

Dispersion protocol for nanoparticle suspension preparation by cup horn sonication

Date

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Version

1.1 English

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1 Scope

This Standard Operating Procedure (SOP) describes the nanoparticle suspension preparation within the nanOxiMet project by using indirect probe sonication with a Cup Horn.

2 Basics

Bringing nanoparticles into suspension is always a challenge and several different protocols are currently used in different scientific studies. However, a dispersion procedure often has to be adapted to the specific material, experiments especially if several, parallel investigations are planned. Consequently, there is not a unique procedure to be applied, only a compromise of the minimal common base, acceptable for the different investigations.

The aim of this Standard Operating Procedure is the description of the in nanOxiMet applied suspension procedure. The procedure was tested prior on a P25 titanium dioxide suspension and revealed an appropriate stability of a suspension for at least 5 min constant particle size distribution and zeta potential (a variance of the z-average 10 % is accepted). This procedure was applied for almost all further materials (not for hydrophobic particles) irrespectively their "ideal" dispersion by this procedures (e. g. high agglomerate status is accepted).

In the follow the SOP describes suitable steps for preparing a nanoparticle suspension in this project.

Stability criteria are e.g.

- Optical observation (no visible sedimentation of the particles)
- Size of the particles in the suspension
- Zeta potential

3 Materials & Instruments

3.1 Materials

The following materials and chemicals are required:

- HPLC Grade water (CAS 7732-18-5)
- Nanomaterial (solid/powder)
- Clean Spatula
- Pipette

3.2 Instruments

Comparable equipment as the mentioned instrument is required:

- Ultrasonication equipment (Bandelin Sonopuls HD2200 ultrasonic homogenizer 200 Watt, Bandelin Cup Horn BB6)

Note: The usage and maintenance of the instruments will be not described in this SOP. Please refer to the manual.

4 Experimental procedure

4.1 Suspension preparation

For preparing the nanomaterial suspension HPLC Gradient Grade Water is used – CAS 7732-18-5

- a defined amount of the nanomaterial – here ~20 mg – is weighed in a 50 ml plastic centrifuge vial (a variance of 1% is accepted)
- each of the centrifuge vial is filled up to 20 ml (concentration 1 mg/mL) with HPLC Water
- the centrifuge vial is placed in the middle of the Cup Horn. The bottom of the centrifuge vial is placed 1 cm above the sonication unit (s. Figure 1)
- the Cup Horn is filled with 230 mL deionised water
- the suspension is sonicated in the Cup Horn for 10 minutes in sum (2 min effective), with a pulsation pause ratio of 0.2/0.8 (Bandeline Sonoplus HD 2200 or comparable instruments) (Efficient energy input 38.9 W based on calorimetry, Taurozzi et al. 2010)
- for sonication the vial with the suspension is cooled with cold/ice water in the Cup Horn to minimize the heating of the suspension during the sonication

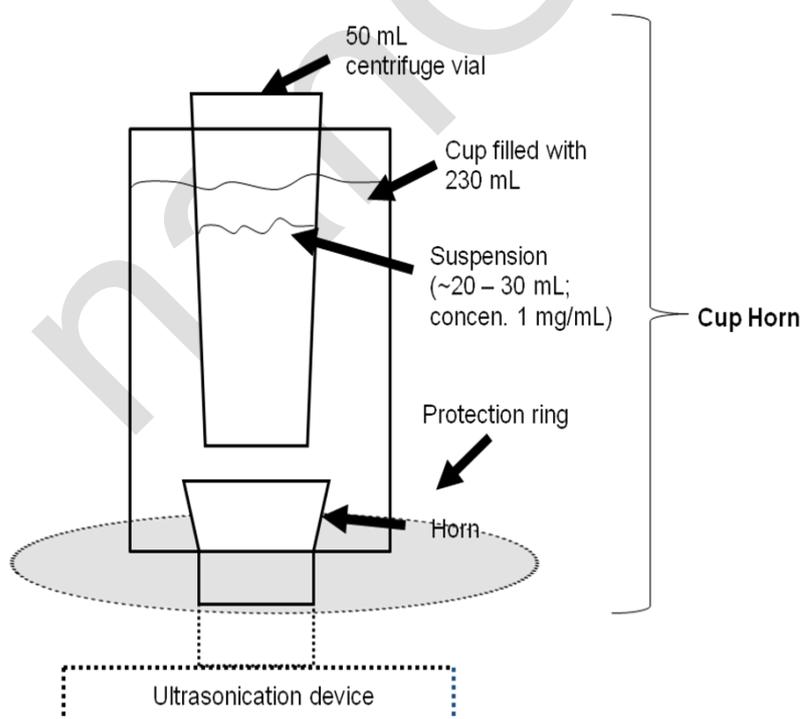


Figure 1. Scheme of the experimental setup (here for Bandelin Cup Horn BB6)

5 Safety precautions

Please follow the safety information and regulations of the working laboratory as well of the materials provider. In general handle with care, wear protective clothing and suitable gloves at any time and labelling the material.

6 Waste disposal

Please follow the disposal advice of the material provider, if available.

nanOxiMet

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Responsible for the implementation of this SOP: Measurement personal

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