Helsinki, 23 November 2015

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DECISION ON SUBSTANCE EVALUATION PURSUANT TO ARTICLE 46(1) OF REGULATION (EC) NO 1907/2006

For di-tert-butyl 3,3,5-trimethylcyclohexylidene diperoxide, CAS No 6731-36-8 (EC No 229-782-3)

Addressees: Registrant(s)[1] of di-tert-butyl 3,3,5-trimethylcyclohexylidene diperoxide

This decision is addressed to all Registrants of the above substance with active registrations on the date on which the draft for the decision was first sent for comment, with the exception of the cases listed in the following paragraph. A list of all the relevant registration numbers subject to this decision is provided as an Annex to this decision.

Registrant(s) holding active registrations on the day the draft decision was sent are *not* addressees of this decision if they are: i) Registrant(s) who had on that day registered the above substance exclusively as an on-site isolated intermediate under strictly controlled conditions and ii) Registrant(s) who have ceased manufacture/import of the above substance in accordance with Article 50(3) of Regulation (EC) No 1907/2006 concerning the Registration, Evaluation, Authorisation and Restriction of Chemicals (REACH Regulation) before the decision is adopted by ECHA.

Based on an evaluation by the Federal Institute of Occupational Safety and Health (BAuA) as the Competent Authority of Germany (evaluating MSCA), the European Chemicals Agency (ECHA) has taken the following decision in accordance with the procedure set out in Articles 50 and 52 of Regulation (EC) No 1907/2006 concerning the Registration, Evaluation, Authorisation and Restriction of Chemicals (REACH Regulation).

This decision is based on the registration dossier(s) on 15 April 2015, i.e. the day until which the evaluating MSCA granted an extension for submitting dossier updates which it would take into consideration.

This decision does not imply that the information provided by the Registrant(s) in the registration(s) is in compliance with the REACH requirements. The decision neither prevents ECHA from initiating compliance checks on the dossier(s) of the Registrant(s) at a later stage, nor does it prevent a subsequent decision under the current substance evaluation or a new substance evaluation process once the present substance evaluation has been completed.

I. Procedure

Pursuant to Article 45(4) of the REACH Regulation the Competent Authority of Germany has initiated substance evaluation for di-tert-butyl 3,3,5-trimethylcyclohexylidene diperoxide, CAS No 6731-36-8 (EC No 229-782-3) based on registration(s) submitted by the Registrant(s) and other relevant and available information and prepared the present

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[1] The term Registrant(s) is used throughout the decision, irrespective of the number of registrants addressed by the decision.

decision in accordance with Article 46(1) of the REACH Regulation.

On the basis of an opinion of the ECHA Member State Committee and due to initial grounds for concern relating to suspected PBT/vPvB properties, di-tert-butyl 3,3,5-trimethylcyclohexylidene diperoxide was included in the Community rolling action plan (CoRAP) for substance evaluation to be evaluated in 2014. The updated CoRAP was published on the ECHA website on 26 March 2014. The Competent Authority of Germany was appointed to carry out the evaluation.

In the course of the evaluation, the evaluating MSCA identified additional concerns regarding the relevant exposure to the environment.

The evaluating MSCA considered that further information was required to clarify the abovementioned concerns.

Therefore, it prepared a draft decision pursuant to Article 46(1) of the REACH Regulation to request further information. It submitted the draft decision to ECHA on 18 November 2014.

Registrant(s) commenting phase

On 27 November 2014 ECHA sent the draft decision to the Registrant(s) and invited them pursuant to Article 50(1) of the REACH Regulation to provide comments on the draft decision.

By 19 January 2015 ECHA received comments from the Registrant(s) of which it informed the evaluating MSCA without delay.

By 2 February 2015 and 15 April 2015 the Registrant(s) submitted updates of the registration dossiers. The evaluating MSCA considered the comments received from the Registrant(s) and the dossier updates. On basis of this information, Section II was amended. The Statement of Reasons (Section III) was changed accordingly.

Commenting by other MSCAs and ECHA

In accordance with Article 52(1) of the REACH Regulation, on 11 June 2015 the evaluating MSCA notified the Competent Authorities of the other Member States and ECHA of its draft decision and invited them pursuant to Articles 52(2) and 51(2) of the REACH Regulation to submit proposals to amend the draft decision within 30 days of the receipt of the notification.

Subsequently, Competent Authorities of the Member States and ECHA submitted proposals for amendment to the draft decision.

On 17 July 2015 ECHA notified the Registrant(s) of the proposals for amendment to the draft decision and invited them pursuant to Articles 52(2) and 51(5) of the REACH Regulation to provide comments on those proposals for amendment within 30 days of the receipt of the notification.

The evaluating MSCA reviewed the proposals for amendment received and amended the draft decision.

Referral to Member State Committee

On 27 July 2015 ECHA referred the draft decision to the Member State Committee.

By 17 August 2015, in accordance to Article 52(2) and Article 51(5), the Registrant(s) provided comments on the proposals for amendment. The Member State Committee took these comments into account.

After discussion in the Member State Committee meeting on 15-17 September 2015, a unanimous agreement of the Member State Committee on the draft decision as modified at the meeting was reached on 17 September 2015 and ECHA took the decision pursuant to Article 52(2) and 51(6) of the REACH Regulation.

II. Information required

Pursuant to Article 46(1) of the REACH Regulation the Registrant(s) shall submit the following information using the indicated test methods (in accordance with Article 13 (3) and (4) of the REACH Regulation) and the registered substance subject to the present decision:

1. Water solubility
   A water solubility test using the shake flask method (EU A.6/OECD 105). Slow stirring conditions shall be used in accordance with EC A.23, OECD (TG) 123 (2006)). In addition to the clear aqueous phase the remaining test solution in the sample container as well as a solution of any test substance (by using a suitable solvent) remaining in the sample container after disposal of the original test solution shall be analysed.

2. Simulation degradation testing

   2 A. Simulation testing on ultimate degradation in surface water (test method: Aerobic mineralisation in surface water – simulation biodegradation test, EU C.25/OECD 309, pelagic test – without additional suspended solids/sediment) as specified in Section III.

   In case the test is technically not feasible due to analytical limitations and this is scientifically justified or the result of the test under request 2A does not allow to conclude that the registered substance is very persistent (vP) according to Annex XIII, 1.1.1/1.2.1 of the REACH Regulation, the following is required:

   2 B. Sediment simulation testing (Aerobic and anaerobic transformation in aquatic sediment systems, EU C.24./OECD 308) as specified in Section III.

Pursuant to Article 46(1) of the REACH Regulation the Registrant(s) shall also submit the following information regarding the registered substance subject to the present decision:

3. Environmental Exposure Assessment: Further information on environmental exposure with regard to the registered substance for all uses and all life cycle steps mentioned in the registration dossiers.

Deadline for submitting the required information

Pursuant to Article 46(2) of the REACH Regulation, the Registrant(s) shall submit to ECHA an update of the registration(s) containing either the information requesting to requests of 1, 2A, 2B, and 3 by **30 August 2018**, or the information requests of 1, 2A or 2B, and 3 by **2 March 2018**, required by this decision[2], including robust study summaries and, where relevant, an update of the Chemical Safety Report.

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[2] The deadline set by the decision already takes into account the time that registrants may require to agree on who is to perform any required tests and the time that ECHA would require to designate a registrant to carry out the test(s) in the absence of the aforementioned agreement by the registrants (Article 53(1) of the REACH Regulation).

III. Statement of reasons

Based on the evaluation of all available information on the registered substance further information is required in order to complete the assessment of whether the substance is PBT or vPvB.

1. Water solubility

The Registrant(s) provided three calculated values for water solubility, a water solubility study in accordance with EEC A.5 and two tests according to the water accommodated fraction (WAF) method.

In the water solubility study (flask method) a value of < 0,57 mg/L after 24 hours with decomposition of the substance was determined. The decomposition of the substance was explained with hydrolysis. However, based on a new hydrolysis study ([redacted]) and solubility information ([redacted]) submitted by the Registrant(s) in response to ECHA's draft decision, the substance is considered as hydrolytically stable. Taking this into account the reliability of the water solubility study is questionable.

The first WAF study shows that the T_0 concentrations achieved using the WAF method (ranging between 20 and 100 µg/L for 1 mg/L WAFs between 7 and 80 µg/L for 0.1 mg/L WAFs) were found to be much higher than the concentrations in treated samples, either filtered (7-14 µg/L) or centrifuged (<2 to 4 µg/L). This study indicates emulsification of the registered substance and adsorption to the centrifugation tubes. From the given data in the study it can be concluded that the formation of an emulsion is highly influencing the results of water solubility testing.

The second WAF study was performed to demonstrate that the loss during the centrifugation step was due to precipitation of undissolved test substance and not to adsorption to the centrifuge tubes. A full mass balance study was performed on a 0.1 and a 1 mg/L WAF and on centrifuged and non-centrifuged samples prepared from those WAFs. In the mass balance equation which accounted for 126% and 104% of the test item respectively for the 0.1 and 1 mg/L WAF loadings, adsorption of the substance to the centrifuge tubes was described by the Registrant(s) as a minor route of loss while the vast majority of substance was claimed to be found in non-dissolved (centrifugable) form, as a semi-stable emulsion in the aqueous solutions. However, from the analysed samples of the dissolved test substance and the remaining aqueous solution, adsorption cannot be ruled out without uncertainty as the remaining test solution has been combined before analysis with a solution gained from rinsing the centrifuge tube.

In another study ([redacted]) the influence of the stirring speed over time was assessed. Results demonstrated that the stirring energy can be concluded to be the main factor determining the concentration of dispersed test material present during these studies. At a fixed stirring speed the concentration remains steady and was achieved quickly (after 2 hours (1 hour stirring + 1 hour resting). Higher stirring speeds resulted in higher analytical recoveries that also remained stable whilst the stirring speed was constant. High speed and full speed agitation caused large spikes in the measured concentrations and rest periods resulted in a reversible decrease of the measured concentrations.

Based on these data the Registrant(s) determine a water solubility value of 4 µg/L. However, the very low value of 4 µg/L is not in line with QSAR estimated values (e.g. 34 µg/L (WATERNT program). Furthermore, this value of 4 µg/L matches only with the very high log K_{ow} of 7.56 (KOWWIN, water solubility estimated from log K_{ow}), but the basis for this estimate seems erroneously high.

In summary, a valid water solubility study is not available and with the presented information many uncertainties remain regarding the water solubility of the registered substance. Also studies that clearly indicate the saturation point or explain the loss during centrifugation (precipitation vs adsorption) have not been provided.

Nevertheless, the water solubility is important to assess studies with aquatic exposure, such as the bioconcentration studies.

Available data on bioaccumulation of the registered substance were assessed with the following result: The registered substance has a log K_{ow} of ≥ 6.5 indicating a high bioaccumulation potential. This is supported by available bioconcentration tests using *Cyprinus carpio* (MITI-study).

The bioconcentration test (OECD 305 C = NITE-test) has been performed at two concentrations. Actual measurements showed that the test concentrations are 0.013 and 0.15 mg/L. It appears that the bioconcentration is high (BCF >5000) and comparable for the two concentrations, yielding almost equal kinetic BCF values of around 7000. In the OECD technical guideline the following is mentioned: "Also for substances where the concentrations tested have to be near the solubility limit for practical reasons, testing at least two concentrations is recommended, because this can give insight into the reliability of the exposure concentrations." Effects that would be observed if the water solubility is exceeded would be erroneously low BCF values and a lower BCF in the higher water concentration compared to the lower water concentration. This is because dosing above the water solubility would result in effective dissolved exposure concentrations that are lower than the nominal concentrations. This effect increases when the solubility is exceeded to a larger extent, i.e. at higher exposure concentration. As stated, none of these effects is observed. This could be an indication that the water solubility is at least in the range of the tested exposure concentrations (0.013-0.15 mg/L).

Therefore, pursuant to Article 46(1) of the REACH Regulation, the Registrant(s) are required to carry out the following study using the registered substance subject to this decision: Water solubility test (Flask method, EU A.6/OECD 105) with expansion of the test design to a full mass balance of the test substance.

By way of derogation from the guideline the shake flask method shall be used instead of the column elution method in order to avoid decomposition of the registered substance during evaporation of the solvent which would be part of the preparation of the carrier material for the column elution method.

In addition, during the test suitable stirring conditions (slow stirring in accordance with EC A.23, OECD (TG) 123 (2006)) should be chosen to avoid overestimation due to emulsification.

To assess the study without uncertainty of loss of test substance during centrifugation (precipitation vs adsorption) in expansion of the normal test design a full mass balance of the following samples shall be analysed:

- the clear aqueous phase,
- the remaining test solution in the sample container,
- a solution of any test substance (by using a suitable solvent) remaining in the sample container after disposal of the original test solution.

Dependent on the water solubility a reassessment of the current BCF test and a new BCF test (e.g. OECD 305 I, aqueous test) might be needed.

2. Simulation degradation testing

The Registrant(s) provided two screening tests on biodegradation. According to the test following test guideline OECD 301C the registered substance is not readily biodegradable (0% BOD and 12% elimination measured by GC-analysis after 28 days). In an enhanced ready biodegradability test according to test guideline OECD 301D 37% mineralization was observed after 112 days. It has to be taken into account that the test system might only have been stable up to a test period of 60 days. Therefore, the degradation value at day 56 (27 %) might be the more reliable result of this screening test. Nevertheless, in either case the pass level for ready biodegradability (60%) is failed. Based on the results of the screening tests it cannot be concluded that the registered substance is not persistent. Therefore higher tier simulation tests on biodegradation behaviour of the substance need to be conducted by the Registrant(s) to draw a conclusion regarding the P/vP criteria.

In addition, an anaerobic test on water/sediment system (according to EPA subdivision N pesticide 162-3 (anaerobic aquatic metabolism) with some deviations) was conducted. However, the result is not conclusive and suitable for persistence assessment. Only one water/sediment system was tested instead of two. The water/sediment system was stored seven months before use which may have resulted in a decrease of the microbial activity. Information on the precautions taken to preserve anaerobic conditions during transport, handling, storage and testing is missing and no anaerobic conditions were measured at the start of the test. It is unclear to which percentage the test substance or its metabolites (3,3,5 trimethylcyclohexane (also an impurity) and tert-butanol) adsorbed to sediment. The given half-life of 4.3 days (whole system; only three sampling times (0, 7 and 14 days)) probably reflects only dissipation (e.g. adsorption to organic material or volatilisation). Neither was information given on the extraction method nor on the LOQ. Besides, the long storing (seven months) and pre-incubation period (five weeks) may have changed the composition of the sediment and probably the sediment did not reflect a naturally occurring compartment anymore.

No simulation degradation test under aerobic conditions is currently available.

Hence, because of the insufficient data it is not possible to decide about the P/vP-criteria according to Annex XIII of the REACH Regulation.

The Registrant(s) noted in their comments that the registered substance is only used industrially and always enters a sewage treatment plant prior to release to the environment. Therefore, they proposed a sewage treatment simulation test according to OECD Guideline 303A to determine the level of removal in the treatment plant and to establish if there are indeed emissions to soil, surface water and sediment. The total removal of the substance from the wastewater was claimed to be >99.5%. In the meantime an update of the registration dossier, including an OECD 303A study, was submitted. According to this study the removal was attributed to 15.4% adsorption onto sludge, 82.6% biodegradation and 2% volatilization. Nevertheless, only a draft report summary was submitted. It could neither be reconstructed and evaluated if the poor water solubility was considered (alleged initial concentration of test substance 10 mg/L) nor whether the reported result of biodegradation reflects primary biodegradation or only elimination (i.e. removal from the water phase due to volatilization, sorption and degradation).

The Registrant(s) further noted that the OECD 303A test alone is not sufficient to address persistency concerns and proposed additionally a series of enhanced prolonged closed bottle tests. However, the available screening data, including an enhanced ready biodegradability test, have not proven the substance non-persistent. Based on the available screening test results and considering the properties of the substance, additional biodegradation screening testing (including enhanced prolonged biodegradation testing) is not anticipated to provide any such evidence which would set in question the weight of the available results. Based on REACH Annex XIII, Section 1.1.1, Section 3.2.1 and introductory section, degradation half-lives from degradation simulation tests carried out under relevant conditions in water, sediment and/or soil shall be considered for the assessment of P/vP properties. Such results are produced by OECD 307, OECD 308 or OECD 309 test guidelines. The OECD 303A test does not produce degradation half-lives for these three compartments but for degradation in sludge/biofilm. Therefore, and considering that test conditions provided in the OECD 303A guideline do not represent conditions which could be considered as surrogate of conditions in compartments water, sediment and soil, also the OECD 303A test can only be used as screening level information in the PBT assessment. Considering the available data on the substance, additional screening level information is not needed at this stage.

The modelling of the distribution of the registered substance in a sewage treatment plant (STP) (ECHA REACH Guidance on information requirements and chemical safety assessment Chapter R.16: Environmental Exposure Estimation, Version: 2.1, October 2012) shows the following steady state mass distribution in STP: 6.6% to water, 76% to sludge and 17.4% to air. Furthermore, the modelling of the distribution in the environment based on Level III fugacity modelling (EPIWEB 4.1) shows: once released to the aquatic environment the registered substance will partition predominantly into sediment (95.3%), but also that, despite of the low water solubility and strong adsorption potential of the registered substance, almost 5% will distribute to surface water.

According to Technical Guidance R.7.9 (ECHA REACH Guidance on Information Requirements and Chemical Safety Assessment Chapter R.7b: Endpoint specific guidance, Version 2.0, November 2014) the compartment of concern is to be considered if new data is to be generated. The registered substance has a high adsorption tendency and therefore soil (via STP sludge deposition) as well as sediment are considered to be the compartments of concern. The registered substance is used as regulator for polymerization processes in the manufacture of resins, rubbers, polymers or as a reactive processing aid for various purposes (treatment of articles, production of chemical products etc.). The knowledge on the use and environmental emission profile is currently not detailed, but based on available information it is likely that it may reach surface water, soil and sediment. Direct emission and emission to STPs will result in exposure of surface water. This will also take place as a result of environmental transport and partitioning processes from sediments and soil. Consequently, besides soil and sediment, surface water is also an environmental compartment of concern even though the relative share of the mass distribution to surface water is smaller than to in particular sediment. However, as will be addressed in the following, it is evaluated that results of testing in sediment will be difficult to interpret and therefore not optimal as the basis for drawing a robust conclusion.

The registered substance will adsorb to solid and suspended matter and will most likely form high percentages of Non Extractable Residues (NER). The Registrant(s) have not provided either in their registration dossier or in their comments to the draft decision or in their comments to the PfAs by MSCAs and ECHA any evidence to refute this assumption. Interpretation of NER is, however, not straightforward and still a topic of much scientific and regulatory debate, e.g. because the composition of NER – such as share of parent and metabolites - and the irreversibility of the binding remain unclear. The REACH guidance states that NER is to be considered as removal but also makes clear that removal alone is insufficient for persistence assessment which requires information about degradation (i.e. transformation) (Chapter R.11.4.1.1 of Guidance on Information Requirements and Chemical Safety Assessment Chapter R.11: PBT/vPvB assessment, Version 2.0, November,2014).

For evaluating the registered substance for persistency it is needed to obtain convincing evidence that e.g. dissipation is not only caused by mere adsorption processes but also enough data to be able to conclude about the degradation rate. To this end a test according to OECD 309 mineralisation in surface water in its pelagic version shall be performed. NER formation in such a test is low, which minimize the above mentioned interpretation problems related to the likely NER formation.

Whereas initially parallel simulation testing in accordance with OECD 308 and OECD 309 was proposed, a sequential testing is now requested following several proposals for amendment.

The simulation tests shall be conducted in sequence with the test under 2A (OECD 309) to be conducted first. While the results of the test OECD 309 plays a significant role in the interpretation of the results of OECD 308, this sequential testing may allow the study OECD 308 to be waived. In case the test OECD 309 does not allow to conclude that the registered substance meets the criteria for very persistent (vP) according to Annex XIII, 1.1.1 / 1.2.1 of the REACH Regulation, the test OECD 308 is required.

Furthermore, another PfA was submitted suggesting to request a soil degradation study (test method: Aerobic degradation in soil – simulation biodegradation test, OECD 307) as it is assumed that soil will be the main compartment of exposure. The evaluating MSCA takes the view that additional simulation testing in the soil compartment (OECD 307) might be considered necessary in case neither of the two tests allows to conclude that the registered substance is very persistent (vP) according to Annex XIII, 1.1.1 / 1.2.1 of the REACH Regulation. Therefore, the study could be requested in the follow-up evaluation if necessary.

A further PfA was submitted on the deadline for submission of the requested information. It was suggested to extend the deadline from 21 to 30 months to allow sufficient time for sequential testing. In light of the sequential testing strategy the deadline was extended to either 33 or 27 months depending whether both or only one of the simulation degradation tests are being performed in accordance with the Decision.

In connection to the PfAs relating to the simulation testing strategy, the Registrant(s) suggested conducting a test on inherent biodegradability (Zahn-Wellens-Test) to decide on persistency of the registered substance. Furthermore they suggested conducting a soil / sediment toxicity test according to ISO 10872 (2010) as no toxicity data on soil are available yet. Using the results from the OECD 303A already available the Registrant(s) suggested adjusting the defaults for risk assessment and conducting a risk assessment on soil and sediment afterwards. Based on the result of the risk assessment the appropriate simulation test should be chosen if the results of the Zahn-Wellens-Test show that the substance might be persistent.

Annex XIII of the REACH Regulation distinguishes between screening of P and vP properties and the definitive assessment of P and vP properties — i.e. comparison with the numeric P, B, and T-criteria. In case screening information is indicating a substance might fulfill the PBT- or vPvB-criteria, the Registrant(s) need to provide further information according to Annex XIII 3.2 (see Annex XIII 2.1).

With regard to assessing the P-criterion of the registered substance, all information provided by the Registrant(s) so far indicate persistency. The proposed Zahn-Wellens Test would only add another piece of screening information. There is no indication and it is not scientifically justifiable why another screening test, i.e. Zahn-Wellens Test, should lead to different results. Therefore the Registrant(s) are requested to provide sufficient information to conclude on the P-criterion according to Guidance R11.1.3.1 (Guidance on Information Requirements and Chemical Safety Assessment Chapter R.11: PBT/vPvB assessment, version 2.0, November 2014) by generation of data as described in sections 2A and 2B of the present Decision.

During the Member State Committee discussion it was noted that there may be technical difficulties to conduct the OECD 309 due to the low water solubility of the substance. Therefore, if conducting the OECD 309 is not technically feasible due to analytical limitations and this is scientifically justified the test can be omitted and the OECD 308 shall be conducted immediately.

2A. Simulation testing on ultimate degradation in surface water (test method: Aerobic mineralisation in surface water - simulation biodegradation test, EU C.25/ OECD 309, pelagic test – without additional suspended solids/sediment)

The test system simulates mineralisation in surface water. It either uses surface water only (pelagic test), or surface water with addition of suspended solids or sediment as inoculum (suspended sediment test). It is the aim to test the substance in a test system with a small surface area for adsorption. Thus the pelagic version of the OECD 309 shall be performed.

Based on the above, the test system should be such that NER-formation is kept to a minimum. This is possible by choosing to perform the test in its pelagic version of OECD 309, i.e. without addition of sediment/suspended solids.

It is important that metabolites/ degradation products are identified /sufficiently characterized relative to the PBT properties. To this end the following conditions shall be fulfilled:

- The initial concentration of the substance in the test water should not exceed the water solubility. The registered substance shall be radiolabelled due to its low water solubility for an appropriate verification of the degradation kinetics and pathways. The Registrant(s) shall provide justification for the location of the radiolabel on the molecule.
- The test shall be done as pelagic test without addition of sediment.
- Metabolites shall be identified and/or sufficiently quantified and characterized as regards their PBT properties (at a concentration of ≥ 0.1 % w/w unless it can be demonstrated that this is technically not possible).
- If relevant degradants resulting from the reaction with metal ions should also be considered in the simulation study as this potentially appears to be an important degradation pathway in the environment. An indication that the reaction with metals may be of importance for the registered substance comes from the 21-day Daphnia reproduction study which was adjusted to address concerns with organic peroxides reacting with metal ions.
- The test guideline OECD 309 stipulates a test duration of 60 days but also states that it may be extended to a maximum of 90 days. It further describes that the test duration may be prolonged to several months if the provisions of Annex 3 of the guideline are followed. Annex 3 describes the semi-continuous procedure which shall prevent deterioration of the system by keeping inoculum viable. However, this procedure includes replacement of water with freshly sampled water and may result in loss of a part of the substance. Hence, account of this should be made either in the procedure of the testing and/ or when evaluating the results of the study. In any case test water renewal shall be started at the latest possible time (e.g. after 60 days) and the number of subsequent repetitions of water removal shall be restricted to a minimum. It is necessary to closely check the test concentration just before and

after each test water renewal if this is employed. All procedures which could make interpretation more difficult or make it more difficult to extrapolate to the behaviour of the substance in the environment should be avoided as far as possible.

- Sufficient measurements shall be performed to enhance the possibility of establishing a reliable kinetic modelling. The guideline OECD 309 stipulates that a minimum of 5 sampling points are required during the degradation phase. This refers to the test duration of 60 days, or 90 days if a semi-continuous procedure is used. A tight pattern of measurements at 1, 6, 12 and 24 hours and at day 7, 14, 28 and 56 and at the end of the test shall be made. If the test is longer than 60 days measurements should be made at regular intervals thereafter but for no longer than a month in agreement with the OECD 309 guideline, which states that more measurements can easily be done although it does not give a fixed time schedule.
- The REACH Guidance (cf. Table R.16-9) defines the average environmental temperature for the EU as 12°C and this is the reference temperature for the assessment of persistency in PBT/vPvB assessment. However, an elevated temperature enhances vitality of the inoculum, the rate of metabolite formation and the possibility of metabolite identification as compared to a lower temperature. In order to achieve this, the Registrant(s) are requested to perform the test at 20°C (293K) but to correct back to 12°C (285K) using the Arrhenius equation. Alternatively the kinetic part of the study may be performed at 12°C (285K) with no need for such an Arrhenius normalisation.
- Test evaluation shall be comprehensive (cf. the procedures and approaches usually used for biocides/ pesticides).

To assess persistence it is necessary to differentiate between mere elimination and degradation processes (cf. REACH Guidance R 11.4.1.1). To this end for the registered substance detection and identification of metabolites shall be provided. This is also based on indications in available data.

Pursuant to Article 46(1) of the REACH Regulation, the Registrant(s) are requested to submit the following study on the registered substance subject to this decision unless it is technically not feasible due to analytical limitations and this is scientifically justified: Simulation testing on ultimate degradation in surface water (test method: Aerobic mineralisation in surface water - simulation biodegradation test, EU C.25/ OECD 309, pelagic test – without additional solids/sediment).

2B. Sediment simulation testing (Aerobic and anaerobic transformation in aquatic sediment systems, EU C.24./OECD 308)

In case the OECD 309 test is not technically feasible due to analytical limitations and this is scientifically justified by the Registrant(s) or does not allow to conclude that the vP criterion for water according to Annex XIII, 1.1.1 / 1.2.1 of the REACH Regulation is met, a sediment simulation degradation test (OECD 308) is required.

As indicated, sediment is also a compartment of concern: the registered substance is highly adsorptive and therefore it will adsorb rapidly and to a high degree to sediment particles. A high degree of non-extractable residues (NER) is expected to be generated in the OECD 308 which may cause challenges for proper test data interpretation.

The following conditions of testing should be fulfilled:

- The initial concentration of the substance in the test water should not exceed the water solubility. The registered substance shall be radiolabelled due to its low water solubility for an appropriate verification of the degradation kinetics and pathways. The Registrant(s) shall provide justification for the location of the radiolabel on the molecule.

- Metabolites shall be identified and/or sufficiently quantified and characterized as regards their PBT properties (at a concentration of ≥ 0.1 % w/w unless it can be demonstrated that this is technically not possible).
- If relevant degradants resulting from the reaction with metal ions should also be considered in the simulation study as this potentially appears to be an important degradation pathway in the environment. An indication that the reaction with metals may be of importance for the registered substance comes from the 21-day Daphnia reproduction study which was adjusted to address concerns with organic peroxides reacting with metal ions.
- Test duration is preferred to be prolonged to 120 or even 180 days to facilitate comparison of data with the persistency trigger values. Experience[3] shows that an extension to 120 days or even longer is possible without reducing significance of data even though the test guideline states that test duration normally should not exceed 100 days.
- Measurements shall be done for modelling the degradation kinetics. The guideline OECD 308 stipulates that the number of sampling times should be at least six including zero time for a test duration of 100 days. This is insufficient for a difficult substance like the registered substance, which is expected to adsorb rapidly to sediment. The test regime shall be such that it is possible to follow the adsorption process over time. This is a necessary provision for a successful kinetic modelling when performing the data evaluation because it may be necessary to re-calculate the test concentration and to adequately identify the point in time to use as the starting point the calculation of the half-life. For being able to do this three samples shall be taken on the first day, after 1 hour, 6 and 12 hours; another sample shall be taken after 24 hours followed by sampling times at day 7, 14 and day 28. The following sampling times shall be nearly evenly distributed in a 4 weeks interval. Hence, depending of the total duration of the study, a total of at least 11 sampling time points for a test duration of 180 days shall be included in the study.
- The REACH Guidance (cf. Table R.16-9) defines the average environmental temperature for the EU as 12°C and this is the reference temperature for the assessment of persistency in PBT/vPvB assessment. However, an elevated temperature enhances vitality of the inoculum, the rate of metabolite formation and the possibility of metabolite identification as compared to a lower temperature. In order to achieve this, the Registrant(s) are requested to perform the test at 20°C (293K) but to correct back to 12°C (285K) using the Arrhenius equation. Alternatively the kinetic part of the study may be performed at 12°C (285K) with no need for such an Arrhenius normalisation.
- Test evaluation shall be comprehensive (cf. those usually used for assessment of degradation of biocides/ pesticides). The following aspects are of special interest for test evaluation: Rate and course of kinetics of parent and metabolites in both the sediment and the water phase shall be evaluated. In respect to the water phase results they shall also be compared with the respective results of the OECD 309 study (if the test was conducted) and considered in interpretation. Special consideration shall be given to:
  1. the kinetics in the water phase of both test systems and the differences found.
  2. the kinetics in the water phase compared to the course of NER formation in the sediment phase of the OECD 308,
  3. the time at which metabolites emerge and their succession in the respective test system and
  4. a comparison of the time at which metabolites emerge and their succession in both test systems.

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[3] R&D projects 20667460/03 and *22801*, UBA 2012 and 2013

All of these aspects are needed for the interpretation of the processes observed.

Pursuant to Article 46(1) of the REACH Regulation, the Registrant(s) are required to carry out the following study using the registered substance subject to this decision: Sediment simulation testing (Aerobic and anaerobic transformation in aquatic sediment systems, EU C.24/ OECD 308).

3. Environmental Exposure Assessment: Further information on environmental exposure with regard to the registered substance is required for all uses and all life cycle steps mentioned in the registration dossiers

With the information provided in the registration dossiers the evaluating MSCA cannot conclude on exposure and exposure pathways for the environment from the uses identified by the Registrant(s).

Based on the data provided by the Registrant in an update in April 2015 the evaluating MSCA agrees that classification as hazardous for the environment is not justified. However, the Registrant(s) state in their comments that a hazard has been identified for the sediment based on a chronic sediment study. Therefore, an environmental exposure assessment needs to be provided. The argumentation of some Registrant(s) for waiving the environmental exposure assessment is not comprehensible.

The Registrant(s) state that after manufacturing the substance is used for the formulation of e.g. [redacted]. It is used in the formulation stage as process regulator, used in [redacted] or polymerisation processes.

It is also industrially used as process regulator for polymerisation processes in production of resins, rubbers, and polymers (cross-linking agents, curing agents), for instance during the use of [redacted] or as [redacted] agents in the production of rubbers.

Some Registrant(s) state uses as intermediate under strictly controlled conditions without further specification which uses are intermediate uses. Others exclude intermediate uses. Therefore, the evaluating MSCA cannot retrace in which uses the registered substance is used as intermediate and which uses contribute to exposure of the environment.

For all uses subsequent to manufacture it is stated that the substance is used as such or in a mixture without any further specification in which uses the substance is used as such or in a mixture and without any description of the uses. It remains unclear how the products which are formulated in the life cycle stage "formulation" are used. There is no differentiation in the exposure assessment for the very different formulation processes and uses. The technical function of the substance in the different identified uses is not described and thus it is not clear whether the substance contributes to environmental exposure. Also it remains unclear what kind of articles are treated or produced.

For all uses following formulation it is stated that no subsequent life cycle stages appear. However, according to the description of uses, there is the formulation in materials, the production of articles and the treatment of articles. These uses must have subsequent life cycle steps as e.g. service life or waste stage. Thus, the scope of the exposure scenarios for formulation and industrial uses is unclear.

For all exposure scenarios Registrant(s) used own specific input parameters for emission factors, emission days and dilution factors which deviate from default input parameters provided in the guidelines R12 and R16. Registrant(s) have not provided adequate justification for that, and in particular has not specified the operational conditions and risk management measures that need to be applied. Furthermore, the assignment of tonnages to the different uses is not clear in all exposure scenarios. Assumptions for exposure assessment are not traceable.

For the formulation and industrial uses Registrant(s) assume that the registered substance is rapidly degraded in waste water and thus reduce the emission factor. Taking into account the studies in the registration dossier the evaluating MSCA comes to the conclusion that the substance is not readily degradable. The reduction of the emission factor is therefore not justified. Furthermore, some registrants did not account for the high log Pow and bioaccumulation potential in their environmental exposure assessment.

As the scope of the formulation and industrial uses is unclear and the actual input parameters (emission factor, emission days, dilution factor and tonnages) for the manufacture and all identified uses are not traceable it is not possible to conclude if there is relevant exposure of the environment as well as to humans via the environment and which environmental compartment is affected most.

The modeling of the distribution of the substance in a sewage treatment plant (ECHA REACH Guidance on information requirements and chemical safety assessment Chapter R.16: Environmental Exposure Estimation, Version: 2.1, October 2012; assumption: not readily biodegradable, water solubility 0.004 mg/L) shows the following distribution: 6.6% to water, 76% to sludge and 17.4% to air. The modeling of the distribution in the environment based on Level III fugacity modeling (EPIWEB 4.2) shows: once released to the aquatic environment the substance will partition predominantly into sediment. If the substance is released to soil it will predominantly stay in soil. Therefore sediment and soil are relevant compartments if the registered substance is released to the environment via sewage treatment plant or via sludge application if uses with emission into municipal wastewater treatment plant occur.

Therefore, the Registrant(s) are requested to respectively conduct or revise the environmental exposure assessment taking into account all uses and all environmental compartments covering all life-cycle stages including the waste stage of the registered substance originating from manufacture and all identified uses. The Registrant(s) shall provide full justification for the input parameters used in the exposure scenarios.

For all exposure scenarios the operational conditions, risk management measures and the use shall be described to a level of detail that it is understandable for which purpose the substance is used in which life cycle step, which processes are carried out with the substance and how these processes are operated so that the release is limited to the release factor reported by the Registrant(s). If additional onsite waste water treatment or waste air treatment is needed to limit the release, the corresponding suitable treatment techniques shall be identified in the exposure scenarios.

The Registrant(s) claim that the substance is highly reactive. If this is confirmed (e.g. from simulation tests), then the assessment should take into account the degradation and/or reaction products.

A proposal for amendment was submitted asking to limit the request for exposure information only to sediment as it is assumed that this is the main compartment of concern taking into account the classification as H241 and the effects observed in the chronic sediment toxicity study. However, the ECHA takes the view that sediment and soil both are relevant compartments if the registered substance is released to the environment via sewage treatment plant or via sludge application. Furthermore, for a potential vPvB substance an exposure assessment for the environment is needed independently of PNECs and hazard information. This information is needed for prioritisation and possible risk management.

IV. Adequate identification of the composition of the tested material

In relation to the required experimental studies, the sample of the substance to be used shall have a composition that is within the specifications of the substance composition that are given by all Registrant(s). It is the responsibility of all the Registrant(s) to agree on the tested material to be subjected to the test(s) subject to this decision and to document the necessary information on composition of the test material. The substance identity information of the registered substance and of the sample tested must enable the evaluating MSCA and ECHA to confirm the relevance of the testing for the substance subject to substance evaluation. Finally, the test(s) must be shared by the Registrant(s).

V. Avoidance of unnecessary testing by data- and cost-sharing

In relation to the experimental studies the legal text foresees the sharing of information and costs between Registrant(s) (Article 53 of the REACH Regulation). Registrant(s) are therefore required to make every effort to reach an agreement regarding each experimental study for every endpoint as to who is to carry out the study on behalf of the other Registrant(s) and to inform ECHA accordingly within 90 days from the date of this decision under Article 53(1) of the REACH Regulation. This information should be submitted to ECHA using the following form stating the decision number above at: https://comments.echa.europa.eu/comments_cms/SEDraftDecisionComments.aspx

Further advice can be found at http://echa.europa.eu/regulations/reach/registration/data-sharing

If ECHA is not informed of such agreement within 90 days, it will designate one of the Registrants to perform the stud(y/ies) on behalf of all of them.

VI. Information on right to appeal

An appeal may be brought against this decision to the Board of Appeal of ECHA under Articles 52(2) and 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 the 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[4] by Leena Ylä-Mononen, Director of Evaluation

Annex: List of registration numbers for the addressees of this decision. This annex is confidential and not included in the public version of this decision.

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[4] As this is an electronic document, it is not physically signed. This communication has been approved according to ECHA's internal decision-approval process.