Helsinki, 23 July 2019

Addressee: [redacted]

Decision number: CCH-D-2114476329-37-01/F
Substance name: Quaternary ammonium compounds, C12-C18 (even numbered) alkyltrimethyl chloride
List number: 939-616-8
CAS number: NS
Registration number: [redacted]
Submission number: [redacted]
Submission date: 20/05/2013
Registered tonnage band: 100-1000

## 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. **Robust study summary (RSS) for "Biodegradability" by [redacted] (1989), Ready biodegradability (Annex VII, Section 9.2.1.1. in conjunction with Annex I, Section 3.1.5);**

   **OR**

   **Ready biodegradability (Annex VII, Section 9.2.1.1.; test method: CO2 evolution test, OECD TG 301B) or**

   **Ready biodegradability (Annex VII, Section 9.2.1.1.; test method: MITI test (I), OECD TG 301C) or**

   **Ready biodegradability (Annex VII, Section 9.2.1.1.; test method: Closed bottle test, OECD TG 301D) or**

   **Ready biodegradability (Annex VII, Section 9.2.1.1.; test method: Manometric respirometry test, OECD TG 301F) or**

   **Ready biodegradability (Annex VII, Section 9.2.1.1.; test method: Ready biodegradability – CO2 in sealed vessels (headspace test), OECD TG 310) with the registered substance.**

You have to submit the requested information in an updated registration dossier by ***30 January 2020***. 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 and advice and further observations are provided in Appendix 3.

EUROPEAN CHEMICALS AGENCY

## 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 **Claudio Carlon**, Head of Unit, Hazard Assessment

[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. Ready biodegradability (Annex VII, Section 9.2.1.1.)**

In accordance with Articles 10(a) and 12(1) of the REACH Regulation, a technical dossier registered at 100 to 1000 tonnes per year must contain, as a minimum, the information specified in Annexes VII to IX to the REACH Regulation. The information to be generated for the dossier must fulfil the criteria in Article 13(4) of the same regulation.

"Ready biodegradability" is a standard information requirement as laid down in Annex VII, Section 9.2.1.1. of the REACH Regulation. Adequate information on this endpoint needs to be present in the technical dossier for the registered substance to meet this information requirement.

Pursuant to Articles 10(a)(vii) of the REACH Regulation, the information set out in Annexes VII to XI must be provided in the form of a robust study summary, if required under Annex I. Article 3(28) of the REACH Regulation defines a robust study summary as a detailed summary of the objectives, methods, results and conclusions of a full study report providing sufficient information to make an independent assessment of the study minimising the need to consult the full study report. Guidance on the preparation of the robust study summaries is provided in the ECHA Practical Guide 3: 'How to report robust study summaries'.

In the technical dossier you have provided the following study record to fulfil the standard information requirement of Annex VII, Section 9.2.1.1.: Key study, reliability 2, "Biodegradability" by [redacted] (1989), GLP compliance: not specified, test method: according to OECD Guideline 301D (OECD TG 301D) with the analogue substance Coco alkyl trimethyl ammonium chloride (EC no. 263-038-9).

ECHA notes that you have not provided sufficient information in the technical dossier to allow assessment of reliability of the study. In particular the OECD 301D specifies that:

- the difference between the extremes of replicate values for the removal of the test chemical, at the plateau or at the end of the test, should be less than 20%. In the test guideline description of your robust study summary, your report the following deviation: "*the course of the oxygen decrease was measured in one bottle using a special funnel*". From this statement, ECHA understands that no replicate experiments were conducted. Accordingly, ECHA cannot verify that the above validity criteria was passed;
- Oxygen depletion in the inoculum blank should not exceed 1.5 mg/L after 28 days. ECHA notes that no data on the inoculum blank are reported.

Considering the above mentioned deficiencies in reporting, ECHA cannot verify whether the validity criteria have been fulfilled for the key study.

In your comments on the draft decision, you clarified that triplicate test vessels were used and that the variability of measured oxygen concentrations among replicates was within an acceptable range. You also agreed with ECHA that this study did not fulfil the validity criteria of OECD TG 301D. Firstly, the oxygen consumption after 28 days in the inoculum blank exceeded 1.5 mg/L which you indicate is due to a "*high number of micro-organisms in the inoculum leading to high endogenous respiration*". As further discussed below, ECHA notes that the information provided by you does not allow verification that an adequate inoculum density was used. Secondly, the data provided as part of your comments shows that the residual oxygen concentration after 28 days in the test bottles containing the test material was extremely low (i.e. 0.1 mg/L). Accordingly, ECHA notes that this study fails to meet

another validity criteria of OECD TG 301D (i.e. residual oxygen concentration in all test bottles > 0.5 mg/L). You consider that the low residual oxygen concentration in the bottles containing the test material should not invalidate the study and uphold your conclusion that the substance should be regarded as readily biodegradable.

Considering the failure to meet the validity criteria listed above and also further deficiencies detailed below, ECHA disagrees with your conclusion.

ECHA notes that, contrary to Article 3(28) of the REACH Regulation the documentation of the study is insufficient and does not allow an independent assessment of the adequacy of the study, their results and use for hazard assessment. In particular, in addition to the above, the following elements are not reported:

a. In the test guideline description, your report the following deviation: "*ammonium chloride was omitted from the medium to prevent nitrification*". However, ECHA notes that the test substance contains nitrogen. According to OECD 301D, corrections for uptake of oxygen by nitrification should be made. ECHA notes that you did not report such a correction in your study summary;
b. In the test material description, you report that the test was conducted using Coco alkyl trimethyl ammonium chloride (CAS 61789-18-2, EC no. 263-038-9), 33% in water. ECHA notes that you did not identify this endpoint as being covered by read-across to an analogous substance. In addition, you did not provide sufficient data on the test material identity (i.e. carbon chain length distribution, presence of impurities);
c. You did not report the inoculum concentration at the start of the experiment. As specified in OECD TG 301D the inoculum concentration should be ≤ 5 ml effluent/L or $10^4$-$10^6$ cells/L. Accordingly, ECHA cannot verify that the test conditions were compliant with OECD TG 301D and that the results are reliable;
d. You did not report all results from control and test conditions (and their replicate, if any) in a tabular form.

In your comments on the draft decision regarding point a., you indicate that ammonium chloride is omitted from the test medium to prevent oxygen consumption by nitrifying bacteria and to lower the endogenous oxygen consumption in the BOD (Biochemical Oxygen Demand) bottles. Accordingly, ECHA understands that you agree that nitrification of ammonium can occur which justifies the need to take into account the uptake of oxygen by nitrification as ammonium can also be released via degradation of the test substance (you state that "*Organic nitrogen is always liberated by microorganisms as ammonium when nitrogen is present as primary amine (amino group), secondary amine group, tertiary amine or quaternary ammonium group*"). This raises further uncertainty on the reliability of the study given that the endogenous respiration in the control bottles was far above the limit value of 1.5 mg/L despite the absence of ammonium chloride from the medium.

You also provided a calculation of the $ThOD_{NH3}$ (Theoretical Oxygen Demand without nitrification) and $ThOD_{NO3}$ (Theoretical Oxygen Demand with nitrification) of the test substance to support your assertion that correction for nitrification would not impact the conclusion that the substance is readily biodegradable. However, ECHA notes that the provided calculations are incorrect. It appears that the percentage biodegradation was calculated by comparing the BOD at 28 days in mg $O_2$/mg test material to the ThOD in mg $O_2$/mg active ingredient. When both BOD and ThOD are expressed as active ingredient concentrations, the % biodegradation after 28 days is found to be > 200%. This suggests that the study is invalid.

Based on the above, ECHA concludes that a correction for nitrification should be applied as

it may impact the conclusion on ready biodegradability.

Regarding point b., you agree that the technical dossier does not currently address the read-across between the registered substance and the source substance Coco alkyl trimethyl ammonium chloride. You indicate that you intend to address this issue in your next dossier update.
Regarding point c., you agree that this information is missing and you state that "*activated sludge plant was diluted to a sludge concentration in the BOD bottle of 2 mg DW/litre*". You intend to include this information in your technical dossier following a dossier update. ECHA notes that the sludge concentration expressed as mg DW/litre does not allow verification that the prescribed conditions of OECD TG 301D are met (i.e. ≤5 mL secondary effluent/L corresponding to $10^4$-$10^6$ cells/L).

Regarding point d., you indicate that this information is available and that you intend to provide adequate reporting of the test results in a dossier update.

Finally, you indicate in your comments that you intend to update your dossier to include read-across ready biodegradability studies conducted on alkyl trimethylammonium chloride (TMAC) substances (such as Cetrimonium chloride (C16 TMAC; CAS No. 112-02-7; EC No. 203-928-6) or trimethyloctadecylammonium chloride (C18 TMAC; CAS No. 112-03-8; EC No. 203-929-1). As these source substances have longer carbon chain lengths, you consider that they represent reasonable worst cases for this endpoint.

ECHA notes that limited read-across justification is currently provided. Further justification should address the impact of the properties of the source and target substances (e.g. mono-constituent *versus* UVCB) on the reliability of the prediction. For instance, the differences in the properties of the source and target substances may for e.g. impact their bioavailability (e.g. through their adsorptive properties or via differing micellization behaviour). ECHA also notes that, as described below, the information included in your comments indicates deficiencies and/or uncertainties with the selected read across studies.

On the studies with Cetrimonium chloride (C16 TMAC; CAS No. 112-02-7; EC No. 203-928-6) conducted according to OECD TG 301B, ECHA-S notes the following:

- The amount of substance used to conduct the test is unclear. You state in your comments that 30 mg a.i./L was used but in the attached table 30 mg/L test substance is reported. Based on the information from the registration dossier of EC no. 203-928-6 on the same study, the test material is described as containing 28.9% a.i. Accordingly, 30 mg/L of test substance corresponds to c.a. 6.2 mg DOC/L which is below the minimum test substance concentrations allowed in OECD TG 301B. As specified in ECHA Guidance on Information Requirement and Chemical Safety Assessment, Chapter R.7b, section R.7.9.4.1. (Version 4.0 – June 2017) there is already some flexibility with the inoculum and test substance concentrations in Ready Biodegradability Tests and going beyond the limits defined will change the ratio of substance to inoculum in a way that is deemed to be too favourable.
- You specify that the initial cell concentration in the test was within $10^7$ to $10^8$ cells/L but did not justify that the concentration in suspended solid (SS) was ≤30 mg/L.
- You provided insufficient data to verify if the validity criteria of the selected method were fulfilled. More specifically the inorganic carbon (IC) content of the test substance suspension in the mineral medium at the beginning of the test must be less than 5% of the total carbon content (TC).
- The raw data obtained using the test substance are not provided and ECHA could not verify that the 10d window criteria was fulfilled.
- It is unclear if duplicate inoculum blanks were analysed as required according to

OECD TG 301B.

On the studies with trimethyloctadecylammonium chloride (C18 TMAC; CAS No. 112-03-8; EC No. 203-929-1) conducted according to OECD TG 301D, EU C.6 and ISO 10707, ECHA notes the following:

- Based on the information provided by you, ECHA understands that the test was conducted with predominantly domestic sewage sludge and that river water was used as dilution water. While OECD TG 301 states that mixed inoculum may be acceptable, the bacterial density in the test bottle should range from $10^4$ to $10^6$ cells/L. Insufficient information is provided to estimate if the bacterial density in the test was compliant with the corresponding test guideline.
- The study was conducted at 1 mg/L test substance (corresponding to $ThOD_{NO3}$ of c.a. 3.1 mg/L). Accordingly, the test substance concentration was below the minimum concentration required to conduct the OECD TG 301D test (i.e. 2 mg/L test material and 5 mg ThOD/L).
- No correction for nitrification (see also above) is reported.

Hence, the information provided on this endpoint for the registered substance in the technical dossier and in your comments on the draft decision does not meet the information requirement. Consequently, there is an information gap and it is necessary to provide information for this endpoint.

In order to allow an independent assessment of the study submitted, pursuant to Article 41(1) and (3) of the REACH Regulation you are requested to provide complete robust study summary with the above missing elements for the study.

Alternatively, if you cannot submit a complete RSS or the RSS indicates that the study is not reliable as per the criteria indicated above and/or not adequate to fulfil the information requirement, pursuant to Article 41(1) and (3) of the REACH Regulation, you are requested to submit the following information derived with the registered substance subject to the present decision:

Ready biodegradability (Annex VII, Section 9.2.1.1.; test method: CO2 evolution test, OECD TG 301B) or

Ready biodegradability (Annex VII, Section 9.2.1.1.; test method: MITI test (I), OECD TG 301C) or
Ready biodegradability (Annex VII, Section 9.2.1.1.; test method: Closed bottle test, OECD TG 301D) or

Ready biodegradability (Annex VII, Section 9.2.1.1.; test method: Manometric respirometry test, OECD TG 301F) with the registered substance or

Ready biodegradability (Annex VII, Section 9.2.1.1.; test method: Ready biodegradability – CO2 in sealed vessels (headspace test), OECD TG 310).

Depending on the substance profile, you may conclude on ready biodegradability, by applying the most appropriate and suitable test guideline among those listed in the ECHA *Guidance on information requirements and chemical safety assessment, Chapter R.7b* (version 4.0, June 2017) and in the paragraph above. The test guidelines include the description of their applicability domain.

## 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 23 November 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 took into account your comments and did not amend the request.

ECHA notified the draft decision to the competent authorities of the Member States for proposal(s) 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.

3. In relation to the information required by the present decision, the sample of the substance used for the new tests must be suitable for use by all the joint registrants. Hence, the sample should have a composition that is suitable to fulfil the information requirement for the range of substance compositions manufactured or imported by the joint registrants.

   It is the responsibility of all joint registrants who manufacture or import the same substance to agree on the appropriate composition of the test material and to document the necessary information on their substance composition. In addition, it is important to ensure that the particular sample of the substance tested in the new tests is appropriate to assess the properties of the registered substance, taking into account any variation in the composition of the technical grade of the substance as actually manufactured or imported by each registrant.

   If the registration of the substance by any registrant covers different grades, the sample used for the new tests must be suitable to assess these grades. Finally there must be adequate information on substance identity for the sample tested and the grades registered to enable the relevance of the tests to be assessed.