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Solubility and decomposition temperature of 1,4-bis(arylamino)anthraquinone dyes

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Abstract

A series of 1,4-bis(arylamino)anthraquinone dyes have been synthesized. 1,4-Bis(2,5-di-*tert*-butylanilino)a thraquinone was 200 times more soluble than 1, 4-dianilinoanthraquinone in hexane at 25°C. The decompositi temperatures of the 1,4-bis(arylamino)anthraquinone derivatives were higher than those of 1, 4-diamino- and 1, 4-bi methylamino)anthraquinones. © 1998 Elsevier Science Ltd. All rights reserved.

Keywords: Solubility; Decomposition temperature; 1,4-bis(arylamino)anthraquinone dyes

1. Introduction

Much effort has been made to improve the solubility of dyes in organic solvents. In the case of 3,4,9,10-perylenetetracarboxydiimides, the *N*, *N'*-bis-(1-hexylheptyl) [1], 1,6,7,12-tetraphenoxy [2], and *N*, *N'*-bis[2-(1*H*,1*H*-perfluoroheptyl)phenyl [3] derivatives have been reported to be much more soluble than the unmodified compounds. Perfluoroalkylated anthraquinone [4] and azo dyes [5] have been reported to be more soluble than their respective non-perfluoroalkylated derivatives. The soluble or homogeneously dispersible 3,6-diphenyl-1,4-diketopyrrolo[3,4-c]pyrrole (DPP) protected by a butoxyoxycarbonyl (BOC) group has been reported [6]. Thus, the improvement in the solubili of dyes is interesting from the viewpoint of the applications. We describe here improvement of t less soluble blue 1,4-bis(arylamino)anthraquino dyes, these being important compounds in colo chemistry. The decomposition temperature (*Td*) the anthraquinone dyes are also examined.

2. Results and discussion

2.1. Synthesis

The 1,4-bis(arylamino)anthraquinone dyes were synthesized as shown in Scheme 1.

Leucoquinizarin (**1**) reacted with substitut anilines **2** in propionic acid in the presence of bor acid to give **3** in moderate to good yields.

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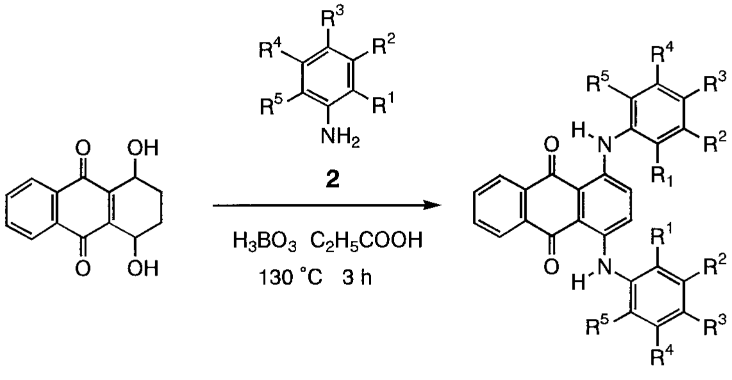
2.2. Solubility

1,4-Diaminoanthraquinone is insoluble in hexane at 25°C. However, the solubilities of the 1,4-bis(methylamino)-, 1,4-dianilino-, and 1,4-bis(1-naphthylamino)anthraquinone dyes were measured to be 0. 1 1, 0. 1 9 and 0.35 mmol dm respectively, suggesting that the 1,4-bis(arylamin derivatives were much more soluble. Therefo the solubilities, of a series of 1,4-bis-(substitut anilino)anthraquinone dyes **3** were examine These results are summarized in Table 1.



1 **3**

Scheme 1.

Table 1
Absorption Band, Solubility, and Decomposition Temperature of 1,4-Bis(arylamino)anthraquinone Dyes

Compound	R^1	R^2	R^3	R^4	R^5	λ_{max}/nm(ε)[a]	Solubility[b]	mp	T
							mmol dm^{-3}	°C	°C
3a	H	H	H	H	H	398 (4690), 591 (8960), 632 (9390)	0.19	223	33
3b	F	H	H	H	H	381 (6620), 577 (11740), 614 (11580)	0.15	—[d]	—
3c	Cl	H	H	H	H	—[c]	—[c]	—[d]	—
3d	Br	H	H	H	H	—[c]	—[c]	—[d]	—
3e	CH_3	H	H	H	H	398 (4760), 590 (11560), 630 (11540)	0.15	225	30
3f	C_2H_5	H	H	H	H	398 (5630), 592 (13600), 633 (14370)	3.89	146	32
3g	C_3H_7	H	H	H	H	400 (5610), 592 (13160), 634 (13440)	9.55	143	32
3h	$CH(CH_3)_2$	H	H	H	H	397 (4450), 591 (11430), 632 (12180)	16.69	129	32
3i	H	$CH(CH_3)_2$	H	H	H	400 (5890), 595 (11920), 636 (12470)	5.80	144	33
3j	H	H	$CH(CH_3)_2$	H	H	403 (6640), 597 (13120), 638 (13840)	0.23	201	33
3k	$C(CH_3)_3$	H	H	H	H	388 (5250), 592 (14840), 638 (15850)	2.18	229	32
3l	C_6H_5	H	H	H	H	403 (6020), 593 (9920), 636 (10430)	0.05	241	35
3m	OCH_3	H	H	H	H	407 (7380), 613 (12310), 639 (12220)	0.15	—[d]	—
3n	CF_3	H	H	H	H	380 (10920), 568 (15270), 608 (13300)	0.25	—[d]	—
3o	CH_3	H	H	H	CH_3	378 (2720), 578 (13650), 623 (14840)	1.38	298	31
3p	C_2H_5	H	H	H	C_2H_5	378 (2880), 580 (15050), 626 (16660)	18.19	209	31
3q	$CH(CH_3)_2$	H	H	H	$CH(CH_3)_2$	379 (1880), 580 (10030), 627 (11250)	19.25	255	31
3r	$CH(CH_3)_3$	H	H	$C(CH_3)_3$	H	378 (3410), 595 (13020), 642 (9590)	40.65	169	29
3s	$CH(CH_3)_2$	H	$CH(CH_3)_2$	H	$CH(CH_3)_2$	379 (1740), 576 (12450), 623 (10050)	20.56	214	31

[a] Measured in hexane.
[b] Measured in hexane at 25°C.
[c] Not measured due to low solubility.
[d] Not measured.

1,4-Bis(2-isopropylanilino)anthraquinone (**3h**) was the most soluble among the 2-, 3-, and 4-isopropyl derivatives **3h–3j**. The solubility of the 1,4-bis(2-substituted anilino)anthraquinone dyes was in the order of the substituent:

$$\mathrm{Cl, Br} < \mathrm{H, F, CH_3, C_6H_5, OCH_3, CF_3} < \mathrm{C(CH_3)_3} < \mathrm{C_2H_5} < \mathrm{C_3H_7} < \mathrm{CH(CH_3)_2}.$$

The 2,6-dialkyl derivatives **3o–3q** were more soluble than the corresponding 2-alkyl derivatives **3e**, **3f**, and **3h**, respectively. Thus, the introduction of bulky alkyl substituents at the 2-and 2,6-positions in the anilino moieties was very effective increasing the solubility. 1,4-Bis(2,5-di-*tert*-butylanilino)anthraquinone (**3r**) showed the highest solubility, being about 200 times more soluble compared with **3a**.The melting point, which represents the intermolecular interactions between the substrates, could be used as a parameter for the solubility. The relationship between the solubility and the melting points is indicated in Fig. 1. The relationship can be classified into two groups: V12 (a) 2-substituted anilino, derivatives **3a–3n** and B) 2, disubstituted anilino derivatives **3o–3s**. In group the solubility was extremely low for compoun whose melting points were higher than *ca* 160° and increased as the melting points decreas when the melting points were lower than 160°C. In group b, the lower the melting poi the higher the solubility.

The relatively high solubility of the 2,6-d ubstituted anilino derivatives could be attribut to the presence of two lypophilic alkyl substitue on both sides of the planar anthraquinone ske ton, which decreased the $\pi-\pi$ intermolecu interactions between the anthraquinone rings a had affinity to hexane. The highest solubility of could be attributed to both the bulky and lyp philic *tert*-butyl groups at the 2- and 5-positions

*2.3. Decomposition temperature (*Td*)*

Monoazo dyes having an *N*, *N*-diaryl moiety ha been reported to have a higher *Td* than tho having an *N,N*-dialkyl group [7]. Therefo

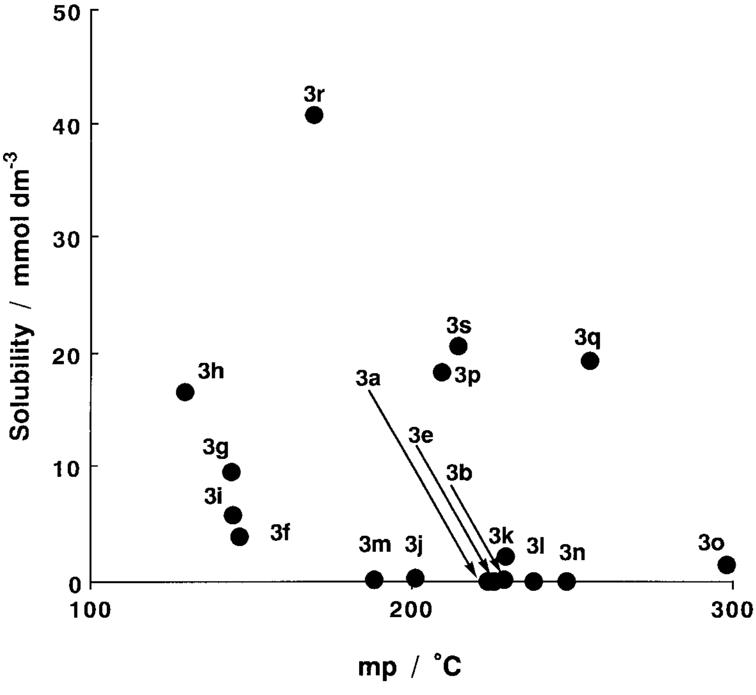


Fig. 1. Relationship between solubility and melting point.

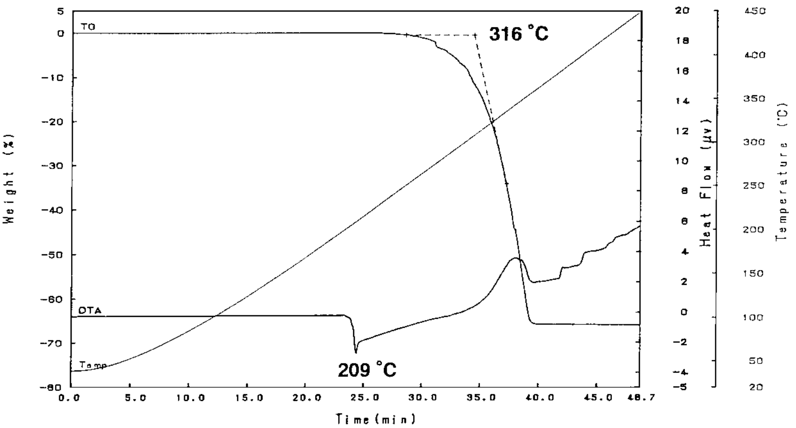


Fig. 2. TG-DTA analysis of **3p**.

investigation of the thermal stability of 1,4-diaminoanthraquinone dyes is of interest. A typical example of the TG-DTA analysis of 1,4-bis(arylamino)anthraquinone dyes is shown in Fig. 2. A sharp endothermic peak was observed at around 209°C, followed by an exothermic peak with decreasing weight. After this measurement, a black residue was formed. These results indicated decomposition of compound **3p** after melting. The *Td*, as defined in the figure, is 316°C. The *Td*'s of selected 1,4-diaminoanthraquinone dyes are also shown in Table 1. The *Td*'s of the 1,4-di(2- and 2,6-disubstituted anilino) (296–356°C) and 1,4-bis(l-naphthylamino) derivatives (351°C) were higher than those of the 1,4-diamino (288°C) and 1,4-bis(methylamino) derivatives (262°C). Thus, the 1,4-bis(arylamino)anthraquinone derivatives were found to be thermally more stable than the 1,4-diamino- and 1,4-bis(methylainino)anthraquinone derivatives.

3. Experimental

3.1. Instruments

Melting points were measured with a Yanagimoto MP-S2 micro melting point apparatus. NMR spectra were obtained by a Jeol α-400 spectrometer. Mass spectra were recorded on a Shimadzu QP-1000 spectrometer. UV spectra were taken on a Shimadzu UV-160A spectrometer. T DTA analysis was performed with a Rigaku TA 200 instrument.

3.2. Materials

Leucoquinizarin (**1**), anilines **2**, and 1,4-dian noanthraquinone were purchased firom Tok Kasei Co., Ltd. 2,4,6-Triisopropylaniline (**2s**) [and 1,4-bis(methylamino)anthraquinone [9] we prepared as described in the literature.

3.3. Synthesis of 1,4-bis(arylamino)anthraquinor dyes 3

To leucoquinizarin **1** (726 mg, 1 mmol) we added propionic acid (1 mmol), the appropria aniline derivative **2** (70 mmol) and boric ac (500 mg, 8 mmol), and heated at 130°C for 3 After the reaction was complete, excess anili was removed by steam-distillation. The resulti precipitate was filtered, dried, purified by colun chromatography, and recrystallized from hexan chloroform. Physical and spectral data are giv below.

*3.3.1. 1,4-Dianilinoanthraquinone (**3a**)*

Yield 84 %; M.p. 223°C ^{1}H NMR (CDC1$_3$) δ 7.17 (t, *J* = 7.3 Hz, 2H), 7.27 (d, *J*=6.1Hz, 4H 7.38 (t, *J* = 7.3 Hz, 4H), 7.45 (s, 2H), 7.74 (dd, = 6.0 and 3.7 Hz, 2H), 8.36 (dd, *J* = 6.0 and 3

Hz, 2H), 12.23 (s, 2H); MS (El, 70 eV) *m*/*z* (%) 390 (100) [M$^+$], 389 (66).

*3.3.2. 1,4-Bis(2-fluoroanilino)anthraquinone (**3b**)*

Yield 85 %; M. p. 228–230°C T; ^{1}H NMR (CDC1$_3$) δ = 7.13-7.21 (m, 6H), 7.36-7.41 (m,4H), 7.77 (dd, *J* = 6.0 and 3.4 Hz, 2H), 8.39 (dd, *J* = 6.0 and 3.4 Hz, 2H), 11.95 (s,2H); MS (El, 70 eV) *m*/*z* (%) 426 (100) [M$^+$], 213 (7).

*3.3.3. 1,4-Bis(2-chloroanilino)anthraquinone (**3c**)*

Yield 60 %; M.p. 282–284 'C; ^{1}H NMR (CDC1$_3$) δ = 7.10 (t, *J* = 7.1 Hz, 2H), 7.26-7.52(m, 8H), 7.78 (dd, *J* = 5.5 and 3.4 Hz, 2H), 8.40 (dd, *J* = 5.5 and 3.4 Hz, 2H), 12.09 (s, 2H); MS (El, 70 eV) *m*/*z* (%) 462 (14) [M$^+$+4], 460 (55) [M$^+$+2], 458 (100) [M$^+$], 424 (18), 423 (23), 387 (15).

*3.3.4. 1,4-Bis(2-bromoanilino)anthraquinone (**3d**)*

Yield 40 %; M.p. 288–290°C; ^{1}H NMR (CDC1$_3$) δ = 7.03 (t, *J* = 7.3 Hz, 2H), 7.32-7.42 (m, 6H), 7.69 (d, *J* = 6.7 Hz, 2H), 7.77 (dd, *J* = 5.8 and 3.4 Hz, 2H), 8.40 (dd, *J* = 5.8 and 3.4 Hz, 2H), 12.05 (s, 2H); MS (El, 70 eV) *m*/*z* (%) 550 (71) [M$^+$+4], 548 (100) [M$^+$+2], 546 (57) [M$^+$], 388 (35), 387 (29), 194 (28).

*3.3.5. 1,4-Bis(2-methylanilino)anthraquinone (**3e**)*

Yield 59 %; M.p. 225°C; ^{1}H NMR (CDC1$_3$) δ= 2.37 (s, 6H), 7.11–7.19 (m, 4H), 7.22 (s, 2H), 7.26-7.32 (m, 4H), 7.77 (dd, *J* = 5.8 and 3.4 Hz, 2H), 8.41 (dd, *J* = 5.8 and 3.4Hz, 2H), 12.16 (s, 2H); MS (El, 70 eV) *m*/*z* (%) 418 (100) [M$^+$], 329 (11), 312 (12).

*3.3.6. 1,4-Bis(2-ethylanilino)anthraquinone (**3f**)*

Yield 89 %; M.p. 146°C; ^{1}H NMR (CDC1$_3$) δ = 1.26 (t, *J* = 7.6 Hz, 6H), 2.74 (q, *J*=7. 6Hz, 4H), 7.17-7.25 (m, 6H), 7.20 (s, 2H), 7.32-7.34 (m, 2H), 7.76 (dd, *J* = 5.9 and 3.4 Hz, 2H), 8.42 (dd, *J* = 5.9 and 3.4 Hz, 2H), 12.22 (s, 2H); MS (EI, 70 eV) *m*/*z* (%) 446 (100) [M$^+$], 310 (24), 77 (19).

*3.3.7. 1,4-Bis(2-propylanilino)anthraquinone (**3g**)*

Yield 28 %; M.p. 143°C ^{1}H NMR (CDC1$_3$) δ = 0.96 (t, *J* = 7.6 Hz, 6H), 1.68 (sextet, *J* = 7.6 Hz, 4H), 2.68 (t, *J* = 7.6 Hz, 4H), 7.17–7.31 (m, 10H), 7.76 (dd, *J* = 5.8 and 3.4 Hz, 2H), 8.42 (dd, *J* 5.8 and 3.4 Hz, 2H), 12.19 (s, 2H); MS (El, 70 e *m*/*z* (%) 474 (100) [M$^+$], 357 (14), 310 (24).

3.3.8. 1,4-Bis(2-isopropylanilino)anthraquinone (3

Yield 84 %; M.p. 129°C ^{1}H NMR (CDC1$_3$) δ 1.26 (d, *J* = 7.3 Hz, 12H), 3.32 (septet, *J* = 6 Hz, 2H), 7.14 (s, 2H), 7.22-7.24 (m, 6H), 7.37-7. (m, 2H), 7.75 (dd, *J* = 5.8 and 3.4 Hz, 2H), 8. (dd, *J* = 5.8 and 3.4 Hz, 2H), 12.23 (s, 2H); M (El, 70 ev) *m*/*z* (%) 474 (100) [M$^+$], 460 (14), 4 (39), 310 (14).

*3.3.9. 1,4-Bis(3-isopropylanilino)anthraquinone (**3i**)*

Yield 47 %; M.p. 144°C; ^{1}H NMR (CDC1$_3$) = 1.27 (d, *J* = 7.3 Hz, 2H), 2.92 (septet, *J* = 6 Hz, 2H), 7.05 (d, *J* = 7.3 Hz, 2H), 7.11 (d, *J* = 7 Hz, 2H), 7.13 (s, 2H), 7.31 (t, *J* = 7.3 Hz, 2H 7.53 (s, 2H), 7.76 (dd, *J* = 5.8 and 3.4 Hz, 2H 8.39 (dd, *J* = 5.8 and 3.4 Hz, 2H), 12.31 (s, 2H MS (El, 70 eV) *m*/*z* (%) 474 (100) [M$^+$].

*3.3.10. 1,4-Bis(4-isopropylanilino)anthraquinone (**3j**)*

Yield 68 %; M.p. 201°C; ^{1}H NMR (CDC1$_3$) δ 1.27 (d, *J* = 7.7 Hz, 12H), 2.93 (septet, *J* = 6.7 H 2H), 7.19 (d, *J* = 7.7 Hz, 4H), 7.25 (d, *J* = 7.7 H 4H), 7.47 (s, 2H), 7.75 (dd, *J* = 5.8 and 3.4 H 2H), 8.39 (dd, *J* = 5.8 and 3.4 Hz, 2H), 12.27 2H); MS (El, 70 eV) *m*/*z* (%) 474 (100) [M$^+$], 4 (44), 222 (24).

*3.3.11. 1,4-Bis(2-tert-butylanilino)anthraquinone (**3k**)*

Yield 77 %; M.p. 229°C; ^{1}H NMR (CDC1$_3$) = 1.47 (s, 18H), 7.01 (s, 2H), 7.19-7.22 (m, 6H 7.49-7.52 (m, 2H), 7.75 (dd, *J* = 5.9 and 3.3 H 2H), 8.43 (dd, *J* = 5.9 and 3.3 Hz, 2H), 12.33 2H); MS (EI, 70 eV) *m*/*z* 502 (100) [M$^+$], 371 (6 341 (40), 324 (22), 133 (23).

3.3.12. 1,4-Bis(2-phenylanilino)anthraquinone (3

Yield 78 %; M.p. 238–239°C; ^{1}H NM (CDC1$_3$) δ=7.18 (s, 2H), 7.24–7.43 (m, 18H 7.65(dd, *J* = 6.0 and 3.2 Hz, 2H), 8.24 (dd, *J* 6.0 and 3.2 Hz, 2H), 11.88 (s, 2H); MS (El, 70 e *m*/*z* (%) 542 (100) [M$^+$], 167 (8).

*3.3.13. 1,4-Bis(2-methoxyanilino)anthraquinone (**3m**)*

Yield 86 %; M.p. 188-190°C; ^{1}H NMR (CDC1_3) δ = 3.92 (s, 6H), 6.93-7.10 (m, 4H), 7.13(t, J = 7.3 Hz, 2H), 7.35 (d, J = 7.9 Hz, 2H), 7.50 (s, 2H), 7.74 (dd, J = 6.0 and 3.4Hz, 2H), 8.40 (dd, J = 6.0 and 3.4 Hz, 2H), 12.10 (s, 2H); MS (El, 70 ev) m/z 450 (100) [M$^+$], 313 (15).

*3.3.14. 1,4-Bis[2-(trifluoromethyl)anilinolanthraquinone (**3n**)*

Yield 18 %; M.p. 248-249°C; ^{1}H NMR (CDC1_3) δ = 7.26 (t, J = 7.3 Hz, 2H), 7.36 (s, 2H), 7.46 (d, J = 7.9 Hz, 2H), 7.53 (t, J=7.3 Hz, 2H), 7.74 (d, J = 7.9 Hz, 2H), 7.77 (dd, J=5.8 and 3.4 Hz, 2H), 8.39 (dd, J=5.8 and 3.4 Hz, 2H), 12.11 (s, 2H); MS (El, 70 eV) m/z (%) 526 (100) [M$^+$], 485 (16), 430 (11), 353 (10), 352 (12), 151 (11).

*3.3.15. 1,4-Bis(2,6-dimethylanilino)anthraquinone (**3o**)*

Yield 47 %; M.p. 298°C; ^{1}H NMR (CDC1_3) δ = 2.20 (s, 12H), 6.56 (s, 2H), 7.21 (s, 6H), 7.77 (dd, J = 5.9 and3.4 Hz, 2H), 8.43 (dd, J = 5.9 and 3.4 Hz, 2H), 11.84 (s, 2H); MS (El, 70 eV) m/z 446 (100) [M$^+$], 77 (35).

*3.3.16. 1,4-Bis(2,6-diethylanilino)anthraquinone (**3p**)*

Yield 25 %; M.p. 209°C; ^{1}H NMR (CDC1_3) δ = 1.13 (t, J = 7.6 Hz, 12H), 2.47 (q, J = 7.6Hz, 2H), 2.51 (q, J = 7.6 Hz, 2H), 2.57 (q, J = 7.6 Hz, 2H), 2.61 (q, J = 7.6 Hz, 2H), 6.54 (s, 2H), 7.15–7.26 (m, 6H), 7.77 (dd, J = 5.9 and 3.4 Hz, 2H), 8.44 (dd, J = 5.9 and 3.4 Hz, 2H), 11.91 (s, 2H); MS (El, 70 eV) m/z (%) 502 (100) [M$^+$], 487 (28), 366 (17), 338 (22), 91 (30), 77 (30).

*3.3.17. 1,4-Bis(2,6-diisopropylanilino)anthraquinone (**3q**)*

Yield 35 %; M.p. 255°C; ^{1}H NMR (CDC1_3) δ = 1.05 (d, J = 6.8 Hz, 12H), 1.17 (d, J = 6.8Hz, 12H), 3.09 (septet, J = 6.9 Hz, 4H), 6.55 (s, 2H), 7.20–7.22 (m, 6H), 7.78 (dd, J = 5.9 and 3.4 Hz, 2H), 8.45 (dd, J = 5.9 and 3.4 Hz, 2H), 11.89 (s, 2H); MS (El, 70 eV) m/z (%) 558 (100) [M$^+$], 5
(33).

*3.3.18. 1,4-Bis(2,5-di-*tert*-butylanilino)anthraquino*
*(**3r**)*

Yield 67 %; M.p. 169°C ^{1}H NMR (CDC1_3) δ
1.25 (s, 18H), 1.47 (s, 18H), 7.03 (s, 2H), 7.18
2H), 7.20 (d, J = 7.9 Hz, 2H), 7.42 (d, J = 7.9 H
2H), 7.75 (dd, J = 5.8 and 3.4 Hz, 2H), 8.44 (dd,
= 5.8 and 3.4 Hz, 2H), 12.38 (s, 2H); MS (El,
eV) m/z (%) 614 (100) [M$^+$], 380 (20), 57 (78).

*3.3.19. 1,4-Bis(2,4,6-triisopropylanilino)anthraquinone (**3s**)*

Yield 11 %; M.p. 214°C; ^{1}H NMR (CDC1_3)
= 1.17 (d, J = 6.8 Hz, 12H), 1.33 (d, J = 6.8 H
2411), 2.98 (septet, J = 6.8 Hz, 211), 3.11 (septe
J = 6.8 Hz, 4H), 6.70 (s, 2H), 7.11 (s, 4H), 7.
(dd, J = 6.8 and 3.7 Hz, 2H), 8.60 (dd, J 6.8 a
3.7 Hz, 2H), 12.02 (s, 2H); MS (El, 70 eV) m/z (
643 (100) [M$^+$], 628 (21), 426 (25).

*3.3.20. 1,4-Bis(1-naphthylamino)anthraquinone (**3***

Yield 14 %; M.p. 285 °C; NMR (CDC1_3) δ
7.21 (s, 2H), 7.45–7.67 (m, 4H), 7.52–7.75(m, 4H
7.74 (t, J = 5.4 Hz, 2H), 7.80–7.82 (m, 2H), 7.8
7.91 (m, 2H), 8.18 (dd, J = 5.9 and 3.4 Hz, 2H
8.48 (dd, J = 5.9 and 3.4 Hz, 2H), 12.65 (s, 2); M
(El, 70 eV) m/z (%) 490 (100) [M$^+$], 127 (19), 1
(30), 77 (15).

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