## Submission for TIC by Steve Diamond, 6/07/098

Title: Phototoxicity of Selected Nanomaterials

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Abstract: Quantification of exposure to nanomaterials is critical for assessing their environmental hazard and risk. This is an immediate issue for nano-TiO2 because it is one of more common nanomaterials now in commerce, and is difficult to analyze using common acid-digestion techniques. To address this problem, we have developed an analytical method for the quantification of nano-titanium dioxide (TiO2) in water matrices and fish tissue using a microwave digestion system with closed vessels and furnace atomic absorption (FAA). Due to the insolubility of nano-TiO2, strong acids are required for dissolution; therefore, two acid regimes were investigated in the current study, including 2:1 HF: HNO3 (v/v) and 1:1 H2SO4: HNO3 (v/v). In addition, two digestion programs were compared, including a ramped and single heating duration and temperature. With immediate sample analysis, an acid regime of 2:1 HF: HNO3 (v/v), with single combination of heating duration and temperature (35 min at 85% power) yielded the highest analyte recoveries of 93.2 to 122% based on spiked concentrations of 1.00 to 100 mg/L. This method requires shorter process times and is simpler than traditional methods. Storage of TiO2 samples for  $\geq$  24-h resulted in decreased recoveries, possibly due to binding of TiO2 to storage vessel surfaces. Followup analyses are underway to refine specific sample storage and preparation approaches that will minimize or eliminate these adsorbance losses. During FAA analysis, tailing of the titanium absorption peak was observed. In an attempt to reduce tailing, several parameters (e.g., increasing the atomization temperature and use of a fluoride modifier) were modified resulting in little to no change of absorption signals. Additional research includes optimization of FAA parameters to reduce peak tailing and the digestion of fish tissue.

**Impact Statement:** Nanotechnology is an emerging field that will produce hundreds of new and novel substances that have the potential for biological activity not typical of their bulk forms. One such material is TiO2, which also presents the added challenge of being extremely recalcitrant to common analytical dissolution methods. We present here the refinement of a digestion method for TiO2 that is efficient, repeatable, and accurate. The work will be of significant value to researchers studying TiO2 and enable analyses required for quantifying effect levels, kinetics, and other aspect of environmental hazard and risk.