ECHA
EUROPEAN CHEMICALS AGENCY

Helsinki, 23 July 2019

Addressee: [redacted]

Decision number: CCH-D-2114476326-43-01/F
Substance name: Quaternary ammonium compounds, (hydrogenated tallow alkyl)trimethyl, chlorides
EC number: 263-005-9
CAS number: 61788-78-1
Registration number: [redacted]
Submission number: [redacted]
Submission date: 17/05/2013
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. **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.

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.

You have sought to adapt this information requirement according to Annex XI, Section 1.5. of the REACH Regulation by providing a study record for a ready biodegradability study (OECD TG 301D) entitled [redacted] 2010) with the analogue substance Tallowtrimethylammonium chloride (EC no. 232-447-4).

ECHA notes that in your read-across justification document attached to Section 13.2 of your IUCLID dossier you specify that the information requirement for this endpoint is covered using data from C16-C18 and C18 unsat., TMAC (i.e. CAS 68002-61-9). There is an inconsistency between this analogue substance and the analogue substance (CAS 8030-78-2) reported in the study record in Section 5.2.1. of your IUCLID dossier.

In your comments on the draft decision, you explain that the identity of the substance originally referred to as 'Tallowtrimethylammonium chloride' (CAS number 8030-78-2) has been updated to C16-C18 and C18 unsat., TMAC (i.e. CAS 68002-61-9) following the application of the principles described in the OECD Guidance for Characterising Oleochemical Substances for Assessment Purposes (OECD STA 193, ENV/JM/MONO(2014)6). You provide a certificate of analysis of the test material used to conduct the study. The test material is a commercial product containing 48.8% active ingredient (i.e. quaternary ammonium compounds), 15.2% water, 32.9% 2-propanol and 2.1% impurities (including mainly tallow alkyl dimethyl amine, tallow alkyl dimethyl ammonium chloride and tallow alkyl alcohol). The C-chain length distribution of the active ingredient is also provided.

Based on this information, ECHA agrees that, if water and 2-propanol are excluded, the composition of this test material would fit the substance identity profile of C16-C18 and C18 unsat., TMAC (EC number 268-074-9) as registered under REACH. ECHA advises you to update the description of the test material in your technical dossier.

ECHA considers that a read-across between two very similar trimethyl alkyl ammonium chloride substances may be plausible. However, you provided inadequate information on the identity of the test material used in the reported ready biodegradability study to enable ECHA to fully evaluate the adequacy of the proposed read-across.

In addition to the above inadequacy of your read-across adaptation, ECHA notes that the reported study does not provide the information required by Annex VII, Section 9.2.1.1., because it is not valid.

More specifically, ECHA notes the following:

- the test material used to conduct this study is described as "*Tallowtrimethylammonium chloride (TMAC) (49%); 2-propanol (33%); Water (15%)*". ECHA notes that the test material contains a high percentage of 2-propanol and hence the measured O_2 consumption cannot be attributed solely to the biodegradation of Tallowtrimethylammonium chloride. Accordingly, ECHA concludes that the reported study cannot demonstrate that Tallowtrimethylammonium chloride is readily biodegradable and the test is not considered valid.

In your comments on the draft decision, you acknowledge that co-solvents (2-propanol and water) were present in the test material but you indicate that the presence of 2-propanol was accounted for in the ThOD (Theoretical Oxygen Demand) calculation. You also state that "*therefore, the overall percentage biodegradation calculation for the test substance does take into account possible oxygen consumption due to the biodegradation of 2-propanol and does not overestimate the biodegradability of the active*". You further consider that these two carbon sources "*are degraded by different sets of micro-organisms*".

ECHA disagrees that the test does not overestimate the biodegradability of the active substance. First, it is highly likely that the active ingredient (i.e. the quaternary ammonium compounds) and 2-propanol show different degradation kinetics. Available data on 2-propanol indicates that it degrades fast under the conditions of ready biodegradability tests and it may be assumed that 2-propanol was fully degraded by the end of the study period. Accordingly, the % biodegradation of the active substance would *de facto* be overestimated. ECHA considers your assertion that quaternary ammonium compounds and 2-propanol are degraded by different sets of micro-organisms as insufficient to demonstrate that the addition of a significant amount of an easily metabolized source of carbon in the test did not influence the degradation kinetics of the active substance. Furthermore, ECHA emphasizes that the OECD TG 301 and ECHA Guidance on Information Requirement and Chemical Safety Assessment, Chapter R.7b, section R.7.9.1.1 (Version 4.0 – June 2017) specify that the substance being tested should be the sole source of organic carbon for energy and growth.

In addition, ECHA has noted the following discrepancies with the reported study:

a. you specify that river water without particles was used as an inoculum. However, you did not report the concentration of the inoculum in the test vessels. 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;
b. in the test guideline description, your report the following deviation: "*ammonium chloride was not added to prevent oxygen consumption due to nitrification (omission does not result in nitrogen limitation as shown by the biodegradation of the reference compound)*". However, ECHA notes that the test substance contains nitrogen. According to OECD TG 301D, corrections for uptake of oxygen by nitrification should be made. ECHA notes that you did not report such a correction in your robust study summary;
c. 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;
d. you did not report all results from the control and the test conditions in a tabular form.

In your comments on the draft decision regarding point a., you state that "*as per the guidelines, activated sludge, effluent from biological wastewater treatment plant, river water, soil etc. are suggested as inocula*" and that "*the characterization of the inoculum for*

instance by determining the most probable number (MPN) is not obligatory". You consider that the measured endogenous respiration in the inoculum blank of ≤ 1.5 mg/L is sufficient to justify that the bacterial density was appropriate.

ECHA disagrees with this conclusion. First OECD TG 301D specifies that "*the inoculum is normally derived from the secondary effluent of a treatment plant or laboratory-scale unit receiving predominantly domestic sewage*" and that "*an alternative source for the inoculum is surface water*". Hence, this test guideline does not describe a mixture of activated sludge and river water as an appropriate inoculum. Furthermore, Table 2 of OECD TG 301 specifies the general conditions applying to OECD TG 301D, which includes the determination of inoculum density. Finally, ECHA does not consider the measured endogenous respiration in the inoculum blank as sufficient to demonstrate that an adequate bacterial density was used. Indeed, by omitting the addition of ammonium in the mineral media, the endogenous respiration in the incoculum blank is likely reduced. In addition, the measured oxygen consumption also depends on the residual organic matter added with the inoculum and is therefore not a straightforward estimator of bacterial density.

Regarding point b., 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. Based on this comment, ECHA understands that you agree that nitrification of ammonium can occur.

On the need to account for the uptake of oxygen by nitrification, you 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. ECHA agrees that, if the hypothesis of equal degradation kinetics between the active ingredient and 2-propanol is correct, the lack of correction for nitrification would not be sufficient to fail to pass the 60% biodegradation threshold after 28 days. However, if (i) it is assumed that 2-propanol is fully mineralized and (ii) correction for nitrification is applied as recommended by the guideline, the oxygen consumption corresponding to the active ingredient would fall below the 60% biodegradation threshold. In addition, ECHA notes that impurities (2.1% w/w) and the mean C-chain length of quaternary ammonium compounds as reported in the certificate of analysis are not accounted for in the ThOD calculations which further overestimate the % biodegradation.

Regarding point c., you specify in your comments that the data on the inoculum blank were indicated in section 'Details on results' of the study summary record. You indicated that "*the validity of the test was demonstrated by an endogenous respiration of 1.0 mg/L at Day 28*". You provide a table detailing measured values in inoculum blanks. Low variability was observed between the two replicate test vessels. However, as ammonium was omitted from the mineral medium to prevent nitrification, ECHA considers that the reported endogenous respiration values may underestimate the value that would be expected under the standard conditions of OECD TG 301D. Hence, it remains unclear if the validity criteria relative to the oxygen depletion in the inoculum blank would have been passed under the standard conditions of this test.

Regarding point d., you indicate that you will provide the requested information in an update of your technical dossier

Based on the above and taking into account your comments on the draft decision, your adaptation of the information requirement cannot be accepted.

In your comments on the draft decision , you also acknowledge that the presence of the co-solvent might have impacted the accuracy of the determination of the biodegradability of the active ingredient in the above study. Therefore you intend to update your dossier to include a read-across ready biodegradability study with trimethyloctadecyl-ammonium chloride (C18 TMAC; CAS No. 112-03-8; EC No. 203-929-1).

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 substance 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 study.

On the study 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.

As explained above, the information provided on this endpoint for the registered substance in the technical dossier does not meet the information requirement. Consequently there is an information gap and it is necessary to provide information for this endpoint.

Regarding the test method, 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 below. The test guidelines include the description of their applicability domain.

Therefore, pursuant to Article 41(1) and (3) of the REACH Regulation, you are requested to perform one of the following tests 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).

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 20 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.