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# Adapting standard aquatic and sediment toxicity tests for use with manufactured nanomaterials: Key issues, expert consensus, and recommendations

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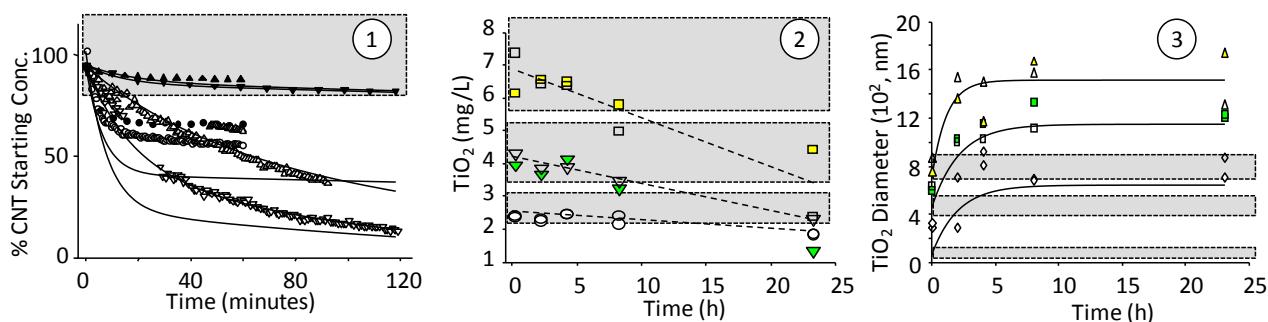
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## 1. Introduction

The rapidly accelerating development and implementation of nanotechnology has inspired vigorous debate about the adequacy of current regulatory frameworks for assuring the safe deployment of manufactured nanomaterials (MNs) in the commercial marketplace (e.g. Beaudrie et al., 2013; Bowman and Hodge, 2007). A critical aspect of these debates is whether standard test protocols currently used in risk assessment are fully adequate for testing the hazard potential of MNs. Standardized testing protocols, and the guidance documents that describe them, are a critical component of risk assessment and for regulatory processes for chemical substances. They describe and prescribe specific techniques and methods for the collection and analyses of data that quantitatively predict, under controlled laboratory conditions, the release, fate, transport, transformation, exposure, and toxicity of chemical substances. The Organization for Economic Cooperation and Development (OECD) has promulgated many test guidelines (TGs) that are used for these purposes. The subset of these TGs that focus on toxicity in aquatic, sediment and soil organisms is the OECD's Section 2: Effects on Biotic Systems (OECD, 2014). These TGs also provide for modification of methods and approaches for testing problematic substances, e.g. those that are difficult to maintain in solution. However, while these TGs are generally adequate for testing MNs they will require additional guidance to address several MN-specific issues that preclude achieving consistent and repeatable results (e.g., Handy et al., 2012; Kühnel and Nickel, 2014; Diamond, 2009).

The identified gaps in TGs generally derive from the particulate or fibrous nature of MNs. MN exposures typically involve colloids or particle-sediment mixtures rather than dissolved chemicals. As exemplified in Fig. 1, MNs in test media typically undergo extensive agglomeration, settling, particle dissolution, and transformations during exposure and media renewal periods (Handy et al., 2012; Lowry et al., 2012) giving rise to unique challenges for exposure-response estimation, and suggesting the need for exposure metrics based on particle number or surface area, in addition to mass concentration.



**Figure 1. Examples of changes in nanoparticle stability: 1, loss of carbon nanotube (-OH, -COOH, and unfunctionalized) concentration with and without dissolved organic carbon (Kennedy et al. 2008); 2, loss of TiO<sub>2</sub> concentration (Ma et al 2010); 3, agglomeration of TiO<sub>2</sub> (Ma et al 2010). Shaded regions indicate +/- 20% change from initial values.**

## 2. Results and discussion

In this presentation we discuss the findings from a work group meeting held in Washington D.C. in July, 2014. This meeting (part of ongoing efforts of the OECD's Working Party on Manufactured Materials) was attended by 23 experts from 6 countries. The topics discussed focused on key limitations of current aquatic bioassay study designs for testing MNs and the knowledge gaps that preclude or hinder the use and usability of these broadly-applicable standard aquatic toxicity tests for MNs. Specific issues included:

- The state of knowledge concerning MN behaviours in test systems; e.g. agglomeration, settling, dissolution, material modifications (e.g. nano-silver sulfidation, DOC coatings), and the variability and heterogeneity in these behaviours, and their relevance for hazard assessment.
- Current limitations of methods and approaches for measuring and monitoring MN characteristics during ecotoxicity assays.
- How to address MN losses, and exposure variability that will likely exceed the +/- 20% concentration threshold identified in current test guidelines; where it is suggested that exceedance of this threshold be addressed with increased analyses, more frequent or continuous renewal of media, use of dispersants or stabilizers, etc. Also, whether this (or an alternate) threshold for exposure variability should also include characteristics other than mass concentration (Fig. 1).
- Whether testing of very unstable MNs should include water-column assays, or focus primarily on sediment testing.
- Assay procedures including preparation of stock media, methods for preparing dilution series, mixing of MNs in sediments, the potential use of settling periods, an accommodated fraction approach, or elutriate testing.

## 3. Conclusions

The ultimate goal of this workgroup meeting is the drafting of an OECD Guidance Document on aquatic and sediment hazard testing of MNs. As this work is ongoing, further resolution of the testing issues described above and development of draft guidance will be discussed. Potential research focused on information needs are also identified. Guidance on the difficult issues faced by regulatory testing laboratories will help reduce uncertainties in assessing the environmental risk of developing nanotechnologies. Standardized guidance will also foster the generation of consistent hazard information that can eventually lead to better predictive tools. This benefit extends to non-regulatory, exploratory testing as well.

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