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Title: Metrological compatibility of nanoparticle size analysis

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Abstract: There is interest within the measurement community to establish a metrological basis for a relevant concept of 'size' for nanoparticles [1-3]. Such efforts are nontrivial because there is no single definition of a size measurand for particle diameter that is entirely satisfactory for all descriptions, situations, and applications of nanoparticles. The acknowledged need to report various instrumental and measurement conditions under which size information is obtained leads to questions of methods divergence and is therefore an integral part of any particle size analysis standard, including those of the International Standards Organization (ISO).

This presentation outlines an ISO-compatible framework for the investigation of methods divergence in the case of nanoparticles. This approach is based upon work which has already appeared in the literature, together with a few novel aspects, which we argue, may be realized within the context of nanoparticle size analysis. First, an *intra-method* analysis is implemented according to the work of [4] in which a model equation, various submodels for influence quantities, and uncertainty estimates for each measurement process are expressed in a consistent fashion. A concise representation of the entire measurement process is clearly a desirable starting point when attempting to compare methods. Next an *inter-method* analysis based on the principle of metrological compatibility is adopted from [5]. Metrological compatibility is defined in VIM3 [6] and serves as the basis for the definition of a pairwise comparison metric for measurement results. Results obtained using this approach will be compared with those from methods based on other statistical criteria [7-9] with the goal of identifying the methods best suited for nanoparticle applications.

In this work, we generalize the notion of metrological compatibility and analogous criteria, often derived using pairwise comparisons [5], to reflect the fact that even certified nanoparticle reference materials are inherently distributions of particle size (as well as other properties). To do so, data from a series of well-characterized reference materials are analyzed collectively in order to map the full functional relationship of the model equation. Functional measurement results are, in a sense, no longer related to a specific sample, though these data remain referenced in a linear approximation to an ideal, purely size dependent description. This starting point allows us to identify deviations from ideality arising from other sources, such as higher moments of the distribution, particle shape variations, and heterogeneous composition. In this generalized form *inter-method* analysis, coupled with expert, or scientific, judgment,

is used to revise individual, *intra-method* uncertainty estimates based upon the correlated, combined results.

Selected results for ensemble and individual particle size measurements from an ongoing interlaboratory comparison of a four-member series of reference polystyrene nanoparticles will be used to demonstrate a detailed application of this approach. Measurement protocols and materials requirements that must be established in order to carry out the analysis will be discussed in the talk.

References:

[1] K. Ehara and H. Sakurai, Metrologia **47** S83 (2010).
[2] T. P. Linsinger, G. Roebben, C. Solans, and R. Ramsch, Trends in Anal. Chem. **30** 18 (2011).
[3] F. Meli et al., Measurement Sci. Technol. **23** 125005 (2012).
[4] K.D. Sommer and B.R.L. Siebert, Metrologia **43** S200 (2006).
[5] R.N. Kacker, R. Kessel, and K. D. Sommer, J. Res. NIST **115** 453 (2010); R. Kessel, R.N. Kacker, and K.D. Sommer, J. Res. NIST **116** 809 (2011).
[6] JCGM 200:2008(E/F); Section 2.47.
[7] A.L. Rukhin, Metrologia, **46** 323 (2009).
[8] A. Possolo and B. Toman, Metrologia **44** 464 (2007).
[9] B. Toman and A. Possolo, Accredit. Qual. Assur. **14** 553 (2009); B. Toman and A. Possolo, Accredit. Qual. Assur. **15** 653 (2010).