Helsinki, 10 April 2014

Decision/annotation number: Please refer to the REACH-IT message which delivered this communication (in format SEV-D-XXXXXXXXXX-XX-XX/F)

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

For N-(1,4-dimethylpentyl)-N'-phenylbenzene-1,4-diamine, CAS No 3081-01-4 (EC No 221-374-3), registration number: [redacted]

Addressee: Registrant of N-(1,4-dimethylpentyl)-N'-phenylbenzene-1,4-diamine

This decision is addressed to all Registrant(s) of the above substance with active registrations on the date on which the draft for the decision was first sent, with the exception of the cases listed in the following paragraph.

Registrant(s) meeting the following criteria are *not* addressees of this decision: i) Registrant(s) who exclusively use the above substance as an on-site isolated intermediate and 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 Umweltbundesamt GmbH on behalf of the Competent Authority of Austria (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 does not take into account any updates of the Registration of the Registrant after 31 October 2013, the date upon which the draft decision was circulated to the other Competent Authorities of the Member States and ECHA pursuant to Article 52(1) of the REACH Regulation.

This decision does not imply that the information provided by the Registrant in the registrations is in compliance with the REACH requirements. The decision neither prevents ECHA from initiating compliance checks on the dossiers of the Registrant 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 Austria has initiated substance evaluation for N-(1,4-dimethylpentyl)-N'-phenylbenzene-1,4-diamine, CAS No 3081-01-4 (EC No 221-374-3) based on registration dossiers submitted by the Registrant 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 Environment/Suspected PBT; Exposure/Wide dispersive use, N-(1,4-dimethylpentyl)-N'-phenylbenzene-1,4-diamine was included in the Community rolling action plan (CoRAP) for substance evaluation pursuant to Article 44(2) of the REACH Regulation to be evaluated in 2012. The CoRAP was published on the ECHA website on 29 February 2012. The Competent Authority of Austria was appointed to carry out the evaluation.

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 28 February 2013.

On 20 March 2013 ECHA sent the draft decision to the Registrant and invited him pursuant to Article 50(1) of the REACH Regulation to provide comments within 30 days of the receipt of the draft decision.

By 19 April 2013 ECHA received comments from the Registrant of which it informed the evaluating MSCA without delay.

The evaluating MSCA considered the Registrant's comments received and did amend Section II of the draft decision. The comments were reflected in Section III of the draft decision (Statement of reasons).

On 31 October 2013 ECHA notified the Competent Authorities of the Member States of its draft decision and invited them pursuant to Article 51(1) of the REACH Regulation to submit proposals for amendment of the draft decision within 30 days of the receipt of the notification.

Subsequently, proposals for amendment to the draft decision were submitted.

On 5 December 2013 ECHA notified the Registrant 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 the 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 accordingly.

On 16 December 2013 ECHA referred the draft decision to the Member State Committee.

By 7 January 2014, in accordance to Article 51(5), the Registrant provided comments on the proposal(s) for amendment. The Member State Committee took the comments of the Registrant on the proposals for amendment into account.

After discussion in the Member State Committee meeting on 3-7 February 2014, a unanimous agreement of the Member State Committee on the draft decision as modified at the meeting was reached on 3 February 2014. ECHA took the decision pursuant to Article 51(6) of the REACH Regulation.

II. Information required

Pursuant to Article 46(1) of the REACH Regulation the Registrant shall submit the following information using the indicated test methods and instructions, and the registered substance subject to the present decision:

1. Hydrolysis as function of pH (test method: Hydrolysis as function of pH, EU C.7/OECD 111); Amendment of test: the higher tier tests shall be conducted as a function of pH until 90% hydrolysis of the primary hydrolysis products is observed or for 30 days whichever comes first. The primary hydrolysis products shall be investigated like the parent compound during the test following the guidance of the test method;

2. Soil simulation testing (test method: Aerobic and anaerobic transformation in soil, EU C.23/OECD 307). The soil simulation test shall be performed with at least one soil having a pH < 5, as at lower pH-values the substance is assumed to be hydrolytically more stable. The test shall be performed at a temperature of 12°C and using radiolabelled material (via ring-labelling) to assess potential metabolites/ transformation products and the quantity of bound residues;

3. 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 N-(1,4-dimethylpentyl)-N'-phenylbenzene-1,4-diamine. Another test shall be performed with five days old test item concentrations to allow the generation of hydrolysis products.

4. Effects on soil micro-organisms (test method: Soil microorganisms: nitrogen transformation test, EU C.21/OECD 216);

5. Environmental exposure assessment
   A quantitative exposure assessment for the substance N-(1,4-dimethylpentyl)-N'-phenylbenzene-1,4-diamine for the exposure scenario "Use of tyres and general rubber goods" based on a quantitative exposure model or monitoring data.

Pursuant to Article 46(1) of the REACH Regulation the Registrant shall submit robust study summaries for the information required in Section II.

Pursuant to Article 46(2) of the REACH Regulation, the Registrant shall submit to ECHA by 10 April 2016 an update of the registration dossier containing the information required by this decision.

III. Statement of reasons

Based on the evaluation of all relevant information submitted on N-(1,4-dimethylpentyl)-N'-phenylbenzene-1,4-diamine 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 substance constitutes a risk regarding Environment/Suspected PBT; Exposure/Wide dispersive use.

Information is required in order to enable the evaluating MSCA to assess the properties of the substance and to decide whether it is persistent, bioaccumulative or toxic (PBT) and whether risks arising from wide dispersive uses are controlled.

Additionally, concerns were identified for micro-organisms in sewage treatment plants and in soil. Important data on a sensitive endpoint are missing, rendering hazard and risk assessments for sewage treatment plants and soil inadequate. Both compartments are target compartments due to the identified applications and wide dispersive uses, potentially leading to unacceptable risk for these compartments.

Moreover the need for a hydrolysis study was identified as it is a key issue for the PBT assessment, for the assessment of aquatic toxicity and for the exposure assessment.

These data are thus needed to clarify the suspected concerns. Without the requested information it will not be possible to verify whether there remains an uncontrolled risk with the substance that shall be subject to further risk management measures.

1. Hydrolysis as function of pH

Hydrolysis is a key issue in PBT assessment, assessment of aquatic toxicity and exposure assessment of 7PPD (abbreviation for targeted substance N-(1,4-dimethylpentyl)-N'-phenylbenzene-1,4-diamine) and its hydrolysis products. The registered substance is expected to hydrolyse fast within hours and under environmentally relevant conditions. The primary hydrolysis products are considered to be more stable in aqueous solution and co-determine the hazard of the substance. The secondary hydrolysis products are also expected to have a high toxic potential. As the formation and degradation of hydrolysis products is uncertain and unknown, the fate of the hydrolysis products needs to be investigated for describing the degradation pathway at environmentally relevant pH-values. Furthermore, relevant environmental hydrolysis products need to be identified for risk assessment. The identity, kinetic and the mass balance of hydrolysis are required for identifying the most critical compartments and estimating the risk. A refined hydrolysis test is also required to substantiate the read across approach which is currently broadly applied in the registration dossier, as the read-across substances are assumed by the Registrant to follow the same hydrolysis pattern.

Both in his initial comments and in his comments to the proposal for amendments, the Registrant claims that the hydrolysis behaviour is adequately understood. Nevertheless, it is considered crucial to provide clear evidence regarding the hydrolysis processes, as they are of outmost importance for the risk assessment of 7PPD:

1) Reliable hydrolysis data are needed to estimate the behaviour of 7PPD and its hydrolysis products in the environment to allow a good quality risk assessment.
2) Hydrolysis is used as a core argument in the read across justification provided for the assessment of the ecotoxicity of the registered substance. In particular, the Registrant is using studies on hydrolysis products and on similar substances instead of studies for 7PPD itself as an adaptation to information requirements of Annex VII to X of the REACH Regulation.

At the moment crucial data of the hydrolysis process are not available.
For 7PPD itself, only two studies are available that are non-conform to OECD guidelines. These studies indicate half-lives of 5.15 hours and 3.5 hours for 7PPD at pH 7. Only data for pH7 are available. In one of these studies, one hydrolysis product has been identified.

More data are available for the structurally similar substances N-1,3-dimethylbutyl-N´-phenyl-p-phenylenediamine (6PPD) and N-(1,3-dimethylbutyl)-N'-(phenyl)-1,4-benzoquinonediimine (CAS No. 52870-46-9, abbreviation: 6QDI) which is the oxidized form of 6PPD. Using read across data from 6PPD and 6QDI it can be assumed that step 1 hydrolysis products are 4-hydroxydiphenylamine and benzoquinone-monoimine. The third hydrolysis product would be an aliphatic amine:1,3-dimethylbutylamine for 6PPD and 1,4-

dimethylpentylamine for 7PPD. Nevertheless, no identification nor quantification of these amine hydrolysis products have been provided leading to the lack of a proper mass balance. No quantitative estimates (in particular for the calculation of half-lives) are available for the hydrolysis products 4-hydroxydiphenylamine and benzoquinone-monoimine and the aliphatic amine.

For step 2, hydrolysis data from 6QDI show that a further hydrolysis of 4-hydroxydiphenylamine and benzoquinone-monoimine into p-Benzoquinone, p-Hydroquinone and aniline occurs. Nevertheless, a proper mass balance and a description of the kinetics are missing also for these secondary hydrolysis products, and thus the behaviour and fate of 7PPD cannot currently be assessed comprehensively.

In conclusion, contrary to what is claimed by the Registrant, the hydrolysis behaviour of the substance under evaluation cannot be regarded as adequately understood due to the lack of crucial information, in particular regarding hydrolysis products and their half-lives. As 7PPD is expected to be hydrolysed rapidly, the resulting hydrolysis products are considered to be of major relevance for the environment. Therefore, the draft decision was not amended after the Registrant's comments and information on the hydrolytical formation and degradation of the first and second step hydrolysis products are still required.

2. Soil simulation testing

The Registrant provided data waiving arguments according to section 1.2 of REACH Annex XI on the basis that there is sufficient weight-of-evidence from several independent sources of information leading to the assumption that the substance does not biodegrade. In aquatic ready biodegradation tests for N-(1,4-dimethylpentyl)-N'-phenylbenzene-1,4-diamine as well as from other phenylene-diamines and in enhanced biodegradation test for N-(1,4-dimethylpentyl)-N'-phenylbenzene-1,4-diamine data no relevant sign for biodegradation was observed. The Registrant speculated that a mixture with soil extract might have been used in biodegradation tested, although this was not stated in the available studies. The Registrant argued that in domestic sewage a lot of different bacteria are present and it is assumed that terrestrial bacteria would behave in a similar manner. Therefore the Registrant concluded that biodegradation in the terrestrial compartment will not occur.

ECHA rejects the waiving arguments for several reasons:
A soil simulation test is required to assess the persistence and the formation of potential degradation products of the substance N-(1,4-dimethylpentyl)-N'-phenylbenzene-1,4-diamine.

It is acknowledged that the substance is not readily biodegradable as shown by several studies, but it can be assumed for the substance to hydrolyse fast (half-life of approximately 5 hours at pH 7). Nevertheless, it cannot be concluded that no biodegradation is possible under more natural and favourable settings than ready biodegradability tests. Here, it has to be noted that one assumed hydrolysis products is assumed to be readily biodegradable. Regarding PBT/vPvB assessment there is a concern that yet unknown metabolites, which are potentially PBT/vPvB substances, are formed.

Nevertheless, N-(1,4-dimethylpentyl)-N'-phenylbenzene-1,4-diamine has also a high potential for adsorption with a Koc value of 11480. Particularly for the soil compartment it has to be clarified whether the substance is adsorbed and/or hydrolysed rapidly. If the substance adsorbs more rapidly than hydrolysis takes place, N-(1,4-dimethylpentyl)-N'-phenylbenzene-1,4-diamine might fulfil the persistence criterion as defined in Annex XIII of the REACH Regulation.

Considering the use of the substance N-(1,4-dimethylpentyl)-N'-phenylbenzene-1,4-diamine

including the use in tyres (in rubber matrix as antioxidant) the soil compartment is a target compartment of N-(1,4-dimethylpentyl)-N'-phenylbenzene-1,4-diamine. Also distribution modelling using Level III Fugacity model and the PBT profiler[1], show that soil has the highest percentage of distribution (70.6 % and 65% respectively).

In case the P criterion for soil is not met, and because the exposure of sediment is considered significant, subsequently also sediment simulation testing may be considered and requested in a follow-up by the evaluating MSCA after studying the dossier update from the Registrant.

If the P criterion is fulfilled, further evaluation regarding the bioaccumulation criterion will be needed. While the assumed hydrolysis products are considered to have a low bioaccumulation potential (the log Kow values of the assumed hydrolysis products is < 3 and the highest measured BCF (Bio-Concentration Factor) value is 58), N-(1,4-dimethylpentyl)-N'-phenylbenzene-1,4-diamine itself fulfils the screening criterion for the B criterion of the PBT assessment, as the estimated log Kow is 5.2. No measured data are available for the BCF for N-(1,4-dimethylpentyl)-N'-phenylbenzene-1,4-diamine. Therefore, the outcome of the soil simulation test needs to be awaited (potential fulfilment of the P criterion) to conclude whether further evaluation of bioaccumulation is necessary.

In his comments, the Registrant accepted to perform a soil simulation study on 7PPD, but requested that the justification is amended regarding the PBT assessment and Specific Target Organ Toxicity Repeated Exposure category 2 (STOT-RE 2) classification.

Regarding PBT assessment, the Registrant is citing a statement of the TEC-NES PBT working group regarding the bioaccumulation criterion of N-1,3-dimethylbutyl-N´-phenyl-p-phenylenediamine (6PPD) in which the bioaccumulation criterion is deemed to be not fulfilled due to its rapid degradability in water. Nevertheless, statements of the former TEC-NES PBT working group are not legally binding and may be outdated due to new data or knowledge. Therefore, the draft decision was not amended.

Regarding the assessment of STOT-RE 2 classification the evaluating MSCA deems the criteria to be fulfilled. Nevertheless, the wording was changed and has no mentioning of the classification any longer as it is not necessary for the justification of the soil simulation test and does not provide information regarding potential future requirements.

Regarding the potential consideration of a sediment simulation test, the Registrant also expressed concerns about the relevance to perform in addition a sediment simulation test, having regard to the fast hydrolysis rate in water and because he considers that degradation patterns in soil and sediment are likely to be similar. Nevertheless, the requirement to perform a sediment simulation test might arise if deemed necessary by the evaluating MSCA at a later stage.

Regarding the T criterion, ECHA agrees with the Registrant that in a preliminary assessment N-(1,4-dimethylpentyl)-N'-phenylbenzene-1,4-diamine needs to be considered to fulfil the T criterion.

Summing up the provided reasons a soil simulation test is needed to clarify whether the persistence criterion according to Annex XIII of REACH is fulfilled and/or whether metabolites are formed, which are potentially PBT or vPvB substances according to Annex XIII of REACH. The required test shall be performed according to the test guideline "Aerobic and anaerobic transformation in soil" (EU C.23/OECD 307). The soil simulation test shall be

[1] The PBT-profiler is a computational tool for predicting the P, B and T criteria of a substance based on its structure; see http://www.pbtprofiler.net/

performed with at least one soil having a pH < 5, as at lower pH-values the substance is assumed to be hydrolytically more stable.

Radiolabelled material (via ring-labelling) shall be used for the identification and the quantification of the potential metabolites/ transformation products and of the bound residues.

The test shall be performed at a temperature of 12°C. The substance subject to the present decision is used and released within the context of the REACH Regulation in the EU. Therefore, the Registrant is requested to perform the study at 12 °C (285K) as this temperature is indicated in the Guidance on information requirements and chemical safety assessment Chapter R.16., Table R.16-9 (version 2.1 October 2012) as the average environmental temperature for the EU to be used in the chemical safety assessment including PBT assessment. The Registrant wishes to perform the test at 20°C, although he indicates in his comments that he understands the reason why the test shall be performed at 12°C and acknowledges that the recalculation to another temperature is scientifically questionable. However he points out that in Annex XIII of REACH no temperature is indicated for the half-life assessment and for the following assessment of the persistence criterion He also highlights that for the hydrolysis test (OECD test guideline 111) and the OECD biodegradation tests (OECD 301 test guideline series) the recommended temperature range is 20 to 23°C. Nevertheless, ECHA considers that performing the test at the temperature of 12°C is within the applicable test conditions of the Test Guideline OECD 307 and is the appropriate temperature for chemical safety assessment in the EU, including the PBT assessment. There is a fundamental difference between OECD 301 tests and an OECD 307 test in that the OECD 301 tests are screening tests that are designed to offer the possibility to come to a conclusion for cases that are rather clear-cut. In contrast, an OECD 307 test is a simulation test and therefore circumstances in this test should be as similar to real life situation as possible.

To avoid unnecessary testing, this test may be performed for the evaluation of N,N'-bis(1,4-dimethylpentyl)-p-phenylenediamine (EC 221-375-9, short name: 77PD) as 7PPD is an acceptable read across substance for 77PD. The Registrant shall thus consider data sharing.

3. Activated sludge respiration inhibition testing

Sewage treatment plants (STP) and their micro-organisms need to be protected to guarantee proper waste water treatment. The high importance of aquatic micro-organisms for risk assessment is in particular reflected by the fact, that a valid test is considered to be a necessary information requirement already for substances produced or imported in quantities of ≥ 10 tonnes (see Annex VIII of REACH).

The Registrant provided data on activated sludge respiration inhibition with respect to substance N-1,3-dimethylbutyl-N'-phenyl-p-phenylenediamine. Although no read across justification was included in the registration dossier, read across was done with data on substance N-1,3-dimethylbutyl-N'-phenyl-p-phenylenediamine ([redacted][2]): The study was performed in 1984 and is only rewritten by [redacted] resulting in an IC50 (half maximal inhibitory concentration) value of 420 mg/L. Nevertheless, the original study report is exceedingly poor in description and contains mainly some handwritten tables, making even comparison with quality criteria of guideline protocols impossible. A non-guideline-conform temperature of around 27°C, instead of 20 ±2°C was used. The test concentration is far above water solubility. ECHA rates this study Klimisch 3.

[2] [redacted] Activated Sludge, Respiration Inhibition Test with [redacted]. Testing laboratory: [redacted]. Report date: 2012-09-28.

The Registrant also included data on the assumed secondary hydrolysis product hydroquinone as the Registrant argues that the formation of hydroquinone is hampering biodegradation. The toxicity towards bacteria was stated to be seen in a test with cyanobacteria *Microcystis aeroruginosa*, which resulted in a 8 day Toxicity Threshold (equivalent to a 3% effect concentration) of 1mg/L (Bringmann G. and Kühn R., 1978[3]). Also another test is provided for hydroquinone resulting in a 2h IC50 (half maximal inhibitory concentration) of 71 mg/L test material (nominal) based on respiration rate (Chan C-M et al, 1999[4]). A luminescence-based scanning respirometer, using a ruthenium (II) complex as the oxygen-sensing element was used. These test procedures differ vastly from standard protocols, resulting in an unclear reliability.

No data on inhibition of nitrification are presented neither for N-(1,4-dimethylpentyl)-N'-phenylbenzene-1,4-diamine nor for any of the hydrolysis products assumed by the Registrant, although this endpoint might be the most sensitive endpoint, as data from literature suggest (literature research performed by the evaluating MSCA). Quinone-like compounds do have an inhibitory effect on nitrification as stated e.g. by Suárez-Ojeda et al, 2010.[5]

Summing up, due to the lack of data on a probably sensitive endpoint (inhibition of nitrification) resulting in serious concern regarding the hazard and risk assessment of STP and their micro-organisms testing is required to gain data on toxicity on aquatic micro-organisms of N-(1,4-dimethylpentyl)-N'-phenylbenzene-1,4-diamine itself as well as of its hydrolysis products. Testing on hydrolysis products is needed as it might take several days until sewage water reaches the sewage treatment plant and hydrolysis might already have occurred, leading to the generation of potentially toxic compounds. The generated data will constitute the crucial data for the hazard and risk assessment for the STP.

The required tests shall be performed according to the test guideline "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 N-(1,4-dimethylpentyl)-N'-phenylbenzene-1,4-diamine. Another test shall be performed with a five days old test item suspension to allow the generation of hydrolysis products.

4. Effects on soil micro-organisms

Soil is a critical compartment for risk assessment. Because of the use of the substance N-(1,4-dimethylpentyl)-N'-phenylbenzene-1,4-diamine including the use in tyres the soil compartment is a target compartment of N-(1,4-dimethylpentyl)-N'-phenylbenzene-1,4-diamine. Also distribution modelling using Level III Fugacity model and the PBT profiler show that soil has the highest percentage of distribution (70.6 % and 65% respectively).

The Registrant provided data waiving according to section 3.2a(ii) of REACH Annex XI based on the following justification: "*No further testing in soil is needed. A PNEC can be derived from results of available test data in soil organisms, taking full account of the uncertainty resulting from the omission of additional soil tests. The PNEC soil derived from 2 chronic studies is appropriate for risk assessment purposes*".

[3] Bringmann G., Kühn R. (1978). Testing for substances their toxicity threshold: Model organisms Microcystis (Diplocystis) aeruginosa and Scendesmus quadricauda. Mitt Internat Verein Limnol 21: 275-284.
[4] Chan C-M, Lo W, Wong K-Y, Chung W-F (1999). Monitoring the toxicity of phenolic chemicals to activated sludge using a novel optical scanning respirometer. Chemosphere 39: 1421-1432.
[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.

The use of the equilibrium partitioning method is not possible and was not applied by the Registrant, as the log Kow of N-(1,4-dimethylpentyl)-N'-phenylbenzene-1,4-diamine is higher than 5 and adsorption potential is high. Moreover, high toxicity was observed in the aquatic tests. Two long-term tests on soil organisms (earthworms, plants) are available: A no observed effect concentration (NOEC) for terrestrial plants of 7.8 mg/kg dw was obtained from testing with *Brassica napus* with respect to shoot length ([redacted]). The NOEC (56d) for reproduction was 100 mg/soil dw from a test with earthworms ([redacted]).

The provided reasons for waiving were not accepted, as the substance is released to the environment and has wide dispersive use. Moreover, data on soil micro-organisms are crucial, as soil micro-organisms might be the most sensitive trophic level in the soil compartment. Data on toxicity on the presumed hydrolysis product hydroquinone, which is known as inhibitor of ammonium oxidation, suggest serious concern in this respect: see Suárez-Ojeda et al, 2010[6] (literature research performed by the evaluating MSCA).

Summing up, due to lack of data on a probably sensitive endpoint (inhibition of nitrification) resulting in serious concern regarding the hazard and risk assessment of soil testing is required to gain data on toxicity on soil micro-organisms. The generated data will provide crucial data for hazard and risk assessment for the soil compartment.

In his comments, the Registrant made the following objections regarding this study request, including:

- that ECHA does not accept new tests in ecotoxicology which are not performed under good laboratory practice (GLP),
- that the lower assessment factor resulting from the availability from this study would at least partially compensate for a potential lower effect value and
- that an exposure assessment and risk assessment have been performed.

These objections could not be followed. Firstly, the cited study of Suarez-Ojeda et al (2010) is only cited to demonstrate that in the scientific community data revealing a concern regarding the toxicity to microorganisms are available. To perform studies according to GLP is an obligation stemming from Article 13(4) of the REACH Regulation. Non-GLP studies need thorough evaluation regarding their validity but are usable for chemical safety assessment. Secondly, microorganisms might be the most sensitive species and hence might result in a lower predicted no effect concentration (PNEC) – even if a lower assessment factor is applied. Lastly, the Registrant's argument that an exposure and risk assessments have been performed is not deemed appropriate as the information in the dossier is precisely believed to be currently insufficient to carry out a proper risk assessment. Consequently, the decision was not amended after the Registrant's comments.

Therefore a test on soil micro-organisms is needed to evaluate the risk for the soil compartment, which is a receiving compartment of N-(1,4-dimethylpentyl)-N'-phenylbenzene-1,4-diamine. The required tests shall be performed according to the test guideline "Soil microorganisms: nitrogen transformation test (EU C.21/OECD 216)

5. Environmental exposure assessment

Regarding the high tonnage, wide dispersive use and the highly ecotoxic properties of 7PPD, the conclusion of negligible exposure to the environment is not accepted for the exposure scenario "Use of tyres and general rubber goods" covering the release of abraded tyre wear

[6] 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.

particles via traffic. High tonnages of tyre wear particles containing residues of N-(1,4-dimethylpentyl)-N'-phenylbenzene-1,4-diamine are expected to be released into the environment.

In his comments, the Registrant argues that he should be allowed to perform the exposure assessment using state-of-the-art knowledge about the fate of anti-degradants in tyres in the exposure assessment. In particular, the use of the study of Baumann and Ismeyer, 1998[7] and of Marwood et al.[8] were addressed.

The cited studies of Marwood et al. concern ecotoxity tests which were performed with the leachate of tyre wear particles for pelagic ecotoxicity tests and with tyre wear particles mixed into the sediment for ecotoxicity testing on sediment organisms. The studies of Marwood et al. need to be rated unreliable as the setting of the test conditions are inappropriate and the substance concentrations were not quantifiable: For pelagic tests, sediment was mixed with tyre wear particles containing antioxidants like 7PPD in a concentration of about 1%. Visual examination of the overlying waters after one hour settling period indicated that the residual turbidity was still too high for successful testing, so the overlying water was centrifuged. As 7PPD and its related substances are highly adsorptive, the adsorption of potentially released substance to sediment could in this case lead to a lower toxicity. Also only a small percentage of the contained substance might have leached out of the particles during the short time of exposure. This is confirmed by low leaching percentages found in the leachates in the acute studies, although also these results are hampered by the fact of analytical inconsistencies. Also for the tests regarding toxicity towards sediment dwelling organisms no confirmed substance concentrations are available. In contrast, in real environmental conditions, the leaching process from tyre wear particles can take months to years (depending on the breakdown rate of the particles) and exceeds the duration of ecotoxicity tests by far. The conditions in the environment are not reflected by the studies of Marwood et al. The complete release of 7PPD through the breakdown of the tyre wear particles has to be assumed, although it might take a long time. To use the studies of Marwood *et al.* and their low toxicity results as an argument not to perform a quantitative exposure assessment is therefore not accepted. Moreover, the use of the Marwood et al studies for exposure assessment is not deemed to be appropriate modelling and use of state-of-the-art knowledge.

Based on the Baumann and Ismeyer, 1998 study the Registrant indicated that the concentration of the substance in tyre wear particles has been estimated to be only 0.1%, whereas rubber is produced with concentration of about 1%. Thus 0.9% of the substance has been assumed to be consumed i.e. when the substance migrates to the surface of rubber and is oxidised and hydrolysed. However, it is considered that both the remaining substance released from tyre wear particles and the oxidation/hydrolysis products released during the service life of the tyres need to be investigated and considered in the risk assessment.

Therefore, environmental exposure shall be estimated by a quantitative environmental exposure model or by monitoring data.

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[7] Baumann W, Ismeier M (1998) Kautschuk und Gummi, 2 volumes, Daten und Fakten zum Umweltschutz, Berlin, Springer, pp. 272 -275, 307 - 349

[8] Marwood et al. (2010). Chronic Toxicity of tire/Road Wear Particles in Sediment to aquatic organisms. Poster presented at SETAC conference 2010; (Link to WBCSD)
Marwood C. et al. (2011). Acute aquatic toxicity of tire and road wear particles to alga, daphnid, and fish. Ecotoxicology 20, 2079-2089.

IV. Adequate identification of the composition of the tested material

The substance identity information submitted in the registration dossier has not been checked for compliance with the substance identity requirements set out in Section 2 of Annex VI of the REACH Regulation.

In relation to the required tests, the sample of substance used for the new studies shall have a composition that is within the specifications of the substance composition that are given by the Registrant. It is the responsibility of the Registrant 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.

V. General requirements regarding Good Laboratory Practice

ECHA reminds registrants of the requirements of Article 13(4) of the REACH Regulation that ecotoxicological and toxicological tests and analyses shall be carried out in compliance with the principles of good laboratory practice (GLP).

According to Article 13(3) of the REACH Regulation, tests that are required to generate information on intrinsic properties of substances shall be conducted in accordance with the test methods laid down in a Commission Regulation or in accordance with other international test methods recognised by the Commission or the European Chemicals Agency as being appropriate. Thus, the Registrant shall refer to Commission Regulation (EC) No 440/2008 laying down test methods pursuant to Regulation (EC) No 1907/2006 as adapted to technical progress or to other international test methods recognised as being appropriate and use the applicable test methods to generate the information on the endpoints indicated above.

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://echa.europa.eu/regulations/appeals
The notice of appeal will be deemed to be filed only when the appeal fee has been paid.

[redacted]

Jukka Malm
Deputy Executive Director