Anticancer Activity Expressed by a Library of 2,9-Diazaperopyrenium Dications

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SUPPORTING INFORMATION

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S1. Experimental Procedures

Materials and General Methods: All reagents were purchased from commercial suppliers and used without further purification. 3,4,9,10-Tetrakis(chloromethyl)perylene, 1 N,N'-bis(2-[2-hydroxyethoxy]ethyl)perylene-3,4:9,10-tetracarboxylic acid bisimide, 2 N,N'-bis(2,6-diisopropylphenyl)perylene-3,4:9:10-bis(dicarboximide) 3 (9), 1^{2+}, and 4^{2+} were all prepared according to literature procedures. All compounds were purified by preparative RP-HPLC (Shimadzu LC-8A), using a C18 column (Waters, XBridge Prep C18 5µm OBD, 19 × 100 mm). The eluants used were MeCN and H2O, both mixed with 0.1 % (v/v) trifluoroacetic acid (TFA). All compounds were characterized as their hexafluorophosphate salts. The detector was set to λ = 254 nm. Nuclear magnetic resonance (NMR) spectra were recorded at 298 K on a Bruker Avance III 500 spectrometer, with working frequencies of 499.373 and 125.579 MHz for 1H and 13C nuclei, respectively. All 13C NMR spectra were recorded with the simultaneous decoupling of 1H nuclei. Chemical shifts are reported in ppm relative to the signals corresponding to the residual non-deuterated solvents (1.94 and 4.33 ppm for CHD2CN and CHD2NO2, respectively for 1H, 118.26 and 62.8 ppm for CD3CN and CD3NO2, respectively for 13C). Electrospray Ionization (ESI) mass spectra were obtained on an Agilent 6210 LC-TOF high resolution mass spectrometer. In order to ensure solubility of all dications in aqueous media for biological studies, the hexafluorophosphate counterions were replaced by chloride anions by precipitating the dichloride salt from an MeCN solution of the hexafluorophosphate salt after addition of n-butyl ammonium chloride, and subsequent washing with an excess of MeCN to remove any remaining n-butyl ammonium chloride and/or hexafluorophosphate.
Scheme S1: Possible routes towards the synthesis of 1 – 8•2PF₆

**2•2PF₆: Strategy 1.** 3,4,9,10-Tetrakis(chloromethyl)perylene (100 mg, 0.22 mmol) was added to neat ethylamine (20 mL) and the solution was stirred at room temperature for 24 h under N₂. Et₂O (200 mL) was added in order to precipitate the crude product. The solution was filtered and the residue was washed with Et₂O (3 × 20 mL) and dried to give a dark red solid (69 mg, 74%). This crude material (69 mg, 0.17 mmol) was then added to MeCN (50 mL), followed by addition of DDQ (372 mg, 1.4 mmol) and this solution was then stirred under reflux in an O₂ atmosphere for 16 h, before being cooled to room temperature. A 32% HCl solution (5 mL) was added to
precipitate the chloride salt of the crude product. The solution was filtered and the residue was
dissolved in a 50/50 mixture of (Me)_2CO/H_2O, to which excess of aqueous NH_4PF_6 was added to
precipitate the bis(hexafluorophosphate) salt. The solution was filtered and the residue was
washed with H_2O (3 × 20 mL) and dried to give the product 2•2PF_6 as an orange solid (108 mg,
88%). **Strategy 2.** Perylene-3,4,9,10-tetracarboxylic dianhydride (1g, 2.5 mmol), imidazole (7.5
g) and a catalytic amount of zinc acetate were heated to 120 °C with stirring under an N_2
atmosphere. Upon melting of the imidazole, ethylamine (2M in THF, 5 mL) was added and the
mixture was left to stir for 16 h. The crude product was treated with MeOH (30 mL) and 6M
HCl solution (10 mL) and the mixture filtered. The residue was washed with MeOH (2 x 30 mL)
and Et_2O (2 x 30 mL) and dried under vacuum. The residue (1.2 g) was then added to a mixture
of LiAlH_4 (1.3 g, 34 mmol) in anhydrous THF (250 mL), which was heated and stirred under N_2
for 16 h. The solution was cooled to 0 °C and the reaction was quenched with H_2O (1.5 mL),
15 % NaOH solution (1.5 mL) and H_2O (5 mL). The mixture was evaporated to dryness and the
residue was subjected to Soxhlet extraction (CHCl_3) for three days, yielding a crude solid (0.5 g)
after evaporation of CHCl_3. This solid was then added to a mixture of glacial acetic acid (100
mL) and chloranil (1.2 g, 4.9 mmol) which was heated under reflux for 4 h. Upon cooling to
room temperature, the mixture was poured into H_2O (500 mL) and filtered. Excess of aqueous
NH_4PF_6 was then added to the filtrate to precipitate the bis(hexafluorophosphate) salt, which was
washed with H_2O (3 × 20 mL) and dried to give 2•2PF_6 as an orange solid (170 mg, 10%). ^1H
NMR (CD_3NO_2, 500 MHz, 298 K): δ = 9.96 (s, 4H), 9.93 (d, J = 9.4 Hz, 4H), 9.01 (d, J = 9.3
Hz, 4H), 5.34 (q, J = 7.4 Hz, 4H), 2.04 (t, J = 7.4 Hz, 6H) ppm. ^13C NMR (CD_3NO_2, 125 MHz,
298 K): δ = 139.4, 131.0, 130.6, 130.1, 129.9, 128.0, 123.2, 60.3, 17.6 ppm. ESI-MS: calcd for
[M – PF_6]^+, m/z = 531.1425, found: m/z = 531.1406.
**3•2PF₆:** 3,4,9,10-Tetrakis(chloromethyl)perylene (100 mg, 0.22 mmol) was added to neat isopropylamine (20 mL) and the solution was stirred at room temperature for 24 h under N₂. Et₂O (200 mL) was added in order to precipitate the crude product. The solution was filtered and the residue was washed with Et₂O (3 × 20 mL) and dried to give a brown solid (71 mg). This crude material (60 mg) was then added to MeCN (50 mL), followed by addition of DDQ (250 mg, 1.1 mmol) and the solution was then stirred at room temperature for 16 h. A 32% aqueous HCl solution (5 mL) and Et₂O (100 mL) were added to precipitate the dichloride salt of the crude product. The solution was filtered and the residue was dissolved in a 50/50 mixture of (Me)₂CO/H₂O, to which an excess of aqueous NH₄PF₆ was added in order to precipitate the bis(hexafluorophosphate) salt. The solution was filtered and the residue was washed with H₂O (3 × 20 mL) and dried to give 3•2PF₆ as a brown/orange solid (21 mg, 14%). ¹H NMR (CD₃CN, 500 MHz, 298 K): δ = 9.91 (s, 4H), 9.84 (d, 3J = 9.2 Hz, 4H), 8.92 (d, 3J = 9.2 Hz, 4H), 5.50 (sep, 3J = 6.8 Hz, 2H), 1.98 (d, 3J = 6.7 Hz, 12H) ppm. ¹³C NMR (CD₃CN, 125 MHz, 298 K): δ = 137.8, 130.5, 129.8, 129.4, 129.4, 127.5, 122.6, 67.4, 23.8 ppm. ESI-MS: calcd for [M – PF₆]⁺, m/z = 559.1738, found: m/z = 559.1742.

**5•2PF₆:** N,N'-Bis(2-[2-hydroxyethoxy]ethyl)-perylene-3,4,9,10-tetracarboxylic acid bisimide (2.5 g, 4.4 mmol) was added to anhydrous THF (200 mL), followed by slow addition of BH₃-DMS (2M in THF, 50 mL) and the solution was stirred under reflux for 5 days under N₂. After cooling the solution to room temperature, the solution was quenched by the slow addition of MeOH (200 mL). Conc. HCl (6M, 100 mL) was added and the solution was stirred under reflux for 3 h. Upon cooling the solution to room temperature, the solvent was removed under vacuum and the residue was suspended in a saturated K₂CO₃ solution until the pH reached 10. The
suspension was then filtered and the residue was washed with H₂O (3 × 50 mL) and dried to give a brown solid (1.4 g, 62%). This crude material (1.2 g) was then added to a MeCN/CH₂Cl₂ 50:50 mixture (200 mL), followed by addition of DDQ (4.4 g, 19.4 mmol) and this solution was then stirred at RT for 3 days. A 32% aqueous HCl solution (5 mL) and Et₂O (100 mL) were added in order to precipitate the dichloride salt of the crude product. The solution was filtered and the residue was dissolved in H₂O (50 mL), to which excess of aqueous NH₄PF₆ was added so as to precipitate the bis(hexafluorophosphate) salt. The solution was filtered and the residue was washed with H₂O (3 × 20 mL) and dried to give 5•2PF₆ as a brown/orange solid (0.47 g, 13%).

1H NMR (CD₃CN, 500 MHz, 298 K): δ = 9.72 (s, 4H), 8.41 (br s, 4H), 8.18 (d, 3J = 9.0 Hz, 4H), 5.31 (t, 3J = 4.8 Hz, 4H), 4.35 (t, 3J = 4.8 Hz, 4H), 3.78 (s, 8H) ppm. 13C NMR (CD₃CN, 125 MHz, 298 K): δ = 140.1, 128.9, 128.1, 127.9, 127.8, 127.0, 120.6, 73.6, 69.9, 63.6, 61.8 ppm. ESI-MS: calcd for [M – PF₆]+, m/z = 651.1847, found: m/z = 651.1848.

6•2PF₆: 3,4,9,10-Tetrakis(chloromethyl)perylene (100 mg, 0.22 mmol) was added to anhydrous THF (20 mL), followed by addition of p-toluidine (4.8 g) and the solution was stirred at 60°C for 16 h under N₂. After cooling the solution to room temperature, Et₂O (200 mL) was added to precipitate the crude product. The solution was filtered and the residue was washed with Et₂O (3 × 20 mL) and dried to give a brown solid (110 mg, 95%). This crude material (100 mg, 0.19 mmol) was then added to a MeCN/CH₂Cl₂ 50:50 mixture (50 mL), followed by addition of DDQ (530 mg, 2.3 mmol) and this solution was then heated to 40°C for 3 days. Upon cooling the solution to room temperature, a 32% aqueous HCl solution (5 mL) and Et₂O (100 mL) were added to precipitate the dichloride salt of the crude product. The solution was filtered and the residue was dissolved in a 50/50 mixture of (Me)₂CO/H₂O, to which an excess of aqueous
NH₄PF₆ was added to precipitate the bis(hexafluorophosphate) salt. The solution was filtered and the residue was washed with H₂O (3 × 20 mL) and dried to give 6•2PF₆ as a brown/orange solid (20 mg, 13%). ¹H NMR (CD₃CN, 500 MHz, 298 K): δ = 10.07 (s, 4H), 9.78 (d, ³J = 9.3 Hz, 4H), 8.94 (d, ³J = 9.3 Hz, 4H), 7.97 (d, ³J = 8.4 Hz, 4H), 7.73 (d, ³J = 8.4 Hz, 4H), 2.61 (s, 6H) ppm. ¹³C NMR (CD₃CN, 125 MHz, 298 K): δ = 143.6, 142.3, 139.4, 132.2, 130.2, 130.1, 129.8, 129.2, 127.7, 125.9, 122.6, 21.3 ppm. ESI-MS: calcd for [M – 2PF₆]²⁺, m/z = 255.1048, found: m/z = 255.1051.

7•2PF₆: 3,4,9,10-Tetrakis(chloromethyl)perylene (145 mg, 0.32 mmol) was added to neat m-toluidine (20 mL) and the solution was stirred at 60°C for 24 h under N₂. After cooling the solution to room temperature, Et₂O (200 mL) was added to precipitate the crude product. The solution was filtered and the residue was washed with Et₂O (3 × 20 mL) and dried to give a brown solid (165 mg, quant). This crude material (150 mg, 0.29 mmol) was added to a MeCN/CH₂Cl₂ 50:50 mixture (50 mL), followed by addition of DDQ (250 mg, 1.1 mmol) and this solution was heated to 60°C for 16 h. Upon cooling the solution to room temperature, a 32% aqueous HCl solution (5 mL) and Et₂O (100 mL) were added to precipitate the dichloride salt of the crude product. The solution was filtered and the residue was dissolved in a 50/50 mixture of (Me)₂CO/H₂O, to which an excess of aqueous NH₄PF₆ was added to precipitate the bis(hexafluorophosphate) salt. The solution was filtered and the residue was washed with H₂O (3 × 20 mL) and dried to give 7•2PF₆ a brown/orange solid (23 mg, 10%). ¹H NMR (CD₃CN, 500 MHz, 298 K): δ = 10.09 (s, 4H), 9.89 (d, ³J = 9.3 Hz, 4H), 8.99 (d, ³J = 9.3 Hz, 4H), 7.93 (s, 2H), 7.89 (d, ³J = 7.8 Hz, 2H), 7.79 (t, ³J = 7.8 Hz, 4H), 7.73 (d, ³J = 7.8 Hz, 2H), 2.63 (s, 6H) ppm. ¹³C NMR (CD₃CN, 125 MHz, 298 K): δ = 144.6, 142.5, 139.4, 133.3, 131.5, 130.4, 130.2,
8•2PF₆: 3,4,9,10-Tetrakis(chloromethyl)perylene (100 mg, 0.22 mmol) was added to neat 2,6-diisopropylaniline (20 mL) and the solution was stirred at 100°C for 24 h under N₂. After cooling the solution to room temperature, Et₂O (200 mL) was added to precipitate the crude product. The solution was filtered and the residue was washed with Et₂O (3 × 20 mL) and dried to give a brown solid (115 mg, 78%). This crude material (91 mg, 0.14 mmol) was added to MeCN (50 mL), followed by addition of DDQ (250 mg, 1.1 mmol) and this solution was then heated under reflux for 16 h. Upon cooling the solution to room temperature, a 32% aqueous HCl solution (5 mL) and Et₂O (100 mL) were added to precipitate the dichloride salt of the crude product. The solution was filtered and the residue was dissolved in a 50/50 mixture of (CH₃)₂CO/H₂O, to which excess of aqueous NH₄PF₆ was added to precipitate the bis(hexafluorophosphate) salt. The solution was filtered and the residue was washed with H₂O (3 × 20 mL) and dried to give 8•2PF₆ as a brown/orange solid (80 mg, 61%). ¹H NMR (CD₃CN, 500 MHz, 298 K): δ = 10.06 (d, 3J = 9.2 Hz, 4H), 9.93 (s, 4H), 9.03 (d, 3J = 9.2 Hz, 4H), 7.83 (t, 3J = 7.9 Hz, 2H), 7.65 (d, 3J = 7.9 Hz, 4H), 2.12 (sep, 3J = 6.7 Hz, 4H), 1.20 (d, 3J = 6.7 Hz, 24H) ppm. ¹³C NMR (CD₃CN, 125 MHz, 298 K): δ = 145.0, 140.4, 140.3, 133.4, 131.4, 130.6, 130.3, 129.8, 128.1, 126.0, 122.9, 29.3, 24.2 ppm. ESI-MS: calcd for [M – 2PF₆]²⁺, m/z = 325.1830, found: m/z = 325.1839.

A•2PF₆: The crude material (70 mg) produced from the reaction between 3,4,9,10-tetrakis(chloromethyl)perylene and isopropylamine, was acidified addition of conc HCl (6M, 50 mL) and the suspension was filtered to remove insoluble impurities. Excess of NH₄PF₆ was added to the filtrate to precipitate the crude product as a bis(hexafluorophosphate) salt.
Purification by RP-HPLC, followed by conversion back to the bis(hexafluorophosphate) salt using aqueous NH₄PF₆, gave A•2PF₆ as a yellow solid (27 mg, 17%). \(^1\)H NMR (CD₃CN, 500 MHz, 298 K): \(\delta = 8.21 \, (d, \, ^3J = 7.6 \, Hz, \, 4H), \, 7.44 \, (d, \, ^3J = 7.6 \, Hz, \, 4H), \, 4.68 \, (s, \, 8H), \, 3.83 \, (sep, \, ^3J = 6.7 \, Hz, \, 2H), \, 1.51 \, (d, \, ^3J = 6.7 \, Hz, \, 12H) \) ppm. \(^{13}\)C NMR (CD₃CN, 125 MHz, 298 K): \(\delta = 131.6, \, 128.2, \, 126.9, \, 126.2, \, 121.9, \, 59.8, \, 52.0, \, 17.4 \) ppm. ESI-MS: calcd for [(M – PF₆) – HPF₆]\(^+\), \(m/z = 419.2487\), found: \(m/z = 419.2491\).

B•2PF₆: The crude material (1.2 g) produced from the reduction of N,N'-bis(2-[2-hydroxyethoxy]ethyl)-perylene-3,4,9,10-tetracarboxylic acid bisimide, was acidified by addition of conc HCl (6M, 50 mL), and the suspension was filtered to remove insoluble impurities. An excess of aqueous NH₄PF₆ was added to the filtrate to precipitate the crude product as the bis(hexafluorophosphate) salt. Purification by RP-HPLC, followed by conversion back to the bis(hexafluorophosphate) salt using NH₄PF₆ gave B•2PF₆ as a yellow solid (0.53 g, 15%). \(^1\)H NMR (CD₃CN, 500 MHz, 298 K): \(\delta = 8.34 \, (d, \, ^3J = 7.8 \, Hz, \, 4H), \, 7.51 \, (d, \, ^3J = 7.8 \, Hz, \, 4H), \, 4.79 \, (s, \, 8H), \, 3.96 – 3.87 \, (m, \, 4H), \, 3.68 – 3.60 \, (m, \, 8H), \, 3.53 – 3.46 \, (m, \, 4H) \) ppm. \(^{13}\)C NMR (CD₃CN, 125 MHz, 298 K): \(\delta = 131.8, \, 128.3, \, 127.9, \, 126.6, \, 125.9, \, 122.1, \, 72.9, \, 64.5, \, 61.8, \, 55.4, \, 55.0 \) ppm. ESI-MS: calcd for [(M – PF₆) – HPF₆]\(^+\), \(m/z = 511.2591\), found: \(m/z = 511.2599\).
S2. $^1$H and $^{13}$C NMR Spectroscopy

Figure S1. $^1$H NMR Spectrum (500 MHz, CD$_3$NO$_2$, 298 K) of 2•2PF$_6$

Figure S2. $^{13}$C NMR Spectrum (125 MHz, CD$_3$NO$_2$, 298 K) of 2•2PF$_6$
Figure S3. $^1$H NMR Spectrum (500 MHz, CD$_3$CN, 298 K) of 3•2PF$_6$.

Figure S4. $^{13}$C NMR Spectrum (125 MHz, CD$_3$CN, 298 K) of 3•2PF$_6$. 
Figure S5. $^1$H NMR Spectrum (500 MHz, CD$_3$CN, 298 K) of A•2PF$_6$

Figure S6. $^{13}$C NMR Spectrum (125 MHz, CD$_3$CN, 298 K) of A•2PF$_6$
Figure S7. $^1$H NMR Spectrum (500 MHz, CD$_3$CN, 298 K) of 5•2PF$_6$.

Figure S8. $^{13}$C NMR Spectrum (125 MHz, CD$_3$CN, 298 K) of 5•2PF$_6$. 
Figure S9. $^1$H NMR Spectrum (500 MHz, CD$_3$CN, 298 K) of B•2PF$_6$

Figure S10. $^{13}$C NMR Spectrum (125 MHz, CD$_3$CN, 298 K) of B•2PF$_6$
Figure S11. $^1$H NMR Spectrum (500 MHz, CD$_3$CN, 298 K) of 6•2PF$_6$

Figure S12. $^{13}$C NMR Spectrum (125 MHz, CD$_3$CN, 298 K) of 6•2PF$_6$
Figure S13. $^1$H NMR Spectrum (500 MHz, CD$_3$CN, 298 K) of 7•2PF$_6$

Figure S14. $^{13}$C NMR Spectrum (125 MHz, CD$_3$CN, 298 K) of 7•2PF$_6$
Figure S15. $^1$H NMR Spectrum (500 MHz, CD$_3$CN, 298 K) of $8 \cdot 2$PF$_6$

Figure S16. $^{13}$C NMR Spectrum (125 MHz, CD$_3$CN, 298 K) of $8 \cdot 2$PF$_6$
S3. Cell Viability Studies and 50% Inhibitory Concentration Data

Table S1: Cell viability data (%) for compounds 1-8•2Cl (10 μM) for the 10 cell lines tested.

<table>
<thead>
<tr>
<th></th>
<th>FaDu</th>
<th>SKMEL-2</th>
<th>HT-29</th>
<th>HT-1080</th>
<th>HepG2</th>
<th>HL-60</th>
<th>Jurkat</th>
<th>PC-3</th>
<th>HeLa</th>
<th>MDA-MB-231</th>
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<tr>
<td>1</td>
<td>77.83 ± 1.17</td>
<td>142.57 ± 2.37</td>
<td>75.34 ± 3.68</td>
<td>96.06 ± 1.45</td>
<td>27.82 ± 0.84</td>
<td>105.02 ± 3.35</td>
<td>80.63 ± 1.68</td>
<td>96.97 ± 2.02</td>
<td>67.01 ± 2.65</td>
<td>90.80 ± 1.48</td>
</tr>
<tr>
<td>2</td>
<td>87.57 ± 2.26</td>
<td>100.05 ± 1.99</td>
<td>85.96 ± 2.81</td>
<td>96.76 ± 1.61</td>
<td>49.50 ± 2.26</td>
<td>98.03 ± 2.99</td>
<td>82.38 ± 1.23</td>
<td>98.81 ± 2.73</td>
<td>91.02 ± 3.26</td>
<td>93.54 ± 1.54</td>
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<tr>
<td>3</td>
<td>63.06 ± 7.73</td>
<td>93.93 ± 0.83</td>
<td>87.28 ± 1.06</td>
<td>95.88 ± 2.52</td>
<td>123.19 ± 4.36</td>
<td>100.14 ± 0.25</td>
<td>106.99 ± 1.28</td>
<td>58.51 ± 0.38</td>
<td>81.80 ± 1.27</td>
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<td>4</td>
<td>89.87 ± 3.32</td>
<td>89.69 ± 3.63</td>
<td>83.90 ± 2.51</td>
<td>92.72 ± 1.45</td>
<td>90.00 ± 4.93</td>
<td>96.40 ± 2.86</td>
<td>81.32 ± 1.41</td>
<td>82.95 ± 2.42</td>
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<td>153.85 ± 4.06</td>
<td>88.44 ± 6.46</td>
<td>95.38 ± 2.67</td>
<td>44.92 ± 2.49</td>
<td>105.16 ± 3.44</td>
<td>87.92 ± 2.57</td>
<td>89.54 ± 2.82</td>
<td>62.58 ± 1.15</td>
<td>91.07 ± 0.68</td>
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<td>85.70 ± 1.90</td>
<td>88.33 ± 2.60</td>
<td>86.36 ± 8.67</td>
<td>105.90 ± 1.22</td>
<td>98.54 ± 4.08</td>
<td>99.74 ± 3.59</td>
<td>83.79 ± 1.48</td>
<td>93.78 ± 3.76</td>
<td>100.87 ± 0.68</td>
<td>100.17 ± 0.93</td>
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<td>7</td>
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<td>80.51 ± 4.42</td>
<td>100.24 ± 0.82</td>
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<td>101.64 ± 3.14</td>
<td>76.52 ± 2.51</td>
<td>85.25 ± 1.32</td>
<td>98.93 ± 1.41</td>
<td>96.56 ± 0.90</td>
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<tr>
<td>8</td>
<td>20.55 ± 1.14</td>
<td>7.18 ± 2.50</td>
<td>14.94 ± 1.68</td>
<td>35.65 ± 1.83</td>
<td>66.77 ± 5.15</td>
<td>51.11 ± 3.41</td>
<td>28.86 ± 1.87</td>
<td>45.56 ± 1.22</td>
<td>32.32 ± 1.80</td>
<td>66.13 ± 3.49</td>
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Table S2: 50% Inhibitory concentration (IC$_{50}$) data (μM) for compounds A•2Cl, B•2Cl, 8•2Cl and doxorubicin against selected cell lines.

<table>
<thead>
<tr>
<th></th>
<th>FaDu</th>
<th>SKMEL-2</th>
<th>HT-29</th>
<th>HepG2</th>
<th>Jurkat</th>
<th>HeLa</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>N/A</td>
<td>N/A</td>
<td>0.62 ± 0.08</td>
<td>1.29 ± 0.11</td>
<td>8.55 ± 0.17</td>
<td>N/A</td>
</tr>
<tr>
<td>B</td>
<td>N/A</td>
<td>9.16 ± 7.09</td>
<td>N/A</td>
<td>N/A</td>
<td>5.16 ± 0.18</td>
<td>1.79 ± 0.08</td>
</tr>
<tr>
<td>8</td>
<td>4.81 ± 0.16</td>
<td>12.32 ± 0.19</td>
<td>0.51 ± 0.26</td>
<td>N/A</td>
<td>N/A</td>
<td>N/A</td>
</tr>
<tr>
<td>Dox</td>
<td>1.88 ± 0.59$^a$</td>
<td>3.78 ± 0.22</td>
<td>0.46 ± 0.07</td>
<td>1.16 ± 0.25</td>
<td>0.007$^b$</td>
<td>1.45 ± 0.05</td>
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</table>
Figure S17. 50% Inhibitory concentration plots with 8•2Cl for (a) FaDu, (b) SKMEL-2, and (c) HT-29 cancer cell lines. (d) Structural formula for 8•2Cl.
Figure S18. 50% Inhibitory concentration plots with A•2Cl for (a) HT-29, (b) HepG2, and (c) Jurkat cancer cell lines. (d) Structural formula for A•2Cl.
Figure S19. 50% Inhibitory concentration plots with B•2Cl for (a) SKMEL-2, (b) Jurkat, and (c) HeLa cancer cell lines. (d) Structural formula for B•2Cl.

S4. Dynamic Light Scattering Data

Table S3: DI water, no addatives

<table>
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<th>Diameter Intensity Measurement (nm)</th>
<th>Integrated Intensity Peak Percentage</th>
<th>Intensity FWHM (Intensity) (± nm)</th>
<th>Integrated Volume Peak Percent</th>
<th>Diameter Number Measurement (nm)</th>
<th>Integrated Number Peak Percentage</th>
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No peaks were detected when running the DI water control
Table S4: pH 7.2 Phosphate Buffer Solution (PBS), no additives

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No peaks were detected when running the Phosphate Buffer Solution control

Table S5: RPMI 1640 medium containing 5% fetal bovine serum, no additives

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<th>Integrated Volume Peak Percent</th>
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<th>Integrated Number Peak Percentage</th>
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<td>52.00</td>
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Table S6: 8•2Cl (10μL, 8mM) added to DI water

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<th>Diameter Intensity Measurement (nm)</th>
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<th>Intensity FWHM (Intensity) (± nm)</th>
<th>Integrated Volume Peak Percent</th>
<th>Diameter Number Measurement (nm)</th>
<th>Integrated Number Peak Percentage</th>
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<td>50.76</td>
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<td>0.217</td>
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<td>80.0</td>
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Table S7: 8•2Cl (20μL, 8mM) added to DI water

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<th>Intensity FWHM (Intensity) (± nm)</th>
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<tr>
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<td></td>
<td>(95.83 nm)</td>
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<td></td>
<td>50.9%</td>
<td></td>
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<td></td>
<td>(260.8 nm)</td>
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### Table S8: $\text{8•2Cl} (30\mu\text{L}, 8\text{mM})$ added to DI water

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<th>Intensity FWHM (Intensity) (± nm)</th>
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<td>102.5</td>
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<td>83.97</td>
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<tr>
<td>2</td>
<td>0.223</td>
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<td>80.96</td>
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<td>15.27</td>
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<td>73.85</td>
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### Table S9: $\text{8•2Cl} (30\mu\text{L}, 8\text{mM})$ added to DI water approximately 4 hours after addition

<table>
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<th>Diameter Intensity Measurement (nm)</th>
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<td></td>
<td>59.85</td>
<td>10.1</td>
<td>7.441</td>
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### Table S10: $\text{8•2Cl} (10\mu\text{L}, 8\text{mM})$ added to pH 7.2 Phosphate Buffer

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<td>179.1</td>
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<td>309.7</td>
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<td>86.8</td>
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### Table S11: $\text{8•2Cl} (20\mu\text{L}, 8\text{mM})$ added to pH 7.2 Phosphate Buffer

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<th>Integrated Volume Peak Percent</th>
<th>Diameter Number Measurement (nm)</th>
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<td>0.324</td>
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<td>55.2</td>
<td>235.7</td>
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Table S12: 8•2Cl (20µL, 8mM) added to pH 7.2 Phosphate Buffer approximately 4 hours after addition

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<th>Intensity FWHM (Intensity) (± nm)</th>
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Table S13: 8•2Cl (10µL, 8mM) added to RPMI 1640 medium containing 5% fetal bovine serum

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<th>Intensity FWHM (Intensity) (± nm)</th>
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<th>Diameter Number Measurement (nm)</th>
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Table S14: 8•2Cl (20µL, 8mM) added to RPMI 1640 medium containing 5% fetal bovine serum

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<th>Intensity FWHM (Intensity) (± nm)</th>
<th>Integrated Volume Peak Percent</th>
<th>Diameter Number Measurement (nm)</th>
<th>Integrated Number Peak Percentage</th>
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<td>350.8</td>
<td>76.5</td>
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<td>-</td>
<td>-</td>
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<tr>
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<td>1.342</td>
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<td>8.762</td>
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<td></td>
<td></td>
<td>45.00</td>
<td>11.6</td>
<td>10.84</td>
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<td>34.74</td>
<td>-</td>
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<td>-</td>
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<td>8.601</td>
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<td>-</td>
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<td></td>
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<td>30.59</td>
<td>12.0</td>
<td>5.770</td>
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<td>25.48</td>
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<td>1.260</td>
<td>96.3</td>
<td>7.766</td>
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</table>
### Table S15: 8•2Cl (30μL, 8mM) added to RPMI 1640 medium containing 5% fetal bovine serum

<table>
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<th>Run #</th>
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<th>Intensity FWHM (Intensity) ± nm</th>
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### Table S16: 8•2Cl (30μL, 8mM) added to RPMI 1640 medium containing 5% fetal bovine serum approximately 4 hours after addition

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<th>Intensity FWHM (Intensity) ± nm</th>
<th>Integrated Volume Peak Percent</th>
<th>Diameter Number Measurement (nm)</th>
<th>Integrated Number Peak Percentage</th>
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### Table S17: 1•2Cl (10μL, 4mM) added to DI water

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<th>Integrated Volume Peak Percent</th>
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<th>Integrated Number Peak Percentage</th>
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### Table S18: 1•2Cl (20μL, 4mM) added to DI water

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<th>Integrated Volume Peak Percent</th>
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<th>Integrated Number Peak Percentage</th>
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<td>530.9</td>
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<td>494</td>
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### Table S19: 1•2Cl (20μL, 4mM) added to DI water approximately 4 hours after addition

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<th>Intensity FWHM (Intensity) (± nm)</th>
<th>Integrated Volume Peak Percent</th>
<th>Diameter Number Measurement (nm)</th>
<th>Integrated Number Peak Percentage</th>
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### Table S20: 1•2Cl (10μL, 4mM) added to Phosphate Buffer Solution

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<th>Diameter Number Measurement (nm)</th>
<th>Integrated Number Peak Percentage</th>
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### Table S21: 1•2Cl (10μL, 4mM) added to RPMI 1640 medium containing 5% fetal bovine serum

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<th>Intensity FWHM (Intensity) (± nm)</th>
<th>Integrated Volume Peak Percent</th>
<th>Diameter Number Measurement (nm)</th>
<th>Integrated Number Peak Percentage</th>
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### Table S22: 1•2Cl (20μL, 4mM) added to RPMI 1640 medium containing 5% fetal bovine serum

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<th>Intensity FWHM (Intensity) (± nm)</th>
<th>Integrated Volume Peak Percent</th>
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Table S23: 1•2Cl (20μL, 4mM) added to RPMI 1640 medium containing 5% fetal bovine serum approximately 4 hours after addition

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Table S24: 3•2Cl (10μL, 4mM) added to DI water

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Table S25: 3•2Cl (20μL, 4mM) added to DI water

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Table S26: 3•2Cl (30μL, 4mM) added to DI water

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<th>Integrated Volume Peak Percent</th>
<th>Diameter Number Measurement (nm)</th>
<th>Integrated Number Peak Percentage</th>
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### Table S27: 3•2Cl (30μL, 4mM) added to DI water approximately 4 hours after addition

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### Table S28: 3•2Cl (10μL, 4mM) added to Phosphate Buffer Solution (80 μM)

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<th>Diameter Number Measurement (nm)</th>
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### Table S29: 3•2Cl (10μL, 4mM) added to Phosphate Buffer Solution (80 μM) approximately 4 hours after addition

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<th>Diameter Number Measurement (nm)</th>
<th>Integrated Number Peak Percentage</th>
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### Table S30: 3•2Cl (10μL, 4mM) added to RPMI 1640 medium containing 5% fetal bovine serum

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<th>Diameter Number Measurement (nm)</th>
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Table S31: 3•2Cl (20μL, 4mM) added to RPMI 1640 medium containing 5% fetal bovine serum

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<th>Integrated Volume Peak Percent</th>
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Table S32: 3•2Cl (20μL, 4mM) added to RPMI 1640 medium containing 5% fetal bovine serum approximately 4 hours after addition

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<th>Integrated Volume Peak Percent</th>
<th>Diameter Number Measurement (nm)</th>
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Table S33: 5•2Cl (10μL, 8mM) added to DI water

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No peaks observed for PEG-DAPP at this concentration in DI water

Table S34: 5•2Cl (20μL, 8mM) added to DI water

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No peaks observed for PEG-DAPP at this concentration in DI water
Table S35: 5•2Cl (30μL, 8mM) added to DI water

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No peaks observed for PEG-DAPP at this concentration in DI water

Table S36: 5•2Cl (30μL, 8mM) added to DI water approximately 4 hours after addition

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No peaks observed for PEG-DAPP at this concentration in DI water

Table S37: 5•2Cl (10μL, 8mM) added to pH 7.2 Phosphate Buffer Solution

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No peaks observed for PEG-DAPP at this concentration in PBS water

Table S38: 5•2Cl (20μL, 8mM) added to pH 7.2 Phosphate Buffer Solution

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S30
### Table S39: 5•2Cl (30μL, 8mM) added to pH 7.2 Phosphate Buffer Solution

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### Table S40: 5•2Cl (30μL, 8mM) added to pH 7.2 Phosphate Buffer Solution approximately 4 hours after addition

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<th>Diameter Number Measurement (nm)</th>
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### Table S41: 5•2Cl (10μL, 8mM) added to RPMI 1640 medium containing 5% fetal bovine serum

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Table S42: 5•2Cl (20μL, 8mM) added to RPMI 1640 medium containing 5% fetal bovine serum

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Table S43: 5•2Cl (30μL, 8mM) added to RPMI 1640 medium containing 5% fetal bovine serum

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<th>Integrated Volume Peak Percent</th>
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<th>Integrated Number Peak Percentage</th>
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Table S44: 5•2Cl (30μL, 8mM) added to RPMI 1640 medium containing 5% fetal bovine serum approximately 4 hours after addition

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<th>Integrated Intensity Peak Percentage</th>
<th>Intensity FWHM (Intensity) (± nm)</th>
<th>Integrated Volume Peak Percent</th>
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S5. References


