ECHA
EUROPEAN CHEMICALS AGENCY

Decision number: CCH-D-2114293124-52-01/F Helsinki, 3 June 2015

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

For diphenyl ether, CAS No 101-84-8 (EC No 202-981-2), 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 diphenyl ether, CAS No 101-84-8 (EC No 202-981-2), submitted by [redacted] (Registrant). The scope of this compliance check decision is limited to the standard information requirements of Annexes VII to X, Section 9 of the REACH Regulation (ecotoxicological information) and of Annex I, Sections 3 to 6 of the REACH Regulation (Chemical Safety Report, specifically, environmental hazard assessment, PBT and vPvB assessment, environmental exposure assessment and environmental risk characterisation).

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 4 September 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 compliance check was initiated on 11 October 2013.

On 13 December 2013 ECHA sent the draft decision to the Registrant and invited him in accordance with Article 50(1) to provide comments on the draft decision. That draft decision was based on submission number [redacted]

On 23 January 2014 ECHA received comments from the Registrant on the draft decision and on 30 April 2014 the Registrant updated his registration dossier with the submission number [redacted]

The ECHA Secretariat considered the Registrant's comments and update.
On basis of this information, Section II was amended. The Statement of Reasons (Section III) was changed accordingly.

On 4 September 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, proposals for amendment to the draft decision were submitted.

On 10 October 2014 ECHA notified the Registrant of the proposals for amendment to the draft decision and invited him pursuant to Article 51(5) of the REACH Regulation to provide comments on the proposals for amendment within 30 days of the receipt of the notification.

The ECHA Secretariat reviewed the proposals for amendment received and amended the draft decision.

On 20 October 2014 ECHA referred the draft decision to the Member State Committee.

By 10 November 2014, in accordance to Article 51(5), the Registrant provided comments on the proposals for amendment. In addition, the Registrant provided comments on the draft decision. The Member State Committee took the comments of the Registrant on the proposals for amendment into account. The Member State Committee did not take into account the Registrant's comments on the draft decision as they were not related to the proposals for amendment made and are therefore considered outside the scope of Article 51(5).

After discussion in the Member State Committee meeting on 8-11 December 2014, a unanimous agreement of the Member State Committee on the draft decision as modified at the meeting was reached on 11 December 2015.

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

II. Information required

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

Pursuant to Articles 41(1), 41(3), 10(a) (vii), 12(1)(e), 13 and Annexes VII and VIII 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. Growth inhibition study on aquatic plants (Annex VII, 9.1.2.; as specified in section III.A.1. of this decision);
2. Activated sludge respiration inhibition testing (Annex VIII, 9.1.4.; test method: Activated sludge, respiration inhibition test (carbon and ammonium oxidation), OECD 209).

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 requests in this decision, or to fulfil otherwise the information requirements with a valid and documented adaptation, will result in a notification to the Enforcement Authorities of the Member States.

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

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

Environmental exposure assessment and risk characterisation as specified in section III.B. (Annex I, sections 5 and 6).

C. Deadline for submitting the required information

Pursuant to Articles 41(4) and 22(2) of the REACH Regulation the Registrant shall submit to ECHA by **10 June 2016** an update of the registration dossier containing the information required by this decision.

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 derived from the application of Annexes VII to XI

Pursuant to Articles 10(a) (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. Growth inhibition study on aquatic plants (Annex VII, 9.1.2.)

"Growth inhibition study on aquatic plants" is a standard information requirement as laid down in Annex VII, Section 9.1.2. 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 Registrant initially submitted an old non-GLP study with *Selenastrum capricornutum* (new name: *Pseudokirchnerella subcapitata*) ([redacted], 1980). In his update of 30 April 2014, the Registrant provided in addition a new key study for the same species ([redacted] 2005) and a QSAR result (ECOSAR v.1.11, US EPA).

According to Annex XI, Section 1.1.2. of the REACH Regulation, data from experiments not carried out according to GLP shall meet the following conditions in order to be considered equivalent to data generated by the corresponding test methods referred to in Article 13(3) of the REACH Regulation:

1) adequacy for the purpose of classification and labelling and/or risk assessment;
2) adequate and reliable coverage of the key parameters foreseen to be investigated in the corresponding test methods referred to in Article 13(3);

3) exposure duration comparable to or longer than the corresponding test methods referred to in Article 13(3) if exposure duration is a relevant parameter; and
4) adequate and reliable documentation of the study is provided.

The study summary provided for the algae study of [redacted], 1980 does not include information on whether the validity criteria, as defined in the OECD test guideline 201, have been fulfilled[1].

Furthermore, no analytical monitoring of the test concentrations has been performed for this study, and there is no indication that the test procedure has been modified to minimise volatilisation losses of the substance (e.g. sealed vessels, reduced headspace). The substance is relatively volatile: the Registrant indicates a Henry's Law constant of 0.00012 $atm.m^3.mol^{-1}$ (12 $Pa.m^3.mol^{-1}$) in the CSR. Therefore, losses of the substance during the performance of this study cannot be excluded. As only nominal concentrations are reported in the robust study summary, the actual tested concentrations at the end of the study cannot be verified and the EC50 values reported for this study thus cannot be considered reliable.

Therefore the study of [redacted], 1980 does not meet the conditions set out in Annex XI, Section 1.1.2. of the REACH Regulation and thus cannot be considered equivalent to data generated by the corresponding test methods referred to in Article 13(3). It cannot be regarded as adequate, in particular for the purpose of the risk assessment and classification and labelling of the substance.

The newly submitted additional study on algae from [redacted] 2005 seems to be in itself appropriate to meet the information requirement of Annex VII, 9.1.2., in particular because the test concentrations have been measured. However, ECHA cannot yet establish compliance with the respective information requirement as the measured concentrations show that after 72h the test concentrations fell systematically below 60% of the nominal concentrations and as low as 45% as shown in Table 1-1 of document "[redacted]" attached to the IUCLID dossier. The results provided in the dossier (72h-EC50 of 0.405 mg/L and 72h-NOEC of 0.25 mg/L) have been calculated from concentrations measured at the beginning of the test (i.e. measured initial concentrations) and do not reflect the rather drastic decrease of the test concentrations by the end of the test period. Furthermore, ECHA notes that according to Table 1-2 of document "[redacted]" attached to the IUCLID dossier, no significant decrease of the test substance concentrations was observed after 72h in the blank test (control with test substance and no algae). For meeting the information requirement of Annex VII, 9.1.2. the Registrant shall thus clarify why a drop of concentrations was observed during the actual test, whereas this was not observed in the blank test. Alternatively, the Registrant shall calculate new EC50 and NOEC based on concentrations measured at the end of the test period or, if appropriate, based on time-weighted mean concentrations.

[1] These quality criteria are as follows:
- The biomass in the control cultures should have increased exponentially by a factor of at least 16 within the 72-hour test period. If this criterion is not met because the species grow slower, then the period should be extended to obtain at least a 16-fold growth in control cultures, while the growth has to be exponential throughout the test period. The test period may be shortened to at least 48 h to maintain unlimited exponential growth during the test, as long as the minimum multiplication factor of 16 is reached.
- The mean coefficient of variation for section-by-section specific growth rates (days 0-1, 1-2 and 2-3, for 72-hour tests) in the control cultures must not exceed 35 %. This criterion applies to the mean value of coefficients of variation calculated for replicate control cultures.
- The coefficient of variation of average specific growth rates during the whole test period in replicate control cultures must not exceed 7 % in tests with *Pseudokirchneriella subcapitata* and *Desmodesmus subspicatus*. For other less frequently tested species, the value should not exceed 10 %.

In addition, ECHA notes that the newly submitted QSAR result (ECOSAR v.1.11, US EPA) is provided as weight of evidence. As specified in Annex XI, 1.2. of the REACH Regulation, a weight of evidence approach could only be applied if several independent source of information are available. As shown above, the study of [redacted], 1980 is deemed to be inadequate, and the results calculated for the study of [redacted], 2005 (EC50 and NOEC) shall be clarified or be recalculated and thus currently cannot be considered valid. The weight of evidence proposed by the Registrant thus cannot be accepted as such.

Therefore, pursuant to Article 41(1) and (3) of the REACH Regulation, the Registrant is requested to provide adequate information on growth inhibition on aquatic plants with the registered substance subject to the present decision. In order to demonstrate that the results of the newly submitted study from [redacted]., 2005 comply with the information requirement of Annex VII, 9.1.2., the Registrant shall either:

- justify why he has calculated EC50 and NOEC values from initial measured concentrations and ignored the fact that the test concentrations dropped drastically by the end of the test period, or
- calculate new EC50 and NOEC values based on concentrations measured at the end of the test period or based on time-weighted mean concentrations.

Moreover, ECHA notes that in the dossier submitted on 30 April 2014, the PNEC values have not been revised. The Registrant shall take into account the new results for the derivation of PNECs, and the chemical safety report shall be amended accordingly.

Note for consideration by the Registrant:

The Registrant is reminded that according to ECHA *Guidance on information requirements and chemical safety assessment* (version 1.2, November 2012), Chapter R7b, Figure R.7.8-4 page 56, if there is neither adequate information on short-term toxicity for three trophic levels nor any other adequate toxicity information (e.g. on long-term toxicity), and if there is no mitigating factor indicating that aquatic toxicity is unlikely, then tests on algae (referred to under Section II.A.2 of this decision) and *Daphnia* (referred to under Section II.A.1 of this decision) are to be conducted. Furthermore, if there is no indication that fish is likely to be less sensitive, the Registrant shall consider performing also a short-term toxicity testing on fish (Annex VIII 9.1.3.), starting with a limit test.

2. Activated sludge respiration inhibition testing (Annex VIII, 9.1.4.)

"Activated sludge respiration inhibition testing" is a standard information requirement as laid down in Annex VIII, Section 9.1.4. 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.

Column 2 of Annex VIII, Section 9.1.4. specifies that this study does not need to be conducted if:

- there is no emission to a sewage treatment plant, or
- there are mitigating factors indicating that microbial toxicity is unlikely to occur, for instance the substance is highly insoluble in water, or
- the substance is found to be readily biodegradable and the applied test concentrations are in the range of concentrations that can be expected in the influent of a sewage treatment plant.

The Registrant has waived activated sludge respiration inhibition testing using the following justification:

> *"Diphenyl oxide (DPO or Diphenyl Ether) is readily biodegradable and due to limited exposure to sewage treatment plants (STPs), no further toxicity testing with microorganisms is proposed. The PNECstp is derived from results of ready biodegradability test conducted at 100 mg/L, showing no apparent toxicity/inhibition of the microbial inoculum".*

The justification for omission information on activated sludge respiration inhibition provided by the Registrant does not meet the criteria of either the specific adaptation rules of Column 2 of Annex VIII, section 9.1.4., or the general adaptation rules of Annex XI:

- Firstly, the Registrant considers that the registered substance is readily biodegradable: 76% degradation was observed after 20 days (64% degradation after 5 days) in a BOD (Biochemical Oxygen Demand) test at a test material concentration of 5.6 mg/L (The Dow Chemical Company, 1976). However, for calculating the PNEC for sewage treatment plants (PNECstp), the Registrant has used the concentration of 100 mg/L as a no observed effect concentration (NOEC) for sewage treatment plant (STP) microorganisms. The concentration of 100 mg/L is actually the test concentration used for another biodegradation study presented in the registration dossier (modified MITI test from Chemicals Inspection and Testing Institute, Japan, 1992). In this latter test, only 6.3% degradation was observed after 14 days at the test concentration of 100 mg/L. As the biodegradation tests lack toxicity controls and the toxicity study on STP microorganisms is omitted, it is not possible to rule-out that the registered substance might have toxic effects on microorganisms at high test concentrations. Therefore the available information does not support 100 mg/L being a NOEC for STP microorganisms.

- Secondly, the Registrant claims that there is limited exposure to sewage treatment plants. However, even if supposedly limited, the Registrant implicitly acknowledges that exposure still occurs and omission of this study based on column 2 of Annex VIII, 9.1.4 can be based only on no emission to a sewage treatment plant. Therefore hazard to STP microorganisms needs to be addressed.

Therefore, the adaptations proposed by the Registrant cannot be accepted and 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.

In his comments submitted on 23 January 2014, the Registrant agreed to conduct the requested activated sludge respiration inhibition study (Annex VIII, 9.1.4.; test method: Activated sludge, respiration inhibition test (carbon and ammonium oxidation), OECD 209), and committed to initiate the testing by the end of 2014. ECHA takes note of the Registrant's intention to conduct the requested study according to the requested test guideline. By 4 September 2014, the date upon which ECHA notified its draft decision to the Competent Authorities of the Member States, this information is still missing in the registration dossier. Therefore ECHA has not amended Section II of this decision with regard to this information requirement.

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: activated sludge respiration inhibition testing, test method:

Activated sludge, respiration inhibition test (carbon and ammonium oxidation), OECD 209. The study result shall be taken into account for the derivation of PNECstp, and the chemical safety report shall be amended accordingly.

B. 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 which shall document the chemical safety assessment conducted in accordance with Article 14(2) to (7) and with Annex I of the REACH Regulation.

According to Article 14(4) of the REACH Regulation, if the registered substance fulfils the criteria for any of the hazard classes of Annex I to Regulation (EC) No 1272/2008 listed in Article 14(4) of the REACH Regulation or is assessed to be a PBT or vPvB, the chemical safety assessment (CSA) shall include an exposure assessment and risk characterisation. The exposure assessment shall be carried out according to section 5 of Annex I and shall include exposure scenarios and exposure estimations for the registered substance. The exposure assessment shall consider all stages of the life-cycle of the substance resulting from the manufacture and identified uses and shall cover any exposures that may relate to the identified hazards. Annex I, section 6 of the REACH Regulation requires the Registrant to characterise the risk for each exposure scenario.

Pursuant to Annex I, section 5.2.1. of the REACH Regulation, the exposure estimation entails three elements: (1) emission estimation, (2) assessment of chemical fate and (3) pathways and estimation of exposure levels. Emission estimation shall be performed under the assumption that the risk management measures (RMMs) and operational conditions (OCs) described in the exposure scenarios (ES) have been implemented. These RMMs and OCs should be included in the ESs provided in a Chemical Safety Report (CSR).

According to the *Guidance on information requirements and chemical safety assessment Chapter R.16: Environmental Exposure Estimation* (ECHA, version: 2.1, October 2012) the exposure scenario should contain information (about operational conditions and risk management measures) based on which the assumed release factors and daily use rates can be justified. The Guidance further indicates that sector specific environmental release categories (spERCs) developed by industrial sector organisations may be used instead of the more conservative default environmental release categories (ERCs) of ECHA's guidance. As far as possible, spERCs have to be linked to the RMM and OC driving the release estimation.

The substance is self-classified as Eye Irritant. 2A and as Aquatic Chronic 2. As the substance meets the criteria for classification, the CSA shall include exposure assessment and risk characterisation.

In the previous version of the dossier (submission number [redacted]), the Registrant presented 6 exposure scenarios:
- ES1: manufacture of the substances;
- ES2: formulation of fragrances and substances;
- ES3: chemical processing (intermediates & solvents);
- ES4: transportation and (re)packing of substances and mixtures;
- ES5: heat transferring of fluids (industrial);
- ES6: consumer use of fragrances.

Two issues regarding the environmental exposure assessment and risk characterisation were identified in the previous version of the dossier (submission number [redacted]) and were addressed in the draft decision sent to the Registrant on 13 December 2013:

- lack of environmental exposure and risk assessments and lack of justification for assuming absence of environmental releases for ES1, ES3, ES4 and ES5,

- applicability of spERC for ES2.

1. Lack of environmental exposure and risk assessments and lack of justification for assuming absence of environmental releases for ES1, ES3, ES4 and ES5

For exposure scenarios ES1, ES3, ES4 and ES5, ECHA noted that the Registrant claimed that environmental releases were not expected to occur due to "*tightly controlled containment systems*". In particular, the operational conditions and the risk management measures necessary to achieve the absence of environmental releases were not specified in the CSR.

Furthermore, the Registrant did not provide environmental exposure and risk assessments for any of these 4 exposure scenarios. No justification was provided either.

The claim that only tightly controlled containment systems are used is debatable in view of the further scenarios that were described for the exposure assessment of workers, e.g. during "equipment cleaning and maintenance", "bulk transfers", "transfer from/pouring from containers", "drum and small package filling", "waste treatment and storage". By nature, these processes are not expected to be strictly controlled with regards to exposure. In addition, the CSR indicated in several instances that local exhaust ventilation (LEV) should be used, but did not specify how the contaminated air should be treated to ensure that there is not emission of the substance into the environment.

2. Applicability of spERC for ES2

For the environmental exposure estimation for exposure scenario ES2 ("Formulation of fragrances and substances"), ECHA noted that the Registrant had used release factors that were said to be based on so called spERC "AISE1". The release factors used for ES2 were very low: 0.02% for the air, 0.01% for water, 0% for soil. By comparison, the default release factors recommended by ECHA Guidance R.16 for ERC2 (Formulation of mixtures) are: 2.5% for the air, 2% for water, 0.01% for soil.

Based on the AISE internet website[2], there is no such spERC with denomination "AISE1". This denomination may be outdated. As no further information is available on the spERC invoked by the Registrant, ECHA could not assess whether the release factors that were used by the Registrant for exposure scenario ES2 were relevant. In particular, there was no information on whether this spERC should apply to large, medium or small scale plants. This is relevant as in general, the smaller the plant is, the larger are the release factors.

3. Outcome

In his comments submitted on 23 January 2014, the Registrant agreed to conduct the changes requested in the draft decision sent on 13 December 2013 and committed to amend the chemical safety report accordingly by the end of 2014. However, the updated

[2]http://www.aise.eu/reach

registration dossier submitted on 30 April 2014 (submission number [redacted]) does not contain an exposure assessment and risk characterisation anymore.

ECHA takes note of the Registrant's intention to provide revised environmental exposure assessment and risk characterisation. However, ECHA notes that by 4 September 2014, the date upon which ECHA notified its draft decision to the Competent Authorities of the Member States, information on exposure assessment and risk characterisation is totally missing in the registration dossier.

Therefore, pursuant to Article 41(1)(c) and 41(3) of the REACH Regulation, the Registrant is requested to develop exposure scenarios relevant for the registered substance and to provide environmental exposure and risk assessments. If he assumes absence of environmental release for these exposure scenarios, he shall then provide a clear and detailed justification (e.g. based on RMMs and/or OCs and/or substance properties). If he intends to use spERCs, he shall then clearly specify the applicability domain of those spERCs (e.g. size of the formulating plant, link to the RMM and OC driving the release estimation). Alternatively, exposure estimation can be based on relevant ERC default release factors as set out in ECHA's Guidance, refined on the basis of RMMs and/or OCs and/or substance properties as appropriate. In any case, the CSR shall be amended accordingly.

Note for consideration by the Registrant:

ECHA notes that the Registrant suggested in his comments on the proposals for amendments that he carries out an OECD 301F test, in order to ascertain the ready biodegradability of the substance and the need for a biodegradation simulation test. The follow up to the present decision will consider the compliance of the endpoints on biodegradation as per Annex IX 9.2. based on the information available in the dossier at that point of time and decide on further regulatory measures, including the need for a further biodegradation study, as appropriate.

IV. Adequate identification of the composition of the tested material

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://www.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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