



SUPPLEMENTARY MATERIAL TO

**Synthesis and dyeing performance of some amphiphilic naphthalimide azo disperse dyes on polyester fabrics**

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ANALYTICAL AND SPECTRAL DATA OF THE SYNTHESIZED COMPOUNDS

**5-Nitroacenaphthene (2).** Yield: 85 %; yellow needles; m.p.: 101–102 °C; Anal. calcd. for C<sub>12</sub>H<sub>9</sub>NO<sub>2</sub>: C, 72.35; H, 4.55; N, 7.03 %. Found: C, 72.18; H, 4.57; N, 7.04 %; FT-IR (KBr, cm<sup>-1</sup>): 3085, 2946, 1620 1596, 1508; <sup>1</sup>H-NMR (300.1 MHz, CDCl<sub>3</sub>, δ / ppm): 3.43–3.50 (4H, *m*, CH<sub>2</sub>), 7.31(1H, *d*, *J* = 7.8 Hz, H1), 7.45 (1H, *d*, *J* = 7.0 Hz, H9), 7.67–7.75 (1H, *m*, H2), 8.47 (1H, *d*, *J* = 7.7 Hz, H8), 8.54 (1H, *d*, *J* = 8.6 Hz, H3); <sup>13</sup>C-NMR (75.5 MHz CDCl<sub>3</sub>, δ / ppm) 30.5, 30.6, 117.9, 120.1, 121.2, 122.7, 124.4, 127.8, 131.7, 140.1, 146.6, 155.7.

**4-Nitro-1,8-naphthalic anhydride (3).** Yield: 74 %; colourless needles; m.p.: 231–232 °C; Anal. calcd for C<sub>12</sub>H<sub>5</sub>NO<sub>5</sub>: C, 59.27; H, 2.07; N, 5.76 %. Found: C, 59.17; H, 2.02; N, 5.75 %; FT-IR (KBr, cm<sup>-1</sup>): 3078, 2914 2849, 1789, 1756, 1624, 1526; <sup>1</sup>H-NMR (300.1 MHz, DMSO-*d*<sub>6</sub>, δ / ppm): 8.09 (1H, *t*, *J* = 7.8 Hz, H6), 8.54 (1H, *d*, *J* = 8.0 Hz, H7), 8.61–8.66 (2H, *m*, H2 & H5), 8.73 (1H, *d*, *J* = 8.7 Hz, H3); <sup>13</sup>C-NMR (75.5 MHz, DMSO-*d*<sub>6</sub>, δ / ppm): 120.0, 122.8, 124.0, 124.3, 129.8, 130.3, 130.5, 131.0, 133.2, 149.5, 159.4, 160.0.

**4-Nitro-N-dodecyl-1,8-naphthalimide (4).** Yield: 74 %; white crystals; m.p.: 94–96 °C; Anal. calcd for C<sub>24</sub>H<sub>30</sub>N<sub>2</sub>O<sub>4</sub>: C, 70.22; H, 7.37; N, 6.82 %. Found C, 70.21; H, 8.15; N, 6.91 %; FT-IR (KBr, cm<sup>-1</sup>): 3081, 2918, 2849, 1699, 1655, 1622, 1593; <sup>1</sup>H-NMR (300.1 MHz, CDCl<sub>3</sub>, δ / ppm): 0.85 (3H, *t*, *J* = 6.9 Hz, CH<sub>3</sub>), 1.25–1.76 (20H, *m*, aliphatic H), 4.18 (2H, *t*, *J* = 7.7 Hz, *N*-CH<sub>2</sub>), 7.98 (1H, *dd*, *J* = 8.7 & 7.3 Hz, H6), 8.39 (1H, *d*, *J* = 8.0 Hz, H7), 8.68–8.82 (2H, *m*, 2H & H5), 8.85 (1H, *d*, *J* = 8.9 Hz, H3); <sup>13</sup>C-NMR (75.5 MHz, CDCl<sub>3</sub>, δ / ppm): 14.1, 22.7, 27.1, 28.0, 29.3, 29.4, 29.5, 29.5, 29.6 (2C), 31.9, 40.9, 123.0, 123.7, 123.9, 127.0, 129.1, 129.2, 129.7, 129.9, 132.4, 149.5, 162.4, 163.3.

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*4-Amino-N-dodecyl-1,8-naphthalimide (5)*. Yield: 84 %; orange crystals; m.p.: 139–141 °C; Anal. calcd for C<sub>24</sub>H<sub>32</sub>N<sub>2</sub>O<sub>2</sub>: C, 75.75; H, 8.48; N, 7.36 %. Found: C, 74.76; H, 8.11; N, 7.03 %; FT-IR (KBr, cm<sup>-1</sup>) 3351, 3242, 2994, 2984, 2918, 2819, 1681, 1653, 1628; <sup>1</sup>H-NMR (300.1 MHz, CDCl<sub>3</sub>, δ / ppm): 0.85 (3H, *t*, *J* = 6.9 Hz, CH<sub>3</sub>), 1.24–1.74 (20H, *m*, aliphatic H), 4.15 (2H, *t*, *J* = 7.6 Hz, *N*-CH<sub>2</sub>), 4.96 (2H, *s*, NH<sub>2</sub>), 6.87 (1H, *d*, *J* = 8.1 Hz, H3), 7.64 (1H, *dd*, *J* = 8.3 & 7.4 Hz, H6), 8.09 (1H, *d*, *J* = 8.4 Hz, H2), 8.40 (1H, *d*, *J* = 8.1 Hz, H5), 8.58 (1H, *dd*, *J* = 7.3 & 0.89 Hz, H7); <sup>13</sup>C-NMR (75.5 MHz, CDCl<sub>3</sub>, δ / ppm): 14.1, 22.6, 27.1, 28.1, 29.3, 29.4 (2C), 29.5, 29.6 (2C), 31.8, 40.2, 109.5, 112.3, 120.0, 123.2, 124.9, 126.6, 129.7, 131.4, 133.6, 148.9, 163.9, 164.5; MS(EI) (*m/z* (%)): 380 (M<sup>+</sup>), 380 (90), 212 (100), 169 (37), 141 (20), 43 (12).

*(Z)-2-Dodecyl-6-((2-hydroxynaphthalen-1-yl)diazanyl)-benzo[de]isoquinoline-1,3(2H)-dione (7a)*. Yield: 80.9 %; m.p.: 146–148 °C; FT-IR (KBr, cm<sup>-1</sup>) 3387, 3062, 2953, 2922, 2850, 1699, 1655, 1623, 1587, 1382; <sup>1</sup>H-NMR (300.1 MHz, CDCl<sub>3</sub>, δ / ppm): 0.85 (3H, *t*, *J* = 7.0 Hz, CH<sub>3</sub>), 1.25–1.70 (20H, *m*, aliphatic H), 4.03 (2H, *t*, *J* = 7.7 Hz, *N*-CH<sub>2</sub>), 6.55 (1H, *d*, *J* = 9.6 Hz), 7.26–7.44 (3H, *m*), 7.53 (1H, *d*, *J* = 9.6 Hz), 7.68 (1H, *m*), 8.04 (1H, *d*, *J* = 8.3 Hz), 8.15 (1H, *d*, *J* = 7.8 Hz), 8.25 (1H, *d*, *J* = 8.4 Hz), 8.43–8.48 (2H, *m*), 16.91 (1H, *brs*, OH); <sup>13</sup>C-NMR (75.5 MHz, CDCl<sub>3</sub>, δ / ppm) 14.1, 22.7, 27.2, 28.1, 29.3, 29.4 (2C), 29.5 (2C), 29.6, 31.9, 40.5, 111.4, 118.6, 121.2, 122.3, 123.0, 126.1, 127.1, 127.6, 128.4, 128.8 (2C), 129.1, 129.7, 131.3, 132.1, 132.5 (2C), 142.7, 143.4, 163.3, 163.7, 180.0; MS(EI) (*m/z* (%)): 535 (M<sup>+</sup>), 535 (69), 380 (18), 212 (36), 143 (100), 43 (55).

*(Z)-6-((2,3-Dihydroxynaphthalen-1-yl)diazanyl)-2-dodecyl-1H-benzo[de]isoquinoline-1,3(2H)-dione (7b)*. Yield: 82.3 %; m.p.: 174–177 °C; FT-IR (KBr, cm<sup>-1</sup>): 3375, 3063, 2921, 2851, 1699, 1658, 1618, 1588, 1382; <sup>1</sup>H-NMR (300 MHz, CDCl<sub>3</sub>, δ / ppm): 0.88 (3H, *t*, *J* = 6.9 Hz, CH<sub>3</sub>), 1.26–1.74 (20H, *m*, aliphatic H), 4.03 (2H, *t*, *J* = 6.9 Hz, *N*-CH<sub>2</sub>), 6.52 (1H, *d*, *J* = 9.6 Hz), 7.26–7.41 (3H, *m*), 7.49 (1H, *d*, *J* = 9.6 Hz), 7.65 (1H, *m*), 7.99 (1H, *d*, *J* = 8.1 Hz), 8.20 (1H, *d*, *J* = 8.4 Hz), 8.40 (1H, *d*, *J* = 6.0 Hz), 8.41–8.44 (2H, *m*), 16.67 (1H, *s*, OH); MS(EI) (*m/z* (%)): 551 (M<sup>+</sup>), 551 (6), 380 (74), 212(100), 169 (13), 43 (11).

*(Z)-6-((4-(Dimethylamino)phenyl)diazanyl)-2-dodecyl-1H-benzo[de]isoquinoline-1,3(2H)-dione (7c)*. Yield: 96.4 %; m.p.: 114–116 °C; FT-IR (KBr, cm<sup>-1</sup>): 3073, 2921, 2850, 1693, 1657, 1604, 1588, 1349; <sup>1</sup>H-NMR (300 MHz, CDCl<sub>3</sub>, δ / ppm): 0.87 (3H, *t*, *J* = 6.8 Hz, CH<sub>3</sub>), 1.24–1.74 (20H, *m*, aliphatic H), 3.16 (6H, *t*, *J* = 4.8 Hz, 2CH<sub>3</sub>), 4.16 (2H, *t*, *J* = 7.6 Hz, *N*-CH<sub>2</sub>), 6.74 (2H, *d*, *J* = 9.0 Hz), 7.72 (1H, *m*), 7.88 (1H, *d*, *J* = 8.0 Hz), 7.95 (2H, *d*, *J* = 9.0 Hz), 8.59 (2H, *m*), 9.15 (1H, *dd*, *J* = 8.4 & 1.0 Hz); <sup>13</sup>C-NMR (75 MHz, CDCl<sub>3</sub>, δ / ppm): 14.1, 22.6, 27.2, 28.1, 29.3, 29.4 (2C), 29.6 (2C), 31.9, 40.3, 40.5, 40.9, 111.4, 112.1, 121.9, 122.4, 126.6, 126.8, 129.1, 130.5, 131.2, 131.7, 133.7, 144.5, 151.7,

153.3, 163.9, 164.3; MS(EI) ( $m/z$  (%)): 512 ( $M^+$ ), 512 (100), 380 (27), 212 (50), 120 (90), 43 (13).

(*Z*)-6-((4-(Diethylamino)phenyl)diazenyl)-2-dodecyl-1*H*-benzo[*de*]isoquinoline-1,3(2*H*)-dione (**7d**). Yield: 91.4 %; m.p.: 137–140 °C; FT-IR (KBr,  $\text{cm}^{-1}$ ): 3072, 2919, 2851, 1692, 1656, 1603, 1599, 1388;  $^1\text{H-NMR}$  (300.1 MHz,  $\text{CDCl}_3$ ,  $\delta$  / ppm): 0.87 (3H, *t*,  $J = 6.9$  Hz,  $\text{CH}_3$ ), 1.19 (6H, *t*,  $J = 6.8$  Hz,  $2\text{CH}_3$ ), 1.25–1.79 (20H, *m*, aliphatic H), 3.50 (4H, *q*,  $J = 7.1$  Hz,  $2\text{CH}_2$ ), 4.18 (2H, *t*,  $J = 7.7$  Hz, *N*- $\text{CH}_2$ ), 6.76 (2H, *d*,  $J = 9.0$  Hz), 7.81 (1H, *dd*,  $J = 8.3$  & 7.7 Hz), 7.93 (1H, *d*,  $J = 8.0$  Hz), 8.0 (2H, *d*,  $J = 9.0$  Hz), 8.61–8.66 (2H, *m*), 9.20 (1H, *dd*,  $J = 8.4$  & 1.0 Hz);  $^{13}\text{C-NMR}$  (75.5 MHz,  $\text{CDCl}_3$ ,  $\delta$  / ppm): 12.7, 14.1, 22.7, 27.2, 28.2, 29.3, 29.4 (2C), 29.5, 29.6 (2C), 31.9, 40.5, 44.9, 111.1, 112.1, 121.8, 122.5, 126.6, 126.7, 129.1, 129.2, 130.6, 131.2, 131.9, 144.3, 151.3, 152.1, 164.1, 164.5; MS(EI) ( $m/z$  (%)): 540 ( $M^+$ ), 540 (93), 212 (25), 148 (100), 43 (17).

#### IR, NMR AND MASS SPECTRA FOR ALL COMPOUNDS

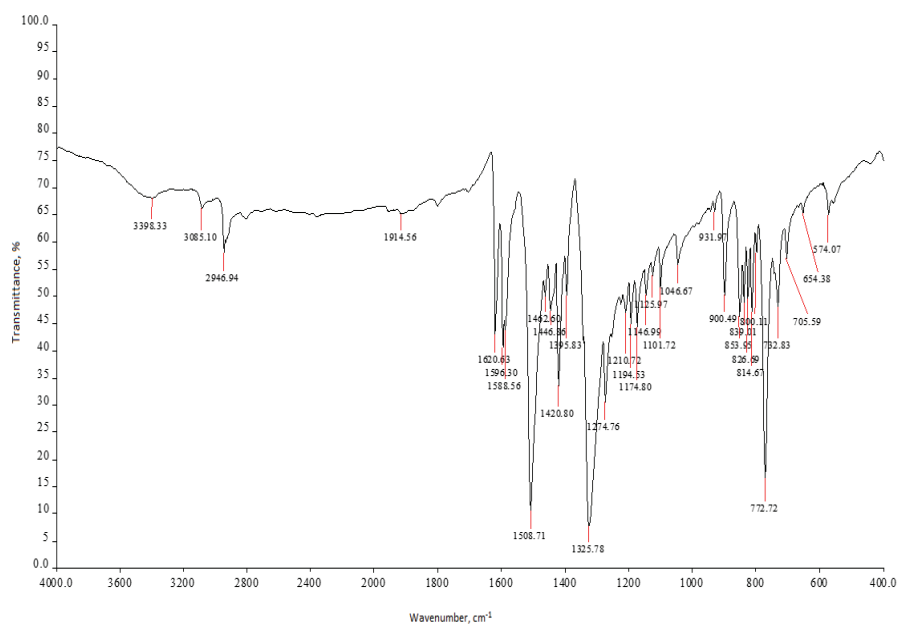
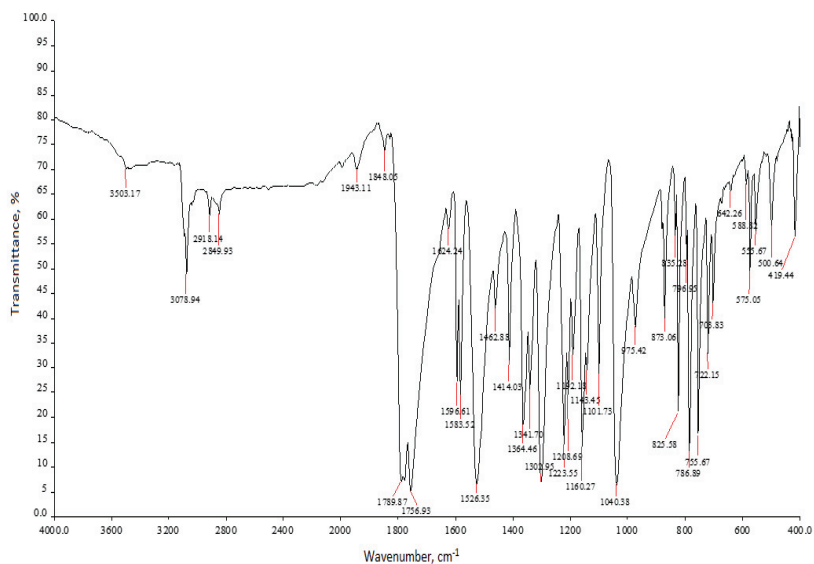
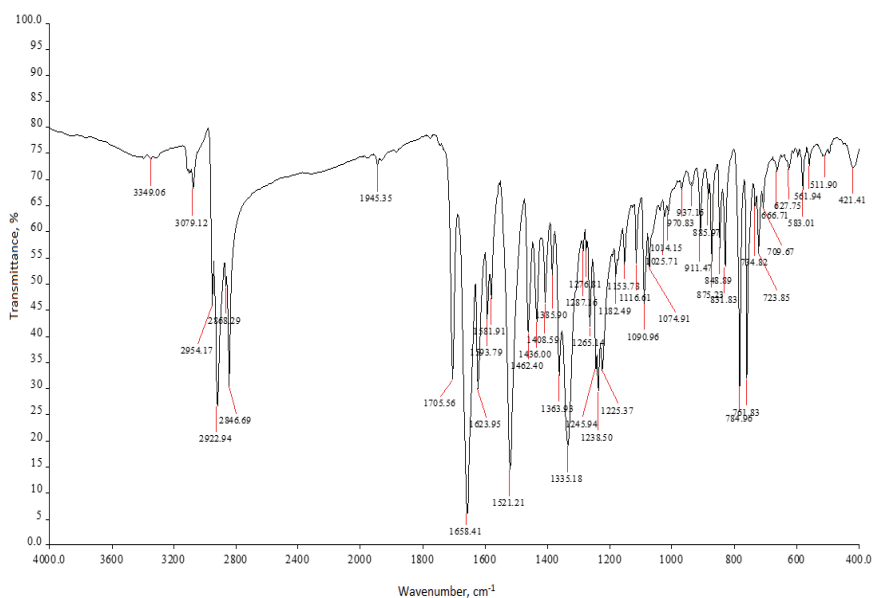


Fig. S-1. FT-IR for 5-nitroacenaphthene (**2**).

Fig. S-2. FT-IR for 4-nitro-1,8-naphthalic anhydride (**3**).Fig. S-3. FT-IR for 4-nitro-N-dodecyl-1,8-naphthalimide (**4**).

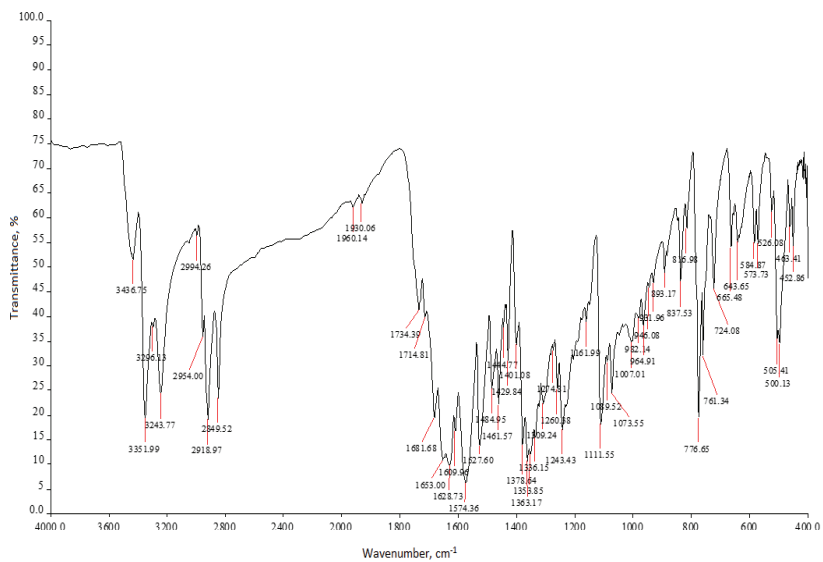


Fig. S-4. FT-IR for 4-amino-N-dodecyl-1,8-naphthalimide (5).

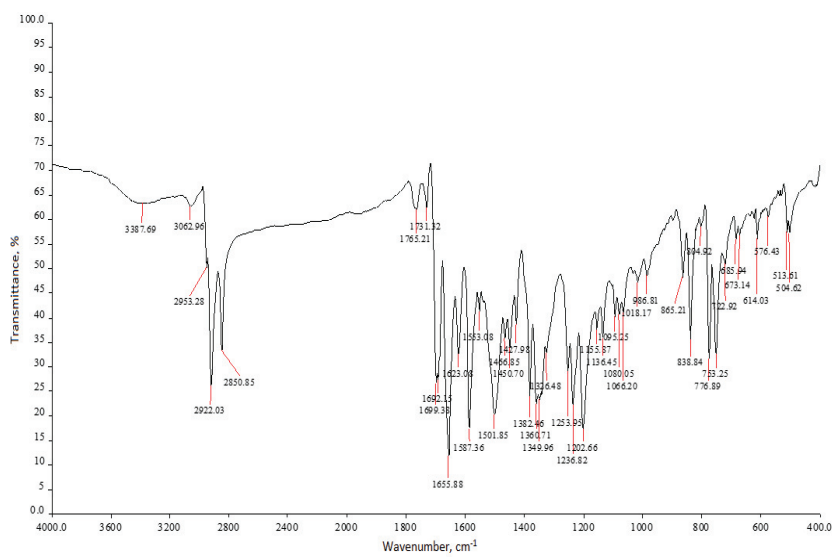


Fig. S-5. FT-IR for dye 7a.

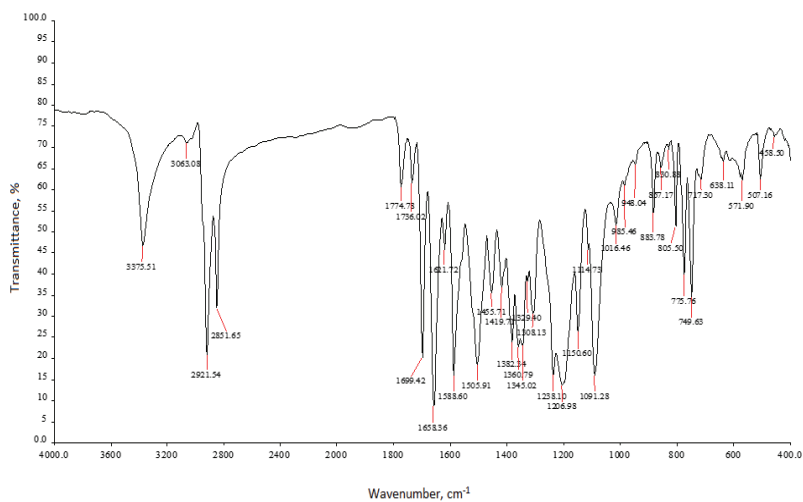


Fig. S-6. FT-IR for dye 7b.

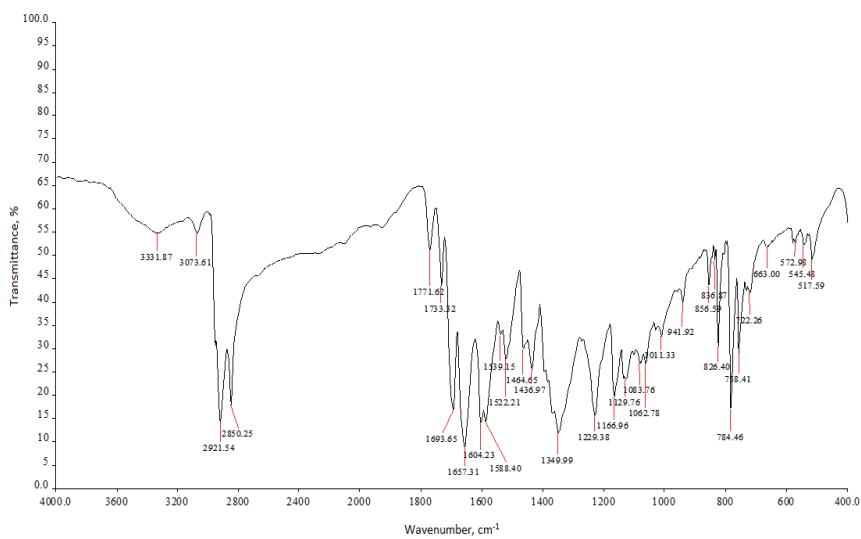


Fig. S-7. FT-IR for dye 7c.

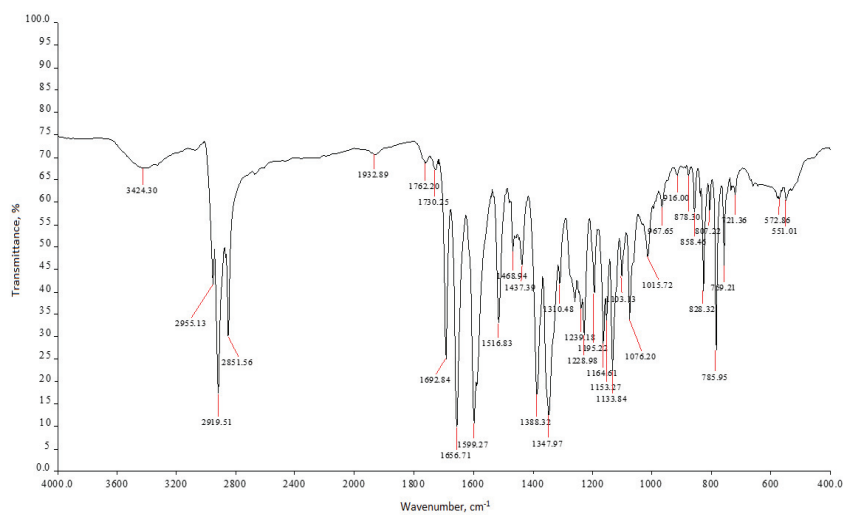
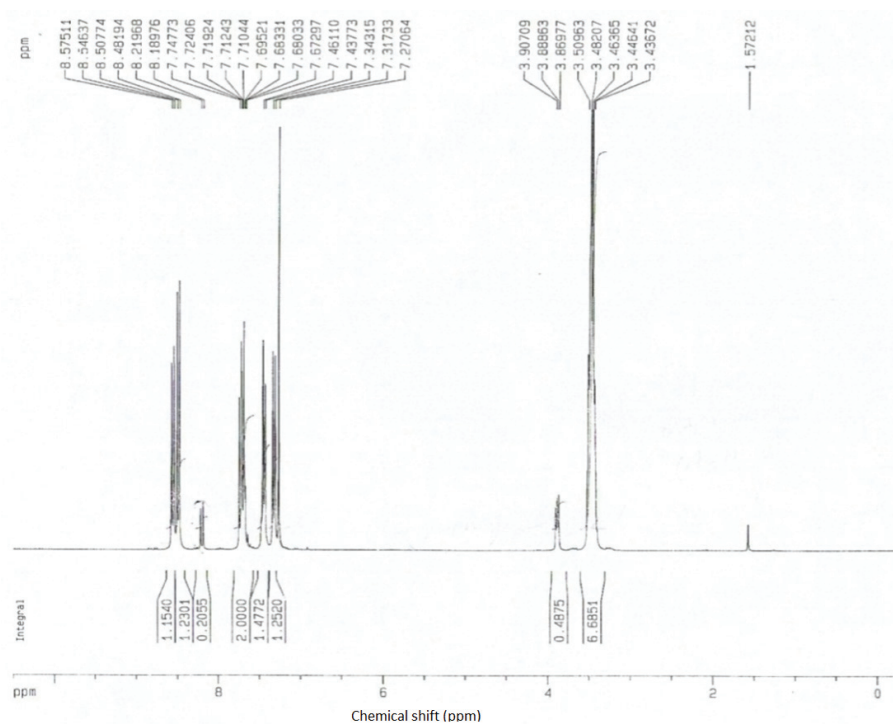
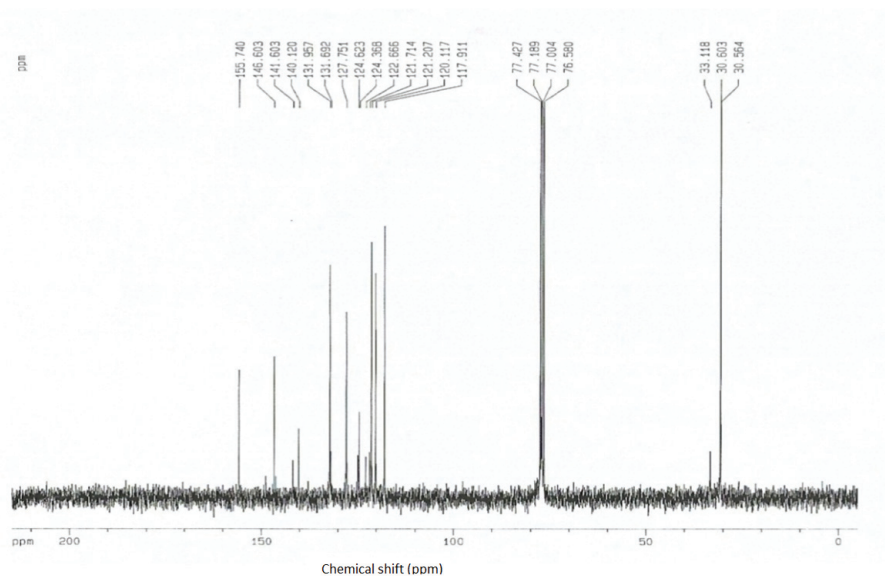
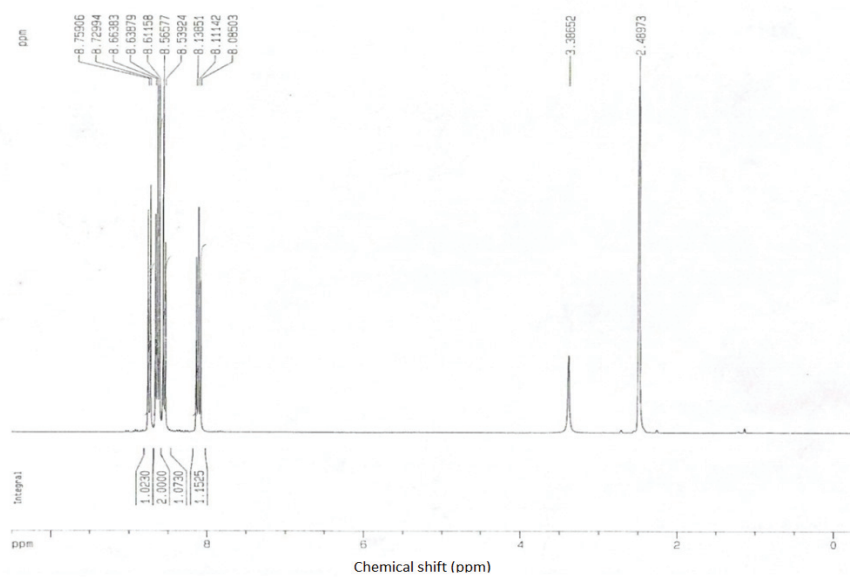


Fig. S-8. FT-IR for dye 7d.

Fig. S-9. <sup>1</sup>H-NMR for 5-nitroacenaphthene (2).

Fig. S-10.  $^{13}\text{C}$ -NMR for 5-nitroacenaphthene (**2**).Fig. S-11.  $^1\text{H}$ -NMR for 4-nitronaphthalic anhydride (**3**).



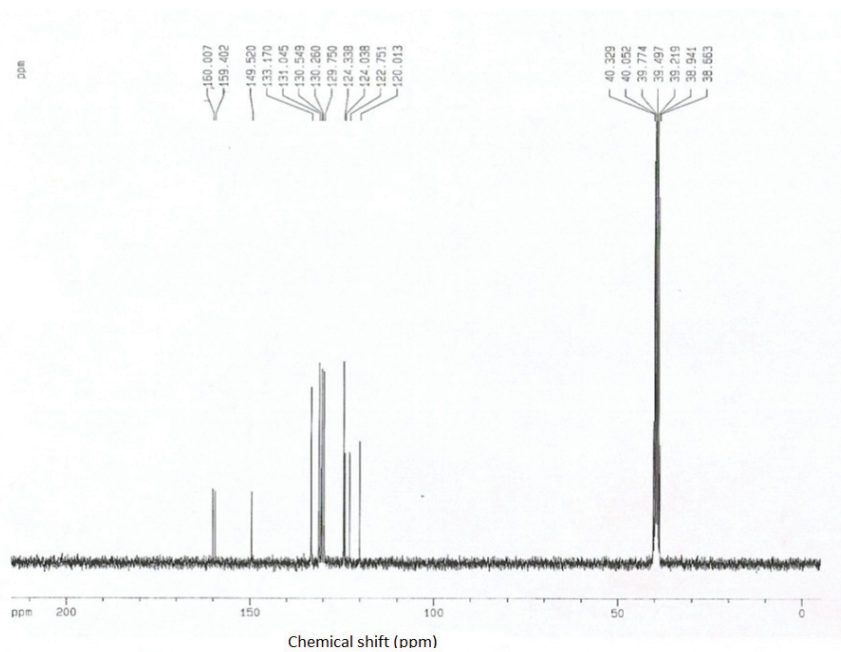


Fig. S-12.  $^{13}\text{C}$ -NMR for 4-nitronaphthalic anhydride (**3**).

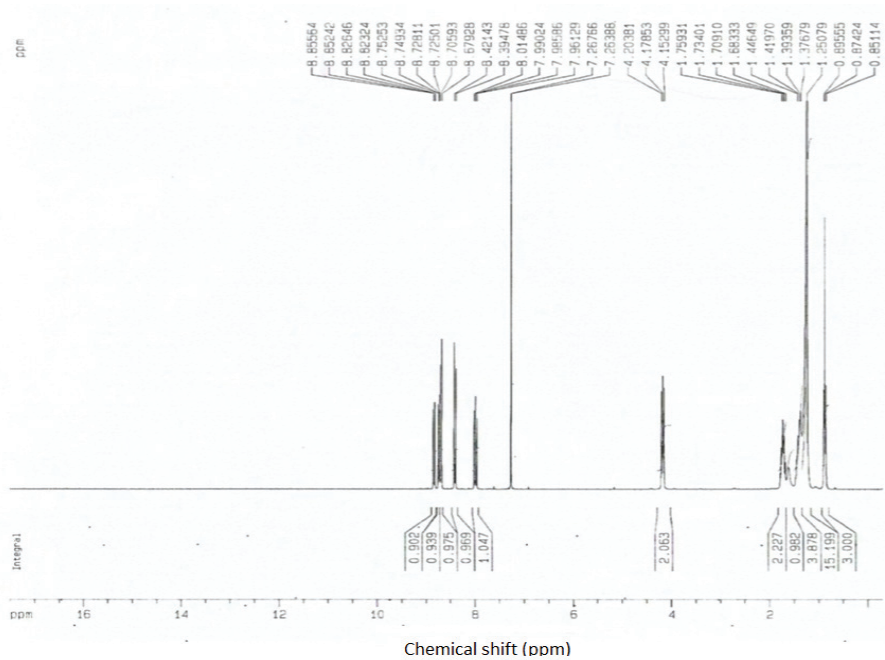
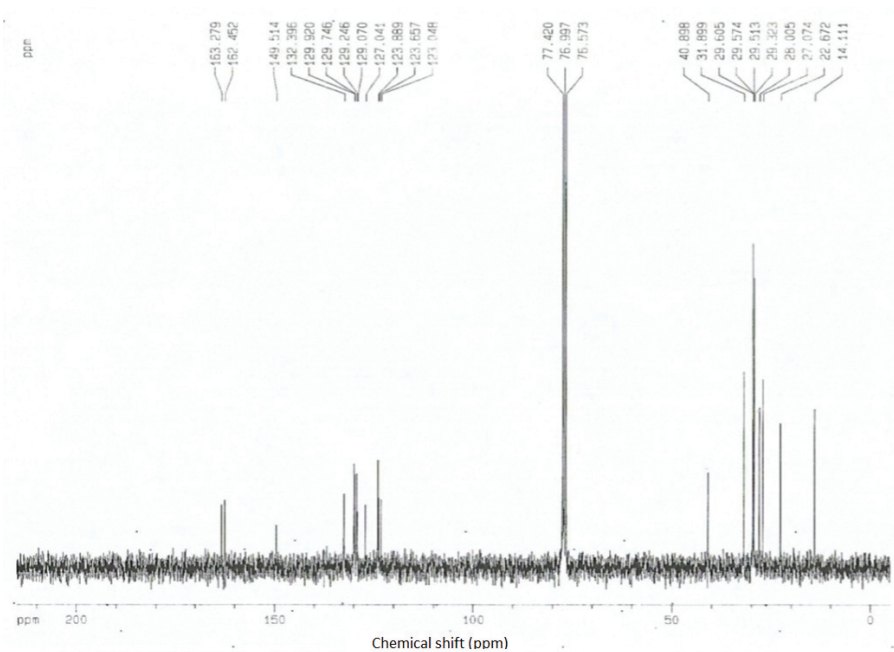
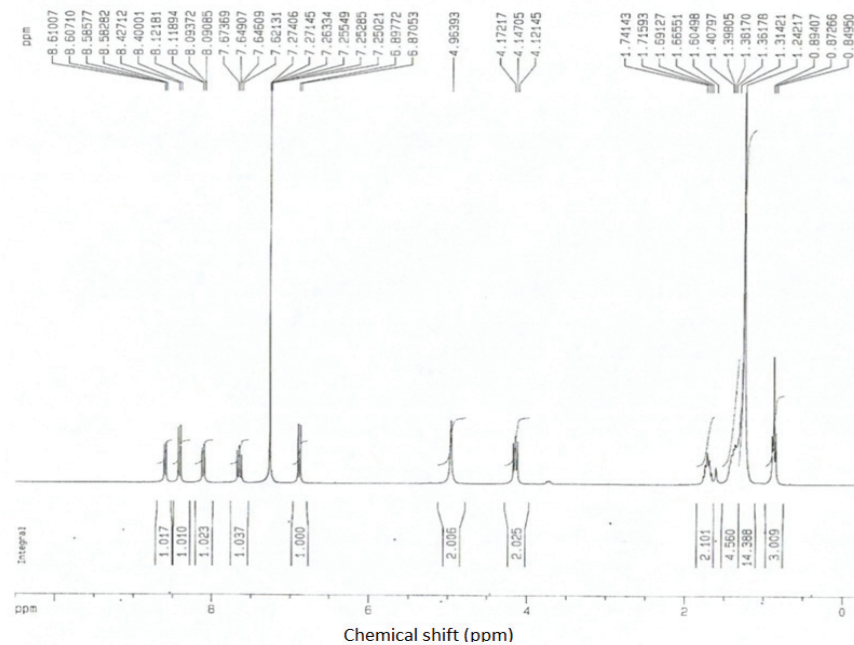


Fig. S-13.  $^1\text{H}$ -NMR for 4-nitro-*N*-dodecyl-1, 8-naphthalimide (**4**).

Fig. S-14.  $^{13}\text{C}$ -NMR for 4-nitro-*N*-dodecyl-1,8-naphthalimide (4).Fig. S-15.  $^1\text{H}$ -NMR for 4-amino-*N*-dodecyl-1,8-naphthalimide (5).

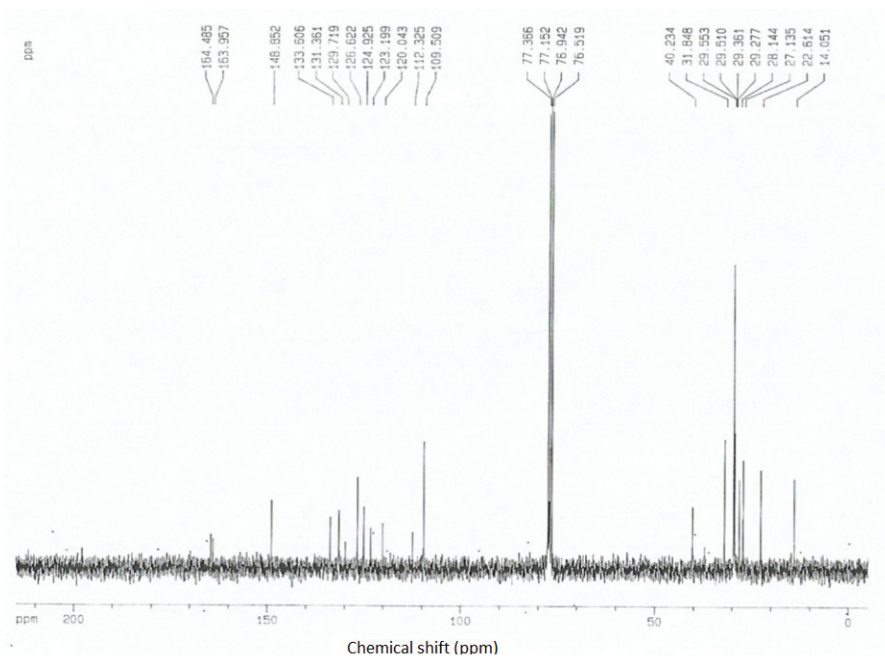


Fig. S-16.  $^{13}\text{C}$ -NMR for 4-amino-*N*-dodecyl-1,8-naphthalimide (**5**).

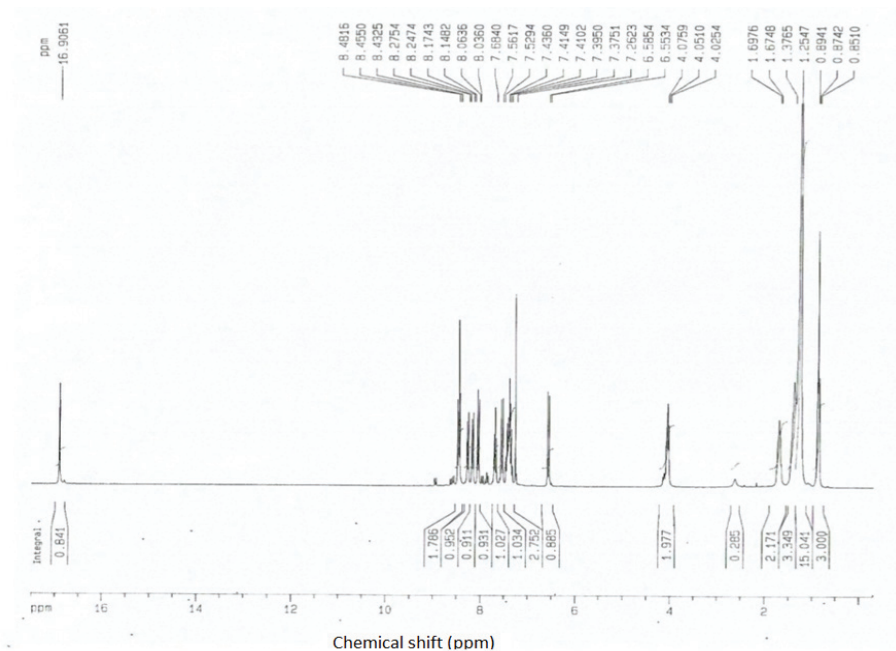
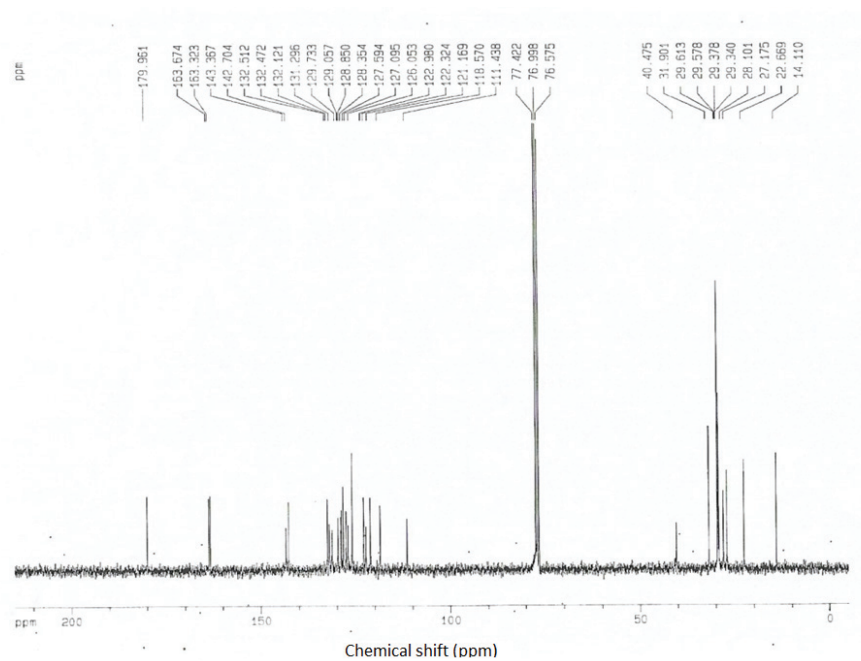
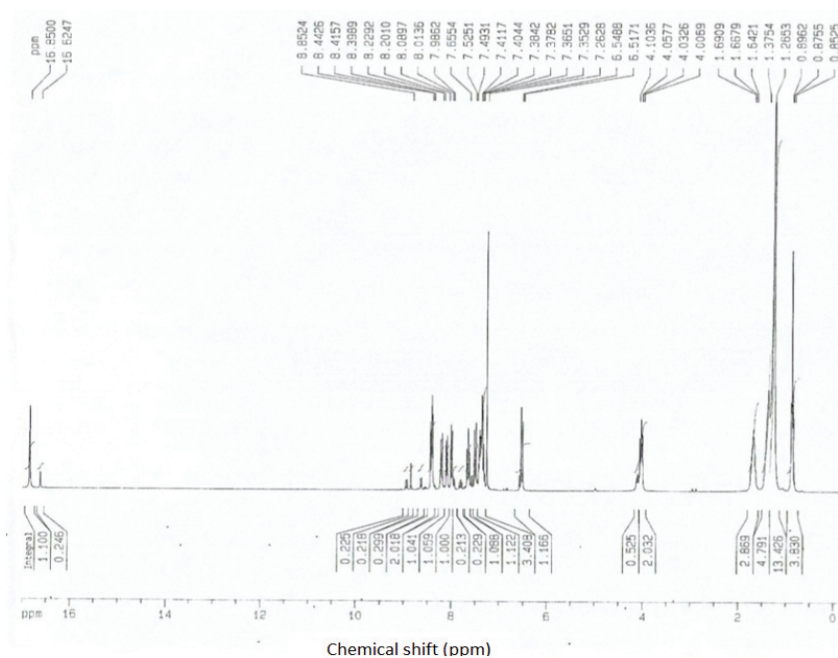


Fig. S-17.  $^1\text{H}$ -NMR for dye **7a**.

Fig. S-18.  $^{13}\text{C}$ -NMR for dye **7a**.Fig. S-19.  $^1\text{H}$ -NMR for dye **7b**.

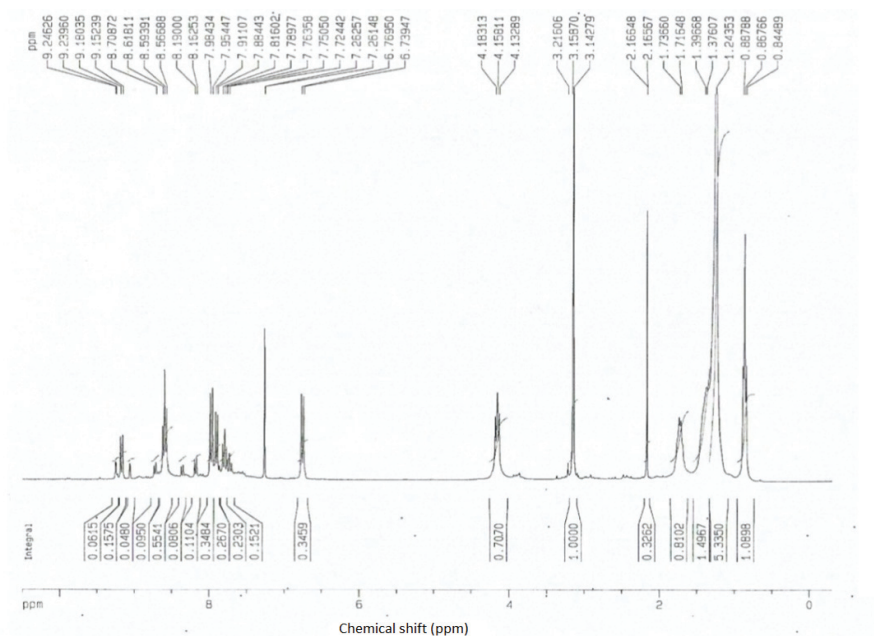


Fig. S-20. <sup>1</sup>H-NMR for dye 7c.

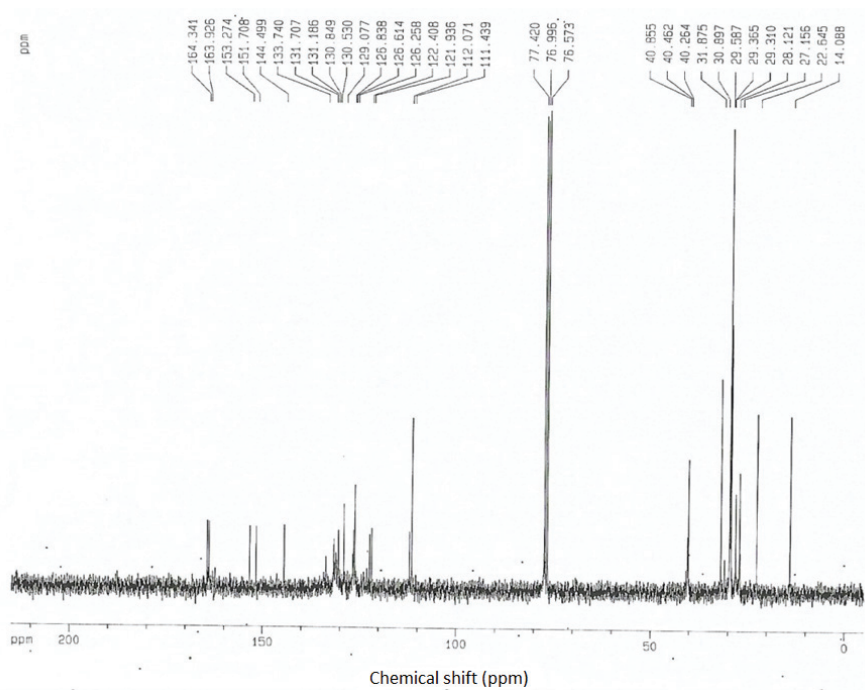
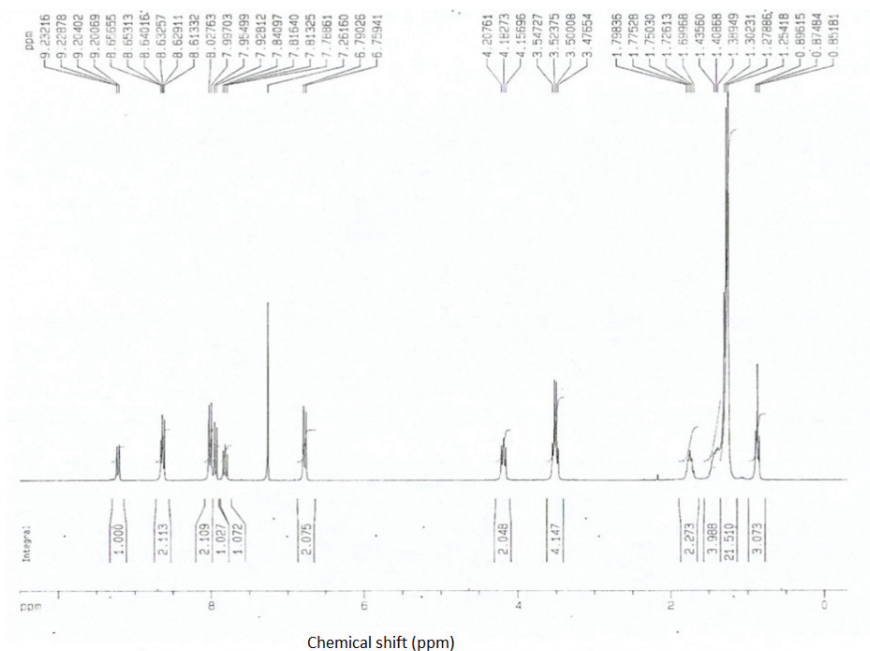
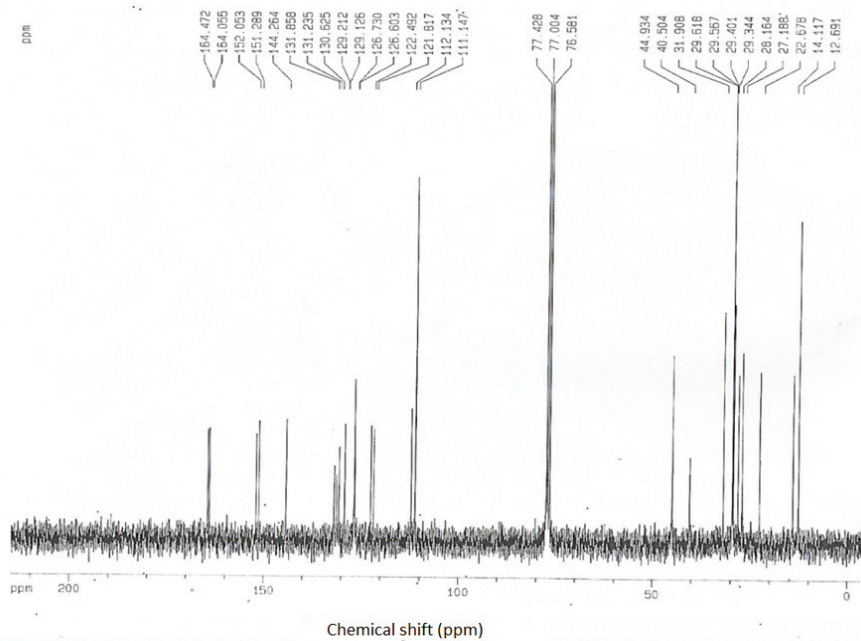


Fig. S-21. <sup>13</sup>C-NMR for dye 7c.

Fig. S-22.  $^1\text{H-NMR}$  for dye **7d**.Fig. S-23.  $^{13}\text{C-NMR}$  for dye **7d**.

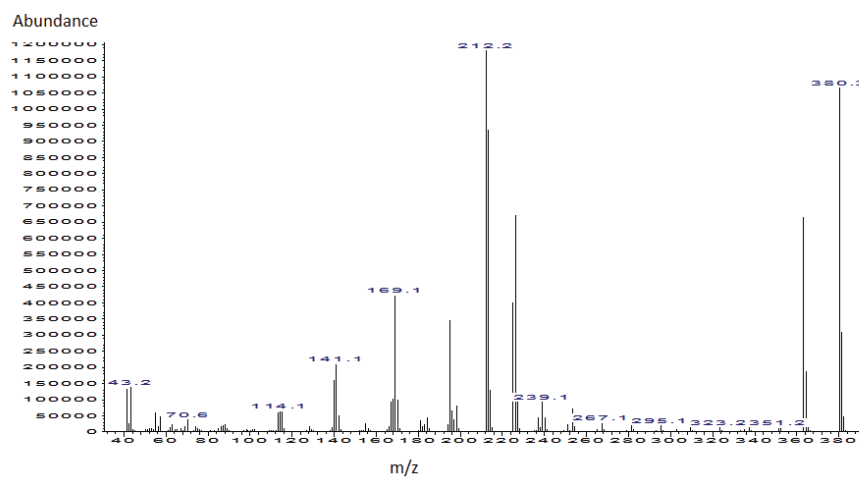


Fig. S-24. MS for 4-amino-N-dodecyl-1,8-naphthalimide (5).

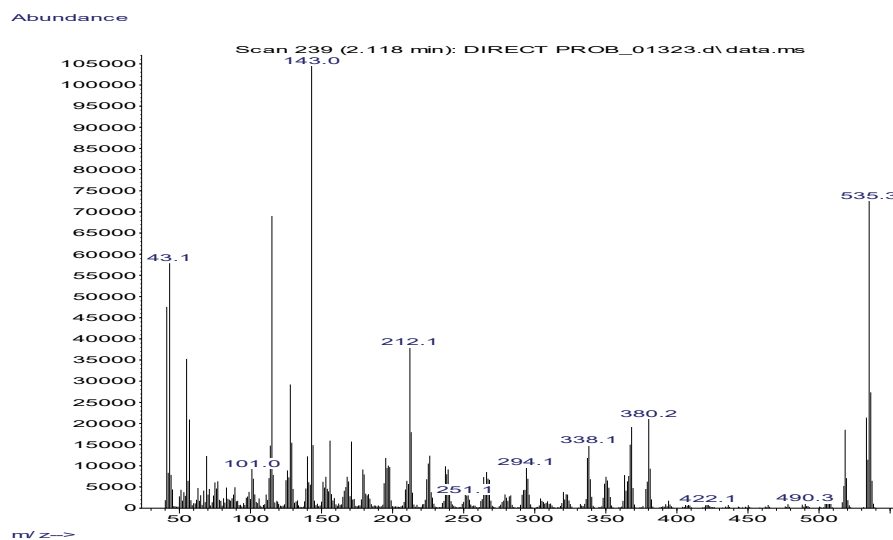


Fig. S-25. MS for dye 7a.

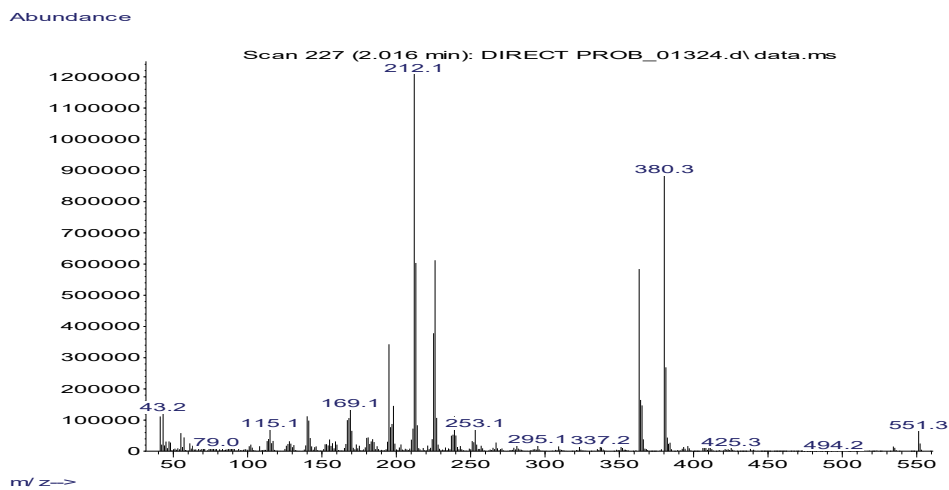


Fig. S-26. MS for dye 7b.

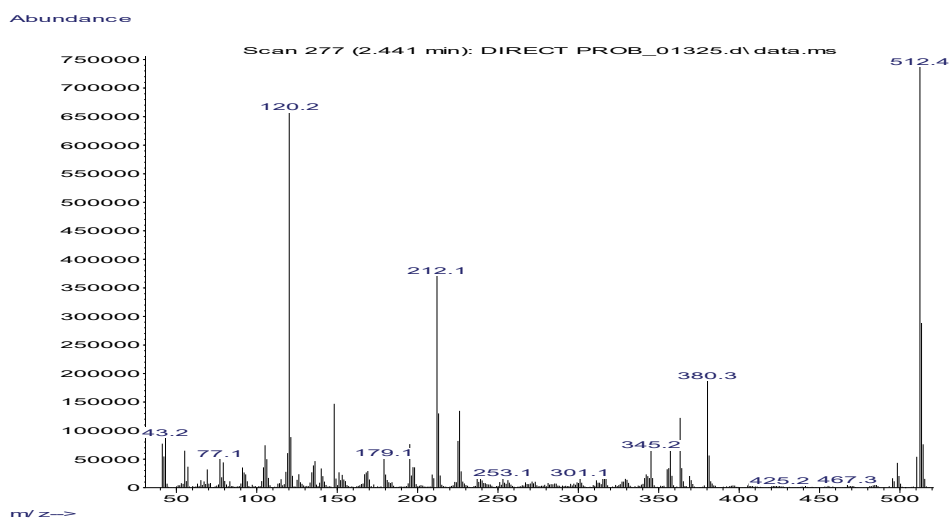
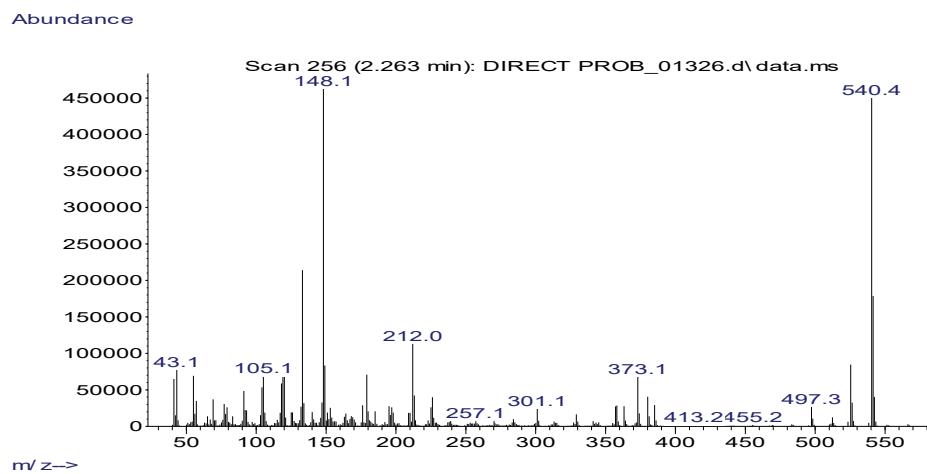


Fig. S-27. MS for dye 7c.



Fig. S-28. MS for dye **7d**.