Supplementary Information for *Chem. Commun.*

**Globally Folded Structure Based on Localized Rigidification: Helical Oligo(*m*-phenylene ethynlenes)**

Xiaowu Yang,*a* Mako Furukawa,*b* Shoujian Li,∗c Wendy E. Gardinier,*a* Eric J. Bukowski,*a* Frank V. Bright,*a* Chong Zheng,*c* Xiao Cheng Zeng,*b* and Bing Gong*a*

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*a* **Department of Chemistry**

University at Buffalo, The State University of New York
Buffalo, New York 14260, USA
Fax: (+1) 716 645 6963; Tel: (+1) 716 645 6800
E-mail: bgong@chem.buffalo.edu

*b* **Department of Chemistry**

University of Nebraska-Lincoln
Lincoln, Nebraska 68588, USA

*c* **Department of Chemistry**

Northern Illinois University
DeKalb, Illinois 60115, USA.
I. 1D $^1$H NMR Spectra

2d

3

4
II. Chemical shifts of protons $t1$-$t4$ and $a1$-$a2$ vs the chain length of oligomers 2d-6. (b)

III. The chemical shifts of protons $t1$-$t4$ of hexamer 6 vs temperature.
IV. Concentration-dependent changes of amide $^1$H chemical shifts of tetramer 4

(500 MHz, CDCl$_3$, 295 K)

0.063 mM

0.125 mM

0.25 mM

0.5 mM

1 mM

2 mM

4 mM

8 mM
V. Chemical shifts (ppm) of the aromatic and ethyl protons on the end residues of oligomers 2d-6

<table>
<thead>
<tr>
<th>Compd</th>
<th>H1</th>
<th>H2</th>
<th>H3</th>
<th>H4</th>
<th>H5</th>
<th>H6</th>
<th>H(CH3)</th>
<th>H(CH2)</th>
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<tbody>
<tr>
<td>2d</td>
<td>7.71</td>
<td>7.57</td>
<td>7.43</td>
<td>8.16</td>
<td>7.58</td>
<td>8.87</td>
<td>1.25/1.35</td>
<td>3.73/3.81</td>
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<tr>
<td>3</td>
<td>7.73</td>
<td>7.62</td>
<td>7.49</td>
<td>8.18</td>
<td>7.58</td>
<td>8.86</td>
<td>1.2-1.38</td>
<td>3.75/3.76</td>
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<td>4</td>
<td>7.74</td>
<td>7.63</td>
<td>7.51</td>
<td>8.20</td>
<td>7.57</td>
<td>8.86</td>
<td>1.2-1.39</td>
<td>3.71/3.76</td>
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<td>5</td>
<td>7.70</td>
<td>7.58</td>
<td>7.49</td>
<td>8.19</td>
<td>7.57</td>
<td>8.86</td>
<td>1.12/1.22</td>
<td>3.57/3.67</td>
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<tr>
<td>6</td>
<td>7.40</td>
<td>6.92</td>
<td>7.12</td>
<td>8.00</td>
<td>7.41</td>
<td>8.76</td>
<td>0.79/1.10</td>
<td>3.05/3.54</td>
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VI. Temperature-dependent changes of amide and aromatic $^1$H chemical shifts
(500 MHz, 2 mM in CDCl$_3$)

1. Tetramer 4
Temperature-dependent $^1$H chemical shift values of tetramer 4.

<table>
<thead>
<tr>
<th>Temp. (°C)</th>
<th>$Ht1$</th>
<th>$Ht2$</th>
<th>$Ht3$</th>
<th>$Ht4$</th>
<th>$Ha$</th>
<th>$Hb$</th>
<th>$Hc$</th>
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</table>

2. Hexamer 6
Temperature-dependent $^1$H chemical shift values of hexamer 6.

<table>
<thead>
<tr>
<th>Temp. ($^\circ$C)</th>
<th>Ht1</th>
<th>Ht2</th>
<th>Ht3</th>
<th>Ht4</th>
<th>Ha</th>
<th>Hb/Hc/Hd</th>
<th>He</th>
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<tr>
<td>20</td>
<td>7.405</td>
<td>6.916</td>
<td>7.120</td>
<td>8.003</td>
<td>8.980</td>
<td>9.106</td>
<td>9.274</td>
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<tr>
<td>0</td>
<td>7.385</td>
<td>6.842</td>
<td>7.082</td>
<td>8.008</td>
<td>9.249</td>
<td>9.038</td>
<td>8.950</td>
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<td>-10</td>
<td>7.375</td>
<td>6.798</td>
<td>7.066</td>
<td>8.015</td>
<td>9.228</td>
<td>-</td>
<td>-</td>
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<tr>
<td>-20</td>
<td>7.369</td>
<td>6.739</td>
<td>7.035</td>
<td>8.024</td>
<td>9.181</td>
<td>-</td>
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VII. 2D NMR Spectra

1. Partial NOESY Spectrum of Tetramer 4

NOEs between the amide protons and ester side chains α-CH₂ protons
(500 MHz, CDCl₃, 263K, con. = 8 mM, mixing time = 0.3 s)
2. Partial NOESY spectrum of hexmer 6
Contacts between the methyl group and side chains methene protons
(500 MHz, CDCl₃, 263K, con. = 8 mM, mixing time = 0.3 s)
(terminal CH₃ of ester groups)
VIII. UV Spectra of Oligomers 2d-6 at Room Temperature

(a) 10 \mu M in chloroform

(b) 2 \mu M in chloroform

(c) 2 \mu M in chloroform/methanol (v/v 1/1)
IX. Fluorescence Experiments and Emission Spectra of Oligomers 2d-6 at Room Temperature

1. Experimental

The solvent for all spectroscopic experiments was acetonitrile.

All absorbance spectra were recorded on a Milton-Roy model 1201 UV-Vis spectrophotometer.

All fluorescence experiments (low micromolar n-mer) were performed with an SLM-AMINCO 48000 multiharmonic Fourier (MHF) spectrofluorometer (Spectronic Instruments). The excitation and emission spectral bandpasses were kept at 4 nm and 2 nm, respectively. All spectra were background corrected. The background contribution was generally small except for the dimer species where the emission was at least an order-of-magnitude weaker in comparison to the other n-mers.

Table 1 list the positions of the longest wavelength absorbance transition and the emission of each n-mer.

<table>
<thead>
<tr>
<th>Species</th>
<th>Absorbance Maximum (nm)</th>
<th>Emission Maxima (nm)</th>
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<tbody>
<tr>
<td>Dimer</td>
<td>320</td>
<td>420 (w)</td>
</tr>
<tr>
<td>Trimer</td>
<td>318</td>
<td>435</td>
</tr>
<tr>
<td>Tetramer</td>
<td>324</td>
<td>440</td>
</tr>
<tr>
<td>Pentamer</td>
<td>330</td>
<td>430,525</td>
</tr>
<tr>
<td>Hexamer</td>
<td>332</td>
<td>420,530</td>
</tr>
</tbody>
</table>

Typical emission spectra for the n-mers is shown in Figure 1 (excitation = 350 nm). The overall spectroscopy shows that the red-edge absorbance maxima shift slightly to the red as the n-mer increases from dimer to hexamer. The molar absorptivity for these transitions do not change significantly, remaining at 3-4,000 cm⁻¹M⁻¹. The highest energy emission feature remains between 420 and 440 nm in each n-mer and the emission from the dimer is significantly weaker in comparison to the other n-mers. However, a new emission feature appears for the pentamer and more so for the hexamer to the red side of the emission spectrum. The emission wavelength-dependent excitation scans of the pentamer and hexamer[1] (not shown) are consistent with the red-edge emission arising from ground pre-association of the chromophores.

2. Emission spectra of 2d-6
X. Computational Details

The computations were carried out using the Gaussian 98 revision A.9. The geometry of each conformation was optimized at the B3LYP/6-31G(d,p) level. The two optimized structures were used to compute the single point energy at the MP2/6-31G(d) level. One of the C-Ph bonds of the optimized conformer 1a structure was rotated 180 degrees to get the structure of conformer 2b. The same was done for conformer 2b to get a structure of 2a.

Results and Discussions

Geometry and Energy. Conformer 2a, which has a hydrogen bond, is the stable conformation. Conformer 2b, the one with no hydrogen bonding, is 5.78 kcal/mol higher in energy. When we rotate the C-Ph bond of conformer 1a to create conformer 2b, we see an energy barrier of 7.19 kcal/mol higher than the energy of conformer 2a. This barrier occurs when the bond is rotated 100 to 110 degrees towards conformer 2b. When rotating the C-Ph bond from conformer 2b to 2a, we see an energy barrier of about 1.21 kcal/mol. Again, this happens at around 100 degrees towards conformer 2b.

We note that by simply rotating the C-Ph bond 180 degrees from conformer 2a, does not create the optimized structure of conformer 2b. This is due to the fact that the hydrogen bonding causes conformer 2a to be a completely planar conformation whereas conformer 2b which has the intramolecular H-bond interrupted is not completely planar. To get a better understanding of the energy barriers, the energies were studied by rotating one of the C-Ph bonds of the optimized conformer 2a model to make conformer 2b, and then from the optimized structure of conformer 2b to make conformer 2a.

As noted above, the energy barrier occurs when the C-Ph bond is rotated 100 to 110 degrees towards conformer 2b from the optimized structure of conformer 2a. The major factor in this barrier is due to the fact that the hydrogen bonding is a strong one with an O–H bond distance of 1.92 Å, and an N-H–O angle of 176.65 degrees.
XI. Synthetic Procedures

General

All chemicals were purchased from Aldrich or Acros and were used as received unless otherwise noted. Triethylamine was dried from sodium and degassed before use. The coupling reactions were carried out under dry argon. All reactions were followed by thin-layer chromatography (precoated 0.25 mm silica gel plates from Aldrich), and silica gel column chromatography was carried out with silica gel 60 (mesh 230-400). The $^1$HNMR spectra and $^{13}$C NMR were recorded on a spectrometer of 500 MHz. NMR chemical shifts are reported in ppm relative to internal standard TMS, and coupling constant, $J$, is reported in Hertz (Hz). The following splitting patterns are designed as s, singlet; d, double; t, triplet; q, quartet; b, broad; m, multiplet.
Methyl 5-acetylaminobenzoate (1a). Compound 1a was synthesized starting from commercially available 3-nitrobenzoic acid which was initially esterified (98.9%) followed by reduction of the nitro group and protection of the corresponding amino group using acetic anhydride (94.3%) to afford a white solid. TLC, R_f = 0.40 (petroleum ether/EtOAc, 1/2), ¹H NMR (500 MHz, CDCl₃): δ 2.20 (s, 3H, Ac), 3.90 (s, 3H, MeO), 7.39 (t, 1H, Ar-H), 7.65 (s, 1H, NH), 7.78 (d, 1H, Ar-H, J = 7.5 Hz), 7.92 (d, 1H, Ar-H, J = 7.5 Hz), 8.02 (s, 1H, Ar-H).

Methyl 5-acetamido-2-nitrobenzoate (1b). Compound 1a (35 g, 181 mmol) was added to concentrated H₂SO₄ (120 mL) cooled in an ice-water bath. To this solution, a cooled mixture of 70% HNO₃ (12.3 mL) and con. H₂SO₄ (58 mL) was added dropwise over a period of 0.5 h at 0 °C. After being stirred for 20 min., the reaction mixture was poured into cracked ice (1 kg) and the mixture was extracted with dichloromethane (150 mL × 3). The combined extracts were then washed with aqueous NaHCO₃ and water respectively, dried over anhydrous Na₂SO₄, filtered and concentrated to provide a brown residue. The resulting solid was recrystallized from dichloromethane to afford 24 g (55.7%) of 1b as a pale yellow needle. TLC, R_f = 0.36 (petroleum ether/EtOAc, 1/3). ¹H NMR (500 MHz, CDCl₃): δ 2.24 (s, 3H, Ac), 3.93 (s, 3H, MeO), 7.61 (d, 1H, Ar-H), 7.86 (dd, 1H, Ar-H), 7.94 (b, 1H, NH), 7.98 (d, 1H, Ar-H, J = 7.5 Hz). ¹³C NMR (125 MHz, CDCl₃): δ 25.11, 54.03, 119.49, 121.18, 126.35, 129.27, 142.46, 142.55, 167.28, 167.78.
**Methyl 5-amino-2-nitrobenzoate (1c).** To a solution of 1b (2.17 g, 9.12 mmol) in MeOH (50 mL) was added con. H₂SO₄ (0.9 mL) with stirring. The solution was refluxed for 0.5 h and the solvent was removed in vacuo to provide a pale yellow oil. The oil was dissolved in dichloromethane (30 mL) and washed with NaHCO₃, water and brine. After washing, the organic layer was dried over Na₂SO₄ and filtered. The filtrate was then concentrated in vacuo to yield a light yellow solid, which was recrystallized from EtOAc to afford 1.65 g (92.3 %) of 1c as a colorless solid. TLC, R_f = 0.24 (petroleum ether/EtOAc, 3/2). ¹H NMR (500 MHz, CDCl₃): δ 3.92 (s, 3H, MeO), 4.56 (s, 2H, NH₂), 6.61 (m, 2H, Ar-H), 7.94 (d, 1H, Ar-H, J=9.5 Hz). ¹³C NMR (125 MHz, CDCl₃): δ 53.19, 112.99, 114.31, 127.23, 132.11, 151.77, 167.54.

**Methyl 5-amino-4-iodo-2-nitrobenzoate (1d).** To a vigorously stirred solution of 1c (4.42 g, 22.6 mmol) in glacial acetic acid (20 mL) was added dropwise a solution of ICl (3.67 g, 22.6 mmol) in glacial acetic acid (5 mL). After 10 min., a yellow precipitate appeared and the solution was allowed to stir for another 12 h. Upon completion, the yellow solution was poured into water (50 mL), and filtered to yield a yellow solid, which was recrystallized from MeOH to afford 5.7 g (78.3 %) of the yellow solid 1d. TLC, R_f = 0.42 (petroleum ether/EtOAc, 3/2). ¹H NMR (500 MHz, CDCl₃): δ 3.91 (s, 3H, MeO), 4.88 (b, 2H, NH₂), 6.73 (s, 1H, Ar-H), 8.42 (s, 1H, Ar-H). ¹³C NMR (125 MHz, CDCl₃): δ 53.57, 81.67, 94.81, 111.87, 114.38, 131.46, 136.17, 151.61.

**Methyl 5-N-acetylamino-4-iodo-2-nitrobenzoate (1e).** To a solution of 1d (1.6 g, 4.97 mmol) in dichloromethane (15 mL) was added con. H₂SO₄ (0.85 mL) at 0 °C. After 5 min., a solution of acetic anhydrous (1.17 mL, 12.38 mmol) in dichloromethane (5 mL) was added dropwise at 0 °C. Upon addition, the mixture was warmed to room temperature and stirred for 1 h until completion detected by TLC. The resulting solution was washed with water, aqueous NaHCO₃, and brine, dried over anhydrous Na₂SO₄ and filtered. The resulting filtrate was evaporated in vacuo to yield a pale yellow solid, which was recrystallized from MeOH to afford 1.6 g (93.8 %) of 1e as a white solid. TLC, R_f = 0.48 (petroleum ether/EtOAc, 1/2). ¹H NMR (500 MHz, CDCl₃): δ 2.31 (s, 3H, Ac), 3.93 (s, 3H, MeO), 7.71 (b, 1H, NH), 8.44 (s, 1H, Ar-H), 8.67 (s, 1H, Ar-H). ¹³C NMR (125 MHz, CDCl₃): δ 25.56, 51.81, 101.0, 111.24, 126.11, 126.26, 127.31, 148.29, 166.02, 168.43.

**Methyl 5-N-acetylamino-4-iodo-2-aminobenzoate (1f).** To a mixture of 1e (5.6 g, 15.4 mmol) in absolute ethanol (88 mL) and glacial acetic acid (88 mL) was added iron powder (2.58 g, 46.7 mmol). The mixture was then heated to reflux for 2 h. The red reaction solution was allowed to cool to room temperature, diluted with water (300 mL), and extracted with dichloromethane (100 mL×3). The combined organic extracts were then washed with water, and dried over anhydrous Na₂SO₄ and filtered. The resulting filtrate was evaporated in vacuo to yield a pale yellow solid, which was recrystallized from MeOH to afford 4.36 g (84.8 %) of 1f as a white needle. TLC, R_f = 0.36 (petroleum ether/EtOAc, 1/2). ¹H NMR (500 MHz, CDCl₃): δ 2.22 (s, 3H, Ac), 3.87 (s, 3H, MeO), 5.69 (b, 2H, NH₂), 7.06 (b, 1H, NH), 7.18 (b, 1H, Ar-H), 8.29 (s, 1H, Ar-H). ¹³C NMR (125 MHz, CDCl₃): δ 24.19, 51.81, 101.0, 111.24, 126.11, 126.26, 127.31, 148.29, 167.87, 168.43.
Methyl 5-N-acetylamino-4-iodo-2-[3,3-diethyl-1-triazenyl] benzoate (1g). A solution of sodium nitrite (0.49 g, 7.1 mmol) in water (4.5 mL) was cooled to 0 °C and then added dropwise over a 10 min. period to a 0 °C solution of 1f (2.13 g, 6.38 mmol) and con. hydrochloric acid (1.8 mL) in water (12 mL) and acetonitrile (20 mL). The mixture was stirred for 0.5 h at 0 °C and then added dropwise to a solution of diethylamine (2.33 mL, 22 mmol) and potassium carbonate (2.9 g, 21 mmol) which was pre-cooled to 0 °C. During the addition, more diethylamine (2.3 mL) was added to the reaction solution. After addition, the mixture was warmed to room temperature for 0.5 h. During that period, a yellow solid precipitated. Upon completion, the solution was extracted with dichloromethane (80 mL×2). The combined organic extracts were then washed with brine and water, dried over anhydrous Na₂SO₄, filtered and concentrated in vacuo to yield a yellow solid, which was recrystallized from MeOH to afford 2.39 g (86.2 %) of 1g as a colorless needle. TLC, Rf = 0.23 (petroleum ether/EtOAc, 3/2). ¹H NMR (500 MHz, CDCl₃): δ 1.31 (b, 6H, Me), 2.24 (s, 3H, Ac), 3.74 (b, 4H, CH₂), 3.85 (s, 3H, MeO), 7.35 (b, 1H, NH), 7.86 (s, 1H, Ar-H), 8.41 (s, 1H, Ar-H). ¹³C NMR (125 MHz, CDCl₃): δ 11.09, 14.52, 24.71, 41.80, 49.20, 52.08, 93.94, 121.97, 127.15, 128.79, 134.63, 146.74, 167.83, 167.98.

5-N-Acetylamino-4-iodo-2-[3,3-diethyl-1-triazenyl] benzoic acid (1h). A solution of 1g (1.4 g, 3.35 mmol) in MeOH (9 mL) was heated to reflux, to which was added dropwise aqueous NaOH (3.7 mL, 1N). The resulting solution was refluxed for 0.5 h and then cooled down to room temperature. Upon cooling, water (20 mL) was introduced. The solution was then extracted with Et₂O and the water phase was neutralized to pH 3 with 1N HCl. After filtration, the solid was dried in vacuo to afford 1.27 g (93.8 %) of 1h as a white solid. ¹H NMR (500 MHz, CDCl₃): δ 1.31 (t, 3H), 1.44 (t, 3H), 2.24 (s, 3H, Ac), 3.78 (q, 2H), 3.95 (q, 2H), 7.43 (s, 1H, NH), 8.12 (s, 1H, Ar-H), 8.71 (s, 1-H, Ar-H), 13.96 (s, 1H, COOH). ¹³C NMR (125 MHz, CDCl₃): δ 10.27, 14.36, 24.44, 43.47, 51.03, 98.62, 122.24, 125.98, 126.26, 135.98, 145.30, 166.34.

Octyl 5-N-acetylamino-4-iodo-2-[3,3-diethyl-1-triazenyl] benzoate (1i). A solution of the white acid 1h (3.0 g, 7.43 mmol), DCC (1.6 g, 7.77 mmol), and DMAP (0.27 g, 2.21 mmol) in dichloromethane (40 mL) was stirred for 1 h at room temperature, to which was added n-octanol (1.11 g, 8.54 mmol) and more DCC (1.6 g, 7.77 mmol) respectively. The reaction mixture was stirred for 48 h at rt., filtered, and concentrated to give a red oil, which was purified by silica gel flash column chromatography (petroleum ether/ EtOAc: 4/1) to provide 3.27 g (85.3 %) of 1i as a pale yellow oil. TLC, Rf = 0.33 (petroleum ether/EtOAc, 2/1). ¹H NMR (500 MHz, CDCl₃): δ 0.88 (t, 3H), 1.22-1.39 (m, 16H), 1.72 (m, 2H), 2.20 (s, 3H, Ac), 3.75 (b, 4H, CH₂), 4.23 (t, 2H, CH₂O), 7.42 (s, 1H, Ar-H), 7.87 (s, 1H, NH), 8.37 (s, 1H, Ar-H). ¹³C NMR (125 MHz, CDCl₃): δ 11.46, 14.33, 14.78, 22.88, 24.93, 26.12, 28.89, 29.39, 29.50, 32.03, 41.91, 49.40, 51.03, 98.62, 122.24, 125.98, 126.26, 135.98, 145.30, 166.34.

Octyl 5-N-acetylamino-4-iodo-2-[3,3-diethyl-1-triazenyl]-4-[2-(1,1-trimethylsilyl)-1-ethynyl] benzoate (1k). A 50 mL flask under argon was charged with 1i (0.77 g, 1.49 mmol), dichlorobis(triphenylphosphine) palladium (II) (21 mg, 0.030 mmol), copper(I) iodide (5.7 mg, 0.03 mmol), and triethylamine (23 mL). The solution was then stirred and warmed to 40 °C, to which was
added dropwise degassed trimethylsilylacetylene (220 ul, 1.54 mmol) by syringe. The reaction mixture was allowed to stir for 12 h at the same temperature, filtered and then concentrated to yield a dark oil. The oil was purified by flash silica gel column chromatography (petroleum/EtOAc 8/1) to afford 0.68 g (93.8 %) of 1k as a colorless solid. TLC, Rf = 0.39 (petroleum ether/EtOAc, 5/1). $^1$H NMR (500 MHz, CDCl3): δ 0.30 (s, 9H, SiMe3), 0.88 (t, 3H, 1.29 (b, 16H), 1.72 (m, 2H), 2.20 (s, 3H, Ac), 3.73 (b, 4H, CH2), 4.26 (t, 2H, CH2O), 7.49 (s, 1H, Ar-H), 7.92 (s, 1H, NH), 8.61 (s, 1H, Ar- H). $^{13}$C NMR (125 MHz, CDCl3): δ 0.099, 11.89, 14.27, 14.33, 22.84, 24.94, 26.16, 28.89, 29.36, 29.47, 32.00, 41.97, 49.23, 65.54, 100.18, 103.35, 114.36, 119.21, 121.61, 128.69, 136.17, 145.05, 167.85, 168.09. Anal. Calcd for C26H42N4O3Si.C, 64.16; H, 8.70; N, 11.51. Found C, 64.14; H, 8.60; N, 11.61.

Methyl 5-N-acetylamino-2-[3,3-diethyl-1-triazenyl]-4-[2-(1,1-trimethylsilyl)-1-ethynyl] benzoate (1j). Compound 1j was prepared from 1g (6.12 g, 14.64 mmol) as described for 1k to afford a brown oil. The oil was purified by silica gel column chromatography (petroleum ether/EtOAc 2/1) to afford 5.58 g (98.2 %) of 1j as a pale yellow solid. TLC, Rf = 0.38 (petroleum ether/EtOAc, 2/1).

$^1$H NMR (500 MHz, CDCl3): δ 0.30 (s, 9H, SiMe3), 1.26 (b, 6H, CH3), 2.20 (s, 3H, Ac), 3.73 (b, 4H, CH2), 3.86 (s, 3H, MeO), 7.49 (s, 1H, Ar-H), 7.92 (b, 1H, NH), 8.63 (s, 1H, Ar-H). $^{13}$C NMR (125 MHz, CDCl3): δ 0.39, 11.09, 14.57, 25.26, 42.08, 49.83, 52.57, 100.4, 103.85, 114.92, 119.70, 121.96, 128.29, 136.41, 145.55, 168.19, 166.65. Anal. Calcd for C19H28N4O3Si: C, 58.73; H, 7.26; N, 14.42. Found C, 58.77; H, 7.26; N, 14.48.

Methyl 5-N-acetylamino-2-[3,3-diethyl-1-triazenyl]-4-ethynyl benzoate (1l). To a solution of 1j (0.50 g, 1.29 mmol) in methanol (8 mL) was added potassium carbonate (12 mg, 0.087 mmol). The solution was stirred for 5 min. at room temperature, diluted with water (10 mL), and then extracted with dichloromethane (10 mL x 20). The pale yellow extracts were washed with water, dried over Na2SO4 and concentrated to yield a oil. The oil was purified by silica gel column chromatography (petroleum ether/EtOAc, 1/1) to afford 0.39 g (96.2 %) of 1l as a pale yellow solid. TLC, Rf = 0.57 (petroleum ether/EtOAc, 1/3).

$^1$H NMR (500 MHz, CDCl3): δ 1.26 (m, 6H, CH3), 2.22 (s, 3H, Ac), 3.53 (s, 1H, CCH), 3.72 (m, 4 H, CH2), 3.86 (s, H, MeO), 7.53 (s, 1H, Ar-H), 7.82 (s, 1H, NH), 8.62 (s, 1H, Ar-H). $^{13}$C NMR (125 MHz, CDCl3): δ 25.02, 52.27, 79.18, 85.21, 113.54, 119.84, 122.50, 128.36, 136.20, 145.29, 168.11, 168.32.

Methyl 5-N-acetylamino-2-iodo-4-[2-(1,1-trimethylsilyl)-1-ethynyl] benzoate (1m). To a sealed tube was added 1j (2.39 g, 6.16 mmol) and iodomethane (10 mL) under argon. The mixture was stirred for 12 h at 120 °C. After filtration the filtrate was concentrated to yield a brown oil, which was purified by silica gel column chromatography (petroleum ether/EtOAc 3/1) to afford 2.09 g (81.9 %) 1m as a white solid. TLC, Rf = 0.53 (petroleum ether/EtOAc, 2/1).

$^1$H NMR (500 MHz, CDCl3): δ 0.31 (s, 9H, SiMe3), 2.22 (s, 3H, Ac), 3.92 (s, 3H, MeO), 7.90 (s, 1H, Ar-H), 7.99 (s, 1H, Ar-H), 8.86 (s, 1H, Ar-H). $^{13}$C NMR (125 MHz, CDCl3): δ -0.041, 25.02, 52.90, 85.46, 97.90, 106.29, 116.10, 120.95, 136.03, 139.42, 143.37, 166.54, 168.25.
**Dimer 2c.** A dry flask was charged with 1l (0.77 g, 2.44 mmol), 1m (0.64 g, 1.54 mmol), bis(dibenzylideneacetone) palladium (28 mg, 0.031 mmol), triphenylphosphine (48 mg, 0.18 mmol), copper (I) iodide (5.9 mg, 0.031 mmol), dry acetonitrile (60 mL), and triethylamine (15 mL) under argon. The mixture was degassed three times and stirred for 12 h at 70 °C. The resulting solution was then filtered and concentrated to yield a dark oil, which was purified by silica gel column chromatography (petroleum ether/EtOAc 1/1) to afford 0.89 g (95.8 %) of dimer 2c as a yellow solid. TLC, Rf = 0.50 (petroleum ether/EtOAc, 1/3). 1H NMR (500 MHz, CDCl3): δ 0.33 (s, 9H, SiMe3), 1.28 (m, 6H, CH3), 2.26 (s, 3H, Ac), 2.42 (s, 3H, Ac), 3.75 (m, 4H, CH2), 3.87 (s, 3H, MeO), 3.94 (s, 3H, MeO), 7.54 (s, 1H, Ar-H), 7.76 (s, 1H, Ar-H), 8.03 (s, 1H, NH), 8.86 (s, 1H, Ar-H), 9.14 (s, 1H, Ar-H), 9.22 (s, 1H, NH). 13C NMR (125 MHz, CDCl3): δ -0.03, 24.71, 24.76, 52.21, 52.88, 90.41, 94.75, 98.38, 106.61, 114.39, 118.70, 119.85, 120.90, 121.73, 128.14, 130.86, 136.82, 136.88, 137.74, 139.04, 144.84, 165.94, 168.37, 168.53, 169.84. Anal. Calcd for C31H37N5O6Si: C, 61.67; H, 6.18; N, 11.60. Found C, 61.76; H, 6.22; N, 11.64.

**Octyl 5-N-acetylamino-2-[3,3-diethyl-1-triazenyl]-4-ethynyl benzoate (1o).** To a solution of the compound 1k (0.95 g, 1.95 mmol) in MeOH (15 mL) was added potassium carbonate (15 mg, 0.11 mmol) as described for 1l to yield a brown oil. The oil was then purified by silica gel column chromatography (petroleum ether/EtOAc: 2/1) to afford 0.73 g (90.2 %) of 1o as a light yellow oil. TLC, Rf = 0.48 (petroleum ether/EtOAc, 1/1). 1H NMR (500 MHz, CDCl3): δ 0.87 (t, 3H), 1.26-1.38 (m, 16H), 1.72 (m, 2H), 2.20 (s, 3H, Ac), 3.55 (s, 1H), 3.72 (b, 4H), 7.54 (s, 1H, Ar-H), 7.91 (s, 1H, NH), 8.58 (s, 1H, Ar-H). 13C NMR (125 MHz, CDCl3): δ 11.41, 14.28, 14.67, 22.83, 24.94, 25.98, 26.14, 28.85, 29.48, 31.98, 32.97, 41.80, 49.26, 63.06, 65.57, 79.08, 85.35, 113.56, 119.75, 122.40, 128.843, 136.17, 145.08, 168.18, 168.33.

**Octyl 5-N-acetylamino-2-iodo-4-[2-(1,1,-trimethylsilyl)-1-ethynyl] benzoate (1p).** The compound was synthesized from 1k (0.97 g, 1.99 mmol) by a similar procedure for 1m to yield a light yellow oil. The oil was then purified by silica gel column chromatography (petroleum ether/EtOAc, 15/1, 10/1) to afford a colorless oil, which was stood overnight to afford 0.81 g (79.4 %) of 1p as a white wax. TLC, Rf = 0.58 (petroleum ether/EtOAc, 5/1). 1H NMR (500 MHz, CDCl3): δ 0.31 (s, 9H, SiMe3), 0.88 (t, 3H), 1.29 (m, 10H), 1.72 (m, 2H), 2.20 (s, 3H, Ac), 4.32 (t, 2H, CH2O), 7.91 (s, 1H, NH), 7.97 (s, 1H, Ar-H), 8.83 (s, 1H, Ar-H). 13C NMR (125 MHz, CDCl3): δ 14.29, 22.83, 25.00, 26.11, 28.72, 28.85, 29.38, 31.97, 66.30, 85.25, 97.94, 106.08, 115.91, 120.83, 136.66, 139.43, 143.20, 166.32, 168.20.

**Dimer 2e.** Following the procedure as described for 2c, the acetylene 1o (0.74 g, 1.79 mmol), 1p (0.76 g, 1.48 mmol), bis(dibenzylideneacetone) palladium (22 mg, 0.024 mmol), copper (I) iodine (4.7 mg, 0.024 mmol), and triphenylphosphine (33 mg, 0.12 mmol) in dry triethylamine (20 mL) was stirred at 70 °C for 24 h under argon. The reaction solution was then concentrated to yield a dark oil, which was purified by silica gel column chromatography (petroleum ether/EtOAc: 5/1) to afford 1.05 g (88.9 %) of dimer 2e as a yellow solid. TLC, Rf = 0.63 (petroleum ether/EtOAc, 2/1). 1H NMR (500 MHz, CDCl3): δ 0.33 (s, 9H, SiMe3), 0.88 (m, 6H), 1.27-1.44 (m, 26H), 1.74 (m, 2H), 1.80 (m, 2H), 2.26 (s, 2H, Ac), 2.41 (s, 2H, Ac), 3.76 (m, 4H, CH2), 4.27(t, 2H), 4.33 (t, 2H), 7.55 (s, 1H), 7.75 (s, 2H).
Dimer 2f. To a solution of dimer 2e (0.58 g, 0.72 mmol) in MeOH (8 mL) and dichloromethane (1 mL) was added potassium carbonate (5 mg, 0.026 mmol). The mixture was allowed to stir for 10 min. at room temperature and then water (8 mL) was added. Upon addition, the solution was extracted with dichloromethane (10 mL × 3) and the resulting organic extracts were then washed with water (10 mL), dried over Na₂SO₄, filtered and concentrated to afford 0.51 g (96.5%) of dimer 2f as a pure pale yellow oil. TLC, Rf = 0.39 (petroleum ether/EtOAc, 1/3). ¹H NMR (500 MHz, CDCl₃): δ 0.88 (m, 6H), 1.28-1.45 (m, 26H), 1.78 (m, 4H), 2.26 (s, 3H, Ac), 2.40 (s, 3H, Ac), 3.66 (s, 1H, CCH), 3.74 (b, 4H), 4.27 (m, 4H, CH₂O), 7.52 (s, 1H, Ar-H), 7.74 (s, 1H, Ar-H), 7.96 (s, 1H, NH), 8.84 (s, 1H, Ar-H), 9.12 (s, 1H, NH), 9.18 (s, 1H, Ar-H). ¹³C NMR (125 MHz, CDCl₃): δ 14.27, 22.84, 24.69, 25.04, 26.11, 28.73, 29.35, 29.37, 29.41, 29.49, 31.97, 32.01, 65.51, 66.22, 77.72, 87.54, 90.42, 94.60, 114.10, 114.90, 118.68, 119.60, 121.36, 121.74, 128.81, 131.61, 137.44, 137.79, 139.32, 144.59, 165.42, 168.37, 168.60, 169.82.

2-[2-(2-Methoxyethoxy) ethoxy]ethyl 2-iodobenzoate (1r). To a solution of 2-iodobenzoic acid 1q (1.26 g, 5.08 mmol) and triethylamine (2.5 mL) in dichloromethane (20 mL) was added dropwise trimethylacetyl chloride (1.8 mL) at 0°C. The reaction solution was allowed to warm to room temperature and stirred for 1h. Then the solution was then cooled in an ice-water bath, to which a solution of triethyleneglycol monomethyl ether (1.05 g, 6.40 mmol) in dichloromethane (10 mL) was added dropwise. The reaction mixture was allowed to stir for 12 h at rt., washed with water, dried over Na₂SO₄, filtered, and concentrated to afford a red oil. The oil was purified by silica gel column chromatography (petroleum ether /EtOAc, 3/1) to afford 1.92 g (96.0 %) of 1r as a pale yellow oil. TLC, Rf = 0.48 (CHCl₃/acetone, 10/1). ¹H NMR (500 MHz, CDCl₃): δ 3.35 (s, 3H, MeO), 3.52 (m, 2H), 3.63-3.85 (m, 6H), 3.85 (t, 2H), 4.89 (t, 2H), 7.15 (m, 1H), 7.40 (m, 1H), 7.83 (d, 1H, J = 7.5 Hz), 8.15 (d, 1H, J = 8.0 Hz), 8.87 (s, 1H), 9.76 (d, 1H). ¹³C NMR (125 MHz, CDCl₃): δ 27.34, 59.16, 63.67, 64.80, 69.10, 70.78, 72.08, 94.29, 128.06, 131.26, 132.84, 15.19, 141.40, 166.51.

Dimer 2d. A mixture of the acetylene 1l (78 mg, 0.25 mmol), 1r (112 mg, 0.28 mmol), bis(dibenzylideneacetone) palladium (4.6 mg, 0.0053 mmol), copper (I) iodine (1 mg, 0.0052 mmol), and triphenylphosphine (6.9 mg, 0.025 mmol) in triethylamine (15 mL) was heated at 70°C under argon for 12 h. The solution was concentrated to yield a brown oil, which was purified by PTLC (CH₂Cl₂/acetone, 10/1) to afford 130 mg (89.1%) of 2d as a yellow solid. TLC, Rf = 0.34 (CHCl₃/acetone, 8/1). ¹H NMR (500 MHz, CDCl₃): δ 1.28 (m, 6H), 2.40 (s, 3H, Ac), 3.36 (s, MeO, 3H), 3.52 (t, CH₂O, 2H), 3.63-3.76 (m, 10 H), 3.87 (m, 5H), 4.48 (t, CH₂O, 2H), 7.43 (t, 1H), 7.58 (m, 2H), 7.71 (d, 1H, J = 7.5 Hz), 8.15 (d, 1H, J = 8.0 Hz), 8.87 (s, 1H), 9.27 (s, 1H). ¹³C NMR (125 MHz, CDCl₃): δ 24.76, 52.22, 59.22, 64.85, 69.18, 70.82, 70.86, 70.99, 72.13, 90.79, 95.60, 114.50, 119.89, 122.02, 124.17, 128.14, 128.55, 130.21, 131.13, 132.73, 134.21, 137.77, 144.90, 165.85, 168.54, 169.77.

1H), 8.03 (s, 1H), 8.84 (s, 1H), 9.16 (s, 1H), 9.22 (s, 1H). ¹³C NMR (125 MHz, CDCl₃): δ -0.009, 14.33, 22.87, 24.74, 25.08, 26.13, 26.19, 28.74, 28.91, 29.38, 29.40, 29.43, 29.52, 31.99, 32.04, 65.53, 66.23, 90.41, 94.73, 98.43, 106.47, 114.17, 118.66, 119.62, 120.79, 121.69, 128.77, 131.33, 136.83, 137.80, 139.06, 144.63, 165.52, 168.32, 169.88.
**Trimer 3.** Following the coupling procedure as described for 2e, the mixture of the acetylene 2f (139 mg, 0.19 mmol), 1r (102 mg, 0.26 mmol), bis(dibenzylideneacetone) palladium (3.5 mg, 0.0038 mmol), copper (I) iodine (1 mg, 0.0052 mmol), and triphenylphosphine (5.2 mg, 0.019 mmol) in triethylamine (10 mL) was heated at 70 °C under argon for 5 h. The solution was then filtered and concentrated to yield a dark oil, which was purified by silica gel column chromatography (dichloromethane/MeOH: 30/1, 20/1) to afford 131 mg (69.4%) of 3 as a yellow solid. TLC, Rf = 0.45 (CHCl3/acetone, 10/1). 1H NMR (500 MHz, CDCl3): δ 0.88 (6H, s), 1.39 (26H, m), 1.74 (2H, m), 1.82 (2H, m), 2.42 (3H, Ac), 2.45 (3H, Ac), 3.36 (3H, MeO), 3.53 (2H, m), 3.67-3.76 (10H, m), 3.87 (2H, t), 4.27 (2H, t), 4.32 (2H, t), 4.49 (2H, t), 7.58 (1H, s), 7.65 (1H, Ar-H), 7.72 (d, 1H, J = 7.5 Hz), 8.17 (d, 1H, J = 8.0 Hz), 8.86 (s, 1H), 9.27 (s, 1H), 9.38 (s, 1H), 9.45 (s, 1H). 13C NMR (125 MHz, CDCl3): δ 14.09, 22.65, 24.49, 24.54, 24.63, 24.68, 25.92, 25.97, 28.56, 28.70, 29.17, 29.22, 29.30, 31.79, 31.82, 59.06, 64.79, 65.28, 65.94, 68.94, 70.65, 70.68, 70.82, 71.94, 89.01, 89.85, 94.79, 97.40, 114.15, 115.57, 117.90, 121.25, 121.46, 121.52, 123.39, 128.44, 130.20, 131.09, 131.20, 132.73, 134.12, 136.84, 136.88, 137.62, 140.57, 144.45, 165.56, 165.69, 168.08, 169.68, 170.11. Anal. Calcd for C56H75N5O11. C, 67.65; H, 7.60; N, 7.04. Found C, 67.80; H, 7.63; N, 7.09.

**Trimer 3a.** To a sealed tube was added with trimer 3 (0.23 g, 0.23 mmol) and iodomethane (1.5 mL). The mixture was stirred for 24 h at 120 °C to yield a brown residue. The residue was then purified by silica gel column chromatography (dichloromethane/MeOH: 15/1, 10/1) to afford 173 mg (73.7 %) of 3a as a yellow solid. TLC, Rf = 0.52 (CHCl3/acetone, 10/1). 1H NMR (500 MHz, CDCl3): δ 0.88 (6H, m), 1.29-1.46 (20H, m), 1.81 (2H, m), 2.41 (3H, Ac), 2.44 (3H, Ac), 3.36 (3H, MeO), 3.52 (2H, t), 3.64-3.73 (6H, m), 3.88 (2H, t), 4.31 (4H, m), 4.46 (2H, m), 7.47 (1H, Ar-H), 7.59 (1H, Ar-H), 7.67 (1H, J = 7.5 Hz), 7.75 (1H, Ar-H), 8.01 (1H, Ar-H), 8.15 (1H, J = 8 Hz), 9.08 (s, 1H, Ar-H), 9.27 (s, 1H, NH), 9.38 (1H, Ar-H), 9.40 (s, 1H, NH). 13C NMR (125 MHz, CDCl3): δ 14.33, 22.87, 24.78, 24.81, 24.89, 26.13, 26.12, 28.72, 29.39, 29.43, 32.00, 32.02, 59.24, 59.27, 65.01, 66.18, 66.22, 69.11, 70.85, 71.00, 72.12, 84.79, 87.83, 88.95, 97.20, 97.94, 115.87, 116.17, 117.31, 121.40, 123.43, 129.17, 130.13, 131.11, 131.31, 132.98, 134.39, 135.87, 137.20, 140.96, 141.19, 143.27, 165.57, 165.73, 166.39, 170.30, 170.36.

**Tetramer 4.** Following the coupling procedure as described for trimer 3, the compound 1o (31 mg, 0.075 mmol), the trimer 3a (51 mg, 0.050 mmol), bis(dibenzyldieneacetone) palladium (1 mg, 0.0011 mmol), copper (I) iodine (0.5 mg, 0.0026 mmol), and triphenylphosphine (1.5 mg, 0.0056 mmol), in triethylamine (15 mL) was heated at 70 °C under argon for 28 h. The solution was then filtered and concentrated to give a dark oil, which was purified by silica gel column chromatography (dichloromethane/acetone: 10/1) to give 48.5 mg (74.3 %) of 4 as a yellow solid. TLC, Rf = 0.53 (CHCl3/acetone, 5/1). 1H NMR (500 MHz, CDCl3): δ 0.88 (9H, m), 1.29-1.46 (36H, m), 1.76 (2H, m), 1.83 (4H, m), 2.42 (3H, Ac), 2.47 (6H, Ac), 3.37 (3H, MeO), 3.53 (2H, m), 3.65-3.89 (12H, m), 4.27 (2H, CH2O), 4.33 (4H, CH2O), 4.51 (2H, t), 7.51 (1H, Ar-H, J = 8 Hz), 7.57 (1H, J = 7 Hz), 7.63 (1H, t), 7.75 (1H, J = 7 Hz), 8.17 (1H, s), 8.20 (1H, J = 7 Hz), 8.86 (s, 1H), 9.29 (s, 1H), 9.39 (s, 1H), 9.42 (s, 1H), 9.46 (s, 1H), 9.49 (s, 1H). 13C NMR (125 MHz, CDCl3):
δ 14.12, 22.66, 24.54, 24.62, 24.71, 25.92, 25.98, 28.54, 28.55, 28.70, 29.18, 29.24, 29.31, 31.80, 31.83, 59.07, 64.84, 65.29, 65.92, 66.09, 68.93, 70.65, 70.67, 71.92, 88.17, 88.78, 89.69, 94.92, 97.14, 97.71, 114.22, 115.74, 115.77, 117.12, 119.47, 121.22, 121.35, 121.53, 123.25, 128.33, 129.01, 130.14, 130.90, 131.14, 131.25, 132.74, 134.15, 136.60, 136.89, 137.00, 137.65, 140.54, 141.11, 144.41, 165.56, 165.66, 168.11, 169.76, 170.25, 170.27. Anal. Calcd for C75H98N6O14. C, 68.89; H, 7.55; N, 6.43; O, 17.43. Found C, 68.66; H, 7.56; N, 6.40.

**Pentamer 5.** A mixture of dimer 2f (47 mg, 0.065 mmol), trimer 3a (50.9 mg, 0.05 mmol), bis(dibenzylideneacetone) palladium (2 mg, 0.0022 mmol), copper (I) iodine (0.5 mg, 0.0026 mmol), and triphenylphosphine (3.1 mg, 0.012 mmol) in triethylamine (10 mL) was stirred at 70 °C under argon for 28 h. After removal of solvent, the resulting residue was purified by silica gel PTLC (dichloromethane/acetone: 10/1) to afford 64 mg (60.8 %) of yellow solid 5. TLC, Rf = 0.47 (CHCl3/acetone, 5/1). 1HNMR (500 MHz, CDCl3): δ 0.88 (s, 12H), 1.32 (m, 46H), 1.73 (m, 2H), 1.83 (m, 6H), 2.42 (s, 3H, Ac), 2.48 (s, 9H), 3.37 (s, 3H), 3.55 (t, 2H), 3.73 (m, 10 H), 3.89 (m, 2H), 4.32(m, 8H), 4.49 (t, 2H), 7.41 (t, 1H), 7.46 (t, 1H), 7.51 (s, 1H), 7.63 (d, 1H, J=8.0 Hz), 7.75 (s, 1H), 7.78, 7.80 (d, 2H), 8.15 (d, 1H, J= 7.5 Hz), 8.82 (s, 1H), 9.20 (s, 1H), 9.33, 9.36, 9.38, 9.39 (q, 4H), 9.42 (s, 1H). 13CNMR (125 MHz, CDCl3): δ 14.12, 22.68, 24.68, 24.75, 25.96, 28.58, 28.74, 29.21, 29.29, 29.33, 31.83, 35.90, 64.88, 65.25, 65.92, 66.08, 66.14, 68.96, 70.67, 70.70, 70.84, 71.96, 87.95, 88.03, 88.71, 89.59, 94.82, 95.01, 97.29, 97.48, 97.75, 114.25, 115.74, 115.80, 115.92, 117.02, 117.08, 117.78, 119.40, 121.08, 121.19, 121.28, 121.37, 121.44, 123.19, 128.32, 130.01, 130.69, 131.08, 131.15, 132.60, 136.60, 136.89, 137.10, 140.59, 141.07, 141.22, 144.28, 165.45, 165.58, 168.03, 169.75, 170.25, 170.27, 170.35. Anal. Calcd for C_{75}H_{98}N_{6}O_{14}. C, 68.89; H, 7.55; N, 6.43; O, 17.43. Found C, 68.66; H, 7.56; N, 6.40.

**Dimer 2g.** A mixture of 1m (0.52 mg, 1.25 mmol), 1o (0.58 g, 1.40 mmol), bis(dibenzylideneacetone) palladium (21 mg, 0.023 mmol), copper (I) iodine (4.4 mg, 0.023 mmol), and triphenylphosphine (31 mg, 0.12 mmol) in triethylamine (30 mL) was stirred at 70 °C under argon for 12 h. The solution was then filtered and concentrated to yield a brown residue, which was purified by silica gel column (petroleum/EtOAc: 3/2) to afford 0.81g (92.4 %) of the dimer 2g as a yellow solid. TLC, Rf = 0.47 (petroleum ether/EtOAc, 1/1). 1H NMR (500 MHz, CDCl3): δ 0.33 (s, 9H, SiMe3), 0.88 (t, 3H), 1.29 (m, 16 H), 1.74 (t, 2H, CH2), 2.25 (s, 3H, Ac), 2.42 (s, 3H, Ac), 3.75 (b, 4H, CH2), 3.94 (s, 3H, MeO), 4.27 (t, 2H, CH2O), 7.54 (s, 1H, Ar-H), 7.75 (s, 1H, Ar-H), 8.03 (s, 1H, NH), 8.88 (s, 1H, Ar-H), 9.12 (s, 1H, Ar-H), 9.21 (s, 1H, NH). 13C NMR (125 MHz, CDCl3): δ -0.032, 14.29, 22.85, 24.72, 25.03, 26.18, 28.91, 29.38, 29.50, 32.02, 52.89, 65.50, 90.50, 94.61, 98.40, 106.57, 114.10, 118.76, 119.64, 120.90, 120.90, 121.71, 128.86, 130.84, 136.84, 137.79, 139.03, 144.62, 165.94, 168.29, 168.35, 169.81. Anal. Calcd for C_{38}H_{51}N_{5}O_{6}Si. C, 65.02; H, 7.32; N, 10.13. Found C, 64.87; H, 7.22; N, 10.13.

**Dimer 2h.** To a solution of the dimer 2g (0.57 g, 0.81 mmol) in MeOH (10 mL) was added potassium carbonate (6 mg, 0.043 mmol) and the reaction was carried out as described for 2f to afford 0.46 g (90.2 %) of the desired 2h as a yellow oil. TLC, Rf = 0.55 (petroleum ether/EtOAc, 1/3). 1H NMR (500 MHz, CDCl3): δ 0.88 (t, 3H), 1.29 (m, 16 H), 1.74 (m, 2H), 2.26 (s, 3H, Ac), 2.42 (s, 3H, Ac).
Trimer 3b. A mixture of 2h (263 mg, 0.42 mmol), 1p (300 mg, 0.58 mmol), bis(dibenzylideneacetone) palladium (7.6 mg, 0.0083 mmol), copper (I) iodine (2 mg, 0.01 mmol), and triphenylphosphine (15 mg, 0.056 mmol) in dry triethylamine (28 mL) was stirred at 70 °C under argon for 24 h. The solution was then filtered and concentrated to yield a brown oil, which was purified by silica gel column (petroleum ether/dichloromethane/MeOH: 3/1/0.5) to afford 280 mg (66.0 %) of 3b as a yellow oil. TLC, Rf = 0.51 (petroleum ether/CHCl3/acetone, 1/1/0.5). 1H NMR (500 MHz, CDCl3): 0.88 (m, 6H), 1.29-1.46 (m, 26 H), 1.75 (t, 2H), 1.81 (t, 2H), 2.26 (s, 3H, Ac), 2.42 (s, 6H, Ac), 3.75 (b, 4H, MeO), 4.29 (m, 4H, CH2O), 7.54 (s, 1H, Ar-H), 7.73 (s, 1H, Ar-H), 8.01 (s, 1H, NH), 8.87 (s, 1H, Ar-H), 9.17 (s, 1H), 9.22 (s, 1H, NH), 9.32 (s, 1H, NH), 9.34 (s, 1H, Ar-H). 13C NMR (125 MHz, CDCl3): δ -0.05, 14.28, 22.66, 22.71, 24.65, 24.72, 25.75, 26.01, 28.54, 28.56, 28.68, 29.22, 29.32, 29.38, 29.39, 31.83, 31.86, 52.64, 59.01, 64.86, 64.98, 66.08, 66.13, 66.22, 68.96, 70.64, 70.66, 70.76, 71.93, 78.03, 87.69, 87.74, 87.79, 88.09, 89.51, 94.43, 97.34, 97.68, 97.73, 97.98, 113.58, 115.77, 115.79, 115.85, 115.89, 117.02, 117.12, 117.85, 118.65, 120.62, 120.79, 122.69, 128.52, 129.09, 129.45, 130.05, 130.11, 130.24, 130.65, 132.20, 134.29,
136.80, 137.19, 137.47, 140.39, 140.97, 141.11, 143.48, 164.76, 164.81, 164.89, 164.99, 165.50, 167.94, 169.87, 170.27, 170.32, 170.40, 170.45. Anal. Calcd for C\textsubscript{106}H\textsubscript{130}N\textsubscript{8}O\textsubscript{20}. C, 69.33; H, 7.14; N, 6.10. Found C, 69.26; H, 7.21; N, 5.87.