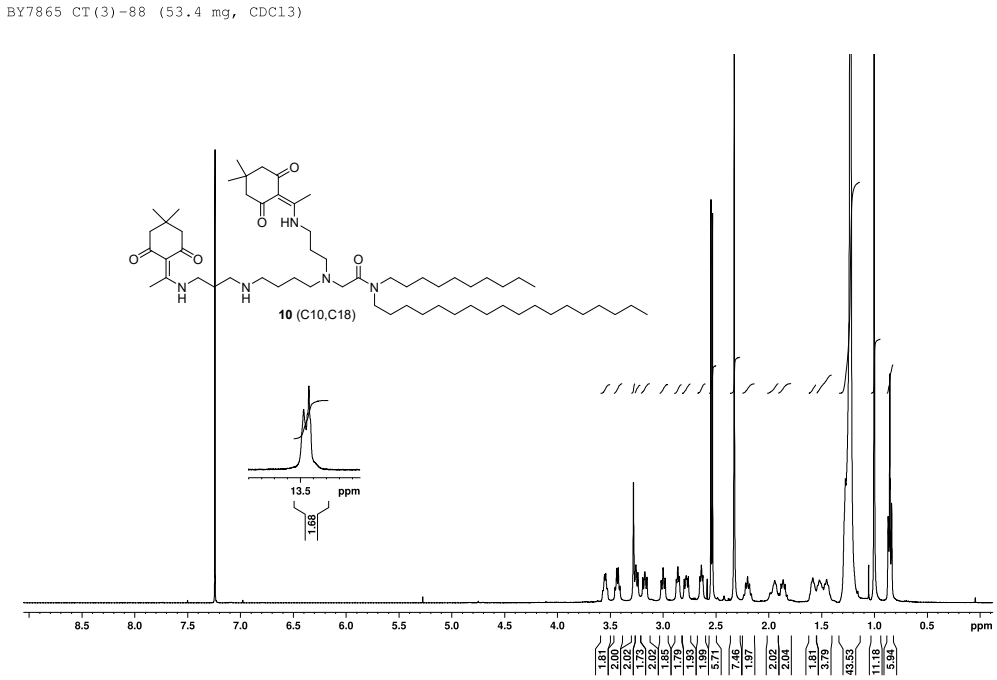


Fig. S44 ^{1}H NMR Spectrum (400 MHz, $CDCl_3$) of compound **10 (C10,C18)**

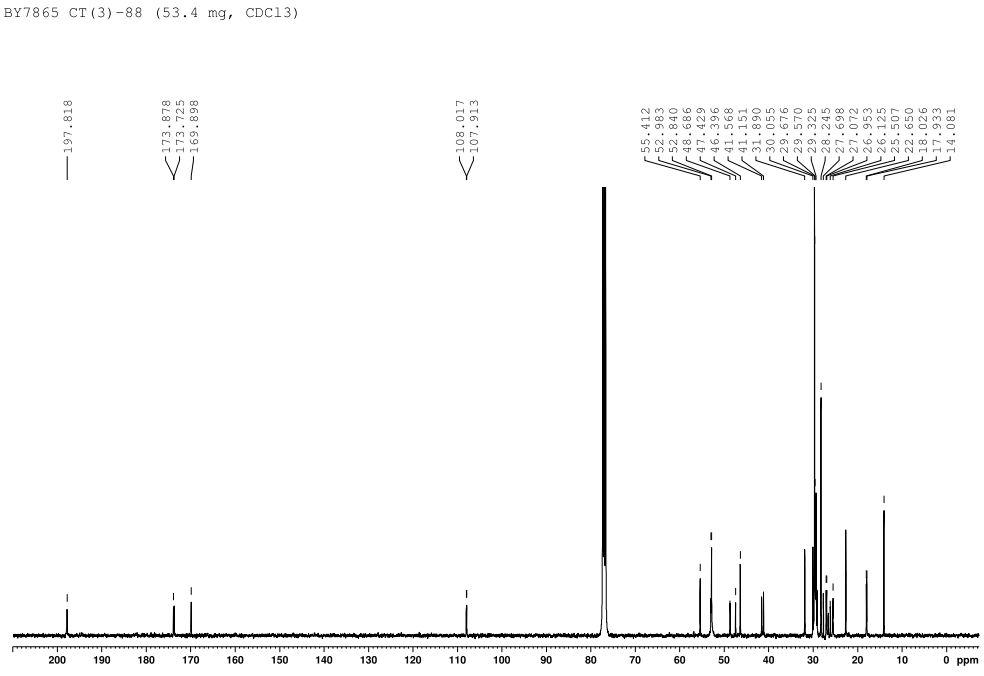


Fig. S45 ^{13}C NMR Spectrum (100 MHz, $CDCl_3$) of compound **10 (C10,C18)**

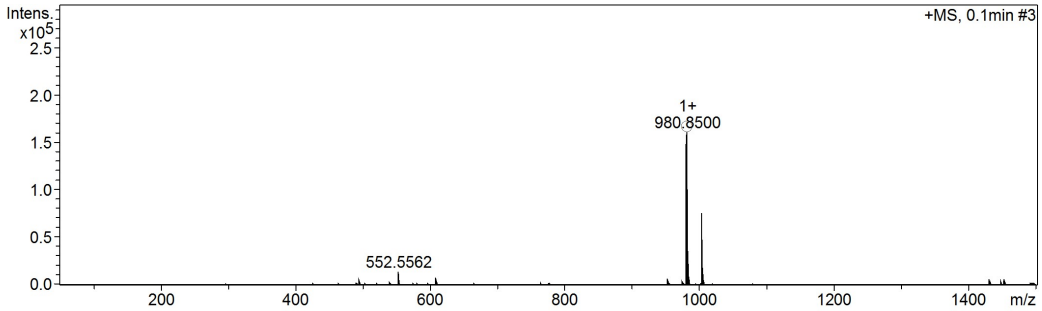
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\Data\Boon-ek\ESI\BY-HRMS 1566 (pos).d	Acquisition Date	12/27/2020 11:54:21 AM
Method	tune_wide_wow 2-7-62.m	Operator	RU
Sample Name	CT(3)-88	Instrument	micrOTOF 8213750.10411
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.5 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



Meas. m/z	#	Ion Formula	m/z	|err| [mDa]	err [ppm]	Mean err [ppm]	rdb	N-Rule	e⁻ Conf	mSigma
980.850009	1	C60H110N5O5	980.850148	0.1	-0.1	0.7	8.5	ok	even	15.1

Fig. S46 HRMS Spectrum of compound **10 (C10,C18)**

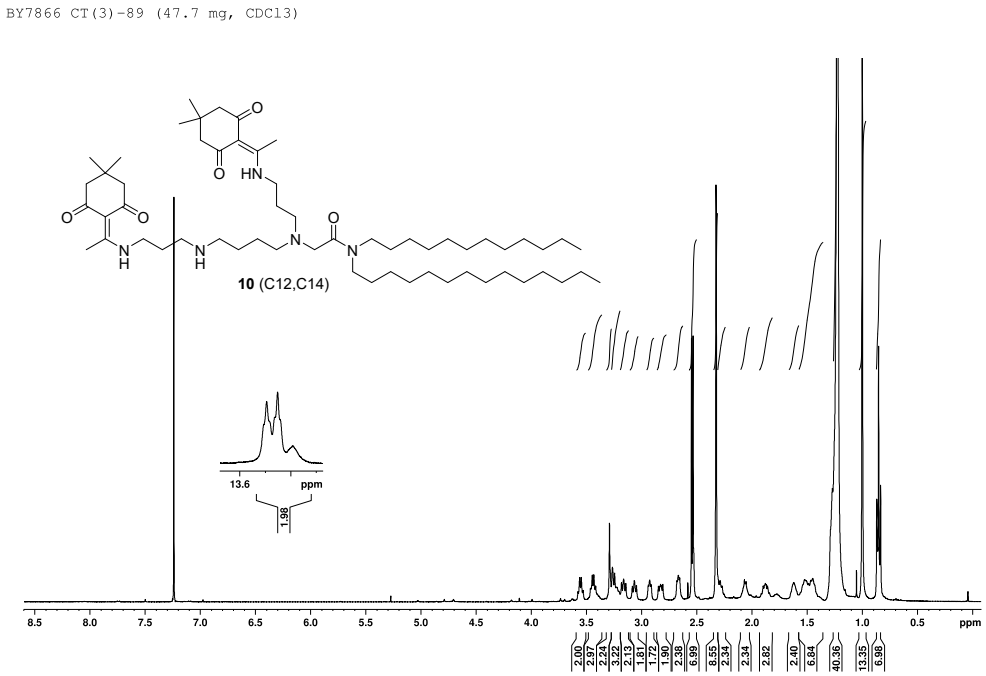


Fig. S47 ^{1}H NMR Spectrum (400 MHz, $CDCl_3$) of compound **10 (C12,C14)**

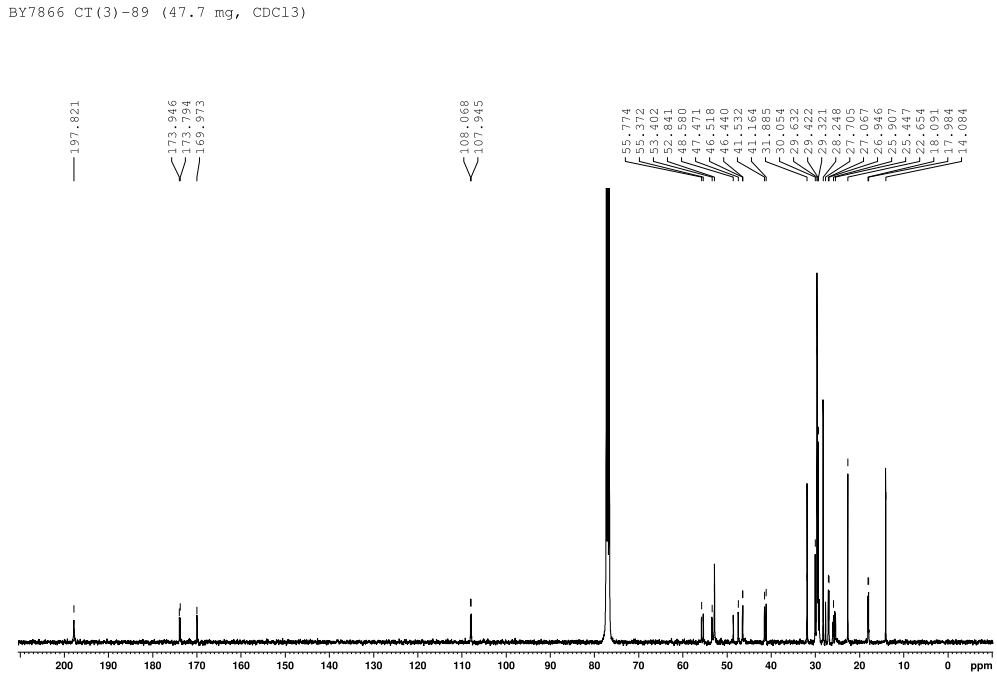


Fig. S48 ^{13}C NMR Spectrum (100 MHz, $CDCl_3$) of compound **10 (C12,C14)**

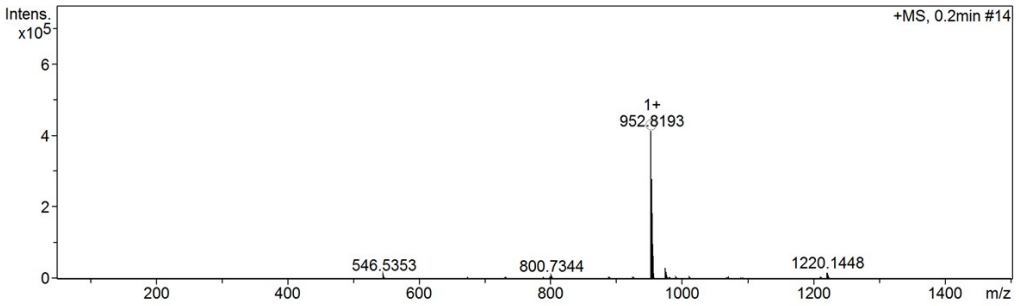
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\Data\Boon-ek\ESI\BY-HRMS 1527 (pos).d	Acquisition Date	12/22/2020 3:14:07 PM
Method	tune_wide-RU.m	Operator	RU
Sample Name	CT(3)-89	Instrument	micrOTOF 8213750.10411
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.3 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	4000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



Meas. m/z	#	Ion Formula	m/z	|err| [mDa]	err [ppm]	Mean err [ppm]	rdb	N-Rule	e⁻ Conf	mSigma
952.819313	1	C58H106N5O5	952.818848	0.5	-0.5	-0.3	8.5	ok	even	6.7

Fig. S49 HRMS Spectrum of compound **10 (C12,C14)**

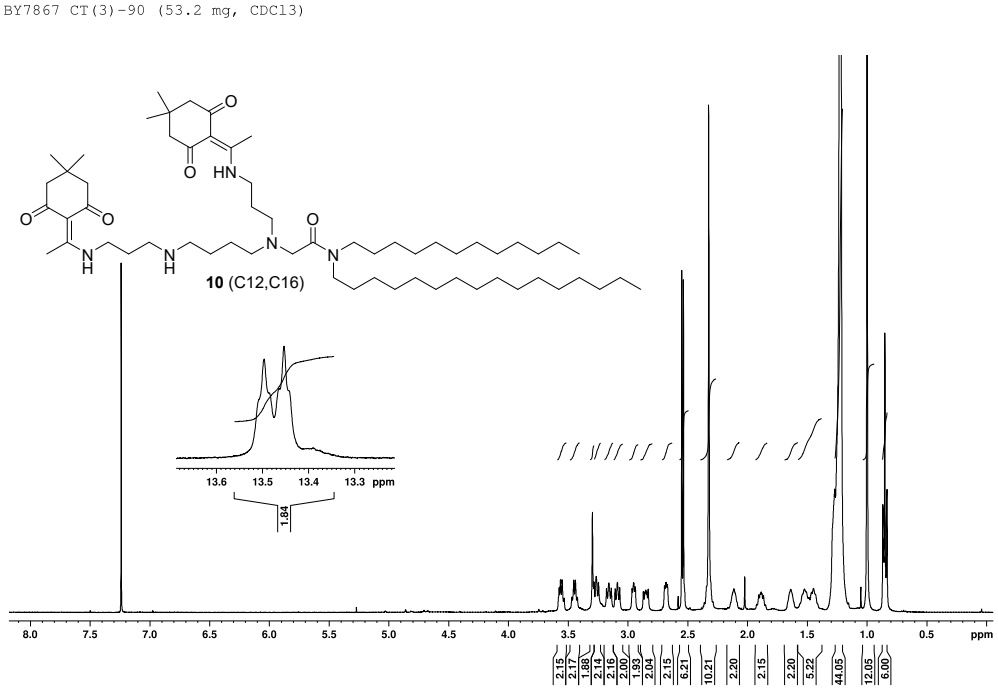


Fig. S50 ^{1}H NMR Spectrum (400 MHz, $CDCl_3$) of compound **10 (C12,C16)**

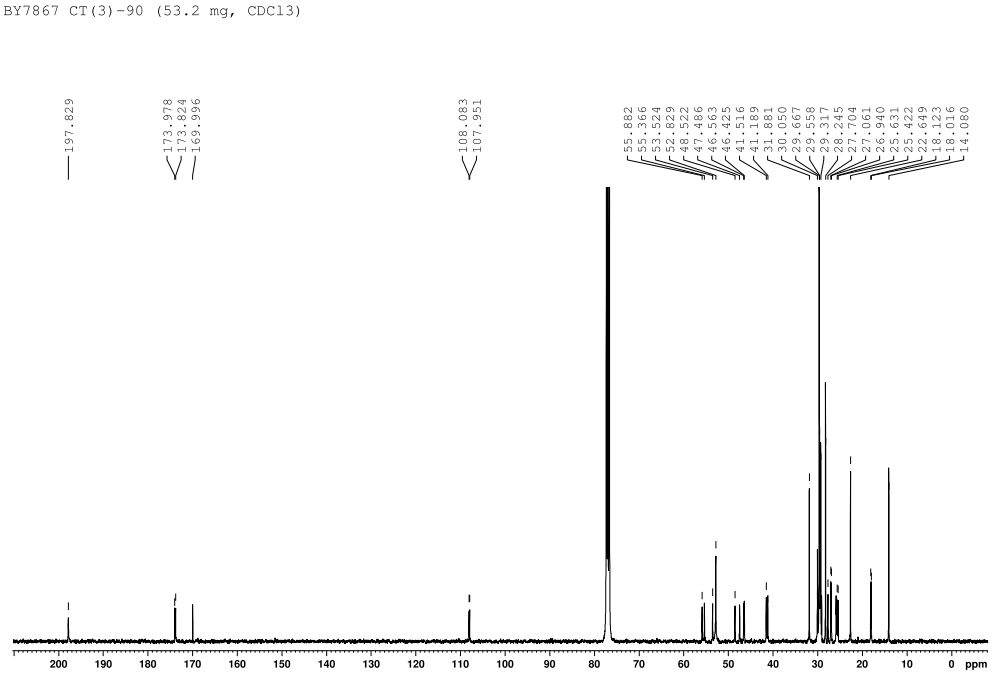


Fig. S51 ^{13}C NMR Spectrum (100 MHz, $CDCl_3$) of compound **10 (C12,C16)**

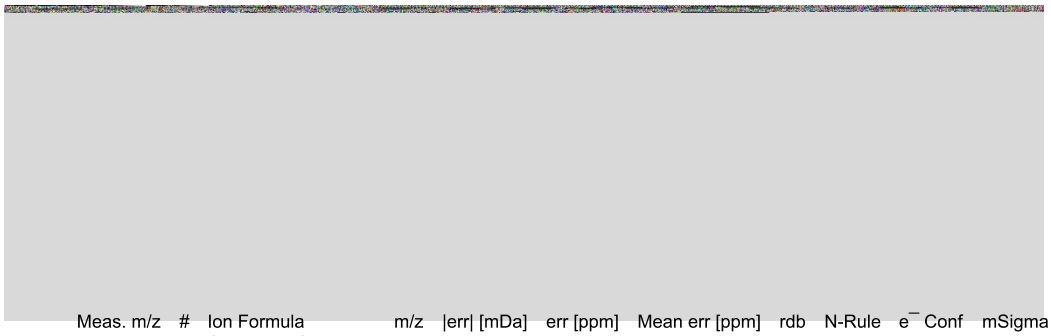
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\Data\Boon-ek\ESI\BY-HRMS 1527 (pos).d	Acquisition Date	12/22/2020 3:14:07 PM
Method	tune_wide-RU.m	Operator	RU
Sample Name	CT(3)-89	Instrument	micrOTOF 8213750.10411
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.3 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	4000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



Meas. m/z	#	Ion Formula	m/z	|err| [mDa]	err [ppm]	Mean err [ppm]	rdb	N-Rule	e⁻ Conf	mSigma
952.819313	1	C58H106N5O5	952.818848	0.5	-0.5	-0.3	8.5	ok	even	6.7

Bruker Compass DataAnalysis 4.1 printed: 12/26/2020 11:37:53 AM by: RU 1 of 1

Fig. S52 HRMS Spectrum of compound **10 (C12,C16)**

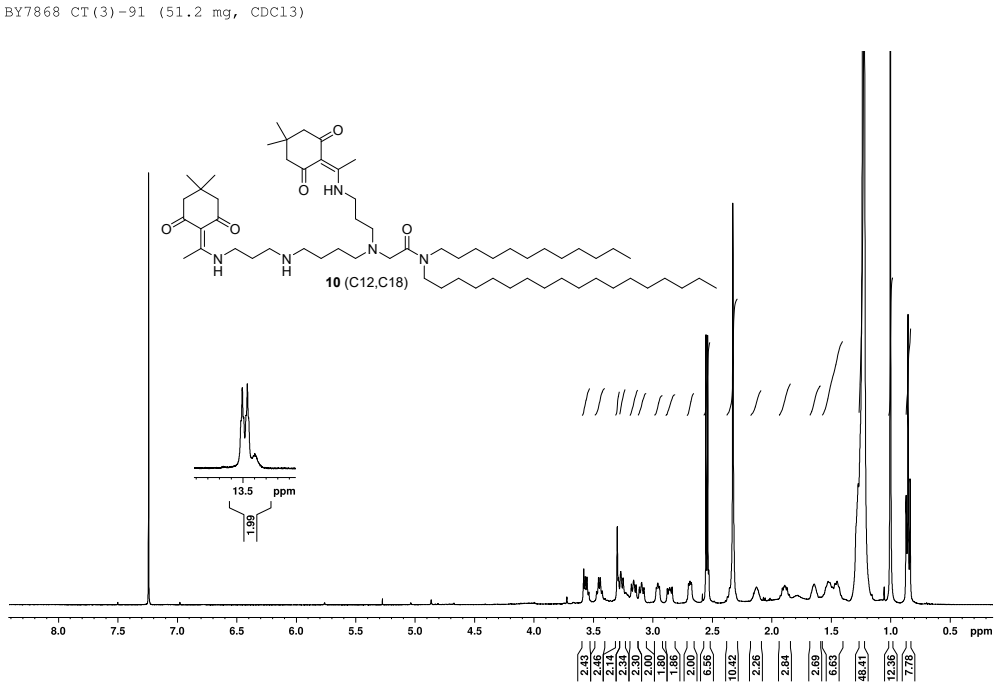


Fig. S53 ^{1}H NMR Spectrum (400 MHz, $CDCl_3$) of compound **10 (C12,C18)**

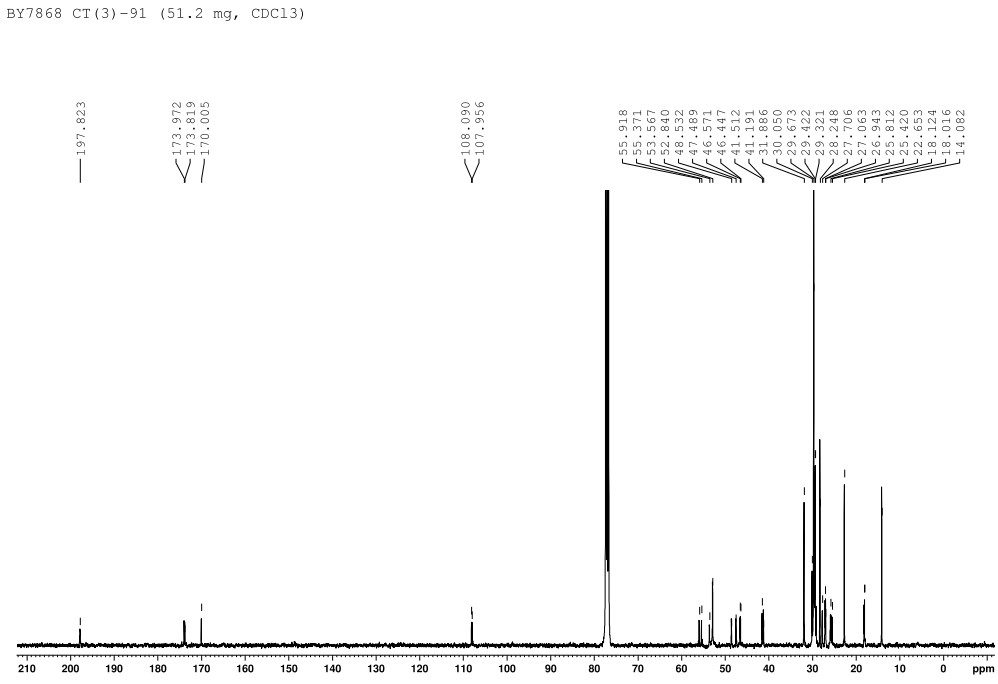


Fig. S54 ^{13}C NMR Spectrum (100 MHz, $CDCl_3$) of compound **10 (C12,C18)**

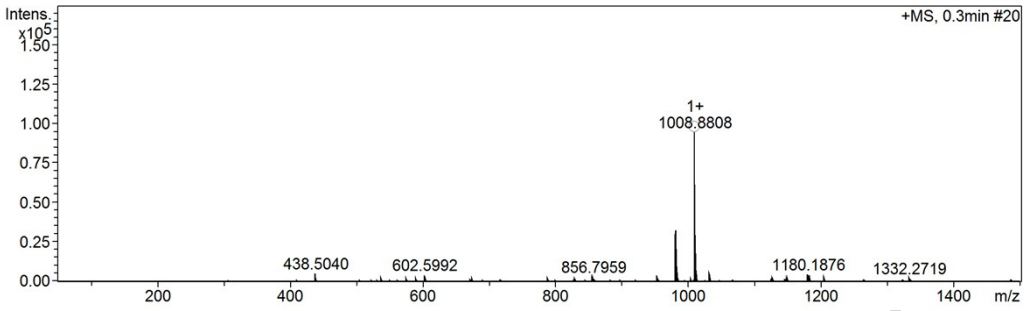
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\Data\Boon-ek\ESI\BY-HRMS 1529 (pos).d	Acquisition Date	12/22/2020 3:16:25 PM
Method	tune_wide-RU.m	Operator	RU
Sample Name	CT(3)-91	Instrument	micrOTOF 8213750.10411
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.3 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	4000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



Meas. m/z	#	Ion Formula	m/z	|err| [mDa]	err [ppm]	Mean err [ppm]	rdb	N-Rule	e⁻ Conf	mSigma
1008.880790	1	C62H114N5O5	1008.881448	0.7	-0.7	-0.1	8.5	ok	even	37.0

Fig. S55 HRMS Spectrum of compound **10 (C12,C18)**

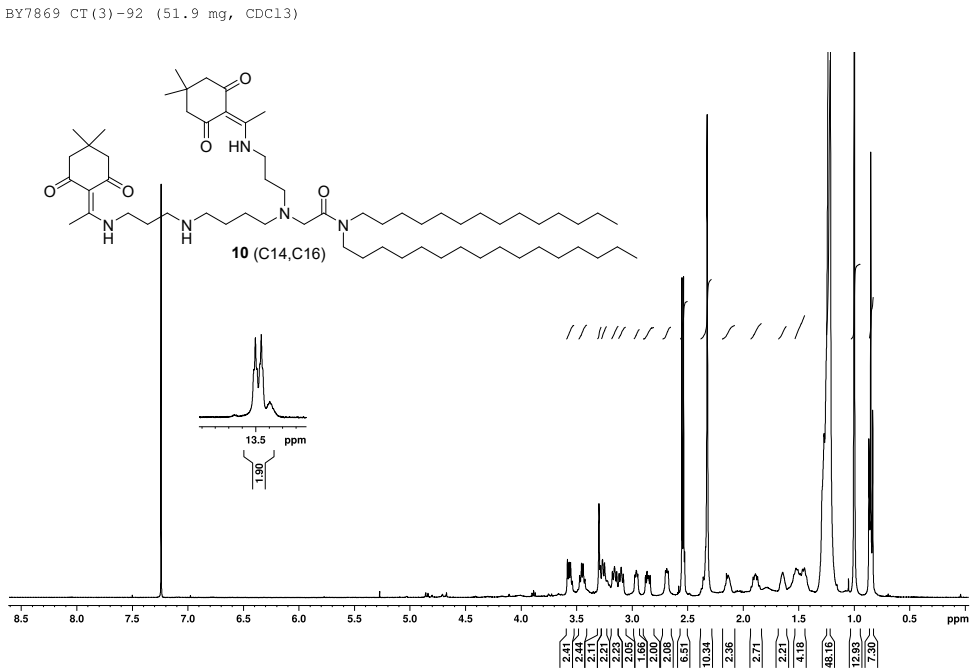


Fig. S56 ^{1}H NMR Spectrum (400 MHz, $CDCl_3$) of compound **10 (C14,C16)**

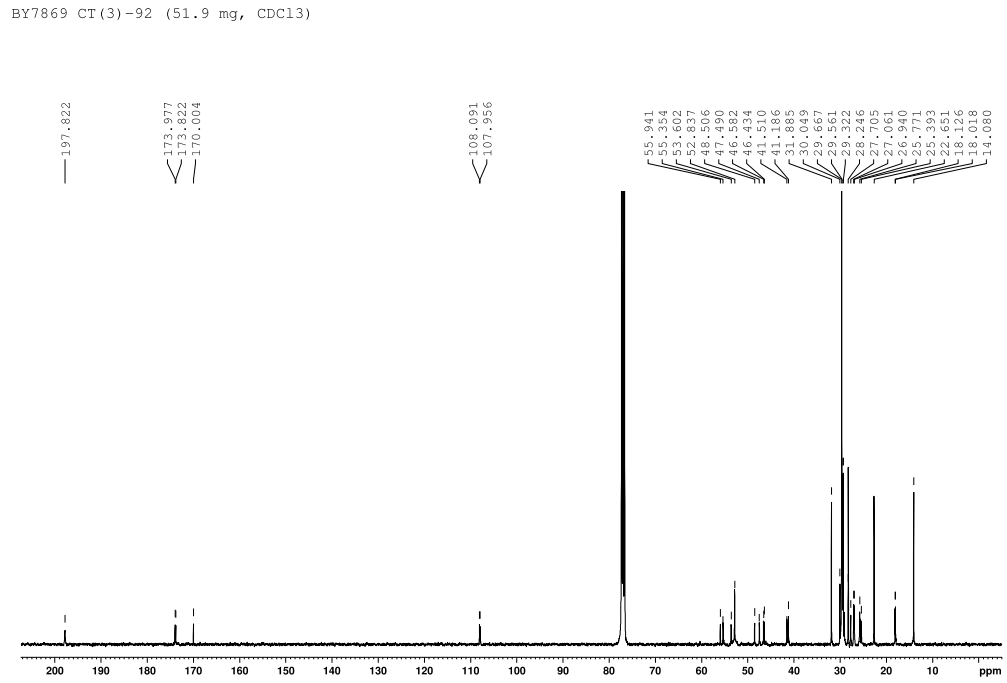


Fig. S57 ^{13}C NMR Spectrum (100 MHz, $CDCl_3$) of compound **10 (C14,C16)**

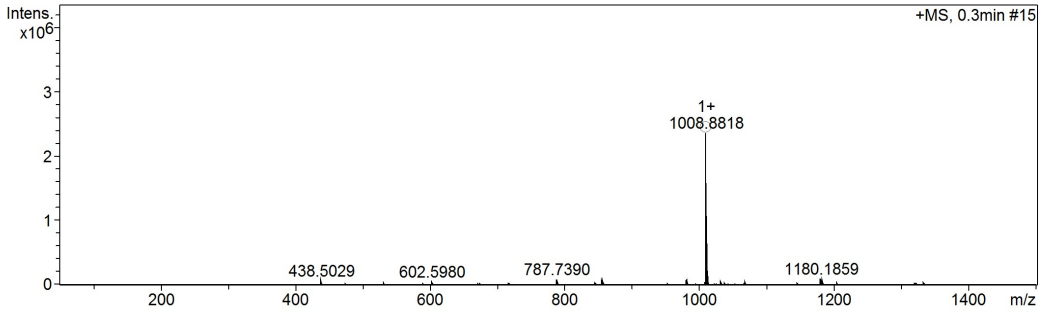
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\Data\Boon-ek\ESI\BY-HRMS 1530 (pos).d	Acquisition Date	12/22/2020 3:17:33 PM
Method	tune_wide-RU.m	Operator	RU
Sample Name	CT(3)-92	Instrument	micrOTOF 8213750.10411
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.3 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	4000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



Meas. m/z	#	Ion Formula	m/z	|err| [mDa]	err [ppm]	Mean err [ppm]	rdb	N-Rule	e⁻ Conf	mSigma
1008.881759	1	C62H114N5O5	1008.881448	0.3	0.3	-1.2	8.5	ok	even	140.5

Fig. S58 HRMS Spectrum of compound **10 (C14,C16)**

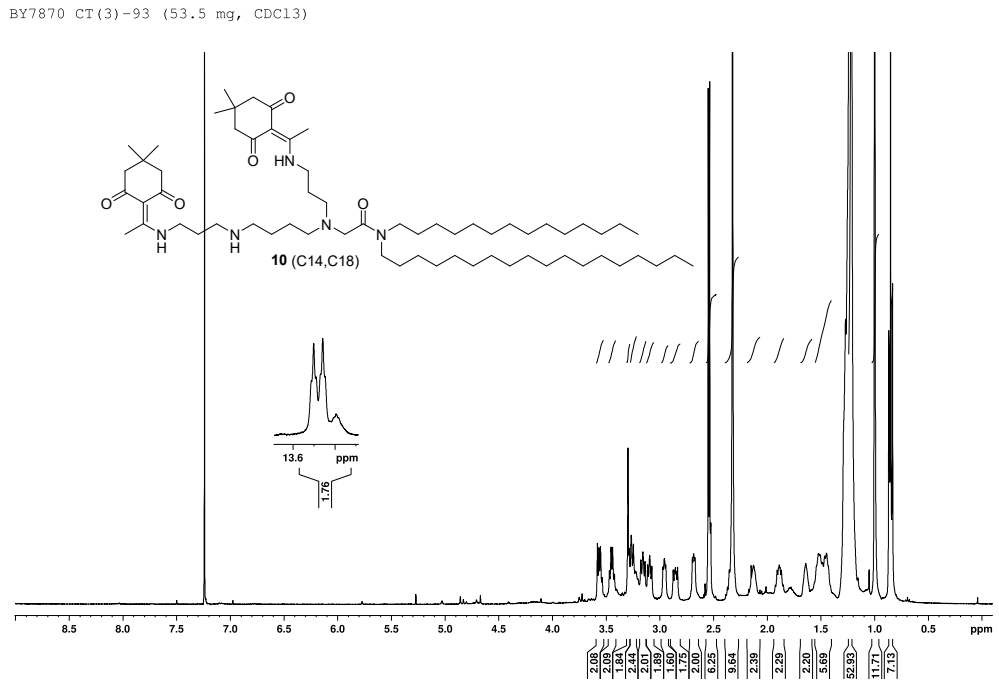


Fig. S59 ^{1}H NMR Spectrum (400 MHz, $CDCl_3$) of compound **10 (C14,C18)**

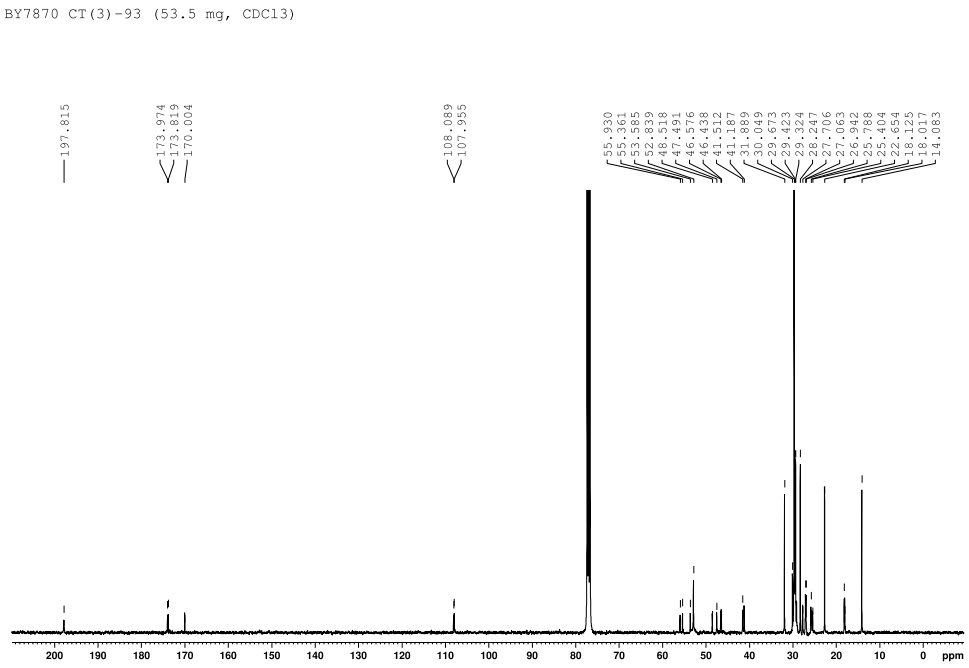


Fig. S60 ^{13}C NMR Spectrum (100 MHz, $CDCl_3$) of compound **10 (C14,C18)**

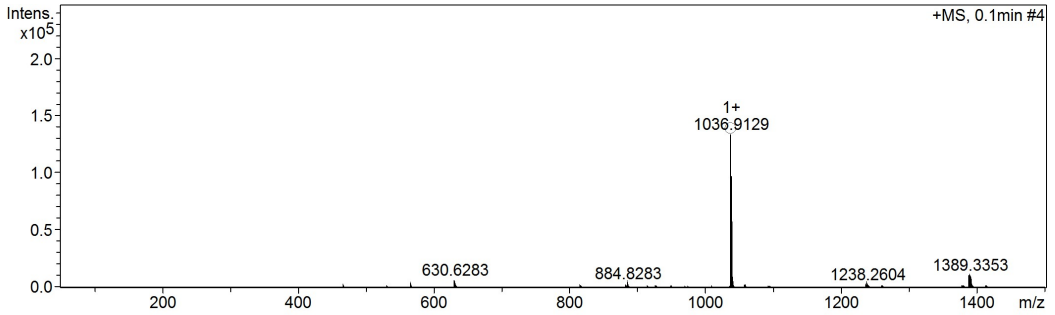
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\Data\Boon-ek\ESI\BY-HRMS 1531 (pos).d	Acquisition Date	12/22/2020 3:20:54 PM
Method	tune_wide-RU.m	Operator	RU
Sample Name	CT(3)-93	Instrument	micrOTOF 8213750.10411
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.3 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	4000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



Meas. m/z	#	Ion Formula	m/z	|err| [mDa]	err [ppm]	Mean err [ppm]	rdb	N-Rule	e⁻ Conf	mSigma
1036.912928	1	C64H118N5O5	1036.912748	0.2	-0.2	0.3	8.5	ok	even	3.7

Fig. S61 HRMS Spectrum of compound **10 (C14,C18)**

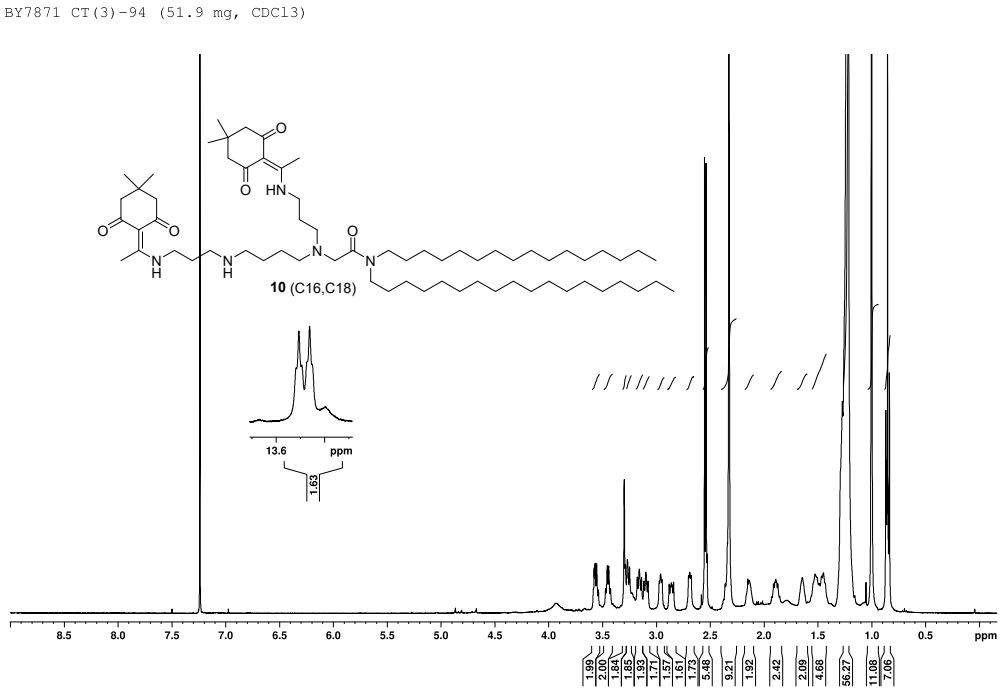


Fig. S62 ^{1}H NMR Spectrum (400 MHz, $CDCl_3$) of compound **10 (C16,C18)**

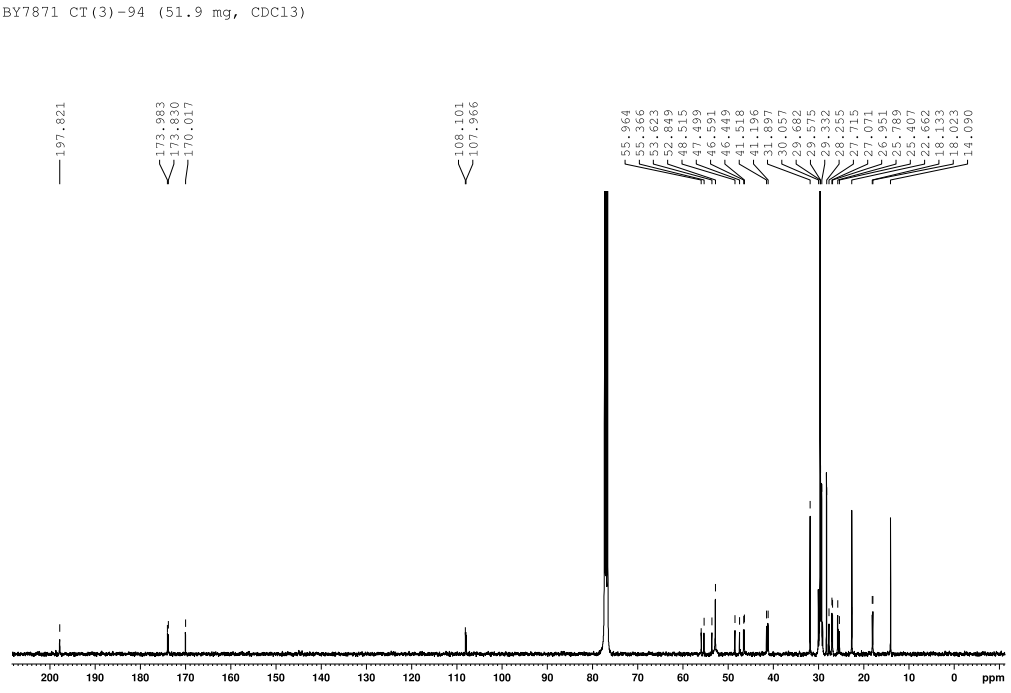


Fig. S63 ^{13}C NMR Spectrum (100 MHz, $CDCl_3$) of compound **10 (C16,C18)**

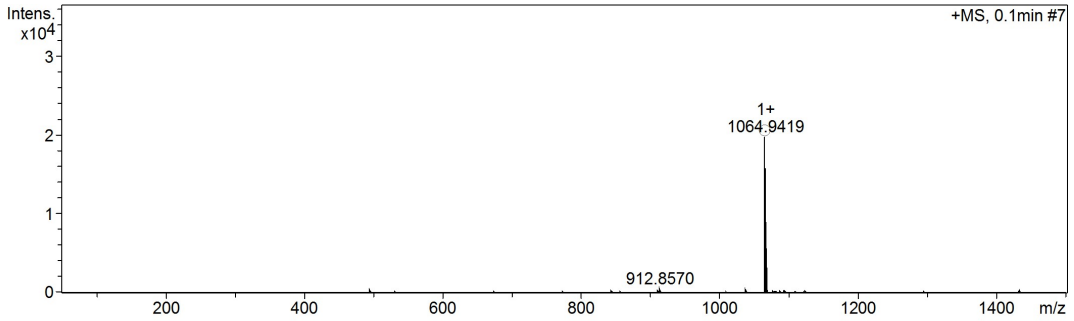
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\Data\Boon-ek\ESI\BY-HRMS 1532 (pos).d	Acquisition Date	12/22/2020 3:21:40 PM
Method	tune_wide-RU.m	Operator	RU
Sample Name	CT(3)-94	Instrument	micrOTOF 8213750.10411
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.3 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	4000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



Meas. m/z	#	Ion Formula	m/z	|err| [mDa]	err [ppm]	Mean err [ppm]	rdb	N-Rule	e⁻ Conf	mSigma
1064.941946	1	C66H122N5O5	1064.944048	2.1	2.0	2.0	8.5	ok	even	20.4

Fig. S64 HRMS Spectrum of compound **10 (C16,C18)**

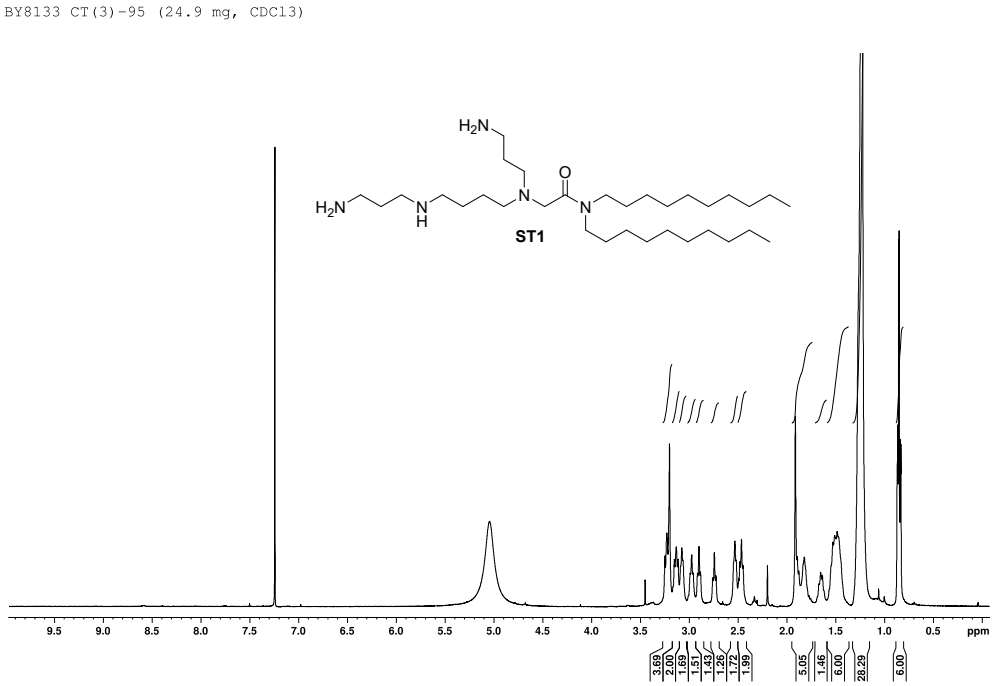


Fig. S65 ^{1}H NMR Spectrum (400 MHz, $CDCl_3$) of lipid **ST1**

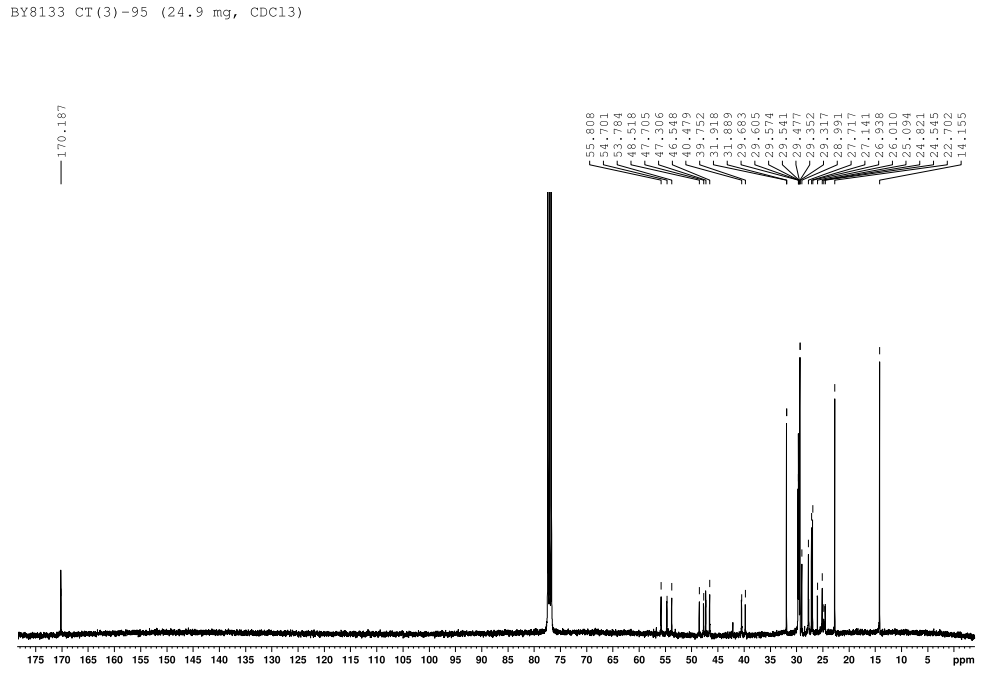


Fig. S66 ^{13}C NMR Spectrum (100 MHz, $CDCl_3$) of lipid **ST1**

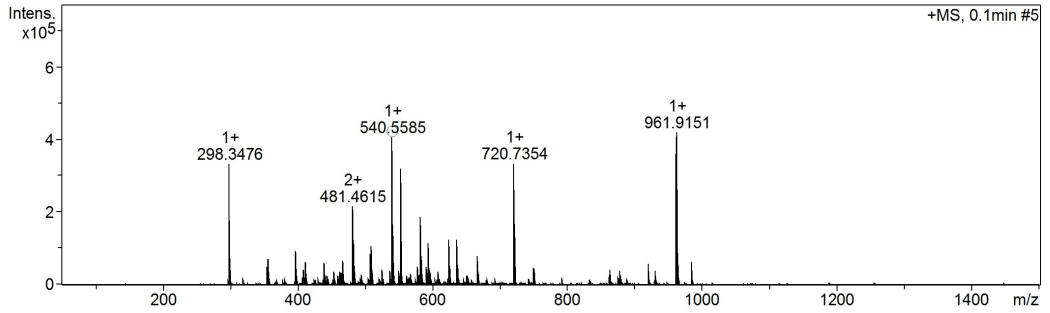
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\Data\Boon-ek\ESI\BY-HRMS 1560 (pos).d	Acquisition Date	12/25/2020 2:52:07 PM
Method	tune_wide_wow 2-7-62.m	Operator	RU
Sample Name	CT(3)-95	Instrument	micrOTOF 8213750.10411
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.5 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



Meas. m/z	#	Ion Formula	m/z	|err| [mDa]	err [ppm]	Mean err [ppm]	rdb	N-Rule	e⁻ Conf	mSigma
540.558541	1	C32H70N5O	540.557488	1.1	1.9	-1.4	0.5	ok	even	1.9

Fig. S67HRMS Spectrum of lipid **ST1**

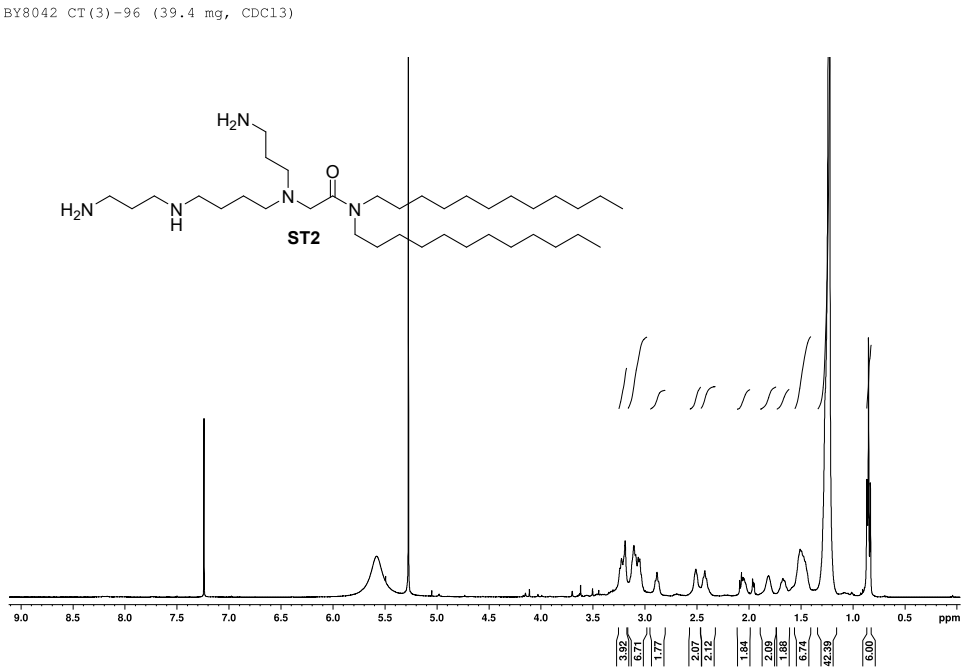


Fig. S68 ^{1}H NMR Spectrum (400 MHz, $CDCl_3$) of lipid **ST2**

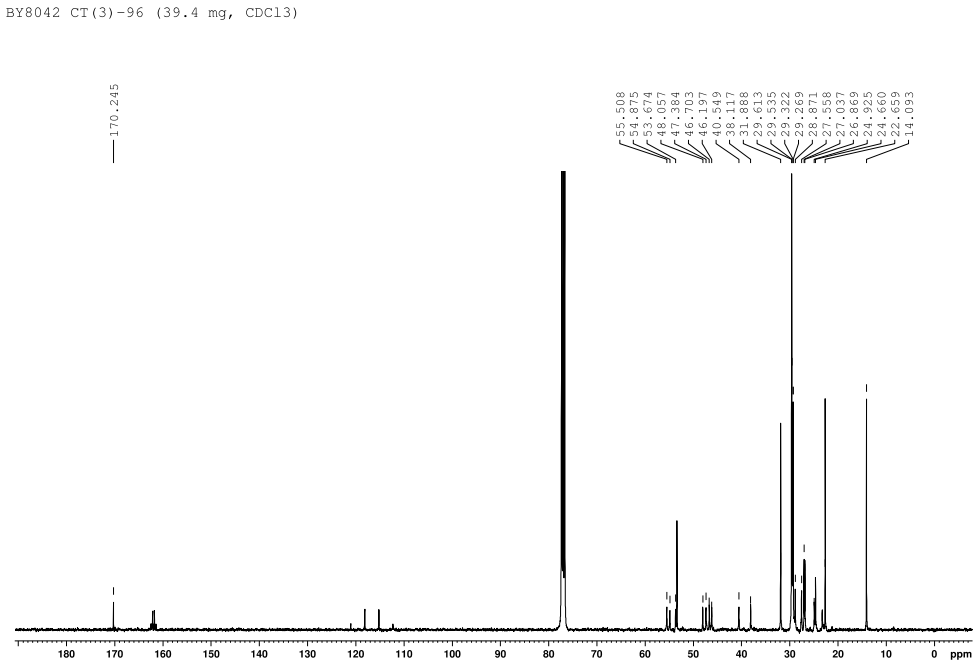


Fig. S69 ^{13}C NMR Spectrum (100 MHz, $CDCl_3$) of lipid **ST2**

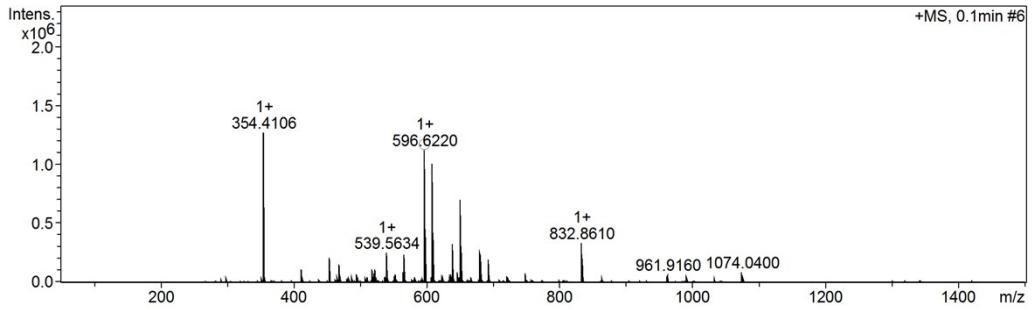
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\Data\Boon-ek\ESI\BY-HRMS 1561 (pos).d	Acquisition Date	12/25/2020 2:52:59 PM
Method	tune_wide_wow 2-7-62.m	Operator	RU
Sample Name	CT(3)-96	Instrument	micrOTOF 8213750.10411
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.5 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



Meas. m/z	#	Ion Formula	m/z	|err| [mDa]	err [ppm]	Mean err [ppm]	rdb	N-Rule	e¯ Conf	mSigma
596.622041	1	C36H78N5O	596.620089	2.0	-3.3	-2.7	0.5	ok	even	6.7

Fig. S70 HRMS Spectrum of lipid **ST2**

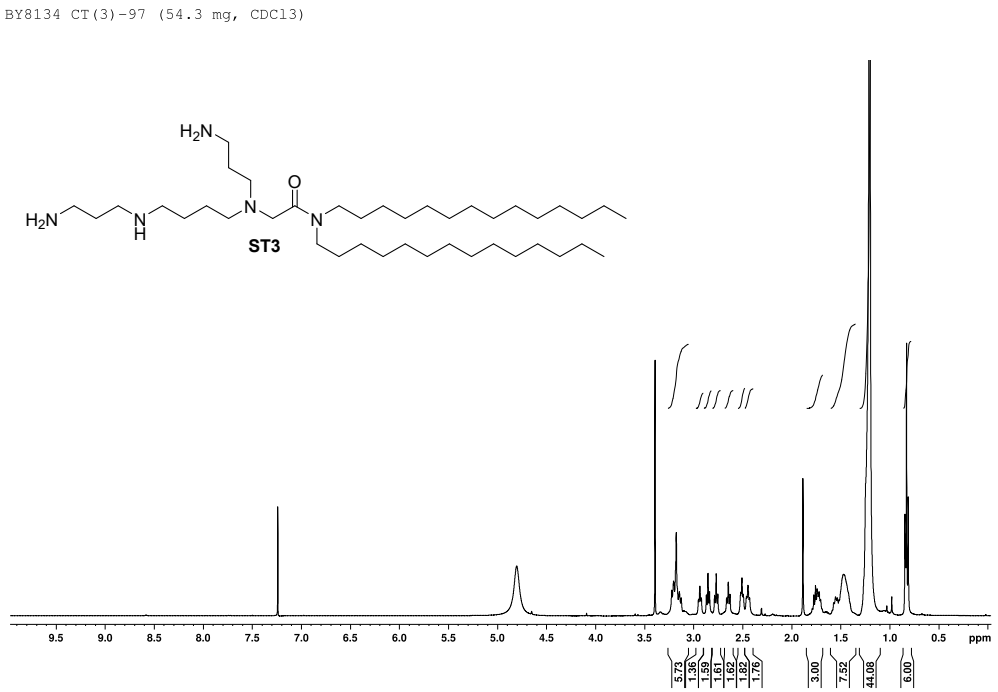


Fig. S71 ^{1}H NMR Spectrum (400 MHz, $CDCl_3$) of lipid **ST3**

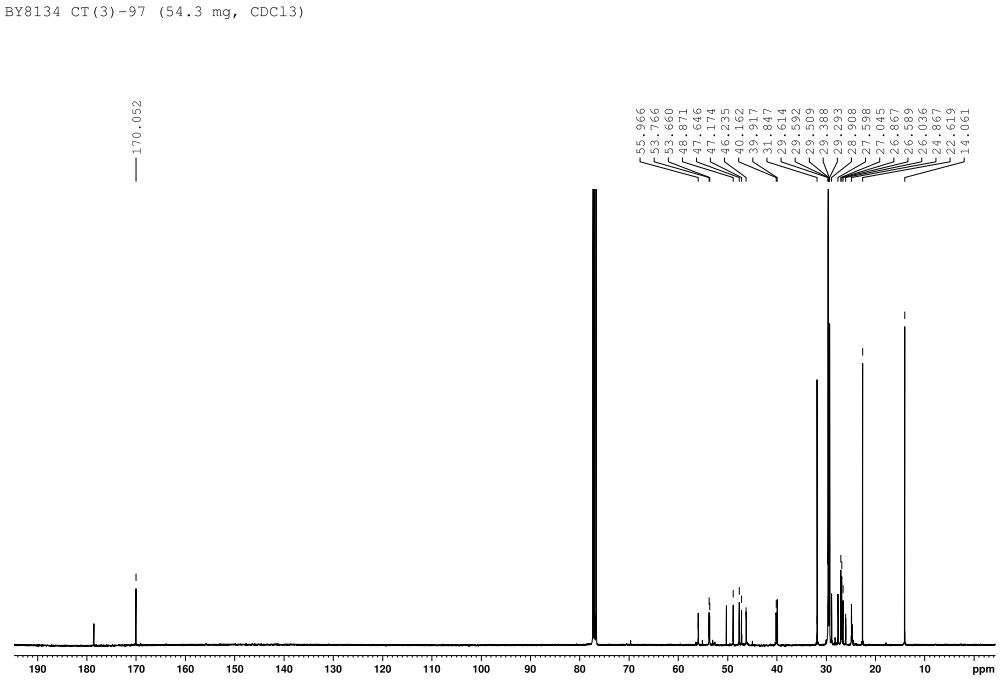


Fig. S72 ^{13}C NMR Spectrum (100 MHz, $CDCl_3$) of lipid **ST3**

Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\Data\Boon-ek\ESI\BY-HRMS 1561 (pos).d	Acquisition Date	12/25/2020 2:52:59 PM
Method	tune_wide_wow 2-7-62.m	Operator	RU
Sample Name	CT(3)-96	Instrument	micrOTOF 8213750.10411
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.5 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste

Meas. m/z	#	Ion Formula	m/z	|err| [mDa]	err [ppm]	Mean err [ppm]	rdb	N-Rule	e⁻ Conf	mSigma
596.622041	1	C36H78N5O	596.620089	2.0	-3.3	-2.7	0.5	ok	even	6.7

Fig. S73 HRMS Spectrum of lipid **ST3**

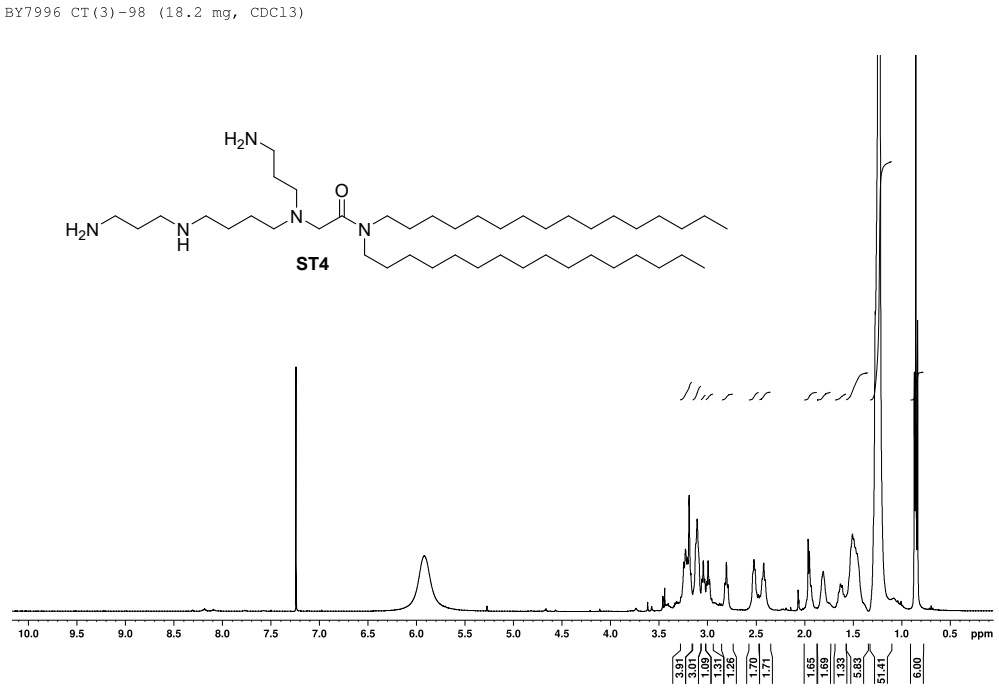


Fig. S74 ^{1}H NMR Spectrum (400 MHz, $CDCl_3$) of lipid **ST4**

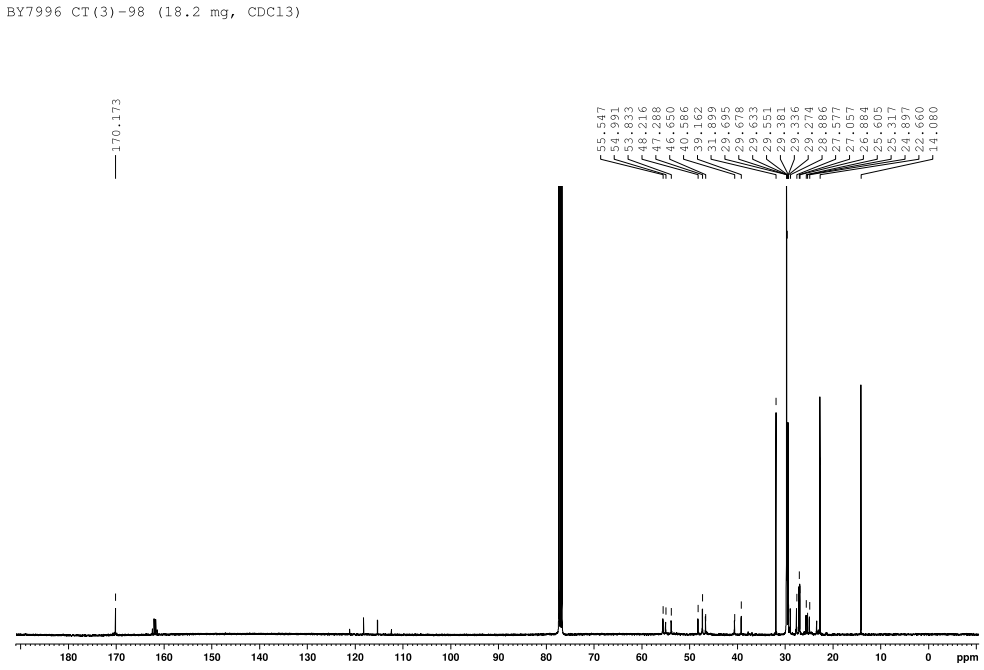


Fig. S75 ^{13}C NMR Spectrum (100 MHz, $CDCl_3$) of lipid **ST4**

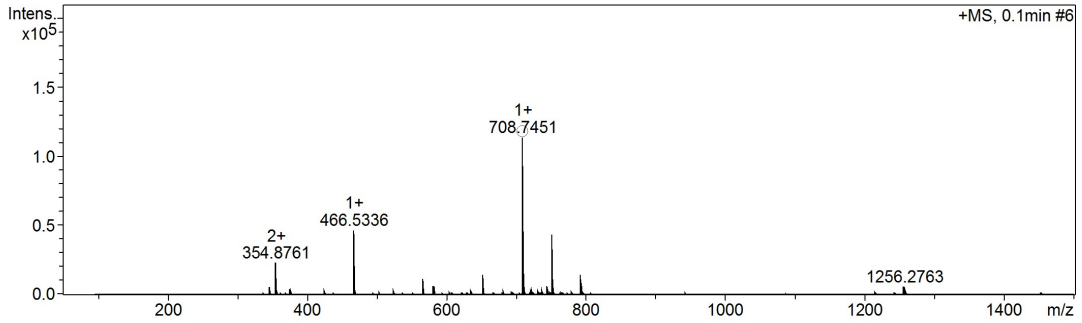
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\Data\Boon-ek\ESI\BY-HRMS 1500 (pos).d	Acquisition Date	12/15/2020 3:31:15 PM
Method	tune_wide(pos).m	Operator	RU
Sample Name	CT(3)-98	Instrument	micrOTOF 8213750.10411
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.3 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	100 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	4000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



Meas. m/z	#	Ion Formula	m/z	|err| [mDa]	err [ppm]	Mean err [ppm]	rdb	N-Rule	e⁻ Conf	mSigma
708.745082	1	C44H94N5O	708.745289	0.2	-0.3	0.3	0.5	ok	even	10.0

Bruker Compass DataAnalysis 4.1 printed: 12/26/2020 10:34:39 AM by: RU 1 of 1

Fig. S76 HRMS Spectrum of lipid **ST4**

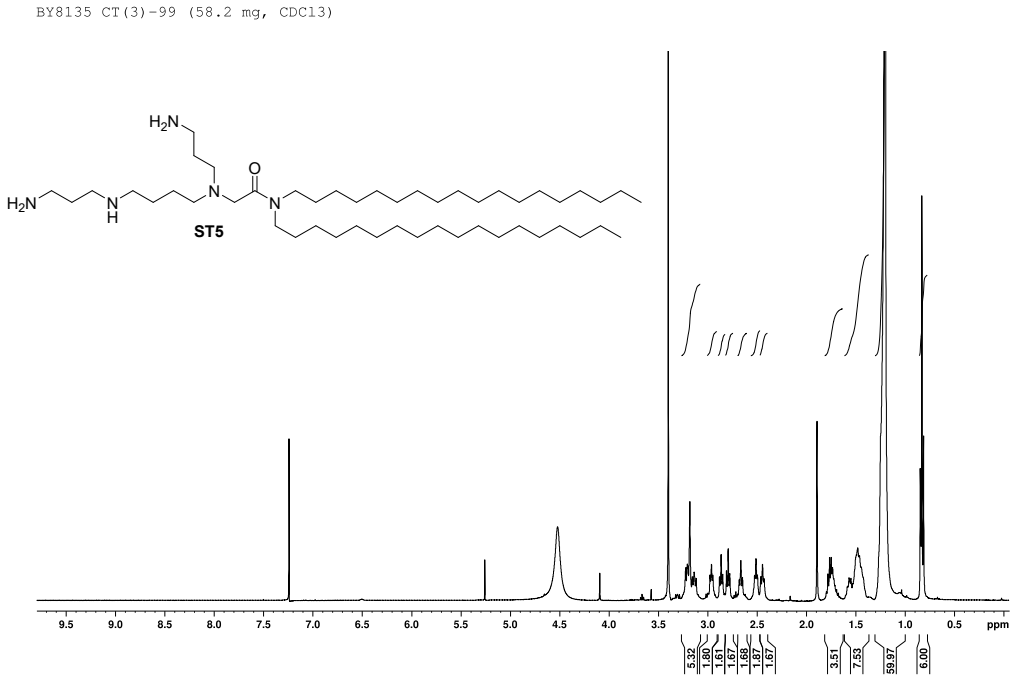


Fig. S77 ^{1}H NMR Spectrum (400 MHz, $CDCl_3$) of lipid **ST5**

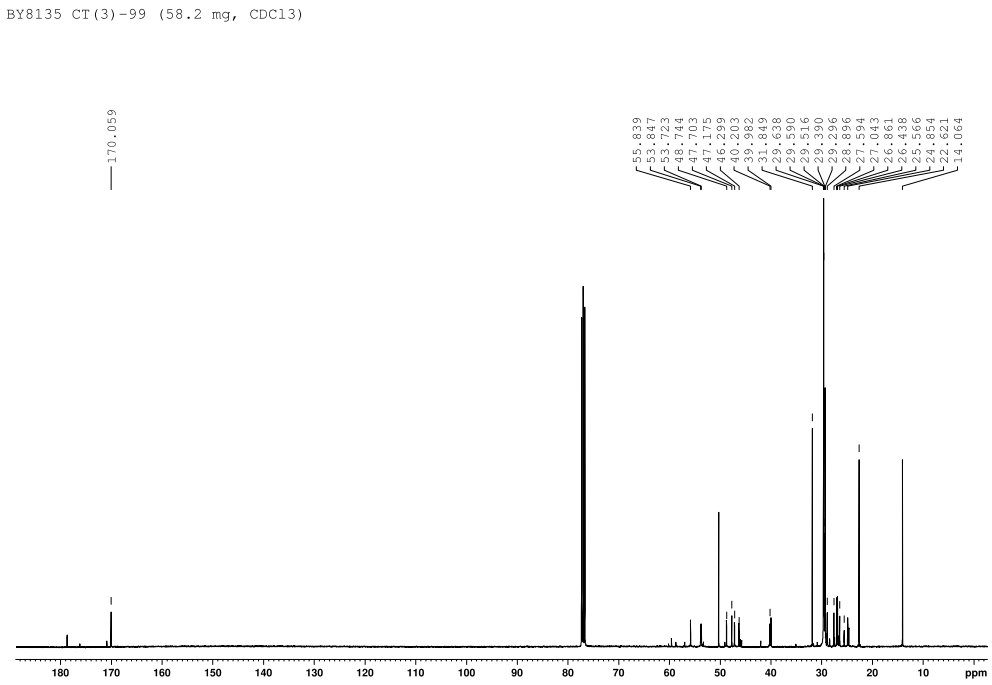


Fig. S78 ^{13}C NMR Spectrum (100 MHz, $CDCl_3$) of lipid **ST5**

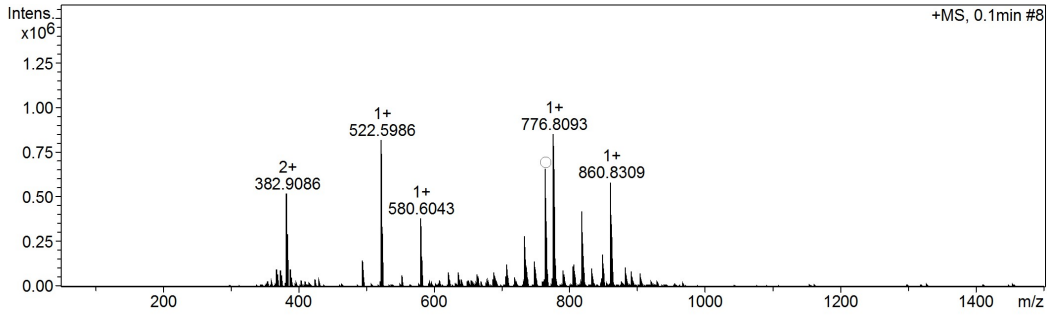
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\Data\Boon-ek\ESI\BY-HRMS 1563 (pos).d	Acquisition Date	12/25/2020 2:56:35 PM
Method	tune_wide_wow 2-7-62.m	Operator	RU
Sample Name	CT(3)-99	Instrument	micrOTOF 8213750.10411
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.5 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



Meas. m/z	#	Ion Formula	m/z	|err| [mDa]	err [ppm]	Mean err [ppm]	rdb	N-Rule	e⁻ Conf	mSigma
764.809400	1	C48H102N5O	764.807889	1.5	2.0	-1.4	0.5	ok	even	4.2

Fig. S79 HRMS Spectrum of lipid **ST5**

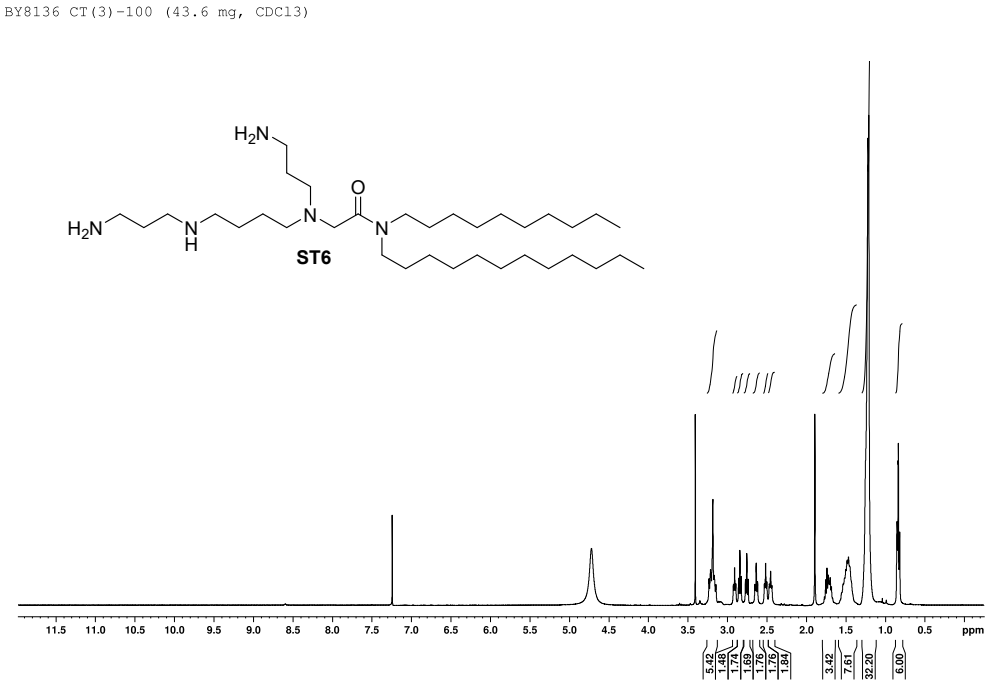


Fig. S80 ^{1}H NMR Spectrum (400 MHz, $CDCl_3$) of lipid **ST6**

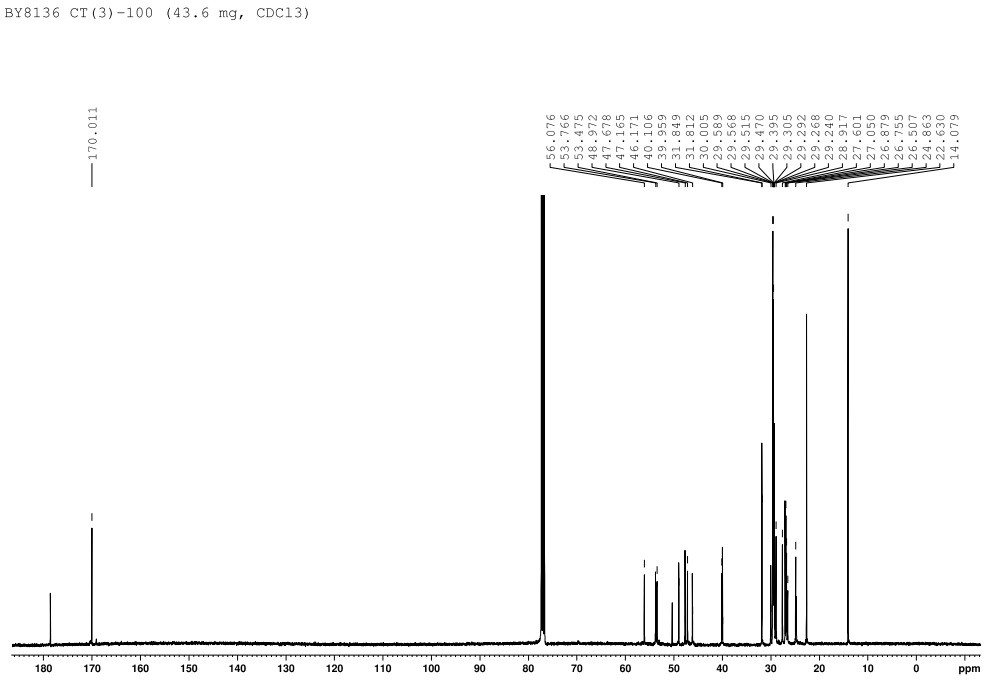


Fig. S81 ^{13}C NMR Spectrum (100 MHz, $CDCl_3$) of lipid **ST6**

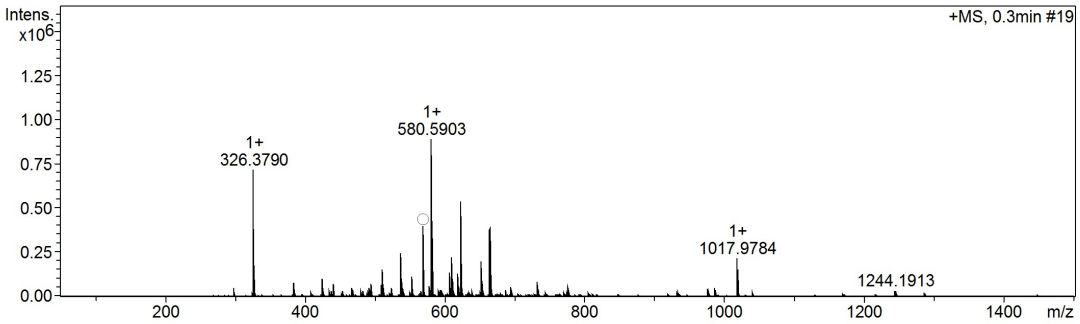
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\Data\Boon-ek\ESI\BY-HRMS 1564 (pos).d	Acquisition Date	12/25/2020 2:58:39 PM
Method	tune_wide_wow 2-7-62.m	Operator	RU
Sample Name	CT(3)-100	Instrument	micrOTOF 8213750.10411
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.5 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



Meas. m/z	#	Ion Formula	m/z	|err| [mDa]	err [ppm]	Mean err [ppm]	rdb	N-Rule	e⁻ Conf	mSigma
568.589904	1	C34H74N5O	568.588788	1.1	2.0	-1.5	0.5	ok	even	7.8

Fig. S82 HRMS Spectrum of lipid **ST6**

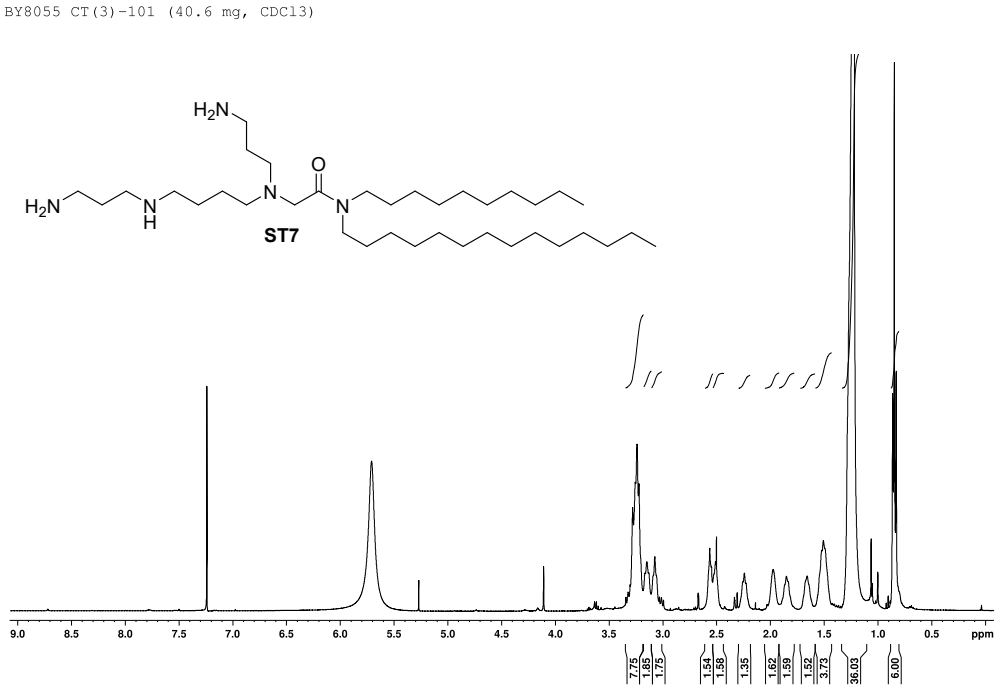


Fig. S83 ^{1}H NMR Spectrum (400 MHz, $CDCl_3$) of lipid **ST7**

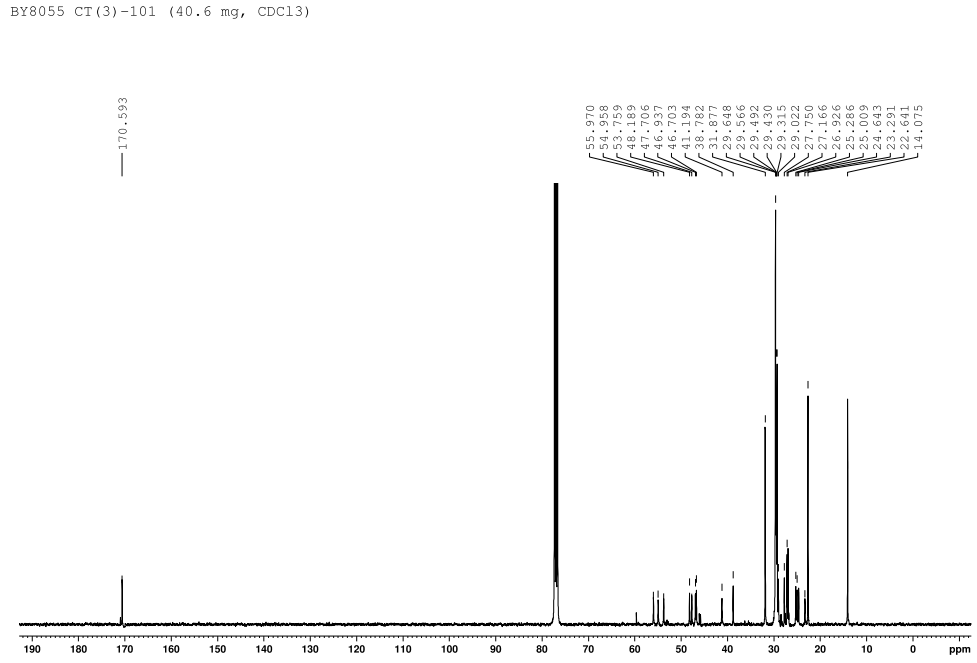


Fig. S84 ^{13}C NMR Spectrum (100 MHz, $CDCl_3$) of lipid **ST7**

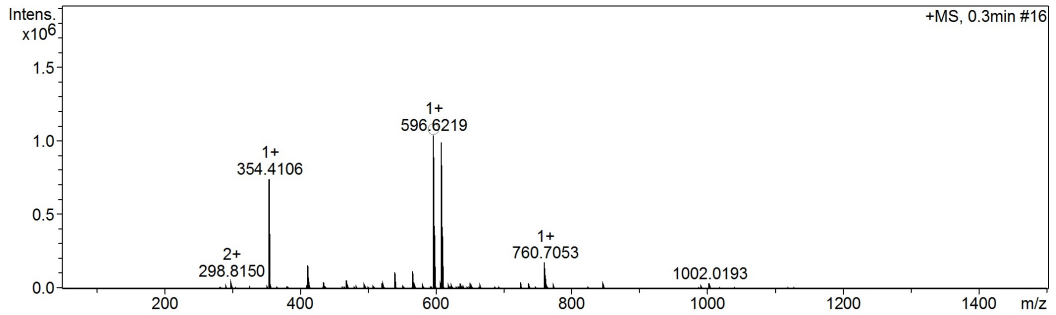
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\Data\Boon-ek\ESI\BY-HRMS 1565 (pos).d	Acquisition Date	12/25/2020 2:59:40 PM
Method	tune_wide_wow 2-7-62.m	Operator	RU
Sample Name	CT(3)-101	Instrument	micrOTOF 8213750.10411
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.5 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



Meas. m/z	#	Ion Formula	m/z	|err| [mDa]	err [ppm]	Mean err [ppm]	rdb	N-Rule	e⁻ Conf	mSigma
596.621902	1	C36H78N5O	596.620089	1.8	3.0	-2.6	0.5	ok	even	4.6

Fig. S85 HRMS Spectrum of lipid **ST7**

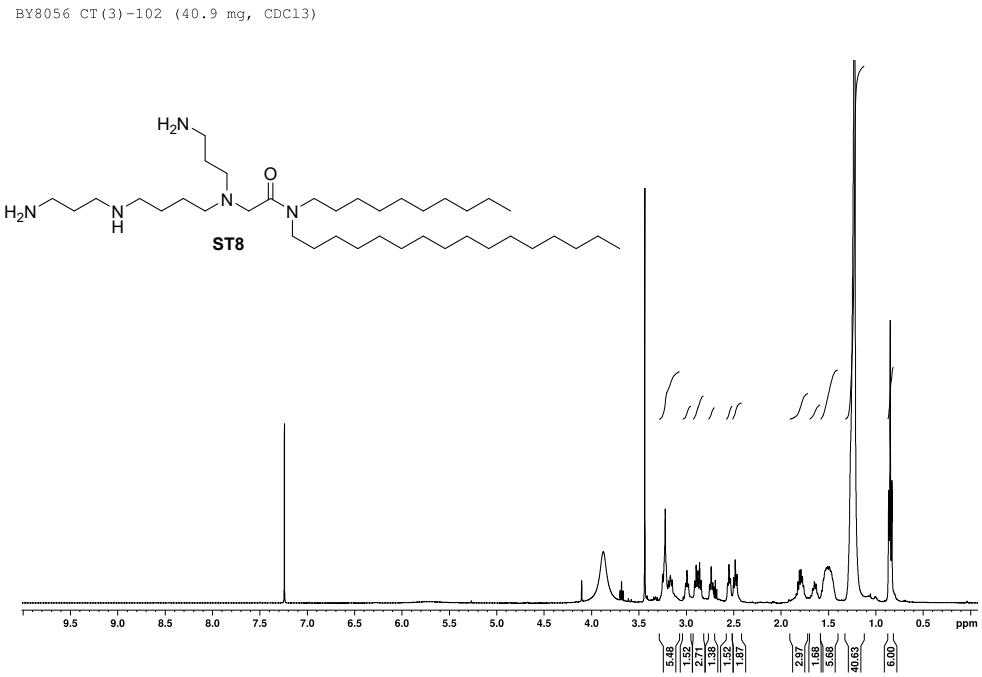


Fig. S86 ^{1}H NMR Spectrum (400 MHz, $CDCl_3$) of lipid **ST8**

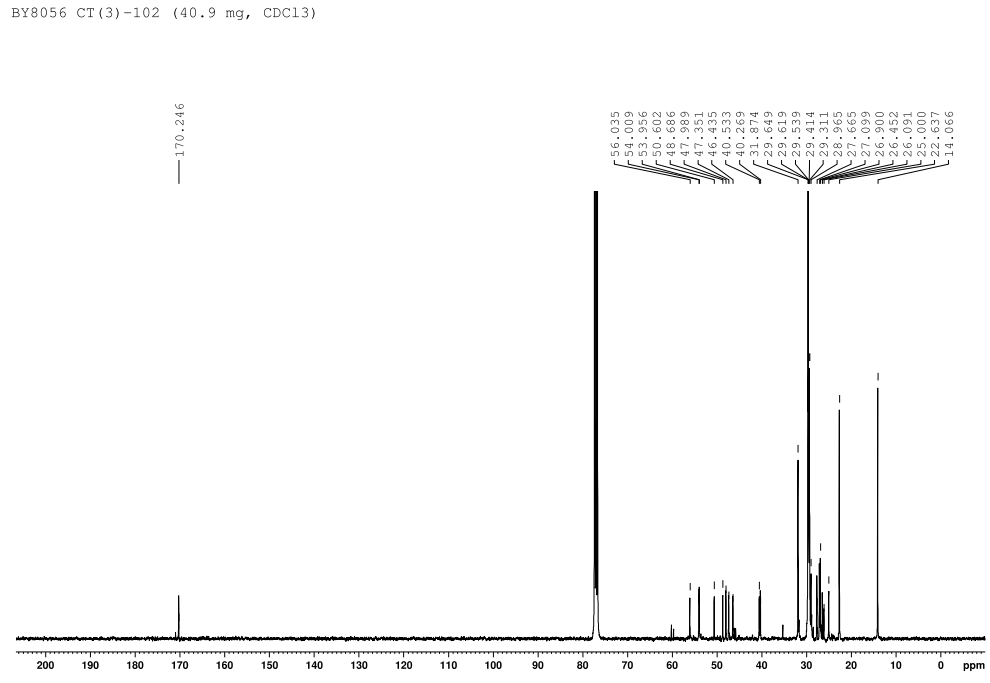


Fig. S87 ^{13}C NMR Spectrum (100 MHz, $CDCl_3$) of lipid **ST8**

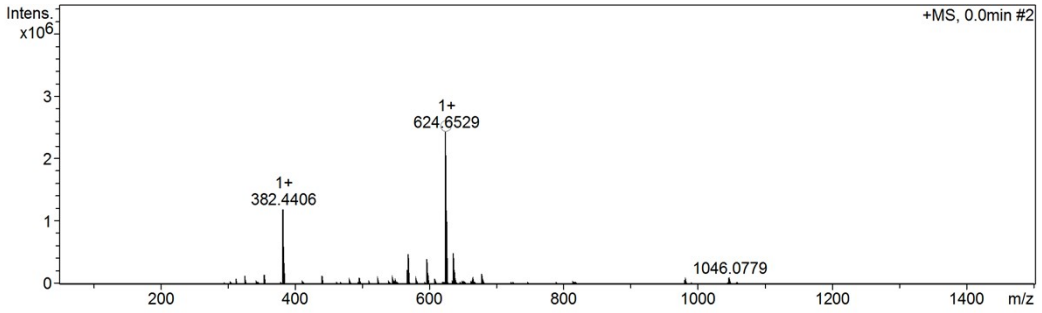
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\Data\Boon-ek\ESI\BY-HRMS 1567 (pos).d	Acquisition Date	12/27/2020 11:55:16 AM
Method	tune_wide_wow 2-7-62.m	Operator	RU
Sample Name	CT(3)-102	Instrument	micrOTOF 8213750.10411
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.5 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



Meas. m/z	#	Ion Formula	m/z	|err| [mDa]	err [ppm]	Mean err [ppm]	rdb	N-Rule	e⁻ Conf	mSigma
624.652894	1	C38H82N5O	624.651389	1.5	-2.4	-1.4	0.5	ok	even	22.2

Fig. S88 HRMS Spectrum of lipid **ST8**

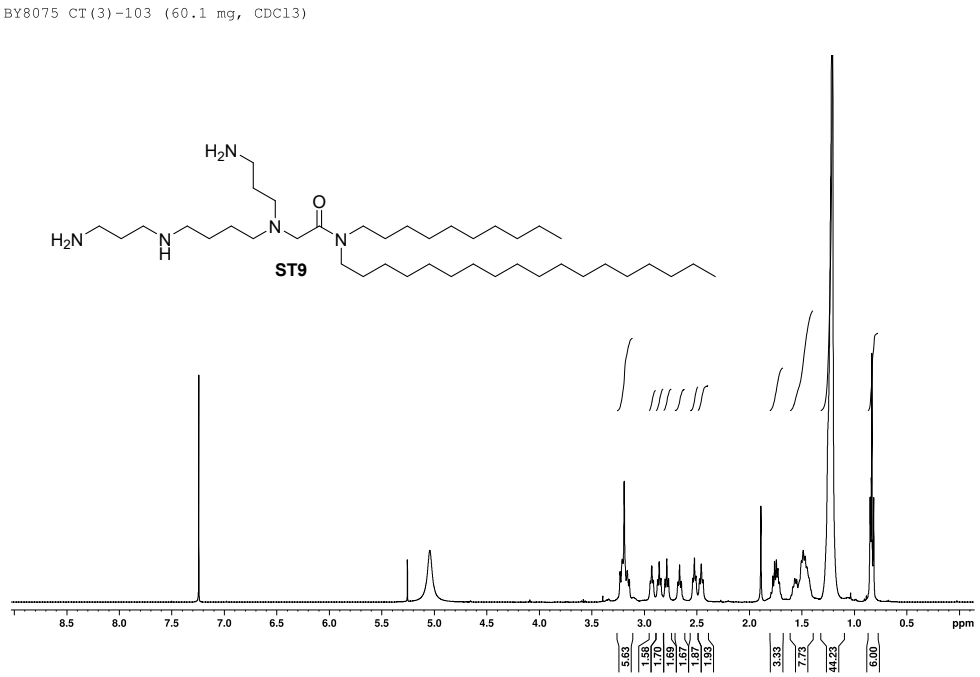


Fig. S89 ^{1}H NMR Spectrum (400 MHz, $CDCl_3$) of lipid **ST9**

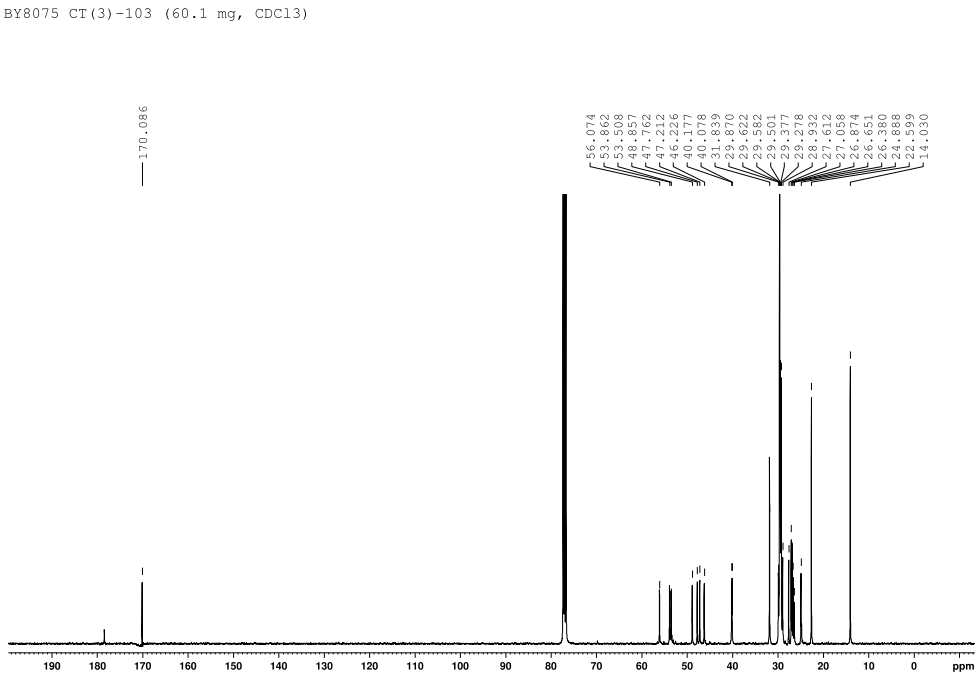


Fig. S90 ^{13}C NMR Spectrum (100 MHz, $CDCl_3$) of lipid **ST9**

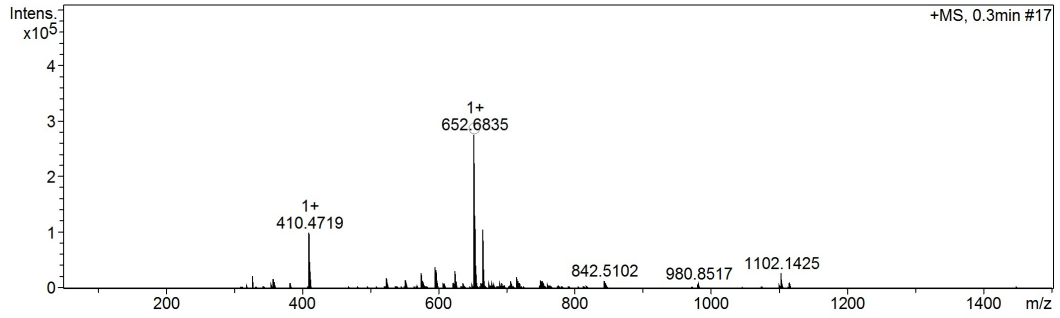
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\Data\Boon-ek\ESI\BY-HRMS 1568 (pos).d	Acquisition Date	12/27/2020 11:56:34 AM
Method	tune_wide_wow 2-7-62.m	Operator	RU
Sample Name	CT(3)-103	Instrument	micrOTOF 8213750.10411
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.5 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



Meas. m/z	#	Ion Formula	m/z	|err| [mDa]	err [ppm]	Mean err [ppm]	rdb	N-Rule	e⁻ Conf	mSigma
652.683477	1	C40H86N5O	652.682689	0.8	-1.2	-0.6	0.5	ok	even	7.4

Fig. S91 HRMS Spectrum of lipid **ST9**

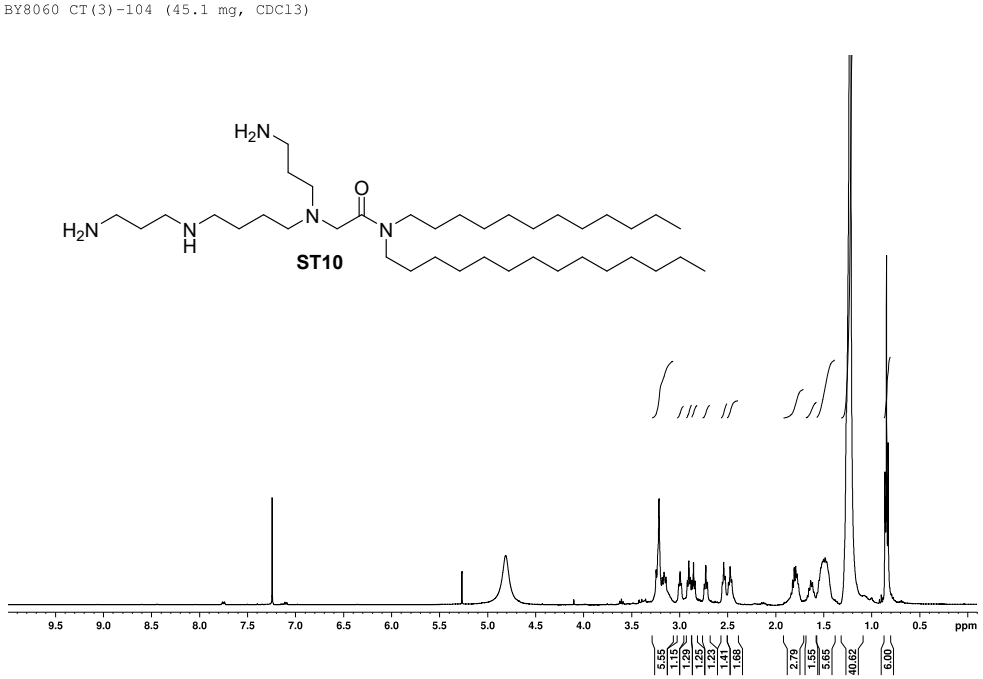


Fig. S92 ^{1}H NMR Spectrum (400 MHz, $CDCl_3$) of lipid **ST10**

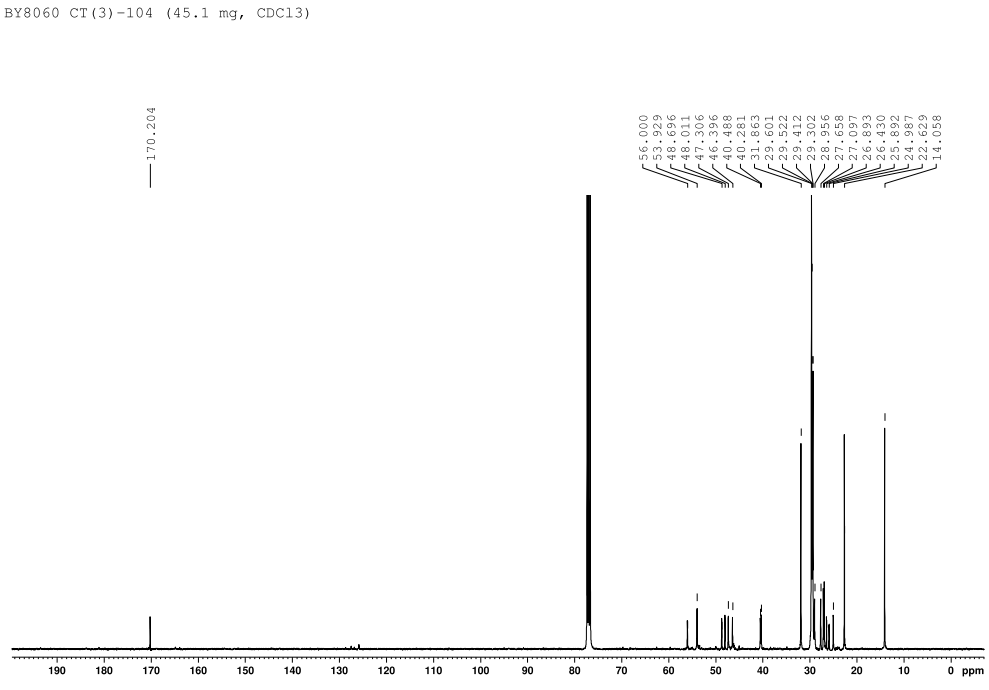


Fig. S93 ^{13}C NMR Spectrum (100 MHz, $CDCl_3$) of lipid **ST10**

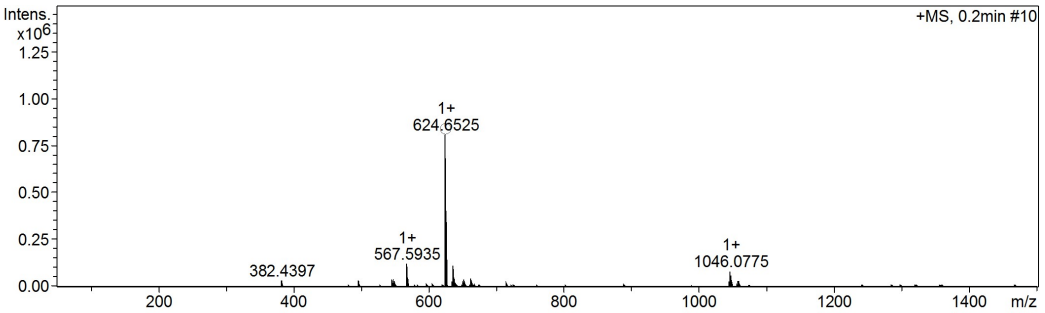
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\Data\Boon-ek\ESI\BY-HRMS 1515 (pos).d	Acquisition Date	12/22/2020 2:59:02 PM
Method	tune_wide-RU.m	Operator	RU
Sample Name	CT(3)-104	Instrument	micrOTOF 8213750.10411
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.3 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	4000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



Meas. m/z	#	Ion Formula	m/z	|err| [mDa]	err [ppm]	Mean err [ppm]	rdb	N-Rule	e⁻ Conf	mSigma
624.652514	1	C38H82N5O	624.651389	1.1	1.8	-1.2	0.5	ok	even	31.7

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Fig. S94 HRMS Spectrum of lipid **ST10**

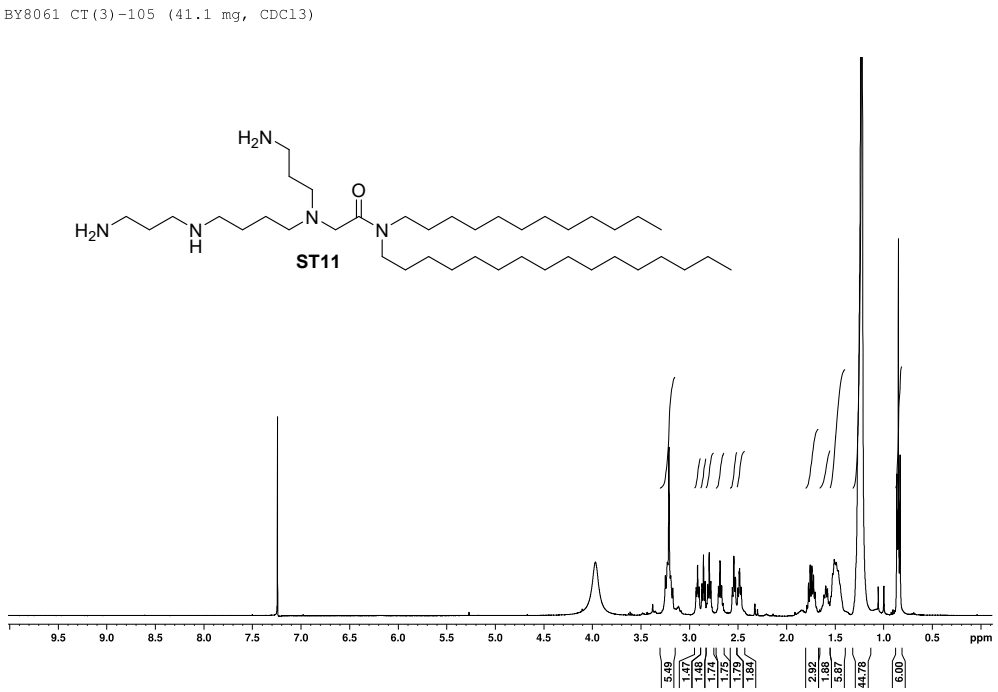


Fig. S95 ^{1}H NMR Spectrum (400 MHz, $CDCl_3$) of lipid **ST11**

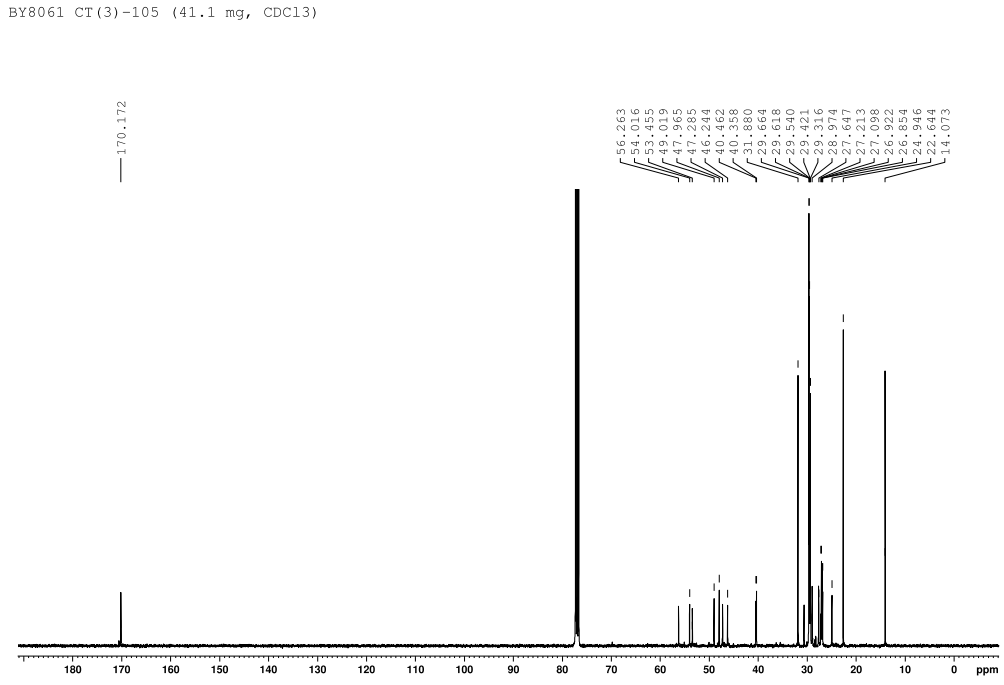


Fig. S96 ^{13}C NMR Spectrum (100 MHz, $CDCl_3$) of lipid **ST11**

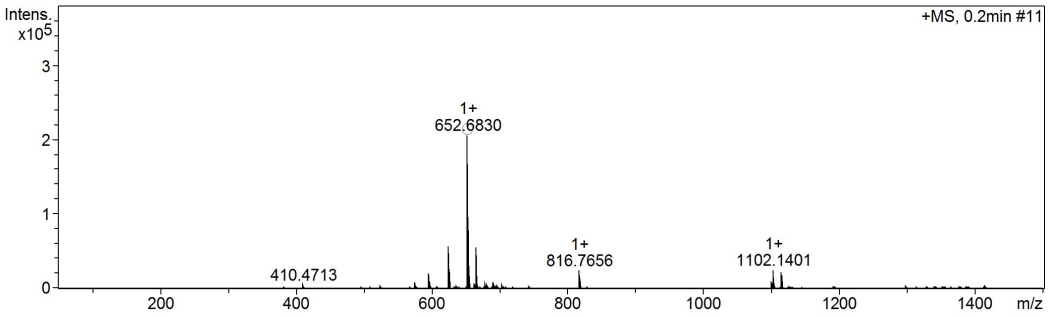
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\Data\Boon-ek\ESI\BY-HRMS 1516 (pos).d	Acquisition Date	12/22/2020 3:00:12 PM
Method	tune_wide-RU.m	Operator	RU
Sample Name	CT(3)-105	Instrument	micrOTOF 8213750.10411
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.3 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	4000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



Meas. m/z	#	Ion Formula	m/z	|err| [mDa]	err [ppm]	Mean err [ppm]	rdb	N-Rule	e⁻ Conf	mSigma
652.682991	1	C40H86N5O	652.682689	0.3	0.5	-0.2	0.5	ok	even	3.5

Fig. S97 HRMS Spectrum of lipid **ST11**

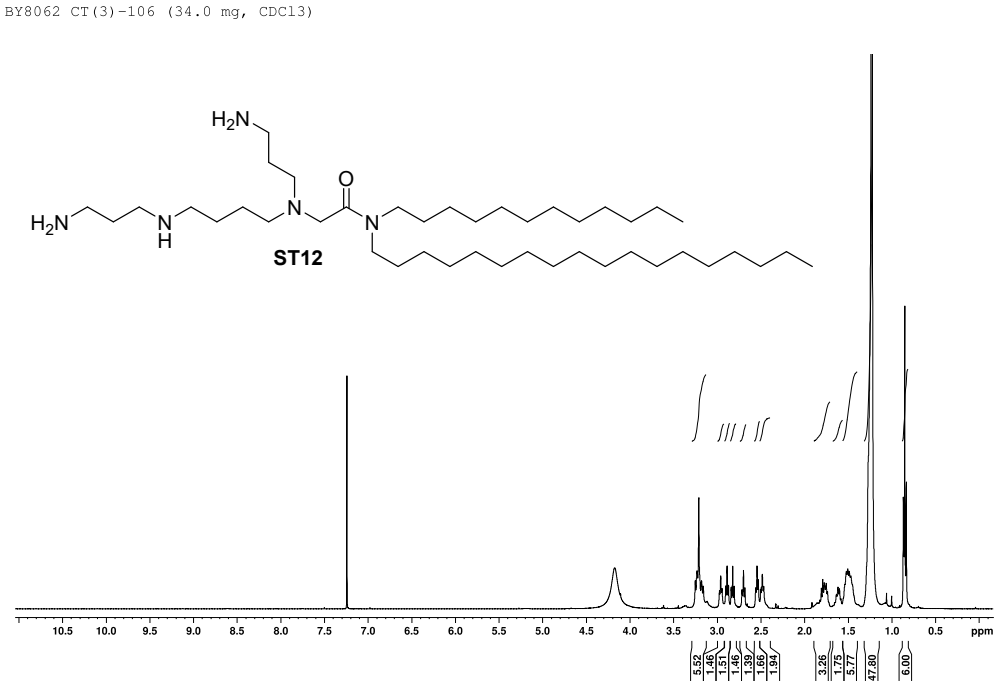


Fig. S98 ^{1}H NMR Spectrum (400 MHz, $CDCl_3$) of lipid **ST12**

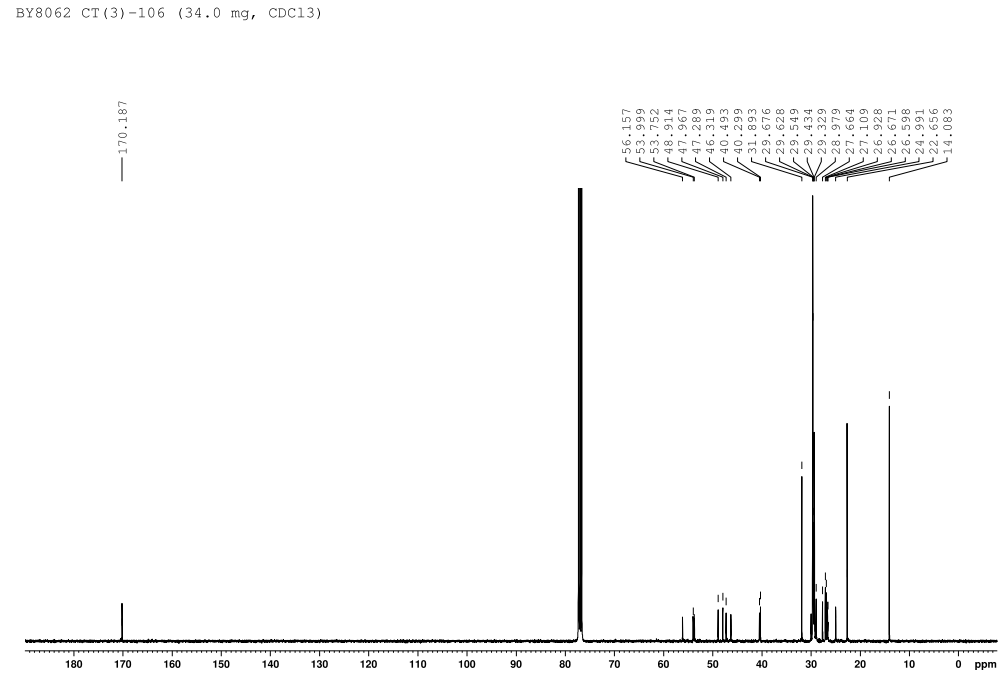


Fig. S99 ^{13}C NMR Spectrum (100 MHz, $CDCl_3$) of lipid **ST12**

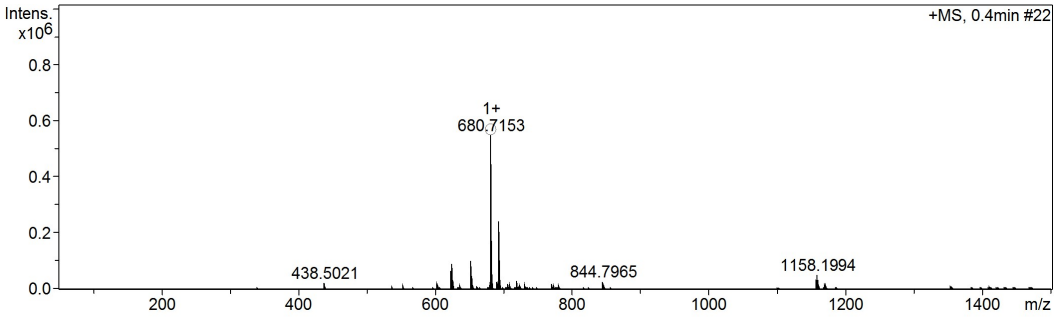
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\Data\Boon-ek\ESI\BY-HRMS 1517 (pos).d	Acquisition Date	12/22/2020 3:01:52 PM
Method	tune_wide-RU.m	Operator	RU
Sample Name	CT(3)-106	Instrument	micrOTOF 8213750.10411
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.3 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	4000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



Meas. m/z	#	Ion Formula	m/z	|err| [mDa]	err [ppm]	Mean err [ppm]	rdb	N-Rule	e⁻ Conf	mSigma
680.715343	1	C42H90N5O	680.713989	1.4	-2.0	-0.7	0.5	ok	even	19.0

Fig. S100 HRMS Spectrum of lipid **ST12**

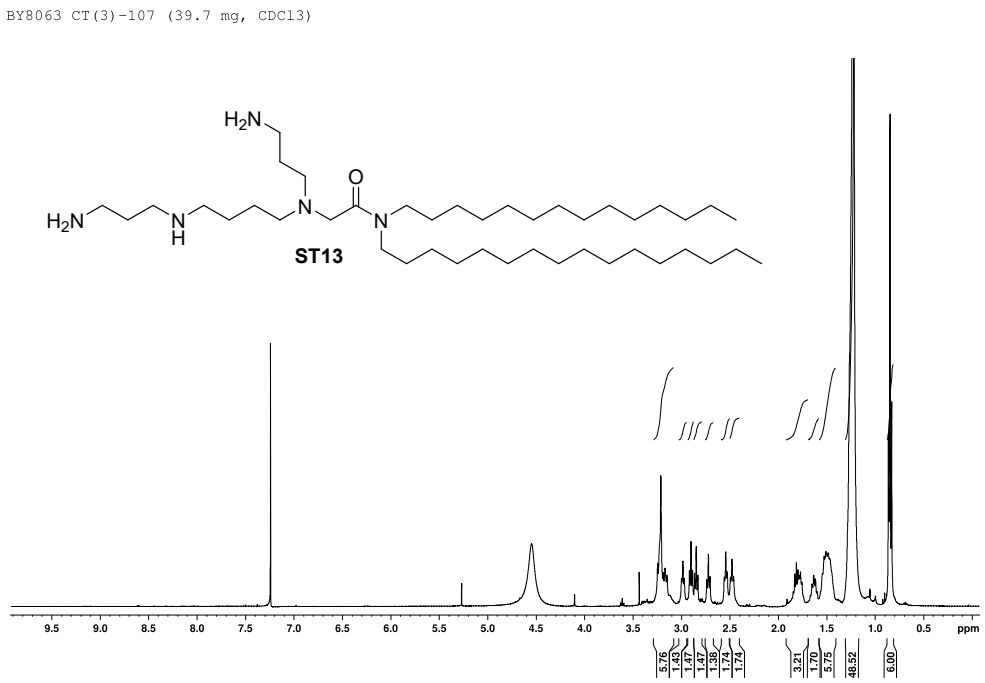


Fig. S101 ^{1}H NMR Spectrum (400 MHz, $CDCl_3$) of lipid **ST13**

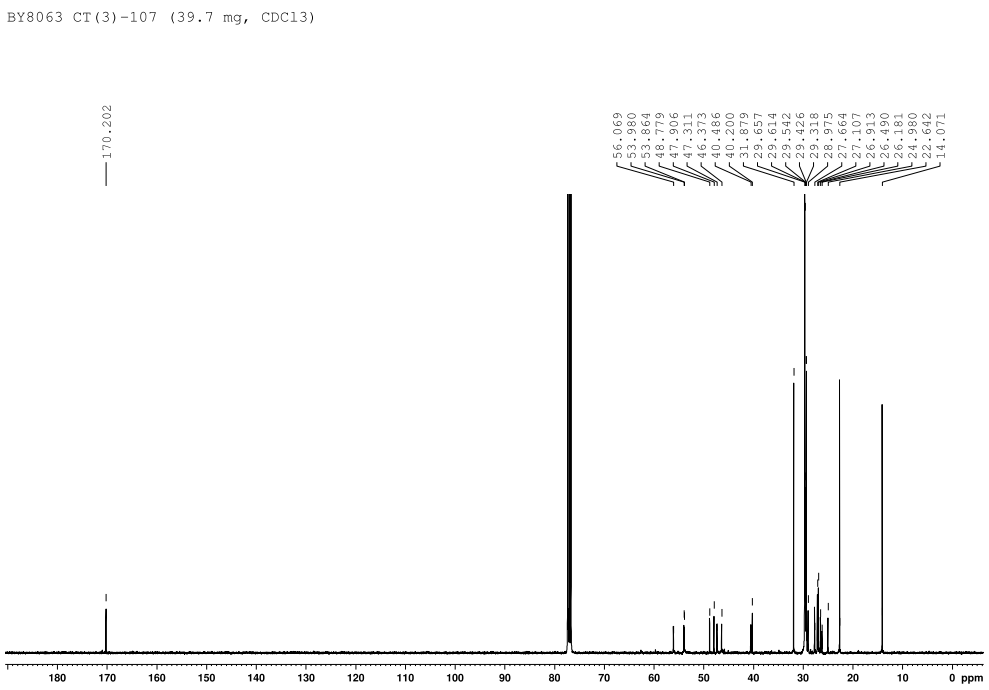


Fig. S102 ^{13}C NMR Spectrum (100 MHz, $CDCl_3$) of lipid **ST13**

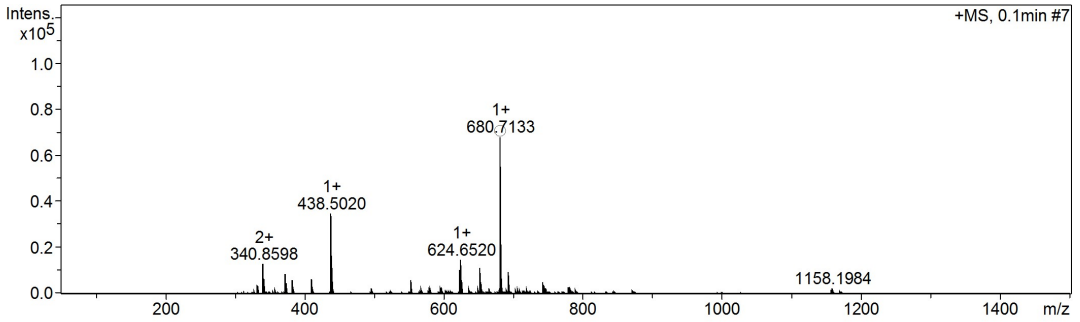
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\Data\Boon-ek\ESI\BY-HRMS 1518 (pos).d	Acquisition Date	12/22/2020 3:03:20 PM
Method	tune_wide-RU.m	Operator	RU
Sample Name	CT(3)-107	Instrument	micrOTOF 8213750.10411
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.3 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	4000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



Meas. m/z	#	Ion Formula	m/z	|err| [mDa]	err [ppm]	Mean err [ppm]	rdb	N-Rule	e⁻ Conf	mSigma
680.713282	1	C42H90N5O	680.713989	0.7	1.0	1.7	0.5	ok	even	8.5

Fig. S103 HRMS Spectrum of lipid **ST13**

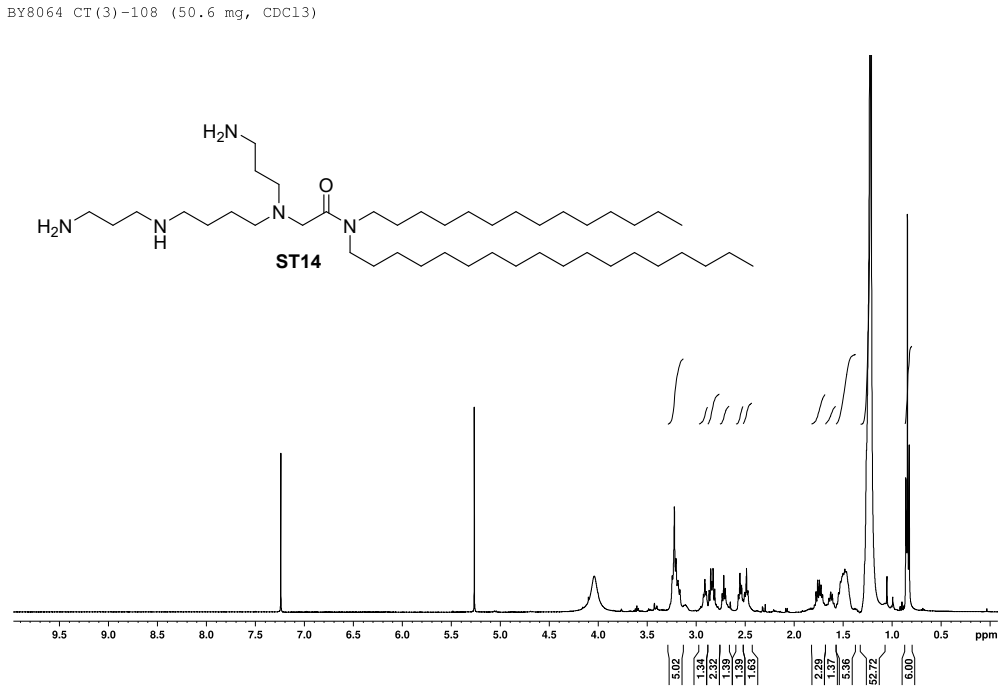


Fig. S104 ^{1}H NMR Spectrum (400 MHz, $CDCl_3$) of lipid **ST14**

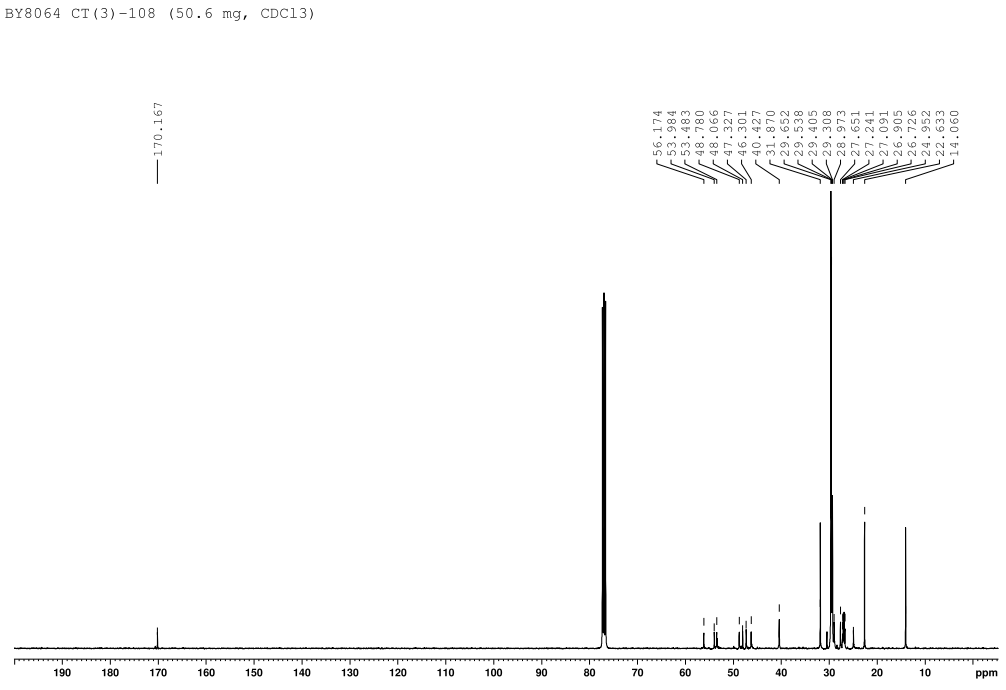


Fig. S105 ^{13}C NMR Spectrum (100 MHz, $CDCl_3$) of lipid **ST14**

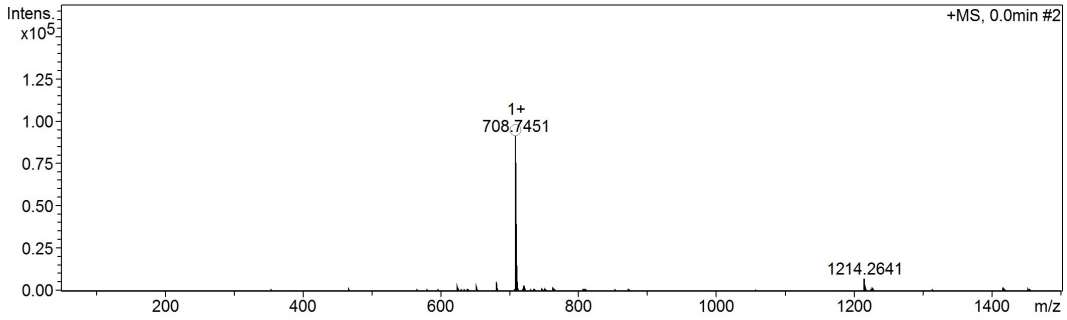
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\Data\Boon-ek\ESI\BY-HRMS 1519 (pos).d	Acquisition Date	12/22/2020 3:04:20 PM
Method	tune_wide-RU.m	Operator	RU
Sample Name	CT(3)-108	Instrument	micrOTOF 8213750.10411
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.3 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	4000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



Meas. m/z	#	Ion Formula	m/z	|err| [mDa]	err [ppm]	Mean err [ppm]	rdb	N-Rule	e⁻ Conf	mSigma
708.745142	1	C44H94N5O	708.745289	0.1	-0.2	0.4	0.5	ok	even	10.2

Fig. S106 HRMS Spectrum of lipid **ST14**

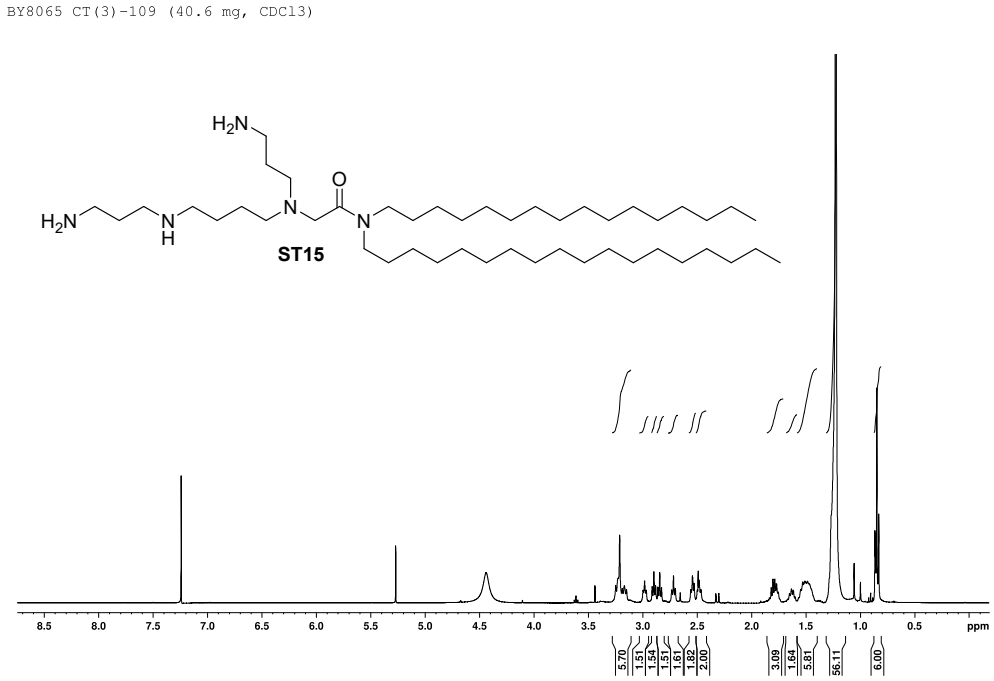


Fig. S107 ^{1}H NMR Spectrum (400 MHz, $CDCl_3$) of lipid **ST15**

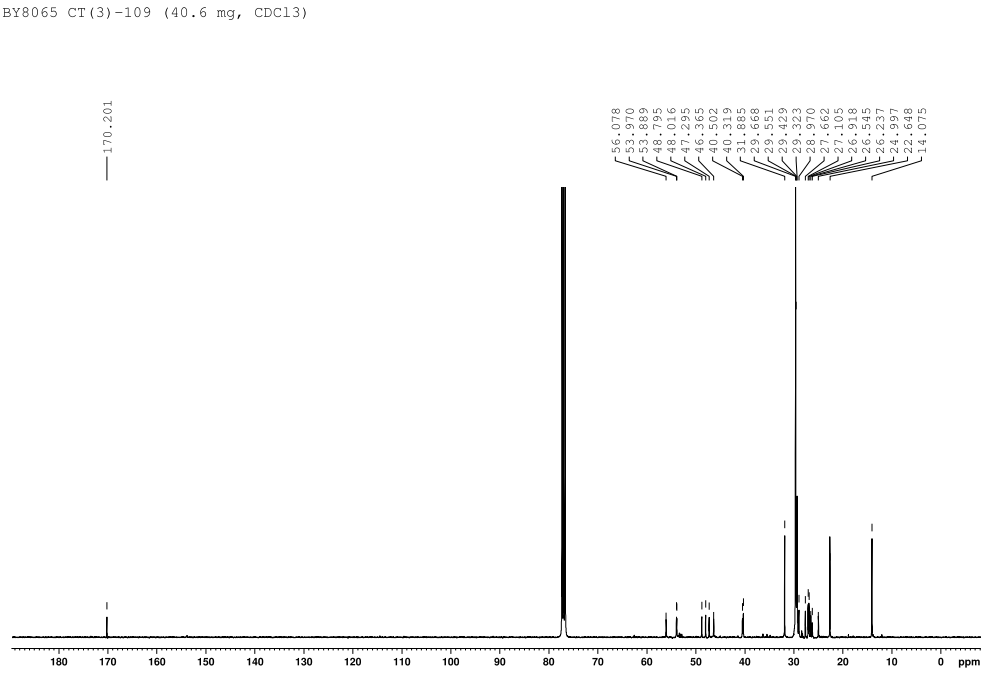


Fig. S108 ^{13}C NMR Spectrum (100 MHz, $CDCl_3$) of lipid **ST15**

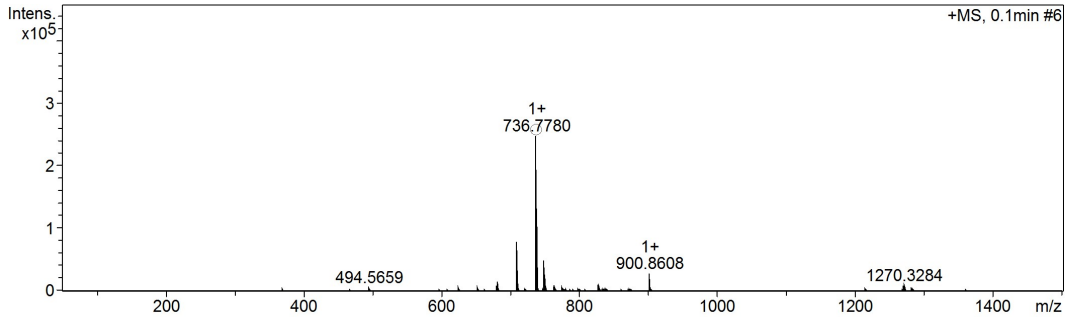
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\Data\Boon-ek\ESI\BY-HRMS 1520 (pos).d	Acquisition Date	12/22/2020 3:05:21 PM
Method	tune_wide-RU.m	Operator	RU
Sample Name	CT(3)-109	Instrument	micrOTOF 8213750.10411
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.3 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	4000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



Meas. m/z	#	Ion Formula	m/z	|err| [mDa]	err [ppm]	Mean err [ppm]	rdb	N-Rule	e⁻ Conf	mSigma
736.778032	1	C46H98N5O	736.776589	1.4	-2.0	-1.0	0.5	ok	even	5.3

Fig. S109 HRMS Spectrum of lipid **ST15**