EUROPEAN CHEMICALS AGENCY

Helsinki, 11 December 2018

Addressee: [redacted]

Decision number: CCH-D-2114453527-44-01/F
Substance name: Benzenesulfonic acid, C14-44-branched and linear alkyl derivs., calcium salts, overbased
EC number: 294-233-7
CAS number: 91696-74-1
Registration number: [redacted]
Submission number: [redacted]
Submission date: 16/04/2014
Registered tonnage band: Over 1000

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 substance (Annex VI, Section 2.1.) of the registered substance;**

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

3. **High-pressure liquid chromatogram, gas chromatogram (Annex VI, Section 2.3.6.) on the registered substance;**

You have to submit the requested information in an updated registration dossier by ***18 March 2019***. You also have to 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 and advice and further observations are provided in Appendix 3.

The scope of this compliance check decision is limited to the standard information requirements of Annex VI, Section 2 to 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, has to 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 ***Jos Mossink***, Head of Unit, Substance Identification and Data Sharing

[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

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

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

"Name or other identifier of the substance" is an information requirement as laid down in Annex VI, Section 2.1 of the REACH Regulation. The name and other identifiers are used to identify the substance in an unambiguous manner and are therefore fundamental for substance identification. Adequate information needs to be present in the technical dossier for the registered substance to meet this information requirement.

ECHA notes that you identified the registered substance as of Unknown or Variable composition, Complex reaction products or Biological materials (UVCB). Information required to be provided according to Annex VI, Section 2.1. of the REACH Regulation on the naming of UVCB substances such as the registered substance shall consist of two parts: (a) the chemical name and (b) a more detailed description of the manufacturing process, as indicated in section 4.3 of the Guidance for identification and naming of substances under REACH and CLP (Version: 2.1, May 2017) – referred to as "the Guidance" hereinafter.

According to the Guidance, the description of the manufacturing process shall include information on the chemical identity of the starting materials and information on the most relevant steps of the process.

Manufacturing process

The information which you have provided regarding the used starting materials and manufacturing process is not sufficient to verify the identity of your substance.

You provided in IUCLID section 1.1 the EC and CAS numbers referring to "Benzenesulfonic acid, C14-44-branched and linear alkyl derivs., calcium salts, overbased", and in the IUPAC name field the following chemical name: "Petroleum, mono-C14-44 branched and linear saturated alkaryl derivatised benzenesulphonic acid calcium salts". In addition, you provided the following description in section 1.1 of the IUCLID dossier: "*The number of carbon units in the alkaryl chain are actually* [redacted] *and considered to be branched and linear saturated chains based upon knowledge of the source of the starting products and manufacturing process and are named based upon the information available from the supplier of the starting material. Analytical data estimates >* [redacted] *wt% of these chain lengths. It is, however, not possible to adequately establish the relative proportion of each of the groups as the isometric ratio of the molecule is highly complex and highly variable.*" No further information was provided on the manufacturing process of the substance.

ECHA notes that you did not specify the exact identity and composition of the alkaryl chain-containing which is used as starting material in the manufacturing process, although in the "Description" you indicated that information on the starting material composition is available from the supplier. ECHA further notes that the information provided on the alkaryl chain length distribution in the "Description" field ([redacted]) is not consistent with the provided identifiers, where the chain length distribution is given as C14-44. As the composition of the starting materials is an important factor determining the composition of the registered substance, it is a necessary element for the identification of the registered substance itself.

Furthermore, the description of the manufacturing process included in IUCLID Section 1.1 does not contain any indication on the identities of other starting materials, or on the ratio of reactants and does not specify the manufacturing process parameters (such as temperature and pressure) which are necessary to obtain the registered substance, or the manufacturing process steps. Specification of the ratio of reactants and identification of process steps and process parameters that may affect the substance composition is essential for the identification of the registered substance.

ECHA therefore concludes that the manufacturing process has not been provided to a sufficient level of detail for the identification of the registered UVCB substance.

Therefore, you shall provide the missing information on the manufacturing process description. This information shall include:

- The exact identities and ratios of all the starting materials used in the process.
- Compositional information on the alkaryl starting material including:
    - the upper and lower concentration levels of the (groups of) constituents presenting the same carbon number, and
    - alkyl chain type (e.g. linear, branched; if relevant)
    - if the starting material used to obtain the overbased calcium salts is the alkaryl derivatised benzenesulphonic acid (as relevant):
        - ratio of mono- and dialkylated constituents
        - ratio of ortho/meta/para isomers (for mono-alkylated constituents)
        - information on whether the alkyl groups are connected to the benzene ring from the terminal carbon and/or from the secondary carbons (i.e. "sec-alkyl").
- Specifications of all relevant process steps and process parameters, including temperature and pressure values, and any other process steps and their parameters including purification step(s) (if any) which are necessary to obtain the registered substance and which may affect the substance composition.

<u>Lube Oil</u>

In addition, the structural formula in section 1.1 of the IUCLID dossier includes "Lube Oil".

However, the identity and concentration of the "Lube Oil" is unclear as no further information regarding this has been provided in the manufacturing process or in the reported composition record. ECHA therefore concludes that the identity of the substance cannot be verified as the identity and role of "Lube Oil" is not clear.

Therefore, you shall provide the missing information on the identity and concentration of the "Lube Oil" identified in the structural formula in section 1.1 of your dossier. This information shall include:

- Role, identities and compositions of the "Lube Oil", as relevant:
    - Role: as explained in more detail below in section 2 of this Appendix in relation to the "Composition of the substance",
    - Identifiers: EC/CAS identifiers and chemical name(s),
    - Composition: as explained in more detail below in section 2 of this Appendix in relation to the "Composition of the substance";

<u>Chemical name</u>

Furthermore, you have provided in IUCLID section 1.1 for the substance the EC and CAS numbers 294-233-7 and 91696-74-1, respectively, corresponding to "Benzenesulfonic acid, C14-44-branched and linear alkyl derivs., calcium salts, overbased". In the IUPAC name field you provided the chemical name "Petroleum, mono-C14-44 branched and linear saturated alkaryl derivatised benzenesulphonic acid calcium salts".

As ECHA notes in the paragraphs above, the information on the alkyl descriptor provided in the Description in section 1.1 of the IUCLID dossier is not consistent with the EC and CAS identifiers and with the chemical name. ECHA notes that this provided information on the alkaryl chain is not consistent with the provided identifiers. The composition and the information on the alkaryl chain is a necessary element for the identification of the registered substance itself. In addition, the identifiers do not mention the "lube Oil", and it is not clear how the calcium carbonate is taken into account in the name (if present in the substance, as explained below under "Composition of the registered substance").

Therefore, you shall clarify the information on the alkaryl chains and on the alkyl descriptor in general. As indicated above this information shall include information on the alkyl chain lengths, alkyl chain type, mono- and/or dialkylation, the connection of the alkyl chain to the benzene ring, and isomer distribution (as relevant).

Regarding the "lube Oil" ECHA notes that if a substance contains a solvent that cannot be removed without affecting the stability of the substance or changing the composition, the solvent becomes part of the substance, needs to be reported in the composition in section 1.2 as constituent and depending on the content may need to be included in the substance name.

You shall also clarify how the calcium carbonate (if present in the substance) is addressed in the chemical name.

If the currently provided chemical name is not specific for the registered substance, you shall provide a new chemical name. The chemical name should reflect the identity and composition of the registered substance in terms of the aspects addressed above, and in the section "Composition of the registered substance" below.

As for the reporting of the information in IUCLID 6, the manufacturing process description for the registered substance shall be reported in the "Description of composition" field in IUCLID 6 section 1.2.

All information in the dossier needs to refer consistently to one substance.

If there is the need to revise the chemical name, and the current EC and CAS identifiers do not sufficiently describe the composition and identity of the substance, you shall:

- Report the appropriate chemical name in the IUPAC name field in section 1.1;
- If the CAS number is not appropriate to describe the substance which is the subject of the current registration, you shall delete from the dossier the CAS information currently assigned to the substance and provide instead any available CAS information specifically corresponding to the substance. If you deem it appropriate, you can however specify the current CAS information as "related CAS information" for the registered substance;
- In case the current identifiers are not appropriate to describe the registered substance, you should not remove or modify at this stage the EC entry 294-233-7 for technical reasons, the registration being linked to that EC entry in REACH-

IT. To ensure unambiguous identification of the registered substance, you should however indicate, in the "Remarks" field of the reference substance in IUCLID section 1.1, the following: "The EC number 294-233-7 currently assigned does not specifically correspond to the registered substance. This identifier cannot be modified or deleted at this stage in the present registration update for technical reasons". You should also specify, in the same "Remarks" field, any available and appropriate EC number for the substance. Any available CAS entry for the registered substance should be reported under the "CAS information" header of the reference substance in IUCLID section 1.1.

You should note that ECHA has established a process, subject to certain conditions, enabling registrants to adapt the EC identifier of an existing registration, while maintaining the regulatory rights already conferred to the substance concerned.

Pending the resolution of the non-compliances addressed in the present decision, any possible adaptation of the identifier can only become effective once ECHA is in a position to establish unambiguously the identity of the substance intended to be covered by you with this registration. Should the information submitted by you as a result of the present decision enable ECHA to identify the substance unambiguously and result in a need to modify the identifier of the substance, the process of adapting the identifier will be considered relevant. In that case, ECHA will inform you in due time as to when and how the identifier adaptation process shall be initiated.

In any case, you should note that the application of the process of adapting the identifier does not affect your obligation to fulfil the requirements specified in this decision.

Further technical details on how to report the identifiers of UVCB substances in IUCLID 6 are available in the Manual "How to prepare registration and PPORD dossiers" on the ECHA website.

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

Identity and composition of alkyl benzenesulfonate derivatives

According to Article 3(1) of the REACH Regulation, a substance is defined as a chemical element and its compounds in the natural state or obtained by any manufacturing process, including any additive necessary to preserve its stability and any impurity deriving from the process used, but excluding any solvent which may be separated without affecting the stability of the substance or changing its composition.

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

- All known constituents and 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.

In the registration dossier you have identified the registered substance as a UVCB substance and specified in IUCLID section 1.2 that the substance contains [redacted]% of the registered substance "Benzenesulfonic acid, C14-44-branched and linear alkyl derivs., calcium salts, overbased". You provided the following description for this generic group of constituents: "*The number of carbon units in the alkaryl chain are actually* [redacted] *and considered to be branched and linear saturated chains based upon knowledge of the source of the starting products and manufacturing process and are named based upon the information available from the supplier of the starting material. Analytical data estimates >* [redacted] *wt% of these chain lengths. It is, however, not possible to adequately establish the relative proportion of each of the groups as the isometric ratio of the molecule is highly complex and highly variable*."
In the analytical report "[redacted].pdf" attached in IUCLID section 1.4 you indicate that "*The chromatogram shows the substance is predominantly* [redacted]."

Furthermore, the results included in the attachment "[redacted].pdf" indicate signals due to the "[redacted]" and "[redacted]". In the attachment "[redacted].pdf" in IUCLID section 1.4 you mention that "*The actual structures vary greatly to include linear and branched species as well as dialkyl species at C32 and greater. These results suggest that both mono- and dialkylated constituents are present in the substance.*"

The constituents resulting from the chemical transformations are essentially reported under the generic group in the composition section of the IUCLID dossier. Additional information was provided in the remarks indicating that the alkaryl chain length is actually [redacted] and the chains are considered to be both branched and linear. In addition, the analytical information included in section 1.4 indicates that more specific compositional information is available on the constituents in the substance.

However, no further information has been provided in section 1.2 on the identities and concentrations of the specific constituents covered by the group of constituents "Benzenesulfonic acid, C14-44-branched and linear alkyl derivs., calcium salts, overbased". E.g. no information was provided on the upper and lower concentration levels of different alkylbenzenesulfonate blocks presenting the same carbon number, on the ratios of the mono- and dialkylated constituents, or on the ratios of ortho/meta/para isomers.

Therefore, you are requested to report the composition of your subtance with sufficient information on the identities and concentration of the constituents of the substance. If it is not possible to identify and report each constituent separately in section 1.2 of your dossier, you may group the constituents. It may not be possible to provide detailed information on constituents and the composition of the substance on the basis of analysing the registered substance. In such case, in order to enable the identification of the substance, you should compensate this lack of information by providing information on composition in section 1.2 of the IUCLID dossier on the basis of the manufacturing process and the starting materials composition.

You shall ensure that the reported composition is consistent with the description of the process used for the manufacturing of the registered substance, including the identity of the starting materials used. You shall also ensure that the composition is verifiable and therefore supported by a description of the analytical methods for the quantification of the constituents required to be reported, as required under Annex VI, section 2.3.7., including the results from these methods. Considering the complexity of the substance, (additional) compositional information can be provided on the basis of the starting materials composition and the manufacturing process.
Therefore, you are accordingly requested to complete and correct the information provided on the composition of the registered substance. You shall report:

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

The identification of these other constituents must be provided for ECHA to establish the composition of the substance as manufactured and to use the compositional information as one identifier for the registered substance.

For constituents/groups of constituents that correspond to "Benzenesulfonic acid, mono-[redacted]-alkyl derivs., calcium salts", the following information is required:

- Relative content of the different alkyl benzenesulfonate derivatives according to the carbon number of the alkyl chain, and if relevant, according to the carbon backbone type (branched/linear);
- Content of monoalkylated and dialkylated constituents (such as the [redacted] and [redacted] mentioned in the HPLC results)
- The overall ratio of ortho/meta/para isomers;
- Information on if in the constituents the alkyl chain is connected from the terminal carbon to the benzene ring, or from a secondary carbon (sec-alkyl)
- Any other constituents required to be reported separately, and currently covered by the generic group of constituents.

<u>Lube Oil</u>

In addition, as noted in section 1 of Annex 1, the structural formula included IUCLID section 1.1 includes "Lube Oil". However, you did not report any constituent referring to "Lube Oil" in the composition record in section 1.2.

If the "Lube Oil" is part of your substance composition it should be reported in section 1.2 of the IUCLID dossier and you are requested to provide necessary information regarding the identity and concentration of this constituent.

Therefore, you shall clarify the identity and concentration of "Lube Oil" which is indicated in the structural formula in section 1.1. You are requested to provide EC/CAS identifiers and chemical name(s) for Lube Oil as well as report typical concentration and concentration ranges for this consituent. If the "Lube Oil" has a variable composition (UVCB-type) you should provide information of its composition based on UVCB requirements.

Calcium carbonate

Furthermore, the structural formula in IUCLID section 1.1 analytical results included in section 1.4 suggest that calcium carbonate is present in the substance. CaCO3 is included in the structural formula, ICP-analysis in IUCLID section 1.4 shows the presence of calcium (attachment "[redacted].pdf"), and the IR spectrum includes signals assigned to carbonate (attachment "[redacted].pdf". The identifiers of the substance (EC, CAS, chemical name) and the generic composition in 1.2 do not include specifically calcium carbonate.

If calcium carbonate is present in the substance, you need to report this in section 1.2 of your dossier.

Therefore, you shall clarify if calcium carbonate is present in the substance, and (if appropriate) report it in section 1.2. If calcium carbonate is present in your substance you are requested to report the minimum, maximum and typical concentration, in the appropriate fields in IUCLID. In addition, you are requested to report if the calcium carbonate is present in the substance as particles or not. In the case calcium carbonate is present in the form of particles; you are also requested to report information on the nature of these particles (e.g. size and size distribution).

All the above information shall be included in section 1.2 of the registration dossier.

You shall ensure that the reported composition is consistent with the description of the process used for the manufacturing of the registered substance, including the identity of the starting materials used. You shall also ensure that the composition is verifiable and therefore supported by a description of the analytical methods for the quantification of the constituents required to be reported, as required under Annex VI, section 2.3.7., including the results from these methods. Considering the complexity of the substance, (additional) compositional information can be provided on the basis of the starting materials composition and the manufacturing process. If the provided analytical results are obtained in the presence of "Lube Oil" this should be clearly stated in the report.

Further technical details on how to report the composition of UVCB substances in IUCLID are available in the Manual "How to prepare registration and PPORD dossiers" on the ECHA website.

3. High-pressure liquid chromatogram, gas chromatogram (Annex VI, Section 2.3.6.)

According to Annex VI, section 2.3.6 of the REACH Regulation, the registration needs to contain a high-pressure liquid chromatogram (HPLC) or a gas chromatogram (GC).

You have provided in section 1.4 results of LC-MS and HPLC analysis in the attachments "[redacted]" and "[redacted].pdf". However, no peak table(s) based on the results was provided. Also, no other alternative quantification results were provided.

The provided information is not sufficient to fulfil the information requirement as you have not provided a peak table or other alternative quantification results that would confirm the composition of your substance.

Therefore, you are requested to submit a GC or an HPLC chromatogram and the required chromatographic data (retention times, peak areas/results and quantifications) that supports the identification and/or quantification of the substance.

You shall ensure that the description of the analytical methods used for the recording of the chromatographic data is specified in the dossier, in line with the requirements under Annex VI section 2.3.7. This information shall be sufficient for the methods to be reproduced and shall therefore include details of the experimental protocol followed, any calculation made and the results obtained. You shall ensure that the provided analytical information is sufficient to verify the composition of the substance reported in section 1.2.

The information shall be included in section 1.4 of the registration dossier.

Appendix 2: Procedural history

An informal call with you was held on 19 December 2016, giving you the opportunity to revise substance identity issues. During the call you agreed to update your dossier addressing substance identity issues by 27 February 2017. As no update was received by the agreed deadline, ECHA contacted you by email and phone and you agreed to provide an updated dossier. However, to date ECHA has not received your updated dossier.

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 28 September 2017.

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.

ECHA did not receive any comments by the end of the commenting period.

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 requests in this decision, or to otherwise fulfil the information requirements with a valid and documented adaptation, will result in a notification to the enforcement authorities of your Member State.