EUROPEAN CHEMICALS AGENCY

Decision number: CCH-D-2114309036-60-01/F Helsinki, 28 September 2015

DECISION ON A COMPLIANCE CHECK OF A REGISTRATION PURSUANT TO ARTICLE 41(3) OF REGULATION (EC) NO 1907/2006

For 2-Butenoic acid, 4-oxo-4-(tridecylamino)-, (Z)-, branched, EC No 283-219-6 (CAS No 84583-68-6), registration number: [redacted]

Addressee: [redacted]

The European Chemicals Agency (ECHA) has taken the following decision in accordance with the procedure set out in Articles 50 and 51 of Regulation (EC) No 1907/2006 concerning the Registration, Evaluation, Authorisation and Restriction of Chemicals (REACH Regulation).

I. Procedure

Pursuant to Article 41(1) of the REACH Regulation ECHA has performed a compliance check of the registration for 2-Butenoic acid, 4-oxo-4-(tridecylamino)-, (Z)-, branched, EC No 283-219-6 (CAS No 84583-68-6), submitted by [redacted] (Registrant). The scope of this compliance check decision is limited to the standard information requirement of Annex VI, Section 2 of the REACH Regulation.

This decision is based on the registration as submitted with submission number [redacted], for the tonnage band of 100 to 1000 tonnes per year. This decision does not take into account any updates after 17 April 2015, i.e. the date when the draft decision was notified to the Registrant under Article 50(1) of the REACH Regulation.

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

The compliance check was initiated on 16 March 2015.

On 17 April 2015 ECHA sent the draft decision to the Registrant and invited him to provide comments within 30 days of the receipt of the draft decision.

On 22 May 2015 ECHA received comments from the Registrant on the draft decision.

The ECHA Secretariat considered the Registrant's comments. The information is reflected in the Statement of Reasons (Section III) whereas no amendments to the Information Required (Section II) were made.

On 23 July 2015 ECHA notified the Competent Authorities of the Member States of its draft decision and invited them pursuant to Article 51(1) of the REACH Regulation to submit proposals for amendment of the draft decision within 30 days of the receipt of the notification.

As no proposal for amendment was submitted, ECHA took the decision pursuant to Article 51(3) of the REACH Regulation.

II. Information required

A. Information in the technical dossier related to the identity of the substance

Pursuant to Articles 41(1), 41(3), 10(a)(ii) and Annex VI, Section 2 of the REACH Regulation the Registrant shall submit the following information for the registered substance subject to the present decision:

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

Pursuant to Articles 41(4) and 22(2) of the REACH Regulation the Registrant shall submit to ECHA by **4 January 2016** an update of the registration dossier containing the information required by this decision, including, where relevant, an update of the Chemical Safety Report.

III. Statement of reasons

Pursuant to Article 41(3) of the REACH Regulation, ECHA may require the Registrant to submit any information needed to bring the registration into compliance with the relevant information requirements.

A. Information in the technical dossier related to the identity of the substance

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.

(a) Description of the analytical methods is a formal information requirement of Annex VI Sections 2.3.7 of the REACH Regulation.

According to Annex VI Section 2 of the REACH Regulation, for each substance, the information given in this section shall be sufficient to enable each substance to be identified.

ECHA notes the following:

The Registrant provided an analytical report in section 1.4 of the IUCLID dossier in support of the composition of the registered substance. The reported composition in section 1.2 includes:

[redacted]

The composition information therefore shows that the substance includes constituents that bear C13 branched alkyl chains and show the specific Z stereoisomerism around the double bond present in the structure.
ECHA notes the following in relation to the analytical report provided:

- The Registrant has included a gas chromatogram (pages 12-14 of the analytical report) and the relative peak table showing a number of peaks. The

area % of the two largest peaks sum up to approximately [redacted]%. These peaks have not been identified in the report.

- The above-mentioned gas chromatogram (pages 12-14 of the analytical report) shows also a number of peaks identified as "[redacted]" having a cumulative area percentage of approximately [redacted]%. However, ECHA notes that in the composition information of the IUCLID dossier the Registrant reported the presence of "[redacted]." at a maximum concentration level of [redacted]% (w/w). The inconsistency between the maximum concentration level reported in the composition and the result of the chromatographic analysis has not been explained by the Registrant.

- The Registrant has provided the result of an HPLC analysis (pages 27-30 of the analytical report). The identification of the peaks shown in the chromatogram is missing in the analytical report.

- The Registrant has also provided a separate LC/MS analysis (pages 31-35 of the analytical report) that identifies the main peaks of the given chromatogram. The constituents identified by the Registrant correspond to structures bearing C11, C12, C13 and C14 alkyl chains. These results together with the result of the HPLC analysis (page 27-30 of the analytical report) indicate that the registered substance includes predominantly groups of constituents having C12 and C13 alkyl chains. However a clear description of the method including calculations used for quantifying these constituents has not been provided.

- The Registrant has provided a quantitative ^{1}H-NMR method with an internal standard in the analytical report in section 1.4 (pages 10-12 of the analytical report). In the analytical report, the Registrant stated that the results of the quantitative method provide the content of a group of constituents "[redacted]". However the utilized quantitative NMR method is unable to differentiate between alkyl chains with different length (e.g. C12 and C13) of the substance. It is therefore not clear how the quantification of this group of constituents was carried out.

- The result of a ^{13}C-NMR analysis is also included in the analytical report (pages 24-25). Such analysis refers to the substance *2-Butenoic acid, 4-oxo-4-(tridecylamino)-, (Z)-, branched*. The spectrum shows four signals in the region from 120 ppm to 140 ppm that have been assigned to the two carbon atoms corresponding to the double bond. The reason why four signals would be generated in this region is not clear, considering that the spectrum refers to a substance showing Z stereoisomerism. On the basis of this information it cannot be concluded that the registered substance consists only of constituents showing the specific Z stereoisomerism. In addition the Registrant indicated: "The spectrum shows the expected signals for a mixture of isomers with the given chemical compound". Such statement creates ambiguity as it may refer to the isomerism relating to the branched alkyl chains with different lengths or to the E/Z stereoisomerism around the double bond.

Based on the analytical information attached in section 1.4, the registered substance includes constituents bearing C11, C12, C13 and C14 alkyl chains.
Information on how these groups of constituents have been quantified has not been

included in the analytical report provided.

In addition, the information given is not sufficient to conclude whether the registered substance consists only of constituents showing the specific Z stereoisomerism around the double bond or if E stereoisomers are also present in the registered substance.

Furthermore the result of the chromatographic analysis does not support the concentration level of the group of constituents "[redacted]" reported in section 1.2 of the IUCLID dossier.

Therefore, the information provided is not sufficient to support the quantification of the groups of constituents required to be reported in the IUCLID dossier.

The Registrant is accordingly requested to provide a description of the analytical methods used for the identification and quantification of the constituents and groups of constituents required to be reported in the composition of the registered substance.

In particular, the Registrant shall describe the method used to quantify the groups of constituents bearing alkyl chains of different chain lengths. In addition, a clarification on the E/Z stereoisomerism around the double bond shall be given. If different stereoisomers are present in the substance, a description of the method used for quantifying the groups of constituents corresponding to the different stereoisomers shall be provided.

The description 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.

Taking into account the complexity of the composition of the registered substance, information on the identification and quantification of its groups of constituents may be derived by combining information on the manufacturing process and results of the qualitative and quantitative analysis of the starting materials.

The Registrant is also requested to provide clear and consistent information that is sufficient for the quantification of the group of constituents "[redacted]"

The Registrant outlined in the comments to the draft decision how he intends to address the information requirement, description of the analytical methods Annex VI Sections 2.3.7. by providing three documents entitled "[redacted]", analytical reports "[redacted]" and "[redacted]". In these attachments, the Registrant has provided comments to the draft decision and has as well provided new information that has not been available earlier.

Irrespective of whether the newly provided information may be sufficient to meet the information requirement addressed in this decision, ECHA can already point out the following:

- The information in the comments would seem to be in line with the expectations in the decision. However, the information to be provided by the Registrant will be assessed on the basis of the updated dossier.

- Regarding ECHA's observation on the presence of two unidentified peaks in the gas chromatogram provided, the Registrant explained in his comments how these peaks have been identified. ECHA notes that the Registrant does not propose to provide such explanation in the registration dossier. A clarification on how the two peaks have been identified is necessary for determining how the composition of the registered substance was derived and shall be included in the IUCLID dossier.

- The Registrant provided information on how he intends to address the observation made by ECHA in relation to the quantification of the groups of constituents present in the registered substance. The Registrant proposed to include information on the fraction of the individual homologues together with an explanation of the calculations in the updated registration dossier.

  When addressing such issue, the Registrant also stated "*Feedback from ECHA as to whether the GC/FI-MS from 1 OL00059 or the LC/UV data from 11 L00486 shall be used to this purpose is welcome.*"

  ECHA notes that the two methods proposed lead to partially distinct results. The Registrant shall derive the composition on the basis of the most appropriate method that shall be chosen taking into account experience and knowledge of the robustness of the method used. When reporting the composition of registered substance the Registrant shall take into account the uncertainty of the results obtained. The Registrant shall ensure that the concentration levels of the constituents present in the substance are not underestimated.

- The Registrant provided a comment addressing the request for clarifying the E/Z stereoisomerism of the registered substance. In his comment, the Registrant stated: "*....Indeed, from the NMR-data, no conclusions can be made regarding the stereoisomerism of the homologues constituting the main component. Thus, neither can a quantitation of the ratio of stereo-isomers be obtained.*"
  ECHA points out that information on how the Registrant concluded on the presence of E and Z stereoisomers is necessary for establishing the identity of the registered substance. As specified in the draft decision, such information may be based also on data derived from the starting materials and from manufacturing process information.

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

The Registrant assigned EC number 283-219-6, CAS number 84583-68-6, and an IUPAC name corresponding to "2-Butenoic acid, 4-oxo-4-(tridecylamino)-, (Z)-, branched" in section 1.1 of the registration dossier. ECHA observes that such name

refers to a substance of Unknown, or Variable Composition, or of Biological origin (UVCB substance) that consists of a multitude of constituents showing different branched alkyl chains with the same carbon chain length.

Information required to be provided according to Annex VI section 2.1 of the REACH Regulation on the naming of UVCB substances shall consist of two parts: (1) the chemical name and (2) a more detailed description of the manufacturing process, as indicated in chapter 4.3 of the Guidance for identification and naming of substances under REACH and CLP (Version: 1.3, February 2014) - referred to as "the Guidance" thereinafter.

The chemical name "2-Butenoic acid, 4-oxo-4-(tridecylamino)-, (Z)-, branched" assigned to the registered substance indicates that it corresponds to a substance which includes constituents showing C13 branched alkyl chains and showing the specific Z stereoisomerism around the double bond present in the structure

ECHA notes, however, that based on the analytical information attached in section 1.4 of the IUCLID dossier, the registered substance includes constituents bearing C11, C12, C13 and C14 alkyl chains. As explained in section III (a) of the present decision, the chromatographic information provided indicates that the registered substance includes predominantly groups of constituents having C12 and C13 alkyl chains.

In addition, the analytical data provided indicates that the registered substance may include groups of constituents showing not only Z but also E stereoisomerism around the double bond. The Registrant shall note that according to the Guidance substances consisting of E and Z isomers are regarded as different substance than substances consisting of the individual isomers (cf. chapter 5 "Criteria for checking if substances are the same", example of "1,2-Dibromoethylene" on page 45).

Therefore, the information provided on the identifiers of the registered substance is not consistent with the analytical data attached in section 1.4 of the IUCLID dossier.

The Registrant is accordingly requested to revise the chemical name and ensure that it is representative of the specific substance which is the subject of this registration and is consistent with the analytical data attached in section 1.4 of the IUCLID dossier.

In particular, reference to the correct E/Z stereoisomerism shall be made in the chemical name of the registered substance.

Furthermore, for substances such as the registered substance that consist of one or more alkyl group(s) with variable alkyl chain attached to a functional group, the name shall represent the actual overall contribution of these groups in the composition. For this purpose, constructing the chemical name on the basis of the main alkyl groups having the same carbon number (i.e. those alkyl groups present at an upper concentration level > 10% (w/w)) is appropriate as long as they altogether compose at least 80% (w/w) of the registered substance. In case of constituents with alkyl groups, each present with an upper concentration level >10%, that altogether do not compose ≥ 80% (w/w) of the substance, all alkyl groups, regardless of their concentration, should be considered for the naming of the registered substance.

ECHA notes that the Registrant has stated that the technological process is "Chemical reaction" in section 3.1. The Registrant has not provided any other information on the manufacturing process.

The identity of the starting materials used to produce the registered substance has not been described. Other elements of the manufacturing process description which are essential for the identification of the registered substance are also missing from the dossier. In particular, the ratio of reactants used, the nature of any catalyst used, specifications of any other relevant manufacturing process parameters, and relevant isolation steps have not been indicated.

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

In line with the above observation, the Registrant shall provide the missing information on the manufacturing process description. This information shall include:

- Information on the starting material in terms of identity
- Ratio of reactants
- Solvent used
- Specifications of nature of any catalyst used and the process parameters that are determinant for the composition of the registered substance
- Relevant isolation steps

As for the reporting of the information in IUCLID, the chemical name and manufacturing process description shall be specified in the "IUPAC name" and "Description" field in IUCLID section 1.1, respectively.

In the comments to the draft decision concerning name or other identifier of the substance (Annex VI, 2.1.), the Registrant indicated his intention to update the composition information stating "*according the information of the analytical reports, the substance will be renamed as an UVCB substance in an update*" of the registration.

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

Annex VI, section 2.3 of the REACH Regulation requires that each registration dossier contains sufficient information for establishing the composition of the registered substance and therefore its identity.

In that respect, according to chapter 4.3 of the Guidance, for UVCB substances presenting a large number of constituents, such as the registered substance, 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.

In the present dossier, the Registrant reported the groups of constituents [redacted]

The presence of other constituents was not indicated in the reported composition.

In relation to the group of constituents "2-Butenoic acid, 4-oxo-4-(tridecylamino)-, (Z)-, branched" ECHA observes that the name provided indicates that this group contains only C13 branched alkyl chains and a double bond where the relative orientation of the functional groups is Z. However, as explained in section III (a) of the present decision ECHA notes that

- based on the analytical information given in section 1.4, the registered substance includes constituents bearing C11, C12, C13 and C14 alkyl chains. Furthermore, the chromatographic information provided indicates that the registered substance includes predominantly groups of constituents having C12 and C13 branched alkyl chains.

  Groups of constituents having C11, C12 and C14 branched alkyl chains have not been reported in the composition information of the IUCLID dossier.

- the ^{13}C-NMR analysis provided in the analytical report indicates that the registered substance may include groups of constituents showing not only Z but also E stereoisomerism around the double bond that have not been reported in the composition information.

The Registrant shall note that 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.

In relation to the group of constituents "[redacted]." ECHA notes that the Registrant reported an upper concentration level corresponding to < [redacted]% (w/w). However, the GC/FID analysis provided in section 1.4 (page 13 of the analytical report) indicates that the registered substance may include significant concentration levels of solvent up to approximately [redacted]%.

The Registrant shall note also that to the extent that the quantity of "[redacted]" reported in the composition is not a solvent which can be removed without affecting the stability of the substance or changing its composition, its constituents shall be regarded as constituents of the registered substance. Accordingly, if the solvent is present in the substance in significant concentration levels according to the analytical data, the identity and concentration level of the constituents and groups of constituents originating from this solvent need to be specified to reflect as far as possible the actual identity of the solvent. This information has not been reported in section 1.2 of the IUCLID dossier.

ECHA notes also that the Registrant provided the result of a gas chromatographic analysis showing the presence of naphthalene at a concentration level equal to [redacted]% (w/w). Naphthalene is subject to harmonised according to Annex VI of Regulation (EC) No 1272/2008 (CLP Regulation). According to CLP Regulation, harmonized classification for naphthalene as aquatic acute 1 is triggered at a concentration level of 0.1%. According to the Guidance constituents relevant for the classification of the registered substance shall be identified and reported individually. ECHA notes that naphthalene has not been reported in section 1.2 of the IUCLID dossier.

Furthermore the Registrant provided the result of a GC/MS analysis (page 15-17 of the analytical report) that indicates the presence of a "[redacted]" in the registered substance. Such group of constituents has not been reported in section 1.2 of the IUCLID dossier.

ECHA therefore concludes that the compositional information has not been provided to the required level of detail.

The Registrant is accordingly requested to correct the information provided on the composition of the registered substance. The Registrant shall report

- All constituents present in the substance with a concentration of ≥ 10 %
- All constituents relevant for the classification and/or PBT assessment of the substance, including naphthalene and
- Other constituents shall be identified by a generic description of their chemical nature. For the constituents that shall be identified by a generic description of their chemical nature the reporting of the constituents according to groups presenting the same carbon number and the same stereochemistry (i.e. E/Z stereoisomerism) is necessary for ECHA to establish the composition of the substance

If "[redacted]" is a solvent that cannot be removed without affecting the stability of the substance, its identification must here again 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 the hydrocarbon constituents originating from the "[redacted]", the reporting of the different aromatic constituents presenting the same number of aromatic cycles in their structure (mono-aromatic, di-aromatic, ...) is necessary as a baseline for ECHA to establish the composition of the substance. For each group of constituents, quantitative information on the carbon number distribution shall also be specified to conclude on the compositional profile of the constituents within the group. In addition any specific constituent that is relevant for the classification and/or PBT assessment of the substance shall be reported.

The Registrant shall note also that if "[redacted]." contributes extensively to the composition the chemical name of the registered substance shall refer to the presence of "[redacted]." to reflect as far as possible its actual identity.

Regarding how to report the composition in IUCLID, the following applies:

The Registrant shall indicate the composition of the registered substance in IUCLID Section 1.2.

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, shall be reported in the appropriate fields in IUCLID. Information on

the carbon number distribution within the relevant groups of constituents shall be specified in the Description field of the reference substance for that group.

Further technical details on how to report the composition of UVCB substances in IUCLID are available in paragraphs 2.1 and 2.2.2 of the Data Submission Manual – Part 18: How to report the substance identity in IUCLID 5 for registration under REACH (version: 2.0, July 2012) on the ECHA website.

In the comments to the draft decision concerning composition of the substance (Annex VI, 2.3.), the Registrant indicated his intention to update the composition information stating "*The composition of the substance will be adapted to the UVCB requirements and will be submitted in a dossier update.*"
In addition, the Registrant provided

1. A clarification on the observation made by ECHA in relation to the inconsistency between the reported concentration level of the group of constituents "[redacted]", and the supporting analytical data included in the registration dossier.

2. A clarification on the detection of the group of constituents "[redacted]" in the sample used for carrying out the GC/MS analysis.

Irrespective of whether the newly provided information may be sufficient to meet the information requirement addressed in the decision, ECHA can already point out the following: the information in the two above-mentioned clarifications would seem to be in line with the expectations in the decision in relation to the two groups of constituents. However, the information to be provided by the Registrant will be assessed on the basis of the updated dossier.

IV. Information on right to appeal

An appeal may be brought against this decision to the Board of Appeal of ECHA under Article 51(8) of the REACH Regulation. Such an appeal shall be lodged within three months of receiving notification of this decision. Further information on the appeal procedure can be found on ECHA's internet page at http://www.echa.europa.eu/regulations/appeals. The notice of appeal will be deemed to be filed only when the appeal fee has been paid.

Authorised[1] by Claudio Carlon, Head of Unit, Evaluation E2.

[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.