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Persistent Fluorescence of Perylene Dyes by Steric Inhibition of Aggregation

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Dedicated to Professor Rolf Huisgen on the occasion of his 80th birthday

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Abstract—The control of the self-organisation of dye molecules in solution by bulky peripheral groups has been demonstrated. Consequently, fluorescent dyes could be prepared with a persistent fluorescence, even at very high concentrations because of a complete suppression of aggregation. Fluorescence quantum yields of unity were found.

Introduction

Intermolecular interactions are the driving forces for the formation of supramolecules (compare Ref. 1 and references therein) as well as for the aggregation of dyes. The molecular stacking in such aggregates is one of the most important reasons for an efficient fluorescence quenching[2,3] with increasing concentrations of dyes and usually excludes concentrated solution of dyes from fluorescence applications.

Results and Discussion

Perylene dyes[4] (perylene-3,4:9,10-tetracarboxylic bisimides, **2**) are of special interest because of their high photostabilities and fluorescence quantum yields. However, fluorescence applications of **2** are limited to rather diluted solutions because of fluorescence quenching by aggregation.[5,6] Moreover, we found this fluorescence quenching effect even more pronounced for derivatives of **2** with low solubilities; compare also Ref. 7.

We investigated the effect of steric shielding of the chromophores for preventing such a self-organisation in order to obtain fluorescent dyes for high concentrations (Figs. 1 and 2).

Synthesis

We tried to attach the bulky 2,4,6-triphenylphenyl group to the nitrogen atoms of perylene-3,4:9,10-tetracarboxylic bisimides such as **2a** and therefore condensed 2,4,6-triphenylaniline with the bisanhydride **1**. However, no **2b** could be obtained, rather the decarboxylation product **3b**; for this reaction compare Refs. 8,9. The bulky 4-aminotetraphenylmethane is less shielded at the amino group than the former amine and can be condensed with **1** to **2c**. The steric shielding of this bulky terminus becomes obvious from the fact that the bisimide **2c** cannot be hydrolyzed[10] under the conditions of other bisimides. Bulky purely aliphatic amines were obtained by the alkylation of acetonitrile with long-chain secondary alkyl halogenides and subsequent reduction. The condensation of such a highly branched amine with **1** gave the aliphatically shielded **2d**.

The partially shielded derivatives **5c** and **5e** could be prepared by the reaction of the monoanhydride **4a** where the 1-hexyl heptyl substituent at the nitrogen atom ('swallow-tail' substituent[11]) guarantees a high solubility. The derivative **5b** with an extended aromatic sphere could be obtained from the iminoimide **5a** and tetraphenyl phthalic anhydride.

Spectroscopic investigations

The UV/vis absorption and fluorescence spectra of **2** and **3** remain nearly uninfluenced by the bulky substituents: **2d** corresponds to dyes with simple aliphatic substituents such as **2a** (1-hexylheptyl substituent) and **2c** to aromatically substituted dyes. The same results are obtained for **3**.

The fluorescence quantum yields for the bisimides **2c**, **2d**, **5b**, **5c** and **5e** are close to unity in diluted solutions and correspond to **2a**.[12] The quantum yield of **3b** is lower (84%, in chloroform), but is close to previously reported yields of perylene-3,4-dicarboxylic imides.[13]

However, in concentrated solutions the bulky substituents induce completely different fluorescence properties in novel

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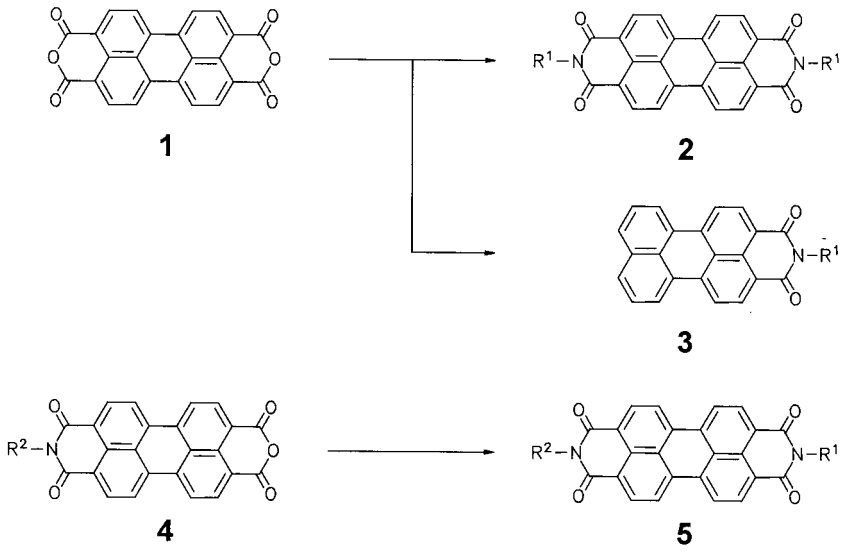

Figure 1.

dyes because of the inhibition of aggregation and, together with this, the inhibition of concentration quenching. Although the solutizing effect of the tetraphenylmethane substituent in **2c** is low so that this dye exhibits more pigment-like properties the intense fluorescence of **2c** persists until saturation in solutions. Moreover, **2c** exhibits a pronounced red solid-state fluorescence (see Fig. 3), whereas the solid-state of the majority of other derivatives of **2** is only weakly fluorescent because of the stacking of chromophores in the crystalline state. The solubility is appreciably increased by the exchange of one tetraphenylmethane substituent by the solutizing 1-hexylheptyl group such as in **5c** and a strong fluorescence is observed even in concentrated solution, however, not in the solid-state. The steric demand of the *N*-terminus can be further increased by the tetraphenylphthalic imide such as in **5b**.

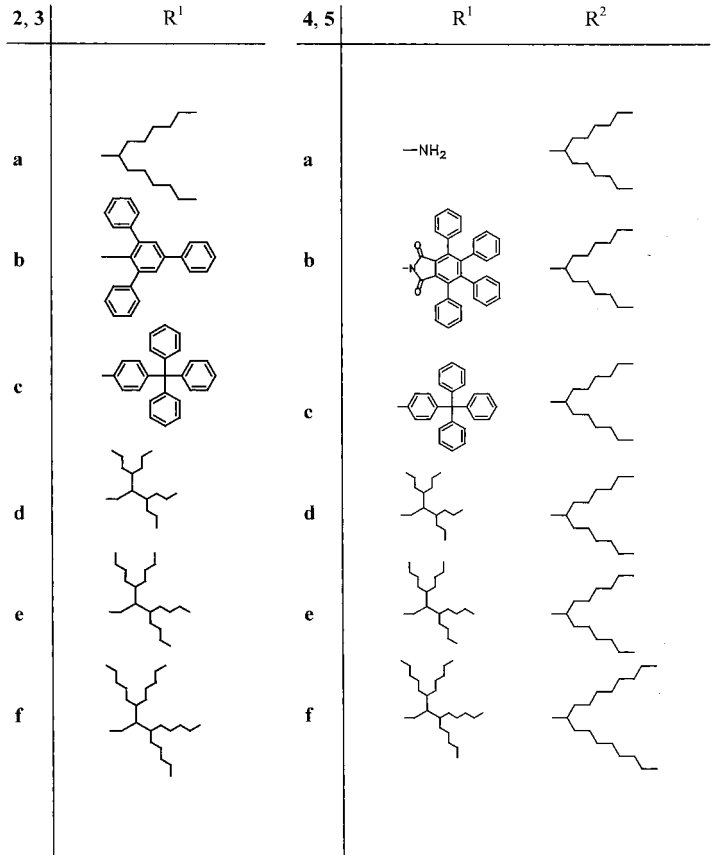

Figure 2.

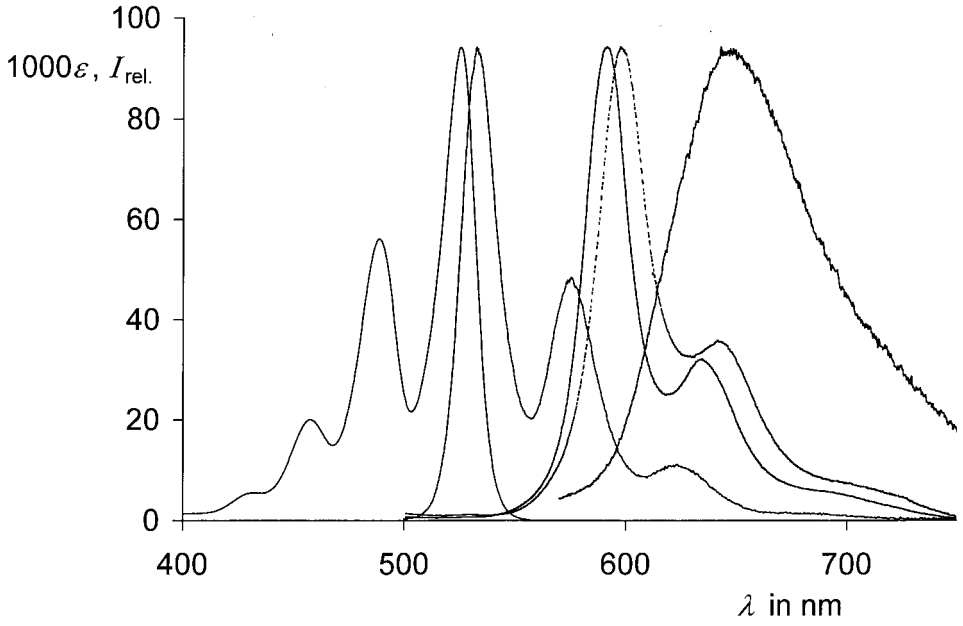

Figure 3. UV/vis absorption and quantum corrected fluorescence spectra of perylene bisimides. From left to right: absorption and fluorescence spectra of **2d** in chloroform and solid-state fluorescence spectra of **2d**, **2e** and **2f**.

Even more pronounced effects were obtained by the purely aliphatic and highly branched *N*-termini such as in **2d**. The solutizing effect of these multiple 'swallow-tail' substituents is so dominant that highly concentrated and dark solutions can be prepared with still persistent fluorescence. Strong fluorescence can be observed at the surface of such solutions. The solid-state fluorescence of the series **2d**…**2f** is also pronounced. Interestingly, the rather short chains in **2d** induce a bright orange shade resembling concentrated solutions of the dye, whereas the colour is shifted to orange red for **2e** and to red for **2f**. Possibly, the longer aliphatic side-chains in **2f** are interacting with themselves forming a 'closed shell' so that shielding effect in the solid-state is not as pronounced as for **2d**.

Conclusion

The self-organisation of molecules, an important process for the formation of aggregates of chromophores, can be efficiently suppressed by the construction of a repulsive outer sphere of bulky groups. The problem of concentration quenching of fluorescene could be solved with this concept so that dyes with a persistent strong fluorescence even in high concentrated solution could be prepared. The easy detectable interaction of chromophores proved to be a suitable tool for the recognition of intermolecular organisation processes and thus is also important for supramolecular chemistry.

Experimental

General procedure for perylene-3,4:9,10-bis(dicarboximides)

6.0 mmol of a primary amine, perylene-3,4:9,10-tetracarboxylic-3,4:9,10-bisanhydride (980 mg, 2.50 mmol) and imidazole (20 g) were heated at 140°C for 3 h. Water (10 ml) was added after cooling and 2 N hydrochloric acid (70 ml). The precipitate was collected by vacuum filtration after stirring for 12 h, washed with distilled water (100 ml), dried and purified by column separation (silica gel, chloroform).

***N*,*N*′-Bis-(4′-tritylphenyl)-perylene-3,4:9,10-bis(dicarboximide)(2c).** 4-Tritylaniline[14] (2.00 g, 5.96 mmol), perylene-3,4:9,10-tetracarboxylic-3,4:9,10-bisanhydride (980 mg, 2.50 mmol) and imidazole (20 g) were allowed to react according to the general procedure and purified by a second column separation (silica gel, chloroform/ethanol 20:1). Yield 2.14 g (85%) of **2c** as a bright red solid, mp >360°C. R_f ($CHCl_3$): 0.22. IR (KBr): $\tilde{\nu}$=3435 cm^{-1} (s br.), 2924 (w, CH aliphat.), 2853 (w), 1709 (s, C=O), 1671 (s), 1593 (m, CC arom.), 1579 (w), 1493 (w), 1432 (w), 1404 (w, CC arom.), 1356 (s), 1256 (m), 1177 (w), 1122 (w), 1035 (w), 958 (w), 834 (w), 810 (m, CH arom.), 745 (m), 702 (m). UV ($CHCl_3$): λ_{max} (ϵ)=459.6 nm (14850), 490.9 (51740), 527.8 (90200). Fluorescence ($CHCl_3$): λ_{max}= 538 nm, 577. Solid-state fluorescence: λ_{max}=593 nm. MS (70 eV): *m*/*z* (%): 1027 (7) [M^+], 1026 (9), 952 (7), 951 (27), 950 (76) [M^+-Ph], 949 (100), 691 (9), 437 (10), 436 (15), 243 (27), 241 (17), 165 (18). $C_{74}H_{46}N_2O_4$ (1027.2): calcd C 86.52, H 4.51, N 2.73; found C 85.58, H 4.56, N 2.65.

Reaction of 1 with 2,4,6-triphenylaniline. 2,4,6-Triphenylaniline (230 mg, 0.72 mmol), zinc acetate dihydrate (160 mg, 0.73 mmol), **1** (140 mg, 0.36 mmol) and imidazole (15 g) were allowed to react (autoclave, 24 h, 175°C) according to the general procedure and purified by a second column separation (silica gel, chloroform/ethanol 20: 1). No **2b** could be detected, but a small amount of **3b**. Yield 1 mg (0.3%) of **3b** as a red powder, mp >360°C. R_f ($CHCl_3$/ethanol 10:1): 0.71. R_f ($CHCl_3$): 0.27. IR (KBr): $\tilde{\nu}$=3436 cm^{-1} (s br.), 3057 (w, CH arom.), 1698 (s,

C=O), 1656 (s), 1592 (m, CC arom.), 1577 (m), 1494 (w), 1458 (w), 1434 (w), 1408 (w), 1362 (s), 1293 (w), 1248 (w), 1199 (w), 1178 (w), 1137 (w), 1032 (w), 921 (w), 890 (w), 812 (m), 758 (w), 701 (w). ^{1}H NMR ($CDCl_3$): δ=7.01–7.05 (m, 1H, phenyl-H), 7.09–7.12 (m, 3H, phenyl-H), 7.30–7.34 (m, 2H, phenyl-H), 7.36–7.43 (m, 7H, phenyl-H), 7.52–7.54 (m, 2H, perylen-H), 7.63–7.66 (m, 6H, phenyl-H), 7.80 (d, J=8.0 Hz, 2H, perylene-H), 8.24 (d, J=8.0 Hz, 2H, perylene-H), 8.32 (d, J=7.4 Hz, 2H, perylene-H), 8.35 (d, J=8.0 Hz, 2H, perylene-H). UV/Vis ($CHCl_3$): λ_{max} (ϵ)=353.6 nm (4060), 450.5 (sh., 14240), 485.5 (28050), 510.1 (26950). Fluorescence ($CHCl_3$): λ_{max}=537 nm, 570. MS (70 eV): *m/z* (%): 627 (11), 626 (47), 625 (100) [M^+], 608 (7), 581 (20), 580 (41), 312 (7), 304 (7), 276 (6).

***N,N′*-Bis(2-(1-propylbutyl)-3-propyl-hexyl)-perylene-3,4:9,10-bis(dicarboximide) (2d).** **1** (890 mg, 2.3 mmol) and 2-(1-propylbutyl)-3-propyl-hexylamine (1.20 g, 4.97 mmol) were allowed to react according to the general procedure. Yield 1.46 g (77%) of **2d** as orange fluorescent crystal needles, mp 335°C. R_f (silica gel, chloroform): 0.80. IR (KBr): $\tilde{\nu}$=3436 cm^{-1} m, 2957 s, 2929 s, 2871 m, 1698 s, 1659 s, 1595 s, 1580 w, 1443 w, 1404 w, 1335 m, 1249 w, 1176 w, 1107 w, 853 w, 811 m, 784 w. ^{1}H NMR (300 MHz, $CDCl_3$): δ=0.78 (t, J=7.2 Hz, 12H, $4CH_3$), 0.91 (t, J=7.3 Hz, 12H, $4CH_3$), 1.20 (m_c, 16H, $8CH_2$), 1.33 (m_c, 16H, $8CH_2$), 1.66 (m_c, 4H, 4 γ-CH), 2.21 (t, J=7.4 Hz, 2H, 2β-CH), 4.24 (d, J=7.4 Hz, 4H, 2α-CH_2), 8.60 (d, J=8.0 Hz, 4H, perylene), 8.66 (d, J=8.0 Hz, 4H, perylene). ^{13}C NMR (75 MHz, $CDCl_3$): δ=14.41, 14.55, 21.18, 21.47, 33.43, 34.53, 37.56, 39.38, 39.71, 123.07, 123.40, 126.50, 129.35, 131.41, 134.61, 163.74. UV/Vis ($CHCl_3$): λ_{max} (ϵ)=434.1 nm (7350), 458.5 (21700), 488.9 (56900), 525.9 (94200). Fluorescence ($CHCl_3$): λ_{max}=534 nm, 574, 621. Fluorescence quantum yield ($CHCl_3$, reference *N,N′*-(1-hexylheptyl)-perylene-3,4:9,10-bis(dicarboximide) with Φ=100%): 99%. Solid-state fluorescence: λ_{max}=596 nm. MS (70 eV): *m/z* (%): 840 (11), 839 (36), 838 (58) [M^+], 783 (26), 782 (44), 740 (11), 627 (9), 617 (12), 616 (39), 615 (63), 614 (12), 560 (26), 559 (45), 558 (12), 517 (14), 418 (10), 417 (9), 406 (12), 405 (44), 404 (67), 403 (20), 393 (17), 392 (68), 391 (100), 390 (87), 373 (16), 345 (9), 199 (16), 111 (9), 97 (15), 91 (14), 83 (24), 71 (11), 69 (41), 57 (58), 55 (32). $C_{56}H_{74}N_2O_4$ (839.2): calcd C 80.15, H 8.89, N 3.34; found C 80.33, H 8.79, N 3.57.

***N,N′*-Bis(-2-(1-butylpentyl)-3-butylheptyl)-perylene-3,4:9,10-bis(dicarboximide) (2e).** **1** (300 mg, 0.8 mmol), 3-butyl-2-(1-butylpentyl)-heptylamine (600 mg, 2.0 mmol) and imidazole (900 gm) were allowed to react (160°C, 3 h) according to the general procedure. Yield 600 mg (81%) of **2e**, mp 238°C. R_f (silica gel/$CHCl_3$): 0.81. IR (KBr): $\tilde{\nu}$=2923 cm^{-1} s, 2857 s, 1691 s, 1651 s, 1594 s, 1579 s, 1508 w, 1467 m, 1446 m, 1404 s, 1372 s, 1338 s, 1247 m, 1177 m, 1130 w, 1110 w, 1016 w, 871 w, 816 s, 796 w, 749 m. ^{1}H NMR (300 MHz, $CDCl_3$): δ=0.82 (t, J=7.1 Hz, 12H, $4CH_3$), 0.88 (t, J=7.2 Hz, 12H, $4CH_3$), 1.20–1.55 (m, 34H, $17CH_2$), 1.65 (m_c, 18H, $8CH_2$+2γ-CH), 2.14 (m_c, 2H, 2β-CH), 4.22 (d, J=7.2 Hz, 4H, 2α-CH_2), 8.56 (d, J=8.1 Hz, 4H, perylene), 8.63 (d, J=8.1 Hz, 4H, perylene). ^{13}C NMR (75 MHz, $CDCl_3$): δ=14.06 (CH_3), 14.21 (CH_3), 22.97 (CH_2), 23.13 (CH_2), 29.60 (CH_2), 30.39 (CH_2), 30.63 (CH_2), 30.78 (CH_2), 31.91 (CH_2), 38.13 (γ-CH), 39.37 (β-CH), 39.68 (α-CH_2), 122.98 (perylene), 123.34 (perylene), 131.33 (perylene), 134.50 (perylene), 163.67 (N–C=O). UV/Vis ($CHCl_3$): λ_{max} (ϵ)=458.2 nm (18700), 488.2 (51500), 525.1 (84800). Fluorescence ($CHCl_3$): λ_{max}=534 nm, 574, 621. Fluorescence quantum yield ($CHCl_3$, reference *N,N′*-(1-hexylheptyl)-perylene-3,4:9,10-bis(dicarboximide) with Φ=100%): 97%. Solid-state fluorescence: λ_{max}=587.9 nm. MS (70 eV): *m/z* (%): 951 (70) [M^++1], 950 (100) [M^+], 824 (3) [M^++1−C_9H_{19}], 823 (5) [M^+−C_9H_{19}], 684 (5) [M^++1−$C_{19}H_{39}$], 683 (7) [M^+−$C_{19}H_{39}$], 673 (24), 672 (70), 671 (88), 544 (2) [671−C_9H_{19}], 541 (3), 418 (4), 417 (5) [544C_9H_{19}], 405 (26), 404 (35) [671−$C_{19}H_{39}$], 403 (10), 393 (19), 392 (63), 391 (68), 390 (52), 373 (13), 346 (6), 345 (7), 111 (8), 97 (18), 71 (12), 69 (20), 57 (18), 55 (20). $C_{64}H_{90}N_2O_4$ (951.4): calcd C 80.80, H 9.53, N 2.94; found C 81.10, H 9.65, N 3.18.

***N,N′*-Bis(2-(1-pentylhexyl)-3-pentyl-octyl)-perylene-3,4:9,10-bis(dicarboximide) (2f).** **1** (200 mg, 0.51 mmol) and the rough mixture of amines containing 1-amino-2-(1-pentylhexyl)-3-pentyl-octane (400 mg) were allowed to react according to the general procedure. Yield 300 mg (55%) of **2f** as red, fluorescent crystal needles, mp 195–196°C. R_f (silica gel, chloroform): 0.83. R_f (silica gel, toluene): 0.65. IR (KBr): $\tilde{\nu}$=3441 cm^{-1} w, 2958 s, 2928 s, 2857 s, 1693 s, 1658 s, 1594 s, 1583 w, 1467 m, 1406 m, 1332 m, 1247 m, 1110 w, 871 w, 813 s, 796 w, 751 m. ^{1}H NMR (300 MHz, $CDCl_3$): δ=0.74 (t, J=6.9 Hz, 12H, $4CH_3$), 0.88 (t, J=6.4 Hz, 12H, $4CH_3$), 1.00–1.18 (m, 16H, $8CH_2$), 1.20–1.35 (m, 32H, $16CH_2$), 1.66 (m_c, 4H, 4γ-CH), 2.14 (t, J=7.1 Hz, 2H, 2β-CH), 4.22 (d, J=7.2 Hz, 4H, 2α-CH_2), 8.53 (d, J=8.13 Hz, 4H, perylene), 8.62 (d, J=8.0 Hz, 4H, perylene). ^{13}C-DEPT-NMR (75 MHz, $CDCl_3$): δ=14.41, 14.58, 23.01, 23.18, 28.21, 28.37, 30.10, 31.39, 32.53, 32.74, 38.43 (γ-CH), 39.69 (β-CH), 40.03 (α-CH_2), 123.35, 131.71. ^{13}C NMR (75 MHz, $CDCl_3$): δ=14.41, 14.58, 23.01, 23.18, 28.21, 28.37, 30.10, 31.39, 32.53, 32.74, 38.43, 39.69, 40.03, 123.35, 123.77, 126.76, 129.67, 131.71, 134.85, 164.02. UV/Vis ($CHCl_3$): λ_{max} (ϵ)=434.1 nm (4750), 458.5 (17800), 489.1 (50500), 525.9 (85400). Fluorescence ($CHCl_3$): λ_{max}=534 nm, 574, 621. Fluorescence quantum yield ($CHCl_3$, reference *N,N′*-(1-hexylheptyl)-perylene-3,4:9,10-bis(dicarboximide) with Φ=100%): 97%. Solid-state fluorescence: λ_{max}=624 nm. MS (70 eV): *m/z* (%): 1066 (8), 1065 (30), 1064 (77), 1063 (100) [M^+], 908 (5) [M^+−$C_{11}H_{23}$], 881 (8), 729 (18), 728 (49), 727 (57), 726 (12), 545 (7), 521 (6), 450 (6), 405 (15), 404 (21), 393 (7), 392 (34), 391 (41), 390 (34), 368 (21), 367 (79), 366 (9), 365 (7), 297 (12), 296 (73), 268 (10), 213 (10), 142 (7), 125 (6), 111 (7), 100 (29), 97 (12), 85 (11), 83 (13), 71 (16), 69 (18), 57 (26), 55 (19). $C_{72}H_{106}N_2O_4$ (1063.6): calcd C 81.30, H 10.04, N 2.63; found C 81.24, H 9.46, N 2.75.

***N*-(1-Hexylheptyl)-*N′*-(4′-tritylphenyl)-perylene-3,4:9,10-bis(dicarboximide) (5c).** *N*-(1-Hexylheptyl)-perylene-3,4:9,10-tetracarboxylic-3,4-anhydride-9,10-carboximide[10] (**4a**, 270 mg, 0.45 mmol), 4-tritylaniline (600 mg, 1.79 mmol) and imidazole (15 g) were allowed to react according to the general procedure. The precipitate was purified by column separation (silica gel, chloroform/ethanol 20:1) and a second column separation (silica gel, chloroform).

Yield 230 mg (56%) of **5c** as a dark red solid, mp 292–293°C. R_f ($CHCl_3$): 0.24. IR (KBr): $\tilde{\nu}$=3435 cm^{-1} (s br.), 3056 (w), 3030 (w), 2953 (w, CH aliphat.), 2926 (w), 2856 (w), 1698 (s, C=O), 1660 (s), 1594 (m, CC arom.), 1579 (w), 1507 (w), 1493 (w), 1433 (w), 1343 (s), 1254 (m), 1175 (w), 1137 (w), 1124 (w), 1107 (w), 1035 (w), 966 (w), 853 (w), 811 (w), 747 (m), 702 (w). ^{1}H NMR ($CDCl_3$): δ=0.83 (t, J=6.7 Hz, 3H, CH_3), 1.23–1.32 (m, 16H, $8CH_2$), 1.83–1.90 (m, 2H, CH_2), 2.22–2.30 (m, 2H, CH_2), 5.16–5.19 (m, 2H, CH), 7.21–7.30 (m, 19H, phenyl-H), 8.60–8.73 (m, 8H, perylene-H). ^{13}C NMR ($CDCl_3$): δ=14.06, 22.59, 26.94, 29.22, 31.76, 32.36, 54.83, 64.91, 123.10, 123.30, 126.08, 126.41, 126.65, 127.34, 127.53, 127.60, 129.54, 129.81, 131.23, 131.78, 132.10, 132.72, 134.28, 135.10, 146.46, 147.36, 163.59. UV ($CHCl_3$): λ_{max} (ϵ)=366.7 nm (4650), 430.0 (sh., 6560), 458.8 (21150), 490.6 (56350), 527.0 (93430). Fluorescence ($CHCl_3$): λ_{max}=535 nm, 575. MS (70 eV): *m/z* (%): 892 (12), 891 (35) [M^+], 890 (51), 815 (9), 814 (19), 813 (47) [$M^+-C_6H_6$], 711 (8), 710 (26), 709 (51) [$M^+-C_{13}H_{26}$], 633 (15), 632 (54) [$M^+-C_{13}H_{26}$-Ph], 631 (100) [$M^+-C_{13}H_{26}-C_6H_6$], 541 (7), 374 (14), 373 (51), 346 (17), 345 (25), 319 (7), 317 (8), 316 (26), 243 (28), 241 (27), 239 (9), 165 (26), 69 (7), 55 (11). $C_{62}H_{54}N_2O_4$ (891.1): calcd C 83.57, H 6.11, N 3.14; found C 83.41, H 6.07, N 3.17.

***N*-(1-Hexylheptyl)-*N'*-(3,4,5,6-tetraphenylphthalimidyl)-perylene-3,4:9,10-bis(dicarboximide) (5b). 4a** (160 mg, 0.272 mmol), 3,4,5,6-tetraphenylphthalic acid anhydride (346 mg, 0.765 mmol) and imidazole (2 g) were allowed to react (130°C, 3 h) according to the general procedure and purified by column separation (silica gel, chloroform/acetone 15:1). Yield 210 mg (75%) of **5b** as a dark red powder, mp 312°C. R_f (silica gel, chloroform/acetone 15:1): 0.19. IR (KBr): $\tilde{\nu}$=3436 cm^{-1} s, 2927 m, 2856 w, 1749 s, 1726 m, 1700 s, 1660 s, 1594 s, 1579 m, 1444 w, 1405 m, 1337 s, 1325 s, 1281 w, 1251 w, 1211 w, 1174 w, 1128 w, 1029 w, 965 w, 942 w, 856 w, 810 m, 780 w, 768 w, 756 w, 739 m, 654 w, 641 w, 562 w, 496 w. ^{1}H NMR (300 MHz, $CDCl_3$): δ=0.82 (t, J=6.8 Hz, 6H, $2CH_3$), 1.16–1.4 (m, 16H, $8CH_3$), 1.87 (m_c, 2H, β-CH_2), 2.26 (m_c, 2H, β-CH_2), 5.19 (m_c, 1H, α-CH), 6.80–6.84 (m, 4H, phenyl), 6.91–6.97 (m, 6H, phenyl),71.3–7.23 (m, 10H, phenyl), 8.52 (d, J=8.3 Hz, 2H, perylene), 8.58 (d, J=8.1 Hz, 2H, perylene), 8.59 (d, J=8.2 Hz, 2H, perylene), 8.67 (m_c, 2H, perylene). ^{13}C NMR (75 MHz, $CDCl_3$): δ=14.5, 23.0, 27.3, 29.6, 30.1, 32.1, 32.8, 55.3, 122.5, 123.3, 124.1, 124.7, 126.6, 126.9, 127.1, 127.5, 127.8, 127.9, 129.8, 129.9, 130.2, 130.4, 131.1, 132.7, 134.3, 135.6, 136.2, 138.2, 140.9, 149.2, 160.6, 163.2. UV/Vis ($CHCl_3$): λ_{max} (ϵ)=352.8 nm (8800), 368.0 (7100), 434 (6000), 459.6 (19100), 491.1 (51100), 527.4 (84100). Fluorescence ($CHCl_3$): λ_{max}=539 nm, 577, 631 sh. Solid-state fluorescence: λ_{max}=628 nm. MS (70 eV): *m/z* (%): 1023 (5), 1022 (13), 1021 (16) [M^+], 1005 (3), 937 (1), 936 (1), 900 (3), 842 (6), 841 (20), 840 (40), 839 (37) [$M^+-C_{13}H_{26}$], 838 (8), 822 (2), 762 (1) [$M^+-C_{13}H_{26}$ C_6H_5], 453 (6), 452 (34), 451 (100) [$(C_6H_5)_4$ $C_6C_2O_2NH^+$=R^+], 450 (28), 449 (5), 448 (7), 434 (4), 433 (6), 432 (9), 422 (4), 420 (4), 408 (5), 407 (13), 406 (5), 405 (7), 404 (7), 403 (5), 402 (7), 401 (3), 392 (4), 391 (9), 390 (15) [M^++2H−$C_{13}H_{26}$–R], 389 (5), 388 (4), 379 (7), 378 (8), 377 (11), 376 (13), 375 (5), 374 (11) [$R^+-C_6H_5$], 373 (6), 372 (5), 364 (6), 363 (8), 328 (4), 327 (5), 303 (4), 302 (8), 301 (4), 194 (4), 188 (6), 187 (5), 181 (5), 180 (4), 174 (3), 55 (4). $C_{69}H_{55}N_3O_6$ (1022.2): calcd C 81.07, H 5.43, N 4.11; found C 81.13, H 5.83, N 3.81.

***N*-(1-Hexylheptyl)-*N'*-(2-(1-propylbutyl)-3-propyl-hexyl)-perylene-3,4:9,10-bis(dicarboximide) (5d). 4a** (1.27 g, 2.20 mmol), 2-(1-propylbutyl)-3-propyl-hexylamine (900 mg, 3.7 mmol) were allowed to react according to the general procedure. Yield 980 mg (50%) of **5d**; mp 148°C. R_f (silica gel, toluene): 0.41. IR (KBr): $\tilde{\nu}$=2957 cm^{-1} s, 2930 s, 2860 m, 1697 s, 1653 s, 1595 s, 1580 m, 1457 w, 1405 m, 1338 s, 1251 m, 1177 w, 855 w, 810 m, 747 m. UV/Vis ($CHCl_3$): λ_{max} (ϵ)=434.1 nm (4730), 458.5 (18000), 489.1 (50500), 526.0 (85200). Fluorescence ($CHCl_3$): λ_{max}=534 nm, 574, 621. Fluorescence quantum yield ($CHCl_3$, reference *N,N'*-(1-hexylheptyl)-perylene-3,4:9,10-bis(dicarboximide) with Φ=100%): 99%. ^{1}H NMR (300 MHz, $CDCl_3$): δ=0.80 (m_c, 18H, $6CH_3$), 1.16–1.57 (m, 34H, 2γ-CH+$16CH_2$), 1.89 (m_c, 2H, β-CH_2), 2.24 (m_c, 3H, β-CH+β-CH_2), 4.22 (d, J=7.1 Hz, 2H, α-CH_2), 5.18 (quint, J=7.0 Hz, 1H, α-CH), 8.53 (d, J=8.1 Hz, 2H, perylene), 8.58 (d, J=8.3 Hz, 2H, perylene), 8.61 (d, J=8.0 Hz, 4H, perylene). ^{13}C NMR (75 MHz, $CDCl_3$): δ=14.44, 14.76, 14.90, 21.51, 21.81, 22.99, 27.37, 29.63, 32.17, 32.79, 33.75, 34.85, 37.86, 39.67, 40.00, 55.20, 123.36, 123.68, 126.73, 129.66, 129.94, 131.27, 131.70, 134.87, 164.02. MS (70 eV): *m/z* (%): 798 (55) [M^++1], 797 (100) [M^+], 615 (5) [M^++1−$C_{13}H_{27}$], 573 (78) [M^++1−$C_{16}H_{33}$], 572 (26) [$M^+-C_{16}H_{33}$], 574 (38), 405 (13), 404 (27), 403 (13), 392 (35), 391 (85), 390 (97), 374 (3), 373 (10), 345 (3), 379 (6), 167 (8), 150 (3), 149 (24),111 (3), 104 (3), 97 (5), 85 (15), 83 (23), 71 (7), 70 (8), 69 (18), 67 (3), 57 (32), 56 (6), 55 (18), 48 (3), 47 (7). $C_{53}H_{68}N_2O_4$: calcd 796.5179; found 796.5161 (MS).

***N*-(2-(1-Butylpentyl)-3-butyl-heptyl)-*N'*-(1-hexylheptyl)-perylene-3,4:9,10-bis(dicarboximide) (5e). 4a** (440 mg, 0.76 mmol), 2-(1-butylpentyl)-3-butyl-heptylamine (250 mg, 0.84 mmol) were allowed to react according to the general procedure. Yield 490 mg (76%) of **5e** as red, fluorescent needles, mp 131°C. R_f (silica gel, chloroform): 0.61. R_f (silica gel, toluene): 0.55. IR (KBr): $\tilde{\nu}$=3442 cm^{-1} m, 2957 s, 2928 s, 2857 s, 1699 s, 1660 s, 1597 s, 1580 s, 1507 w, 1467 m, 1447 m, 1406 s, 1339 s, 1254 m, 1176 m, 1131 w, 1112 w, 1017 w, 853 m, 810 s, 799 w, 748 m. ^{1}H NMR (300 MHz, $CDCl_3$): δ=0.78 (t, J=6.7 Hz, 12H, $4CH_3$), 0.88 (t, J=7 Hz, 6H, $2CH_3$), 1.17–1.35 (m, 40H, $20CH_2$), 1.66 (m_c, 2H, γ-CH), 1.85 (m_c, 2H, β-CH_2), 2.16–2.24 (m, 3H, β-CH_2+β-CH), 4.22 (d, J=7.3 Hz, 2H, α-CH_2), 5.16 (quint, J=7.6, 1H, α-CH), 8.57 (d, J=8.1 Hz, 2H, perylene), 8.58 (d, J=8.1 Hz, 2H, perylene), 8.65 (d, J=8.0 Hz, 4H, perylene). ^{13}C NMR (75 MHz, $CDCl_3$): δ=14.44, 14.48, 14.63, 22.99, 23.54, 17.35, 29.62, 30.80, 31.19, 32.33, 32.78, 38.53, 39.78, 40.08, 55.18, 123.42, 123.79, 126.91, 129.75, 129.99, 131.79, 134.96, 164.10. UV/Vis ($CHCl_3$): λ_{max} (ϵ)=433.0 nm (6430), 458.1 (19800), 488.9 (53300), 525.9 (88900). Fluorescence ($CHCl_3$): λ_{max}=534 nm, 574, 621. Fluorescence quantum yield ($CHCl_3$, reference *N,N'*-(1-hexylheptyl)-perylene-3,4:9,10-bis(dicarboximide) with Φ=100%): 100%. Solid-state fluorescence: λ_{max}=605 nm. MS (70 eV): *m/z* (%): 855

(7), 854 (20), 853 (31) [M^+], 586 (5), 585 (7), 575 (12), 574 (40), 573 (77), 572 (19), 405 (14), 404 (29), 403 (14), 393 (10), 392 (41), 391 (95), 390 (100), 373 (16), 345 (8), 279 (10), 250 (9), 167 (13), 149 (26), 126 (19), 111 (14), 97 (16), 85 (23), 71 (37), 70 (12), 69 (23), 57 (40), 55 (35). $C_{57}H_{76}N_2O_4$ (853.3): calcd C 80.24, H 8.98, N 3.28; found C 80.44, H 8.96, N 3.40.

***N*-(2-(1-Pentylhexyl)-3-pentyloctyl)-*N'*-(1-octylnonyl)-perylene-3,4:9,10-bis(dicarboximide) (5f).** *N*-(1-Octylnonyl)-perylene-3,4:9,10-tetracarboxylic-3,4-anhydride-9,10-imide (**4f**, 300 mg, 0.48 mmol) and the rough 1-amino-2-(1-pentylhexyl)-3-pentyl-octane (300 mg) were allowed to react according to the general procedure. *First fraction:* trace amount of *N*-(2-bis(1-pentylhexyl)-3-pentyl-octyl)-*N'*-(1-octylnonyl)-perylene-3,4:9,10-bis(dicarboximide). R_f (silica gel, chloroform): 0.98. R_f (silica gel, toluene): 0.86. UV/Vis ($CHCl_3$): λ_{max}=434.1 nm, 458.5, 489.1, 526.0. Fluorescence ($CHCl_3$): λ_{max}=534 nm, 574, 621. MS (70 eV): *m/z* (%): 1120.8 (1), 1119.8 (3) [M^+], 1118.8 (3), 1117.8 (1), 1077.7 (1), 1077.6 (1), 1036.8 (1), 1035.7 (3), 1034.7 (3), 1022.7 (1), 1021.7 (3) [$M^+-C_8H_{16}$], 1020.7 (1), 1007.7 (1), 1006.6 (1), 963.6 (1) [$M^+-C_{11}H_{24}$], 937.5 (1), 936.6 (1), 882.5 (1) [$M^+-C_{17}H_{34}$], 810.4 (1) [$M^+-2\times C_{11}H_{24}$], 783.4 (2), 699.4 (2), 685.3 (1), 667.6 (1), 654.4 (1) [$M^+-3\times C_{11}H_{24}$], 639.6 (1), 628.6 (1) [$M^+-CH_2-C(C_{11}H_{24})_3$], 628.5 (1), 627.6 (2), 626.6 (5), 624.5 (2), 623.5 (2), 622.6 (1), 528.5 (9), 472.4 (20), 471.4 (11), 401.3 (13), 374.3 (14), 373.3 (9), 360.3 (17), 359.3 (10), 331.2 (9), 318.2 (17), 359.3 (9), 331.2 (9), 318.2 (17), 317.2 (14), 303.2 (12), 295.2 (18), 278.2 (37), 251.2 (8), 247.2 (19), 220.2 (13). 213.2 (10), 206.1 (12), 183.1 (10), 177.1 (11), 155.1 (9), 154.1 (27), 141.1 (16), 125.1 (11), 124.1 (28), 112.1 (10), 111.1 (18), 109.1 (10), 99.1 (11), 98.1 (13), 97.1 (35), 96.1 (21), 95.1 (19), 85.1 (34), 84.1 (18), 83.1 (37), 82.0 (18), 71.1 (51), 70.0 (35), 69.0 (64), 68.0 (13), 67.0 (28), 57.1 (100), 56.1 (47), 55.1 (93), 54.0 (13), 53.0 (10). *Second fraction:* Yield 250 mg (54%) of **5f** as red, fluorescent needles, mp 108°C. R_f (silica gel, chloroform): 0.70. R_f (silica gel, toluene): 0.60. IR (KBr): $\tilde{\nu}$=3440 cm^{-1} m, 2958 s, 2926 s, 2855 s, 1698 s, 1658 s, 1595 s, 1579 s, 1508 w, 1467 m, 1446 m, 1405 s, 1339 s, 1253 m, 1174 m, 1130 w, 1112 w, 1016 w, 851 m, 810 s, 799 w, 747 m. ^{1}H NMR (300 MHz, $CDCl_3$): δ=0.72 (t, *J*=7.2 Hz, 6H, $2CH_3$), 0.80 (t, *J*=6.9 Hz, 6H, $2CH_3$), 0.89 (t, *J*=7.0 Hz, 6H, $2CH_3$), 1.17–1.48 (m, 60H, $30CH_2$), 1.64 (m_c, 2H, 2γ-CH), 1.90 (m_c, 2H, β-CH_2), 2.15–2.27 (m, 3H, β-CH_2+β-CH), 4.22 (d, *J*=7.3 Hz, 2H, α-CH_2), 5.15 (quint, *J*=7.5 Hz, 1H, α-CH), 8.59 (d, *J*=8.3 Hz, 4H, perylene), 8.66 (d, *J*=8.2 Hz, 4H, perylene). ^{13}C NMR (75 MHz, $CDCl_3$): δ=14.07, 14.18, 22.62, 22.77, 26.97, 27.80, 27.98, 29.24, 29.50, 29.55, 31.00, 31.83, 32.13, 32.38, 39.68, 54.77, 123.04, 123.31, 131.42, 134.54, 164.23. UV/Vis ($CHCl_3$): λ_{max} (ϵ)=534.1 nm (5230), 458.9 (18700), 489.9 (52100), 526.0 (87700). Fluorescence ($CHCl_3$): λ_{max}=534 nm, 574, 621. Fluorescence quantum yield ($CHCl_3$ reference *N,N'*-(1-hexylheptyl)-perylene-3,4:9,10-bis(dicarboximide) with Φ=100%): 98%. Solid-state fluorescence: λ_{max}=610 nm. MS (70 eV): *m/z* (%): 968 (7), 967 (25), 966 (69), 965 (100) [M^+], 869 (9), 868 (28), 867 (42), 727 (5), 642 (6), 641 (7), 631 (14), 630 (50), 629 (90), 628 (40), 405 (10), 404 (17), 403 (9), 392 (9), 391 (30), 390 (92), 373 (6), 281 (16), 126 (8), 97 (15), 83 (17), 72 (44), 69 (20), 59 (88), 57 (26). $C_{65}H_{92}N_2O_4$ (965.4): calcd C 80.87, H 9.60, N 2.90; found C 80.89, H 9.57, N 3.06.

1-Cyano-1-(1-propylbutyl)-2-propylpentane. 1.6 m *tert*-Butyllithium solution in pentane (40 ml, 0.064 mol) was evaporated under argon below 50°C. Anhydrous ether (13 ml, 78°C) and a solution of anhydrous acetonitrile (700 mg, 17 mmol) in anhydrous ether (13 ml) were added. The mixture was warmed up to 40°C (evolution of 2-methylpropane), a solution of bromoheptane (16 g, 89 mmol) in anhydrous tetrahydrofuran (18 ml) was added dropwise with stirring, kept at this temperature for 6 h with stirring, warmed up to room temperature, quenched by the addition of water (17 ml), extracted with ether, dried (magnesium sulphate) and distilled in vacuo. Yield 4.15 g (20%) of 1-cyano-1-(1-propylbutyl)-2-propylpentane, bp 97°C/2 mbar. IR (film): $\tilde{\nu}$=2978 s, 2935 s, 2868 s, 2222 w (CN), 1742 s, 1387 m, 737 w. ^{1}H NMR (300 MHz, $CDCl_3$): δ=0.83 (t, *J*=6.9 Hz, 6H, $2CH_3$), 0.90 (t, *J*=7.1 Hz, 6H, $2CH_3$), 1.2–1.4 (m, 18H, CH, CH_2), 2.49 (t, *J*=7.0 Hz, 1H, HC–CN). ^{13}C NMR (75 MHz, $CDCl_3$): δ=13.8, 14.9, 19.5, 35.5, 36.5, 43.5, 121.4. MS (70 eV): *m/z* (%): 238 (5) [M^++1], 194 (49) [M^+-C_3H_7], 96 (100) [$M^+-C_3H_7-C_7H_{14}$], 57 (17).

1-Cyano-1-(1-butylpentyl)-2-propylpentane. Sodium (1.5 g, 65 mmol) was dissolved in liquid ammonia (20 ml, 78°C). Ferric nitrate (200 mg, 0.83 mmol) was added. After 2 h a solution of anhydrous acetonitrile (600 mg, 15 mmol) and 5-bromoundecane (12 g, 58 mmol) in anhydrous ether (12 ml) was added dropwise with stirring. Ammonia was evaporated (3 h) and anhydrous ether was added (20 ml). After stirring for 16 h anhydrous ethanol was cautiously added and then 2 N sulphuric acid (50 ml). The reaction product was obtained by extraction with ether, drying (magnesium sulphate), and vacuum fractionation by means of a Vigreux column (20 cm). Yield 200 mg (5%) of 1-cyano-1-(1-butylpentyl)-2-propylpentane as viscous oil, bp 113°C/0.3 mbar. IR (film/KBr): $\tilde{\nu}$=2978 s, 2935 s, 2868 s, 2223 w (CN), 1387 m, 737 w. ^{1}H NMR (300 MHz, $CDCl_3$): δ=0.905 (t, *J*=7 Hz, 6H, $2CH_3$), 0.915 (t, *J*=7 Hz, 6H, $2CH_3$), 1.42–1.08 (m, 24H, $12CH_2$), 1.61 (m_c, 2H, β-CH), 2.52 (t, *J*=7 Hz, 1H, HC–CN). ^{13}C NMR (75 MHz, $CDCl_3$): δ=13.97 (CH_3), 14.01 (CH_3), 14.14 (CH_3), 22.85 (CH_2), 22.90 (CH_2), 23.12 (CH_2), 28.10 (CH_2), 28.87 (CH_2), 30.39 (CH_2), 30.45 (CH_2), 30.55 (CH_2), 30.81 (CH_2), 36.51 ((HC)$_2$CH–CN), 39.55 (H*C*–CN), 120.95 (CN). MS (70 eV): *m/z* (%): 293 (2) [M^+], 264 (5) [$M^+-C_2H_5$], 250 (7) [$M^+-C_3H_7$], 237 (17), 236 (89) [$M^+-C_4H_9$], 194 (9), 166 (8) [$M^+-C_9H_{19}$], 110 (100) [M^++1−$C_4H_9-C_9H_{19}$], 82 (8), 71 (13), 57 (12), 55 (12). $C_{20}H_{39}N$ (293.6): calcd C 81.84, H 13.39, N 4.77; found C 81.91, H 13.52, N 4.63.

1-Cyano-1-(1-pentylhexyl)-2-pentylheptane. Acetonitrile (1.5 g, 37 mmol) and 6-bromoundecane (35.0 g, 149 mmol) were allowed to react analogously to 1-cyano-1-(1-butylpentyl)-2-propylpentane. Yield 1.8 g (25%) of 1-cyano-1-(1-pentylhexyl)-2-pentylheptane as viscous oil bp 148°C/1.5 mbar. IR (film/KBr): $\tilde{\nu}$=2972 cm^{-1} s, 2924 s, 2853 s, 2223 w, 1465 s, 1379 m, 728 w. ^{1}H NMR (300 MHz, $CDCl_3$): δ=0.88 (t, *J*=8 Hz, 6H, CH_3), 1.34–1.18 (m, 16H, CH_2), 1.68 (quint, *J*=4 Hz, 1H, β-CH), 2.32

(d, J=8 Hz, 2H, H_2C–CN). ^{13}C NMR (75 MHz, $CDCl_3$): δ=14.09 (CH_3), 22.68 (CH_2), 27.81 (CH_2), 30.64 (CH_2), 32.34 (CH_2), 35.11 (β-CH), 39.57 (H_2C-CN), 118.92 (CN).

1-Amino-3-propyl-2-(1-propylbutyl)-hexane. Lithium aluminium hydride (410 mg, 11 mmol) was dissolved in anhydrous diethylether (30 ml), a solution of 1-cyano-1-(1-propylbutyl)-2-propylpentane (1.66 g, 7.00 mmol) in anhydrous ether (12 ml) was added dropwise, refluxed with stirring for 3 h, cooled with ice, aqueous sodium hydroxide was added (2 ml, 20%), extracted with ether, dried (magnesium sulphate) and distilled in vacuo. Yield 1.8 g (56%) of 1-amino-3-propyl-2-(1-propylbutyl)-hexane, bp 102°C/2 mbar. IR (film): $\tilde{\nu}$=3380 cm^{-1} w (NH_2), 2956 s (CH_3), 2928 s (CH_2), 2872 s (CH_3), 2859 s (CH_2), 1667 w (NH_2), 1064 w (C–N), 790 w (NH_2), 730 w (NH_2). ^1H NMR (300 MHz, $CDCl_3$): δ=0.80 (t, J=8.0 Hz, 6H, $2CH_3$), 0.83 (t, J 8.0 Hz, 6H, $2CH_3$), 1.1–1.4 (m, 16H, CH_2), 1.4–1.5 (m, 3H, CH), 2.60 (d, J=6.9 Hz, α-CH_2). ^{13}C NMR (75 MHz, $CDCl_3$): δ=14.9 (CH_3), 19.9–37 (CH_2), 38.0 (β-CH), 40.8 (α-CH_2).

1-Amino-3-butyl-2-(1-butylpentyl)-heptylamine. 1-Cyano-1-(1-pentylhexyl)-2-pentylheptane (1.0 g, 3.4 mmol) and lithium aluminium hydride (200 mg, 5.3 mmol) were allowed to react analogously to 1-amino-3-propyl-2-(1-propylbutyl)-hexane. Yield 830 mg (82%) of oily 1-amino-3-butyl-2-(1-butylpentyl)-heptylamine, mp 150°C/(1 2) mbar. IR (film/KBr): $\tilde{\nu}$=3380 cm^{-1} w (NH_2), 2956 s (CH_3), 2928 s (CH_2), 2872 s (CH_3), 2859 (CH_2), 1667 w (NH_2), 1466 m (CH_2), 1446 m (CH_2), 1380 m (CH_3), 1064 w (C–N), 790 w (NH_2), 730 w (NH_2). ^1H NMR (300 MHz, $CDCl_3$): δ=0.89 (t, J=8 Hz, 6H, $2CH_3$), 0.90 (t, J=8 Hz, 6H, $2CH_3$), 1.1–1.4 (m, 24H, $12CH_2$), 1.42–1.5 (m, 3H, 3CH), 2.66 (d, J=4 Hz, 2H, α-CH_2). ^{13}C NMR (75 MHz, $CDCl_3$): δ=14.10 (CH_3), 14.12 (CH_3), 22.68 (CH_2), 22.84 (CH_2), 23.12 (CH_2), 23.15 (CH_2), 28.05 (CH_2), 29.80 (CH_2), 29.99 (CH_2), 30.20 (CH_2), 30.38 (CH_2), 30.45 (CH_2), 30.51 (CH_2), 30.78 (CH_2), 31.73 (CH_2), 34.80 (α-CH_2), 36.50 (γ-CH), 36.67 (γ-CH), 38.04 (β-CH). MS (70 eV): m/z (%): 297 (5) [M^+], 280 (39) [M^+-NH_3], 236 (100) [$M^+-1-NH_3-C_3H_7$], 223 (22) [$M^+-NH_3-C_4H_9$], 209 (64) [$M^+-NH_3-C_5H_{11}$], 170 (23) [$M^+-C_9H_{19}$], 168 (22), 154 (24) [$M^+-NH_3+1-C_9H_{19}$], 153 (20) [$M^+-NH_3-C_9H_{19}$], 141 (31), 140 (62) [$M^+-C_9H_{19}$ $H_2C{=}NH_2$], 126 (17), 112 (31), 111 (23), 110 (95) [$M^+-NH_3-C_3H_7-C_9H_{19}$], 98 (35), 97 (37) [$M^+-NH_3-C_9H_{19}-C_4H_9$], 85 (35), 83 (39) [$M^+-C_9H_{19}-H_2C{=}NH_2-C_4H_9$], 71 (54), 70 (35), 69 (38), 57 (58), 56 (26), 55 (54).

1-Amino-2-(1-pentylhexyl)-3-pentyl-octane. 1-Cyano-1-(1-pentylhexyl)-2-pentylheptane (1.0 g) prepared analogously to 1-cyano-1-(1-propylbutyl)-2-propylpentane containing some 1-cyano-1,1-*bis*-(1-pentylhexyl)-2-pentylheptane was allowed to react analogously to 1-amino-3-propyl-2-(1-propylbutyl)-hexane with lithium aluminium hydride (410 mg, 11 mmol). Yield 760 mg of a viscous, oily mixture of 1-amino-3-pentyl-2-(1-pentylhexyl)-octane and some 1-amino-2,2-*bis*-(1-pentylhexyl)-3-pentyl-octane and was used without further purification.

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