Helsinki, 01 October 2015

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

For mixture of two components: 1. N-(1,3-dimethylbutyl)-N´-phenyl-p-phenylenediamine 2. N1-(1,3-dimethylbutyl)-N4-(4-(1-methyl-1-phenylethyl)phenyl)benzene-1,4-diamine, CAS No not available (EC No 448-020-2)

Addressee: Registrant(s)[1] of mixture of two components: 1. N-(1,3-dimethylbutyl)-N´-phenyl-p-phenylenediamine 2. N1-(1,3-dimethylbutyl)-N4-(4-(1-methyl-1-phenylethyl)phenyl)benzene-1,4-diamine

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 comments, 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.

Registrants holding active registrations on the day the draft decision was sent for comments 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 Ministry of Economy of the Slovak Republic, department Centre for Chemical Substances and Preparations as the Competent Authority of the Slovak Republic (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 29 April 2014, i.e. the day on which the draft decision was notified to the Registrant(s) pursuant to Article 50(1) of the REACH Regulation.

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 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 the Slovak

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

Republic has initiated substance evaluation for mixture of two components: 1. N-(1,3-dimethylbutyl)-N´-phenyl-p-phenylenediamine 2. N1-(1,3-dimethylbutyl)-N4-(4-(1-methyl-1-phenylethyl)phenyl)benzene-1,4-diamine, CAS No not available (EC No 448-020-2) based on registration(s) submitted by the Registrant(s) and other relevant and available information and prepared the present 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 Human health/CMR; Environment/Suspected PBT; Exposure/High RCR, mixture of two components: 1. N-(1,3-dimethylbutyl)-N´-phenyl-p-phenylenediamine 2. N1-(1,3-dimethylbutyl)-N4-(4-(1-methyl-1-phenylethyl)phenyl)benzene-1,4-diamine was included in the Community rolling action plan (CoRAP) for substance evaluation to be evaluated in 2013. The updated CoRAP was published on the ECHA website on 20 March 2013. The Competent Authority of the Slovak Republic was appointed to carry out the evaluation.

The evaluating MSCA considered that further information was required to clarify the following concerns: Environment/Suspected PBT; Exposure/High RCR. 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 19 March 2014.

On 29 April 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 within 30 days of the receipt of the draft decision.

Registrant commenting phase

By 5 June 2014 ECHA received comments from the Registrant(s) of which it informed the evaluating MSCA without delay.

The evaluating MSCA considered the comments received from the Registrant(s). The information contained therein is reflected in the Statement of Reasons (Section III) whereas no amendments to the Information Required (Section II) were made.

Commenting by other MSCAs and ECHA

In accordance with Article 52(1) of the REACH Regulation, on 5 March 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, five Competent Authorities of the Member States and ECHA submitted proposals for amendment (PfAs) to the draft decision.

On 10 April 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 20 April 2015 ECHA referred the draft decision to the Member State Committee.

By 11 May 2015 ECHA did not receive any comments from the Registrant(s) to the proposals for amendment to the draft decision.

After discussion in the Member State Committee meeting on 8-11 June, a unanimous agreement of the Member State Committee on the draft decision, as modified at the meeting, was reached on 9 June 2015.

ECHA took the decision pursuant to Article 52(2) and Article 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 method and instructions in accordance with Article 13 (3) and (4) of the REACH Regulation and the registered substance subject to the present decision:

1. Dissociation constant (test method: calculation for both components as specified in section III);

2. Water solubility (test method: EU A.6./OECD 105/ as specified in section III);

3. Tiered approach strategy for the assessment of the persistence potential and overall PBT/vPvB potential:

   - Tier 1: Ready biodegradability (test method: Closed bottle test, OECD 301 D); Test shall be performed at the test substance concentration close to the lower value of the test concentration range required according to the test method so that suspected inhibition of microbial activity due to toxicity is avoided. Specific chemical analysis shall be carried out to assess primary degradation of the registered substance and to determine the concentration of main degradation products formed. Correction for oxygen uptake for interference by nitrification, toxicity and abiotic controls shall be performed;

     Tier 2: Soil simulation testing (test method: Aerobic and anaerobic transformation in soil, EU C.23./OECD 307) if the results of the Closed bottle test do not indicate that both components (i.e. N-(1,3-dimethylbutyl)-N´-phenyl-p-phenylenediamine and N1-(1,3-dimethylbutyl)-N4-(4-(1-methyl-1-phenylethyl)phenyl)benzene-1,4-diamine) of the registered substance can be excluded as fulfilling the screening criterion for persistence. Pending on the outcome of Tier 1, the soil simulation study shall be conducted with the registered substance as such or with the relevant component(s) of the registered substance (i.e. N-(1,3-dimethylbutyl)-N´-phenyl-p-phenylenediamine and N1-(1,3-dimethylbutyl)-N4-(4-(1-methyl-1-phenylethyl)phenyl)benzene-1,4-diamine) . The soil simulation test shall be performed at a temperature of 12°C and with at least one soil having a pH < 5. Information on the rate of degradation in soil, identification of degradation products formed and quantity of bounds residues is requested.

   In case neither of the parent component is assessed to be P/vP following the Closed bottle test (e.g. due to rapid primary degradation), the Registrant(s) shall determine what further information is required to conclude the PBT /vPvB assessment of the

degradants.

In view of the new information obtained, the Registrant(s) shall revise the PBT/vPvB assessment including the assessment of degradation products and impurities and update the CSR.

4. Activated sludge respiration inhibition testing (test method: Activated sludge, respiration inhibition test (carbon and ammonium oxidation), OECD 209). The respiration rate regarding carbon oxidation and ammonium oxidation shall be measured. One test shall be performed with freshly prepared test concentrations of the registered substance. Another test shall be performed with five days old test concentrations of the registered substance to allow the generation of hydrolysis products.

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:

5. Exposure assessment:

A quantitative exposure assessment relating to human health for the registered substance for the industrial use in polymer industry covering exposure scenarios for manufacture of synthetic rubber, production of [redacted] and general rubber goods, use of [redacted] and rubber goods based on a quantitative exposure model or monitoring data.

- A quantitative exposure assessment relating to environment for the mixture of two components: 1. N-(1,3-dimethylbutyl)-N´-phenyl-p-phenylenediamine 2. N1-(1,3-dimethylbutyl)-N4-(4-(1-methyl-1-phenylethyl)phenyl)benzene-1,4-diamine for the production and industrial use in polymer industry covering exposure scenarios for manufacture of synthetic rubber, production of [redacted] and general rubber goods, use of [redacted] and rubber goods including waste stage based on a quantitative exposure model or monitoring data. The information required in points 1, 2, 3 and 4 of this decision shall be taken into account for the environmental exposure assessment and risk assessment.

6. Information related to chemical safety assessment/personal protective equipment:

Provide information on the specification of personal protective equipment and the duration of use for all scenarios where the use of personal protective equipment is advised.

In particular:

a) the type of material, thickness and breakthrough times of the gloves and the duration of use for all exposure scenarios where the use of gloves is advised.

b) specifying for air-purifying respirators, the proper purifying element (cartridge or canister), the adequate particulate filters and the adequate masks, or self-contained breathing apparatus for the scenarios where the use of respiratory protection is advised.

Pursuant to Article 46(2) of the REACH Regulation, if the results of the Closed bottle test indicate that both components of the registered substance can be excluded as fulfilling the screening criterion for persistence and therefore the soil simulation testing at point 3 Tier 2 is not required, the Registrant(s) shall submit to ECHA by 24 months from the date of the decision an update of the registration(s) containing the information in points 1, 2, 3 (Tier 1), 4, 5 and 6 required by this decision.

If the results of the Closed bottle test do not indicate that both parent components of the registered substance can be excluded as fulfilling the screening criteria for persistence, the soil simulation testing at point 3 Tier 2 is required and the Registrant(s) shall submit to ECHA by **08 April 2019** from the date of the decision an update of the registration(s) containing the information in points 1, 2, 3 (Tier 1 and Tier 2), 4, 5 and 6 required by this decision. In both cases the update shall include robust study summaries and, where relevant, an update of the Chemical Safety Report.

III. Statement of reasons

Based on the evaluation of all relevant information submitted on the registered substance and other relevant and available information, ECHA concludes that further information is required in order to enable the evaluating MSCA to complete the evaluation of whether the registered substance constitutes a risk for the environment due to suspected PBT and Exposure/High RCR.

1. Dissociation constant

The value of dissociation constant (pKa) is important for the assessment of the registered substance behaviour in environment.

Though the value of dissociation constant is required according to Annex IX of REACH, there is no data regarding dissociation constant in the registration dossier. The Registrant(s) provided justification for waiving the study (the substance is practically not soluble in water). The exact value of water solubility is not yet determined. The calculations of pKa values separately for the component 1: 1. N-(1,3-dimethylbutyl)-N´-phenyl-p-phenylenediamine (CAS No 793-24-8) and for the component 2: N1-(1,3-dimethylbutyl)-N4-(4-(1-methyl-1-phenylethyl)phenyl)benzene-1,4-diamine (CAS No 194478-84-7) of the substance are possible and shall be provided instead of measured values.

According to data from registration documentation for component 1 of the substance (CAS No 793-24-8), the dissociation constants (calculated by using ACD/Labs, v. 7.00) are pKa (HL/H+L) = 6.73±0.32 and pKa (H2L/H+HL) = -0.71±0.40 at 25 °C (ECHA, 2013)[2]. The calculation shows that both the neutral and the mono-protonated forms are present at environmental relevant pH.

In the comment on the draft decision the Registrant(s) state that they fully respect evaluation of the substance by the evaluationg MSCA. However, the Registrant(s) stated that they were not able to give final statement to the draft decision.

ECHA points out that the study of hydrolysis as function of pH indicates that the registered substance undergoes significant abiotic hydrolysis in water compartment under aerobic conditions. Its intensity depends on temperature and pH. The study results show, that component 2 (CAS No 194478-84-7) undergoes the abiotic degradation more slowly compared to component 1 (CAS No 793-24-8) of the registered substance. The values of DT50 less than 12 hours are at pH 7, temperature 15°C and 25°C for both components of the substance and at pH 10, temperature 25°C for component 1 (CAS NO 793-24-8). According to Column 2 of the REACH Annex IX a study does not need to be conducted if the substance is hydrolytically unstable (Half-life less than 12 hours). Therefore the calculations of pKa values separately for the both component of the registered substance shall be provided instead of measured values.

Information on dissociation constant is required in order to enable the evaluating MSCA to

[2] http://echa.europa.eu/

assess the substance behaviour in the environment. The dissociation constant value impacts appropriate interpretation of ecotoxicity results and influences PEC calculation in the risk assessment. Dissociation constant is needed in order to enable the Registrant(s) to consider integrated testing strategy for water solubility according to ECHA guidance for information requirement and chemical safety report, Chapter R7.1.7.

Therefore, pursuant to Article 46(1) of the REACH Regulation, the Registrant(s) are required to provide the following information: Dissociation constant (test method: as specified above and according to the ECHA Guidance for information requirement and chemical safety report, Chapter R7.1.17).

2. Water solubility

The water solubility is an essential parameter in ecotoxicological testing and evaluation. The determination of reliable value of water solubility (for both components of the substance) is essential for the proper risk assessment of the substance.

The registration dossier contains a water solubility study (OECD TG No. 105, Column elution method) for the mixture of two components: 1. N-(1,3-dimethylbutyl)-N´-phenyl-p-phenylenediamine 2. N1-(1,3-dimethylbutyl)-N4-(4-(1-methyl-1-phenylethyl)phenyl)benzene-1,4-diamine. However, the study is of inadequate quality and is not satisfactory according to OECD TG 105. Based on the data given in the original study report, the equilibrium could not be established for the lower flow rate – the concentrations differ by more than ±30% in a random fashion. In addition, the data shows that the measured solubility was higher with the lower flow rate but the test with halving of the flow rate was not conducted. The mean concentration values obtained from two tests with different flows differ by far more than 30%. There is no mention whether hydrolytic stability and acid dissociation constant of the registered substance had been considered in this study. The value of water solubility of the registered substance under the evaluation is stated to be < 1 mg/L for both components (below the limit of detection) at 20°C, pH 6, the exact value of water solubility of the substance is not determined. The study could be considered as limit test performed up to the detection limit of analytical method used. It is necessary to underline that the value of < 1 mg/L for both components was determined only based on the detection of component 1 (CAS No 793-8) as in the performed study the problem with detection of component 2 (CAS No 194478-84-7) was reported.

Considering the hydrolytical instability of the registered substance, the evaluating MSCA used EPI Suite v4.1 (WSKOW v1.42) for estimation of water solubility. Water solubility for component 1 (CAS No 793-24-8) is 1.879 mg/L and for component 2 (CAS No 194478-84-7) is 0.0022 mg/L at 25°C (based on estimated log Kow values). Estimated values at 25°C based on measured/user entered log Kow value are 2.2 mg/L for component 1 (CAS No 793-24-8) and 0.0104 mg/L for component 2 (CAS No 194478-84-7).

According to data from registration documentation for component 1 (CAS No 793-24-8) the water solubility is 1.1 mg/L at ambient temperature, pH was not reported and water solubility at 50°C is circa 1 mg/L, pH was not reported (ECHA, 2013).

The estimations show that the values of water solubility of the components differ considerably (by two and more orders), which was not considered in the water solubility test. The analytical method used was not optimised for as low concentration as needed for identification and quantification of component 2. Developing new analytical method (reliable analytical protocol) is required to measure and quantify both components of the substance in water.

In the comment on the draft decision the Registrant(s) state that fully respect evaluation of the substance by the evaluating MSCA. However, the Registrant(s) stated that they were

not able to give final statement to the draft decision. They asked the experts for the statement regarding the issue of isolation of two components and synthesis of the standard products of decomposition of substance, which is not available on the market. ECHA points out that reliable analytical protocol with a validated limit of quantification (LOQ) is required to measure and quantify low concentration of component 1 (CAS No 793-24-8) and component 2 (CAS No 194478-84-7) in water. ECHA does not require the synthesis of the standard products of decomposition of the mixture.

As the value of water solubility is the crucial parameter for the environmental part of evaluation, reliable value of water solubility of the substance shall be determined by using integrated testing strategy for water solubility according to ECHA Guidance on information requirements and chemical safety assessment, Chapter R.7.1.7. Reliable analytical protocol to measure and quantify both components of the registered substance in water shall be used. To address this requirement, separate tests shall be conducted for the two components. If this is not possible in practice, the Registrant(s) shall explain how the chosen test method is suitable for establishing the water solubilities of both components. The test shall be performed under conditions that ensure that abiotic degradation does not occur during the study. The Registrant(s) shall refer to the difficult substances guidance. For example, pH adjustment may be necessary

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 method: EU A.6./OECD 105/ as specified above).

Notes for consideration by the Registrant(s):
Alternatively, the water solubility of the substance may be substantiated with a documented, reliable and adequate QSAR value; The QSAR must meet the requirements of Annex XI, paragraph 1.3. If the QSAR prediction is not acceptable to the evaluating MSCA then the Registrant(s) would need to conduct a test.

3. Tiered approach strategy for the assessment of the persistence potential and overall PBT/vPvB potential:

- Tier 1: Ready biodegradability (test method: Closed bottle test, OECD 301 D)

  Closed Bottle Test for ready biodegradability performed at lower test substance concentration is required as a first step for clarification of persistency of the substance.

  In the registration dossier there are three tests on ready biodegradation (OECD TG 301 B, 301 D, 301 F) that indicate 46%, 33% and 9% degradation after 28 days, respectively. A modified MITI (II) test (OECD TG 302C) indicates 18% degradation after 28 days.

  However, the biodegradability (ready and inherent biodegradability) studies are of low quality with the exception of the Modified Sturm Test (OECD 301 B) which indicates 46% degradation. This study is of best quality from among the submitted biodegradability studies. The used test substance concentration was 18.2 mg/L; test method requires 10 – 20 mg/L. The used test concentration in Closed Bottle Test was too high (36.4 mg/L – 36. 8 mg/L); test method requires 2 – 10 mg/L, usually 2 – 5 mg/L. The study as such including the reporting is of low quality; although the concerned registrant assigned the study as a key study ECHA considers it as not reliable as a key study for Chemical Safety Assessment (CSA). Similar case regarding the reporting and quality of the study is Manometric Respirometry Study indicating 9% degradation after 28 days; the initial test concentration was 100 mg/L; ECHA rates the study as not reliable study for CSA. As for the Modified MITI (II) test, the study is of low

quality and was not performed in line with OECD TG 302C method. The Registrant(s) did not assign the reliability; ECHA rates the study as not reliable study for CSA.

However, considering that the water solubility of the substance is very low (less than 1 mg/L) the used test concentrations (18, 2 mg/L - 100 mg/L) in all available biodegradability tests are rather high and thus significantly increases the probability of the inhibition to the microbial population.

A clear trend is observed in used test concentrations and biodegradability percentage in the biodegradability studies i.e. the higher the test concentrations, the less the percentage of biodegradability. A possible explanation of the low biodegradation is the formation of hydrolysis products p-benzoquinone/p-hydroquinone which are very toxic to bacteria. Contradictory to this seems to be the value of EC50 > 2000mg/L from activated sludge respiration inhibition test. This test however is regarded as unreliable (performed far above the water solubility, nitrification was not under consideration, the test duration was short , only 3 hours, to enable formation of degradation products inhibiting the microorganisms).

To clarify the potential of persistency, it is required at the first step to repeat the ready biodegradability study (Closed Bottle Test, OECD 301 D) as the basic respiration in this study is the lowest and perform it at a test concentration close to the lower value of the test concentration range recommended in the test guideline (usually 2 – 10 mg/L); so that suspected inhibition of microbial activity due to toxicity be avoided. Considering poor water solubility of the registered substance Annex III of the OECD 301 should be taken into account.

In the OECD 301D test, a toxicity control must be included and if inhibition by test substance is suspected the test should be repeated as instructed in the test guideline, using, e.g., a lower test substance concentration. ECHA further notes that, in Annex II of the OECD 301, it is stated that if the inhibition due to toxicity is to be avoided, it is suggested that the test substance concentrations should be less than 1/10 of the EC_{50} values (or less than EC20 values) obtained in toxicity testing. Therefore it is recommended to the Registrant(s) to conduct activated sludge respiration inhibition test as required in this decision before the ready biodegradability testing.

As the Registrant(s) did not provide any data on degradation products (only possible hydrolysis products have been identified) the study shall be performed with request for specific chemical analysis to assess primary degradation and to determine concentrations of main degradation products.

Correction for oxygen uptake for interference by nitrification shall be conducted as nitrification is expected. Also abiotic controls shall be performed as the registered substance undergoes significant abiotic degradation.

In the comment on the draft decision the Registrant(s) state that fully respect evaluation of the substance by the evaluating MSCA. However, the Registrant(s) stated that they were not able to give final statement to the draft decision. They asked the experts for the statement regarding the issue of isolation of two components and synthesis of the standard products of decomposition of substance, which is not available on the market.

ECHA points out that according to requirement of Annex XIII of REACH (Regulation 253/2011) the identification of PBT/vPvB substances shall take account of the PBT/vPvB properties of relevant constituent of the registered substance and relevant transformation and/or degradation products. If the substance is potentially PBT/vPvB it is proportionate to request to generate/isolate the test substance / targeted

constituents for further testing although it would be difficult. ECHA does not require the synthesis of the standard products of decomposition of the mixture.

Available information on biodegradability of hydrolysis products indicates that p-benzoquinone / p- hydroquinone, acetophenone and aniline are ready biodegradable but 4- hydroxydiphenylamine, benzoquinone-monoimine, benzophenone are not readily biodegradable and can be considered as preliminary fulfilling persistency criterion.

- Tier 2: Soil simulation testing (test method: Aerobic and anaerobic transformation in soil, EU C.23./OECD 307)

In case the results of the Closed bottle test do not indicate that both parent components (i.e. N-(1,3-dimethylbutyl)-N´-phenyl-p-phenylenediamine and N1-(1,3-dimethylbutyl)-N4-(4-(1-methyl-1-phenylethyl)phenyl)benzene-1,4-diamine) can be excluded as fulfilling the screening criterion for persistence, further information to clarify the potential of persistency is needed. If neither parent components is assessed to be P/vP following the Closed bottle test (e.g. due to extensive primary transformation), the Registrant(s) shall determine what further information is required to conclude the PBT/vPvB assessment of the degradants.

According to tonnage level of the registered substance (Annex IX of REACH), simulation testing shall be required to elucidate the potential of persistency of the registered substance (and its degradation products) in relevant environmental compartments.

In the registration dossier there is no reference to any simulation study addressing the degradation half-lives and biotransformation of the registered substance and its degradation products in particular environmental compartments. The Registrant(s) provided exposure based waiving justification for the degradation simulation tests. However, information on uses of the registered substance during the whole life cycle does not provide sufficient evidence to exclude that the registered substance exposes the soil compartment.

The results of adsorption/desorption screening test (OECD TG 121) indicate high adsorption potential of the registered substance (Koc = 3200 for component 1 and Koc= 25000 for component 2) and the likely very low water solubility of especially component 2 will most probably make a degradation simulation test in water technically very difficult/practically impossible.

Furthermore, estimations of the environmental distribution using Level III Fugacity model and PBT profiler[3] indicate the main target environmental compartments for the components of the registered substance are sediment (14% for component 1, 60% for component 2) and, predominantly, soil (76% for component 1 , 38% for component 2). The main target environmental compartment for the possible hydrolysis products is predominantly soil (from 54% to 87%).

Considering the high adsorption potential of the registered substance, the very low water solubility and environmental distribution based estimated high exposure potential to soil from uses of the registered substance during the whole life cycle, soil simulation testing with request for determination of the degradation half-lives and the identification of transformation products of the registered substance shall be required to clarify the potential of persistency.

[3] http://www.pbtprofiler.net

Depending of chemical identity of the transformation products which will be reported by the requested soil simulation test reports, ECHA, on the basis of the evaluation by the evaluating MSCA, may in a follow up evaluation decide whether further soil simulation degradation testing of specific degradation products is required (e.g. to conduct tests for measuring of their primary degradation half-life). This may be required in order to clarify the potential PBT properties of these transformation products – and furthermore, if the persistency is confirmed, adequate tests on bioaccumulation and toxicity (ecotoxicity and mammalian toxicity) may also be required on these degradation products if not available.

Information from soil simulation study is also needed for further clarification of potential for bioaccumulation and biomagnification of the registered substance as well as of the degradation products. Further information on bioaccumulation may be requested after the persistence of the registered substance is clarified.

Considering the overall picture of the PBT potential of the registered substance the T criterion is fulfilled based on Registrant(s) self-classification as Reprotox 1B and STOT RE 1 and is borderline case based on the results of both available aquatic chronic toxicity studies that report NOEC of 0.01 mg/l (based on nominal concentrations). The Registrant(s) concluded in their registration dossier that the T criterion is met.

Summing up the provided reason, persistency potential of the registered substance should be clarified in tiered approach:

Tier 1: Ready biodegradability (test method: Closed bottle test, OECD 301 D); Test shall be performed at the test substance concentration close to the lower value of the concentration range required according to test method so that suspected inhibition of microbial activity due to toxicity is avoided. Specific chemical analysis shall be carried out to assess primary degradation of the registered substance and to determine the concentration of main degradation products formed. Correction for oxygen uptake for interference by nitrification, toxicity and abiotic controls shall be performed;

Tier 2: Soil simulation testing (test method: Aerobic and anaerobic transformation in soil, EU C.23./OECD 307) if the results of the Closed bottle test do not indicate that both parent components of the registered substance can be excluded as fulfilling the screening criterion for persistence; I Pending on the outcome of Tier 1, the soil simulation study shall be conducted with the registered substance as such or with the relevant component(s) of the registered substance; The soil simulation test shall be performed at a temperature of 12°C and with at least one soil having a pH < 5, as at lower pH-values the registered substance is assumed to be hydrolytically more stable. Information on rate of degradation in soil, identification of degradation products formed and quantity of bounds residues is requested.

In case neither of the parent component is assessed to be P/vP following the Closed bottle test (e.g. due to rapid primary degradation), the Registrant(s) shall determine what further information is required to conclude the PBT /vPvB assessment of the degradants.

In view of the new information obtained, the Registrant(s) shall revise the PBT/vPvB assessment including the assessment of degradation products and impurities and update the CSR.
The PBT/vPvB assessment should be conducted in accordance with ECHA Guidance on information requirements and chemical safety assessment, Chapter R.11: PBT Assessment (version2.0., November 2014).

4. Activated sludge respiration inhibition testing

The concern was identified for microorganisms in sewage treatment plant. Important data is missing for hazard and risk assessment in the compartments of sewage treatment plant.

In the registration dossier there is one test of activated sludge respiration inhibition (OECD TG 209). Effect concentrations on the basis of respiration inhibition after 3 hours are EC20=189-745 mg/L, EC50≥2000 mg/L, EC80≥2000 mg/L. However, there are several limitations to this test. Even though in the registration documentation reliability 1 is assigned to the study, the concerned registrant did not use the value of 2000 mg/L for PNEC derivation and reported that there is no reliable standard microbial inhibition test data available as the results of toxicity study to microorganisms are above water solubility.

The test concentration is far above the water solubility of the registered substance (<1 mg/L) and it can be assumed that, due to short test duration, the major amount of the registered substance will be insolubilized and thus not available to micro-organisms. The available data from the registration dossier on ECHA website[4] indicate toxic effect of hydroquinone (as possible hydrolysis product) on aquatic micro-organisms. No data on inhibition of nitrification are presented in the study report, however the inhibitory effect on nitrification might be a sensitive endpoint, as the possible products of hydrolysis (hydroquinone and other quinone-like compounds) might be toxic to aquatic micro-organisms. The data from literature show that nitrification was progressively inhibited as quinone-like compounds concentration was increased, with IC50 values at 1h of exposure time of 3.1 ± 0.5 mg/L for hydroquinone and 2.8 ± 0.4 mg/L for p-benzoquinone as reported by Suárez-Ojeda et al, 2010[5]

The micro-organisms in the sewage treatment plant should be protected to ensure proper waste water treatment. Reliable data on inhibition of nitrification as a probably sensitive endpoint are missing in the registration dossier which results in concern regarding the hazard and risk assessment of sewage treatment plant and its microorganisms. Therefore, toxicity tests on aquatic microorganisms for the registered substance and its hydrolysis products shall be performed. The testing of hydrolysis product is needed as the registered substance undergoes significant abiotic degradation in water compartment under aerobic conditions, leading to the generation of compounds potentially toxic to micro-organisms. One test shall be performed with five days old test item to allow generation of hydrolysis products.

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: Activated sludge respiration inhibition testing (test method: Activated sludge, respiration inhibition test (carbon and ammonium oxidation), OECD 209); The respiration rate regarding carbon oxidation and ammonium oxidation shall be measured. One test shall be performed with freshly prepared test item concentrations of the registered substance. Another test shall be performed with five days old test item concentration to allow the generation of hydrolysis products.

5. Exposure assessment

Based on information in the registration dossier and other relevant and available

[4] http://echa.europa.eu/
[5] Suárez-Ojeda M E, Guisasola A, Carrera J. 2010 Inhibitory impact of quinone-like compounds over partial nitrification. Chemosphere Volume 80, Issue 4, June 2010, Pages 474-480.

information, potential concern regarding Exposure/High RCR cannot be excluded.

The registered substance is used for manufacture of synthetic rubber and subsequently for the production of [redacted] and rubber articles. The exposure part of chemical safety report is primarily focused on production and identified uses, which covers two exposure scenarios: Manufacture of the registered substance (ES1) and industrial use of the registered substance by polymer industry (ES2). For ES1, detailed operational conditions and adequate risk management measures are considered. For ES2, no exact data are available as stated by concerned registrant, however processes in closed system with high level of emission protection or industrial uses with high level of occupational exposure control are considered. The exposure scenarios for production of [redacted] and general rubber goods, use of [redacted] and general rubber goods are not included.

- Exposure assessment relating to human health: Exposure of workers to the registered substance is considered to be minimal since most large industrial users have mechanized materials handling systems. However some operations are carried out in systems of more opened character (coagulation tanks, sieves etc.).
  Regarding the identified use/exposure scenarios, all RCRs related to inhalation and dermal exposure for workers are below 1. The derivation of these values was recalculated based on input data given but results different from those presented by the Registrant(s) were obtained, not supporting the RCRs below 1 for all scenarios presented. Moreover, gaps in the input data and documentation of exposure assessment as well as several uncertainties in the relevant part of CSR, leads to doubts about correctness of results and credibility of risk characterisations of the registered substance for human health (workers). Based on these findings it was concluded that a possible risk for workers after inhalation and dermal exposure in more open systems cannot be excluded. As the registered substance is a non-threshold skin sensitizer, any dermal exposure needs to be avoided.

  The registration dossier shall be amended with relevant information on uses by workers at industrial sites, uses by professional workers and uses by consumers. The exposure scenarios for production of [redacted] and general rubber goods, use of [redacted] and rubber goods covering rubber article shall be included and the exposure assessment and risk characterisation for these scenarios shall be performed. Exposure estimation for workers shall contain description of operational conditions and activities in production of [redacted] and general rubber goods and all information needed for refinement of exposure estimation. Exposure for professional workers for uses of [redacted] mounting and dismounting and handling of technical rubber goods shall be included. Consumer exposure from use of [redacted] or rubber article shall be included only where relevant.

- Environmental exposure assessment: Releases of the registered substance into the environment are expected during production mainly via wastewater and via exhaust gases from processing when this involves heating (formulation) during industrial and professional uses. As mentioned above, the exposure scenario for use of the registered substance in production of [redacted] and rubber articles is not included. The exposure scenario for use of [redacted] and general rubber goods covering the release of abraded [redacted] wear particles via [redacted] are not included although high tonnages of [redacted] wear particles containing residues of the registered substance are expected to be released to the environment. There is no data on leaching of the registered substance from [redacted] wear particles over the time leaching periods, no information on exposure to possible toxic degradation products. There is no information on how the articles are handled in the waste stage and whether this can lead to release of the registered substance or toxic degradation products. Moreover, there is several incorrect/incomplete information in the relevant part of the CSR. In some cases, the values of the same parameter (i.e. PECs,

PNECs) in different tables differ significantly and even by orders of magnitude. Therefore, the CSR shall be amended with proper data for the assessment of environmental exposure. The input parameters for exposure estimation should be reported properly.

The new data regarding water solubility, activated sludge respiration inhibition and degradability need to be taken into account for the subsequent environmental exposure and risk assessment. More detailed data are required for risk assessment of the registered substance and degradation products. One of the major points for clarification is the relevance of degradation products.

Therefore, 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:

- A quantitative exposure assessment relating to human health for the registered substance for the industrial use in polymer industry covering exposure scenarios for manufacture of synthetic rubber, production of [redacted] and general rubber goods, use of [redacted] and rubber goods based on a quantitative exposure model or monitoring data. A quantitative exposure assessment relating to environment for the registered substance for the production and industrial use in polymer industry covering exposure scenarios for manufacture of synthetic rubber, production of [redacted] and general rubber goods, use of [redacted] and rubber goods including waste stage based on a quantitative exposure model or monitoring data. The information required in points 1, 2, 3 and 4 of this decision shall be taken into account for the environmental exposure assessment and risk assessment.

6. Information related to chemical safety assessment/personal protective equipment:

Based on information in the registration dossier and other relevant and available information, potential concern regarding Exposure/High RCR cannot be excluded.

Personal protective equipment (PPE: gloves, goggles and protection) are mentioned, but no characteristics are provided in the dossier. There is a possible risk for workers as inhalation and dermal exposure cannot be excluded. In CSR Section 9 PPEs were considered but not adequately specified. PPE specification is a requirement of Annex II, 8.2.1. and the efficacy is needed to assess residual exposure occurring to workers when PPE are used. Article 14(6) as well as Annex I, 5.2.4 of the REACH Regulation require registrants to identify and apply appropriate measures to adequately control the risks identified in a CSR. The exposure shall be estimated and risks shall be characterised in the CSR under the assumption that relevant risk management measures have been implemented.

Pursuant to Annex VI, section 5 and Annex II, section 0.1.2. of the REACH Regulation the information provided in the registration dossier shall be consistent with that in the Safety Data Sheet (SDS). The requirements of Safety Data Sheets are specified in Annex II of the REACH Regulation. Annex II, section 8.2.2.2. requires the Registrant to describe the relevant RMM in detail (e.g. for hand protection - the type of gloves to be worn shall be clearly specified based on the hazard of the substance or mixture and potential for contact and with regard to the amount and duration of dermal exposure; for respiratory protection - the type of protective equipment to be used shall be specified based on the hazards and potential for exposure including air-purifying respirators, specifying the proper purifying element, the adequate particulate filters and the adequate mask, or self-contained breathing apparatus) in order to minimise the exposure for workers handling the registered substance.

Not all materials are well suited to protect against exposure to all substances, mixtures or materials. This has to be specified further to match the specific substances. A concern is raised if workers are not properly informed to use the right type of e.g. gloves to protect themselves against exposure to chemicals. The use of unsuited material may even result in higher level of exposure, than not using any protection at all, as the inside of contaminated gloves, may be covered with migrated substance — and the skin inside a glove is often humid — corresponding to exposure under occlusion.

Information on the specification of personal protective equipment shall be provided for all scenarios where the use of personal protective equipment is advised.

Therefore, 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:
Information related to chemical safety assessment on the specification of personal protective equipment and the duration of use for all scenarios where the use of personal protective equipment is advised. In particular:
a) the type of material, thickness and breakthrough times of the gloves and the duration of use for all exposure scenarios where the use of gloves is advised. This is of specific concern as the registered substance is a skin sensitizer.
b) specifying for air-purifying respirators, the proper purifying element (cartridge or canister), the adequate particulate filters and the adequate masks, or self-contained breathing apparatus for the scenarios where the use of respiratory protection is advised. This is a specific concern in relation to processes with increased temperature due to increased likelihood of inhalation.

IV. Adequate identification of the composition of the tested material

In relation to the required experimental stud(y/ies), 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. 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://echa.europa.eu/regulations/appeals. The notice of appeal will be deemed to be filed only when the appeal fee has been paid.

Authorised[6] 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.

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