List of Useful ISO Standards relating to nanoparticle definitions and Characterisation

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NB: Some of the documents are more mature than others. None of the documents are specific to magnetic nanoparticles at present. All of the ISO documents are subject updates and ammendments as as the relevent fields develop. All new documents should build on, or acknowledge and correct, the existing literature. I would be very interested to receive any feedback that the experts at this COST meeting might have on any of the listed documents. For information on magnetic charactisation of nanoparticles please see the final section 1.1.8.

Definitions, Characteristics and Measurements Relating to MNPs

1.1.1 General

Technical Specification PD CEN ISO/TS 17200:2015 – Nanotechnology – nanoparticles in powder form – characteristics and measurements Lists fundamental characteristics which are commonly determined for nanoparticles in dry powder form. The technical specification prescribes specific measurement methods for each of these characteristics. The following sections are the most relevant:

Section 5: Sample preparation defines a set of fundamental characteristics of nanoparticle powders as well as the units of measurement and acceptable measurement methods. It contains some basic thoughts on appropriate sample preparation, and references ISO 14488 for detailed guidelines on this topic (see sampling section).

Section 6.1 Provides a list of techniques for analysing the chemical composition depending on the particle type.
Section 6.2 References standards EN13925-1:2003 and JIS0131:1996 for guidelines on the correct protocol for analysing crystal structure and average crystallite size using XRD.
Section 6.5 Provides guidelines for measuring the average size and standard deviation of particles using TEM. It references ISO13322-1 for guidelines on image processing techniques

ISO 9276: Representation of results of particle size analysis – This multipart standard contains a variety of advice in analysis techniques and presentation of experimental data resulting from particle size analysis. Of particular interest to the NanoMag work are:

Part 2: Calculation of average particle sizes/diameters and moments from particle size distributions
Part 3: Adjustment of an experimental curve to a reference model
Part 6: Descriptive and quantitative representation of particle shape and morphology

PD ISO/TR 13097:2013(E) – Guidelines for the characterisation of dispersion stability – This Technical Report (ISO Document class) addresses the characterisation of the stability of liquid dispersions by monitoring the change in one or more system parameters over time. It covers both the real-time

measurement of solutions, and the use of modelling to make predictions based on the measurement of related physical parameters. In the case of extremely stable solutions it considers the possibility of accelerating the aging process in order to reduce the necessary measurement time (mechanical, thermal and physic-chemical procedures). The content of this document should be considered during standardisation development relating to those measurements involving the stability of MNP suspensions.

ISO 26824:2013 Particle characterisation of particulate systems – vocabulary is a published standard providing a dictionary of agreed definitions relating to particle size analysis. It contains sections defining terms which are of direct relevance to nanoparticle standardisation including

Section 4: Representation of particle shape analysis
Section 7: Dynamic light scattering
Section 10: Small angle X-ray scattering method
Section 16: Characterisation of particle dispersions in liquids
Section 17: Methods for zeta potential determination

Any future standardisation work should either conform to the definitions contained within this standard, or else provide explanations of amendments to the exisiting literature.

1.1.2 X-ray diffraction

EN 13925-1:2003 (E) - Non-destructive testing — X-ray diffraction from polycrystalline and amorphous materials

This European Standard specifies the general principles of X-ray diffraction from polycrystalline and amorphous materials. This is known by the term "X-ray Powder Diffraction (XRPD)", and is now applied to powders, bulk materials, thin film, and others. As the method can be used for various types of materials and to obtain a large variety of information, this standard reviews a large number of types of analysis but remains non-exhaustive.

At present we are not aware of any standardisation documents which relate to neutron scattering analysis of nanoparticles or powders.

1.1.3 Small angle X-ray scattering

ISO 17867:2015 – Particle size analysis – Small-angle X-ray scattering – Is a published ISO standard on the use of SAXS for the analysis of particles in the size range 1-100nm suspended in a solvent. It covers the measurement principles, common apparatus, sample preparation, measurement procedure and data processing. The document must be taken into consideration in any NanoMag standardisation work.

DD ISO/TS 13762:2001-Particle size analysis – Small angle X-ray scattering method – Is a technical specification pertaining to the use of SAXS in the analysis of particle powders and suspensions with particle sizes in the range 1-300 nm. The technical specification describes its purpose as being to **facilitate the comparison of size analysis made in different laboratories.** The specification is only applicable to particles that can be regarded as isotropic and spherically shaped.

1.1.4 Electron Microscopy (SEM/TEM)

ISO 13322-1 - Particle size analysis — Image analysis methods - Part 1: Static image analysis methods – This standard is referenced for TEM analysis of nanoparticle sizes in PD CEN ISO/TS 17200:2015. It contains guidelines on digital imaging of static nanoparticles using SEM/TEM techniques. Stationary imaging of immobilised nanoparticles is conducted within the NanoMag project. The standard again references ISO 14488 for sampling guidelines (see sampling section). It does not cover data analysis in much detail.

1.1.5 Dynamic Light Scattering (1.1.6 also relevant to this)

ISO 13321:1996 –Methods for determination of particle size distribution – Part 8: Photon correlation spectroscopy – describes the application of the photon correlation spectroscopy technique to provide a measurement of the average particle size of particles dispersed in liquid as well as a measure of the broadness of the size distribution. It is applicable to particle sizes from a few nanometers to about 1 µm. It provides a procedure for data analysis based on the assumptions that the particles are isotropic and spherically shaped. The techniques described in ISO 13321 are limited to low particle concentrations as the methods described contain no compensations to account for multiple scattering events in more concentrated solutions.

ISO 22412:2008 – Particle size analysis – Dynamic light scattering - Elaborates upon the methodology described within ISO 13321 and describes a standard method for measuring the size and size distribution of sub-micrometer nanoparticle solutions across a broad concentration range from the dilute to concentrated.

1.1.6 Zeta potential (Using DLS)

ISO 13099-2:2012 Colloidal systems – Methods for zetapotential determination Part 2: Optical methods – Specifies two methods of measurement of electrophoretic mobility of particles suspended in a liquid: video microscopy and electrophoretic light scattering (based on same principles as DLS). The surface charge and zeta-potential can be calculated from these measurements. ISO13099-1 contains the standardised theoretical models for conducting this analysis.

ISO13099-1:2012 Colloidal systems —Methods for zeta-potential determination Part 1: Electroacoustic and electrokinetic Phenomena - When calculating zeta-potential from measured data there are a number of possible models depending on the type of dispersion used and measurement conditions. There is no single model that is valid for all real systems. Theories are divided into two groups: elementary theories and advanced theories. This standard provides a framework for both.

1.1.7 Inductively Coupled Plasma Mass Spectrometry

ISO 17294-1:2006 Water quality – Application of inductively coupled plasma mass spectrometry – Part 1:General Guidelines Specifies the essential principle of ICP-MS, the necessary apparatus, and provides general directions for determination of elements in water. It contains some discussion of the likely interference in readings which may occur and describes some of the likely causes.

ISO 17294-2 Water quality – Application of inductively coupled plasma mass spectrometry – Part 2: Determination of 62 elements – Specifies a method for the determination of 62 elements within water with threshold concentrations for detection ranging between 0.1 µg/l a 1 µg/l depending on the

element. It details a large number of potential interference effects due to isobars, doubly charged ions and polyatomic ions that can skew results. It cites ISO17294-1 – 6.2 for a detailed discussion of non-spectral interference effects which may require consideration. The standard specifies the criteria required for the reagents necessary for measurements, apparatus, sampling techniques (ISO 5667), sample digestion methods, measurement procedures and calibration requirements.

Determination of iron (oxide) concentration, which is the most relevant MNP core material, is affected by interference with ArO^+ as described in *ISO 17294-1:2006.* It is therefore not included among the 62 elements in *ISO 17294-2.* Nevertheless, modern ICP-MS instruments operating in medium- or high-resolution modes can easily discriminate Fe^+ from ArO^+. Therefore these standards can be used in the context of MNP characterization without essential modifications.

ISO 11885:2007 - Water quality — Determination of selected elements by inductively coupled plasma optical emission spectrometry (ICP-OES) – Published standard. Specifies a method for determining the dissolved elements, elements bound to particles and total content of elements in a water sample. Techniques for detecting aluminium, antimony, arsenic, barium, beryllium, bismuth, boron, cadmium, calcium, chromium, cobalt, copper, gallium, indium, iron, lead, lithium, magnesium, manganese, molybdenum, nickel, phosphorus, potassium, selenium, silicon, silver, sodium, strontium, sulfur, tin, titanium, tungsten, vanadium, zinc and zirconium. Taking into account the specific and additionally occurring interferences, these elements can also be digests of water, sludges and sediments (for example, digests of water as specified in ISO 15587-1 or ISO 15587-2).). The method is suitable for mass concentrations of particulate matter in waste water below 2 g/l.

ISO/TS 13278:2011 Nanotechnologies -- Determination of elemental impurities in samples of carbon nanotubes using inductively coupled plasma mass spectrometry. This standard might be relevant when using ICP-MS to determine elemental impurities in samples of MNPs.

ISO/DTS 19590:draft Nanoparticles — Detection and sizing of nanoparticles in aqueous media via single particle inductively coupled plasma mass spectrometry. When approved, this standard has a potential to be used in determination of MNP core size and concentration in addition to iron content measurements.

1.1.8 Magnetic Measurements of MNPs

At present no standards exist for measurements that contribute to the magnetic characterisation of MNP solutions. This applies to both DC and AC based techniques. This lack of documentation within the field indicates the timely creation of the NanoMag consortium and emphasises the importance of the work within this emerging industry. The standardisation of the definitions and measurement of these characteristics will be a key area for development in the future. An early-stage material specification ISO/TS 19807 “Liquid suspensions of magnetic nanoparticles” is currently being developed by ISO, with myself and Quentin Pankhurst providing the UK contribution to the drafting process through the British Standards Institute.