Figure S1. $^1$H NMR Spectra for PEG-BR. The PEG-BR product was methanol extracted, dried, and re-dissolved in DMSO-d$_6$ (5 mg/mL) for $^1$H NMR spectroscopy. The peak at 2.5 ppm is due to DMSO-d$_6$, and the peak at 3.25 ppm is due to a trace amount of water. Data confirm that PEG-BR was successfully synthesized.
Figure S2. GPC Traces of PEG-BR and Its Precursors. PEG-BR and its precursors, PEG-NH₂ (Laysan Bio, 2.0 kDa) and BR (Alfa Aesar), were dissolved in HPLC-grade THF (1 mg/mL) for GPC analysis (filtered with a 450 nm PTFE filter prior to GPC).
Figure S3. GPC Traces of PEG-BR before and after UV irradiation. PEG-BR NPs (micelles) in PBS were irradiated with UV-A (0.56 J/cm$^2$ or 61.6 J/cm$^2$). PEG-BR was extracted from these solutions with DCM, dried, and re-dissolved in HPLC-grade THF (1 mg/mL) for GPC analysis (filtered with a 450 nm PTFE filter prior to GPC).
Figure S4. DLS Size Data for UV-A-Irradiated PEG-BR NPs. PEG-BR NPs (micelles) at a concentration of 0.1 mg/mL in PBS were irradiated with UV-A light (peak emission at 365 nm) at a fluence of 0.56 or 61.6 J/cm². DLS size measurements were conducted immediately after formulation, and after UV-A exposure. The size of PEG-BR micelles was not influenced by UV-A irradiation. Error bars represent standard deviation (N = 1). Note: Experiment conducted using filtered micelles.
Figure S5. Absorbances and Fluorescences of Irradiated PEG-BR NPs. PEG-BR NPs (micelles) were suspended at 0.1 mg/mL in PBS. Absorbance (left) and fluorescence (right, excitation wavelength 200 nm) measurements were performed using a quartz cuvette with 1 cm path length at 1 nm wavelength intervals. PBS was used as the blank reference for absorbance measurements.
Figure S6. Absorbances and Fluorescences of Irradiated CWO NPs. Uncoated CWO NPs were suspended at 0.1 mg/mL in PBS. Absorbance (left) and fluorescence (right, excitation wavelength 200 nm) measurements were performed using a quartz cuvette with 1 cm path length at 1 nm wavelength intervals. PBS was used as the blank reference for absorbance measurements.
Figure S7. Dot Plots from FACS Analysis. HN31 cells were seeded in T-25 cell culture flasks at a density of $6 \times 10^5$ cells/flask. Cells were incubated in a growth medium containing CWO NPs or PEG-BR/CWO NPs at a NP concentration of 0.1 mg/mL (based on CWO NP concentration) for 4 h prior to 8 Gy X-ray exposure. X-ray irradiation was performed at a dose rate of 2 Gy/min using a 320 kV X-ray irradiator. Cells were collected via trypsin treatment at 24 h post irradiation, double stained with FITC Annexin V and Ethidium Homodimer III, and analyzed on a BD LSRFortessa Cell Analyzer using the FITC and PE laser lines. (A) Signals from cellular debris were first gated out on a 2D dot plot of forward scatter (FSC) vs. side scatter (SSC) intensities (demonstrated for unstained PBS-treated unirradiated cells). (B) Cell doublets were next gated out based on size, i.e., by excluding signals with FSC intensity greater than approximately 2× median of the FSC intensity for unstained PBS-treated unirradiated cells (demonstrated for (Left) unstained PBS-treated unirradiated cells and (Right) unstained PEG-BR/CWO NP-treated unirradiated cells). This identical gating criterion was applied to all stained/unstained, CWO NP and PEG-BR/CWO NP-treated, irradiated/unirradiated subgroups. (C) On top of (A) and (B) above, non-HN31 signals were gated out based on complexity by comparing FITC vs. SSC intensity plots for stained/unstained, CWO NP and PEG-BR/CWO NP-treated, irradiated/unirradiated subgroups with the unstained PBS-treated unirradiated control (demonstrated for (Left) unstained PBS-treated unirradiated cells and (Right) unstained PEG-BR/CWO NP-treated unirradiated cells). (D) Within the resultant cell population, FITC/PE-negative and FITC/PE-positive subpopulations were identified based on the quadrant criteria established using data from unstained and single-stained PBS-treated X-ray-irradiated controls (the same quadrant criteria were applied for all subgroups). The different quadrants represent different cell states: Q1 (upper left) = early apoptosis, Q2 (upper right) = apoptosis, Q3 (lower left) = live, Q4 (lower right) = necrosis. See Figure 12 for actual population distributions. (E) The validity of this procedure was tested by examining data from unstained PEG-BR/CWO NP-treated unirradiated cells. As shown in (E), all false signals from PEG-BR which were originally present in the FITC and/or PE-positive quadrants (B and C) have been completely gated out using the above procedure.
Figure S8. Tumor Doubling Time Estimation. Tumor volume data from unirradiated mouse groups at early time points were fit to the power-law expression, $\frac{V}{V_0} = 2^{t_d/t}$, $V_0 = 100$ and 120 mm$^3$ for the 4 Gy (left; Figure 12) and 8 Gy (right; Figure 14) experiments, respectively. The resultant $t_d$ values are given in Table 1.
Figure S9. Mouse Body Weight Trends. Mouse body weights recorded as a function of time following first NP injection for mice tested in the study shown in Figures 13 and 14.
**Figure S10. Mouse Body Weight Trends.** Mouse body weights recorded as a function of time following first NP injection for mice tested in the study shown in Figures 15 and 16.