Development of OECD Test Guidelines and Guidance documents: Dustiness of Manufactured Nanomaterials

ATEX, non-HARN, HARN and exposure models

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December 16, 2020

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This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No 885931

BACKGROUND – THE OECD PROPOSAL
Dustiness of Manufactured Nanomaterials

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Gov4Nano meeting the needs of nanotechnology

✓ Dustiness = a key parameter

- Relevant in assessing the risk assessment of exposure
- Potentially useful to support the identification of hazardous scenarios such as dispersion, fire, explosion

Determination of the dustiness of MNs (1 TG)

i) Non-HARNs and HARNs

and its subsequent use of dustiness data (2 GDs) for

i) exposure modelling, and

ii) to improve ATEX safety approaches

2 EU Projects:

- Gov4Nano (non-HARN NP)
- NanoHarmony (HARN and highly reactive NP)

Scientific basis for Dustiness testing of ENMs (1 new TG) & (2 new GDs)

Nanoobjects to be tested:
SiO_2: NM 203, NM204, 244 FP
TiO_2: PC 105 (MSSA) anatase, NM 103, NM105, 244 FP
+ other HARNs (TBD) & reactive materials

Support the current and foreseeable future regulatory needs
(ex: Reg 2020/878, Reg 2018/1881, Dir 99/92- 2014/34)

April 2022: First draft TG at WNT
April 2023: First Draft GD at WNT

(*) CEN/TC 137 (TC 137 WI 061)

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DUSTINESS OF NANOMATERIALS: FLOW OF TESTING PLAN AND USE

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nanoPowder Phys-chem Characteristics → Dust generation → Dust Measurements (TG) → Use of Measurements (GD)

Dust generation
Rotating drum method (RD)
Small rotating drum (SRD)
Vortex shaker method (VS)
Venturi dustiness testing device
Fluidizer
Continuous drop (CD)-Palas Dust View II
Continuous drop method (CD)

- Respirable Dustiness mass (mg/kg)
- Respirable Dustiness number (1/mg)
- Respirable Number emission rate (1/mg.s)
- Number based initial dustiness kinetics (1/mg.s^2)
- Time-based dustiness kinetics: time required to generate 50 % of the total number of particles released (s)
- Time-averaged number-based aerodynamic particle size distribution as dN/dlogD_p (number of modes and first/second modal aerodynamic equivalent diameters; µm)
- Morphology (L, d, for fibers object counting - optional to non-HARN)

Occupational Exposure modelling:
- Predicted exposure level comparisons to OEL for various exposure scenarios

ATEX (explosion risk):
- Workplace aerosol Mass concentration < LEL?
- Improve ATEX safety procedures

- Test Applicability of generated dustiness data to predict exposure levels
- Compare modeled versus real exposure scenarios data

Non-HARN Aerosol Emission

HARN Aerosol Emission

- Intra validation (robustness, accuracy)
- Inter lab (assessment of variability between labs) on similar methods

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7 METHODS TO BE CONSIDERED FOR THE TG

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Methods	Procedure/standard used	Number of participants involved in the testing
Rotating drum method (RD)	EN 17199-2:2019 (& EN15051-2:2006)	**6** (**TNO**, IOM, ITC- Universitat Jaume I, INRS, NIOSH/CDC, and BIAC/CHEMOURS)
Small rotating drum (SRD)	EN 17199-4:2019	**5** (**NRCWE**, BASF, KRISS, INRS, and CIOP-PIB)
Vortex shaker method (VS)	EN 17199-5:2019	**6** (**INRS**, KRISS, CEA, KTH, LNE, INERIS)
Venturi dustiness testing device	Boundy et. al. (Ann Occup Hyg. 2006 Jul; 50(5):453-8)	**3** (**NIOSH/CDC**, IPT- University of Wuppertal, and KTH)
Fluidizer	Dirk Broßell et al., Powder Technology. Volume 342, 2019, Pages 491-508	**3** (**BAuA**, NRCWE, and CYPRUS Institute)
Continuous drop (CD)-Palas Dust View II	Palas	**1** **BAM**
Continuous drop method (CD)	EN 17199-3:2019	**2** (**TNO**, ITC- Universitat Jaume I)

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RELEVANT ITEMS DISCUSSED ON DUSTINESS PROGRAMME

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1. Do you consider that all methods have potential to support regulatory information requirements for dustiness testing?

Comparability is key!

Yes but only on the already standardized methods.

Yes - but only if the test results can actually be used in exposure modelling.

Clear guidelines for which method to use for which scenario and caveats will be needed.

Methods requiring small amounts of materials are advantageous for using them in a safety-by design approach

Yes. Each of the methods should be matched and recommended to represent/mimic a certain set of activities.

2. Do you think this dustiness testing programme will provide enough generated data and knowledge to establish a method-specific ranking scheme for NMs?

There might be limitations but that it is a good starting point as 7 methods will deliver data on six different materials.

dustiness thresholds - It was expressed that errors need to be taken into account when assigning powders to a specific **band of dustiness level**. Qualitative assessment will need a very careful description in the TG

Hopefully the data can be added to the previous generated data

Not completely but at least we can see some general tendencies or converging results to derive method-specific ranking schemes.

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RELEVANT ITEMS DISCUSSED ON DUSTINESS PROGRAMME

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3. Do you consider that method-specific ranking schemes will be useful for down-stream users to assess ATEX risk and/or exposure assessment?

Certainly yes!

for ATEX risk, many additional factors are required.

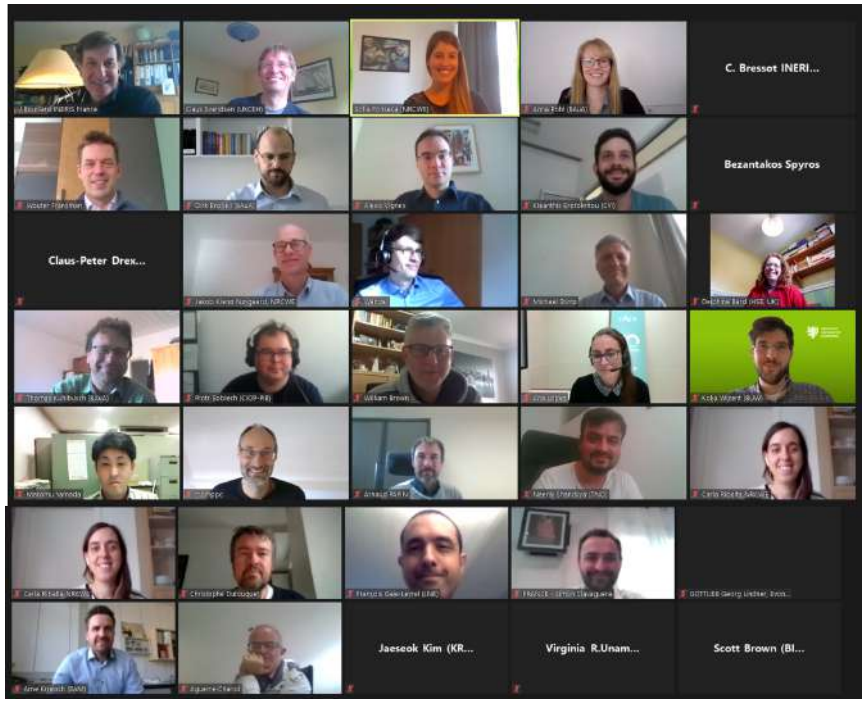
The handling activity in the respective scenario should be associated to the method.

For ATEX it will give an insight on the persistence of the cloud and the propagation and thereby can help to assess the explosibility.

For exposure assessment: yes, if the aerosol generation is adequately simulating the occupational activity.

4. Should the mandatory measurands be harmonized and determined for all the methods to the extent possible?

Consensus was found for the common template already built and that the measurands should be harmonized within the limitations of the different methods and that the standards will be followed wherever possible.



DUSTINESS TESTING OF HIGH ASPECT RATIO NANOMATERIALS (HARN)

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What is the most important measurand regarding HARN dustiness?

- Fibre number concentration
- EM analysis in combination with particle counting
- Distinguishing fibres from other particles

What is your viewpoint towards aerosol instrumentation to determine measurands for HARN dustiness testing?

- Use of electron microscopy for determination of dustiness measurands and morphological characterization
- ELPI instruments (counting of objects plus morphological characterisation of collected samples using EM possible)
- Particles counter instruments as complementary technique, however no distinction between spherical and fibrous objects possible

Is it viable to formulate an electron microscopy aerosol sample analysis procedure applicable for both SEM and TEM?

- Different sampling procedures and sample preparations for SEM and TEM
- Image analysis software plus detailed description for image processing and counting rules required

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RELEVANT ITEMS DISCUSSED ON DUSTINESS PROGRAMME – ATEX ISSUES

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1. Do you agree to add some ATEX safety provisions in the TG (caveat) with an accompanying flowchart to determine the reactive materials ?

Yes, but TG should remain consistent with already EU existing standards (EN 17199: 1-5) and contradiction should be avoided

Include a caveat in the TG: occupational, including ATEX risks, should be assessed according to national regulations – Details of ATEX risks assessment are reported in the associated GD

The GD on dustiness & ATEX will accompany the TG and give additional information when dealing with reactive materials through the proposed flowchart.

2. Use of dustiness to support the determination of ATEX area

Challenges for GD ATEX are similar to GD on exposure modelling: importance of the characterisation of the source term but focused on the total amount of airborne particles / initial powder mass in the system (as well as the dustiness kinetics = particle flowrate into the air). Possibility to determine near field concentration in relation to the critical concentration for explosibility (LEL) in a defined control volume.

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DUSTINESS TESTING OF HIGH ASPECT RATIO NANOMATERIALS (HARN)

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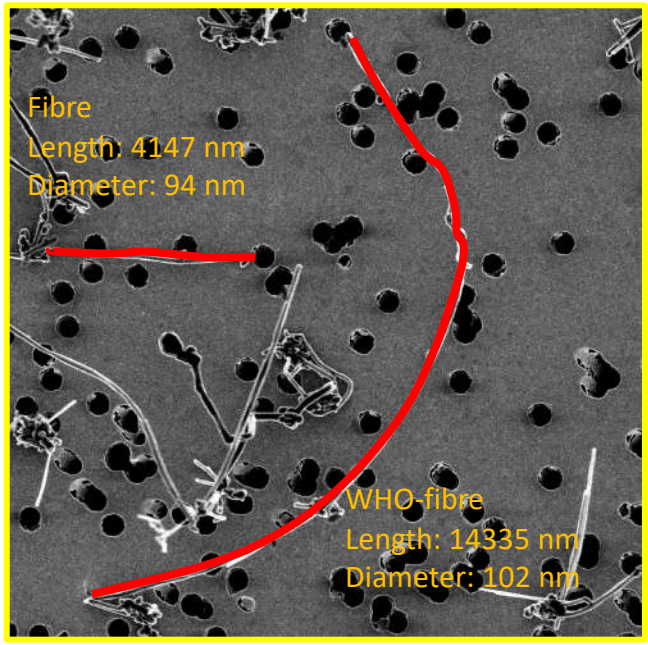
Consent approach

- Morphological characterization of aerosol samples from dustiness tests
- Counting rules and classification parameters
- Extrapolation of results taking into account the relative error
- Consensus found on importance of software image analysis tools and harmonized counting rules



Official call for contribution in an intra- and inter-laboratory comparison of dustiness test methods for HARN

Inter- and Intra-laboratory comparison test of dustiness testing of HARN including morphology characterization using electron microscopy (April – September 2021)

We are looking for interested partner laboratories, using dustiness test methods plus morphology analysis via electron microscopy to participate in the testing programme.

Contact jacques.bouillard@ineris.fr and pohl.anna@baua.bund.de

OECD
BETTER POLICIES FOR BETTER LIVES

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USE OF DUSTINESS DATA FOR OCCUPATIONAL EXPOSURE MODELLING

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BACKGROUND

$$S_c = DI_m \cdot H \cdot \frac{dM}{dt} \cdot LC$$

- Emission source: critical parameter for exposure modelling
- To use dustiness index (DI) for emission source (S_C) characterization it needs to be "scaled" (H) for different activities and dustiness methods

GOAL

- Improve source characterization by refining H and adding dustiness kinetics for at least pouring processes and two dustiness methods (SRD and CD)
- Testing of improved emission source term in mass-balance models

OUTCOME

- Improved understanding of dustiness link to emission source and its use for exposure modelling
- First step to expand to other processes and dustiness methods

RESEARCH NEEDS:

- **High quality measurement data for model testing is needed**
- **Harmonized libraries with descriptors for:** i) Emission sources (process & material), and ii) dustiness

DUSTINESS TESTING

1 TG: generation of dustiness data and method-specific dustiness ranking schemes for future regulatory risk management classification of powders

1 GD : use of dustiness data for exposure assessment modelling

1 GD: use of dustiness data to support ATEX industrial risk analysis

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- SOPs to be written (by end of December 2020), Experimental testing (round robin) and reporting of preliminary results (by September 2021).
  - **Final TG draft submission to WNT for comments and endorsement (April 2022)**
  - **Final GD submission to WNT for comments and endorsement (June 2022-April-2023)**

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