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A Click Reaction for Fluorescent Labelling: Application of the 1,3-Dipolar Cycloaddition Reaction

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The copper-catalysed 1,3-dipolar cycloaddition of azides with terminal alkynes was use to introduce a highly fluorescent and photostable perylene-tetracarboxdiimide label. Fluorescent labelling of human hormones and applications are discussed.

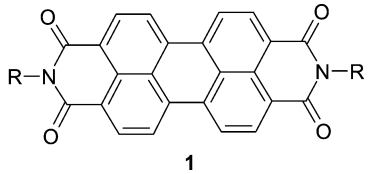
Introduction

Fluorescence labelling is becoming more and more important for chemistry, because of its simple and sensitive detection.[1] This is of special interest for pharmacologically active substrates. However, individual methods have to be developed to introduce the label in the various types of such compounds. A universal and efficient method capable of introducing a fluorescent label into these various compounds would bring appreciable progress to this area. The 1,3-dipolar cycloaddition[2] is of interest for such a labelling procedure, because the reaction can be manipulated to tolerate the majority of functional groups.

Results and Discussion

We applied the cycloaddition of azides with terminal alkynes[3] for the linking of substrates with fluorophores and catalysed[4] the reaction with copper(I) salts. This type of cycloaddition is well known as the "click reaction",[5] because of its efficiency and rapidity.

The azide group can be introduced into various substrates by nucleophilic displacement reactions and requires a terminal alkyne for labelling. Perylene-3,4:9,10-tetracarboxdiimides **1** are suitable fluorophores[6] to act as labels, because they posses extraordinarily high photostability, fluorescence quantum yields[7] close to 100%, thermal stability and chemical inertness and tolerance to various reactive reagents.



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Bifunctional derivatives of **1** were applied for insertion into oligonucleotides,[8] and tetrafunctionalised derivatives[8a] were used for the construction of light-harvesting systems by the "click reaction"; the low solubility of such structures[8c] proved to be a problem (for further information see also ref.[9]). Readily soluble, monofunctionalised derivatives are still lacking. For the labelling experiments, we started with anhydride carboximide **2**, where the long-chain secondary 1-hexylheptyl group ("swallow-tail substituent"[10]) was attached to the nitrogen atom for solubilisation, and it was condensed with 4-aminophenylacetylene to form **3a**. The "click-reaction" of this terminal alkyne was demonstrated with benzyl azide, which afforded labelled compound **4a** (Scheme 1). As an alternative, propargylic amine was condensed with **2** to form lower homologue **3b**, where the chromophore is closer to the linking position. The "click-reaction" of the latter proceeds similarly to the first example as was shown with **4b** and **4c**.

Linking with **3** requires the introduction of an azide group into the substrate; this may be a limitation for some substrates. As an alternative, the two linking structures may be interchanged for substrates where alkyne structures can be introduced. To this end, we condensed (4-aminomethylphenyl)methanol with **2**, activated the resulting alcohol **5** by bromination and converted it into azide **6**. The "click-reaction" of the latter to various labelled compounds **7** was successful, even with Michael systems (**7e**). Functional groups such as ether groups (**7a** and **7b**) or amino groups (**7c** and **7d**) do not interfere with the reaction. Restrictions are given by severe steric hindrance with *tert*-butyl groups (3,3-dimethylbut-1-yne) or for phenyl acetylene, and iodated triazoles are often formed as byproducts (compare ref.[11]). The usefulness of the reaction is demonstrated with the labelling of the pharmacologically active compounds

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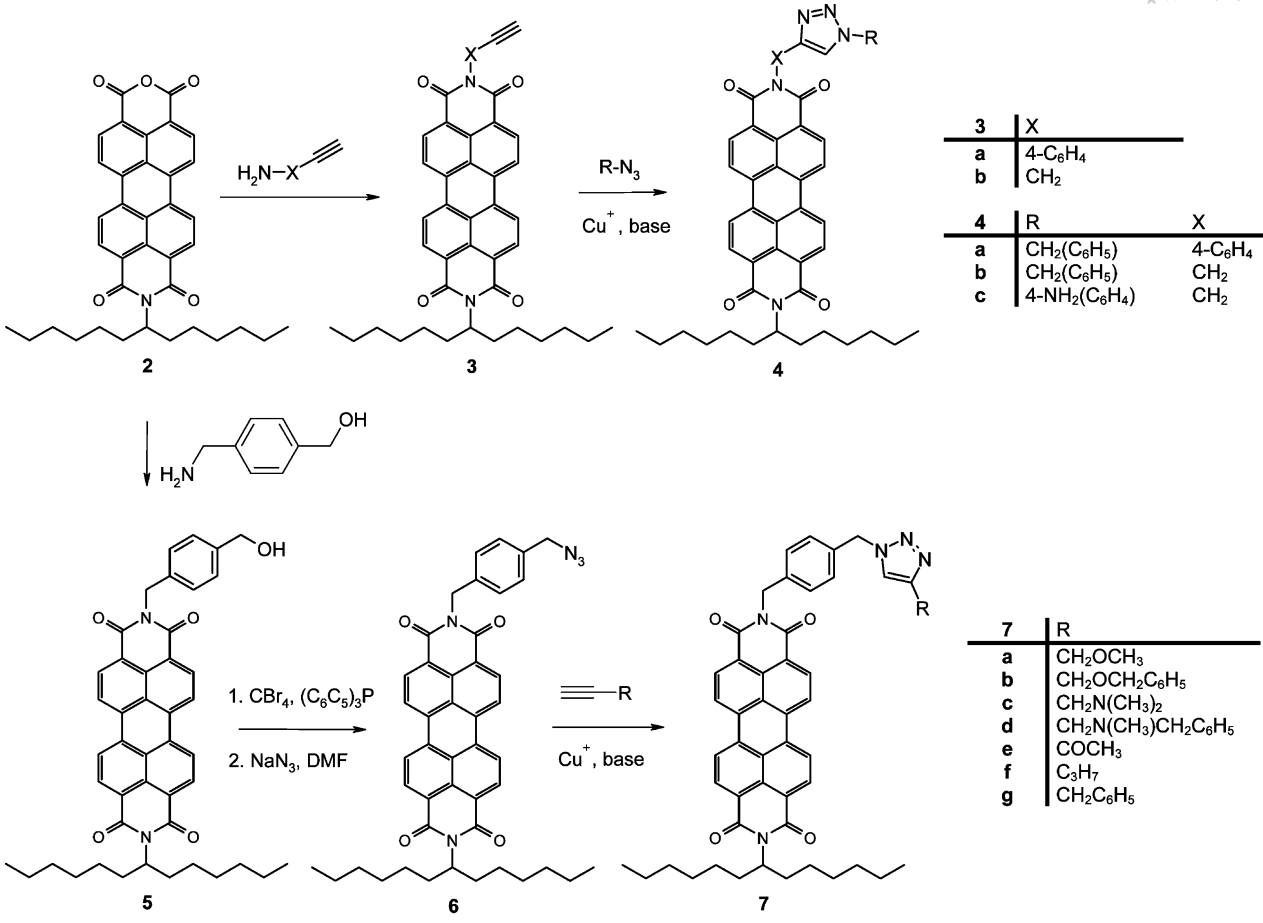


3	X
a	4-C_6H_4
b	CH_2

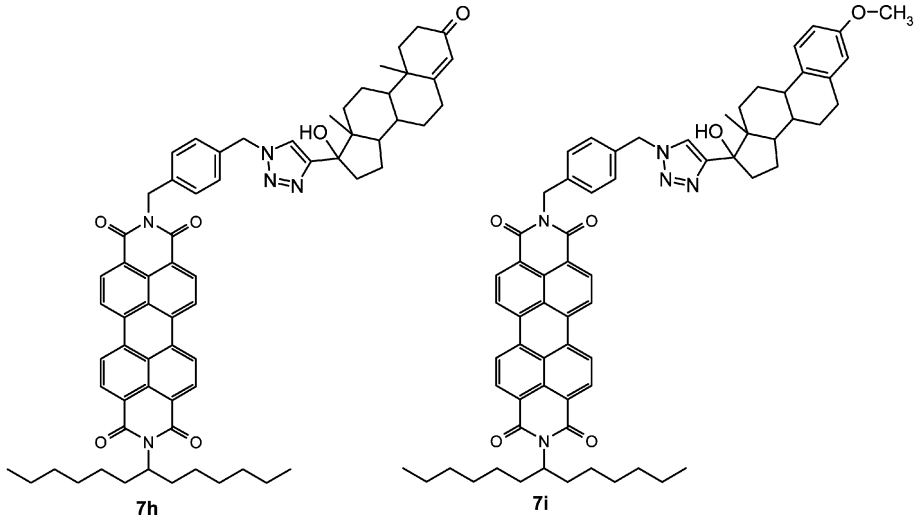
4	R	X
a	$CH_2(C_6H_5)$	4-C_6H_4
b	$CH_2(C_6H_5)$	CH_2
c	4-$NH_2(C_6H_4)$	CH_2

7	R
a	CH_2OCH_3
b	$CH_2OCH_2C_6H_5$
c	$CH_2N(CH_3)_2$
d	$CH_2N(CH_3)CH_2C_6H_5$
e	$COCH_3$
f	C_3H_7
g	$CH_2C_6H_5$

Scheme 1. Labelling with perylene fluorophores.

ethisterone and mestranol to form **7h** and **7i**, respectively. Such structures are of interest for the determination of hormones by competitive receptor assays, for example, for the detection of their illegal use such as for doping applications or for the detection of endocrinic disruptors in the environment.

The UV/Vis absorption and fluorescence spectra of labelled compounds **4** and **7** are congruent with those of **1** with aliphatic R substituents[12] (see Figure 1). The fluorescence quantum yields of all derivatives are close to 100%, which is a good prerequisite for sensitive detection (see Experimental Section). The high fluorescence quantum yields of



7c and **7d**, respectively, are remarkable, because electron transfer from the electron-rich amino group to the electronically excited chromophore could proceed, which would cause fluorescence quenching.[13] Obviously, there is no such interference, and thus, the fluorescence labelling of a broad variety of substrates by means of **3** and **6** becomes possible. The labelled compounds are readily soluble in lipophilic solvents. For more hydrophilic media, mixtures between DMSO and water[8c] may be used, as could micelle-forming detergents.[14]

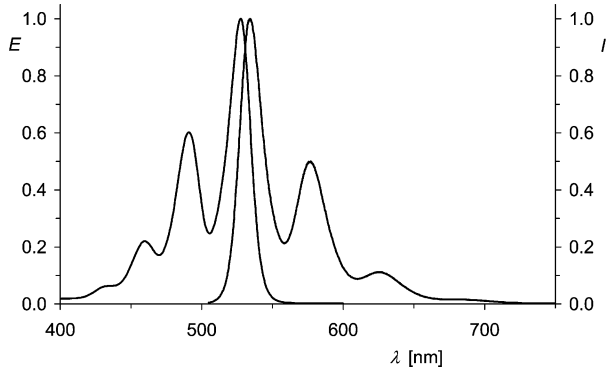


Figure 1. Absorption (left, *E*) and fluorescence (right, *I*) spectrum of **7i** in chloroform.

Conclusions

The "click variant" of the 1,3-dipolar cycloaddition is a useful method to introduce a fluorescent label into various complex structures. The mild reaction conditions make this method suitable for labile natural products or their analogous. Proximity of the chromophore to the substrate and electron-rich groups do not interfere with high fluorescence quantum yields or with the labelling reaction.

Experimental Section

General: IR spectra: Perkin–Elmer 1420 Ratio Recording Infrared Spektrometer, FT 1000. UV/Vis spectra: Varian Cary 5000 and Bruins Omega 20; fluorescence spectra: Perkin–Elmer FS 3000 (totally corrected); NMR spectroscopy: Varian Vnmrs 600 (600 MHz); mass spectrometry: Finnigan MAT 95.

***N*-[1-(4-Ethynylphenyl)]-*N*′-(1-hexylheptyl)perylene-3,4:9,10-tetracarboxdiimide (3a):** A mixture of *N*-(1-hexylheptyl)perylene-3,4:9,10-tetracarboxylic-3,4-anhydride-9,10-carboximide (**2**, 1.50 g, 2.62 mmol),[15] 4-ethynylaniline (378 mg, 3.14 mmol) and imidazole (10.0 g) was heated at reflux (bath 100 °C) for 90 min, cooled and treated with a small amount of ethanol. The crude material was precipitated by the addition of 2 N HCl, and the solid was collected by vacuum filtration, washed with 2 N HCl and dried at 110 °C in air. The crude product was purified by column separation (silica gel, chloroform), dissolved in a small amount of dichloromethane and precipitated with methanol. The precipitate was collected by vacuum filtration and dried at 110 °C in air. Yield: 543 mg (31%), dark-red solid, m.p. >300 °C. R_f (silica gel, chloroform) = 0.09. IR (KBr): $\tilde{\nu}$ = 2955 (m), 2923 (m), 2855 (m), 1696 (s), 1655 (s), 1592 (s), 1576 (s), 1506 (m), 1483 (w), 1458 (w), 1432 (m), 1403 (m), 1340 (s), 1301 (m), 1251 (s), 1196 (m), 1174 (s), 1137 (m), 1124 (m), 1108 (m), 1020 (w), 966 (m), 890 (w), 840 (m), 808 (s), 795 (s), 744 (s), 725 (m) cm^{-1}. ^1H NMR (600 MHz, $CDCl_3$, 25 °C): δ = 0.83 (t, $^3J_{H,H}$ = 7.1 Hz, 6 H, CH_3), 1.21–1.36 (m, 16 H, CH_2), 1.85–1.91 (m, 2 H, β-CH_2), 2.22–2.28 (m, 2 H, β-CH_2), 3.16 (s, 1 H, C≡CH), 5.19 (tt, $^3J_{H,H}$ = 5.8 Hz, $^3J_{H,H}$ = 9.3 Hz, 1 H, α-CH), 7.33–7.70 (m, 4 H, H_{arom}), 8.62–8.73 (m, 8 H, $H_{perylene}$) ppm. ^{13}C NMR (150 MHz, $CDCl_3$, 25 °C): δ = 14.04, 22.58, 26.94, 29.21, 31.76, 32.37, 54.85, 78.44, 83.16, 123.14, 123.26, 123.58, 126.61, 126.90, 129.00, 129.73, 130.05, 130.06, 131.30, 132.10, 133.39, 134.42, 135.48, 135.59, 163.57 ppm. UV/Vis ($CHCl_3$): λ_{max} (E_{rel}) = 459 (0.22), 490 (0.61), 527 (1.0) nm. Fluorescence ($CHCl_3$): λ_{max} (I_{rel}) = 539 (1.0), 582 (0.40) nm. Fluorescence quantum yield ($CHCl_3$, λ_{exc} = 491 nm, E_{491nm} = 0.0257 cm^{-1}, **1a** with Φ = 1.00): 1.00. HRMS: calcd. for $C_{45}H_{40}N_2O_4$ 672.298; found 672.297, Δ = 1 mmu. $C_{45}H_{40}N_2O_4$ (672.8): calcd. C 80.33, H 5.90, N 4.16; found C 79.10, H 5.92, N 4.03.

***N*-[4-(1-Benzyl-1*H*-[1,2,3]triazol-4-yl)phenyl]-*N*′-(1-hexylheptyl)perylene-3,4:9,10-tetracarboxdiimide (4a):** *N*-(4-Ethynylphenyl)-*N*′-(1-hexylheptyl)perylene-3,4:9,10-tetracarboxdiimide (**3**, 100 mg, 0.149 mmol) was dissolved in THF (20 mL) and treated with benzyl azide (19.8 mg, 0.149 mmol), copper(I)iodide (28.4 mg, 0.149 mmol) and *N*-ethylbis(isopropyl)amine (19.3 mg, 0.149 mmol). The mixture was stirred at room temperature for 2 d and then filtered. The solvent was evaporated in vacuo, and the residue was purified by column separation (silica gel; dichloromethane/methanol, 100:1; elution of the starting material and one by-product) and the resulting material was dissolved in a small amount of dichloromethane, precipitated with methanol, collected by vacuum filtration and dried at 110 °C in air. Yield: 20.0 mg (17%), red dye, m.p.>300 °C. R_f (silica gel; dichloromethane/methanol, 30:1) = 0.69. IR (KBr): $\tilde{\nu}$ = 2953.5 (m), 2921.9 (m), 2853.4 (m), 1690.2 (m), 1644.8 (s), 1591.5 (s), 1576.0 (m), 1505.1 (w), 1480.9 (w), 1455.9 (w), 1434.0 (w), 1403.5 (w), 1343.0 (m), 1249.8 (m), 1199.0 (w), 1175.9 (m), 1126.3 (w), 1107.0 (w), 1076.1 (w), 1058.3 (w), 1024.7 (w), 980.0 (w), 964.2 (w), 863.3 (w), 843.5 (w), 810.8 (w), 796.0 (w), 746.8 (w), 727.8 (w), 696.0 (w) cm^{-1}. ^1H NMR (600 Hz, $CDCl_3$, 25 °C): δ = 0.83 (t, $^3J_{H,H}$ = 7.1 Hz, 6 H, CH_3), 1.23–1.36 (m, 16 H, CH_2), 1.85–1.90 (m, 2 H, β-CH_2), 2.22–2.29 (m, 2 H, β-CH_2), 5.17–5.22 (m, 1 H, α-CH), 5.73 (s, 2 H, CH_2), 7.33–7.47 (m, 9 H, H_{arom}), 8.21 (s, 1 H, C=CH-N), 8.68–8.77 (m, 8 H, $H_{perylene}$) ppm. ^{13}C NMR (150 MHz, $CDCl_3$, 25 °C): δ = 14.04, 22.58, 26.93, 29.21, 29.70, 31.76, 32.38, 55.05, 123.11, 123.25, 123.38, 127.80, 128.25, 128.54, 128.89, 129.00, 129.58, 129.92, 131.95, 135.28, 163.55 ppm. UV/Vis ($CHCl_3$): λ_{max} (E_{rel}) = 459 (0.23), 490 (0.61), 527 (1.0) nm. Fluorescence ($CHCl_3$): λ_{max} (I_{rel}) = 539 (1.0), 582 (0.40) nm. Fluorescence quantum yield ($CHCl_3$, λ_{exc} = 491 nm, E_{491nm} = 0.0257 cm^{-1}, reference: **1a** with Φ = 1.00): 1.00. HRMS: calcd. for $C_{52}H_{47}N_5O_4$ 805.362; found 805.360, Δ = 2 mmu.

(1-Hexylheptyl)-*N*′-propargylperylene-3,4:9,10-tetracarboxdiimide (3b): A mixture of *N*-(1-hexylheptyl)perylene-3,4:9,10-tetracarboxylic-3,4-anhydride-9,10-carboximide (**2**, 1.14 g, 1.98 mmol)[15] and imidazole (20 g) were heated at 90 °C, treated with propargylamine (2 mL), and kept at that temperature for 2 h. The mixture was then treated with a few mL of ethanol, quenched by the addition of 2 N HCl (100 mL) and cooled. The solid was collected by vacuum filtration, washed with 2 N HCl, dried in air at 110 °C for 16 h, dissolved in a small amount of chloroform and purified by column separation (silica gel, chloroform). Yield: 780 mg (64%), red dye, m.p. >300 °C. R_f (silica gel, chloroform) = 0.12. IR (ATR): $\tilde{\nu}$ = 3253.3 (m), 2925.0 (m), 2922.6 (m), 2855.5 (m), 1694.0 (m), 1654.3

(s), 1593.3 (s), 1578.6 (m), 1506.4 (w), 1464.0 (w), 1433.4 (m), 1403.3 (m), 1353.0 (w), 1334.5 (s), 1246.1 (m), 1171.5 (m), 1134.6 (w), 1124.5 (w), 1097.0 (w), 984.2 (w), 852.8 (w), 845.2 (w), 807.5 (s), 746.0 (m), 721.1 (w), 633.9 (w) cm^{-1}. ^1H NMR (600 MHz, $CDCl_3$, 25 °C): δ = 0.82 (t, ^3J = 7.0 Hz, 6 H, CH_3), 1.22–1.36 (m, 16 H, CH_2), 1.84–1.90 (m, 2 H, β-CH_2), 2.22–2.28 (m, 2 H, β-CH_2), 2.24 (s, 1 H, C≡CH), 4.98 (d, ^4J = 2.4 Hz, 2 H, CH_2), 5.10–5.28 (m, 1 H, α-CH), 8.50–8.66 (m, 8 H, $H_{perylene}$) ppm. ^{13}C NMR (150 MHz, $CDCl_3$, 25 °C): δ = 14.0, 22.6, 26.9, 29.2, 29.6, 31.8, 32.4, 54.9, 70.8, 78.3, 122.7, 122.9, 123.2, 126.2, 126.4, 129.4, 131.7, 135.0, 162.5 ppm. UV/Vis ($CHCl_3$): λ_{max} (E_{rel}) = 459 (0.22), 490 (0.60), 526 (1.0) nm. Fluorescence ($CHCl_3$): λ_{max} (I_{rel}) = 539 (1.0), 582 (0.40) nm. Fluorescence quantum yield ($CHCl_3$, λ_{exc} = 491 nm, E_{491nm} = 0.0285 cm^{-1}, reference: **1a** with Φ = 1.00): 1.00. HRMS: calcd. for $C_{40}H_{38}N_2O_4$ 610.284; found 610.285, Δ = 1 mmu.

***N*-{1-[(4-Aminophenyl)-1*H*-[1,2,3]triazol-4-yl]methyl}-*N*′-(1-hexylheptyl)perylene-3,4:9,10-tetracarboxdiimide (4c):** Prepared according to the procedure outline for **4a** with the use of *N*-(1-hexylheptyl)-*N*′-(propargyl)perylene-3,4:9,10-tetracarboxdiimide (**3b**, 500 mg, 0.819 mmol), 4-azidoaniline hydrochloride (140 mg, 0.819 mmol), copper(I)iodide (312 mg, 1.64 mmol) and *N*-ethylbis(isopropyl)amine (318 mg, 2.46 mmol) in THF (125 mL). The residue was dissolved in dichloromethane, purified by column separation (silica gel; dichloromethane/methanol, 50:1; elution of the starting material and 10:1 for collecting the product). The resulting product was dissolved in a small amount of dichloromethane, precipitated with methanol, collected by vacuum filtration and dried in air at 110 °C. Yield: 289 mg (47%), m.p. >300 °C. R_f (silica gel; dichloromethane/methanol, 30:1) = 0.43. IR (KBr): $\tilde{\nu}$ = 3368.0 (w), 2952.3 (m), 2923.4 (m), 2854.6 (m), 1692.6 (s), 1650 (s), 1592.3 (s), 1576.2 (s), 1519.7 (m), 1483.9 (w), 1456.4 (w), 1435.2 (m), 1403.2 (m), 1376.7 (w), 1335.5 (s), 1299.9 (m), 1249.5 (m), 1195.0 (w), 1171.6 (m), 1127.8 (w), 1096.2 (w), 1052.2 (w), 990.5 (w), 935.5 (w), 851.5 (w), 829.9 (m), 808.1 (s), 796.2 (w), 769.7 (w), 752.2 (m), 740.1 (m), 671.4 (w), 692.0 (w), 671.4 (w), 635.5 (w), 612.4 (w), 588.2 (w) cm^{-1}. ^1H NMR (600 MHz, $CDCl_3$, 25 °C): δ = 0.83 (t, ^3J = 6.9 Hz, 6 H, CH_3), 1.21–1.36 (m, 16 H, CH_2), 1.85–1.90 (m, 2 H, β-CH_2), 2.22–2.29 (m, 2 H, β-CH_2), 3.95 (s, 1 H, NH_2), 5.19 (tt, ^3J = 5.8 Hz, ^3J = 9.3 Hz, 1 H, α-CH), 5.52 (s, 2 H, CH_2-N), 6.94–7.08 (m, 4 H, H_{arom}), 8.53–8.65 (m, 8 H, $H_{perylene}$) ppm. UV/Vis ($CHCl_3$): λ_{max} (E_{rel}) = 458 (0.22), 490 (0.60), 527 (1.0) nm. HRMS: calcd. for $C_{46}H_{44}N_6O_4$ 745.350; found 745.351, Δ = 1 mmu.

***N*-{1-(Benzyl-1*H*-[1,2,3]triazol-4-yl)methyl}-*N*′-(1-hexylheptyl)perylene-3,4:9,10-tetracarboxdiimide (4b):** Prepared according to the procedure outline for **4c** with the use of *N*-(1-hexylheptyl)-*N*′-(propargyl)perylene-3,4:9,10-tetracarboxdiimide (**3b**, 100 mg, 0.164 mmol), benzyl azide (22.0 mg, 0.164 mmol), copper(I)iodide (42.0 mg, 0.328 mmol) and *N*-ethylbis(isopropyl)amine (60.0 mg, 0.328 mmol). Yield: 84 mg (69%), red dye, m.p. >300 °C. R_f (silica gel; chloroform/methanol, 30:1) = 0.12. IR (ATR): $\tilde{\nu}$ = 2952.2 (m), 2922.1 (m), 2854.1 (m), 1687.6 (m), 1642.9 (s), 1592.6 (s), 1577.6 (m), 1505.9 (w), 1456.9 (w), 1433.8 (m), 1402.9 (m), 1341.1 (s), 1248.0 (m), 1216.6 (w), 1166.5 (m), 1125.0 (w), 1050.9 (w), 986.3 (w), 848.9 (w), 809.1 (s), 793.8 (w), 764.3 (w), 752.4 (m), 705.1 (w), 623.9 (w) cm^{-1}. ^1H NMR (600 MHz, $CDCl_3$, 25 °C): δ = 0.82 (t, ^3J = 7.0 Hz, 6 H, CH_3), 1.22–1.36 (m, 16 H, CH_2), 1.84–1.90 (m, 2 H, β-CH_2), 2.22–2.28 (m, 2 H, β-CH_2), 5.18 (tt, ^3J = 5.8 Hz, ^3J = 9.4 Hz, 1 H, CH-N), 5.47 (s, 2 H, CH_2-N), 5.57 (s, 2 H, CH_2-N), 7.31–7.36 (m, 5 H, H_{aryl}), 7.74 (s, 1 H, $H_{triazole}$), 8.19–8.48 (m, 8 H, $H_{perylene}$) ppm. ^{13}C NMR (150 MHz, $CDCl_3$, 25 °C): δ = 14.0, 22.6, 27.0, 29.2, 31.8, 32.4, 54.4, 54.8, 122.6, 122.6, 122.9, 123.7, 125.8, 125.9, 128.3, 128.7, 129.0, 129.1, 129.2, 131.2, 134.4, 134.5, 143.4, 162.7 ppm. UV/Vis ($CHCl_3$): λ_{max} (E_{rel}) = 459 (0.22), 490 (0.61), 527 (1.0) nm. Fluorescence ($CHCl_3$): λ_{max} (I_{rel}) = 539 (1.0), 582 (0.40) nm. Fluorescence quantum yield ($CHCl_3$, λ_{exc} = 491 nm, E_{491nm} = 0.0232 cm^{-1}, reference: **1a** with 1.00): 1.00. HRMS: calcd. for $C_{47}H_{46}N_5O_4$ 744.355; found 744.353, Δ = 2 mmu.

(4-Aminomethylphenyl)methanol: The synthetic procedure of ref.[16] was followed; however, it required some modifications: $LiAlH_4$ (5.8 g, 152.5 mmol) was dispersed in THF (100 mL), treated dropwise with 4-cyanobenzaldehyde (5.00 g, 38.1 mmol) in THF (50 mL), heated at reflux with stirring for 5 h, cooled, treated with 2 M NaOH, separated from the solid by filtration, treated with dichloromethane (2 × 400 mL), dried with $MgSO_4$, filtered and evaporated. The product was pure enough for the subsequent condensation. Yield: 3.28 g (57%), colourless solid, m.p. 79 °C. ^1H NMR (200 MHz, $CDCl_3$, 25 °C): δ = 3.77 (s, 2 H, CH_2NH_2), 4.60 (s, 2 H, CH_2OH), 7.14–7.31 (m, 4 H, H_{aryl}) ppm. HRMS: calcd. for $C_8H_{11}NO$ 137.084; found 137.082, Δ = 2 mmu.

***N*-(1-Hexylheptyl)-*N*′-(4-hydroxymethylbenzyl)perylene-3,4:9,10-tetracarboxdiimide (5):** A mixture of *N*-(1-hexylheptyl)perylene-3,4:9,10-tetracarboxylic-3,4-anhydride-9,10-carboximide (**2**, 1.00 g, 1.74 mmol)[15] and imidazole (20 g) were heated at 140 °C, treated with (4-aminomethylphenyl)methanol (290 mg, 2.09 mmol), stirred at this temperature for 2 h, cooled and treated with a few mL of ethanol. The crude material was precipitated by the addition of 2 N HCl, and the mixture (100 mL) was allowed to stand for 1 h. The crude solid was collected by vacuum filtration, washed with 2 N HCl, dried in air (110 °C, 16 h), dissolved in a small amount of chloroform and purified by column separation (silica gel; chloroform/methanol, 30:1). Yield: 755 mg (62%), red dye, m.p. >300 °C. R_f (silica gel; chloroform/methanol, 30:1) = 0.42. IR (ATR): $\tilde{\nu}$ = 3492.0 (m), 2952.5 (s), 2922.7 (s), 2855.2 (s), 1691.3 (s), 1646.8 (s), 1591.9 (s), 1575.6 (s), 1507.9 (w), 1463.9 (w), 1436.1 (w), 1403.3 (m), 1338.6 (s), 1248.2 (m), 1172.2 (m), 1128.6 (w), 1017.8 (w), 852.2 (w), 823.9 (w), 808.2 (w), 783.8 (w), 752.0 (w), 724.8 (w), 644.3 (w), 627.9 (w) cm^{-1}. ^1H NMR (600 MHz, $CDCl_3$, 25 °C): δ = 0.82 (t, ^3J = 7.0 Hz, 6 H, CH_3), 1.22–1.36 (m, 16 H, CH_2), 1.84–1.90 (m, 2 H, β-CH_2), 2.22–2.28 (m, 2 H, β-CH_2), 4.66 (s, 2 H, CH_2-OH), 5.18 (tt, ^3J = 5.8 Hz, ^3J = 9.4 Hz, 1 H, CH-N), 5.40 (s, 2 H, CH_2-N), 7.34 (d, ^3J = 8.2 Hz, 2 H, H_{aryl}), 7.58 (s, ^3J = 8.2 Hz, 2 H, H_{aryl}), 8.56–8.67 (m, 8 H, $H_{perylene}$) ppm. ^{13}C NMR (150 MHz, $CDCl_3$, 25 °C): δ = 14.0, 22.6, 26.9, 29.2, 31.8, 32.4, 43.5, 54.8, 65.1, 123.0, 123.1, 123.2, 126.4, 126.5, 127.2, 129.3, 129.4, 129.5, 131.7, 134.3, 136.5, 140.3, 163.4 ppm. UV/Vis ($CHCl_3$): λ_{max} (ε) = 529 (82900), 492 (51200), 463 (22500) nm. Fluorescence ($CHCl_3$): λ_{max} (I_{rel}) = 539 (1.00), 582 (0.40) nm. Fluorescence quantum yield ($CHCl_3$, λ_{exc} = 491 nm, E_{491nm} = 0.0269 cm^{-1}, reference: **1a** with 1.00): 1.00. HRMS: calcd. for $C_{45}H_{44}N_2O_5$ 692.324; found 692.324, Δ = 0 mmu. $C_{45}H_{44}N_2O_5$ (692.8): calcd. C 78.01, H 6.40, N 4.04; found C 78.03, H 6.36, N 3.94.

***N*-(4-Bromomethylbenzyl)-*N*′-(1-hexylheptyl)perylene-3,4:9,10-tetracarboxdiimide:** *N*-(1-Hexylheptyl)-*N*′-(4-hydroxymethylbenzyl) perylene-3,4:9,10-tetracarboxdiimide (**5**, 500 mg, 0.72 mmol) was suspended in chloroform (50 mL) and treated in a modified Appel reaction[17] with tetrabromomethane (5.00 g, 15.2 mmol) and triphenylphosphane (3.64 g, 13.7 mmol; complete dissolution of the dye). The volatile materials were evaporated in vacuo, and the resulting crude material was dissolved in a small amount of chloroform and purified by column separation (silica gel; chloroform/methanol, 60:1). Yield: 485 mg (89%), red dye, m.p. >300 °C. R_f (silica gel; chloroform/methanol, 50:1) = 0.85. IR (ATR): $\tilde{\nu}$ = 2953.1 (m), 2923.1 (m), 2854.7 (m), 1696.0 (s), 1647.7 (s), 1592.3

(s), 1576.1 (s), 1507.6 (w), 1466.3 (w), 1435.7 (m), 1403.6 (m), 1336.2 (s), 1300.2 (m), 1250.3 (m), 1199.1 (m), 1168.6 (m), 1124.5 (w), 1107.2 (w), 1079.5 (w), 1019.3 (w), 987.7 (m), 848.6 (m), 808.1 (s), 785.5 (w), 750.5 (m), 740.1 (m), 724.0 (w), 652.0 (w), 628.3 (w), 615.9 (w) cm^{-1}. ^1H NMR (600 MHz, $CDCl_3$, 25 °C): δ = 0.83 (t, ^3J = 7.0 Hz, 6 H, CH_3), 1.21–1.38 (m, 16 H, CH_2), 1.85–1.91 (m, 2 H, β-CH_2), 2.22–2.28 (m, 2 H, β-CH_2), 4.46 (s, 2 H, CH_2-Br), 5.19 (tt, ^3J = 5.8 Hz, ^3J = 9.4 Hz, 1 H, CH-N), 5.38 (s, 2 H, CH_2-N), 7.33 (d, ^3J = 8.3 Hz, 2 H, H_{aryl}), 7.56 (d, ^3J = 8.3 Hz, 2 H, H_{aryl}), 8.52 (d, ^3J = 8.1 Hz, 2 H, $H_{perylene}$), 8.55 (d, ^3J = 8.1 Hz, 2 H, $H_{perylene}$), 8.61 (d, ^3J = 8.1 Hz, 2 H, $H_{perylene}$), 8.63–8.66 (m, 2 H, $H_{perylene}$) ppm. ^{13}C NMR (150 MHz, $CDCl_3$, 25 °C): δ = 14.0, 22.6, 26.9, 29.2, 31.8, 32.4, 33.2, 43.3, 54.8, 122.9, 123.0, 123.2, 126.3, 126.4, 129.2, 129.4, 129.5, 129.6, 131.6, 134.2, 134.9, 137.2, 137.3, 163.3 ppm. UV/Vis ($CHCl_3$): λ_{max} (ε) = 529 (86500), 491 (52000), 463 (19200) nm. Fluorescence ($CHCl_3$): λ_{max} (I_{rel}) = 539 (1.00), 582 (0.40) nm. Fluorescence quantum yield ($CHCl_3$, λ_{exc} = 491 nm, E_{491nm} = 0.0269 cm^{-1}, reference: **1a** with 1.00): 1.00. HRMS: calcd. for $C_{45}H_{43}BrN_2O_4$ 755.242; found 755.242, Δ = 0 mmu. $C_{45}H_{43}BrN_2O_4$ (755.2): calcd. C 71.52, H 5.73, N 3.71; found C 71.34, H 5.76, N 3.65.

***N*-(4-Azidomethylbenzyl)-*N*′-(1-hexylheptyl)perylene-3,4:9,10-tetracarboxdiimide (6):** *N*-(4-Bromomethylbenzyl)-*N*′-(1-hexylheptyl)-perylene-3,4:9,10-tetracarboxdiimide (400 mg, 0.53 mmol) was dispersed in DMF (50 mL) and treated in a modified procedure to that outlined in ref.[18] with sodium azide (72 mg, 1.1 mmol). The mixture was stirred at 80 °C for 2 h and then cooled, and the crude product was precipitated by the addition of distilled water (50 mL). The solid was collected by vacuum filtration, washed with distilled water and methanol and dried in vacuo at 60 °C for 1 h. The product was analytically pure without further treatment. Yield: 353 mg (93%), red dye, m.p. >300 °C. R_f (silica gel; chloroform/methanol, 50:1) = 0.85. IR (ATR): $\tilde{\nu}$ = 2956.0 (m), 2922.9 (m), 2855.0 (m), 2100.1 (m), 1695.0 (s), 1647.9 (s), 1592.8 (s), 1576.8 (s), 1507.5 (w), 1466.3 (w), 1436.1 (m), 1403.6 (m), 1338.1 (s), 1301.1 (m), 1249.3 (m), 1171.8 (m), 1124.2 (w), 1107.7 (w), 984.4 (m), 848.2 (m), 808.1 (s), 771.9 (w), 743.8 (m), 723.0 (w), 667.7 (w), 628.1 (w), 605.6 (w) cm^{-1}. ^1H NMR (600 MHz, $CDCl_3$, 25 °C): δ = 0.83 (t, ^3J = 7.0 Hz, 6 H, CH_3), 1.21–1.38 (m, 16 H, CH_2), 1.85–1.91 (m, 2 H, β-CH_2), 2.22–2.30 (m, 2 H, β-CH_2), 4.30 (s, 2 H, CH_2-N_3), 5.19 (tt, ^3J = 5.8 Hz, ^3J = 9.4 Hz, 1 H, CH-N), 5.37 (s, 2 H, CH_2-N), 7.29 (d, ^3J = 8.2 Hz, 2 H, H_{aryl}), 7.60 (d, ^3J = 8.0 Hz, 2 H, H_{aryl}), 8.42 (d, ^3J = 8.1 Hz, 2 H, $H_{perylene}$), 8.48 (d, ^3J = 8.1 Hz, 2 H, $H_{perylene}$), 8.53 (d, ^3J = 8.1 Hz, 2 H, $H_{perylene}$), 8.62 (d, ^3J = 13.6 Hz, 2 H, $H_{perylene}$) ppm. ^{13}C NMR (150 MHz, $CDCl_3$, 25 °C): δ = 14.0, 22.6, 27.0, 29.2, 31.8, 32.4, 43.3, 54.8, 54.9, 122.8, 123.1, 126.2, 126.3, 128.4, 129.3, 129.4, 129.7, 131.5, 134.0, 134.7, 134.8, 137.2, 163.2 ppm. UV/Vis ($CHCl_3$): λ_{max} (ε) = 526 (84600), 491 (52000), 461 (20200) nm. Fluorescence ($CHCl_3$): λ_{max} (I_{rel}) = 539 (1.00), 582 (0.40) nm. Fluorescence quantum yield ($CHCl_3$, λ_{exc} = 491 nm, E_{491nm} = 0.0269 cm^{-1}, reference: **1a** with 1.00): 1.00. HRMS: calcd. for $C_{45}H_{43}N_5O_4$ 717.327; found 717.327, Δ = 0 mmu. $C_{45}H_{43}N_5O_4$ (717.9): calcd. C 75.29, H 6.04, N 9.76; found C 75.06, H 6.00, N 9.27.

General Procedure for the "Click Reaction" of Perylene Azide 6 with Terminal Alkynes: A mixture of *N*-(4-azidomethylbenzyl)-*N*′-(1-heptylhexyl)perylene-3,4:9,10-tetracarboxdiimide (**6**, 100 mg, 0.139 mmol), the terminal alkyne (0.556 mmol), copper(I)iodide (132 mg, 0.695 mmol) and *N*-ethylbis(isopropyl)amine (72.0 mg, 0.556 mmol) in THF (15 mL) was stirred at room temperature for 16 h. The mixture was filtered, and the solvent was evaporated in vacuo. The crude material was dissolved in a small amount of chloroform and purified by column separation (silica gel; dichloromethane/methanol, 100:1).

***N*-(1-Hexylheptyl)-*N*′-4-(4-methoxymethyl[1,2,3]triazol-1-ylmethyl)-benzylperylene-3,4:9,10-tetracarboxdiimide (7a):** Methyl propargyl ether (40.0 mg, 0.556 mmol) was allowed to react according to the general procedure. Yield: 68 mg (63%), red dye, m.p. >300 °C. R_f (silica gel; chloroform/methanol, 40:1) = 0.49. IR (ATR): $\tilde{\nu}$ = 2952.3 (m), 2923.2 (m), 2854.2 (m), 1691.1 (m), 1650.4 (s), 1592.6 (s), 1576.9 (m), 1506.7 (w), 1455.8 (w), 1434.9 (m), 1403.0 (m), 1331.6 (s), 1247.8 (m), 1169.1 (m), 1124.8 (w), 1093.8 (w), 1049.2 (w), 1022.6 (w), 982.1 (w), 851.9 (w), 808.6 (m), 795.1 (w), 743.8 (m), 720.8 (w), 626.2 (w) cm^{-1}. ^1H NMR (600 MHz, $CDCl_3$, 25 °C): δ = 0.83 (t, ^3J = 7.0 Hz, 6 H, CH_3), 1.21–1.38 (m, 16 H, CH_2), 1.85–1.91 (m, 2 H, β-CH_2), 2.22–2.30 (m, 2 H, β-CH_2), 3.33 (s, 3 H, CH_3), 4.50 (s, 2 H, CH_2-O) 5.18 (tt, ^3J = 5.8 Hz, ^3J = 9.4 Hz, 1 H, CH-N) 5.35 (s, 2 H, CH_2-N), 5.48 (s, 2 H, CH_2-N), 7.24 (d, ^3J = 8.2 Hz, 2 H, H_{aryl}), 7.40 (s, 1 H, $CH_{triazole}$), 7.57 (d, ^3J = 8.0 Hz, 2 H, H_{aryl}), 8.39–8.62 (m, 8 H, $H_{perylene}$) ppm. ^{13}C NMR (150 MHz, $CDCl_3$, 25 °C): δ = 14.0, 22.6, 26.9, 29.2, 31.7, 32.4, 43.2, 53.9, 54.8, 58.3, 65.4, 65.9, 122.8, 123.1, 126.1, 126.2, 128.1, 128.4, 129.2, 129.4, 129.7, 129.9, 131.5, 133.8, 134.7, 137.8, 163.2 ppm. UV/Vis ($CHCl_3$): λ_{max} (ε) = 527 (83700), 491 (52500), 463 (21100) nm. Fluorescence ($CHCl_3$): λ_{max} (I_{rel}) = 539 (1.0), 582 (0.40) nm. Fluorescence quantum yield ($CHCl_3$, λ_{exc} = 491 nm, E_{491nm} = 0.0234 cm^{-1}, reference: **1a** with 1.00): 1.00. HRMS: calcd. for $C_{49}H_{49}N_5O_5$ 787.371; found 787.370, Δ = 1 mmu. $C_{45}H_{43}N_5O_4$ (717.9): calcd. C 74.69, H 6.27, N 8.89; found C 74.36, H 6.14, N 8.83.

Byproduct *N*-(1-Hexylheptyl)-*N*′-4-(5-iodo-4-methoxymethyl[1,2,3]triazol-1-ylmethyl)benzylperylene-3,4:9,10-tetracarboxdiimide: ^1H NMR (600 MHz, $CDCl_3$, 25 °C): δ = 0.83 (t, ^3J = 7.0 Hz, 6 H, CH_3), 1.21–1.38 (m, 16 H, CH_2), 1.85–1.91 (m, 2 H, β-CH_2), 2.22–2.30 (m, 2 H, β-CH_2), 3.35 (s, 3 H, CH_3), 4.46 (s, 2 H, CH_2-O) 5.18 (tt, ^3J = 5.8 Hz, ^3J = 9.4 Hz, 1 H, CH-N) 5.33 (s, 2 H, CH_2-N), 5.54 (s, 2 H, CH_2-N), 7.24 (d, ^3J = 8.2 Hz, 2 H, H_{aryl}), 7.55 (d, ^3J = 8.0 Hz, 2 H, H_{aryl}), 8.39–8.62 (m, 8 H, $H_{perylene}$) ppm. HRMS: calcd. for $C_{49}H_{48}IN_5O_5$ 913.269; found 913.269, Δ = 0 mmu.

***N*-4-(4-Acetyl[1,2,3]triazol-1-ylmethyl)benzyl-*N*′-(1-hexylheptyl)perylene-3,4:9,10-tetracarboxdiimide (7e):** But-3-yn-2-one (38.0 mg, 0.556 mmol) was allowed to react according to the general procedure. Yield: 60 mg (54%), red dye, m.p. >300 °C. R_f (silica gel; chloroform/methanol, 50:1) = 0.45. IR (ATR): $\tilde{\nu}$ = 2952.4 (m), 2921.8 (m), 2853.4 (m), 1688.9 (m), 1678.2 (s), 1648.4 (s), 1593.9 (s), 1577.0 (m), 1530.4 (w), 1516.2 (w), 1506.3 (w), 1459.0 (w), 1436.9 (m), 1423.2 (w), 1403.4 (m), 1335.4 (s), 1294.4 (w), 1247.1 (m), 1168.2 (m), 1124.9 (w), 1105.9 (w), 982.9 (w), 810.6 (s), 744.0 (m), 720.7 (w), 625.8 (w) cm^{-1}. ^1H NMR (600 MHz, $CDCl_3$, 25 °C): δ = 0.82 (t, ^3J = 7.0 Hz, 6 H, CH_3), 1.21–1.38 (m, 16 H, CH_2), 1.85–1.91 (m, 2 H, β-CH_2), 2.22–2.30 (m, 2 H, β-CH_2), 2.62 (s, 3 H, CH_3), 5.18 (tt, ^3J = 5.8 Hz, ^3J = 9.4 Hz, 1 H, CH-N) 5.38 (s, 2 H, CH_2-N), 5.51 (s, 2 H, CH_2-N), 7.26 (d, ^3J = 8.4 Hz, 2 H, H_{aryl}), 7.62 (d, ^3J = 8.4 Hz, 2 H, H_{aryl}), 7.87 (s, 1 H, $CH_{triazole}$), 8.39–8.62 (m, 8 H, $H_{perylene}$) ppm. ^{13}C NMR (150 MHz, $CDCl_3$, 25 °C): δ = 14.0, 22.6, 26.9, 27.1, 29.2, 31.7, 32.4, 43.2, 54.2, 54.8, 122.8, 122.9, 123.2, 125.2, 126.3, 126.4, 128.6, 129.4, 129.5, 130.2, 131.7, 132.9, 134.9, 138.3, 148.3, 163.3, 192.8 ppm. UV/Vis ($CHCl_3$): λ_{max} (ε) = 529 (85200), 492 (52100), 463 (22200) nm. Fluorescence ($CHCl_3$): λ_{max} (I_{rel}) = 539 (1.0), 582 (0.40) nm. Fluorescence quantum yield ($CHCl_3$, λ_{exc} = 491 nm, E_{491nm} = 0.0246 cm^{-1}, reference: **1a** with 1.00): 1.00. HRMS: calcd. for $C_{49}H_{47}N_5O_5$ 785.356; found 785.356, Δ = 0 mmu. $C_{45}H_{43}N_5O_4$ (717.9): calcd. C 74.88, H 6.03, N 8.91; found C 74.55, H 5.97, N 8.81.

Byproduct *N*-4-(4-Acetyl-5-iodo[1,2,3]triazol-1-ylmethyl)benzyl-*N*′-(1-hexylheptyl)perylene-3,4:9,10-tetracarboxdiimide: HRMS: calcd. for $C_{49}H_{46}IN_5O_5$ 911.254; found 911.255, Δ = 1 mmu.

***N*-4-(4-Benzyloxymethyl[1,2,3]triazol-1-ylmethyl)benzyl-*N*′-(1-hexylheptyl)perylene-3,4:9,10-tetracarboxdiimide (7b):** Benzyl propargyl ether (82.0 mg, 0.556 mmol) was allowed to react according to the general procedure. Yield: 84 mg (70%), red dye, m.p. >300 °C. R_f (silica gel; chloroform/methanol, 100:1) = 0.43 IR (ATR): $\tilde{\nu}$ = 2952.5 (m), 2922.4 (m), 2853.7 (m), 1691.2 (m), 1651.2 (s), 1592.5 (s), 1576.5 (m), 1514.8 (w), 1506.3 (w), 1453.3 (w), 1434.8 (m), 1402.8 (m), 1333.1 (s), 1301.1 (w), 1248.0 (m), 1217.7 (w), 1170.0 (m), 1124.9 (w), 1105.9 (w), 1047.3 (w), 1022.6 (w), 984.5 (w), 852.0 (w), 808.9 (m), 742.6 (m), 721.7 (w), 625.9 (w) cm^{-1}. ^{1}H NMR (400 MHz, CD_2Cl_2, 25 °C): δ = 0.79 (t, ^3J = 7.0 Hz, 6 H, CH_3), 1.21–1.38 (m, 16 H, CH_2), 1.80–1.89 (m, 2 H, β-CH_2), 2.17–2.27 (m, 2 H, β-CH_2), 4.44 (s, 2 H, CH_2), 4.52 (s, 2 H, CH_2), 5.12 (tt, ^3J = 5.8, ^3J = 9.4 Hz, 1 H, CH-N), 5.13 (s, 2 H, CH_2-N), 5.44 (s, 2 H, CH_2-N), 7.18–7.24 (m, 7 H, H_{aryl}), 7.43 (s, 1 H, $H_{triazole}$), 7.54 (d, ^3J = 8.2 Hz, 2 H, H_{aryl}), 8.31 (d, ^3J = 8.2 Hz, 2 H, $H_{perylene}$), 8.48 (d, ^3J = 8.2 Hz, 2 H, $H_{perylene}$), 8.53 (d, ^3J = 8.2 Hz, 2 H, $H_{perylene}$), 8.60–8.64 (m, 2 H, $H_{perylene}$) ppm. ^{13}C NMR (150 MHz, $CDCl_3$, 25 °C): δ = 14.0, 22.59, 26.6, 29.2, 31.7, 32.4, 43.2, 54.1, 54.86, 63.5, 72.6, 122.6, 122.7, 123.0, 126.0, 126.1, 127.6, 127.7, 127.8, 127.9, 128.3, 128.4, 129.1, 129.3, 130.0, 131.3, 133.6, 134.5, 137.6, 137.8, 163.0 ppm. UV/Vis ($CHCl_3$): λ_{max} (ε) = 525 (84600), 490 (52000), 461 (20200) nm. Fluorescence ($CHCl_3$): λ_{max} (I_{rel}) = 539 (1.0), 582 (0.40) nm. Fluorescence quantum yield ($CHCl_3$, λ_{exc} = 491 nm, E_{491nm} = 0.0275 cm^{-1}, reference: **1a** with 1.00): 1.00. HRMS: calcd. for $C_{55}H_{53}N_5O_5$ 863.406; found 863.408, Δ = 0 mmu. $C_{55}H_{53}N_5O_5$ (864.0): calcd. C 76.45, H 6.18, N 8.11; found C 75.31, H 6.18, N 7.85.

Byproduct *N*-4-(5-Iodo-4-benzyloxymethyl[1,2,3]triazol-1-ylmethyl)-benzyl-*N*′-(1-hexylheptyl)perylene-3,4:9,10-tetracarboxdiimide: MS (DEI+, 70 eV): *m*/*z* (%) = 992.4 (1) [M + 2H]$^+$, 991.4 (6) [M + H]$^+$, 990.4 (14) [M]$^+$, 676.7 (39), 677.7 (18), 678.7 (5), 494.2 (86), 495.2 (57), 496.2 (16), 390.1 (23), 391.1 (14), 374.1 (27), 346.1 (24), 91.1 (100).

***N*-(1-Hexylheptyl)-*N*′-4-(4-propyl[1,2,3]triazol-1-ylmethyl)benzylperylene-3,4:9,10-tetracarboxdiimide (7f):** 1-Pentyne (38.0 mg, 0.556 mmol) was allowed to react according to the general procedure. Yield: 65 mg (59%), red dye, m.p. >300 °C. R_f (silica gel; chloroform/methanol, 100:1) = 0.10. IR (ATR): $\tilde{\nu}$ = 3472.9 (br. m), 2923.8 (m), 2854.0 (m), 1692.0 (m), 1651.6 (s), 1592.5 (s), 1576.8 (m), 1506.7 (w), 1434.5 (m), 1402.9 (m), 1377.8 (w), 1332.5 (s), 1248.3 (m), 1218.3 (w), 1170.1 (m), 1124.8 (w), 1105.1 (w), 1046.1 (w), 1020.8 (w), 984.8 (w), 852.6 (w), 808.7 (s), 743.3 (m), 720.8 (w), 626.8 (w) cm^{-1}. ^{1}H NMR (600 MHz, $CDCl_3$, 25 °C): δ = 0.82 (t, ^3J = 7.0 Hz, 6 H, CH_3), 0.90 (t, ^3J = 8.2 Hz, 3 H, CH_3), 1.21–1.38 (m, 16 H, CH_2), 1.60–1.65 (m, 2 H, CH_2) 1.86–1.92 (m, 2 H, β-CH_2), 2.22–2.30 (m, 2 H, β-CH_2), 2.65 (t, ^3J = 7.6 Hz, 2 H, CH_2), 5.18 (tt, ^3J = 5.8 Hz, ^3J = 9.4 Hz, 1 H, CH-N) 5.35 (s, 2 H, CH_2-N), 5.47 (s, 2 H, CH_2-N), 7.23 (d, ^3J = 8.2 Hz, 2 H, H_{aryl}), 7.22 (s, 1 H, $CH_{triazole}$), 7.57 (d, ^3J = 8.2 Hz, 2 H, H_{aryl}), 8.37–8.60 (m, 8 H, $H_{perylene}$) ppm. ^{13}C NMR (150 MHz, $CDCl_3$, 25 °C): δ = 13.7, 14.0, 22.5, 22.6, 27.0, 27.3, 29.2, 31.7, 32.3, 43.3, 54.1, 54.9, 121.0, 122.7, 122.8, 122.8, 123.1, 126.1, 126.2, 127.9, 128.3, 129.2, 129.4, 129.6, 129.8, 131.4, 133.8, 133.9, 134.7, 137.8, 163.2 ppm. UV/Vis ($CHCl_3$): λ_{max} (ε) = 525 (86100), 490 (51900), 461 (21600) nm. Fluorescence ($CHCl_3$): λ_{max} (I_{rel}) = 539 (1.0), 582 (0.40) nm. Fluorescence quantum yield ($CHCl_3$, λ_{exc} = 491 nm, E_{491nm} = 0.0297 cm^{-1}, reference: **1a** with 1.00): 1.00. HRMS: calcd. for $C_{50}H_{51}N_5O_4$ 785.394; found 785.394, Δ = 0 mmu. $C_{45}H_{43}N_5O_4$ (717.9): calcd. C 76.41, H 6.54, N 8.91; found C 76.06, H 6.53, N 8.78.

Byproduct *N*-(1-Hexylheptyl)-*N*′-4-(5-iodo-4-propyl[1,2,3]triazol-1-ylmethyl)benzylperylene-3,4:9,10-tetracarboxdiimide: ^{1}H NMR (400 MHz, CD_2Cl_2, 25 °C): δ = 0.82 (t, ^3J = 7.0 Hz, 6 H, CH_3), 0.91 (t, ^3J = 8.2 Hz, 3 H, CH_3), 1.21–1.38 (m, 16 H, CH_2), 1.65–1.70 (m, 2 H, CH_2), 1.86–1.92 (m, 2 H, β-CH_2), 2.22–2.30 (m, 2 H, β-CH_2), 2.58 (t, ^3J = 7.6 Hz, 2 H, CH_2), 5.18 (tt, ^3J = 5.8 Hz, ^3J = 9.4 Hz, 1 H, CH-N) 5.33 (s, 2 H, CH_2-N), 5.51 (s, 2 H, CH_2-N), 7.23 (d, ^3J = 8.2 Hz, 2 H, H_{aryl}), 7.54 (d, ^3J = 8.0 Hz, 2 H, H_{aryl}), 8.37–8.60 (m, 8 H, $H_{perylene}$) ppm. HRMS: calcd. for $C_{50}H_5IN_5O_4$ 911.289; found 911.288, Δ = 0 mmu.

***N*-4-{4-[(Benzylmethylamino)methyl][1,2,3]triazol-1-ylmethyl}-benzyl-*N*′-(1-hexylheptyl)perylene-3,4:9,10-tetracarboxdiimide (7d):** *N*-Benzyl-*N*-methylpropargylamine (89.0 mg, 0.556 mmol) was allowed to react according to the general procedure. Yield: 89 mg (73%), red dye, m.p. >300 C. R_f (silica gel; chloroform/methanol, 70:1) = 0.20. IR (ATR): $\tilde{\nu}$ = 3486.8 (br. m), 2923.9 (m), 2853.9 (m), 1692.1 (m), 1651.8 (s), 1592.5 (s), 1576.7 (m), 1506.7 (w), 1434.6 (m), 1402.8 (m), 1377.6 (w), 1332.3 (s), 1248.4 (m), 1217.9 (w), 1170.1 (m), 1124.9 (w), 1105.2 (w), 1045.7 (w), 1021.1 (w), 984.8 (w), 852.9 (w), 808.7 (s), 743.6 (m), 720.5 (w), 626.5 (w) cm^{-1}. ^{1}H NMR (600 MHz, $CDCl_3$, 25 °C): δ = 0.82 (t, ^3J = 7.0 Hz, 6 H, CH_3), 1.21–1.38 (m, 16 H, CH_2), 1.60–1.65 (m, 2 H, CH_2) 1.86–1.92 (m, 2 H, β-CH_2), 2.22–2.30 (m, 2 H, β-CH_2), 2.28 (s, 3 H, N-CH_3), 2.68 (s, 2 H, N-CH_2), 2.81 (s, 2 H, N-CH_2), 5.18 (tt, ^3J = 5.8 Hz, ^3J = 9.4 Hz, 1 H, CH-N) 5.37 (s, 2 H, CH_2-N), 5.48 (s, 2 H, CH_2-N), 7.24 (d, ^3J = 8.4 Hz, 2 H, H_{aryl}), 7.34 (s, 1 H, $CH_{triazole}$), 7.28–7–37 (m, 5 H, H_{aryl}), 7.58 (d, ^3J = 8.4 Hz, 2 H, H_{aryl}), 8.46–8.65 (m, 8 H, $H_{perylene}$) ppm. ^{13}C NMR (150 MHz, $CDCl_3$, 25 °C): δ = 14.0, 22.6, 26.9, 29.2, 31.7, 32.4, 43.3 53.9, 54.8, 122.8, 122.9, 123.2, 126.2, 126.4, 128.3, 128.5, 129.4, 129.5, 129.9, 131.6, 133.9, 134.8, 137.7, 163.3 ppm. UV/Vis ($CHCl_3$): λ_{max} (ε) = 525 (85600), 490 (52000), 461 (21700) nm. Fluorescence ($CHCl_3$): λ_{max} (I_{rel}) = 539 (1.0), 582 (0.40) nm. Fluorescence quantum yield ($CHCl_3$, λ_{exc} = 491 nm, E_{491nm} = 0.0303 cm^{-1}, reference: **1a** with 1.00): 1.00. HRMS: calcd. for $C_{56}H_{55}N_6O_4$ 877.441; found 877.439, Δ = 2.2 mmu. $C_{56}H_{56}N_6O_4$ (877.1): calcd. C 76.69, H 6.44, N 9.58; found C 76.74, H 6.11, N 9.56.

***N*-4-(4-Benzyl[1,2,3]triazol-1-ylmethyl)benzyl-*N*′-(1-hexylheptyl)perylene-3,4:9,10-tetracarboxdiimide (7g):** 3-Phenyl-1-propyne (65 mg, 0.56 mmol) was allowed to react according to the general procedure. Yield: 33 mg (28%), red dye, m.p. >300 °C. R_f (silica gel; chloroform/methanol, 50:1) = 0.10. IR (ATR): $\tilde{\nu}$ = 2952.5 (m), 2923.5 (m), 2854.7 (m), 1689.0 (m), 1678.2 (m), 1648.0 (s), 1594.1 (s), 1577.1 (m), 1530.0 (w), 1436.2 (w), 1423.2 (w), 1403.5 (m), 1355.4 (m), 1335.9 (s), 1294.5 (w), 1247.2 (m), 1217.3 (w), 1201.9 (w), 1168.1 (w), 1124.9 (w), 1106.1 (w), 1037.8 (w), 1021.3 (w), 982.7 (w), 863.3 (w), 810.8 (m), 744.1 (w), 720.8 (w), 625.8 (w) cm^{-1}. ^{1}H NMR (600 MHz, $CDCl_3$, 25 °C): δ = 0.82 (t, ^3J = 7.0 Hz, 6 H, CH_3), 1.21–1.38 (m, 16 H, CH_2), 1.86–1.92 (m, 2 H, β-CH_2), 2.22–2.30 (m, 2 H, β-CH_2), 4.06 (s, 2 H, CH_2-Ph) 5.18 (tt, ^3J = 5.8 Hz, ^3J = 9.4 Hz, 1 H, CH-N) 5.34 (s, 2 H, CH_2-N), 5.43 (s, 2 H, CH_2-N), 7.15–7.24 (m, 7 H, H_{aryl}), 7.25 (s, 1 H, $CH_{triazole}$), 7.55 (d, ^3J = 6.7 Hz, 1 H, H_{aryl}), 8.39–8.65 (m, 8 H, $H_{perylene}$) ppm. ^{13}C NMR (150 MHz, $CDCl_3$, 25 °C): δ = 14.0, 22.6, 26.9, 29.2, 31.7, 32.4, 43.2, 54.8, 122.1, 122.7, 122.8, 122.9, 123.2, 126.2, 126.3, 126.7, 128.0, 128.4, 128.6, 128.7, 129.3, 129.4, 129.6, 129.8, 131.5, 133.3, 134.0, 134.8, 138.0, 163.4 ppm. UV/Vis ($CHCl_3$): λ_{max} (ε) = 525 (86300), 490 (52300), 461 (22100) nm. Fluorescence ($CHCl_3$): λ_{max} (I_{rel}) = 539 (1.0), 582 (0.40) nm. Fluorescence quantum yield ($CHCl_3$, λ_{exc} = 491 nm, E_{491nm} = 0.0314 cm^{-1}, reference: **1a** with

1.00): 1.00. MS (DEI+, 70 eV): *m/z* (%) = 834.9 (9) $[M]^+$, 674.5 (81), 492.2 (100), 373.2 (82), 159.1 (94), 130.1 (51), 91.1 (74), 55.1 (34). $C_{54}H_{51}N_5O_4$ (834.0): calcd. C 77.77, H 6.16, N 8.40; found C 76.46, H 6.15, N 8.26.

***N*-4-(4-Dimethylaminomethyl[1,2,3]triazol-1-ylmethyl)benzyl-*N*′-(1-hexylheptyl)perylene-3,4:9,10-tetracarboxdiimide (7c):** *N*,*N*-Dimethylpropargylamine (46.0 mg, 0.556 mmol) was allowed to react according to the general procedure. Yield: 83 mg (75%), red dye, m.p. >300 °C. R_f (silica gel; chloroform/methanol, 70:1) = 0.15. IR (ATR): $\tilde{\nu}$ = 3487.0 (br. m), 2922.0 (m), 2853.8 (m), 1692.2 (m), 1655.8 (s), 1592.9 (s), 1576.8 (m), 1499.4 (w), 1455.0 (w), 1435.3 (m), 1403.1 (m), 1352.8 (w), 1329.9 (s), 1247.7 (m), 1217.5 (w), 1169.0 (m), 1125.6 (w), 1103.3 (w), 1042.8 (w), 1021.9 (w), 983.3 (w), 853.0 (w), 808.8 (s), 743.7 (m), 720.5 (w), 627.0 (w) cm^{-1}. ^1H NMR (600 MHz, $CDCl_3$, 25 °C): δ = 0.82 (t, ^3J = 7.0 Hz, 6 H, CH_3), 1.21–1.38 (m, 16 H, CH_2), 1.86–1.92 (m, 2 H, β-CH_2), 2.22–2.30 (m, 2 H, β-CH_2), 2.51 (s, 6 H, CH_3), 3.92 (s, 2 H, N-CH_2) 5.18 (tt, ^3J = 5.8 Hz, ^3J = 9.4 Hz, 1 H, CH-N) 5.38 (s, 2 H, CH_2-N), 5.49 (s, 2 H, CH_2-N), 7.24 (d, ^3J = 8.3 Hz, 2 H, H_{aryl}), 7.58 (d, ^3J = 8.3 Hz, 2 H, H_{aryl}), 7.86 (s, 1 H, $CH_{triazole}$), 8.55–8.67 (m, 8 H, $H_{perylene}$) ppm. ^{13}C NMR (150 MHz, $CDCl_3$, 25 °C): δ = 14.0, 22.6, 26.9, 29.2, 31.7, 32.4, 43.3, 43.5, 53.2, 54.0, 54.8, 122.9, 123.0, 123.2, 126.3, 126.5, 128.3, 128.7, 129.4, 129.5, 129.9, 130.2, 131.7, 133.6, 134.2, 134.9, 137.9, 163.4 ppm. UV/Vis ($CHCl_3$): λ_{max} (E_{rel}) = 459 (0.22), 489 (0.59), 528 (1.0) nm. Fluorescence ($CHCl_3$): λ_{max} (I_{rel}) = 539 (1.0), 582 (0.40) nm. Fluorescence quantum yield ($CHCl_3$, λ_{exc} = 491 nm, E_{491nm} = 0.0314 cm^{-1}, reference: **1a** with 1.00): 1.00. MS (DEI+, 70 eV): *m/z* (%) = 801.9 (1) $[M + H]^+$, 800.9 (2) $[M]^+$, 757.8 (15), 676.6 (77), 675.6 (77), 495.2 (65), 494.1 (100), 390.1 (55), 374.1 (12), 346.1 (36), 182.2 (12).

Byproduct *N*-4-(4-Dimethylaminomethyl-5-iodo[1,2,3]triazol-1-ylmethyl)benzyl-*N*′-(1-hexylheptyl)perylene-3,4:9,10-tetracarboxdiimide: ^1H NMR (600 MHz, $CDCl_3$, 25 °C): δ = 0.82 (t, ^3J = 7.0 Hz, 6 H, CH_3), 1.21–1.38 (m, 16 H, CH_2), 1.86–1.92 (m, 2 H, β-CH_2), 2.22–2.30 (m, 2 H, β-CH_2), 2.51 (s, 6 H, CH_3), 3.92 (s, 2 H, N-CH_2) 5.18 (tt, ^3J = 5.8 Hz, ^3J = 9.4 Hz, 1 H, CH-N) 5.40 (s, 2 H, CH_2-N), 5.54 (s, 2 H, CH_2-N), 7.24 (d, ^3J = 8.3 Hz, 2 H, H_{aryl}), 7.63 (d, ^3J = 8.3 Hz, 2 H, H_{aryl}), 8.55–8.67 (m, 8 H, $H_{perylene}$) ppm. HRMS: calcd. for $C_{50}H_{53}IN_6O_4$ 928.318; found 928.320, Δ = 2 mmu.

***N*-(1-Hexylheptyl)-*N*′-4-[4-(17-hydroxy-3-methoxy-13-methyl-7,8,9,11,12,13,14,15,16,17-decahydro-6*H*-cyclopenta[*a*]phenanthren-17-yl)[1,2,3]triazol-1-ylmethyl]benzylperylene-3,4:9,10-tetracarboxdiimide (7i):** Mestranol (174 mg, 0.556 mmol) was allowed to react according to the general procedure. Yield: 120 mg (84%), red dye, m.p. >300 °C. R_f (silica gel; dichloromethane/methanol, 15:1) = 0.26. IR (ATR): $\tilde{\nu}$ = 3487.0 (br. m), 2924.4 (m), 2853.5 (m), 1693.3 (m), 1654.4 (s), 1592.8 (s), 1576.7 (m), 1498.8 (w), 1434.5 (m), 1403.0 (m), 1377.6 (w), 1332.8 (s), 1249.5 (m), 1169.5 (m), 1124.8 (w), 1104.4 (w), 1041.8 (w), 1020.8 (w), 984.8 (w), 852.2 (w), 808.5 (s), 743.5 (m), 720.6 (w), 627.8 (w) cm^{-1}. ^1H NMR (600 MHz, $CDCl_3$, 25 °C): δ = 0.82 (t, ^3J = 7.0 Hz, 6 H, CH_3), 0.96 (s, 3 H, CH_3), 1.19–1.67 (m, 25 H, CH_2, CH), 1.81–1.91 (m, 4 H, β-CH_2, CH_2), 2.00–2.10 (m, 2 H, CH), 2.22–2.32 (m, 2 H, β-CH_2), 2.96–2.82 (m, 2 H, CH_2), 3.72 (s, 3 H, OCH_3), 5.15–5.20 (m, 1 H, CH-N), 5.35 (s, 2 H, CH_2-N), 5.49 (s, 2 H, CH_2-N), 6.53 (d, ^3J = 2.7 Hz, 1 H, H_{aryl}), 6.60 (dd, ^3J = 2.7, 8.6 Hz, 1 H, H_{aryl}), 7.03 (d, ^3J = 8.6 Hz, 1 H, H_{aryl}), 7.25 (d, ^3J = 8.3 Hz, 2 H, H_{aryl}), 7.33 (s, 1 H, $H_{triazole}$), 7.58 (d, ^3J = 8.3 Hz, 2 H, H_{aryl}), 8.40 (d, ^3J = 8.1 Hz, 2 H, $H_{perylene}$), 8.47 (d, ^3J = 8.1 Hz, 2 H, $H_{perylene}$), 8.50 (d, ^3J = 8.1 Hz, 2 H, $H_{perylene}$), 8.55–8–60 (m, 2 H, $H_{perylene}$) ppm. ^{13}C NMR (150 MHz, $CDCl_3$, 25 °C): δ = 14.0, 14.1, 22.6, 23.3, 26.2, 26.9, 27.2, 29.2, 29.7, 31.7, 32.6, 32.9, 37.8, 39.4, 43.2, 43.3, 47.2, 48.4, 50.8, 53.8, 54.8, 55.1, 82.3, 111.3, 113.6, 121.1, 122.7, 122.8, 123.1, 126.1, 126.1, 126.3, 128.2, 129.2, 129.4, 129.8, 131.5, 132.6, 134.0, 134.1, 134.8, 137.7, 137.9, 154.1, 157.3, 163.3 ppm. UV/Vis ($CHCl_3$): λ_{max} (ε) = 525 (84500), 490 (50300), 461 (21000) nm. Fluorescence ($CHCl_3$): λ_{max} (I_{rel}) = 539 (1.0), 582 (0.40) nm. Fluorescence quantum yield ($CHCl_3$, λ_{exc} = 491 nm, E_{491nm} = 0.0251 cm^{-1}, reference: 1a with 1.00): 1.00. HRMS: calcd. for $C_{66}H_{69}N_5O_6$ 1027.522; found 1027.524, Δ = 2 mmu. $C_{66}H_{69}N_5O_6$ (1027.5): calcd. C 77.09, H 6.76, N 6.81; found C 76.63, H 6.88, N 6.81.

***N*-(1-Hexylheptyl)-*N*′-4-[4-(17-hydroxy-10,13-dimethyl-3-oxo-2,3,6,7,8,9,10,11,12,13,14,15,16,17-tetradecahydro-1*H*-cyclopenta-[*a*]phenanthren-17-yl)[1,2,3]triazol-1-ylmethyl]benzylperylene-3,4:9,10-tetracarboxdiimide (7h):** Ethisterone (175 mg, 0.556 mmol) was allowed to react according to the general procedure. Yield: 100 mg (70%), red dye, m.p. >300 °C. R_f (silica gel; chloroform/methanol, 50:1) = 0.10. IR (ATR): $\tilde{\nu}$ = 3476.2 (br. m), 2924.9 (m), 2854.4 (m), 1692.2 (m), 1653.5 (s), 1614.1 (w), 1592.9 (s), 1576.9 (m), 1507.0 (w), 1434.8 (m), 1403.0 (m), 1377.6 (w), 1332.2 (s), 1270.4 (w), 1248.6 (m), 1170.1 (m), 1125.6 (w), 1105.2 (w), 1045.5 (w), 1021.5 (w), 985.5 (w), 853.5 (w), 808.9 (s), 743.9 (m), 720.3 (w), 627.3 (w) cm^{-1}. ^1H NMR (600 MHz, $CDCl_3$, 25 °C): δ = 0.30–0.42 (m, 1 H, CH), 0.65–0.76 (m, 1 H, CH), 0.83 (t, ^3J = 7.0 Hz, 6 H, CH_3), 1.00 (s, 3 H, CH_3), 1.15 (s, 3 H, CH_3), 1.19–, CH_2, 20 H) 1.37 (m, 1.42–1.48 (m, 2 H, CH_2), 1.55–1.63 (m, 2 H, CH_2), 1.77–1.96 (m, 6 H, CH_2, β-CH_2), 2.02–2.09 (m, 1 H, CH), 2.21–2.42 (m, 6 H, CH_2, β-CH_2), 5.15–5.20 (m, 1 H, CH-N), 5.34 (s, 2 H, CH_2-N), 5.44–5.57 (m, 2 H, CH_2-N), 5.67 (s, 1 H, H_{olefin}), 7.23–7.27 (m, 2 H, H_{aryl}), 7.35 (s, 1 H, $H_{triazole}$), 7.60 (d, ^3J = 6.9 Hz, 2 H, H_{aryl}), 8.41–8.63 (m, 8 H, $H_{perylene}$) ppm. ^{13}C NMR (150 MHz, $CDCl_3$, 25 °C): δ = 14.0, 14.2, 17.4, 20.5, 22.6, 23.6, 27.0, 29.2, 31.5, 31.7, 32.4, 32.7, 33.9, 35.6, 36.2, 37.8, 38.5, 43.3, 47.0, 49.1, 53.1, 54.8, 122.7, 122.9, 123.2, 123.8, 126.1, 126.2, 128.3, 129.2, 129.4, 129.8, 131.5, 133.4, 134.8, 138.0, 153.5, 163.2, 171.1, 199.4 ppm. UV/Vis ($CHCl_3$): λ_{max} (ε) = 525 (85600), 491 (52000), 461 (22400) nm. Fluorescence ($CHCl_3$): λ_{max} (I_{rel}) = 539 (1.0), 582 (0.40) nm. Fluorescence quantum yield ($CHCl_3$, λ_{exc} = 491 nm, E_{491nm} = 0.0359 cm^{-1}, reference: **1a** with 1.00): 1.00. HRMS: calcd. for $C_{66}H_{72}N_5O_6$ 1030.549; found 1030.551, Δ = 2 mmu. $C_{66}H_{72}N_5O_6$ (1030.3): calcd. C 76.94, H 6.95, N 6.80; found C 76.74, H 6.64, N 6.67.

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