~~Confidential~~

Helsinki, 31 August 2021

Addressees
Registrant(s) of Joint_Black46 as listed in the last Appendix of this decision

Date of submission of the dossier subject to this decision
30/08/2018

Registered substance subject to this decision ("the Substance")
Substance name: [4-[p,p'-bis(dimethylamino)benzhydrylidene]cyclohexa-2,5-dien-1-ylidene]dimethylammonium m-[[p-anilinophenyl]azo]benzenesulphonate
EC number: 265-449-9
CAS number: 65113-55-5

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

DECISION ON A COMPLIANCE CHECK

Under Article 41 of Regulation (EC) No 1907/2006 (REACH), you must submit the information listed below, by the deadline of ***7 December 2022***.

Requested information must be generated using the Substance unless otherwise specified.

Many of this type of organic pigments are listed in various national inventories of nanomaterials, such as the French nano-particulate substances reporting system.[1] In the case where the Substance is manufactured and/or imported in the European Union in nanoforms by any addressee of the present decision, the REACH Regulation (as amended by Regulation Commission Regulation (EU) 2018/1881) sets out explicit information requirements for nanoforms of substances. Manufacturers and/or importers of nanoforms must have fulfilled these specific information requirements by 1st January 2020. As far as the registration dossiers currently submitted on the Substance by any addressee of the present decision they do not cover any nanoform. Any incompliances identified in the present decision on the Substance relate only to information required on non-nanoforms.

Based on the above, the requested information in this present decision must be generated using exclusively non-nanoforms of the Substance.

A. Information required from all the Registrants subject to Annex VII of REACH

1. Water solubility (Annex VII, Section 7.7.; test method: EU A.6./OECD TG 105

2. Partition coefficient n-octanol/water (Annex VII, Section 7.8.;OECD TG 117 or OECD TG 123)

3. Growth inhibition study aquatic plants (Annex VII, Section 9.1.2.; test method: EU C.3./OECD TG 201)

B. Information required from all the Registrants subject to Annex VIII of REACH

1. Adsorption/ desorption screening (Annex VIII, Section 9.3.1.; test method: OECD TG

[1] "*Dispositif de déclaration des substances à l'état nanoparticulaire* », Decree 2012-232 of French *Conseil d'Etat* of 17 February 2012.

106 or OECD TG 121

C. Information required from all the Registrants subject to Annex IX of REACH

1. Dissociation constant (Annex IX, Section 7.16.; test method OECD TG 112)

Reasons for the request(s) are explained in the following appendices:

- Appendices entitled "Reasons to request information required under Annexes VII to IX of REACH", respectively.

Information required depends on your tonnage band

You must provide the information listed above for all REACH Annexes applicable to you, and in accordance with Articles 10(a) and 12(1) of REACH:

- the information specified in Annexes VII and VIII to REACH, for registration at 10-100 tpa;
- the information specified in Annexes VII, VIII and IX to REACH, for registration at 100-1000 tpa.

You are only required to share the costs of information that you must submit to fulfil your information requirements.

How to comply with your information requirements

To comply with your information requirements you must submit the information requested by this decision in an updated registration dossier by the deadline indicated above. You must also update the chemical safety report, where relevant, including any changes to classification and labelling, based on the newly generated information.

You must follow the general testing and reporting requirements provided under the Appendix entitled "Requirements to fulfil when conducting and reporting new tests for REACH purposes". For references used in this decision, please consult the Appendix entitled "List of references".

Appeal

This decision, when adopted under Article 51 of REACH, may be appealed to the Board of Appeal of ECHA within three months of its notification to you. Please refer to http://echa.europa.eu/regulations/appeals for further information.

Failure to comply

If you do not comply with the information required by this decision by the deadline indicated above, ECHA will notify the enforcement authorities of your Member State.

Authorised[2] under the authority of Christel Schilliger-Musset, Director of Hazard Assessment

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

Appendix A: Reasons to request information required under Annex VII of REACH

1. Water solubility

Water solubility is an information requirement under Annex VII to REACH (Section 7.7).

You have provided the following information:

i) key study (2013) with the Substance

We have assessed this information and identified the following issues:

To fulfil the information requirement, a study must comply with the OECD TG 105, or the EU Method A.6 (Article 13(3) of REACH). These test guidelines describe two methods (the shake flask method and column elution method) for conducting the determination of water solubility. The EU Method A.6 specifies that the method selection must be based on properties of the substance and on a preliminary determination of the following range of solubilities:

1. one which applies to essentially pure substances with low solubilities, (< 10^{-2} grams per litre), and which are stable in water, referred to as the 'column elution method;
2. the other which applies to essentially pure substances with higher solubilities (> 10^{-2} grams per litre), and which are stable in water, referred to as the 'flask method'.

You have used a spectrophotometric non-guideline non-GLP water solubility to test different aqueous extract from the powder. You have not provided any justification for the use of that method nor as to why none of the methods listed above are technically feasible.

You have not reported the limit of detection nor the limit of quantification for the applied method for your Substance.

Furthermore, you indicated in the analysis of the results that it was not possible to distinguish what was measured: soluble particles of the Substance or secondary solubilization of insoluble particles solubilized by the methanol used during the dilution step.

Therefore, you have not demonstrated compliance with Article 13(3) of REACH nor with OECD TG 105 or EU Method A.6 nor have you established that the method, or its results, are adequate to conclude on the solubility of the Substance.

In your comments on the initial draft decision, you provide the following additional information to resolve the issue.

"*According to the OECD TG 123 performed on the substance, the mean water solubility of the substance in water saturated with octanol at 25°C, pH 6.2 (i.e. increasing water solubility of the substance, because of an higher solubility in octanol and higher T°C) is of 0.038 mg/L.*"

However, OECD TG 123 is not a recognised test method (Art. 13(3)) for the determination of water solubility as it provides information on equilibrium concentrations of the pure test substance in 1-octanol saturated with water and water saturated with 1-octanol but not the solubility of the pure substance in water.

Furthermore, based on your comments on the initial draft decision, for the endpoint's adsorption/desorption screening test and dissociation constant determination, ECHA's

understands that you will conduct a new water solubility study according to OECD TG 105. ECHA cannot take into account future studies.

Therefore, the provided information does not fulfil the information requirement.

2. Partition coefficient n-octanol/water

Partition coefficient in n-octanol/water is an information requirement under Annex VII to REACH (Section 7.8).

You have provided the following information:

i) Key study (2013) [redacted].

We have assessed this information and identified the following issues:

A. To fulfil the information requirement, a study must comply with the OECD TG 107 or OECD TG 117 or OECD TG 123 or the EU Method A.8 (Article 13(3) of REACH). These test guidelines describe three methods (the shake flask method, the HPLC method and the slow-stirring method) for conducting the determination of the partition coefficient between water and n-octanol (Log Kow). The EU test method A.8 specifies that the method selection must be based on the properties of the substance and on a preliminary determination of Log Kow using the individual solubilities of the test material in water and n-octanol. This preliminary estimate is considered sufficient only if none of the recommended method are technically feasible due to specific substance properties (*e.g.* surface active substances).

   Your robust study summary reports that the Log Kow of the Substance was an extrapolated value determined using the individual solubilities of the test material in water and n-octanol. You have not provided any justification as to why none of the methods listed above are technically feasible.

B. To provide an acceptable determination of the partition coefficient using individual solubilities in water and n-octanol, the calculation must be based on reliable individual solubilities estimates.

   You used the information discussed under Section A.1 as the water solubility estimated used in the calculation. You report that the n-octanol solubility estimate was determined using a shake flask method followed by filtration and photometric analysis.

   As explained under Section A.1, the information provided in your registration does not fulfil the information requirement. Furthermore, as a similar approach was used to determine n-octanol solubility, similar issues identified under Section A.1 also apply to the determination of n-octanol solubility. Hence, the log Kow value reported in your registration dossier is not reliable.

In your comments on the initial draft decision, you submitted a new log Kow value, supported by a summary of an experimental study according to OECD TG 123. ECHA has assessed the information against the requirements in OECD TG 123. The information you have provided in your comments on the initial draft decision addresses the incompliances identified in this decision for this information requirement. However, as the information is currently not available in your registration dossier, the data gap remains. You should therefore submit this information in an updated registration dossier by the deadline set out in the decision.

On the basis of the above, the information requirement is not fulfilled.

Study design

Considering the properties of the Substance (ionisable organic substance), the Partition Coefficient (n-octanol/water), HPLC Method (test method: OECD TG 117) or alternatively the Partition Coefficient (1-Octanol/Water): Slow-Stirring Method (test method: OECD TG 123) are the most appropriate method to fulfil the information requirement for the Substance.

3. Growth inhibition study aquatic plants

Growth inhibition study aquatic plants is an information requirement under Annex VII to REACH (Section 9.1.2).

In support of your adaptation you have provided the following information:

i) A key study (2013) according to OECD TG 201 on the Substance

We have assessed this information and identified the following issues:

To comply with this information requirement, the test material in a study must be representative for the Substance (Article 10 and Recital 19 of REACH; ECHA Guidance R.4.1).

For study "[redacted]" above, you have identified the test material as "[redacted]", EC number [redacted]

You also indicated that you have based the results of the test on the analysis of the substance [redacted]. According to the information provided in section 1.2. of the IUCLID dossier, [redacted] is an impurity present at less than [redacted] in the substance.

This information is not consistent with the above statement on the test material while it raises question on the representativity of the test material.

Therefore, you have not demonstrated that the analysed material is representative for the whole of the Substance.

Further, to fulfil the information requirement, a study must comply with OECD TG 201 (Article 13(3) of REACH). Therefore, the following specifications must be met:

- exponential growth in the control cultures is observed over the entire duration of the test;
- at least 16-fold increase in biomass is observed in the control cultures by the end of the test;
- 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 is ≤ 35%;
- the coefficient of variation of average specific growth rates during the whole test period in replicate control cultures is ≤ 7% in tests with *Pseudokirchneriella subcapitata* the method for determination of biomass and evidence of correlation between the measured parameter and dry weight are reported;
- the results of algal biomass determined in each flask at least daily during the test period are reported in a tabular form;
- microscopic observation performed to verify a normal and healthy appearance of the inoculum culture are reported. Any abnormal appearance of the algae at the end of the test is reported;

In your comments on the initial draft decision, you have included a summary document to address the incompliances of the submitted OECD TG 201. ECHA has assessed the information against the requirement in OECD TG 201. The information you have provided in your comments on the initial draft decision addresses only the incompliances identified concerning the controls. However, the results on algal biomass (raw data) determined in each flask at least daily during the test period are not present. Furthermore, you have not explained the % of the dissociation product(s) expected nor the actual calculation of the dissociation product(s) relative to the Substance.

Your technical dossier does not include the information listed above.

Therefore, you have not demonstrated that the validity criteria of OECD TG 201 were met and the documentation of this study is insufficient and does not allow an independent assessment of the adequacy of this study, its results and its use for hazard assessment. Therefore, the requirements of OECD TG 201 are not met.

On the basis of the above, the information requirement is not fulfilled.

Study design

The Substance is difficult to test due to its ionic properties. OECD TG 201 specifies that, for difficult to test substances, you must consider the approach described in OECD GD 23 or other approaches, if more appropriate for your substance. In all cases, the approach selected must be justified and documented. Due to the properties of Substance, it may be difficult to achieve and maintain the desired exposure concentrations.

Therefore, you must monitor the test concentration(s) of the Substance throughout the exposure duration and report the results. If it is not possible to demonstrate the stability of exposure concentrations (i.e. measured concentration(s) not within 80-120% of the nominal concentration(s)), you must express the effect concentration based on measured values as described in OECD TG 201. In case a dose-response relationship cannot be established (no observed effects), you must demonstrate that the approach used to prepare test solutions was adequate to maximise the concentration of the Substance in the test solution

Appendix B: Reasons to request information required under Annex VIII of REACH

1. Adsorption/ desorption screening

Adsorption/desorption screening is a standard information requirement under Annex VIII, Section 9.3.1.

We understand that you have adapted this information requirement according to Annex XI, Section 2.

You provided the following justification for the adaptation: "The study was technically not feasible because of its low hydrosolubility"

We have assessed this information and identified the following issues:
Under Annex XI, Section 2, a study can be omitted if it is technically not possible as a consequence of the properties of the substance.

You have claimed that it is not technically feasible due to the low hydrosolubility of the Substance, but not provided any supporting information.

In addition to this, you stated that the recommended test guideline OECD 106 may not be suitable for items with a partition coefficient value greater than 1 x 10^{-4}, the partition coefficient of the test item has been estimated to be a log Kow value of greater than 5.7.

You have not demonstrated why the low solubility justified that the study was not technically feasible. Furthermore, as explained under request A.1, your dossier currently does not include a reliable value on the water solubility of the substance.

As explained under request A.2, your dossier currently does not include a reliable value on the partition coefficient of the Substance. You have provided no argument why an OECD 106 would not be suitable, even less technically impossible, in this case.

Therefore, your adaptation is rejected.

In your comments on the initial draft decision, you state that "it might be possible that an adsorption coefficient study for this substance will not be technically feasible." We understand that you want to adapt this information requirement according to Annex XI Section 2.

Under Section 2 of Annex XI to REACH, a study may be omitted if it is technically not possible to conduct as a consequence of the properties of the substance.

You have provided the following:

- OECD TG 106: you stated that " an OECD TG 106 seems not feasible and that based on the similarity of the REG substance to another similar substance not identified and on the expected low water solubility of the substance are not supported by relevant information.
- Regarding OECD TG 121, you state that "is quite sure that with this methodology, it will only be possible to determine the adsorption coefficient of the counterions". Further analytical data confirming this statement is not provided.

However, your claims are unsubstantiated in the absence of relevant information and, in any case, the claim (eg 'seems' not feasible) does not demonstrate that study is technically not possible.

Therefore, your adaptation is rejected and the information requirement is not fulfilled.

Appendix C: Reasons to request information required under Annex IX of REACH

1. Dissociation constant

Dissociation constant is an information requirement under Annex IX to REACH (section 7.16).

You have provided an adaptation based on Column 2, second indent.

We have assessed this information and identified the following issue(s):

According to Annex IX, Section 7.16, Column 2, the study does not need to be conducted if it is scientifically not possible to perform the test.

You have provided the following justification:

"*The substance is insoluble (solubility > 0.01 mg/L) and the test item has no specific absorbance. Therefore, any dissociation of the test item will not lead to a change in the sample solutions on analysis.*"

In your comments on the initial draft decision, you stated that the suitable analytical method available to distinguish the Substance and its potential dissociation products ([redacted]) is currently performed on another similar substance. In addition you state that, results of the water solubility OECD TG 105 should be available before performing a dissociation constant test or consolidating waiving arguments for the endpoint.

First, you argue that the test is not scientifically *needed* not why it is *not scientifically possible* as provided under Column 2. Second, your argument is speculative and, in any case, as explained under request A.1, your dossier currently does not include a reliable value on the water solubility of the Substance. The information provided in your comments on the initial draft decision, do not change this assessment, as future information cannot be taken into account. You indicated your intension to address this information requirement with a read-across adaptation without further explanation. The information in your comments on the initial draft decision, is not sufficient for ECHA to make an assessment in the absence of any further explanation.

Therefore, your adaptation is rejected and the information requirement is not fulfilled.

Appendix D: Requirements to fulfil when conducting and reporting new tests for REACH purposes

A. Test methods, GLP requirements and reporting

1. Under Article 13(3) of REACH, all new data generated as a result of this decision must be conducted according to the test methods laid down in a European Commission Regulation or to international test methods recognised by the Commission or ECHA as being appropriate.

2. Under Article 13(4) of REACH, ecotoxicological and toxicological tests and analyses must be carried out according to the GLP principles (Directive 2004/10/EC) or other international standards recognised by the Commission or ECHA.

3. Under Article 10(a)(vi) and (vii) of REACH, all new data generated as a result of this decision must be reported as study summaries, or as robust study summaries, if required under Annex I of REACH. See ECHA Practical Guide on How to report robust study summaries[3].

B. Test material

Before generating new data, you must agree within the joint submission on the chemical composition of the material to be tested (Test Material) which must be relevant for all the registrants of the Substance.

1. Selection of the Test material(s)

   The Test Material used to generate the new data must be selected taking into account the following:
   - the variation in compositions reported by all members of the joint submission,
   - the boundary composition(s) of the Substance,
   - the impact of each constituent/ impurity on the test results for the endpoint to be assessed. For example, if a constituent/ impurity of the Substance is known to have an impact on (eco)toxicity, the selected Test Material must contain that constituent/ impurity.

2. Information on the Test Material needed in the updated dossier
   - You must report the composition of the Test Material selected for each study, under the "Test material information" section, for each respective endpoint study record in IUCLID.
   - The reported composition must include all constituents of each Test Material and their concentration values and other parameters relevant for the property to be tested.

This information is needed to assess whether the Test Material is relevant for the Substance and whether it is suitable for use by all members of the joint submission.

Technical instructions on how to report the above is available in the manual on How to prepare registration and PPORD dossiers[4].

[3] https://echa.europa.eu/practical-guides
[4] https://echa.europa.eu/manuals

Appendix E: Procedure

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

ECHA followed the procedure detailed in Articles 50 and 51 of REACH.

The compliance check was initiated on 12 February 2020.

ECHA notified you of the draft decision and invited you to provide comments.

ECHA took into account your comments and amended the request(s) by removing the request for a pre-natal developmental toxicity study.

ECHA notified the draft decision to the competent authorities of the Member States for proposals for amendment.

As no amendments were proposed, ECHA adopted the decision under Article 51(3) of REACH.

Appendix F: List of references - ECHA Guidance[5] and other supporting documents

Evaluation of available information
Guidance on information requirements and chemical safety assessment, Chapter R.4 (version 1.1., December 2011), referred to as ECHA Guidance R.4 where relevant.

QSARs, read-across and grouping
Guidance on information requirements and chemical safety assessment, Chapter R.6 (version 1.0, May 2008), referred to as ECHA Guidance R.6 where relevant.

Read-across assessment framework (RAAF, March 2017)[6]

RAAF - considerations on multiconstituent substances and UVCBs (RAAF UVCB, March 2017)

Physical-chemical properties
Guidance on information requirements and chemical safety assessment, Chapter R.7a (version 6.0, July 2017), referred to as ECHA Guidance R.7a in this decision.

Toxicology
Guidance on information requirements and chemical safety assessment, Chapter R.7a (version 6.0, July 2017), referred to as ECHA Guidance R.7a in this decision.

Guidance on information requirements and chemical safety assessment, Chapter R.7c (version 3.0, June 2017), referred to as ECHA Guidance R.7c in this decision.

Environmental toxicology and fate
Guidance on information requirements and chemical safety assessment, Chapter R.7a (version 6.0, July 2017), referred to as ECHA Guidance R.7a in this decision.

Guidance on information requirements and chemical safety assessment, Chapter R.7b (version 4.0, June 2017), referred to as ECHA Guidance R.7b in this decision.

Guidance on information requirements and chemical safety assessment, Chapter R.7c (version 3.0, June 2017), referred to as ECHA Guidance R.7c in this decision.

PBT assessment
Guidance on information requirements and chemical safety assessment, Chapter R.11 (version 3.0, June 2017), referred to as ECHA Guidance R.11 in this decision.

Guidance on information requirements and chemical safety assessment, Chapter R.16 (version 3.0, February 2016), referred to as ECHA Guidance R.16 in this decision.

Data sharing
Guidance on data-sharing (version 3.1, January 2017), referred to as ECHA Guidance on data sharing in this decision.

OECD Guidance documents[7]
Guidance Document on aqueous–phase aquatic toxicity testing of difficult test chemicals – No 23, referred to as OECD GD 23.

[5] https://echa.europa.eu/guidance-documents/guidance-on-information-requirements-and-chemical-safety-assessment
[6] https://echa.europa.eu/support/registration/how-to-avoid-unnecessary-testing-on-animals/grouping-of-substances-and-read-across
[7] http://www.oecd.org/chemicalsafety/testing/series-testing-assessment-publications-number.htm

Guidance document on transformation/dissolution of metals and metal compounds in aqueous media – No 29, referred to as OECD GD 29.

Guidance Document on Standardised Test Guidelines for Evaluating Chemicals for Endocrine Disruption – No 150, referred to as OECD GD 150.

Guidance Document supporting OECD test guideline 443 on the extended one-generation reproductive toxicity test – No 151, referred to as OECD GD 151.

Appendix G: Addressees of this decision and their corresponding information requirements

You must provide the information requested in this decision for all REACH Annexes applicable to you.

Registrant Name	Registration number	Highest REACH Annex applicable to you
[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]

Where applicable, the name of a third party representative (TPR) may be displayed in the list of recipients whereas ECHA will send the decision to the actual registrant.