Supporting Information

Development of Potent and Selective Inhibitors of *ecto*-5'-Nucleotidase Based on an Anthraquinone Scaffold

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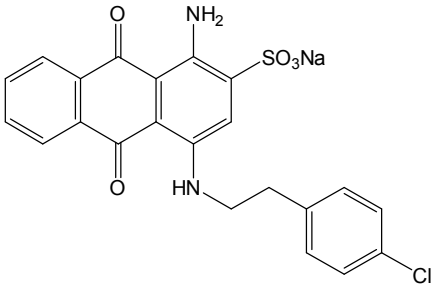
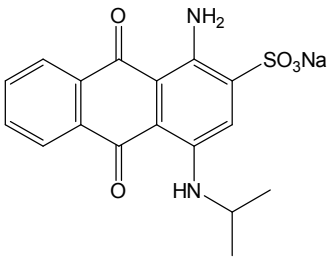
Analytical data of the newly synthesized anthraquinone derivatives

Data for new anthraquinone derivatives as well as for previously described anthraquinone derivatives that have now been synthesized according to a new, recently developed method are given.[1] All other compounds were previously described.[2-4]

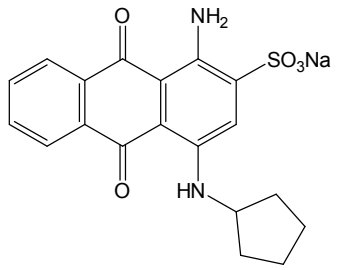


Sodium 1-amino-4-(4-chlorophenethylamino)-9,10-dioxo-9,10-dihydroanthracene-2-sulfonate (11): According to the general procedure: 15 min, 120 °C, 100 W; pressure up to 10 bar. Analytical data: mp > 300 °C, blue powder. ^{1}H-NMR: δ 2.98 (q, 2H, 1'-CH_2), 3.66 (t, 2H, 4-NHC$\underline{H_2}$), 7.37 (m, 4H, 2'-H, 3'-H, 5'-H, 6'-H), 7.79 (m, 2H, 6-H, 7-H), 7.81 (s, 1H, 3-H), 8.21 (m, 2H, 5-H, 8-H), 10.70 (br, 2H, 1-NH_2), (br, 1H, 4-NH not detectable). ^{13}C-NMR: δ 34.50 (1'-CH_2), 43.71 (4-NHCH_2), 109.16 (C-9a), 109.30 (C-4a), 120.93 (C-3), 125.86 (C-5), 126.01 (C-8), 128.50 (C-2', C-6'), 130.76 (C-3', C-5'), 131.16 (C-4'), 132.63 (C-6, C-7), 134.01 (C-10a), 134.09 (C-8a), 138.08 (C-1'), 143.21 (C-4), 143.73 (C-2), 144.96 (C-1), 181.10 (C-9), 181.77 (C-10). LC-MS (m/z): 474 [M-Na+$NH4^+$], 457 $[M-Na]^+$, 455 $[M-Na]^-$. Purity by HPLC-UV (254 nm)-ESI-MS: 99%.

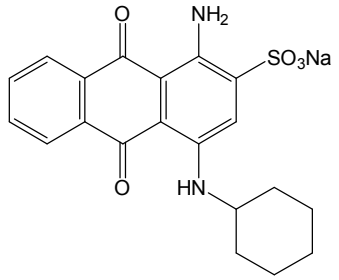


Sodium 1-amino-4-(isopropylamino)-9,10-dioxo-9,10-dihydroanthracene-2-sulfonate (12): According to the general procedure: 5 min, 120 °C, 100 W; pressure up to 10 bar. Analytical data: mp > 300 °C, blue powder. ^{1}H-NMR: δ 1.30 (d, 6H, $2CH_3$), 3.95 (m, 1H, CH), 7.77 (s, 1H, 3-H), 7.79 (m, 2H, 6-H, 7-H), 8.24 (m, 2H, 5-H, 8-H), 10.13 (br, 2H, 1-NH_2), 10.76 (d, 1H, 4-NH). ^{13}C-NMR: δ 23.3 ($2CH_3$), 43.4 (CH), 108.7 (C-9a), 109.2 (C-4a), 121.3, 125.8, 126.0, (C-3, C-5, C-8), 132.5, 132.6, 134.09, 134.13 (C-8a, C-10a, C-6, C-7), 143.1 (C-4), 143.8 (C-2), 144.6 (C-1), 180.8 (C-

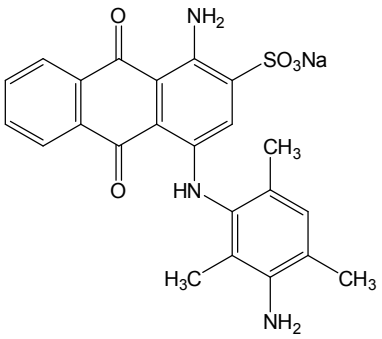
9), 181.6 (C-10). LC-MS (m/z): 378 [M-Na+NH_4^+]$^+$, 361 [M-Na]$^+$, 359 [M-Na]$^-$. Purity by HPLC-UV (254 nm)-ESI-MS: 99.2%.



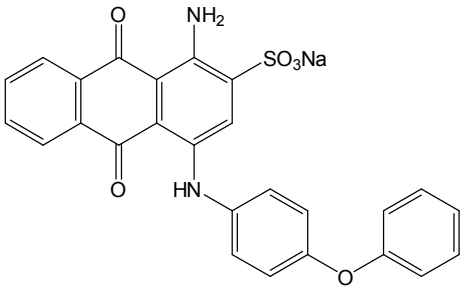
Sodium 1-amino-4-(cyclopentylamino)-9,10-dioxo-9,10-dihydroanthracene-2-sulfonate (13): According to the general procedure: 24 min, 120 °C, 100 W; pressure up to 10 bar. Analytical data: mp > 300 °C, blue powder. ^{1}H-NMR: δ 1.57, 1.67, 1.76, 2.08 (each m, 8H, 2'-H, 3'- H, 4'-H, 5'- H), 4.11 (m, 1H, 1'-H), 7.78 (m, 3H, 3-H, 6-H, 7-H), 8.23 (m, 2H, 5-H, 8-H), 10.60 (br, 2H, 1-NH_2), (s, 1H, 4-NH not detectable). ^{13}C-NMR: δ 23.71 (C-3', C-4'), 33.74 (C-2', C-5'), 53.53 (C-1'), 108.95 (C-9a), 109.22 (C-4a), 121.74 (C-3), 125.83 (C-5), 126.00 (C-8), 132.52 (C-6), 132.61 (C-7), 134.11 (C-10a, C-8a), 143.21 (C-4), 143.69 (C-2), 144.87 (C-1), 180.85 (C-9), 181.65 (C-10). LC-MS (m/z): 404 [M-Na+NH4$^+$], 387 [M-Na]$^+$, 385 [M-Na]$^-$. Purity by HPLC-UV (254 nm)-ESI-MS: 98%.



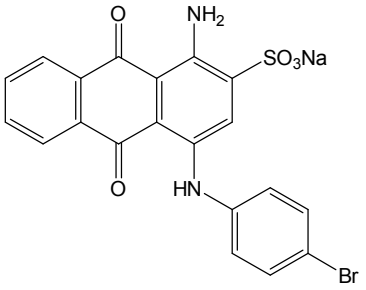
Sodium 1-amino-4-(cyclohexylamino)-9,10-dioxo-9,10-dihydroanthracene-2-sulfonate (14): According to the general procedure: 24 min, 120 °C, 100 W; pressure up to 10 bar. Analytical data: mp > 300 °C, blue powder. ^{1}H-NMR: δ 1.31, 1.43, 1.58, 1.72, 1.98 (each m, 10H, 2'-H, 3'-H, 4'-H, 5'-H, 6'-H), 3.68 (m, 1H, 1'-H), 7.77 (s, 1H, 3-H), 7.79 (m, 2H, 6-H, 7-H), 8.24 (m, 2H, 5-H, 8-H), 10.80 (br, 2H, 1-NH_2), (s, 1H, 4-NH not detectable). ^{13}C-NMR: δ 23.99. 25.28. 32.98 (C-2', C-3', C-4', C-5', C-6'), 50.15 (C-1'), 108.93 (C-9a), 109.29 (C-4a), 121.41 (C-3), 125.85 (C-5), 125.99 (C-8), 132.50 (C-6), 132.60 (C-7), 134.09 (C-10a), 134.12 (C-8a), 143.18 (C-4), 143.77 (C-2), 144.37 (C-1), 180.81 (C-9), 181.60 (C-10). LC-MS (m/z): 401 [M-Na]$^+$, 399 [M-Na]$^-$. Purity by HPLC-UV (254 nm)-ESI-MS: 99%.



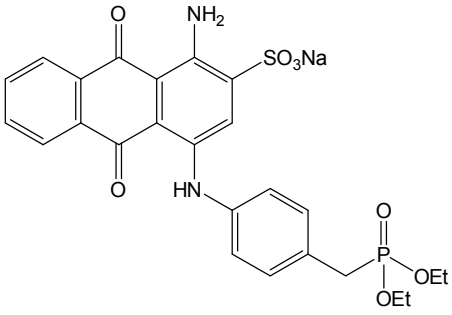
Sodium 1-amino-4-(3-amino-2,4,6-trimethylphenylamino)-9,10-dioxo-9,10-dihydro anthracene-2-sulfonate (21): According to the general procedure: 5 min, 120 °C, 100 W; pressure up to 10 bar. Analytical data: mp > 300 °C, blue powder. ^{1}H-NMR: δ 1.92, 1.97, 2.12 (3 s, 9H, 3CH_3), 4.53 (br, 2H, NH_2), 6.83 (s, 1H, 5'-H), 7.16 (s, 1H, 3-H), 7.83 (m, 2H, 6-H, 7-H), 8.29 (m, 2H, 5-H, 8-H), 10.1 (br, 2H, 1-NH_2), 11.81 (s, 1H, 4-NH). ^{13}C-NMR: δ 12.7, 17.5, 17.9 (3 CH_3), 109.0, 109.2 (C-9a), 118.6 (C-2', C-6'), 120.1, 121.9, 122.4 (C-4a), 126.0, 126.12, 129.5 (C-3, C-5, C-8, C-4', C-5'), 132.7, 132.9, 133.6, 133.9, 134.3 (C-8a, C-10a, C-6, C-7), 143.3 (C-1'), 143.6 (C-4), 143.9, 144.8 (C-2), (C-1), (C-3'), 181.6 (C-9), 182.8 (C-10). LC-MS (m/z): 469 [M-Na+NH_4^+]$^+$, 452 [M-Na]$^+$, 450 [M-Na]$^-$. Purity by HPLC-UV (254 nm)-ESI-MS: 95%.



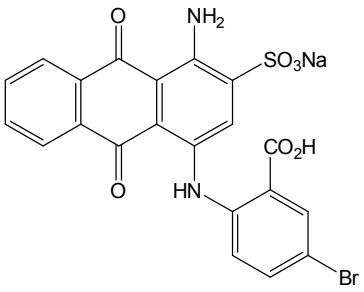
Sodium 1-amino-4-(4-phenoxyphenylamino)-9,10-dioxo-9,10-dihydroanthracene-2-sulfonate (26): According to the general procedure: 10 min, 100 °C, 80 W; pressure up to 10 bar. Analytical data: mp > 300 °C, blue powder. ^{1}H-NMR: δ 7.08 (m, 4H, 2'-H, 3'-H, 5'-H, 6'-H), 7.15 (dd, 1H, 4''-H), 7.30 (d, 2H, 2''-H, 6''-H), 7.41 (dd, 2H, 3''-H, 5''-H), 7.84 (m, 2H, 6-H, 7-H), 7.95 (s, 1H, 3-H), 8.27 (m, 2H, 5-H, 8-H), 10.1 (br, 2H, 1-NH_2), 12.04 (s, 1H, 4-NH). ^{13}C-NMR: δ 109.2 (C-9a), 111.2 (C-2', C-6'), 122.7 (C-4a), 123.4, 126.05, 126.1, 128.6, 128.9, 130.0 (C-3, C-5, C-8, C-4', C-3', C-5', C4''), 132.9, 133.2, 133.7, 134.3, 137.2, 137.8 (C-8a, C-10a, C-6, C-7, C-2'', C-6'', C-3'', C-5''), 141.3 (C-1'), 143.0 (C-4), 144.4 (C-2, C-1), 181.9 (C-9), 182.4 (C-10). LC-MS (m/z): 504 [M-Na+NH_4^+]$^+$, 487 [M-Na]$^+$, 485 [M-Na]$^-$. Purity by HPLC-UV (254 nm)-ESI-MS: 97.7%.



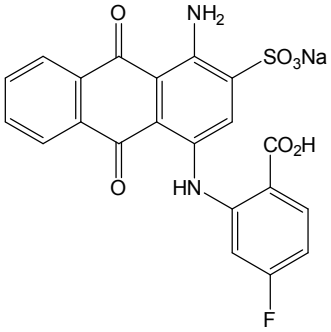
Sodium 1-amino-4-(4-bromophenylamino)-9,10-dioxo-9,10-dihydroanthracene-2-sulfonate (30): According to the general procedure: 20 min, 120 °C, 100 W; pressure up to 10 bar. Analytical data: mp > 300 °C, blue powder. ^{1}H-NMR: δ 7.24 (d, 2H, 2'-H, 6'-H), 7.59 (d, 2H, 3'-H, 5'-H), 7.85 (m, 2H, 6-H, 7-H), 7.97 (s, 1H, 3-H), 8.26 (m, 2H, 5-H, 8-H), 10.05 (br, 2H, 1-NH_2), 11.84 (s, 1H, 4-NH). ^{13}C-NMR: δ 109.5 (C-9a), 112.3 (C-4a), 116.1 (C6'), 122.8 (C-3), 124.7 (C-2'), 124.9 (C-2'), 126.1 (C-5, C-8), 126.2 (C-4'), 132.6 (C-3' and C-5'), 133.0 (C-6), 133.5 (C-7), 133.6 (C-10a), 134.3 (C-8a), 139.1 (C-1'), 140.0 (C-4), 142.8 (C-2), 144.6 (C-1), 182.1 (C-9), 183.0 (C-10). LC-MS (m/z): 473 $[M\text{-}Na]^{+}$, 471 $[M\text{-}Na]^{-}$. Purity by HPLC-UV (254 nm)-ESI-MS: 99.2 %.



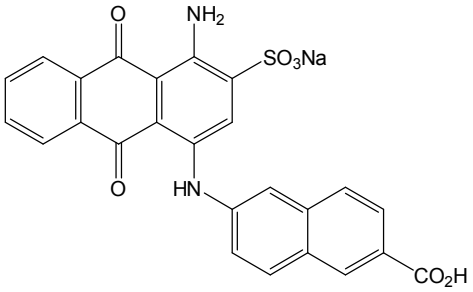
Sodium 1-amino-4-[4-(diethoxyphosphoryl)methylphenyl-amino]-9,10-dioxo-9,10-dihydroanthracene-2-sulfonate (44): According to the general procedure: 5 min, 120 °C, 100 W; pressure up to 10 bar. Analytical data: mp > 300 °C, blue powder. ^{1}H-NMR: δ 1.19 (. t, 6H, 2-CH_3), 3.24 (d, 2H, 4'-CH_2P), 3.96 (m, 4H, 2-OCH_2), 7.22 (d, 2H, 2'-H, 6'-H), 7.34 (dd, 2H, 3'-H, 5'-H), 7.84 (m, 2H, 6-H, 7-H), 8.00 (s, 1H, 3-H), 8.27 (m, 2H, 5-H, 8-H), 10.10 (br, 2H, 1-NH_2), 12.04 (s, 1H, 4-NH). ^{13}C-NMR: δ 16.35, 16.38 (2 CH_3), 31.37, 32.44 (4'-CH_2P), 61.56 (2 OCH_2), 109.26 (C-9a), 111.42 (C-4a), 122.83 (C-3), 123.16 (C-4'), 126.08 (C-2'), 126.17 (C-6'), 128.60 (C-5), 128.68 (C-8), 131.10 (C-5'), 131.15 (C-3'), 132.90 (C-6), 133.28 (C-7), 133.73 (C-10a), 134.29 (C-8a), 137.81 (C-4), 141.09 (C-1'), 142.98 (C-2), 144.48 (C-1), 181.91 (C-9), 182.51 (C-10). ^{15}P-NMR: δ 26.96 (P=O[2OEt]), LC-MS (m/z): 567 $[M]^{+}$, 562 $[M\text{-}Na+NH_4^{+}]^{+}$, 545 $[M\text{-}Na]^{+}$, 543 $[M\text{-}Na]^{-}$. Purity by HPLC-UV (254 nm)-ESI-MS: 98.4%.



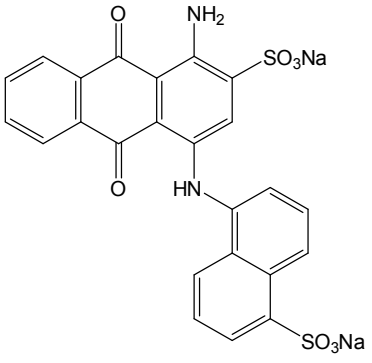
Sodium 1-amino-4-(4-bromo-2-carboxyphenylamino)-9,10-dioxo-9,10-dihydroanthracene-2-sulfonate (47): According to the general procedure: 5 min, 120 °C, 100 W; pressure up to 10 bar. Analytical data: mp > 300 °C, blue powder. ^{1}H-NMR: δ 7.15 (dd, 1H, 5'-H), 7.53 (d, 1H, 6'-H), 7.83 (m, 2H, 6-H, 7-H), 8.01 (d, 1H, 3'-H), 8.09 (s, 1H, 3-H), 8.20 (m, 2H, 5-H, 8-H), 9.95 (br, 2H, 1-NH_2), 12.47 (s, 1H, 4-NH). ^{13}C-NMR: δ 110.4, 112.1, 116.5, 121.6, 125.5, 126.2, 133.1, 133.5, 133.8, 134.2, 135.8, 141.1, 145.1, 182.5 (C-9), 182.7 (C-10). LC-MS (m/z): 534 $[M\text{-}Na+NH_4^+]^+$, 517 $[M\text{-}Na]^+$, 515 $[M\text{-}Na]^-$. Purity by HPLC-UV (254 nm)-ESI-MS: 98.9%.



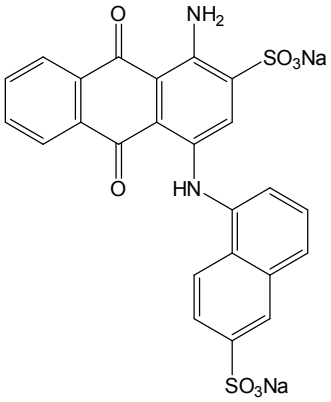
Sodium 1-amino-4-(2-carboxy-5-fluorophenylamino)-9,10-dioxo-9,10-dihydroanthracene-2-sulfonate (48): According to the general procedure: 5 min, 120 °C, 100 W; pressure up to 10 bar. Analytical data: mp > 300 °C, blue powder. ^{1}H-NMR: δ 6.82 (m, 1H, 4'-H), 6.98 (dd, 1H, 6'-H), 7.85 (m, 2H, 6-H, 7-H), 8.02 (dd, 1H, 3'-H), 8.11 (s, 1H, 3-H), 8.20 (m, 2H, 5-H, 8-H), 12.13 (s, 1H, 4-NH). ^{13}C-NMR: δ 104.7, 104.9 (C-9a), 107.6, 107.7 (C-2', C-6'), 110.8, 114.6, 118.3 (C-4a), 126.0, 126.2 (C-3, C-5, C-8, C-4', C-5'), 133.2, 133.6, 133.8, 134.0, 134.2, 134.8 (C-8a, C-10a, C-6, C-7), 140.8 (C-1'), 145.5 (C-4), 145.6 (C-2), (C-1), (C-3'), 166.0-164.0 (d, C-5'), 167.4 (COOH), 182.9 (C-9), 183.4 (C-10). LC-MS (m/z): 474 $[M\text{-}Na+NH_4^+]^+$, 457 $[M\text{-}Na]^+$, 455 $[M\text{-}Na]^-$. Purity by HPLC-UV (254 nm)-ESI-MS: 98.5%.



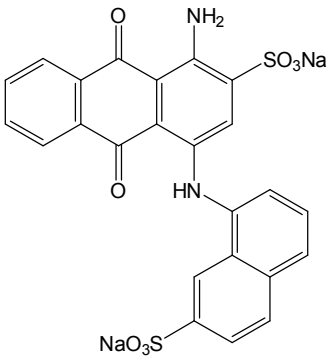
Sodium 1-amino-4-(6-carboxy-2-naphthylamino)-9,10-dioxo-9,10-dihydroanthracene-2-sulfonate (51): According to the general procedure: 5 min, 120 °C, 100 W; pressure up to 10 bar. Analytical data: mp > 300 °C, blue powder. ^{1}H-NMR: δ 7.55 (dd, 1H, 1 naphthalene H), 7.78 (d, 1H, 1 naphthalene H), 7.86 (m, 3H, 6-H, 7-H, 1 naphthalene H), 7.95 (dd, 1H, 1 naphthalene H), 8.15 (d, 1H, 1 naphthalene H), 8.17 (s, 1H, 3-H), 8.27 (m, 2H, 5-H, 8-H), 8.57 (br, 1H, 1 naphthalene H), 12.05 (br, 1H, NH). ^{13}C-NMR: δ 109.6, 113.1 (C4a, C9a), 117.3, 123.3 (C2', C4'), 123.4 (C3), 126.1, 126.2 (2C), 126.9, 127.3, 129.1, 130.5 (C5, C8, C5', C6', C7', C8', C4a', C8a'), 131.1, 133.0, 133.6, 133.6, 134.3, 136.2 (C6, C7, C8a, C10a, C1', C3'), 139.2, 139.8, 142.6, 144.9 (C1, C2, C4), 167.5 (COOH), 182.2 (C9), 183.2 (C10). LC-MS (m/z): 506 $[M\text{-}Na+NH_4^+]^+$, 489 $[M\text{-}Na]^+$, 487 $[M\text{-}Na]^-$. Purity by HPLC-UV (254 nm)-ESI-MS: 99%.



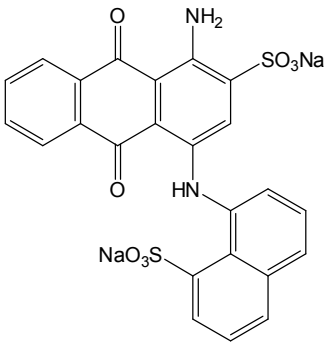
Disodium 1-amino-4-(5-sulfo-1-naphthylamino)-9,10-dioxo-9,10 dihydroanthracene-2-sulfonate (54): According to the general procedure: 5 min, 120 °C, 100 W; pressure up to 10 bar. Analytical data: mp > 300 °C, blue powder. ^{1}H-NMR: δ 7.47 (d, 1H, 1 naphthalene H), 7.52 (dd, 1H, 1 naphthalene H), 7.58 (dd, 1H, 1 naphthalene H), 7.74 (s, 1H, 3-H), 7.87 (m, 2H, 6-H, 7-H), 8.05 (dd, 2H, 1 naphthalene H), 8.32 (m, 2H, 5-H, 8-H), 8.82 (d, 1H, 1 naphthalene H), 10.12 (br, 2H, 1-NH_2), 12.50 (s, 1H, NH). ^{13}C-NMR: δ 109.1, 111.2 (C4a, C9a), 121.8, 122.9 (C2', C4'), 123.3 (C3), 125.6, 125.9 (2C), 126.2, 129.8, 130.5, 132.9, 133.3 (C5, C8, C5', C6', C7', C8', C4a', C8a'), 133.8, 134.4, 134.6, 134.9 (C6, C7, C8a, C10a, C1', C3'), 142.9, 143.2, 144.4, 144.9 (C1, C2, C4), 181.9 (C9), 182.9 (C10). LC-MS (m/z): 542 $[M\text{-}2Na+NH_4^+]^+$, 523 $[M\text{-}2Na]^-$, 261 $[M\text{-}2Na]^{2-}$. Purity by HPLC-UV (254 nm)-ESI-MS: 98%.



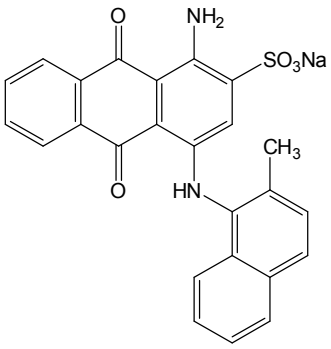
Disodium 1-amino-4-(6-sulfo-1-naphthylamino)-9,10-dioxo-9,10 dihydroanthracene-2-sulfonate (55): According to the general procedure: 5 min, 120 °C, 100 W; pressure up to 10 bar. Analytical data: mp > 300 °C, blue powder. ^{1}H-NMR: δ 7.49 (dd, 1H, 1 naphthalene H), 7.59 (dd, 1H, 1 naphthalene H), 7.80 (dd, 1H, 1 naphthalene H), 7.85 (s, 1H, 3-H), 7.87 (m, 2H, 6-H, 7-H), 7.90 (d, 1H, 1 naphthalene H), 8.04 (d, 1H, 1 naphthalene H), 8.24 (d, 1H, 1 naphthalene H), 8.32 (m, 2H, 5-H, 8-H), 10.1 (br, 2H, 1-NH_2), 12.50 (s, 1H, NH). ^{13}C-NMR: δ 109.3, 111.7 (C4a, C9a), 121.5, 121.7 (C2', C4'), 123.0 (C3), 124.9, 125.0 (2C), 126.18, 126.22, 126.6, 128.5, 132.9 (C5, C8, C5', C6', C7', C8', C4a', C8a'), 133.4, 133.7, 133.8, 134.4, 135.2 (C6, C7, C8a, C10a, C1', C3'), 142.2, 143.1, 144.5, 146.5 (C1, C2, C4), 181.9 (C9), 183.0 (C10). LC-MS (m/z): 542 $[M-2Na+NH_4^+]^+$, 523 $[M-2Na]^-$, 261 $[M-2Na]^{2-}$. Purity by HPLC-UV (254 nm)-ESI-MS: 99.6%.



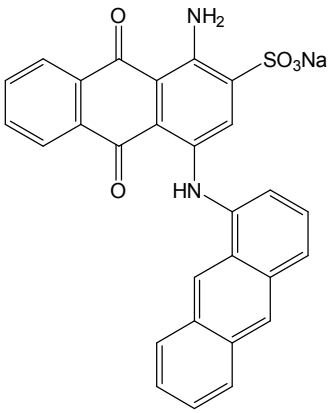
Disodium 1-amino-4-(7-sulfo-1-naphthylamino)-9,10-dioxo-9,10 dihydroanthracene-2-sulfonate (56): According to the general procedure: 5 min, 120 °C, 100 W; pressure up to 10 bar. Analytical data: mp > 300 °C, blue powder. ^{1}H-NMR: δ 7.48 (d, 1H, 1 naphthalene H), 7.58 (dd, 1H, 1 naphthalene H), 7.81 (m, 2H, 1 naphthalene H), 7.87 (m, 2H, 6-H, 7-H), 7.91 (s, 1H, 3-H), 7.97 (d, 1H, 1 naphthalene H), 8.32 (m, 2H, 5-H, 8-H), 8.52 (br, 1H, 1 naphthalene H), 10.15 (br, 2H, 1-NH_2), 12.51 (s, 1H, NH). ^{13}C-NMR: δ 109.3, 111.9 (C4a, C9a), 118.1, 121.0 (C2', C4'), 123.1 (C3), 124.9, 125.0 (2C), 126.2, 126.3, 126.6, 127.9, 128.3 (C5, C8, C5', C6', C7', C8', C4a', C8a'), 132.9, 133.4, 133.7, 134.2, 134.4, 136.0 (C6, C7, C8a, C10a, C1', C3'), 142.0, 143.1, 144.6, 146.7 (C1, C2, C4), 181.9 (C9), 183.1 (C10). LC-MS (m/z): 542 $[M-2Na+NH_4^+]^+$, 523 $[M-2Na]^-$, 261 $[M-2Na]^{2-}$. Purity by HPLC-UV (254 nm)-ESI-MS: 96%.



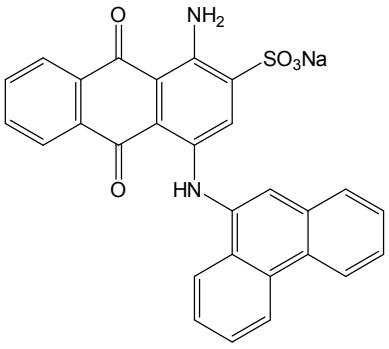
Disodium 1-amino-4-(8-sulfo-1-naphthylamino)-9,10-dioxo-9,10 dihydroanthracene-2-sulfonate (57): According to the general procedure: 5 min, 120 °C, 100 W; pressure up to 10 bar. Analytical data: mp > 300 °C, blue powder. ^{1}H-NMR: δ 7.48 (d, 1H, 1 naphthalene H), 7.58 (dd, 1H, 1 naphthalene H), 7.81 (m, 2H, 1 naphthalene H), 7.87 (m, 2H, 6-H, 7-H), 7.91 (s, 1H, 3-H), 7.97 (d, 1H, 1 naphthalene H), 8.32 (m, 2H, 5-H, 8-H), 8.52 (br, 1H, 1 naphthalene H), 10.15 (br, 2H, 1-NH_2), 12.51 (s, 1H, NH). ^{13}C-NMR: δ 109.3, 111.9 (C4a, C9a), 118.1, 121.0 (C2', C4'), 123.1 (C3), 124.9, 125.0 (2C), 126.2, 126.3, 126.6, 127.9, 128.3 (C5, C8, C5', C6', C7', C8', C4a', C8a'), 132.9, 133.4, 133.7, 134.2, 134.4, 136.0 (C6, C7, C8a, C10a, C1', C3'), 142.0, 143.1, 144.6, 146.7 (C1, C2, C4), 181.9 (C9), 183.1 (C10). LC-MS (m/z): 542 $[M-2Na+NH_4^+]^+$, 523 $[M-2Na]^-$, 261 $[M-2Na]^{2-}$. Purity by HPLC-UV (254 nm)-ESI-MS: 96%.



Sodium 1-amino-4-(2-methyl-1-naphthylamino)-9,10-dioxo-9,10-dihydroanthracene-2-sulfonate (58): According to the general procedure: 20 min, 120 °C, 100 W; pressure up to 10 bar. Analytical data: mp > 300 °C, blue powder. ^{1}H-NMR: δ 2.33 (s, 3H, CH_3), 7.02 (s, 1H, 3-H), 7.50 (m, 2H, 2 naphthalene H), 7.57 (d, 1H, J = 8.5 Hz, 3'-H or 4'-H), 7.79 (m, 1H, 1 naphthalene H), 7.87 (m, 2H, 6-H, 7-H) 7.91 (d, 1H, J = 8.5 Hz, 3'-H or 4'-H), 8.00 (m, 1H, 1 naphthalene H), 8.33 (m, 2H, 5-H, 8-H), 10.10 (br, 2H, 1-NH_2), 12.09 (s, 1H, NH). ^{13}C-NMR: δ 18.7 (CH_3), 109.3, 110.0 (C-4a, C-9a), 122.1 (C-2', C-4'), 122.5 (C-3), 125.9, 126.15, 126.21, 127.27, 127.30, 128.3, 129.2 (C-5, C-8, C-5', C-6', C-7', C-8', C-4a', C-8a'), 131.3, 132.1, 132.9, 133.0, 133.2, 133.3, 133.8, 134.4 (C-6, C-7, C-8a, C-10a, C-1', C-3'), 143.6, 144.0, 144.3 (C-1, C-2, C-4), 181.9 (C-9), 182.7 (C-10). LC-MS (m/z): 476 $[M-Na+NH_4^+]^+$, 459 $[M-Na]^+$, 457 $[M-Na]^-$. Purity by HPLC-UV (254 nm)-ESI-MS: 99.4%.



Sodium 1-amino-4-(1-anthracenylamino)-9,10-dioxo-9,10 dihydro-anthracene-2-sulfonate (59): According to the general procedure: 5 min, 120 °C, 100 W; pressure up to 10 bar. Analytical data: mp > 300 °C, blue powder. ^{1}H-NMR: δ 7.53 (m, 5H, 5 anthracene H), 7.87 (m, 3H, 3-H, 6-H, 7-H), 8.02 (d, 1H, 1 anthracene H), 8.14 (t, 2H, 2 anthracene H), 8.34 (m, 2H, 5-H, 8-H), 8.70 (br, 1H, 1 anthracene H), 8.74 (br, 1H, 1 anthracene H), 10.15 (br, 2H, 1-NH_2), 12.60 (s, 1H, NH). ^{13}C-NMR: δ 109.2, 111.9 (C4a, C9a), 120.1, 120.6 (C2', C4'), 123.3 (C3), 125.5, 125.8, 126.1 (2C), 126.2, 126.3, 127.1, 127.8, 128.0, 128.7, 131.6, 131.7 (C5, C8, C5', C6', C7', C8', C4a', C8a'), 132.3, 132.9, 133.4, 133.8, 134.4, 135.4, (C6, C7, C8a, C10a, C1', C3'), 142.4, 143.1, 144.6 (C1, C2, C4), 181.9 (C9), 183.0 (C10). LC-MS (m/z): 495 $[M-Na]^+$, 493 $[M-Na]^-$. Purity by HPLC-UV (254 nm)-ESI-MS: 96.5%.



Sodium 1-amino-4-(9-phenanthrenylamino)-9,10-dioxo-9,10-dihydroanthracene-2-sulfonate (60): According to the general procedure: 5 min, 120 °C, 100 W; pressure up to 10 bar. Analytical data: mp > 300 °C, blue powder. ^{1}H-NMR: δ 7.71 (m, 3H, 3 phenanthrene H), 7.79 (m, 2H, 2 phenanthrene H), 7.85 (s, 1H, 3-H), 7.88 (m, 2H, 5-H, 8-H), 7.96 (d, 1H, J = 7.85 Hz, 1 phenanthrene H), 8.14 (d, 1H, J = 8.2 Hz, 1 phenanthrene H), 8.33 (m, 2H, 5-H, 8-H), 8.87 (d, 1H, 1 phenanthrene H), 8.96 (d, 1H, J = 7.85 Hz, 1 phenanthrene H), 10.11 (br, 2H, 1-NH_2), 12.46 (s, 1H, NH). ^{13}C-NMR: δ 109.2, 111.5 (C-4a, C-9a), 121.9 (C-2', C-4'), 122.8 (C-3), 123.1, 123.2, 123.8, 126.20, 126.24, 126.8, 127.4, 127.8, 128.4, 128.6, 129.0, 131.2, 131.6 (C-5, C-8, C-5', C-6', C-7', C-8', C-4a', C-8a'), 132.9, 133.4, 133.6, 133.8, 134.4 (C-6, C-7, C-8a, C-10a, C-1', C-3'), 142.9, 143.2, 144.5 (C-1, C-2, C-4), 181.9 (C-9), 183.0 (C-10). LC-MS (m/z): 495 $[M-Na]^+$, 493 $[M-Na]^-$. Purity by HPLC-UV (254 nm)-ESI-MS: 96.5%.

References

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