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Helsinki, 30 November 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
12/11/2021

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 JEIO multi-walled carbon nano tubes (MWCNT) (hereafter, "the Set of Nanoforms") into compliance with the information requirements listed below by the deadline of ***6 June 2024***.

1. **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)**

2. **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.
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 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.

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. 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)

1 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.

1.1.1. Information provided

2 In your dossier, you have reported "*JEIO multi-walled carbon nano tubes (MWCNT)*" as a set of similar nanoforms which boundaries are defined under the Set of Nanoforms in Section 1.2 of your registration dossier. In your comments to the draft decision, you have provided information on characterization of the Set of Nanoforms which was further consolidated in the latest dossier update by providing the document entitled "[redacted].pdf" in the field "*Justification for reporting set of similar nanoforms*" in section 1.2. of the IUCLID dossier.

1.1.2. Assessment of the information provided

3 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.

1.1.2.1. Unclear boundaries of the shape and morphology - Shape

4 The Annex VI section 2.4.4. of the REACH Regulation 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*".

5 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.

6 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.

7 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).

8 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 1.9 and 552 µm and the aspect ratio between 1:163.8 and 1:47977.3. Based on the analytical information included in section 1.4 of your IUCLID dossier the length values are based on the "*end-to-end distance*" of the fibre bundles.

9 Based on the information provided in your comments to the draft decision, we understand that you assume that the fiber bundle length is the same as the length of individual tubes, *i.e.* the constituent particles. However, such assumption is not adequate to meet the data requirement specified in the Annex VI of the REACH Regulation. For elongated particles such as nanotubes, you must report the range of length of the constituent particles and not the length of bundles potentially formed, which in turn should be considered as aggregates or agglomerates of the constituent particles. Indeed, fiber bundles might consist of many constituent particles of different lengths, not corresponding to the provided bundle length. Only the length of constituent particles is considered as characterization parameter to define boundaries of the set of nanoforms based on their shape.

10 As per the information provided in your comments to the draft decision, the bundle length is assumed to be the same as the constituent particle length. However, the data provided in the analytical report does not fully reflect the values reported in section 1.2 of your dossier. For instance, the measured length is reported up to higher values such as 634 µm while data confirming a length down to 1.9 µm can be hardly found. Moreover, the measurements of 'end-to-end distance' of the fiber bundles from electron microscopy images don't appear to be precisely reflecting the bundle length due to their entanglement.

11 Based on the information provided in your comments, the aspect ratio is calculated using the constituent particle size diameter and the "*end-to-end distance*" value of the fibre bundle. Furthermore, you state that "[t]*he Jenotube MWCNT are not rigid and therefore are tangled with a very high aspect ratio*".

12 However, the constituent particle diameter and the "*end-to-end distance*" of the fibre bundle are not comparable because bundles consist of many constituent particles and thus the aspect ratio as reported in your IUCLID dossier 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 i.e. individual particles (carbon nanotubes) and not the fiber bundles.

13 In addition, you claim in the analytical reports included in section 1.4 of IUCLID dossier that "[t]*he Individual CNT tubes in the fiber bundle cannot be separated from each other without CNT tube destruction and CNT tube length cannot be obtained because of the twist and the entanglement of fiber bundles*".

14 However, the claim "*CNT tube length cannot be obtained*" is not fully supported by the SEM images provided in the Section 1.4 of your IUCLID dossier. The electron microscopy images allow individually separated carbon nanotubes to be identified e.g. in

- Figure 17 of report '[redacted].pdf
- Figure 16 of report '[redacted].pdf'
- Figure 14 of report '[redacted].pdf'
- Figures 17-21 and S6-S23 of report '[redacted].pdf'.

15 Thus, your claim is contradicted by this documentary evidence and it must therefore be rebutted.

16 In your comments to the draft decision, you claim that currently no reproducible method exists that can disperse MWCNT bundles into single tubes without breaking the tubes. However, existing scientific literature shows that bundles/agglomerates of carbon nanotubes can be dispersed or deagglomerated for length measurements (see for *e.g.*, dx.doi.org/10.1021/am500424u; DOI: 10.1002/adfm.201402976 or DOI: 10.1126/sciadv.abm3285). You only briefly describe the procedure for sample preparation for electron microscopy measurements and no further optimization of CNT dispersion is provided in terms of solvent used, conditions of ultrasonification or presence of surfactants.

17 Further in your comments to the draft decision, you also state that the similarity of the length of the single tubes to length of the bundles can be explained by the manufacturing process. The manufacturing mechanism of CNT growth is explained in the annex to your comments. However, this description does not *per se* demonstrate that bundles would contain constituent particles of which length is equal to the bundle length.

18 Finally, you report broad range of lengths (1.9-552 µm) of the bundles, but the provided manufacturing process description does not demonstrate that the long bundles would contain constituent particles which always have length equal to length of the bundles. It is neither demonstrated why short constituent particles are not present in long bundles.

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

20 Therefore, you are requested to report in section 1.2 of IUCLID dossier the length and aspect ratio of the constituent particles. You must also in accordance with REACH Annex VI section 2.4.6 describe the analytical methods for determination of the length of the tubes.

21 The description of the analytical methods must be provided in section 1.4 of your IUCLID dossier.

22 In your comments to the draft decision, you agreed with the 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.

1.2. 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)

23 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*".

1.2.1. Information provided

24 In your comments to the draft decision, you have provided information which was further consolidated in your latest dossier update by providing the justification document

"[redacted].pdf" in the field "*Justification for reporting set of similar nanoforms*" in section 1.2. of IUCLID dossier.

1.2.2. Assessment of the information provided

1.2.2.1. Missing explanation addressing the physicochemical properties of nanoforms in the set

25 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[2]. 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?

26 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.

27 In your justification document, you report for dissolution rate and solubility that "*Jenotube MWCNTs are inorganic and are composed of almost 100% pure carbonaceous material, which is insoluble, and have very poor dissolution rate in environmentally and physiologically relevant media*".

28 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 solubility and dissolution rates of different nanoforms part of the set are irrelevant for the joint hazard assessment.

29 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.

30 In your comments on the draft decision, you agree with the deficiencies noted above and you inform that you are currently conducting and planning new studies on dispersion stability (OECD TG 318) with Jenotube 8, water solubility with Jenotube 8 (OECD TG 105), and dustiness (EN 17199) with all nanoforms.

31 In your comments on the draft decision, you indicate that, at the time of dossier submission, you were conducting a water solubility study for Jenotube 8 and you highlight the technical challenge of the analytical method available for carbon based substances: "*Based on general physico-chemical considerations, relevant dissolution of carbon from the four nanoforms is considered highly unlikely*". More specifically, you comment on the limitations of the measurements of water solubility and dissolution rate for all the forms. Consequently,

[2] 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

you claim that you do not commit to perform more measurements for the different forms of Jenotube. In particular, you 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 Jenotubes that is below LOD".* You finally indicate in your comments 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 for the other nanoforms of the set no higher value of water solubility will be reached than the lowest LOD that was technically feasible for test material Jenotube 8*".

32 Although ECHA takes note of your testing strategy for water solubility, your assumptions expressed in your comments are not substantiated. 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. Also, your assumption that no higher value of water solubility will be reached for the other nanoforms of the set is unsubstantiated. In any case, the measurements for the nanoforms of your set are needed to justify, for this characterisation parameter, that a variation within the boundaries of the Set of Nanoforms does not affect the hazard assessment.

33 Moreover, you acknowledge that nonetheless, the dissolution of impurities may be relevant and explain that "*experimental studies according to the OECD Guidance Document (GD) No. 29 (OECD, 2001) with adaptations to nanomaterials were contracted for all four nanoforms and are expected to be completed in the first quarter 2023."*

34 In your comments to the draft decision, you further indicate your intention to investigate the dispersion stability by "*conducting a preliminary test for the remaining nanoforms of the set: In detail, we will prepare a stock solution identical to the Jenotube 8 stock solution in the OECD TG 318 study for each remaining nanoform of the set and visually investigate if each nanoform agglomerates in the same way as Jenotube 8."*

35 However, ECHA highlights that a simple visual inspection is not considered an appropriate method for performing a screening study.

36 ECHA takes note of your intention to generate data on water solubility and dissolution rate, dustiness and dispersion stability to provide information on the potential release of the set of nanoforms, and of your dossier update containing information on some of the physico chemical properties. In any case, the incompliance identified under this section is the absence of justification to demonstrate that the variation of this characterisation parameter(s) of the nanoforms in the Set does not affect the joint hazard assessment of these nanoforms, and this incompliance still remains.

37 Should you intend to pursue the testing strategy you describe in your comment, ECHA can already bring your attention on the following observations. Firstly, the testing strategy you described 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 only. You must also follow the advice provided in OECD GD 318 with regard to the performance of dispersion stability tests.

1.2.2.2. Missing (robust) study summary(ies)

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

39 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. However, currently this information is not available for assessment.

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

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 at least 6 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 and the generation of data to justify the set of nanoforms. Based on this information, ECHA has extended the deadline indicated in the decision from 3 months to 6 months.

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

ECHA received proposal(s) for amendment and modified the draft decision.

ECHA invited you to comment on the proposed amendment(s) and referred the modified draft decision to the Member State Committee.

You did not provide any comments on the proposed amendment(s).

The Member State Committee unanimously agreed on the draft decision in its MSC-84 written procedure. ECHA adopted the decision under Article 51(6) of REACH.

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Appendix 3: Addressees 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.