EUROPEAN CHEMICALS AGENCY

Decision number: CCH-D-2114290169-41-01/F Helsinki, 16 December 2014

DECISION ON A COMPLIANCE CHECK OF A REGISTRATION PURSUANT TO ARTICLE 41(3) OF REGULATION (EC) NO 1907/2006

For p-xylene, CAS No 106-42-3 (EC No 203-396-5), registration number: [redacted]

Addressee: [redacted]

The European Chemicals Agency (ECHA) has taken the following decision in accordance with the procedure set out in Articles 50 and 51 of Regulation (EC) No 1907/2006 concerning the Registration, Evaluation, Authorisation and Restriction of Chemicals (REACH Regulation).

I. Procedure

Pursuant to Article 41(1) of the REACH Regulation ECHA has performed a compliance check of the registration for p-xylene, CAS No 106-42-3 (EC No 203-396-5), submitted by [redacted] (Registrant). The scope of this decision is limited to the standard information requirements of Annex VI, Section 2, Annex IX, Sections 9.1.6 and 9.4.2, Annex X, Sections 9.4.4 and 9.4.6 of the REACH Regulation and the related elements of the chemical safety assessment and chemical safety report.

This decision is based on the registration as submitted with submission number [redacted], for the tonnage band of 1000 tonnes or more per year. This decision does not take into account any updates submitted after 24 July 2014, the date upon which ECHA notified its draft decision to the Competent Authorities of the Member States pursuant to Article 51(1) of the REACH Regulation.

This compliance check decision does not prevent ECHA from initiating further compliance checks on the present registration at a later stage.

The substance subject to the present decision is provisionally listed in the Community rolling action plan (CoRAP) for start of substance evaluation in 2015.

The compliance check was initiated on 2 August 2013.

On 18 December 2013 ECHA sent the draft decision to the Registrant and invited him to provide comments within 45 days of the receipt of the draft decision.

On 3 February 2014 ECHA received comments from the Registrant on the draft decision.

The ECHA Secretariat considered the Registrant's comments.

The information is reflected in the Statement of Reasons (Section III) whereas no amendments to the Information Required (Section II) were made.

On 24 July 2014 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, proposal for amendment to the draft decision were submitted.

On 29 August 2014 ECHA notified the Registrant of the proposal for amendment to the draft decision and invited him pursuant to Article 51(5) of the REACH Regulation to provide comments on the proposal for amendment within 30 days of the receipt of the notification.

The ECHA Secretariat reviewed the proposal for amendment received and did not amend the draft decision.

On 8 September 2014 ECHA referred the draft decision to the Member State Committee.

By 29 September 2014, in accordance to Article 51(5), the Registrant did not provide any comments on the proposal for amendment.

After discussion in the Member State Committee meeting on 28-29 October 2014, a unanimous agreement of the Member State Committee on the draft decision as modified at the meeting was reached on 29 October 2014.

ECHA took the decision pursuant to Article 51(6) of the REACH Regulation.

II. Information required

A. Information in the technical dossier related to the identity of the substance

Pursuant to Articles 41(1)(a), 41(3), 10(a)(ii) and Annex VI, Section 2 of the REACH Regulation the Registrant shall submit the following information for the registered substance subject to the present decision:

1. Composition of each substance (Annex VI, Section 2.3), as specified under section III.A.(1) below;
2. Spectral data (Annex VI, 2.3.5.), as specified under Section III.A.(1) below;

B. Information in the technical dossier derived from the application of Annexes VII to XI

Pursuant to Articles 41(1), 41(3), 10(a)(vi) and/or (vii), 12(1)(e), 13 and Annexes IX, X of the REACH Regulation the Registrant shall submit the following information using the indicated test methods and the registered substance subject to the present decision:

1. Ready biodegradability (Annex VII, 9.2.1.1.; test method: MITI (I) test, OECD 301 C or Closed bottle test, OECD 301 D or Manometric respirometry test, OECD 301 F);
2. Long-term toxicity testing on fish (Annex IX, 9.1.6.1.; test method: Fish, early-life stage (FELS) toxicity test, OECD 210);
3. Effects on terrestrial organisms - Long-term toxicity testing on terrestrial invertebrates (Annex X, 9.4.4.; test method: Earthworm reproduction test (*Eisenia fetida/Eisenia andrei*) (test method: OECD 222), or Enchytraeid reproduction test (test method: OECD 220), or Collembolan reproduction test in soil, OECD 232);
4. Effects on terrestrial organisms – Long-term toxicity testing on plants (Annex X, Section 9.4.); test method: Terrestrial plants, growth test, OECD 208, with at least six species tested (with as a minimum two monocotyledonous species and

four dicotyledonous species) or Soil Quality – Biological Methods – Chronic toxicity in higher plants, ISO 22030;

5. Effects on terrestrial organisms - Effects on soil micro-organisms (Annex IX, 9.4.2.; test method: Soil microorganisms: nitrogen transformation test, EU C.21./OECD 216 and Soil microorganisms: carbon transformation test, EU C.22./OECD 217), as specified under Section III.B below.

C. Information related to chemical safety assessment and chemical safety report

Pursuant to Articles 41(1), 41(3), 10(b), 14 and Annex I of the REACH Regulation the Registrant shall submit in the chemical safety report:

1. Revised predicted no effect concentrations (PNECs) for freshwater and marine water as specified under Section III C below.

Pursuant to Article 41(4) of the REACH Regulation the Registrant shall submit the information in the form of an updated registration to ECHA by **23 September 2016**. The timeline has been set to allow for sequential testing as appropriate.

Note for consideration by the Registrant:

The Registrant may adapt the testing requested above according to the specific rules outlined in Annexes VI to X and/or according to the general rules contained in Annex XI of the REACH Regulation. In order to ensure compliance with the respective information requirement, any such adaptation will need to have a scientific justification, referring to and conforming with the appropriate rules in the respective Annex, and an adequate and reliable documentation.

Failure to comply with the request(s) in this decision, or to fulfil otherwise the information requirement(s) with a valid and documented adaptation, will result in a notification to the Enforcement Authorities of the Member States.

III. Statement of reasons

Pursuant to Article 41(3) of the REACH Regulation, ECHA may require the Registrant to submit any information needed to bring the registration into compliance with the relevant information requirements.

A. Information in the technical dossier related to the identity of the substance

Pursuant to Article 10(a)(ii) of the REACH Regulation, the technical dossier shall contain information on the identity of the substance as specified in Annex VI, Section 2 of the REACH Regulation. In accordance with Annex VI, Section 2 the information provided shall be sufficient to enable the identification of the registered substance.

1. Composition of each substance (Annex VI, 2.3)

ECHA observes that the registration dossier does not contain concentration ranges for the main constituent and impurities listed in Section 1.2. Based on the reported minimum purity of [redacted] % w/w a significant part (up to [redacted] %) of the substance is unknown. ECHA can therefore not verify that all individual impurities required to be identified have been reported in the composition of the registered substance. The Registrant is therefore requested to include the minimum and maximum concentration values for the main constituent and each impurity listed in Section 1.2 of the dossier. For mono-constituent

substances the sum of the minimum purity and the maximum concentrations of each impurity should equal to 100 %.

2. Spectral data (Annex VI, 2.3.5.)

ECHA observes that the registration dossier does not contain any of the spectral data required according to Annex VI Section 2.3.5. of the REACH Regulation to support the identity of the registered substance. An Ultra-Violet (UV) spectrum, Infra-Red (IR) spectrum and Nuclear Magnetic Resonance (NMR) spectrum (or a Mass Spectrum (MS) instead of an NMR spectrum) are an information requirement under Annex VI Section 2.3.5.

According to the Registrant; *"The techniques UV, IR, NMR are not applicable for analysis because they will require sophisticated, advanced and non-routine handling of test samples. Detailed compositional analysis has been obtained by GC."* ECHA cannot accept this justification as it is technically possible and scientifically necessary to provide the missing information.

ECHA regards the required information scientifically necessary to identify the registered substance for the following reasons:
- The substance absorbs in the UV range due to the presence of chromophores in the composition. A UV spectrum representing the absorption of these constituents in the UV range can therefore be recorded;
- The IR spectrum displays characteristic vibration bands of covalent bonds in molecules present in the substance, including characteristic vibration bands from the chemical functionalities expected to be present in the composition;
- NMR spectroscopic analyses such as a 1H-NMR or a 13C-NMR are powerful tools for structure characterisation and elucidation due to characteristic chemical shifts and spin-spin coupling which also reflects the relative abundance of individual atoms.

The Registrant is therefore requested to submit a UV spectrum, an IR spectrum and an NMR spectrum, such as a 1H-NMR or a 13C-NMR. As an alternative to an NMR spectrum, a mass spectrum can be provided. Details of sample preparation (such as solvent identity and concentration) and relevant equipment operating parameters (such as spectrometer frequency) should accompany each spectrum.

As for the reporting of the spectral data in the registration dossier, the information should be included in IUCLID Section 1.4.

The Registrant submitted comments on the draft decision and proposed to address these issues on the Substance Identity with a spontaneous update by the end of 2014.

ECHA acknowledges the Registrant's commitment to update the substance identity information as required by the present decision. However, ECHA notes that the standard information requirement is not yet fulfilled. Therefore, the information requirement is maintained in the decision.

Therefore, Section II.A of the decision is not amended based on the comments provided by the Registrant.

B. Information in the technical dossier derived from the application of Annexes VII to XI

Pursuant to Articles 10(a)(vi) and/or (vii), 12(1)(e) of the REACH Regulation, a technical dossier for a substance manufactured or imported by the Registrant in quantities of 1000

tonnes or more per year shall contain as a minimum the information specified in Annexes VII, VIII, IX, and X of the REACH Regulation.

1. Ready biodegradability (Annex VII, 9.2.1.1)

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

The registration dossier contains a GLP compliant study with the registered substance performed according to OECD 301 F, Manometric respirometry test ([redacted]). The study is assigned reliability 1 (reliable without restrictions) by the Registrant and was chosen as key study. In the robust study summary it is reported that "*This study followed a standard guideline and was conducted to GLP. Greater than 20% variability was observed between replicates at the start and end of the 10 day window. Despite this limitation all three replicates had reached >60% biodegradation by day 11, so this is not considered to have invalidated the results of this test. It is therefore considered suitable for use as the key study for this endpoint.*" However, the validity criterion described in paragraph 24 of the OECD test guideline 301 is not met and, according to the test guideline, the test should be repeated.

A supporting study by Bridie et al. (1979) is provided. This study was published in the open literature and predates the implementation of GLP and OECD guidelines for biodegradation screening tests. According to the Registrant, 44% degradation of p-xylene was found after 5 d based on the BOD/ThOD ratio. In the dossier it is written that *"the level of detail in the publication is very limited. In particular the concentration of the test substance is not reported."* The registrant assigned a reliability score of 2 (reliable with restrictions).

A further supporting study by MITI, 2001 is assigned reliability 4 by the Registrant since the original study report was not reviewed and it could not be confirmed that the validity criteria were met. The study was conducted according to test guideline OECD 301C and found 38% biodegradation after 28 days as measured by BOD and 92% biodegradation measured by GC analysis.

ECHA acknowledges that the available information indicates the substance is biodegradable but no conclusion can be reached on whether the substance meets the criteria for ready biodegradability. Since the key study by [redacted] does not meet the validity criterion of the OECD 301F test guideline, i.e. the variation between the replicates exceeded 20 %, ECHA considers that the study is invalid. ECHA considers that the robust study summary of the supporting study provided (Bridie et al.1979) contains insufficient information to assess its validity. For example, the concentration of the test substance is not reported. The reliability of the second supporting study (MITI, 2001) could not be assessed. ECHA considers that based on the information provided in the dossier, it is not possible to conclude on the endpoint of ready biodegradability.

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

Since the registered substance is volatile, with a calculated Henry's law constant of 623 Pa.m3.mole reported in the Chemical Safety Report the test method selected for ready biodegradability testing shall be suitable for volatile substances. Table 1 of the OECD test

guideline 301 (adopted 17 July 1992) indicates that the following test methods may be suitable for volatile substances: MITI test, OECD 301 C; Closed bottle test, OECD 301 D; Manometric respirometry test, OECD 301 F.

Therefore, pursuant to Article 41(1) and (3) of the REACH Regulation, the Registrant is requested to submit the following information derived with the registered substance subject to the present decision:Ready biodegradability: MITI test, OECD 301 C or Closed bottle test, OECD 301 D or Manometric respirometry test, OECD 301 F.

2. Long-term toxicity testing on fish (Annex IX, 9.1.6.1.)

"Long-term toxicity testing on fish" is a standard information requirement as laid down in Annex IX, Section 9.1.6. of the REACH Regulation. Adequate information on Fish, early-life stage (FELS) toxicity test (Annex IX, 9.1.6.1.), or Fish, short-term toxicity test on embryo and sac-fry stages (Annex IX, 9.1.6.2.), or Fish, juvenile growth test (Annex IX, 9.1.6.3.) needs to be present in the technical dossier for the registered substance to meet this information requirement.

The Registration dossier contains a long-term toxicity study with *Oncorhynchus mykiss* and the read-across substance mixed xylenes (Walsh et al. 1977). The study is assigned reliability 2 (reliable with restrictions) by the Registrant who summarises it as follows: *"Adult fish were exposed to xylene for 56 days in a flow through system. Test concentrations were confirmed by analytical monitoring throughout the exposure period. Changes in behaviour and mortalities were the endpoints reported. No exposure linked mortality or changes in behaviour were observed at any of the test concentration (NOEC >1.3 mg/l). This study predates the implementation of GLP and study guidelines and deviates significantly from standard chronic fish toxicity tests. In particular, the use of adult fish in the chronic test and the choice of endpoints may result in a less sensitive result than if an embryo-larval or juvenile fish test had been conducted."*

As stated by the Registrant himself, the study deviates significantly from currently available standardised chronic fish toxicity tests. The OECD guidelines for long-term toxicity testing on fish cover the exposure of early life-stages, which may be more sensitive towards toxicants than the adult stage. According to ECHA Guidance on information requirements and chemical safety assessment (version 1.2, November 2012), Chapter R7b, Section R.7.8.4, *"Only such studies can be regarded as long-term fish test, in which sensitive life-stages (juveniles, eggs, larvae) are exposed."* In the study by Walsh et al. (1977), only adult fish were exposed. ECHA therefore considers that the study provided does not meet the information requirement of Annex IX, Section 9.1.6. "Long-term toxicity testing on fish".

ECHA notes that the adaptation possibility of Section 1.5. of Annex XI allows registrants to fulfil information requirements by predicting the required data by using the information from the proposed read-across substance. However, as the data submitted is insufficient even for the proposed read-across substance, ECHA does not need to assess whether the conditions for applying the group concept (Annex XI, 1.5. of the REACH Regulation) have been justified by the Registrant.

According to ECHA Guidance on information requirements and chemical safety assessment (version 1.2, November 2012), Chapter R7b, Section R.7.8.4, *"Among the currently available standardised test methods, the FELS toxicity test is considered as the most sensitive of the fish tests. It covers several life stages of the fish from the newly fertilised egg, through hatch to early stages of growth and is also the only suitable test currently available for examining the potential toxic effects of bioaccumulation."* Therefore, ECHA considers that this is the most suitable test method for the registered substance.

The Registrant states in their comments that fish are not the most sensitive test organism. However, ECHA considers that fish, aquatic invertebrates and algae all show a similar level of sensitivity to p-xylene based on the experimental data included in the registration dossier since the acute toxicity effect concentrations differ by less than a factor of 10. According to ECHA Guidance on information requirements and chemical safety assessment (version 1.2, November 2012), Chapter R7b, Section R.7.8.5.3, "*If there is compelling evidence, using these methods, to suggest that the fish value is likely to be at least a factor of about 10 less sensitive than invertebrates or algae there are no further requirements for fish testing.*"

Moreover, the Registrant proposes to use QSAR predictions for the endpoint of long-term fish toxicity. ECHA notes that this additional information is not yet included to the registration dossier and the standard information requirement is not fulfilled. Therefore, the information requirement is maintained in the decision.

Furthermore, the Registrant proposes to use PETROTOX to derive the aquatic PNEC for risk assessment. For ECHA's consideration on the PETROTOX model please refer to Section III.C of this decision.

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

Therefore, pursuant to Article 41(1) and (3) of the REACH Regulation, the Registrant is requested to submit the following information derived with the registered substance subject to the present decision: Fish, early-life stage (FELS) toxicity test (test method: OECD 210).

3., 4. and 5.. Effects on terrestrial organisms (Annex IX and X, 9.4.)

"Effects on terrestrial organisms" are standard information requirements as laid down in Annex IX and X, Section 9.4. of the REACH Regulation. Adequate information on effects on soil micro-organisms (Annex IX, Section 9.4.2.), short-term and long-term toxicity to invertebrates (Annex IX, Section 9.4.1. and Annex X, Section 9.4.4.) and short-term and long-term toxicity to plants (Annex IX, Section 9.4.3. and Annex X, Section 9.4.6.) needs to be present in the technical dossier for the registered substance to meet the information requirements.

a) Toxicity testing on terrestrial invertebrates (Annex IX, 9.4.1. and Annex X, 9.4.4.)

The Registrant proposed to waive testing on terrestrial invertebrates using the following justifications: "*In accordance with column 2 of REACH Annex IX, the short term toxicity to invertebrates study does not need to be conducted as direct and indirect exposure of the soil compartment is unlikely*" and "*In accordance with column 2 of REACH Annex X, the long term toxicity to invertebrates study does not need to be conducted as direct and indirect exposure of the soil compartment is unlikely.*"

However, ECHA considers that direct exposure of the soil compartment is likely since the registered substance is used as an agrochemical. Furthermore, in the Chemical Safety Assessment emissions to soil are indicated for 20 out of 22 Exposure scenarios, for example for professional and consumer use of agrochemicals (Exposure scenarios 15 and 16). Hence, ECHA considers that the adaptation provided by the Registrant does not meet the criteria of either the specific adaptation rules of Column 2 of Annexes IX and X, Sections 9.4, or any of the general adaptation rules of Annex XI. Therefore, the adaptations cannot be accepted.

The Registrant provided comments on the draft decision. In his comments the Registrant confirms that exposure to soil is expected for the use of xylenes in pesticide formulations therefore the column 2 adaptation "direct and indirect exposure of the soil compartment is unlikely" cannot be applied.

The Registrant proposes to use PETROTOX to derive the terrestrial PNECs for risk assessment. For ECHA's consideration on the PETROTOX model please refer to Section III.C of this decision.

ECHA considers that the the standard information requirement is not fulfilled. Therefore, the information requirement is maintained in the decision.

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

The earthworm reproduction test (OECD 222), Enchytraeid reproduction test (OECD 220), and Collembolan reproduction test (OECD 232) are each considered capable of generating information appropriate for the fulfilment of the information requirements for short-term and long-term toxicity to terrestrial invertebrates (Annex IX, 9.4.1, and Annex X, 9.4.4.). ECHA is not in a position to determine the most appropriate test protocol, since this decision is dependent upon species sensitivity and substance properties.

Therefore, pursuant to Article 41(1) and (3) of the REACH Regulation, the Registrant is requested to submit the following information derived with the registered substance subject to the present decision:

Earthworm reproduction test (*Eisenia fetida/Eisenia andrei*) (test method: OECD 222), or Enchytraeid reproduction test (test method: OECD 220), or Collembolan reproduction test in soil (test method: OECD 232).

b) Toxicity testing on terrestrial plants (Annex IX, 9.4.3. and Annex X, 9.4.6.)

The Registrant provides one short-term toxicity test on the read-across substance o-xylene which they summarise as follows: *"A single study to investigate the effects of o-xylene on terrestrial plants was found. This study reported a 14 day EC50 of >1mg/kg nominal, although analytical measurements indicated that <10% nominal remained at the end of the test. The methods used in study were equivalent to OECD 208 (1984). The main deviations from this guideline were a higher clay content than recommended, and the reporting of results at 14 days post sowing, not 14 days post germination of the controls. Insufficient details are given in the paper to allow us to conclude whether the validity criteria of the test were met as results from the controls are not reported. Despite these restrictions this study does allow us to conclude that major effects on the growth of lettuce are not seen at the highest concentration tested although this is probably due to reduced exposure due to volatilisation or biodegradation. The results from this study will not be used to calculate a PNEC in the risk assessment due to these limitations."*

ECHA considers that this study is not suitable for evaluation of the acute toxicity to plants since it is unclear whether the validity criteria of the test guideline were met, the exposure duration is shorter than recommended by the OECD 208 test guideline and control results are not reported.

A supporting study is provided in the registration dossier (IUC4#1/Ch.4.6.2, Ivans 1952). The study is poorly reported. The the read-across substance o-xylene was applied in the vapour phase to detached leaves of runner bean (*Phaseolus multiflorus*) and there was a relatively small difference between concentrations causing no damage and those completely killing the leaves. No indication of the reliability of the study is provided by the Registrant. ECHA considers that the supporting study cannot be used to meet the information requirement of short-term toxicity to plants because there is no information on the reliability and validity of the study. Consequently, there is no valid short-term study available on toxicity to terrestrial plants.

ECHA notes that the adaptation possibility of Section 1.5. of Annex XI allows registrants to fulfil information requirements by predicting the required data by using the information from the proposed read-across substance. However, as the data submitted is insufficient even for the proposed read-across substance, ECHA does not need to assess whether the conditions for applying the group concept (Annex XI, 1.5. of the REACH Regulation) have been justified by the Registrant.

Furthermore, the Registrant has waived long-term toxicity testing on terrestrial plants using the following justification: *"In accordance with column 2 of REACH Annex X, the long term toxicity testing on plants study does not need to be conducted as the chemical safety assessment according to Annex I has not indicated a need to investigate further the effects of the substance and/or degradation products on terrestrial organisms."*

However, ECHA considers that direct exposure of the soil compartment is probable since the registered substance is used as an agrochemical. Furthermore, in the Chemical Safety Assessment emissions to soil are indicated for 20 out of 22 Exposure scenarios and several RCRs for the soil compartment are close to 1. The highest RCR is [redacted] (Exposure Scenario 5: Coatings-Industrial). The RCRs are based on a PNECsoil derived by the Equilibrium Partitioning Method from a PNECaquatic, which was calculated using the PETROTOX model. However, ECHA considers the PETROTOX model in its current form as scientifically not valid. Therefore, in this decision the Registrant is requested to revise the PNECs for freshwater and marine water (see Section II.C above) considering the experimental data available in the dossier and the results from the long-term fish study requested in this decision (see Section II.B above). The revised aquatic PNECs may affect the outcome of the screening assessment for soil risks.

Hence, ECHA considers that the adaptation provided by the Registrant does not meet the criteria of either the specific adaptation rules of Column 2 of Annexes IX and X, Sections 9.4, or any of the general adaptation rules of Annex XI. Therefore, the adaptation cannot be accepted.

The Registrant provided comments on the draft decision and accepted that there is no valid short-term study available on toxicity to terrestrial plants. The Registrant also confirmed that exposure to soil is expected for the use of xylenes in pesticide formulations therefore the column 2 adaptation "direct and indirect exposure of the soil compartment is unlikely" cannot be applied.

The Registrant proposed to use PETROTOX to derive the terrestrial PNECs for risk assessment. For ECHA's consideration on the PETROTOX model please refer to Section III.C of this decision.

ECHA considers that the the standard information requirement is not fulfilled. Therefore, the information requirement is maintained in the decision.

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

The terrestrial plants growth test (OECD 208), with at least six species tested (with as a minimum two monocotyledonous species and four dicotyledonous species) and the chronic toxicity test in higher plants (ISO 22030) are each considered capable of generating information appropriate for the fulfilment of the information requirements for short-term and long-term toxicity to terrestrial plants (Annex IX, 9.4.3, and Annex X, 9.4.6).

Therefore, pursuant to Article 41(1) and (3) of the REACH Regulation, the Registrant is requested to submit the following information derived with the registered substance subject to the present decision:

Terrestrial plants, growth test (test method: OECD 208), with at least six species tested (with as a minimum two monocotyledonous species and four dicotyledonous species), or Soil Quality – Biological Methods – Chronic toxicity in higher plants, ISO 22030.

OECD guideline 208 (Terrestrial plants, growth test) considers the need to select the number of test species according to relevant regulatory requirements, and the need for a reasonably broad selection of species to account for interspecies sensitivity distribution. For long-term toxicity testing, ECHA considers six species as the minimum to achieve a reasonably broad selection. Testing shall be conducted with species from different families, as a minimum with two monocotyledonous species and four dicotyledonous species, selected according to the criteria indicated in the OECD 208 guideline. The Registrant should consider if testing on additional species is required to cover the information requirement.

c) Effects on soil micro-organisms (Annex IX, 9.4.2.)

The Registrant provides a single key study with the read-across substance o-xylene for this endpoint. The study is non-GLP, non-guideline and is published in peer reviewed literature. The Registrant assigns it a reliability 2. The Registrant provides the following summary of the study in Section 6.3.4 of the IUCLID registration dossier: "*A single study to investigate the effects of o-xylene on soil microorganisms was identified. This study reported a 10 hour EC50 of 220mg/kg nominal. Although this study does not follow the standard guidelines for measuring respiration inhibition of soil microorganisms it is well documented and scientifically acceptable. The test design uses a closed system and analytical monitoring. The exposure period is only 10 hours compared to the standard 28 days. Therefore, although we consider this test reliable, this study will not be used to derive the PNEC in the risk assessment.*"

ECHA considers that the study is not suitable to fulfil the information requirement since the test duration is only 10 hours, compared with 28 days which is required by OECD test guidelines OECD 216 and 217. Furthermore, the only parameter measured was oxygen consumption, which does not directly measure the nitrogen transformation activity of the micro-organisms.

ECHA notes that the adaptation possibility of Section 1.5. of Annex XI allows registrants to fulfil information requirements by predicting the required data by using the information from the proposed read-across substance. However, as the data submitted is insufficient even for the proposed read-across substance, ECHA does not need to assess whether the conditions for applying the group concept (Annex XI, 1.5. of the REACH Regulation) have been justified by the Registrant.

The Registrant provided comments on the draft decision and confirmed that exposure to soil is expected for the use of xylenes in pesticide formulations therefore the column 2 adaptation "direct and indirect exposure of the soil compartment is unlikely" cannot be applied.

The Registrant proposed to use PETROTOX to derive the terrestrial PNECs for risk assessment. For ECHA's consideration on the PETROTOX model please refer to Section III.C of this decision.

ECHA considers that the the standard information requirement is not fulfilled. Therefore, the information requirement is maintained in the decision.

The Registrant also proposed to use the equilibrium partitioning approach to derive the terrestrial PNECs. ECHA emphasises that the effects on soil microbial communities are not addressed through the EPM extrapolation method and therefore the adaptation possibility does not apply for this endpoint. Therefore, the information requirement for effects on soil micro-organisms is maintained in the decision as there is currently no relevant, reliable study for toxicity to soil micro-organisms.

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

According to ECHA *Guidance on information requirements and chemical safety assessment* (version 1.1, November 2012), Chapter R.7C, R.7.11.3.1. p112, the nitrogen transformation test is considered sufficient for most non-agrochemicals. However, as the substance has known agrochemical uses, ECHA considers that both the nitrogen and carbon transformation tests should be performed simultaneously.

Therefore, pursuant to Article 41(1) and (3) of the REACH Regulation, the Registrant is requested to submit the following information derived with the registered substance subject to the present decision:

Soil microorganisms: nitrogen transformation test (test method : EU C.21./OECD 216) and Soil microorganisms: carbon transformation test (test method: EU C.22./OECD 217).

Notes for consideration by the Registrant

Subsequently to the requested revision of the PNECaquatic, the Registrant may consider the Integrated Testing Strategy as recommended in section R.7.11.6., Chapter R.7c of the ECHA Guidance on information requirements and chemical safety assessment (version 1.1, November 2012), and determine the need for further testing on terrestrial organisms as requested under Section II.B above. However, ECHA emphasises that the intrinsic properties of soil microbial communities are not addressed through the EPM extrapolation method and therefore the adaptation possibility does not apply for this endpoint.

C. Information related to the chemical safety assessment and chemical safety report

Pursuant to Articles 10(b) and 14(1) of the REACH Regulation the registration shall contain a chemical safety report (CSR) which shall document the chemical safety assessment conducted in accordance with Article 14(2) to (7) and with Annex I of the REACH Regulation.

In accordance with Annex I, 3.3.1. of the REACH Regulation, a PNEC for each environmental sphere shall be established based on the available information and shall be reported in the CSR. While the Registrant has reported experimental key studies in the IUCLID file in Section 6, the results of these key studies are not used for deriving PNEC values.

Non-compliance of PNECs freshwater and marine water

The PNEC values used in the risk assessment in the registration dossier are derived from environmental risk assessment IT tools, namely the PETROTOX IT tool, with an applied assessment factor of 1. PETROTOX is a quantitative structure-activity relationship ((Q)SAR) tool based on the target lipid model (TLM) which predicts the toxicity of petroleum products to aquatic organisms.

As the effect values used in PNEC derivation are derived using a (Q)SAR model, namely the TLM via PETROTOX, and not on the basis of the experimental key studies available to the Registrant, ECHA considers that the Registrant has chosen to adapt the relevant standard information requirements from Annexes VII to X which are necessary for environmental hazard assessment in accordance with Annex I, Section 3. of the REACH Regulation by employing (Q)SARs, in accordance with Annex XI, 1.3. of the REACH Regulation.

Annex XI, Section 1.3. sets out the conditions which must be fulfilled in order for the results of (Q)SARs to be acceptable as a replacement for testing:

- Results are derived from a (Q)SAR model whose scientific validity has been established,
- The substance falls within the applicability domain of the (Q)SAR model,
- Results are adequate for the purpose of classification and labelling and/or risk assessment, and
- Adequate and reliable documentation of the applied method is provided.
- ECHA considers that the results from the application of the PETROTOX tool fail to meet the first condition above as the scientific validity of this tool, and in particular the target lipid model upon which the tool is based, has not been sufficiently established. Consequently, the PETROTOX tool is not suitable for classification and labelling or risk assessment.

PETROTOX in its current form is considered as not scientifically valid for the following reasons[1]:

- Insufficient number of taxonomic groups used in the acute and chronic Species sensitivity distributions (SSDs),
- Shortcomings in the acute-to-chronic ratio (ACR) derivation,
- Shortcomings in the HC5 derivation: the assumption of a normal distribution, which is not met for log CTLBB (critical target lipid body burden) and log ACR (acute to chronic ratio) and the assumption of independent parameters, which is not met for the combination of CTLBB and the universal slope for narcosis,
- Underestimation of chronic toxicity when compared with data from experimental studies.

Consequently ECHA considers that there is an inconsistency between the available experimental data not used in the PNEC derivation and the reported PNECs derived from the PETROTOX tool and that the PNECs for freshwater and marine water in the registration

[1] Emiel Rorije, Eric M.J. Verbruggen & Joop A. de Knecht. Service Request on a critical review of the environmental and physicochemical methodologies commonly employed in the environmental risk assessment of petroleum substances in the context of REACH registrations (05 August 2012, Version 4).

dossier are not valid. Hence, new PNECs shall be derived for the reasons outlined above and also specified further in Annex I.

During the commenting period the Registrant pointed out that the use of PETROTOX for Chemical Safety Assessment is currently being addressed with CONCAWE and any proposed amendments or changes to PETROTOX should be awaited.

ECHA has taken note of the significant investigations proposed by CONCAWE to improve the PETROTOX model. However, ECHA considers that the PETROTOX model is specific for complex petroleum substances whereas the present decision is referred to a well characterized mono-constituent substance. Therefore, in this specific case, ECHA is of the opinion that the effects values used in PNEC derivation should be based on experimental studies.

Therefore, based on the examination of the technical dossier, ECHA concludes that the information therein, submitted by the Registrant for registration of the above mentioned substance for the purpose of registration within the applicable tonnage band of 1000 tonnes or more per year in accordance with Article 6 and 11(1) of the REACH Regulation, does not comply with the requirements of Articles 10(b), 13 and 14 or with Annexes I and XI thereof.

Consequently, the Registrant is requested to submit the information that is needed to bring the registration into compliance with the relevant information requirements.

On the basis of the above considerations, the Registrant is requested to revise the PNECs for freshwater and marine water in an updated registration dossier.

IV. Adequate identification of the composition of the tested material

ECHA stresses that the information submitted by other joint registrants for identifying the substance 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 information required by the present decision, the sample of substance used for the new studies must be suitable for use by all the joint registrants. Hence, the sample should have a composition that is within the specifications of the substance composition that are given by the joint registrants. It is the responsibility of all joint registrants who manufacture or import the same substance to agree on the appropriate composition of the test material and to document the necessary information on their substance composition.

In addition, it is important to ensure that the particular sample of substance tested in the new studies is appropriate to assess the properties of the registered substance, taking into account any variation in the composition of the technical grade of the substance as actually manufactured by each registrant. If the registration of the substance by any registrant covers different grades, the sample used for the new studies must be suitable to assess these grades.

Finally there must be adequate information on substance identity for the sample tested and the grade(s) registered to enable the relevance of the studies to be assessed.

V. Information on right to appeal

An appeal may be brought against this decision to the Board of Appeal of ECHA under Article 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 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]

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Director of Evaluation

Annex I

Detailed justification why the PETROTOX tool in its current form is considered as not scientifically valid

Species sensitivity distribution on acute data

Although the SSD contains 47 species, there is no higher aquatic plant (macrophyte) or blue-green algae (cyanophyte) included. Both are ecologically important organism groups. According to the ECHA *Guidance on information requirements and chemical safety assessment* (May 2008) the minimum species requirements when using the SSD method are fish, a second family in the phylum *Chordata*, a crustacean, an insect, a family in a phylum other than *Arthropoda* or *Chordata*, a family in any order of insect or any phylum not already represented, algae and higher plants. Because higher plants belong to the eight taxonomic groups defined as minimum requirement to perform a SSD this minimum number of taxonomic groups is thus not met. Therefore, although CTLBBs are present for 47 species, the Registrant did not justify why the SSD approach with less than the required taxonomic groups would still be acceptable.

Acute to chronic ratio (ACR)

A number of aspects lead to an underestimation of the ACR in PETROTOX:

- It is noted that in case other acute effects were not observed, behavioural effects were used instead in deriving the ACR used in the IT tools. Behavioural effects occur at lower concentrations than the standard acute endpoints such as mortality, immobility or population growth and consequently will lead to an underestimation of the ACRs. Importantly, because these behavioural effects are not considered in the construction of the target lipid model, it is also not appropriate to use ACRs which are derived based on these effects.
- Overall, the distribution of ACR values used for petroleum substances in the target lipid model does not cover the full range of available ACR values (e.g. the high ACRs for some crustaceans are not taken into account). Therefore, the ACR taking into account all available information[2,3] (including the high ACRs) will lead to a higher ACR than the one used in the IT tools as presented by the Registrant. Consequently the chronic toxicity may be underestimated.
- According to the REACH guidance[4] the minimum species requirements when using the SSD method are fish, a second family in the phylum *Chordata*, a crustacean, an insect, a family in a phylum other than *Arthropoda* or *Chordata*, a family in any order of insect or any phylum not already represented, algae and higher plants. The ACRs available in the IT tools cover eleven species (two algal, three crustacean, four fish, one insect, and one rotifer species). Therefore, the number of species (marginally) meets the REACH guidance requirements for using a chronic SSD: "*...at least 10 NOECs (preferably more than 15)...* " However, the conditions for using a chronic SSD regarding the number and type of taxonomic groups are not met as this set is lacking 2 of the 8 required taxonomic groups "*a family in any order of insect or any phylum not already represented*" and "*higher plants*".
- A comparison of the mean (3.83) and 95th percentile (13) ACRs derived in the TLM with the ACRs derived from experimental data on polycyclic aromatic hydrocarbons (PAH) showed that a substantial number of these PAH ACRs are above the TLM's 95th

[2] Emiel Rorije, Eric M.J. Verbruggen & Joop A. de Knecht. Service Request on a critical review of the environmental and physicochemical methodologies commonly employed in the environmental risk assessment of petroleum substances in the context of REACH registrations (05 August 2012, Version 4).
[3] Verbruggen *et al.*, 2008; Ahlers *et al.*, 2006; Raimondo *et al.*, 2007.
[4] ECHA guidance on information requirements and chemical safety assessment, R.10.3.1.3. (May 2008).

percentile ACR (i.e. >13) [5]. Therefore, the mean and 95th percentile ACR used in the TLM is not protective for at least these PAHs.

Equation used to arrive at a HC5 (hazardous concentration for 5% of species)

For the equation to calculate HC5 to be correct the individual parameters for which the uncertainty is accounted for should be independent of each other and should be log normally distributed. However, the assumption of independence of parameters is not met for the combination of CTLBB and the universal slope for narcosis[2]. For the intercept, the variance is only based on the variance in estimated CTLBBs. However, the variance in the intercept is correlated to the variance of the slope as well (i.e. the higher the slope, the lower the intercept). On top of that, the general intercept of the relationship between the logarithms of K_{mw} and K_{ow} is now assumed to be zero but should be added to these CTLBBs. This intercept has its own variance and is also not independent of the universal slope.

Furthermore, two of the three parameters used in the equation (slope, CTLBB, and ACR) do not follow a log normal distribution. The SSD on the CTLBB does not follow a normal distribution as expected, and the ACR should not be normally distributed based on theoretical considerations, although the choice of data for this parameter is probably more influential on the final result. Therefore, the requirement that the parameters are normally distributed is not met. Essentially, the equation which is used to derive the HC5 which is subsequently used to derive the PNECs is not scientifically valid as the fundamental assumptions made in its derivation have been shown to be incorrect.

Comparison of the HC5 with experimental chronic data

The target lipid model has been compared with a limited number of chronic toxicity studies on petroleum compounds[5]. The chronic HC5 levels are higher than HC5 values derived directly from experimental data for individual substances and HC5 values derived in a comparable way from chronic toxicity data for PAHs and petroleum products. Some reasons for this discrepancy are the selection of ACRs and the dependency of the parameters CTLBB and universal slope for narcosis as explained above. Also a comparison between the chronic values derived with the TLM used in the PETROTOX IT tool and chronic toxicity data from experimental studies showed some differences. In general, acute toxicity of monoaromatics, polycyclic aromatic hydrocarbons, and oil and PAH mixtures were fairly well predicted, with the accuracy within a factor 3 to 5. For chronic toxicity, however, the results were more variable and the toxicity has been shown to be underestimated by up to a factor of 44. This indicates that the TLM used in the PETROTOX IT tool in their present form, with the errors outlined above is not a conservative or protective approach for all relevant substances. Importantly, the Registrant did not provide a thorough validation of the PETROTOX IT tool with high quality experimental acute and chronic data.

ECHA notes that the justification for applying an assessment factor of 1 to the chronic HC5 when deriving the PNEC is not sufficient and does not address all of the uncertainties linked with the PNEC value. In accordance with Annex I, 3.3.1. of the REACH Regulation a PNEC may be calculated by applying an appropriate assessment factor to the effect values. Given the variable composition of many petroleum substances and the uncertainty associated with mass distribution over hydrocarbon blocks and considering also the uncertainty attached to the number and the physico-chemical properties of individual constituents of the library included in the PETROTOX IT tool to represent petroleum substances this assessment factor is not substantiated.

[5] Emiel Rorije, Eric M.J. Verbruggen & Joop A. de Knecht. A critical review of the environmental and physicochemical methodologies commonly employed in the environmental risk assessment of petroleum substances in the context of REACH registrations. 05 August 2012, Version 4.