EUROPEAN CHEMICALS AGENCY

Helsinki, 29 May 2017

Addressee: [redacted]

Decision number: CCH-D-2114360550-57-01/F
Substance name: aluminum zirconium chloride hydroxide
EC number: 260-599-1
CAS number: 57158-29-9
Registration number: [redacted]
Submission number: [redacted]
Submission date: 18.08.2016
Registered tonnage band: 100-1000T

DECISION ON A COMPLIANCE CHECK

Based on Article 41 of Regulation (EC) No 1907/2006 (the 'REACH Regulation'), ECHA requests you to submit information on

1. **Name or other identifier of the registered substance (Annex VI, Section 2.1.)**
2. **Composition of the registered substance (Annex VI, Section 2.3.)**
3. **Description of the analytical methods on the registered substance (Annex VI, 2.3.7.)**

You are required to submit the requested information in an updated registration dossier by **5 September 2017**. You shall also update the chemical safety report, where relevant.

The reasons of this decision are set out in Appendix 1. The procedural history is described in Appendix 2. Advice and further observations are provided in Appendix 3.

The scope of this compliance check decision is limited to the standard information requirement(s) of Annex VI, Section 2 of the REACH Regulation.

Appeal

This decision can be appealed to the Board of Appeal of ECHA within three months of its notification. An appeal, together with the grounds thereof, shall be submitted to ECHA in writing. An appeal has suspensive effect and is subject to a fee. Further details are described under http://echa.europa.eu/regulations/appeals.

Authorised[1] by Ofelia Bercaru, Head of Unit, Evaluation E3

[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.

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Appendix 1: Reasons

Pursuant to Article 10(a)(ii) of the REACH Regulation, the technical dossier shall contain information on the identity of the substance as specified in Annex VI, Section 2 of the REACH Regulation. In accordance with Annex VI, Section 2 the information provided shall be sufficient to enable the identification of the registered substance.

1. Name or other identifier of the substance (Annex VI, Section 2.1.)

In accordance with Annex VI, section 2.1. of the REACH Regulation, the name and other identifiers reported are required to be sufficient to enable the substance identity to be verified.

Chapters 4.1 and 4.2 of the Guidance for identification and naming of substances under REACH and CLP (Version: 1.4, June 2016), referred to as "the SID Guidance" hereinafter, describe well-defined substances as those with fully defined qualitative and quantitative composition. Each constituent of a well-defined substance requires a complete chemical specification, including structural information. This implies that constituents of well-defined substances must have a unique definitive molecular formula. According to Section 4.1 of the SID Guidance, any variations of composition of well-defined substances are specified by the upper and lower limit of the concentration range(s) of the main constituent(s). Consequently, there cannot be variations in the molecular formula. In comparison, for UVCB substances (substances of Unknown or Variable composition, Complex reaction products or Biological materials) the variability is relatively large and/or poorly predictable.

You identified the registered substance as a mono-constituent substance with IUPAC name "Aluminum zirconium chloride hydroxide".

The EC and CAS entries provided in section 1.1 of the dossier correspond to "Aluminum zirconium chloride hydroxide" with undefined stoichiometry (EC no. 260-599-1 and CAS no. 57158-29-9).

In the molecular formula field in section 1.1 you provided the following formula: "AlyZrClx(OH)(3y+4-x)". The x and y in the formula were explained in section 1.2 of the technical dossier in the composition block "Aluminium zirconium chloride hydroxide" in the "Description of composition" field as follows: "where y varies between [redacted] and x from [redacted] and the percentage basicity defined as 100*(3y+4-x)/(3y+4) is between [redacted] to [redacted]%". You reported information on the manufacturing process in the same field.

You provided the following trade names in the "other identifiers" field in section 1.1: [redacted]. These names may refer to different well-defined substances, as explained in detail below.

In section 1.4 you provided two analytical information blocks, as explained in more detail below in section 3 of the current Appendix. The information in these two blocks is indicating different stoichiometric ranges:

- Information in the first block in the attachment "[redacted]". The attachment includes two sets of specifications for the atomic ratios of elements [redacted]:
  - "USP" quality with the range of [redacted] atomic ratio between [redacted] and the range of [redacted] atomic ratio between [redacted]
  - "Standard" quality with the range of [redacted] atomic ratio between [redacted], and the range of [redacted] atomic ratio between [redacted]

- Information in the second block indicates the range of [redacted] atomic ratio to be between [redacted] and the range of [redacted] atomic ratio between [redacted]

ECHA notes that although you identified the registered substance as a well-defined mono-constituent substance, the registration dossier contains such variations of the molecular formula which do not fulfil the requirements for the unambiguous identification of one well-defined mono-constituent substance.

The molecular formula provided in section 1.1 and the further explanation included in section 1.2 are not specific for a mono-constituent substance: "AlyZrClx(OH)(3y+4-x)" and "where y varies between [redacted] and x from [redacted] and the percentage basicity defined as 100*(3y+4-x)/(3y+4) is between [redacted] to [redacted]%". Such a formula is not specific, because it contains a variable x, which ranges from [redacted] to [redacted] and a variable y, which ranges from [redacted] to [redacted]. This does not fulfil the requirement to provide a specific molecular formula for a well-defined mono-constituent substance, where variations are described by the upper and lower limit of the concentration range(s) of the main constituent(s) only.

ECHA notes that the molecular formula provided could potentially cover different substances with specific stoichiometry within the given stoichiometric range. The molecular formula could as well refer to one (or more) UVCB substances with more narrow stoichiometric ranges than currently described in the registration dossier.

Separate EC and CAS numbers are available for the substances with defined stoichiometries: [redacted] (EC [redacted]; CAS [redacted]), [redacted] (EC [redacted], CAS [redacted]), [redacted] ([redacted], CAS [redacted] and [redacted] ([redacted], CAS [redacted]). The trade names reported in the "other identifiers" may refer to some of these compounds with more specific stoichiometry than what is currently reported for the molecular formula in IUCLID sections 1.1 and 1.2.

The information included in IUCLID sections 1.2 on the description of the composition and manufacturing process and in section 1.4 for the analytical data is also not sufficient to conclude on the molecular formula of the substance registered, as e.g. the ratios of the starting materials are not reported.

Therefore, ECHA notes that the molecular formula and the other information in the dossier are not in line with the identification of the substance as mono-constituent, and do not specify sufficiently the identity of the registered substance. The information could potentially cover different substances with specific stoichiometry within the given stoichiometric range, or one (or more) UVCB substances where compositions of "aluminium zirconium chloride hydroxide" includes compounds of different stoichiometry.

Therefore, you are requested to specify the following information in section 1.1 to unambiguously establish the identity of the registered substance:

- If the substance is a well-defined (mono-constituent) substance of specific stoichiometry, e.g. $Al_8Cl_5(OH)_{23}Zr$, "[redacted]": the information in section 1.1 on the name and other identifiers needs to reflect the specific stoichiometry. The IUPAC name of the substance shall also reflect the exact composition of the substance in terms of stoichiometry of the elements or composite entities (such as polyatomic ions).

- If the substance is manufactured such that the composition is highly variable and, as a consequence, the substance cannot be identified as a well-defined substance, the substance shall be rather identified as UVCB substance. Examples of UVCB substances of this kind are outlined in section 4.3.2.1 of the SID Guidance. Should the substance be identified as a UVCB substance, further information is required to appropriately identify the registered substance in accordance with section 4.3 of the SID Guidance. Information required to be provided on the naming of UVCB substances consists of two parts: (1) the chemical name and (2) a more detailed description of the manufacturing process. The description of the manufacturing process shall be sufficiently detailed to allow ECHA to understand which starting materials are used, and how any other steps and process parameters may affect the substance composition and therefore its identity. It shall include, as appropriate, the ratio of reactants and any relevant operating parameters (e.g. temperature and pressure).

    - Accordingly, you will need to specify a chemical name of the substance that is representative of the registered substance.
    - You will also need to provide the missing details of the process used for the manufacturing of the registered substance, including the ratios of the starting materials and any other information that is missing.
    - If the UVCB substance covered by the registration is manufactured according to different manufacturing processes, including the use of different starting materials, or different starting material ratios, the detailed description of the manufacturing process required above shall be reported separately for each manufacturing process. Any corresponding separate composition block will be included in section 1.2. A manufacturing process may be considered different when the sources, the processing steps and/or processing parameters are different. Please note that according to section 4.3 of the SID Guidance the consequence of defining a substance as UVCB is that any significant change of source or process would be likely to lead to a different substance that should be registered again.

Regarding how to report the requested information in IUCLID the following applies:

- The revised chemical name that is representative of the stoichiometry covered shall be included in the IUPAC name field in section 1.1.
- The revised molecular and structural formula and other identifiers, where relevant, shall be included in their respective fields in section 1.1 of the IUCLID dossier.

- The relevant appropriate CAS entry shall be included in the "CAS information" field, if available. If the current CAS entry (CAS number (57158-29-9) and CAS name (Aluminum zirconium chloride hydroxide) are not appropriate to identify the registered substance (e.g. if the substance has a specific stoichiometry) they shall be reported in the "Other identifiers" –field in section 1.1 of the IUCLID dossier. Similarly where the current EC entry is not appropriate to identify the substance as described above, it will need to be revised. However, for technical reasons you are requested at this stage, not to remove or revise the EC entry in the updated dossier. As this registration is linked to this EC entry in REACH-IT, the IT system will not accept the updated dossier as an update when the EC entry has changed. You are requested to include the following in the "Remarks" field of the reference substance: "This EC entry is not appropriate to identify the registered substance. This identifier cannot be modified in the present registration at this stage for technical reasons".
- The manufacturing process shall be reported in section 1.2 in the "Description of composition" field.
- The details of any separate compositions manufactured with different manufacturing process parameters covered by the registration shall be reported separately in section 1.2 and sufficient analytical data for the composition shall be included in section 1.4.
- If trade names are used in the registration dossier, it needs to be clear to which stoichiometry they refer to.

Further information on how to report the chemical name, the molecular and structural formula, other identifiers and the description of the manufacturing process is available on the ECHA Manual on "How to prepare registration and PPORD dossiers" published on the ECHA website at http://www.echa.europa.eu/manuals.

In the comments to the draft decision according to Article 50(1) you have agreed with the requests in the draft decision. You have agreed to change the status (i.e. type) of the substance to a UVCB. In addition, you have indicated your intention to revise the Sections 1.1, 1.2 and 1.4 of IUCLID and address the information requirement in an update of the registration. ECHA notes that such information, including the adequacy of the information to be provided, will be examined by ECHA only after the deadline set in the adopted decision has passed and all the substance identity information requested in this decision has been submitted.

2. Composition of the substance (Annex VI, 2.3.)

According to Annex VI, section 2.3. of the REACH Regulation, sufficient compositional information is required to be reported in a registration dossier such that the substance identity can be verified.

According to chapter 4.2 of the "Guidance for identification and naming of substances under REACH and CLP" (Version 1.4, June 2016) hereafter referred to as the "SID Guidance", the following applies for well-defined substances:

- Each main constituent (i.e. the constituent present at ≥80% for mono-constituent substance or each constituent present at ≥10% and 80% for multi-constituent substance) shall be identified and reported individually; and
- Each impurity present at ≥1% or relevant for the classification and/or PBT assessment of the registered substance shall be identified and reported individually.

- For each constituent, the typical, minimum and maximum concentration levels shall be specified regardless of the substance type.

As a general rule, the compositional information should be completed up to 100%.

According to chapter 4.3 of the SID Guidance for UVCB substances presenting a large number of constituents, the following applies:

- All constituents present in the substance with a concentration of ≥ 10 % shall be identified and reported individually,
- All constituents relevant for the classification and/or PBT assessment of the registered substance shall be identified and reported individually; and
- Other constituents shall be identified by a generic description of their chemical nature.

Furthermore for each constituent required to be reported individually, the IUPAC name, CAS name and CAS number (if available), molecular and structural formula, as well as the minimum, maximum and typical concentration, should be reported in the appropriate fields in IUCLID. For the other constituents to be reported under a generic description, a generic chemical name describing the group of constituents, generic molecular and structural information (if applicable), as well as the minimum, maximum and typical concentration, should be reported in the appropriate fields in IUCLID.
You provided two composition blocks in section 1.2 specific for your legal entity:

- "[redacted]": Here you reported one main constituent with the same generic reference substance name as in section 1.1: "[redacted]" with typical concentration of [redacted]%(w/w) and a concentration range of [redacted]%(w/w). You reported a generic group "[redacted]" as the only impurity with maximum concentration ≤[redacted] %(w/w). You indicated in the "Description of composition" field: "Typical for marketed substance".

- "[redacted]": Also here you reported one main constituent with the same generic reference substance name as in section 1.1: "[redacted]" with typical concentration of [redacted]%(w/w) and a concentration range of [redacted]%(w/w). No impurities were reported. You indicated in the "Description of composition" field: "[redacted]."

As already explained under Section 1 of this Appendix, the generic identification of the substance as "[redacted]" is not considered sufficient for the unequivocal identification of the substance.

From the limited information in section 1.2 of the IUCLID dossier, and due to the inadequacy in the identifiers of the reference substance, the composition of the substance as a well-defined substance of specific stoichiometric ratio, or as a UVCB substance with variable stoichiometry cannot be established. On the basis of the current information it is also not possible to establish the relevance of any specific compositions of the substance (e.g. separate compositions of more specific stoichiometry than that indicated by the reference substance in section 1.1).

Therefore, you shall revise the information in section 1.2 of the dossier in accordance with the information requested for sections 1.1 and 1.4.

If the substance is a mono-constituent substance with specific stoichiometry, this shall be indicated in the "Description of composition" field. The information in section 1.2 shall be modified accordingly: the name of the main constituent should be more specific to the identity of the substance.

If the registered substance is a UVCB substance composed of constituents with different stoichiometries, this shall be indicated in the "Description of composition" field. The composition shall reflect the UVCB nature of the substance. You are also required to further specify in section 1.2 the identities of the constituents of the substance (e.g. [redacted] compounds of specific stoichiometry). The name and other identifiers for each constituent shall reflect the nature of the constituent(s) and specify the stoichiometry, as relevant. If you consider that it is not possible to report more specific constituent(s) in section 1.2 you shall include a robust scientific justification for this. In this case more specific information on the nature of the constituents shall be reported in the Remarks field of each constituent.

If you cover different compositions of the same substance in a registration, you shall report separately the compositional information of each composition. This means that if the substance covered by the registration has two (or more) different compositions then these must be presented separately in section 1.2 in line with what was indicated above for the manufacturing process. Corresponding analytical data that enables the identitification and quantification of each composition listed in section 1.2 shall be included in section 1.4. All compositions reported are required to refer to the same substance identified in section 1.1 of the dossier.

Further technical details on how to report different compositions and the constituents of a substance in IUCLID are available on the ECHA Manual on "How to prepare registration and PPORD dossiers".

In the comments to the draft decision according to Article 50(1) you have agreed with the requests in the draft decision. In addition, you have indicated your intention to revise the Section 1.2 of IUCLID and address the information requirement in an update of the registration. You indicated that during the update/change of the substance (type) to UVCB, you agree to the adaptation of section 1.2, substance composition. You also indicated that you will provide information on the composition in agreement with the requirements for a UVCB. ECHA notes that such information, including the adequacy of the reported composition, will be examined by ECHA only after the deadline set in the adopted decision has passed and all the substance identity information requested in this decision has been submitted.

3. Description of the analytical methods (Annex VI, 2.3.7.)

According to Annex VI, section 2.3.7. of the REACH Regulation, a registration dossier shall report a description of the analytical methods or the appropriate bibliographic references for the identification of the substance and where appropriate for the identification of impurities and additives. The reporting shall be given in sufficient detail that the methods may be reproduced.

Chapter 4.2.3.3 of the SID Guidance states for inorganic substances, such as the registered substance, that IR, UV and NMR spectral data and GC or HPLC chromatograms are not sufficient for their identification and quantification. Instead, information resulting from other analytical techniques , e.g. X-ray diffraction, elementary analysis etc is needed.

You provided two blocks of analytical results in section 1.4:

- The first block included descriptions of analytical methods such as elemental analysis, and for the sample "[redacted]" you provided specifications that include information e.g. on the [redacted] contents. The attachment "[redacted]" includes two sets of specifications for the atomic ratios of elements [redacted]:
  - Quality "USP" with the range of [redacted] atomic ratio between [redacted] and the range of [redacted] atomic ratio between [redacted]
  - "Standard" quality with the range of [redacted] atomic ratio between [redacted] and the range of ([redacted] atomic ratio between [redacted]

  You also provided an IR spectrum.
- The second block included results of tests performed according to the US Pharmacopoeia (determination of: pH, Arsenic, Heavy Metals, Limit of Iron, Content of aluminum, Content of Zirconium, Content of Chloride). You indicated that "no spectral data will be submitted as it is not suitable for this product". Information in the second block indicates the range of [redacted] atomic ratio to be between [redacted], with the "Tested Result" of [redacted]; and the range of [redacted] atomic ratio between [redacted], with the "tested Result" of [redacted]. You did not indicate if the analyses had been carried out on solution or on a solid sample.

The two analytical information blocks are reporting different ranges for the [redacted] and [redacted] atomic ratios and are therefore pointing to different [redacted] stoichiometries. It cannot be concluded based on this information how the results reported in these two analytical information blocks relate to the identity of the substance reported in section 1.1 or to the composition reported in section 1.2. Also, the information is not sufficient to verify if the samples would relate to different compositions of the same substance.

Regarding the IR spectrum, this alone in the absence of e.g. XRD analysis is not sufficient for an inorganic substance to give information on the identities of the constituents required to be reported in the composition.

Therefore, ECHA considers that the analytical data that you provided does not enable the unequivocal identification and quantification of the substance (including the stoichiometry of the relevant substance constituent(s)).

You shall include information on the methods used to identify and quantify all substance constituents in terms of their stoichiometries and phases (including if they are amorphous, crystalline), where relevant.

You may use any method or combination of methods to do this (e.g. elemental analysis, gravimetry, quantitative XRD, BET, etc.). As a minimum you should provide elemental analysis and an XRD analysis. However, this may not be sufficient for the unequivocal identification and quantification of your substance. It may also be appropriate to consider other suitable methods, such as methods that can give more specific information on the Al-containing species, e.g. ^{27}Al-NMR (nuclear magnetic resonance spectroscopy).

Where separate compositions of the substance are registered, sufficient data that will enable the identity and composition of each composition to be verified shall be included.

You shall note that the description of each method used shall be included in such detail that the methods may be reproduced.

The analytical data, including the description of the analytical methods and the actual results of analysis shall be reported in IUCLID section 1.4. You shall ensure that the composition reported in the dossier is consistent with the analytical results obtained.

In the comments to the draft decision according to Article 50(1) you have agreed with the requests in the draft decision. In addition, you have indicated your intention to revise the Section 1.4 of IUCLID and address the information requirement in an update of the registration. You propose the following methods to be used depending on the form in which the substance is put on the market:
Solid substance: XRD (qualitative and quantitative), ICP-OES (identification and quantification of [redacted] as well as impurities such as [redacted], etc.), Titration (quantification of chloride), Karl-Fischer titration (iwater content), HPLC, IR. For the [redacted] (proposed by ECHA in the draft decision) you included the note: Although this method was proposed by the agency, the (lead registrant) states that this method will not provide any useful information that may be used for identification or quantification of the substance and its constituents (here [redacted]).

ECHA notes that in case when the combination of analytical methods you proposed allows unequivocal identification and quantification of your substance in solid, no complementary methods (such as [redacted] must be applied.

Solutions: ICP-OES (identification and quantification of [redacted] as well as impurities such as [redacted], etc.), Titration (quantification of chloride), UV spectroscopy (depending on the formation of specific chloro complexes with e.g. [redacted]+ in aqueous solution, UV spectroscopy may provide additional information; this will be checked)."

You also noted that "any other standard methods do not appear to be appropriate for inorganic aqueous solutions at first sight".

In addition, you mentioned the following methods in the context of the solutions: HPLC, Specific gravity.

Regarding the information for the solutions, ECHA notes that you should ensure that the level of analytical information provided for solutions is sufficient for its unambiguous identification and also that it is comparable with the data recorded for solid substance. For this purpose ECHA recommends you to perform a XRD analysis recorded on a sample isolated from the solution. The results obtained for the solution should be clearly relatable to the composition in 1.2, which should be reported without a solvent which can be removed without changing the composition of the substance.

Nevertheless, such information, including the adequacy of the information to be submitted, will be examined by ECHA only after the deadline set in the adopted decision has passed and all the substance identity information requested in this decision has been submitted.

Appendix 2: Procedural history

For the purpose of the decision-making, this decision does not take into account any updates of your registration after the date when the draft decision was notified to you under Article 50(1) of the REACH Regulation.

The compliance check was initiated on 11 October 2016.

The decision making followed the procedure of Articles 50 and 51 of the REACH Regulation, as described below:

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

In your comments you agreed to the draft decision. ECHA took your comments into account and did not amend the request(s).

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

As no amendments were proposed, ECHA took the decision according to Article 51(3) of the REACH Regulation.

Appendix 3: Further information, observations and technical guidance

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

2. Failure to comply with the request(s) in this decision, or to fulfil otherwise the information requirement(s) with a valid and documented adaptation, will result in a notification to the enforcement authorities of your Member State.