

***Supplementary Information***

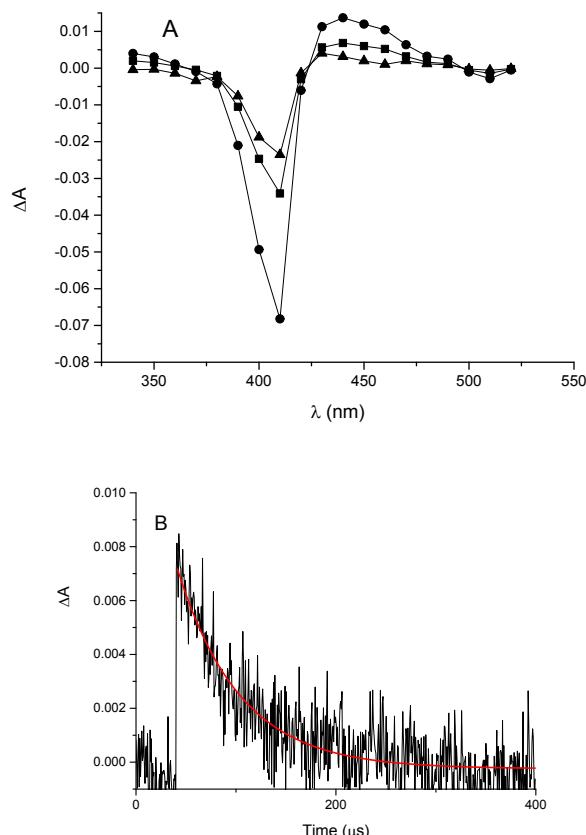
## Polymer nanoparticles with electrostatically loaded multicargo for combined cancer phototherapy

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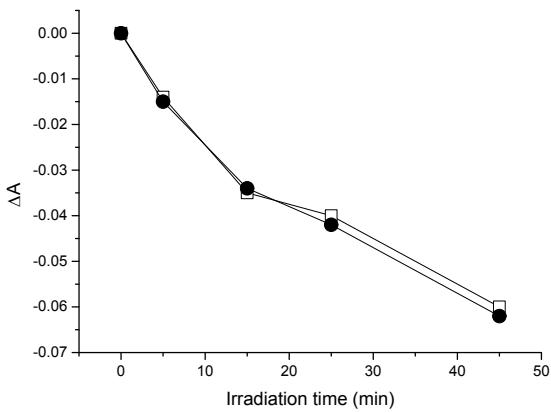
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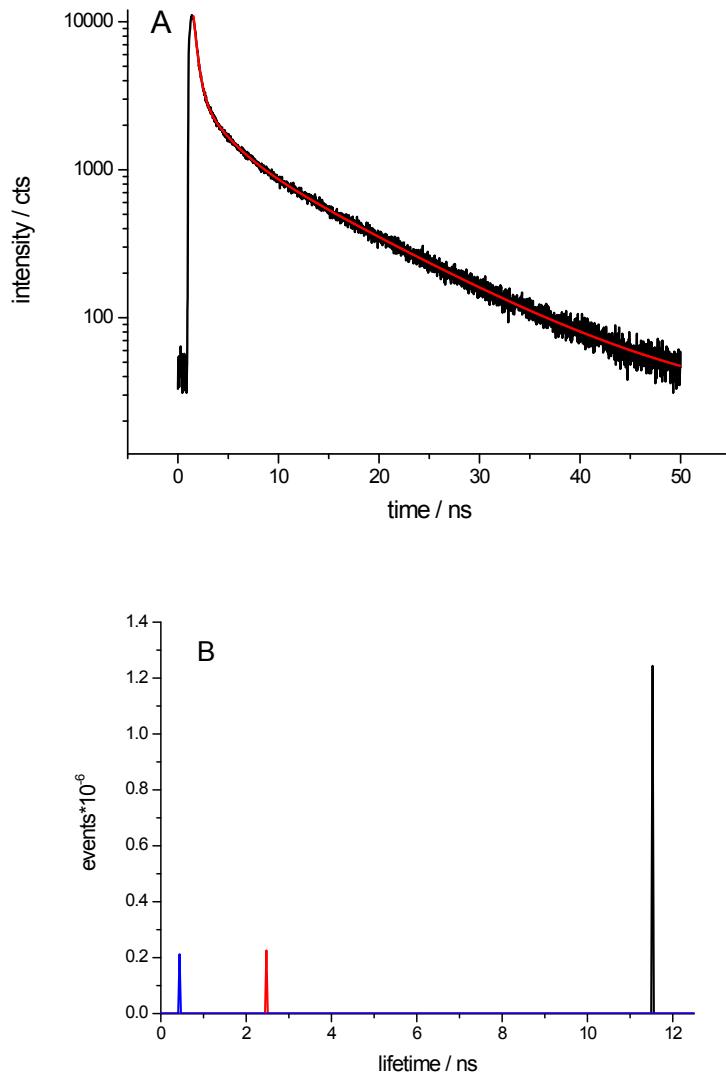
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**Fig. S1** (A) Transient absorption spectra observed 0.1  $\mu$ s ( $\infty$ ), 20  $\mu$ s ( $\circ$ ) and 80  $\mu$ s ( $\bullet$ ) and 300  $\mu$ s ( $\circ$ ) after 532 nm laser excitation ( $E_{532} \sim 10$  mJ/ pulse) of Ar-saturated aqueous dispersions of NP **1** co-loaded with **2**. (B) Decay trace monitored at 440 nm and the related first-order fitting.



**Fig. S2** Absorbance changes observed at 380 nm upon 400 nm light irradiation of NP 1 loaded with 3 (ꝝ), and 2+3 (ꝫ). The data are corrected for the different amount of photons absorbed by the NO photodonor in the two different samples.



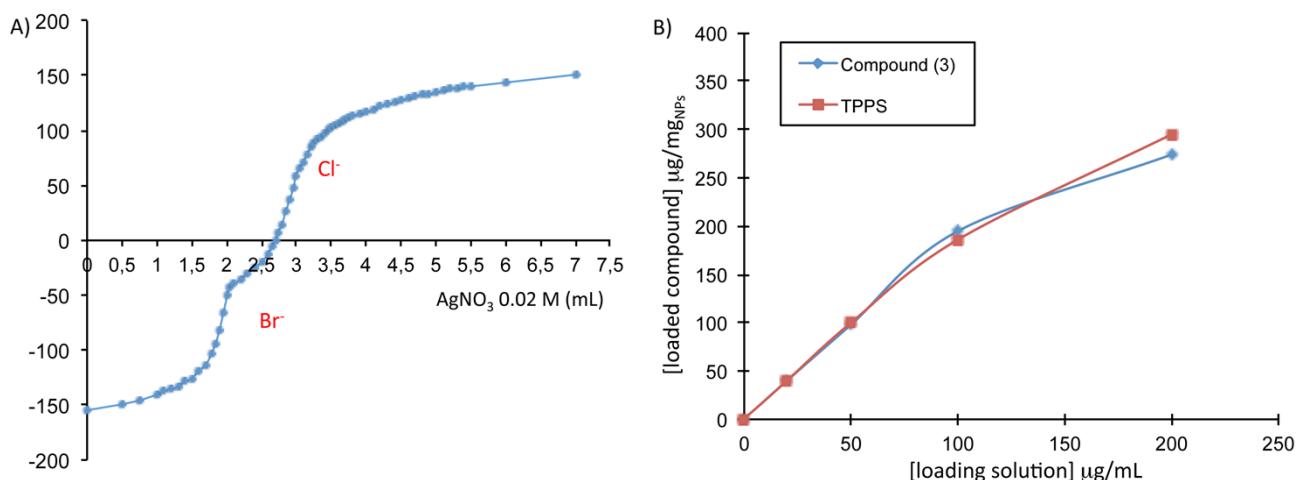
**Fig. S3** (A) Decay calculated for ROI comprising both nucleus and cytoplasm; tail-fit with a three-exponential function gave satisfactory results. (B) Histogram of the three lifetimes obtained after fitting of the selected ROI; pre-exponential factors were fitted while the lifetimes were fixed.

**Table S1.** Hydrodynamic diameter measurements by dynamic light scattering in mQ water of NPs and NPs loaded with **2 + 3** (five measurements).

Sample	Eff.Diam. (nm)	PDI	Baseline Index	Sample	Eff. Diam. (nm)	PDI	Baseline Index
NPs (1°)	102,73	0,02	8,67	NPs + <b>2 + 3</b> (1°)	298,43	0,34	0,00
NPs (2°)	107,00	0,07	4,72	NPs + <b>2 + 3</b> (1°)	292,38	0,32	0,00
NPs (3°)	107,84	0,12	8,83	NPs + <b>2 + 3</b> (1°)	280,53	0,33	0,00
NPs (4°)	106,18	0,10	8,63	NPs + <b>2 + 3</b> (1°)	276,06	0,32	0,00
NPs (5°)	103,10	0,06	0,00	NPs + <b>2 + 3</b> (1°)	276,53	0,34	0,00
Mean	105,37	0,08	6,17	Mean	284,79	0,33	0,00
Std Err	1,04	0,02	1,73	Std Err	4,51	0,00	0,00
Std Dev	2,32	0,04	3,86	Std Dev	10,08	0,01	0,00

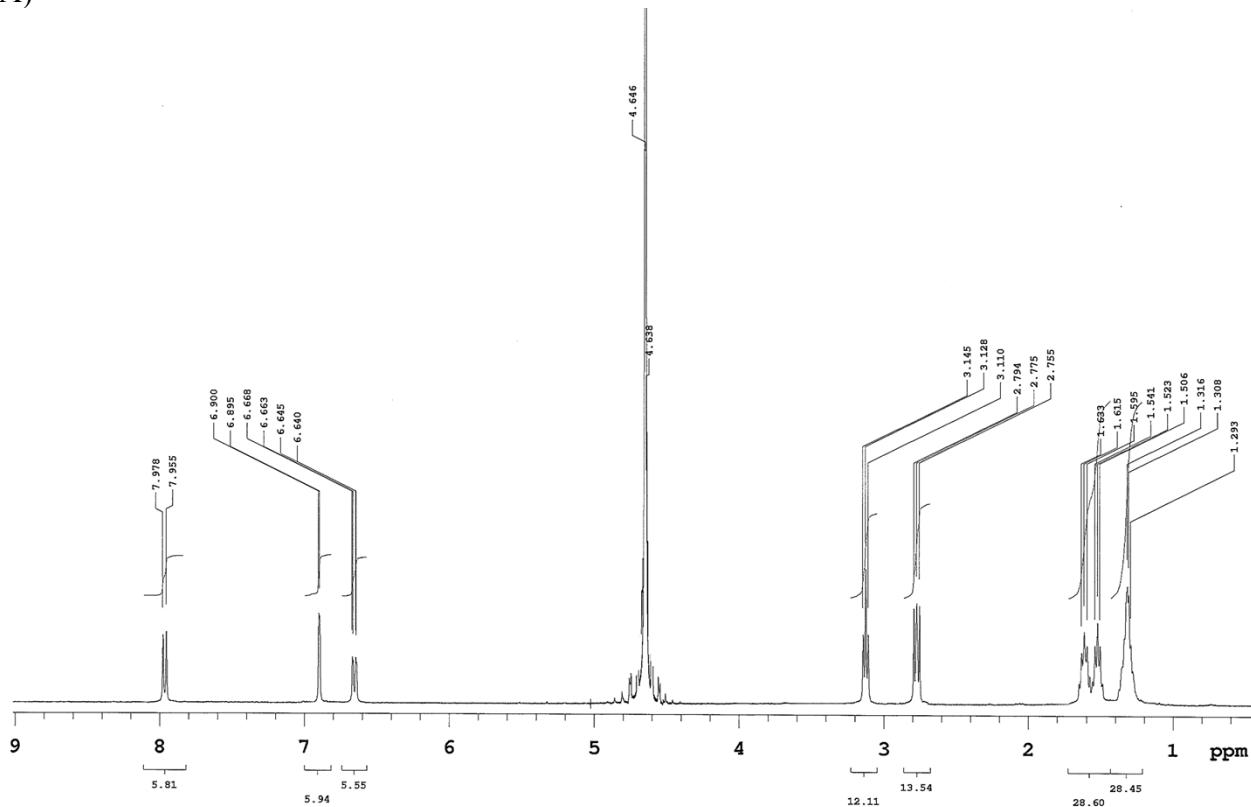
**Table S2.**  $\zeta$ -potential measurements in mQ water of NPs and NPs loaded with **2 + 3** (five measurements).

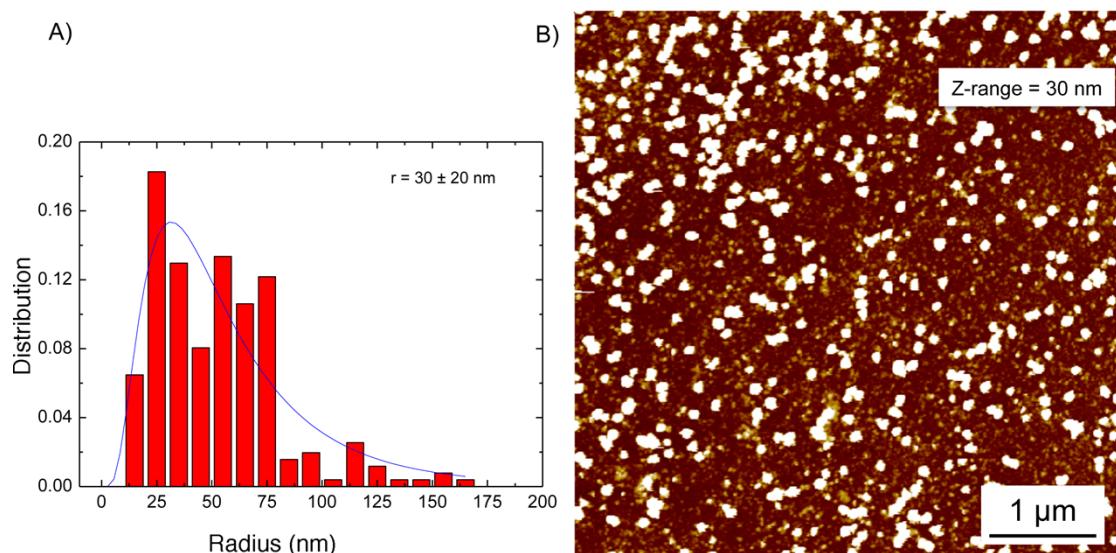
Sample	Zeta Potential (mV)	Mobility ( $\mu$ s)/(V/ cm)	RMS Residual	Sample	Zeta Potential (mV)	Mobility ( $\mu$ s)/(V/cm)	RMS Residual
NPs	58,49	4,57	2,16E-02	NPs + <b>2 + 3</b>	41,76	3,26	1,63E-02
	52,76	4,12	2,31E-02		44,95	3,51	3,17E-02
	50,23	3,92	2,74E-02		41,89	3,27	1,45E-02
	56,05	4,38	3,39E-02		40,60	3,17	1,25E-02
	53,02	4,14	2,63E-02		42,49	3,32	1,62E-02
Mean	54,11	4,23	2,65E-02	Mean	42,34	3,31	1,82E-02
Std Err	1,43	0,11	2,14E-03	Std Err	0,72	0,06	3,44E-03
Std Dev	3,20	0,25	4,79E-03	Std Dev	1,61	0,13	7,69E-03



**Fig. S4** (A) NPs titration curve and (B) loading calibration curve with TPPS and compound **3**.

A)





**Figure S6.** (A) Distribution (%) of nanoparticle radius. B) AFM image of nanospheres deposited on atomic flat silicon Z-range =  $14 \pm 5$  nm