Experimental Section

1 Reagents and general methods

All reagents were purchased from commercial suppliers and were dried and purified when necessary, and β-carboline-3-carboxylic acids **1-13** were synthesized according to our previously published methods [23, 24, 28].

Melting points were determined in capillary tubes on an electrothermal PIF YRT-3 apparatus and without correction. MS spectra were obtained from VG ZAB-HS spectrometer. ^{1}H NMR and ^{13}C NMR spectra were recorded on a Mercury-Plus 300 spectrometer at 300 MHz and 75 MHz, respectively, using TMS as internal standard and $CDCl_3$ as solvent and chemical shifts (δ) were expressed in ppm. HRMS were obtained from ESI-Q-TOF maxis 4G spectrometer. Silica gel F254 were used in analytical thin-layer chromatography (TLC) and silica gel were used in column chromatography respectively.

2 General procedure for the preparation of bivalent β-carbolines 14-44.

A mixture of β-carboline-3-carboxylic acids **1-13** (2.1mmol), anhydrous K_2CO_3 (3.0mmol), anhydrous DMF (50ml) and appropriate dibromoalkane (1.0mmol) was stirred at 80 ℃ for 6-10 h. After completion of the reaction as indicated by TLC, the solution was poured into ice-water and extracted with CH_2Cl_2. The combined organic layer was washed with water and brine, dried over anhydrous Na_2SO_4, filtered and evaporated in vacuum. The residue was purified by column chromatography with CH_2Cl_2/MeOH (50:1) to successfully afford the desirable target products.

2.1 ***1,3-Bis(1,9-dimethyl-β-carboline-3-carboxylic acid)propanediyl ester*****（14）**

Starting from 1,9-dimethyl-β-carboline-3-carboxyl acid (0.51g, 2.1mmol) and 1,3-dibromopropane (1.0mmol). White solid was obtained (0.36g, 69％), mp 186-188℃. ESI-MS m/z: 521.3 (100) $[M+H]^+$. ^{1}H NMR (300MHz, $CDCl_3$): δ 8.36 (2H, s, ArH), 7.83 (2H, d, J=7.8Hz, , ArH), 7.52-7.57 (2H, m, , ArH), 7.20-7.29 (4H, m, ArH), 4.71 (4H, t, J=5.7Hz, C***H***$_2$CH$_2$C***H***$_2$), 3.80 (6H, s, NC***H***$_3$), 2.98 (6H, s, C***H***$_3$), 2.40-2.47 (2H, m, CH$_2$C***H***$_2$CH$_2$); ^{13}C NMR (75 MHz, $CDCl_3$): δ 165.8, 141.7, 136.7, 136.1, 128.2, 127.7, 121.3, 120.8, 120.1, 116.2, 109.8, 63.7, 32.0, 28.7, 24.1. HRMS (ESI) calcd for $C_{31}H_{29}N_4O_4$ 521.2183 $[M+H]^+$, found 521.2185.

2.2 ***1,6-Bis(1,9-dimethyl-β-carboline-3-carboxyl)hexanediyl ester*****（15）**

Starting from 1,9-dimethyl-β-carboline-3-carboxyl acid (0.51g, 2.1mmol) and 1,6-dibromohexane (1.0mmol). White solid was obtained (0.42g, 75％), mp 161-163℃. ESI-MS m/z: 563.4 (100)

$[M+H]^+$. ^1H NMR (300 MHz, $CDCl_3$): δ 8.60 (2H, s, ArH), 8.08-8.11 (2H, m, ArH), 7.58-7.65 (2H, m, ArH), 7.44 (2H, d, *J*=8.4Hz, ArH), 7.28-7.34 (2H, m, ArH), 4.48 (4H, t, *J*=6.6Hz, C***H***$_2$[CH_2]$_4$C***H***$_2$), 4.12 (6H, s, NC***H***$_3$), 3.14(6H, s, C***H***$_3$), 1.90-1.97 (4H, m, CH_2C***H***$_2$[CH_2]$_2$C***H***$_2$$CH_2$), 1.62-1.67 (4H, m, CH_2CH_2[C***H***$_2$]$_2$$CH_2CH_2$); ^{13}C NMR (75 MHz, $CDCl_3$): δ 166.2, 142.4, 141.9, 137.3, 136.9, 128.6, 121.7, 121.5, 120.7, 116.2, 110.0, 109.9, 65.7, 32.6, 29.1, 26.3, 24.4. HRMS (ESI) calcd for $C_{34}H_{35}N_4O_4$ 563.2653 $[M+H]^+$, found 563.2668.

2.3 ***1,10- Bis(1,9-dimethyl-β-carboline-3-carboxyl) decanediyl ester*****（16）**

Starting from 1,9-dimethyl-β-carboline-3-carboxyl acid (0.51g, 2.1mmol) and 1,10-dibromodecane (1.0mmol). Yellow solid was obtained (0.47g, 76％), mp 152-154℃. ESI-MS m/z: 619.4 (100) $[M+H]^+$. ^1H NMR (300 MHz, $CDCl_3$): δ 8.68 (2H, s, ArH), 8.14 (2H, d, *J*=7.5Hz, ArH), 7.59-7.64 (2H, m, ArH), 7.45-7.50 (2H, m, ArH), 7.30-7.35 (2H, m, ArH), 4.43 (4H, t, *J*=6.9Hz, C***H***$_2$[CH_2]$_8$C***H***$_2$), 4.18 (6H, s, NC***H***$_3$), 3.17 (6H, s, NC***H***$_3$), 1.83-1.92 (4H, m, CH_2C***H***$_2$[CH_2]$_6$C***H***$_2$$CH_2$), 1.32-1.51 (8H, m, CH_2CH_2C***H***$_2$C***H***$_2$[CH_2]$_2$C***H***$_2$C***H***$_2$$CH_2CH_2$), 0.84-0.91 (4H, m, $CH_2CH_2CH_2CH_2$ [C***H***$_2$]$_2$$CH_2CH_2CH_2CH_2$); ^{13}C NMR (75 MHz, $CDCl_3$): δ 166.2, 142.5, 142.0, 137.4, 137.0, 128.7, 121.7, 121.6, 120.7, 116.3, 110.0, 65.9, 32.6, 29.8, 29.7, 29.2, 26.4, 24.4. HRMS (ESI) calcd for $C_{38}H_{43}N_4O_4$ 619.3279 $[M+H]^+$, found 619.3293.

2.4 ***1,6-Bis(9-ethyl-1-methyl-β-carboline-3-carboxylic acid)hexanediyl ester*** **(17)**

Starting from 9-ethyl-1-methyl-β-carboline-3-carboxyl acid (0.53g, 2.1mmol) and 1,6-dibromohexane (1.0mmol). White solid was obtained (0.38g, 64％), mp 142-144℃. ESI-MS m/z: 591.3 (100) $[M+H]^+$. ^1H NMR (300 MHz, $CDCl_3$): δ 8.68 (2H, s, ArH), 8.14 (2H, d, *J*=7.8Hz, ArH), 7.57-7.62 (2H, m, ArH), 7.47 (2H, d, *J*=8.1Hz, ArH), 7.28-7.33 (2H, m, ArH), 4.62 (4H, q, *J*=7.2Hz, NC***H***$_2$$CH_3$), 4.47 (4H, t, *J*=6.9Hz, C***H***$_2$[CH_2]$_4$C***H***$_2$), 3.11 (6H, s, C***H***$_3$), 1.89-1.98 (4H, m, CH_2C***H***$_2$[CH_2]$_2$C***H***$_2$$CH_2$), 1.60-1.64 (4H, m, CH_2CH_2[C***H***$_2$]$_2$$CH_2CH_2$), 1.47 (6H, t, *J*=7.2Hz, NCH_2C***H***$_3$); ^{13}C NMR (75 MHz, $CDCl_3$): δ 166.3, 141.5, 141.4, 136.8, 136.5, 128.9, 128.7, 121.8, 120.7, 116.4, 110.0, 65.7, 40.0, 29.1, 26.3, 24.2, 16.1. HRMS (ESI) calcd for $C_{36}H_{39}N_4O_4$ 591.2966 $[M+H]^+$, found 591.2980.

2.5 ***1,8-Bis(9-ethyl-1-methyl-β-carboline-3-carboxyl)octanediyl ester*** **(18)**

Starting from 9-ethyl-1-methyl-β-carboline-3-carboxyl acid (0.53g, 2.1mmol) and 1,8-dibromooctane (1.0mmol). Yellow oil was obtained (0.44g,72％). ESI-MS m/z: 619.3 (100) $[M+H]^+$. ^1H NMR (300 MHz, $CDCl_3$): δ 8.69 (2H, s, ArH), 8.15 (2H, d, *J*=7.8Hz, ArH), 7.57-7.62

(2H, m, ArH), 4.47(2H, d, *J*=8.4Hz, ArH), 7.29-7.34 (2H, m, ArH), 4.63 (4H, q, J=7.2Hz, C***H***$_2$[CH$_2$]$_6$C***H***$_2$), 4.45 (4H, t, *J*=6.9Hz, NC***H***$_2$CH$_3$), 3.12 (6H, s, C***H***$_3$), 1.87-1.93 (4H, m, CH$_2$C***H***$_2$[CH$_2$]$_4$C***H***$_2$CH$_2$), 1.45-1.59 (14H, m, CH$_2$CH$_2$[C***H***$_2$]$_4$CH$_2$CH$_2$, NCH$_2$C***H***$_3$); ^{13}C NMR (75 MHz, CDCl$_3$): δ 166.2, 141.4, 141.3, 136.7, 136.3, 128.8, 128.6, 121.7, 120.6, 116.2, 109.9, 65.8, 39.9, 29.6, 29.2, 26.3, 24.1, 16.2. HRMS (ESI) calcd for $C_{38}H_{43}N_4O_4$ 619.3279 $[M+H]^+$, found 619.3307.

2.6 ***1,9-Bis(9-ethyl-1-methyl-β-carboline-3-carboxylic acid)nonanediyl ester*** **(19)**

Starting from 9-ethyl-1-methyl-β-carboline-3-carboxyl acid (0.53g, 2.1mmol) and 1,9-dibromononane (1.0mmol). Yellow oil was obtained (0.4g, 63％). ESI-MS m/z: 633.5 (100) $[M+H]^+$. ^{1}H NMR (300 MHz, CDCl$_3$): δ 8.69 (2H, s, ArH), 8.14-8.16 (2H, m, ArH), 7.56-7.62 (2H, m, ArH), 7.47 (2H, d, *J*=8.4Hz, ArH), 7.28-7.33 (2H, m, ArH), 4.59-4.66 (4H, m, C***H***$_2$[CH$_2$]$_7$C***H***$_2$), 4.44 (4H, t, *J*=6.9Hz, NC***H***$_2$CH$_3$), 3.12 (6H, s, C***H***$_3$), 1.83-1.92 (4H, m, CH$_2$C***H***$_2$[CH$_2$]$_5$C***H***$_2$CH$_2$), 1.34-1.50 (16H, m, CH$_2$CH$_2$[C***H***$_2$]$_5$CH$_2$CH$_2$, NCH$_2$C***H***$_3$); ^{13}C NMR (75 MHz, CDCl$_3$): δ 166.1, 141.4(2C), 136.7, 136.3, 128.8, 128.6, 121.6(2C), 120.6, 116.1, 109.9, 65.7, 39.8, 29.8, 29.6, 29.2, 26.3, 24.0, 16.1. HRMS (ESI) calcd for $C_{39}H_{45}N_4O_4$ 633.3435 $[M+H]^+$, found 633.3479.

2.7 ***1,4-Bis(9-butyl-β-carboline-3-carboxylic acid)butanediyl ester*** **(20)**

Starting from 9-butyl-β-carboline-3-carboxyl acid (0.56g, 2.1mmol) and 1,4-dibromobutane (1.0mmol). White solid was obtained (0.32g, 54％), mp 168-169℃. FAB-MS m/z 591 (100) $[M+H]^+$; ^{1}H-NMR (300MHz, CDCl$_3$): δ 8.95 (2H, s, ArH); 8.86 (2H, s, ArH); 8.20 (2H, d, *J*=7.8Hz, ArH); 7.60-7.65 (2H, m, ArH); 7.50 (2H, d, *J*=8.4Hz, ArH); 7.32-7.36 (2H, m, ArH); 4.59 (4H, t, *J*=5.4Hz, C***H***$_2$[CH$_2$]$_2$C***H***$_2$); 4.42 (4H, t, *J*=6.9Hz, NC***H***$_2$CH$_2$CH$_2$CH$_3$); 2.11-2.15 (4H, m, CH$_2$[C***H***$_2$]$_2$CH$_2$); 1.85-1.95 (4H, m, NCH$_2$C***H***$_2$CH$_2$CH$_3$); 1.32-1.45 (4H, m, NCH$_2$CH$_2$C***H***$_2$CH$_3$); 0.94 (6H, t, *J*=7.5Hz, NCH$_2$CH$_2$CH$_2$C***H***$_3$). ^{13}C NMR (75 MHz, CDCl$_3$): δ 166.3, 141.7, 138.0, 137.5, 131.9, 128.9, 128.4, 122.3, 121.6, 120.7, 117.9, 110.0, 65.4, 43.7, 31.6, 26.1, 20.8, 14.1. HRMS (ESI) calcd for $C_{36}H_{39}N_4O_4$ 591.2966 $[M+H]^+$, found 591.2982.

2.8 ***1,5-Bis(9-butyl-β-carboline-3-carboxylic acid)pentanediyl ester*** **(21)**

Starting from 9-butyl-β-carboline-3-carboxyl acid (0.56g, 2.1mmol) and 1,5-dibromopentane (1.0mmol). White solid was obtained (0.46g, 77％), mp 149-151℃. ESI-MS m/z: 605.4 (100) $[M+H]^+$. ^{1}H NMR (300 MHz, CDCl$_3$): δ 8.90 (2H, s, ArH), 8.82 (2H, s, ArH), 8.13 (2H, d,

J=7.8Hz, ArH), 7.57-7.62 (2H, m, ArH), 7.46 (2H, d, *J*=7.8Hz, ArH), 7.27-7.32 (2H, m, ArH), 4.51(4H, t, *J*=6.9Hz, C***H***$_2$[CH$_2$]$_3$C***H***$_2$), 4.36(4H, t, *J*=7.2Hz, NC***H***$_2$CH$_2$CH$_2$CH$_3$), 1.95-2.05 (4H, m, CH$_2$C***H***$_2$CH$_2$C***H***$_2$CH$_2$), 1.81-1.91 (4H, m, NCH$_2$C***H***$_2$CH$_2$CH$_3$), 1.68-1.75 (2H, m, CH$_2$CH$_2$C***H***$_2$CH$_2$CH$_2$), 1.29-1.41 (4H, m, NCH$_2$CH$_2$C***H***$_2$CH$_3$), 0.92 (6H, t, *J*=7.2Hz, NCH$_2$CH$_2$CH$_2$C***H***$_3$); ^{13}C NMR (75 MHz, CDCl$_3$): δ 165.9, 141.1, 137.4, 137.1, 131.6, 128.6, 127.8, 121.8, 121.0, 120.4, 117.5, 109.8, 65.3, 43.2, 31.2, 28.8, 22.8, 20.5, 13.9. HRMS (ESI) calcd for C$_{37}$H$_{41}$N$_4$O$_4$ 605.3122 [M+H]$^+$, found 605.3146.

2.9 ***1,6-Bis(9-butyl-β-carboline-3-carboxylic acid)hexanediyl ester*** **(22)**

Starting from 9-butyl-β-carboline-3-carboxyl acid (0.56g, 2.1mmol) and 1,6-dibromohexane (1.0mmol). White solid was obtained (0.48g, 78%), mp 156-157℃; FAB-MS m/z 619 (100) [M+H]$^+$; ^{1}H-NMR (300MHz, CDCl$_3$): δ 8.93 (2H, s, ArH); 8.84 (2H, s, ArH); 8.18 (2H, d, *J*=7.8Hz, ArH); 7.59-7.64 (2H, m, ArH); 7.48 (2H, d, *J*=8.1Hz, ArH); 7.31-7.36 (2H, m, ArH); 4.49 (4H, t, *J*=6.9Hz, C***H***$_2$[CH$_2$]$_4$C***H***$_2$); 4.40 (4H, t, *J*=6.9Hz, NC***H***$_2$CH$_2$CH$_2$CH$_3$); 1.84-1.97 (8H, m, CH$_2$C***H***$_2$[CH$_2$]$_2$C***H***$_2$CH$_2$, NCH$_2$C***H***$_2$CH$_2$CH$_3$); 1.60-1.64 (4H, m, CH$_2$CH$_2$[C***H***$_2$]$_2$CH$_2$CH$_2$); 1.31-1.44 (4H, m, NCH$_2$CH$_2$C***H***$_2$CH$_3$); 0.94 (6H, t, *J*=7.5Hz, NCH$_2$CH$_2$CH$_2$C***H***$_3$); ^{13}C NMR (75 MHz, CDCl$_3$): δ 166.3, 141.7, 137.9, 137.7, 131.9, 128.9, 128.4, 122.3, 121.5, 120.7, 117.8, 110.1, 65.8, 43.7, 31.6, 29.2, 26.2, 20.8, 14.1. HRMS (ESI) calcd for C$_{38}$H$_{43}$N$_4$O$_4$ 619.3279 [M+H]$^+$, found 619.3302.

2.10 ***1,8-Bis(9-butyl-β-carboline-3-carboxylic acid)octanediyl ester*** **(23)**

Starting from 9-butyl-β-carboline-3-carboxyl acid (0.56g, 2.1mmol) and 1,8-dibromooctane (1.0mmol). White solid was obtained (0.36g, 56%), mp 142-144℃. FAB-MS m/z 647 (100) [M+H]$^+$; ^{1}H-NMR (300 MHz, CDCl$_3$): δ 8.94 (2H, s, ArH); 8.85 (2H, s, ArH); 8.20 (2H, d, *J*=7.8Hz, ArH); 7.60-7.65 (2H, m, ArH); 7.50 (2H, d, *J*=8.1Hz, ArH); 7.31-7.36 (2H, m, ArH); 4.47 (4H, t, *J*=6.9Hz, C***H***$_2$[CH$_2$]$_6$C***H***$_2$); 4.42 (4H, t, *J*=7.2Hz, NC***H***$_2$CH$_2$CH$_2$CH$_3$); 1.85-1.92 (8H, m, CH$_2$C***H***$_2$[CH$_2$]$_4$C***H***$_2$CH$_2$, NCH$_2$C***H***$_2$CH$_2$CH$_3$); 1.34-1.51 (12H, m, CH$_2$CH$_2$[C***H***$_2$]$_4$CH$_2$CH$_2$, NCH$_2$CH$_2$C***H***$_2$CH$_3$); 0.94 (6H, t, *J*=7.2Hz, NCH$_2$CH$_2$CH$_2$C***H***$_3$); ^{13}C NMR (75 MHz, CDCl$_3$): δ 166.3, 141.7, 137.9, 137.7, 131.9, 128.9, 128.4, 122.3, 121.5, 120.7, 117.8, 110.1, 65.9, 43.7, 31.6, 29.6, 29.2, 26.3, 20.8, 14.1. HRMS (ESI) calcd for C$_{40}$H$_{47}$N$_4$O$_4$ 647.3592 [M+H]$^+$, found 647.3627.

2.11 ***1,9-Bis(9-butyl-β-carboline-3-carboxylic acid)nonanediyl ester*** **(24)**

Starting from 9-butyl-β-carboline-3-carboxyl acid (0.56g, 2.1mmol) and 1,9-dibromononane (1.0mmol). Yellow oil was obtained (0.52g, 79%). ESI-MS m/z: 661.3 (100) $[M+H]^+$. ^1H NMR (300 MHz, $CDCl_3$): δ 8.93 (2H, s, ArH), 8.85 (2H, s, ArH)；8.19 (2H, d, *J*=7.8Hz, ArH); 7.58-7.64 (2H, m, ArH); 7.49 (2H, d, *J*=8.4Hz, ArH); 7.30-7.36 (2H, m, ArH); 4.39-4.48 (8H, m, $CH_2[CH_2]_7CH_2$, $NCH_2CH_2CH_2CH_3$); 1.84-2.04 (8H, m, $CH_2CH_2[CH_2]_5CH_2CH_2$, $NCH_2CH_2CH_2CH_3$); 1.31-1.51 (14H, m, $CH_2CH_2[CH_2]_5CH_2CH_2$, $NCH_2CH_2CH_2CH_3$); 0.93 (6H, t, *J*=7.2Hz, $NCH_2CH_2CH_2CH_3$); ^{13}C NMR (75 MHz, $CDCl_3$): δ 166.1, 141.3, 137.5, 137.4, 131.6, 128.7, 128.0, 121.9, 121.2, 120.5, 117.5, 109.9, 65.8, 43.3, 31.4, 29.7, 29.5, 29.1, 26.3, 20.6, 14.0. HRMS (ESI) calcd for $C_{41}H_{49}N_4O_4$ 661.3748 $[M+H]^+$, found 661.3789.

2.12 ***1,10-Bis(9-butyl-β-carboline-3-carboxylic acid)decanediyl ester*** **(25)**

Starting from 9-butyl-β-carboline-3-carboxyl acid (0.56g, 2.1mmol) and 1,10-dibromodecane (1.0mmol). White solid was obtained (0.50g, 75%), mp 156-157℃. FAB-MS m/z 675 (100) $[M+H]^+$；^1H-NMR (300 MHz, $CDCl_3$): δ 8.92 (2H, s, ArH); 8.83 (2H, s, ArH); 8.17 (2H, d, *J*=7.8Hz, ArH); 7.57-7.63 (2H, m, ArH); 7.47 (2H, d, *J*=8.4Hz, ArH); 7.30-7.35 (2H, m, ArH); 4.45 (4H, t, *J*=6.9Hz, $CH_2[CH_2]_8CH_2$); 4.39 (4H, t, *J*=6.9Hz, $NCH_2CH_2CH_2CH_3$); 1.83-1.93 (8H, m, $CH_2CH_2[CH_2]_6CH_2CH_2$, $NCH_2CH_2CH_2CH_3$); 1.31-1.50 (16H, m, $CH_2CH_2[CH_2]_6CH_2CH_2$, $NCH_2CH_2CH_2CH_3$); 0.93 (6H, t, *J*=7.2Hz, $NCH_2CH_2CH_2CH_3$). ^{13}C NMR (75 MHz, $CDCl_3$): δ 166.3, 141.6, 137.8, 137.7, 131.8, 128.9, 128.3, 122.2, 121.5, 120.7, 117.7, 110.1, 65.9, 43.6, 31.6, 29.8, 29.7, 29.2, 26.3, 20.8, 14.1. HRMS (ESI) calcd for $C_{42}H_{51}N_4O_4$ 675.3905 $[M+H]^+$, found 675.3946.

2.13 ***1,6-Bis(9-butyl-1-methyl-β-carboline-3-carboxyl)hexanediyl ester*** **(26)**

Starting from 9-butyl-1-methyl-β-carboline-3-carboxyl acid (0.57g, 2.1mmol) and 1,6-dibromohexane (1.0mmol). White solid was obtained (0.41g, 63%), mp 146-148℃. ESI-MS m/z: 647.4 (100) $[M+H]^+$. ^1H NMR (300 MHz, $CDCl_3$): δ 8.67 (2H, s, ArH), 8.12 (2H, d, *J*=7.8Hz, ArH), 7.55-7.60 (2H, m, ArH), 7.44 (2H, d, *J*=8.4Hz, ArH), 7.26-7.32 (2H, m, ArH), 4.45-4.51 (8H, m, $CH_2[CH_2]_4CH_2$, $NCH_2CH_2CH_2CH_3$), 3.09 (6H, s, CH_3), 1.89-1.97 (4H, m, $CH_2CH_2[CH_2]_2CH_2CH_2$), 1.79-1.84 (4H, m, $NCH_2CH_2CH_2CH_3$), 1.59-1.64 (4H, m, $CH_2CH_2[CH_2]_2CH_2CH_2$), 1.37-1.51 (4H, m, $NCH_2CH_2CH_2CH_3$), 0.97 (6H, t, *J*=7.2Hz, $NCH_2CH_2CH_2CH_3$); ^{13}C NMR (75 MHz, $CDCl_3$): δ 166.0, 141.5, 141.2, 136.5, 136.2, 128.4, 121.4, 121.3, 120.3, 116.0, 115.9, 110.0, 65.4, 44.7, 33.0, 29.0, 26.1, 24.1, 20.2, 14.0. HRMS (ESI)

calcd for $C_{40}H_{47}N_4O_4$ 647.3592 $[M+H]^+$, found 647.3635.

2.14 ***1,7-Bis(9-butyl-1-methyl-β-carboline-3-carboxylic acid)heptanediyl ester*** **(27)**

Starting from 9-butyl-1-methyl-β-carboline-3-carboxyl acid (0.57g, 2.1mmol) and 1,7-dibromoheptane (1.0mmol). Yellow oil was obtained (0.38g, 58%). ESI-MS m/z: 661.3 (100) $[M+H]^+$. ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.69 (2H, s, ArH), 8.11 (2H, d, *J*=7.8Hz, ArH), 7.53-7.58 (2H, m, ArH), 7.43 (2H, d, *J*=8.1Hz, ArH), 7.27-7.32 (2H, m, ArH), 4.42-4.56 (8H, m, C***H***$_2$[CH_2]$_5$C***H***$_2$, NC***H***$_2$$CH_2CH_2CH_3$), 3.10 (6H, s, C***H***$_3$), 1.78-1.92 (8H, m, CH_2C***H***$_2$[CH_2]$_3$C***H***$_2$$CH_2$, NCH_2C***H***$_2$$CH_2CH_3$), 1.36-1.53 (10H, m, CH_2CH_2[C***H***$_2$]$_3$$CH_2CH_2$, NCH_2CH_2C***H***$_2$$CH_3$), 0.96 (6H, t, *J*=7.5Hz, $NCH_2CH_2CH_2$C***H***$_3$); ^{13}C NMR (75 MHz, $CDCl_3$): δ 166.2, 141.8, 141.4, 136.7, 136.6, 128.8, 128.5, 121.6, 120.6, 116.2, 116.1, 110.2, 65.7, 45.0, 33.2, 29.4, 29.1, 26.3, 24.2, 20.4, 14.2. HRMS (ESI) calcd for $C_{41}H_{49}N_4O_4$ 661.3748 $[M+H]^+$, found 661.3802.

2.15 ***1,10-Bis(9-butyl-1-methyl-β-carboline-3-carboxylic acid)decanediyl ester*** **(28)**

Starting from 9-butyl-1-methyl-β-carboline-3-carboxyl acid (0.57g, 2.1mmol) and 1,10-dibromodecane (1.0mmol). Yellow oil was obtained (0.42g, 60%). ESI-MS m/z: 703.4 (100) $[M+H]^+$. ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.69 (2H, s, ArH), 8.14 (2H, d, *J*=7.8Hz, ArH), 7.55-7.61 (2H, m, ArH), 7.45(2H, d, *J*=8.4Hz, ArH), 7.28-7.33 (2H, m, ArH), 4.54 (4H, t, *J*=7.2Hz, C***H***$_2$[CH_2]$_8$C***H***$_2$), 4.43 (4H, t, *J*=6.9Hz, NC***H***$_2$$CH_2CH_2CH_3$), 3.10 (6H, s, C***H***$_3$), 1.77-1.91 (8H, m, CH_2C***H***$_2$[CH_2]$_6$C***H***$_2$$CH_2$, NCH_2C***H***$_2$$CH_2CH_3$), 1.31-1.53 (16H, m, CH_2CH_2[C***H***$_2$]$_6$$CH_2CH_2$, NCH_2CH_2C***H***$_2$$CH_3$), 0.97 (6H, t, *J*=7.2Hz, $NCH_2CH_2CH_2$C***H***$_3$); ^{13}C NMR (75 MHz, $CDCl_3$): δ 166.2, 141.9, 141.5, 136.9, 136.7, 128.9, 128.6, 121.7, 120.6, 116.3, 116.2, 110.3, 65.8, 45.2, 33.3, 30.1, 29.9, 29.2, 26.4, 24.2, 20.5, 14.3. HRMS (ESI) calcd for $C_{44}H_{55}N_4O_4$ 703.4218 $[M+H]^+$, found 703.4225.

2.16 ***1,4-Bis(9-benzyl-β-carboline-3-carboxylic acid)butanediyl ester*** **(29)**

Starting from 9-benzyl-β-carboline-3-carboxyl acid (0.66g, 2.1mmol) and 1,4-dibromobutane (1.0mmol). White solid was obtained (0.58g, 88%), mp 210-212℃. ESI-MS m/z: 659.3 (100) $[M+H]^+$. ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.89 (2H, s, ArH), 8.88 (2H, s, ArH), 8.22 (2H, d, *J*=7.8Hz, ArH), 7.57-7.63 (2H, m, ArH), 7.48 (2H, d, *J*=8.4Hz, ArH), 7.33-7.38 (2H, m, ArH), 7.24-7.27 (6H, m, ArH), 7.12-7.15 (4H, m, ArH), 5.61(4H, s, C***H***$_2$Ph), 4.56 (4H, t, *J*=5.4Hz, C***H***$_2$[CH_2]$_2$C***H***$_2$), 2.08-2.12 (4H, m, CH_2[C***H***$_2$]$_2$$CH_2$); ^{13}C NMR (75 MHz, $CDCl_3$): δ 165.9, 141.9,

138.0, 137.7, 135.8, 132.0, 129.2, 129.1, 128.1, 126.6, 122.3, 121.6, 121.0, 117.8, 110.2, 65.2, 47.3, 25.7. HRMS (ESI) calcd for $C_{42}H_{35}N_4O_4$ 659.2653 $[M+H]^+$, found 659.2664.

2.17 ***1,6-Bis(9-benzyl-β-carboline-3-carboxylic acid)hexanediyl ester*** **(30)**

Starting from 9-benzyl-β-carboline-3-carboxyl acid (0.66g, 2.1mmol) and 1,6-dibromohexane (1.0mmol). White solid was obtained (0.54g, 79%), mp 194-196℃. ESI-MS m/z: 687.3 (100) $[M+H]^+$. ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.89 (2H, s, ArH), 8.87 (2H, s, ArH), 8.22 (2H, d, *J*=7.8Hz, ArH), 7.57-7.62 (2H, m, ArH), 7.47 (2H, d, *J*=8.4Hz, ArH), 7.33-7.38 (2H, m, ArH), 7.23-7.28 (6H, m, ArH), 7.12-7.15 (4H, m, ArH), 5.60 (4H, s, C***H***$_2$Ph), 4.48 (4H, t, *J*=6.9Hz, C***H***$_2$[CH_2]$_4$C***H***$_2$), 1.88-1.97 (4H, m, CH_2C***H***$_2$[CH_2]$_2$C***H***$_2$$CH_2$), 1.58-1.62 (4H, m, CH_2CH_2[C***H***$_2$]$_2$$CH_2CH_2$); ^{13}C NMR (75 MHz, $CDCl_3$): δ 166.3, 141.9, 138.2, 138.1, 136.0, 132.3, 129.2, 128.9, 128.3, 126.7, 122.4, 121.8, 121.1, 117.9, 110.4, 65.9, 47.6, 29.2, 26.2. HRMS (ESI) calcd for $C_{44}H_{39}N_4O_4$ 687.2966 $[M+H]^+$, found 687.2976.

2.18 ***1, 8-Bis(9-benzyl-β-carboline-3-carboxylic acid)octanediyl ester*** **(31)**

Starting from 9-benzyl-β-carboline-3-carboxyl acid (0.66g, 2.1mmol) and 1,8-dibromooctane (1.0mmol). White solid was obtained (0.52g, 70%), mp 176-178℃. ESI-MS m/z: 715.3 (100) $[M+H]^+$. ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.90 (2H, s, ArH), 8.88 (2H, s, ArH), 8.22 (2H, d, *J*=7.8Hz, ArH), 7.57-7.62 (2H, m, ArH), 7.48 (2H, d, *J*=8.4Hz, ArH), 7.33-7.38 (2H, m, ArH), 7.24-7.28 (6H, m, ArH), 7.12-7.15 (4H, m, ArH), 5.61 (2H, s, C***H***$_2$Ph), 4.45 (4H, t, *J*=6.9Hz, C***H***$_2$[CH_2]$_6$C***H***$_2$), 1.84-1.93 (4H, m, CH_2C***H***$_2$[CH_2]$_4$C***H***$_2$$CH_2$), 1.45-1.52 (8H, m, CH_2CH_2[C***H***$_2$]$_4$$CH_2CH_2$); ^{13}C NMR (75 MHz, $CDCl_3$): δ 166.3, 141.9, 138.2, 138.0, 136.0, 132.3, 129.2, 128.8, 128.2, 126.7, 122.3, 121.7, 121.1, 117.9, 110.4, 66.0, 47.5, 29.6, 29.2, 26.3. HRMS (ESI) calcd for $C_{46}H_{43}N_4O_4$ 715.3279 $[M+H]^+$, found 715.3318.

2.19 ***1,5-Bis(9-benzyl-1-methyl-β-carboline-3-carboxylic acid)pentanediyl ester*** **(32)**

Starting from 9-benzyl-1-methyl-β-carboline-3-carboxyl acid (0.67g, 2.1mmol) and 1,5-dibromopentane (1.0mmol). White solid was obtained (0.48g, 68%), mp 175-177℃. ESI-MS m/z: 701.3 (100) $[M+H]^+$. ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.73 (2H, s, ArH), 8.13 (2H, d, *J*=7.5Hz, ArH), 7.50-7.55 (2H, m, ArH), 7.21-7.37 (10H, m, ArH), 6.90-6.93 (4H, m, ArH), 5.76 (4H, s, C***H***$_2$Ph), 4.51 (4H, t, *J*=6.9Hz, C***H***$_2$[CH_2]$_3$C***H***$_2$), 2.92 (6H, s, C***H***$_3$), 1.95-2.02 (4H, m, CH_2C***H***$_2$$CH_2$C***H***$_2$$CH_2$), 1.67-1.77 (2H, m, CH_2CH_2C***H***$_2$$CH_2CH_2$); ^{13}C NMR (75 MHz, $CDCl_3$): δ 166.1, 142.2, 142.0, 137.6, 137.3, 137.1, 129.2, 129.0, 128.9, 127.8, 125.5, 121.8, 121.7, 121.1,

116.3, 110.4, 65.5, 48.5, 28.9, 23.8, 23.1. HRMS (ESI) calcd for $C_{45}H_{41}N_4O_4$ 701.3122 $[M+H]^+$, found 701.3114.

2.20 ***1,6-Bis(9-benzyl-1-methyl-β-carboline-3-carboxylic acid)hexanediyl ester*** **(33)**

Starting from 9-1-methyl-benzyl-β-carboline-3-carboxyl acid (0.67g, 2.1mmol) and 1,6-dibromohexane (1.0mmol). White solid was obtained (0.56g, 78%), mp 143-145℃. ESI-MS m/z: 715.3 (100) $[M+H]^+$. ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.74 (2H, s, ArH), 8.19 (2H, d, J=7.8Hz, ArH), 7.52-7.57 (2H, m, ArH), 7.22-7.39 (10H, m, ArH), 6.92-6.95 (4H, m, ArH), 5.80 (4H, s, C$\boldsymbol{H}_2$Ph), 4.48 (4H, t, J=6.9Hz, C$\boldsymbol{H}_2$[CH_2]$_4$C$\boldsymbol{H}_2$), 2.94 (6H, s, C$\boldsymbol{H}_3$), 1.89-1.96 (4H, m, CH_2C$\boldsymbol{H}_2$[CH_2]$_2$C$\boldsymbol{H}_2$$CH_2$), 1.60-1.64 (4H, m, CH_2CH_2[C$\boldsymbol{H}_2$]$_2$$CH_2CH_2$); ^{13}C NMR (75 MHz, $CDCl_3$): δ 166.2, 142.3, 142.0, 137.6, 137.4, 137.2, 129.2, 129.1, 129.0, 127.9, 125.4, 121.9, 121.8, 121.1, 116.3, 110.4, 65.7, 48.6, 29.2, 26.3, 23.9. HRMS (ESI) calcd for $C_{46}H_{43}N_4O_4$ 715.3279 $[M+H]^+$, found 715.3271.

2.21 ***1,6-Bis[9-(3-chlorobenzyl)-β-carboline-3-carboxylic acid]hexanediyl ester*** **(34)**

Starting from 9-(3-chlorobenzyl)-β-carboline-3-carboxyl acid (0.71g, 2.1mmol) and 1,6-dibromohexane (1.0mmol). White solid was obtained (0.45g, 55%), mp 182-184℃. ESI-MS m/z: 755.8 (100) $[M+H]^+$. ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.90 (2H, s, ArH), 8.88 (2H, s, ArH), 8.22 (2H, d, J=7.8Hz, ArH), 7.59-7.65 (2H, m, ArH), 7.36-7.47 (4H, m, ArH), 7.13-7.23 (6H, m, ArH), 6.97-7.00 (2H, m, ArH), 5.58 (4H, s, C$\boldsymbol{H}_2$Ph), 4.49 (4H, t, J=6.9Hz, C$\boldsymbol{H}_2$[CH_2]$_4$C$\boldsymbol{H}_2$), 1.90-1.96 (4H, m, CH_2C$\boldsymbol{H}_2$[CH_2]$_2$C$\boldsymbol{H}_2$$CH_2$), 1.58-1.63 (4H, m, CH_2CH_2[C$\boldsymbol{H}_2$]$_2$$CH_2CH_2$); ^{13}C NMR (75 MHz, $CDCl_3$): δ 166.1, 141.7, 138.4, 138.0, 137.9, 135.2, 132.0, 130.5, 129.4, 129.0, 128.5, 126.9, 124.8, 122.5, 121.8, 121.4, 117.9, 110.2,65.9, 47.0, 29.2, 26.2. HRMS (ESI) calcd for $C_{44}H_{37}Cl_2N_4O_4$ 755.2186 $[M+H]^+$, found 755.2194.

2.22 ***1,9-Bis[9-(3-chlorobenzyl)-β-carboline-3-carboxylic acid]nonanediyl ester*** **(35)**

Starting from 9-(3-chlorobenzyl)-β-carboline-3-carboxyl acid (0.71g, 2.1mmol) and 1,9-dibromononane (1.0mmol). White solid was obtained (0.75g, 94%), mp 148-150℃. ESI-MS m/z: 797.4 (100) $[M+H]^+$. ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.89 (4H, s, , ArH), 8.22 (2H, d, J=7.8Hz, ArH), 7.59-7.64 (2H, m, ArH), 7.36-7.46 (4H, m, ArH), 7.14-7.22 (6H, m, ArH), 6.97-7.00 (2H, m, ArH), 5.59 (4H, s, C$\boldsymbol{H}_2$Ph), 4.46 (4H, t, J=6.9Hz, C$\boldsymbol{H}_2$[CH_2]$_7$C$\boldsymbol{H}_2$), 1.83-1.93 (4H, m, CH_2C$\boldsymbol{H}_2$[CH_2]$_5$C$\boldsymbol{H}_2$$CH_2$), 1.39-1.51 (10H, m, CH_2CH_2[C$\boldsymbol{H}_2$]$_5$$CH_2CH_2$); ^{13}C NMR (75 MHz, $CDCl_3$): δ 166.2, 141.7, 138.5, 138.1, 137.9, 135.2, 132.0, 130.5, 129.4, 129.0, 128.5, 126.9,

124.8, 122.4, 121.8, 121.3, 117.9, 110.2, 66.1, 47.0, 29.8, 29.6, 29.2, 26.3. HRMS (ESI) calcd for $C_{47}H_{43}Cl_2N_4O_4$ 797.2656 $[M+H]^+$, found 7797.2647.

2.23 ***1,3-Bis[9-(3-phenylpropyl)-β-carboline-3-carboxylic acid]propanediyl ester*** **(36)**

Starting from 9-(3-phenylpropyl)-β-carboline-3-carboxyl acid (0.69g, 2.1mmol) and 1,3-dibromopropane (1.0mmol). White solid was obtained (0.48g, 68%), mp 187-189℃. ESI-MS m/z: 701.3 (100) $[M+H]^+$. ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.71 (4H, s, ArH), 8.06 (2H, d, *J*=7.8Hz, ArH), 7.54-7.59 (2H, m, ArH), 7.20-7.32 (10H, m, ArH), 7.12 (4H, d, *J*=6.9Hz, ArH), 4.73 (4H, t, *J*=6.0Hz, $C\boldsymbol{H}_2CH_2C\boldsymbol{H}_2$), 4.19 (4H, t, *J*=7.2Hz, $C\boldsymbol{H}_2CH_2CH_2Ph$), 2.66 (4H, t, *J*=7.5Hz, $CH_2CH_2C\boldsymbol{H}_2Ph$), 2.44-2.52 (2H, m, $CH_2C\boldsymbol{H}_2CH_2$), 2.11-2.20 (4H, m, $CH_2C\boldsymbol{H}_2CH_2Ph$); ^{13}C NMR (75 MHz, $CDCl_3$): δ 166.0, 141.2, 140.3, 137.3, 137.2, 131.7, 128.7, 128.4, 128.0, 126.5, 122.1, 121.2, 120.6, 117.8, 109.8, 63.2, 42.7, 33.2, 30.3, 28.8. HRMS (ESI) calcd for $C_{45}H_{41}N_4O_4$ 701.3122 $[M+H]^+$, found 701.3116.

2.24 ***1,6-Bis[9-(3-phenylpropyl)-β-carboline-3-carboxylic acid]hexanediyl ester*** **(37)**

Starting from 9-(3-phenylpropyl)-β-carboline-3-carboxyl acid (0.69g, 2.1mmol) and 1,6-dibromohexane (1.0mmol). White solid was obtained (0.52g, 70%), mp 179-181℃. ESI-MS m/z: 743.3 (100) $[M+H]^+$. ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.82 (4H, s, ArH), 8.17 (2H, d, *J*=7.5Hz, ArH), 7.56-7.61 (2H, m, ArH), 7.11-7.39 (14H, m, ArH), 4.50 (4H, t, *J*=6.9Hz, $C\boldsymbol{H}_2[CH_2]_4C\boldsymbol{H}_2$), 4.38 (4H, t, *J*=7.2Hz, $C\boldsymbol{H}_2CH_2CH_2Ph$), 2.70 (4H, t, *J*=7.5Hz, $CH_2CH_2C\boldsymbol{H}_2Ph$), 2.19-2.29 (4H, m, $CH_2C\boldsymbol{H}_2[CH_2]_2C\boldsymbol{H}_2CH_2$), 1.89-1.96 (4H, m, $CH_2C\boldsymbol{H}_2CH_2Ph$), 1.59-1.64 (4H, m, $CH_2CH_2[C\boldsymbol{H}_2]_2CH_2CH_2$); ^{13}C NMR (75 MHz, $CDCl_3$): δ 166.3, 141.6, 140.4, 137.8, 137.6, 131.9, 129.0, 128.8, 128.5, 126.6, 122.3, 121.6, 120.8, 117.8, 110.0, 65.9, 43.2, 33.4, 30.6, 29.2, 26.2. HRMS (ESI) calcd for $C_{48}H_{47}N_4O_4$ 743.3592 $[M+H]^+$, found 743.3594.

2.25 ***1,7-Bis[9-(3-phenylpropyl)-β-carboline-3-carboxylic acid]heptanediyl ester*** **(38)**

Starting from 9-(3-phenylpropyl)-β-carboline-3-carboxyl acid (0.69g, 2.1mmol) and 1,7-dibromoheptane (1.0mmol). Yellow oil was obtained (0.34g, 45%). ESI-MS m/z: 757.4 (100) $[M+H]^+$. ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.84 (4H, s, ArH), 8.18 (2H, d, *J*=7.8Hz, ArH), 7.57-7.62(2H, m, ArH), 7.39 (2H, d, *J*=8.4Hz, ArH), 7.12-7.33 (12H, m, ArH), 4.48 (4H, t, *J*=6.9Hz, $C\boldsymbol{H}_2[CH_2]_5C\boldsymbol{H}_2$), 4.41 (4H, t, *J*=7.2Hz, $C\boldsymbol{H}_2CH_2CH_2Ph$), 2.71 (4H, t, *J*=7.5Hz, $CH_2CH_2C\boldsymbol{H}_2Ph$), 2.21-2.31(4H, m, $CH_2C\boldsymbol{H}_2[CH_2]_3C\boldsymbol{H}_2CH_2$), 1.85-1.94 (4H, m, $CH_2C\boldsymbol{H}_2CH_2Ph$), 1.51-1.56 (6H, m, $CH_2CH_2[C\boldsymbol{H}_2]_3CH_2CH_2$); ^{13}C NMR (75 MHz, $CDCl_3$): δ 166.2, 141.4, 140.4,

137.6, 137.5, 131.8, 128.9, 128.7, 128.3, 126.5, 122.2, 121.4, 120.7, 117.8, 109.9, 65.9, 43.0, 33.3, 30.4, 29.4, 29.2, 26.3. HRMS (ESI) calcd for $C_{49}H_{49}N_4O_4$ 757.3748 $[M+H]^+$, found 757.3751.

2.26 *1,6-Bis[9-(3-phenylpropyl)-1-methyl-β-carboline-3-carboxylic acid] hexanediyl ester* (39)

Starting from 9-(3-phenylpropyl)-1-methyl-β-carboline-3-carboxyl acid (0.72g, 2.1mmol) and 1,6-dibromohexane (1.0mmol). White solid was obtained (0.66g, 86%), mp 160-162℃. ESI-MS m/z: 772.0 (100) $[M+H]^+$. ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.67 (2H, s, ArH), 8.13 (2H, d, *J*=8.4Hz, ArH), 7.53-7.58 (2H, m, ArH), 7.17-7.33 (14H, m, ArH), 4.54 (4H, t, *J*=7.8Hz, C***H***$_2$[CH_2]$_4$C***H***$_2$), 4.47(4H, t, *J*=6.9, C***H***$_2$$CH_2CH_2$Ph), 2.97(6H, s, C***H***$_3$), 2.76 (4H, t, *J*=7.5Hz, CH_2CH_2C***H***$_2$Ph), 2.12-2.22 (4H, m, CH_2C***H***$_2$[CH_2]$_2$C***H***$_2$$CH_2$), 1.86-1.94 (4H, m, CH_2C***H***$_2$$CH_2$Ph), 1.60-1.64 (4H, m, CH_2CH_2[C***H***$_2$]$_2$$CH_2CH_2$); ^{13}C NMR (75 MHz, $CDCl_3$): δ 166.3, 141.8, 141.6, 140.5, 137.0, 136.7, 129.0, 128.8, 128.7, 128.5, 126.6, 121.8, 120.8, 116.3, 110.2, 65.7, 44.6, 33.3, 32.4, 29.2, 26.3, 24.1. HRMS (ESI) calcd for $C_{50}H_{51}N_4O_4$ 771.3905 $[M+H]^+$, found 771.3907.

2.27 *1,9-Bis[9-(3-phenylpropyl)-1-methyl-β-carboline-3-carboxylic acid] nonanediyl ester* (40)

Starting from 9-(3-phenylpropyl)-1-methyl-β-carboline-3-carboxyl acid (0.72g, 2.1mmol) and 1,9-dibromononane (1.0mmol). Yellow oil was obtained (0.4g, 49%). ESI-MS m/z: 814.0 (100) $[M+H]^+$. ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.69 (2H, s, ArH), 8.13 (2H, d, *J*=7.8Hz, ArH), 7.53-7.58 (4H, m, ArH), 7.16-7.33 (14H, m, ArH), 4.55 (4H, t, *J*=7.8Hz, C***H***$_2$[CH_2]$_7$C***H***$_2$), 4.44 (4H, t, *J*=6.9Hz, C***H***$_2$$CH_2CH_2$Ph), 2.97 (6H, s, C***H***$_3$), 2.76 (4H, t, *J*=7.5Hz, CH_2CH_2C***H***$_2$Ph), 2.12-2.22 (4H, m, CH_2C***H***$_2$[CH_2]$_5$C***H***$_2$$CH_2$), 1.83-1.92 (4H, m, CH_2C***H***$_2$$CH_2$Ph), 1.41-1.51 (10H, m, CH_2CH_2[C***H***$_2$]$_5$$CH_2CH_2$); ^{13}C NMR (75 MHz, $CDCl_3$): δ 166.2, 141.9, 141.6, 140.5, 137.0, 136.7, 129.0, 128.8, 128.7, 128.5, 126.6, 121.8, 120.8, 116.3, 110.2, 65.9, 44.6, 33.3, 32.4, 29.8, 29.7, 29.2, 26.4, 24.0. HRMS (ESI) calcd for $C_{53}H_{57}N_4O_4$ 813.4374 $[M+H]^+$, found 813.4386.

2.28 *1,6-Bis[1-(4-methoxyphenyl)-9-ethyl-β-carboline-3-carboxylic acid] hexanediyl ester* (41)

Starting from 1-(4-methoxyphenyl)-9-ethyl-β-carboline-3-carboxylic acid (0.73g, 2.1mmol) and 1,6-dibromohexane (1.0mmol). White solid was obtained (0.56g, 73%), mp 161-163℃. ESI-MS m/z: 895.4 (100) $[M+H]^+$. ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.82 (2H, s, ArH), 8.23 (2H, d, *J*=7.8Hz, ArH), 7.54-7.63 (6H, m, ArH), 7.46 (2H, d, *J*=8.4Hz, ArH), 7.32-7.37 (2H, m, ArH), 7.01-7.04 (4H, m, ArH), 4.46 (4H, t, *J*=6.9Hz, C***H***$_2$[CH_2]$_4$C***H***$_2$), 4.06 (4H, q, *J*=7.2Hz, NC***H***$_2$$CH_3$), 3.89 (6H, s, OC***H***$_3$), 1.86-1.96 (4H, m, CH_2C***H***$_2$[CH_2]$_2$C***H***$_2$$CH_2$), 1.57-1.62 (4H, m, CH_2CH_2[C***H***$_2$]$_2$$CH_2CH_2$), 1.01 (6H, t, *J*=7.2Hz, NCH_2C***H***$_3$); ^{13}C NMR (75 MHz, $CDCl_3$): δ 166.4,

160.2, 144.2, 142.3, 137.4, 135.8, 132.2, 130.9, 130.5, 128.8, 122.2, 120.9, 116.5, 113.9, 110.7, 65.7, 55.7, 39.7, 29.1, 26.2, 14.5. HRMS (ESI) calcd for $C_{52}H_{55}N_4O_4$ 895.3913 $[M+H]^+$, found 895.3919.

2.29 ***1,6-Bis[1-(4-methoxyphenyl)-9-(3-phenylpropyl)-β-carboline-3-carboxylic acid]hexanediyl ester*** **(42)**

Starting from 1-(4-methoxyphenyl)-9-(3-phenylpropyl)-β-carboline-3-carboxylic acid (0.92g, 2.1mmol) and 1,6-dibromohexane (1.0mmol). Yellow oil was obtained (0.56 g, 59%). ESI-MS m/z: 1075.5 (100) $[M+H]^+$. ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.83 (2H, s, ArH), 8.23 (2H, d, *J*=7.8Hz, ArH), 7.57-7.60 (6H, m, ArH), 7.31-7.36 (4H, m, ArH), 7.04-7.24 (6H, m, ArH), 7.04-7.06 (4H, m, ArH), 6.92-6.95 (4H, m, ArH), 4.48 (4H, t, *J*=6.6Hz, C***H***$_2$[CH_2]$_4$C***H***$_2$), 4.05 (4H, t, *J*=7.8Hz, C***H***$_2$$CH_2CH_2$Ph), 3.91 (6H, s, OC***H***$_3$), 2.17 (4H, t, *J*=7.8Hz, CH_2CH_2C***H***$_2$Ph), 1.89-1.96 (4H, m, CH_2C***H***$_2$[CH_2]$_2$C***H***$_2$$CH_2$), 1.68-1.75 (4H, m, CH_2C***H***$_2$$CH_2$Ph), 1.54-1.61 (4H, m, CH_2CH_2[C***H***$_2$]$_2$$CH_2CH_2$); ^{13}C NMR (75 MHz, $CDCl_3$): δ 166.4, 160.2, 144.2, 142.5, 140.8, 137.4, 136.0, 132.0, 131.1, 130.5, 128.9, 128.6, 128.3, 126.3, 122.0, 120.9, 116.7, 113.9, 110.7, 65.8, 55.8, 44.7, 33.3, 30.9, 29.2, 26.3. HRMS (ESI) calcd for $C_{66}H_{67}N_4O_{10}$ 1075.4852 $[M+H]^+$, found 1075.4844.

2.30 ***1,6-Bis[1-(3,4,5-trimethoxyphenyl)-9-ethyl-β-carboline-3-carboxylic acid] hexanediyl ester*** **(43)**

Starting from 1-(3,4,5-trimethoxyphenyl)-9-ethyl-β-carboline-3-carboxyl acid (0.85g, 2.1mmol) and 1,6-dibromohexane (1.0mmol). Yellow solid was obtained (0.6g, 67%), mp 117-119℃. ESI-MS m/z: 775.3 (100) $[M+H]^+$. ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.84 (2H, s, ArH), 8.24 (2H, d, *J*=7.5Hz, ArH), 7.60-7.65 (2H, m, ArH), 7.48 (2H, d, *J*=8.4Hz, ArH), 7.33-7.38 (2H, m, ArH), 6.81 (4H, s, ArH), 4.46 (4H, t, *J*=6.9Hz, C***H***$_2$[CH_2]$_4$C***H***$_2$), 4.05 (4H, q, *J*=6.9Hz, NC***H***$_2$$CH_3$), 3.90 (6H, s, OC***H***$_3$), 3.87(12H, s, OC***H***$_3$), 1.89-1.93 (4H, m, CH_2C***H***$_2$[CH_2]$_2$C***H***$_2$$CH_2$), 1.54-1.60 (4H, m, CH_2CH_2[C***H***$_2$]$_2$$CH_2CH_2$), 1.08 (6H, t, *J*=7.2Hz, NCH_2C***H***$_3$); ^{13}C NMR (75 MHz, $CDCl_3$): δ 166.2, 153.2, 144.0, 142.1, 138.4, 136.9, 135.5, 135.1, 130.4, 129.1, 122.1, 121.0, 117.0, 110.7, 106.8, 65.7, 61.3, 56.5, 39.7, 29.2, 26.2, 15.0. HRMS (ESI) calcd for $C_{48}H_{47}N_4O_6$ 775.3490 $[M+H]^+$, found 775.3483.

2.31 ***1,6-Bis[1-(3,4,5-trimethoxyphenyl)-9-(3-phenylpropyl)-β-carboline-3-carboxylic acid]hexanediyl ester*** **(44)**

Starting from 1-(3,4,5-trimethoxyphenyl)-9-(3-phenylpropyl)-β-carboline-3-carboxylic acid (1.04g, 2.1mmol) and 1,6-dibromohexane (1.0mmol). Yellow oil was obtained (0.74g, 69%). ESI-MS m/z: 955.4 (100) $[M+H]^+$. ^1H NMR (300 MHz, $CDCl_3$): δ 8.83 (2H, s, ArH), 8.23(2H, d, *J*=7.5Hz, ArH), 7.57-7.62 (2H, m, ArH), 7.32-7.37 (4H, m, ArH), 7.13-7.25 (6H, m, ArH), 6.96 (4H, d, *J*=7.5Hz, ArH), 6.84(4H, s, ArH), 4.46 (4H, t, *J*=6.9Hz, $CH_2[CH_2]_4CH_2$), 4.03(4H, t, *J*=7.5Hz, $CH_2CH_2CH_2Ph$), 3.93 (6H, s, OCH_3), 3.85 (12H, s, OCH_3), 2.23 (4H, t, *J*=7.8Hz, $CH_2CH_2CH_2Ph$), 1.89-1.93 (4H, m, $CH_2CH_2[CH_2]_2CH_2CH_2$), 1.76-1.81 (4H, m, $CH_2CH_2CH_2Ph$), 1.55-1.62 (4H, m, $CH_2CH_2[CH_2]_2CH_2CH_2$); ^{13}C NMR (75 MHz, $CDCl_3$): δ 166.0, 153.2, 143.9, 142.3, 140.6, 138.5, 136.9, 135.5, 135.0, 130.3, 129.1, 128.6, 128.1, 126.3, 122.1, 121.7, 121.0, 117.0, 110.6, 107.1, 65.6, 61.3, 56.5, 44.7, 33.3, 31.4, 29.1, 26.1. HRMS (ESI) calcd for $C_{62}H_{59}N_4O_6$ 955.4429 $[M+H]^+$, found 955.4438.