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Helsinki, 22 June 2023

Addressee
Registrant of Multi-Walled Carbon Nanotubes as listed in Appendix 3 of this decision

Date of submission of the dossier subject to this decision
26/03/2020

Registered substance subject to this decision ("the Substance")
Substance name: Multi-Walled Carbon Nanotubes (MWCNT), synthetic graphite in tubular shape
EC/List number: 936-414-1

Decision number: Please refer to the REACH-IT message which delivered this communication (in format CCH-D-XXXXXXXXXX-XX-XX/F)

DECISION ON A COMPLIANCE CHECK OF A SET OF NANOFORMS

Based on Article 41 of Regulation (EC) No 1907/2006 (REACH), ECHA requires that you submit the information needed to bring the registration of the Set of Nanoforms K-Nanos (hereafter, "the Set of Nanoforms") into compliance with the information requirements listed below by the deadline of ***2 April 2024.***

1. **Composition of the registered substance (Annex VI, Section 2.3.)**

2. **Characterisation of the clearly defined boundaries of the Set of Nanoforms in accordance with the parameters set out in the sections 2.4.2 to 2.4.5 of Annex VI (introduction to Annex VI)**

3. **Justification that a variation within the boundaries of the Set of Nanoforms does not affect the hazard assessment, exposure assessment and risk assessment of the similar nanoforms in the set (introduction to Annex VI)**

In principle, each different nanoform covered by a registration must be reported and assessed individually. By derogation, it should be possible to group nanoforms of the substance with similar characterisation parameters in a set of similar nanoforms. Consequently, the incompliance(s) described above can be resolved by implementing one of the following actions:

1) by reporting and assessing each single nanoform covered by the currently reported set. This implies:

   a. the characterisation of each nanoform in accordance with sections 2.4.2 to 2.4.5 of Annex VI; and

   b. the submission of information on hazards, exposure and risk specific to each nanoform; and

   c. the reporting of the above information in such a manner that it is clear which hazards, exposure and risk information pertains to each nanoform.

2) by correcting the incompliances of the currently reported set.

3) by grouping the nanoforms covered by the currently reported set in different sets

of nanoforms. This implies that:

a. the boundaries of each set are clearly defined in the parameters in sections 2.4.2 to 2.4.5 of Annex VI; and

b. justification is provided for each set of nanoforms that the hazard, exposure and risk assessment of the nanoforms in the set can be performed jointly; and

c. the reporting of the above information in such a manner that it is clear which hazards, exposure and risk information pertains to each set of nanoforms

4) by reporting some of the nanoforms covered by the current set as single nanoforms and grouping the other nanoforms covered by that set in one or different sets of nanoforms.

Each reporting approach would have to fulfil the conditions set out respectively in option 1) and option 3).

Under Annex VI, a set of similar nanoforms is a group of nanoforms defined by clear boundaries. Based on the information currently provided in the dossier, ECHA cannot determine the actual nanoforms that you intended to cover within the set. Only the Registrant of each nanoform in the set knows the characterisation of that nanoform. Therefore, it is each Registrant's exclusive responsibility 1) to ensure that the boundaries of the set of nanoforms are clearly defined in accordance with sections 2.4.2 to 2.4.5 of Annex VI and 2) to justify that a variation within the boundaries of the set nanoforms does not affect the hazard assessment, exposure assessment and risk assessment of the similar nanoforms in the set.

Consequently, if the information eventually submitted by a Registrant does not enable ECHA to verify that the information in the dossier complies with the requirements set out in this decision, the set of nanoforms will not be considered valid. As a result, all the nanoforms that the set was supposed to cover will be considered as not registered. This could result in national enforcement authorities deciding on possible enforcement actions. The reasons of this decision are set out in Appendix 1. The procedural history is described in Appendix 2.

The scope of this compliance check decision is limited to the standard information requirements of Annex VI applicable to the Set of Nanoforms.

How to comply with your information requirements

To comply with your information requirements, you must submit the information requested by this decision in an updated registration dossier by the deadline indicated above. You must also update the chemical safety report, where relevant, including any changes to classification and labelling, based on the newly generated information.

Appeal

This decision, when adopted under Article 51 of REACH, may be appealed to the Board of Appeal of ECHA within three months of its notification to you. Please refer to http://echa.europa.eu/regulations/appeals.

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Failure to comply

If you do not comply with the information required by this decision by the deadline indicated above, ECHA will notify the enforcement authorities of your Member State.

Authorised[1] under the authority of Mike Rasenberg, Director of Hazard Assessment

Appendix 1: Reasons to request information on the submitted set of similar nanoforms under Annex VI of the REACH Regulation
Appendix 2: Procedure
Appendix 3: Addressees of this decision and their corresponding information requirements

[1] As this is an electronic document, it is not physically signed. This communication has been approved according to ECHA's internal decision-approval process.

Appendix 1: Reasons to request information on the submitted set of similar nanoforms under Annex VI of the REACH Regulation

1.1. Composition of the registered substance (Annex VI, Section 2.3.)

1 Annex VI, sections 2.3.2 and 2.3.3. of the REACH regulation requires reporting of the "*nature of impurities, including isomers and by-products*" and the "*percentage of (significant) main impurities*", respectively. ECHA guidance document 'Guidance for identification and naming of substances under REACH and CLP' (Version 2.1, May 2017) stipulates in section 4.2.1 that, for mono-constituent substances, "*impurities present in a concentration > 1% should be specified by at least one of the following identifiers: chemical name (IUPAC and/or CAS name), CAS-number and EC-number and/or molecular formula. Impurities that are relevant for the classification and/or PBT assessment shall always be specified by the same identifiers, independently from their concentration*".

2 You have defined your substance as a mono-constituent substance and reported the concentration range of the main constituent as [redacted] %(w/w). This means that your substance may contain up to [redacted] % of impurities. The results of analytical data included in section 1.4 of your IUCLID dossier reports [redacted] at concentration range from few ppm to more than one percent, but you have not reported any impurities for the substance in section 1.2 of your IUCLID dossier.

3 Therefore, you are requested to report the impurities in section 1.2 of your registration dossier in such way that the composition accounts to 100 % and in accordance with requirements specified in the 'Guidance for identification and naming of substances under REACH and CLP' (Version 2.1, May 2017).

4 In your comments to the draft decision you agreed with ECHA's finding that information on substance composition is incompliant and you agreed to report the impurities in such a way that the composition accounts to 100 %.

1.2. Characterisation of the clearly defined boundaries of the Set of Nanoforms in accordance with the parameters set out in the sections 2.4.2 to 2.4.5 of Annex VI (introduction to Annex VI)

5 Annex VI of REACH requires that each set of similar nanoforms is identified by clearly defined boundaries in the parameters in the sections 2.4.2 to 2.4.5 of the individual nanoforms within the set.

1.2.1. Information provided

6 In your dossier, you have reported one set of nanoforms named "*K-Nanos*". You have identified the boundaries of your set of nanoforms in Section 1.2 of your registration dossier and in a document entitled "[redacted]".

1.2.2. Assessment of the information provided

7 We have assessed the information you provided and we have identified the following issues on the basis of which we consider that the Set of Nanoforms does not fulfil the requirement for clearly defined boundaries in the parameters in sections 2.4.2 and 2.4.4 of Annex VI.

1.2.2.1. Unclear boundaries of the particle size distribution

8 Annex VI section 2.4.2. of the REACH Regulation requires reporting of "*number-based particle size distribution with indication of the number fraction of constituent particles in*

the size range within 1 nm – 100 nm". In addition, Annex VI section 2.4.6 of REACH Regulation requires reporting the "*description of the analytical methods or the appropriate bibliographical references for the information elements in [sub-section 2.4]. This information shall be sufficient to allow the methods to be reproduced*".

9 Further, ECHA Guidance document 'Appendix for nanoforms applicable to the Guidance on Registration and Substance Identification' outlines the principles for reporting particle size distribution and number fraction of constituent particles for a set of nanoforms.

- Section 4.1 stipulates that for a set of nanoforms, you must report the particle size distribution and the number fraction of constituent particles of the nanoforms included in the set with the smallest and largest d10, d50, and d90 value.
- Section 3.1.1.2.1 specifies that you must provide
  - the particle size distribution as a histogram with a table showing values on which the histogram is based on
  - the description of the particle size distribution measurement method including
    - description of sample preparation
    - instrument parameters
    - functions and calculations applied, as appropriate
    - measurand or precise name of the external dimension of the particles used in the measurement (e.g. minimum Feret diameter or maximum inscribed circle diameter)
    - measurement uncertainty

10 You have reported, in section 1.2 of your IUCLID dossier, the ranges for the d10, d50 and d90 values for "Tube diameter" as [redacted] respectively. In addition, you have reported that the range of lateral dimension of the tubes to be [redacted]. You have provided in section 1.4 the results of analysis of constituent particle diameter distributions of five different nanoforms in format of numerical ranges of values describing the diameter distribution. Furthermore, you have provided a brief description of sample preparation protocol and the instrument parameters of TEM equipment. In addition, you have provided results of laser diffraction based particles size analysis.

11 However, the analytical reports do not include any distribution graphs or tables showing the measured constituent particle diameter values, but only numerical values for range of tube diameters. The description of the sample preparation method is not such that it could be reproduced. You have neither described the measurand or precise name of the external dimension of the particles used in the measurement nor the measurement uncertainty. In addition, laser diffraction based methods cannot provide information on number-based particle size distribution of constituent particles.

12 As explained above, this information is necessary to determine the boundaries of the particle size distribution. Therefore, you are requested to provide analytical data which contains histograms with tables showing values on which the histograms are based on representing the constituent particle diameter distribution of the nanoforms with the smallest and largest d10, d50 and d90 of the nanoforms in the set. Furthermore, in line with draft OECD Test Guideline for particle size and particle size distribution of nanomaterials[2] you must include in the sample preparation description the specification of the dispersion medium, filtering procedure, concentration, dispersing agents, sample

[2] https://www.oecd.org/chemicalsafety/testing/draft-test-guideline-particle-size-distribution-nanomaterials.pdf

dilution, final sample concentration, dispersing procedure as well as sonicator brand/type, calibration of delivered power, energy input, indicated power, amplitude and pulse time, as applicable. Finally, you are also requested to describe the analytical methods used for the constituent particle size distribution measurement in line with the requirements specified in the REACH Annex IV section 2.4.6 and in section 3.1.1.2.1 of the Guidance document 'Appendix for nanoforms applicable to the Guidance on Registration and Substance Identification'.

13 The information must be provided in the section 1.4 of your IUCLID dossier.

14 In your comments to the draft decision, you agreed with the finding that the size information reported is incompliant and you agreed to provide the information requested.

1.2.2.2. Unclear boundaries of the shape and morphology - Shape

15 The REACH Annex VI section 2.4.4. requires reporting of the "*shape, aspect ratio and other morphological characterisation: crystallinity, information on assembly structure including e.g., shell like structures or hollow structures, if appropriate*".

16 Further, Section 4.2 of the 'Appendix for nanoforms applicable to the Guidance on Registration and Substance Identification' outlines the principles for reporting of shape, aspect ratio and other morphological characterisation for a set of similar nanoforms. It stipulates that nanoforms consisting of particles falling under different shape categories must in principle not be part of a same set of similar nanoforms.

17 The 'Appendix for nanoforms applicable to the Guidance on Registration and Substance Identification' also stipulates that the following information must be reported for each set of nanoforms:

a. the shape category of the set, and

b. a list of the specific shapes covered under a certain set, and

c. the range of number of walls or of layers for particles with an assembly structure. The range must reflect the variation between the nanoforms that are part of the set, and

d. an electron microscopy image for each nanoform with a different shape included within the set.

18 In addition, for a set of elongated nanoforms, the following additional information must be provided:

e. the range of the aspect ratios of the different nanoforms covered under the set, and

f. the maximum and minimum length of the nanoforms that are part of the set, and

g. where relevant (e.g., when rigidity is a part of the justification), an indication of the rigidity of the nanoforms that are part of the set (e.g., based on the cross-sectional diameters/widths).

19 In your dossier, you have reported the shape category of the nanoforms in the set to be "*elongated*" and their specific shape to be "*tube*". You have reported the length of the tubes to vary between [redacted] and the aspect ratio between [redacted]. Based on the analytical information included in section 1.4 of your IUCLID dossier the length values are based on the "*bundles*" of the tubes. You have reported the assembly structure

to be "*Multi walled carbon nanotube*" and based on the analytical data included in section 1.4 of your IUCLID dossier, the number of walls of the tubes vary between [redacted].

20 However, the constituent particle diameter and the length of the tube bundles are not comparable because bundles consist of many constituent particles and thus the aspect ratio calculated based on these values is not meaningful. As the shape of the particles and thus also the aspect ratio values are related to the shape of the constituent particles, the aspect ratio must be determined by the length and width of the constituent particles.

21 In your comments to the draft decision, you state that no reproducible method exists that can disperse MWCNT bundles into single tubes without breaking the tubes and therefore it is impossible to perform reliable measurements of single tube length at this point in time. However, existing scientific literature shows that bundles/agglomerates of carbon nanotubes can be dispersed or deagglomerated for length measurements (see for *e.g.* DOI: 10.1021/am500424u; DOI: 10.1002/adfm.201402976 or DOI: 10.1126/sciadv.abm3285).

22 Furthermore, you state in your comments that you assume that the bundle length is similar to single tube length and this assumption is based on and justified by the manufacturing process of K-Nano MWCNT described in the Annex of your comments to the draft decision. However, this description does not *per se* demonstrate that bundles would contain constituent particles of which the length is equal to the bundle length.

23 Finally, you report a broad range of lengths [redacted] of the bundles. However, the provided manufacturing process description does not demonstrate that the long bundles would contain constituent particles which have always a length equal to the length of the bundles. It is neither demonstrated why short constituent particles are not present in long bundles.

24 In the absence of scientific substantiation, your comments to the draft decision could not justify a modification of the draft decision.

25 You have not provided any electron microscopy images to support determination of shape, length and number of the walls of the tubes. Furthermore, you have not described the analytical methods used to determine the length of the tubes (constituent particles) nor provided any indication of the rigidity of the tubes, although carbon tubes with diameter of 30 nm or more have been concluded be potentially rigid[3].

26 Therefore, you are requested to provide electron microscopy image(s) for each nanoform with a different shape. The images must allow to confirm the reported number of the walls of the tubes. You must also describe the analytical methods used for determination of the length of the tubes and you must provide an indication of rigidity of the tubes. The electron microscopy images and the description of the analytical methods must be provided in section 1.4 of your IUCLID dossier and the description of the shape including the number of walls and indication of rigidity in section 1.2 of your IUCLID dossier.

27 In your comments to the draft decision, you agreed with ECHA's finding that shape information is incompliant and you agreed to provide:

- the range of the aspect ratios of the different nanoforms covered under the set (single tube and bundle aspect ratio);
- electron microscopy image(s) for each nanoform with a different shape where the image allows to confirm the reported number of the walls of the tubes;
- a description of the analytical methods used to determine the length of the tubes;
- an indication of the rigidity for tubes with diameter of >= 30 nm.

[3] https://echa.europa.eu/registry-of-clh-intentions-until-outcome/-/dislist/details/0b0236e18195a284

1.2.2.1. Unclear boundaries of the shape and morphology – Crystallinity

28 The Annex VI section 2.4.4. of the REACH Regulation requires reporting of "*shape, aspect ratio and other morphological characterisation: crystallinity, information on assembly structure including e.g., shell like structures or hollow structures, if appropriate*".

29 Further, Section 4.2 of the 'Appendix for nanoforms applicable to the Guidance on Registration and Substance Identification' outlines the principles for reporting of shape, aspect ratio and other morphological characterisation for a set of similar nanoforms. It stipulates that when reporting the information on the crystallinity of a set of nanoforms, you must specifically provide:

30 For a set including only amorphous nanoforms:

- A clear indication that the set includes only amorphous nanoforms

31 For a set including only crystalline nanoforms with one specific crystal structure:

- The name of the specific crystal structure covered
- A clear indication that the set includes nanoforms consisting of particles with only specific crystal structure

32 For a set including crystalline nanoforms where the individual nanoforms consist of particles with more than one crystal structure:

- The names and the ranges (as w/w percentage) of different crystal structures covered by the set

33 For a set including partially crystalline nanoforms:

- The range(s) (as w/w percentage) and the name of different crystal structure(s) and the range of amorphous fraction covered by the set.

34 Any information submitted based on requirements specified above must be consistent.

35 You have reported one crystal structure identified as "*other: carbon nanotube*" and selected "*yes*" for 'Pure structure'. Further, you state in the 'Description' field that "*XRD analysis result show peaks were ascribed to reflections from the carbon nanotubes. Raman Spectroscopy result show the IG/ID (Gmode / Dmode ratio) of* [redacted]".

36 However, the reported information concerning crystallinity is inconsistent. On one hand, reporting "*yes*" for 'Pure structure' implies that the nanoforms in the Set are fully crystalline with crystal structure "*carbon nanotube*". On the other hand, the information provided in the 'Description' field indicates that nanoforms in the Set have IG/ID ratio between [redacted] but you do not provide any justification why these values support the conclusion that the nanoforms are fully crystalline.

37 Therefore, you are requested to report in Section 1.2 of your IUCLID dossier consistent information on crystallinity either by reporting all the crystalline and amorphous phases and the concentration ranges reflecting the boundaries in terms of different crystal structures of the nanoforms in the Set, or by providing representative analytical information which shows that no amorphous phases are present in the nanoforms part of the Set.

38 The analytical information must be provided in Section 1.4 of your IUCLID dossier.

39 In your comments to the draft decision you agreed to "*report the crystalline and amorphous phases, and the percentage crystallinity of the nanoforms*". ECHA takes note of your intention to submit the requested information. Please note that if the crystallinity of the nanoforms in the set varies, a justification must be provided that hazard, exposure, and

risk assessment of the nanoforms with different degree of crystallinity can be performed jointly.

1.3. Justification that a variation within the boundaries of the set of nanoforms does not affect the hazard assessment, exposure assessment and the risk assessment of the similar nanoforms in the set (introduction to Annex VI)

40 Annex VI of the REACH regulation requires that a "*justification shall be provided to demonstrate that a variation within these boundaries does not affect the hazard assessment, exposure assessment and risk assessment of the similar nanoforms in the set*".

1.3.1. Information provided

41 In your dossier, you have provided a document "[redacted]" in the field "*Justification for reporting set of similar nanoforms*" in section 1.2. of IUCLID.

1.3.2. Assessment of the information provided

1.3.2.1. Missing explanation addressing the physicochemical of nanoforms in the set of Nanoforms

42 Section 4 of the 'Appendix for nanoforms applicable to the Guidance on Registration and Substance Identification' explains how to justify that the variation of a characterisation parameter of the nanoforms covered by the set does not change the hazard profile of those nanoforms[4]. More specifically, the justification must contain documented evidence that the registrant has investigated the threshold beyond which a variation of a characteriser will affect the property of the nanoforms included in the set. More specifically, the justification must investigate at minimum the following:

- Does the variation of the characterisation parameters of the different nanoforms within the set impact their dissolution rate and solubility?
- Does the variation of the characterisation parameters of the different nanoforms within the set impact their toxicokinetic behaviour, as well as their fate and (bio)availability?
- Does the variation of the characterisation parameters of the different nanoforms within the set impact their (eco)toxicity? Is there a direct relationship between that variation and the (eco)toxicity?

43 The justification must address separately each characterisation parameter set out in Section 2.4 of Annex VI for which there is a variation among the different nanoforms within the set.

44 In your justification document, you report for dissolution rate and solubility that "*K-Nanos are almost [redacted]% [redacted] carbonaceous material, which is insoluble and have a very poor dissolution rate in environmentally and physiologically relevant media*". For ecotoxicological and fate properties, you consider that the nanoforms are insoluble and unlikely to be systematically distributed.

45 However, while the boundaries of the Set of Nanoforms report a variation of particle size distribution, your justification does not investigate whether this variation impacts solubility and dissolution and does not contain any documented evidence for your claim that differences in dissolution rates of different nanoforms part of the set are irrelevant for the joint hazard assessment.

[4] Section 4.1 (Page 22) and 4.2.2.1 (page 23) of the Appendix for Nanoforms applicable to the Guidance on Registration and the Guidance on Substance Identification

46 In your comments to the draft decision, you inform that you are planning to conduct OECD TG 318 studies with [redacted] and [redacted] and that you encountered numerous technical difficulties with a study on dispersion stability according to OECD TG 318 previously performed with a different registered nanoform of MWCNT. You found that dispersion stability was very low for the investigated MWCNT test material in the screening test of OECD TG 318 and you expect similar results for [redacted] and [redacted].

47 You also indicate that you are "*planning to conduct a preliminary test for the remaining nanoforms of the set, i.e.* [redacted]*, and* [redacted]*, because it is technically extremely difficult and costly to perform the test*." You also add that "[you] *will prepare a stock solution identical to the tested stock solution in the OECD TG 318 studies for each remaining nanoform of the set and visually investigate if each nanoform agglomerates in the same way as for* [redacted] *and* [redacted]". However, a simple visual inspection is not considered an appropriate method for performing a screening study.

48 In your comments to the draft decision you indicate that you are "*planning to conduct a study according to OECD TG 105 with* [redacted] *and* [redacted] *in an extended testing regime*". You highlight the technical challenge of the analytical method available for carbon based substances and comment on the limitations of these measurements (water solubility and dissolution rate) for all the forms. Consequently, you do not commit to perform more measurements for your different nanoforms. You specifically highlight that "*Analytical measurement of MWCNT is a major challenge and in principle technically not feasible... Therefore,* [you] *foresee to establish the lowest LOD technically feasible and establish a value for water solubility for* [redacted] *that is below LOD".* Finally you indicate that: *"Since the water solubility of MWCNT has been well researched and consistently shown to be very low or insoluble,* [you] *think it is justified to assume that no higher value of water solubility will be reached than the lowest LOD that was technically feasible for* [redacted] *and* [redacted]".

49 Although ECHA takes note of your testing strategy for water solubility, your comments do not provide any substantiated justification. More specifically, you do not provide any scientific evidence demonstrating that the methods specified in the latest adaptation of the OECD TG 105 in conjunction with OECD GD 318 do not allow a quantitative measure of solubility and dissolution of your nanoforms.

50 Therefore, your justification does not demonstrate that the variation of this characterisation parameter of the nanoforms in the Set does not affect the joint hazard assessment of these nanoforms. Consequently, you have not established that the hazard assessment of the nanoforms within the set can be performed jointly.

51 In your comment to the draft decision you indicate that you will generate data on water solubility, dustiness and dispersion stability to provide information on the potential release of the set of nanoforms. ECHA takes note of your intention.

52 In any case, the incompliance identified under this section is the absence of explanation addressing the physicochemical properties of nanoforms in the set. A justification must therefore be provided by the deadline indicated.

53 Should you intend to pursue the testing strategy you describe in your comment, ECHA can already bring your attention on the following observations.

54 Firstly, the release of ions or molecules from various impurities (e.g. catalyst residues) due to dissolution can impact the hazard profile of the different nanoforms in the Set. Therefore, you must take account of this release in order to justify the joint hazard assessment of the nanoforms in the Set.

55 Secondly, the testing strategy you describe in your comments may potentially substantiate an explanation addressing the physicochemical properties of nanoforms in the Set only if

the screening test is performed in accordance with OECD TG 318 requirements. The OECD TG 318 specifies that screening tests have to be performed at different pHs and ionic strength and also with NOM (Natural Organic Matter) as explained under OECD TG 318 paragraph 13-23 including in particular figure 2. Information on dispersion stability is sufficient to justify a set in relation to this parameter, if results indicate similar dispersion stabilities under the corresponding conditions. The screening test is sufficient as assessment of dispersion stability only if its results indicate that the nanoforms tested have either <10 % (very low dispersion stability) or > 90 % (very high dispersion stability) of material left in the supernatant under all conditions of the screening. It is not possible to establish this by visual inspection. You must also follow the advice provided in OECD GD 318 with regard to the performance of dispersion stability tests.

56 Thirdly, your testing strategy to generate data on water solubility, dustiness and dispersion stability must clarify in which form the environment or humans can be exposed to the nanoforms covered by the Set (e.g. dissolved form, undissolved form, mixture of both and extend of agglomeration).

1.3.2.2. Missing (robust) study summary(ies)

57 In the absence of robust study summaries or study summaries, ECHA cannot assess the reliability of your justification.

58 In your comments to draft decision you inform that the "*missing robust study summaries will be reported when the final study reports of the new experimental studies are available.*" ECHA takes note of your intention to submit the requested information.

59 Therefore, you have not demonstrated that the hazard assessment of the nanoforms can be performed jointly.

1.3.2.3. Missing justification for joint exposure assessment of the Set of Nanoforms

60 Section 4 of the 'Appendix for nanoforms applicable to the Guidance on Registration and Substance Identification' (Version 2.0 – January, page 22-23) states that a justification must be provided as to "*why the exposure* […] *can also be performed jointly for the set of nanoforms*". It specifically requires that "*a common conclusion on exposure assessment can be reached for the set*". This is demonstrated when the potential release is similar for all the nanoforms in the set with regards to all their respective exposure routes. For example, for airborne exposure, this is demonstrated by similar value of dustiness (or by using a dustiness value that is conservative); for aquatic exposure, it is demonstrated as a minimum by similar dispersion stability, dissolution behaviour and surface functionalisation of all nanoforms within the set.

61 However in your dossier, you have not provided any information on the potential release of the nanoforms.

62 Therefore, you have not demonstrated that there is no variation in the potential release of the nanoforms and that the exposure assessment of all the nanoforms in the set can be performed jointly.

63 Therefore, it is not demonstrated that a common conclusion on exposure assessment can be reached for the set. Hence, the risk assessment of the set of similar nanoforms cannot be performed jointly.

64 In your comments to the the draft decision you indicate that you are currently generating new data (dispersion stability, water solubility and dustiness) and will provide elaborate reasoning on theoretical considerations (on dissolution behaviour) to justify a joint exposure assessment for the set of nanoforms. ECHA takes note of your intention. However, the

incompliance identified under this section is the absence of justification for joint exposure assessment of the Set of Nanoforms. A justification must therefore be provided by the deadline indicated.

1.3.2.4. Hazard/fate data provided only on one nanoform in the Set of Nanoforms

65 To demonstrate that the hazard assessment of the nanoforms in a set of nanoforms can be performed jointly, Annex VI of REACH regulation requires that a "*justification shall be provided to demonstrate that a variation within these boundaries does not affect the hazard assessment, exposure assessment and risk assessment of the similar nanoforms in the set*". Therefore, the registrant must provide hazard information demonstrating that there is a common pattern in the potency of the (eco)toxicological properties and fate of the nanoforms in the set, irrespective of a variation in any characterisation parameter. To establish a common pattern, the registrant must firstly identify the nanoforms with the characterisation parameter values defining the boundaries of the set of nanoforms. Secondly, the registrant must submit scientific evidence that the variation between the nanoforms relating to the same characterisation parameter does not result in a variation of the properties of these nanoforms. This demonstration must be done for each characterisation parameter defining the boundaries of the set of nanoform. The demonstration of the common pattern must also be relevant for all the hazard information requirements applicable to set of nanoforms.

66 In your dossier, you provided hazard information only on one nanoform in the Set (*i.e* [redacted]). However, the reporting of the boundaries for the Set of Nanoforms indicates that that there is a variation of particle size distribution (the length of the tubes varies from [redacted] and the lateral dimension from [redacted]) of the nanoforms covered by the set.

67 A common pattern between variating nanoforms cannot be established based on information on only one of these nanoforms. Conclusion on similarity requires to have comparable information on more than one form to conclude on a common pattern of potency in the set.

68 In your comments on the draft decision, you agree with ECHA's assessment and indicate that you propose to split the set into 2 separate sets and that you "*are planning to generate new data on (eco)toxicological properties and fate of the nanoforms for Set 2 if the manufacturing company decides (after a data gap analysis) to continue with the preparation of a second set of similar nanoforms for* [redacted] *and* [redacted] *The new Set 2 would be ultimately included in our dossier*". You also argue that "*nanoform* [redacted] *covers the boundaries of particle size distribution for Set 1, and thus provided hazard information can demonstrate that there is a common pattern in the potency of the (eco)toxicological properties and fate of the nanoforms included in Set 1.*"

69 ECHA takes note of your intended strategy and will assess the compliance of the information on the new set(s) of nanoforms when it has been submitted as part of a registration dossier.

70 Therefore, you have not established that irrespective of a variation in any characterisation parameter, there is a common pattern of potency of the (eco)toxicological properties of the nanoforms. Therefore, based on the information provided on one form, you did not demonstrate that the hazard assessment of all the nanoforms in this set can be performed jointly.

71 Should you intend to pursue the testing strategy you describe in your comment, ECHA can already bring your attention on the following observations. Regarding the claim that [redacted] covers the boundaries of the particle size distribution for Set 1, this is not demonstrated by any scientific evidence. In addition, you seem to indicate that there is variation in the

particle size distribution. However, by default, one nanoform with specific particle size distribution cannot be representative for other nanoforms with different particle size distribution. According to the nanoform guidance, the nanoforms with smallest and largest d10, d50 and d90 values are the ones which establish the boundaries for a set of nanoforms. Any justification of the set(s) of nanoforms you intend to submit must address the variation in particle size distribution.

1.3.2.5. Hazard data do not cover all the variations of characterisation parameters among the nanoforms in the Set of Nanoforms

72 To demonstrate that the hazard assessment of the nanoforms in a set of nanoforms can be performed jointly, Annex VI of REACH regulation requires that a "*justification shall be provided to demonstrate that a variation within these boundaries does not affect the hazard assessment, exposure assessment and risk assessment of the similar nanoforms in the set*". Thus, in relation to variation of each characterisation parameter, the registrant must provide hazard information demonstrating that there is a common pattern in the potency of the (eco)toxicological properties despite the variation of the characterisers of the nanoforms in the set. The registrant must not necessarily provide hazard information on every information requirement, but the information provided must at least demonstrate that there is a common pattern and that this common patter is relevant to all the applicable information requirements.

73 In your justification for the Set of Nanoforms, you have not reported the variation in the following characterisation parameters: shape, crystallinity and surface treatment.

74 Nevertheless, the reporting of the boundaries for the Set of Nanoforms shows significant variation in particle size distribution. More specifically, the nanoform [redacted], on which the data are generated, has the smallest d50 value of the diameter within the set (*i.e.*, ranging from [redacted]).

75 However, the maximum diameter value of the nanoforms in the set is > 30 nm for [redacted] and [redacted].

76 This value has potentially toxicological significance and, based on available data related to tumour induction, a CLH proposal[5] adopted by RAC in March 2022[6] for MWCNT established a cut-off value of 30 nm for the lower boundary diameter as a proxy for rigidity. However, the nanoforms with a diameter of 30 nm or more are not addressed by your justification. As a result, the provided hazard information on nanoform K-Nano 100 cannot cover all the toxicological concerns that may result from all the nanoforms in the Set, especially those with a diameter of 30 nm or more.

77 In your comments on the draft decision, you agree with ECHA's assessment and "*will therefore divide the current set of nanoforms in two separate sets of nanoforms, i.e. Set 1 and Set 2. Set 1 will consist of nanoforms* [redacted] *and* [redacted]*, and Set 2 will consist of nanoforms* [redacted] *and* [redacted]*. Thus, Set 2 will fall within the defined boundaries of the CLP proposal whereas Set 1 will not. In this way, it will be possible to address toxicological concerns related to tube diameter ≥ 30 nm.*"

78 ECHA takes note of your intention and will assess the compliance of the information on the new set of nanoforms when it has been submitted as part of a registration dossier.

79 Therefore, you have not established that there is a common pattern in the potency of the (eco)toxicological properties taking account of the variation of all the characterisers of concern in sections 2.4.2 to 2.4.5 of Annex VI. Therefore, based on the information

[5] https://echa.europa.eu/registry-of-clh-intentions-until-outcome/-/dislist/details/0b0236e18195a284
[6] Annex (europa.eu)

concerning only some of these characterisers, you did not demonstrate that the hazard assessment of all the nanoforms in this set can be performed jointly.

1.3.2.6. Hazard data provided only on a nanoform outside the Set of Nanoforms

80 In accordance with Annex VI of REACH, the parameters in sections 2.4.2 to 2.4.5 for an individual nanoforms are used to define the boundaries of a set of nanoforms in order to "*allow to conclude that the hazard assessment, exposure assessment and risk assessment of these nanoforms can be performed jointly*". This implies that the justification can be based only on hazard information from nanoforms with characterisation parameter values within the boundaries of the set.

81 However, in your dossier you provided toxicokinetic information generated on a nanoform with diameter and BET surface area which are outside the boundaries defined for the Set of nanoforms. This nanoform, used in toxicokinetic studies within the OECD Working Party, has a diameter of 4.8 nm - 88 nm, length 0.94 µm - <20 µm and a BET surface of 23 - 230 m2/g.

82 In your comments on the draft decision you agree with ECHA's assessment and indicate that you will remove the data. While you indicate that you "*must provide new data on toxicokinetic information for Set 1 and Set 2"* and that you "*will perform a data-gap-analysis for each set and then may perform a new experimental study if no suitable data is available (e.g. published data)*", you do not provide any further hazard information relating to the nanoforms covered by the Set.

83 In the absence of hazard information generated on nanoforms covered by the set and subsequent set(s), you have not justified that the hazard assessment of the nanoforms in this set can be performed jointly.

1.3.2.7. Hazard data provided only on an unknown nanoform

84 Recital 12 of the COMMISSION REGULATION (EU) 2018/1881, stipulates that "*to allow for adequate assessment of the relevance of any physicochemical, toxicological and ecotoxicological information for the different nanoforms, the test material should be appropriately characterised. For the same reasons, test conditions documented and a scientific justification for the relevance and adequacy of the utilised test material as well as documentation for the relevance and adequacy of the information obtained from means other than testing for the different nanoforms should be provided*".

85 However, in your dossier you provided carcinogenicity information generated on a nanoform characterised only by the fibre diameter.

86 In the absence of appropriate characterisation of the test material used in the generation of the data, it is not possible to conclude whether the tested nanoforms is representative for the nanoforms included in the set. Therefore, it cannot be concluded that the hazard assessment of the nanoforms in this set can be performed jointly.

87 In your comments to the draft decision you agree with ECHA's assessment and you indicate that you will remove the carcinogenicity information generated on a nanoform characterised only by the fibre diameter (reference to [redacted] 2012 in your comments). You also indicate your intention to refer to "(*i) BAuA's conclusion on the substance evaluation of MWCNT (EC no. 936-414-1) published in 2020 that defines nanoforms with a diameter < 30 nm lose their rigid fibre shape and (ii) BAuA's follow-up proposal on harmonised classification and labelling of MWC(N)T that exclude nanoforms with a geometric tube diameter < 30 nm from classification for carcinogenicity, i.e. Carc. 1B, H350i*".

88 Should you intend to pursue the intention you describe in your comment, ECHA can already bring your attention on the following observation. The harmonized classification for MWC(N)T of defined dimensions, is based on a "*fibre paradigm mechanism*". As explained in the classification proposal document, fibres may induce carcinogenicity in the lung via other mechanisms as shown for short tangled MWCNT (*e.g.,* Saleh *et al.*, 2020, doi: 10.1186/s12989-020-00382-y).

89 Nevertheless, the information provided in the BAuA's conclusion on substance evaluation of MWCNT does not change the finding that the hazard/fate data provided relate only to an unknown nanoform.

Appendix 2: Procedure

This decision does not prevent ECHA from initiating further compliance checks at a later stage on the registrations present.

ECHA followed the procedure detailed in Articles 50 and 51 of REACH.

The compliance check was initiated on 05 July 2021.

ECHA notified you of the draft decision and invited you to provide comments.

ECHA took into account your comments and did not amend the requests.

In your comments on the draft decision, you requested an extension of the deadline to provide information from 3 to 9 months from the date of adoption of the decision.

On the 30 August 2022 ECHA requested clarifications to substantiate your request for an extension of the deadline, in order for ECHA to understand the need for an extension of the deadline and evaluate a proportionate time. More specifically, ECHA requested information on the detailed actions you intended to take; measurements and the precise nature of the tests you intend to perform; and information on the precise nature of the literature searches (scope, sources, search and quality criteria) you intend to perform.

In response, you provided a more detailed testing plan including the measurements and also searches for data and literature you consider necessary to comply with the decision information requirement and the generation of data to justify the sets of nanoforms. Based on this information, ECHA has extended the deadline indicated in the decision from 3 months to 9 months.

ECHA notified the draft decision to the competent authorities of the Member States for proposals for amendment.

ECHA received proposals for amendment and modified the draft decision.

ECHA invited you to comment on the proposed amendments and referred the modified draft decision to the Member State Committee.

You have provided comments on the draft decision. These comments do not address the proposed amendment(s). Therefore, these comments were not taken into account by the Member State Committee as they were considered to be outside of the scope of Article 51(5).

The Member State Committee unanimously agreed on the draft decision in its MSC-82 written procedure. ECHA adopted the decision under Article 51(6) of REACH.

~~Confidential~~

Appendix 3: Addressee of this decision and their corresponding information requirements

In accordance with Articles 10(a) and 12(1) of REACH, the information requirements for individual registrations are defined as follows:

Registrant Name	Registration number
[redacted]	[redacted]

Where applicable, the name of a third party representative (TPR) may be displayed in the list of recipients whereas ECHA will send the decision to the actual registrant