EUROPEAN CHEMICALS AGENCY

Helsinki, 3 February 2020

Addressees
Registrants of [redacted] listed in the last Appendix of this decision

Date of submission for the dossier subject of this decision
31 October 2018

Registered substance subject to this decision, hereafter 'the Substance'
Substance name: ASPHALT, SULFONATED, SODIUM SALT
EC number: 269-212-0
CAS number: 68201-32-1

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

DECISION ON A COMPLIANCE CHECK

Based on Article 41 of Regulation (EC) No 1907/2006 (REACH), ECHA requests that you submit the information listed below by the deadline of ***10 February 2022***.

A. Requirements applicable to all the Registrants subject to Annex IX of REACH

1. Sub-chronic toxicity study (90-day), oral route (Annex IX, Section 8.6.2.; test method OECD TG 408) in rats with the Substance

2. Pre-natal developmental toxicity study (Annex IX, Section 8.7.2.; test method OECD TG 414) in a first species (rat or rabbit), oral route with the Substance

Conditions to comply with the requests

You are bound by the requests for information corresponding to the REACH Annexes applicable to your own registered tonnage of the Substance at the time of evaluation. Therefore you have to comply with the requirements of Annexes VII, VIII and IX of REACH, if you have registered a substance at 100-1000 tpa.

Appendix A states the reasons for the requests for information to fulfil the requirements set out respectively in Annex IX of REACH.

The test material used to perform the required studies must be selected and reported in accordance with the specifications prescribed in Appendix entitled Observations and technical guidance.

You must submit the information requested in this decision by the deadline indicated above in an updated registration dossier and also update the chemical safety report, where relevant, including any changes to classification and labelling, based on the newly generated information. The timeline has been set to allow for sequential testing where applicable.

Appeal

This decision can be appealed to the Board of Appeal of ECHA within three months of its notification. An appeal, together with the grounds thereof, has to be submitted to ECHA in writing. An appeal has suspensive effect and is subject to a fee. Further details are described under: http://echa.europa.eu/regulations/appeals.

Approved[1] under the authority of Christel Schilliger-Musset, Director of Hazard Assessment

[1] 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 for the requests to comply with Annex IX of REACH

In accordance with Articles 10(a) and 12(1) of the REACH Regulation, a technical dossier registered at 100 to 1000 tonnes or more per year must contain, as a minimum, the information specified in Annexes VII-IX to the REACH Regulation.

1. Sub-chronic toxicity study (90-day), oral route (Annex IX, Section 8.6.2.);

A Sub-chronic toxicity study (90 day) is a standard information requirement in Annex IX to REACH.

You have provided:

1. Screening for reproductive / developmental toxicity study in rats (key study, equivalent to OECD TG 422, GLP compliant, study report 2007).

2. An adaptation according to Annex IX, Section 8.6.2., Column 2 fourth indent. In addition you argue a low bioavailability and low toxicity of the Substance.

We have assessed this information and identified the following issues:

1.) To be considered compliant and enable concluding whether the Substance has dangerous properties and supports the determination of the No-Observed Adverse Effect Level (NOAEL), a study has to meet the requirements of a 90-day repeated dose toxicity study (OECD TG 408). The following key parameter(s) of this test guideline include, among others:

- At least 10 female and 10 male animals should be used at each dose level (including control group)
- dosing of the Substance daily for a period of 90 days until the scheduled termination of the study

You did not provide information equivalent to OECD TG 408. Specifically, the OECD TG 422 you have submitted does not have the required exposure duration of 90 days, because the exposure duration of the screening test is approximately 63 days (for females) and 28 days (for males). Furthermore the organ weight and histopathological investigations in OECD TG 422 are only conducted using 5 animals per sex per group and not 10 per sex per group as in OECD TG 408.

2.) In addition, you provided an adaptation for 90-day study. To adapt standard information requirement for 90-day repeated dose toxicity, the conditions of specific adaptation based on Annex IX, Section 8.6.2, Column 2, or General adaptations, set in Annex XI have to be fulfilled. As indicated in Annex IX, Section 8.6.2, Column 2, you may adapt the information requirement, provided you fulfil the following (cumulative) criteria, including among others that the substance is unreactive, insoluble and there is no evidence of absorption.

Based on the information in the registration dossier:

- The substance is reactive, since it has reactive groups, e.g. alkyl and aryl sulfonates. Furthermore, irritant potency of the Substance is reported in the *in vivo* skin irritation/corrosion and eye irritation studies ([redacted] 2008). Therefore, the Substance cannot be considered as unreactive.

- The reported water solubility of the Substance is approx. 48.5% w/w, which qualifies it as water soluble.

- There are statistically significant effects in clinical chemistry (decreased plasma calcium, phosphorus and urea) in males, reported in the OECD TG 422 study. This means that the Substance is systemically available as a result of absorption in the organism.

Based on this information, you have not met the criteria above, as the substance is reactive, water soluble and absorbable.

Your additional arguments of low bioavailablity and low toxicity of the Substance are not an adaptation option, neither in Annex IX, Section 8.6.2, Column 2, nor in Annex XI.

Hence your adaptation is rejected.

In your comments to the draft decision you agreed with ECHA that the adaptation of Column 2 of Annex IX, Section 8.6.2. is not fulfilled. You further state that the criteria of Annex XI, Section 3.2 (a) are fulfilled, since the solely use of the Substance is as an additive for drilling fluids to reduce torque and drag and there is not significant exposure to workers and to the environment.

As stated in Annex XI, Section 3, you may adapt the information requirement, provided you fulfil all the identified criteria in paragraphs 3.2(a)(i) to (iii) and submit an adequate and scientifically-supported justification, based on a thorough and rigorous exposure assessment in accordance with Section 5 of Annex I, in particular:

(i) "the results of the exposure assessment covering all relevant exposures throughout the life cycle of the substance demonstrate the absence of or no significant exposure in all scenarios of the manufacture and all identified uses as referred to in Annex VI section 3.5.;

(ii) a suitable DNEL or a PNEC can be derived from results of available test data for the Substance taking full account of the increased uncertainty resulting from the omission of the information requirement, and that DNEL or PNEC is relevant and appropriate both to the information requirement to be omitted and for risk assessment purposes;

(iii) the comparison of the derived DNEL or PNEC with the results of the exposure assessment shows that exposures are always well below the derived DNEL or PNEC."

We have assessed this information and identified the following issues:

The first criterion 3.2(a)(ii) requires "*absence of or no significant exposure in all scenarios of the manufacture and all identified uses*". However, we note that in Section 9 of your Chemical Safety Report (CSR) you based your exposure assessment using one exposure scenario PROC 4, for which you estimate dermal systemic exposure [redacted] mg/kg bw and inhalation exposure [redacted] mg/m^3. Further, when comparing those with the general population DNEL, this leads to an RCR of [redacted] for chronic dermal exposure and RCR of [redacted] for combined systemic exposure. We note that those values cannot be considered as "*absence of significant human exposure*". Therefore, criterion 3.2 (a) (i) is not fulfilled.

The second criterion 3.2. (a) (ii) requires that "*a DNEL can be derived from results of available test data*" and that "*DNEL is relevant and appropriate*". However, the worker long-term systemic DNEL for dermal and inhalation exposure, which you derived in your CSR, is based on Screening for reproductive/developmental toxicity study (OECD TG 422) with the

Substance. We underline that such DNEL is not relevant nor appropriate both for the information requirement to be omitted and for risk assessment purposes. The exposure duration of the screening test is shorter (you reported 43 days) than 90-days and the results have a lower statistical power because the number of animals examined per dose group is significantly lower than required by the OECD TG 408. For these reasons, a DNEL derived from screening for reproductive/developmental toxicity study is not appropriate to omit a 90-day toxicity study. Therefore, the criterion 3.2 (a) (ii) is not fulfilled.

Since the DNEL is considered not appropriate, it follows that the third criterion 3.2(a)(iii), exposure results are to be well below the derived DNEL, cannot be fulfilled.

The information you provided in the dossier does not meet the general rules for adaptation of Annex XI, Section 3.2(a) and your adaptation is rejected.

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

Information on the design of the study to be performed (species/route)

Following the criteria provided in Annex IX, Section 8.6.2, Column 2, the oral route is the most appropriate route of administration to investigate repeated dose toxicity[2]. The Substance is a powder, with particle size > 100 microns, which does not raise concern for inhalation. Therefore, the sub-chronic toxicity study must be performed according to the OECD TG 408, in rats and with oral administration of the Substance.

2. Pre-natal developmental toxicity study (Annex IX, Section 8.7.2.) in a first species;

A Pre-natal developmental toxicity (PNDT) study (OECD TG 414) in one species is a standard information requirement under Annex IX to REACH.

You have provided:

1. Screening for reproductive / developmental toxicity study in rats (key study, equivalent to OECD TG 422, GLP compliant, study report 2007).

2. Statement that "*The Pre-natal Developmental Toxicity Study (OECD 414), required in Section 8.7.2 of Annex X, does not need to be conducted because the following reasons apply: • Asphalt, sulphonated, sodium salt use is restricted to a single industrial application, therefore there are no consumer or professional uses; • No adverse effects on reproductive and developmental parameters were detected in a Combined Repeated Dose Toxicity Study with the Reproduction / Developmental Toxicity Screening Test up to the highest dose tested (1000 mg/kg bw/day). The study was conducted according to an appropriate OECD test guideline and in compliance with GLP. • Tonnage has not exceeded 100 tonnes per year since 2010 and the registration will be downgraded to* [redacted]*, therefore proposal of Pre-natal Developmental Toxicity Study (OECD 414) is not appropriate*".

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

[2] ECHA Guidance R.7a, Section R.7.6.2.3.2.

1) In order to be considered compliant and enable assessing if the Substance is a developmental toxicant, the information provided has to meet the requirements of a pre-natal developmental toxicity study (OECD TG 414) in a first species.

You did not provide information equivalent to OECD TG 414 because the OECD TG 422 study does not cover key parameters of a pre-natal developmental study, like examinations of foetuses for skeletal and visceral alterations.

2) To adapt standard information requirement for pre-natal developmental toxicity, the conditions of specific adaptations based on Annex IX, Section 8.7.2, or General adaptations, set in Annex XI have to be fulfilled.

You have not used any of the above adaptation options and arguments you provided in your dossier (as cited above) do not fulfil the conditions set therein. Hence, your adaptation is rejected.

In your comments to the draft decision you agreed with ECHA that the adaptation of Column 2 of Annex IX, Section 8.7.2. is not fulfilled. You further state that the criteria of Annex XI, Section 3.2 (a) are fulfilled, since the solely use of the Substance is as an additive for drilling fluids to reduce torque and drag and there is not significant exposure to workers and to the environment.

As stated in Annex XI, Section 3, you may adapt the information requirement, provided you fulfil all the identified criteria in paragraphs 3.2(a)(i) to (iii) and submit an adequate and scientifically-supported justification, based on a thorough and rigorous exposure assessment in accordance with Section 5 of Annex I, in particular:

(iv) "the results of the exposure assessment covering all relevant exposures throughout the life cycle of the substance demonstrate the absence of or no significant exposure in all scenarios of the manufacture and all identified uses as referred to in Annex VI section 3.5.;
(v) a suitable DNEL or a PNEC can be derived from results of available test data for the Substance taking full account of the increased uncertainty resulting from the omission of the information requirement, and that DNEL or PNEC is relevant and appropriate both to the information requirement to be omitted and for risk assessment purposes;
(vi) the comparison of the derived DNEL or PNEC with the results of the exposure assessment shows that exposures are always well below the derived DNEL or PNEC."

We have assessed the information and identified the following issues:

The first criterion 3.2(a)(ii) requires "*absence of or no significant exposure in all scenarios of the manufacture and all identified uses*". However, we note that in Section 9 of your Chemical Safety Report (CSR) you based your exposure assessment using one exposure scenario PROC 4, for which you estimate dermal systemic exposure [redacted] mg/kg bw and inhalation exposure [redacted] mg/m^3. Further, when comparing those with the general population DNEL, this leads to an RCR of [redacted] for chronic dermal exposure and RCR of [redacted] for combined systemic exposure. We note that those values cannot be considered as "*absence of significant human exposure*". Therefore, criterion 3.2 (a) (i) is not fulfilled.

The second criterion 3.2. (a) (ii) requires that "*a DNEL can be derived from results of available test data*" and that "*DNEL is relevant and appropriate*". However, the worker long-term systemic DNEL for dermal and inhalation exposure, which you derived in your CSR, is based on Screening for reproductive/developmental toxicity study (OECD TG 422) with the

Substance. We underline that such DNEL is not relevant nor appropriate both for the information requirement to be omitted and for risk assessment purposes. According to the footnote in Annex XI, section 3 "*a DNEL derived from a screening test shall not be considered appropriate to omit prenatal developmental toxicity study toxicity study*". Therefore, criterion 3.2 (a) (ii) is not fulfilled.

Since the DNEL is considered not appropriate, it follows that the third criterion 3.2(a)(iii), exposure results are to be well below the derived DNEL, cannot be fulfilled.

The information you provided in the dossier does not meet the general rules for adaptation of Annex XI, Section 3.2(a) and your adaptation is rejected.

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

Information on study design

A PNDT study according to the test method OECD TG 414 should be performed in rat or rabbit as preferred species with oral[3] administration of the Substance.

[3] ECHA Guidance R.7a, Section R.7.6.2.3.2.

Appendix B: Procedural history

For the purpose of the decision-making, this decision does not take into account any updates of registration dossiers after the date on which you were notified the draft decision according to Article 50(1) of the REACH Regulation.

The compliance check was initiated on 09/05/2018.

ECHA notified you of the draft decision and invited you to provide comments within 30 days of the notification.

ECHA took into account your comments and did not amend the request(s).

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 C: Observations and technical guidance

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

2. Failure to comply with the requests in this decision, or to otherwise fulfil the information requirements with a valid and documented adaptation, will result in a notification to the enforcement authorities of the Member States.

3. Test guidelines, GLP requirements and reporting

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

   According to 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.

   According to 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: 'How to report robust study summaries[4]'.

4. Test material

   While selecting the test material you must take into account 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. Any constituents that have harmonised classification and labelling according to the CLP Regulation (Regulation (EC) No 1272/2008) must be identified and quantified using the appropriate analytical methods.

   The OECD Series on Principles of Good Laboratory Practice and Compliance Monitoring, Number 11 [ENV/MC/CHEM(98)16] requires a careful identification of the test material and description of its characteristics. In addition, the Test Methods Regulation (EU) 440/2008, as amended by Regulation (EU) 2016/266, requires that "*if the test method is used for the testing of a* [...] *UVCB* [...] *sufficient information on its composition should be made available, as far as possible, e.g. by the chemical identity of its constituents, their quantitative occurrence, and relevant properties of the constituents*".

   In order to meet this requirement, all the constituents of the test material used for each test must be identified as far as possible.

   Technical Reporting of the test material for UVCB substances

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

The composition of the selected test material must be reported in the respective endpoint study record, under the Test material section. The composition must include all constituents of the test material and their concentration values. Without such detailed reporting, ECHA may not be able to confirm that the test material is relevant for the Substance and to all the registrants of the Substance.

Technical instructions are available in the manual "How to prepare registration and PPORD dossiers" on the ECHA website (https://echa.europa.eu/manuals).

5. List of references of the ECHA Guidance documents[5]

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 in this decision.

Read-Across Assessment Framework (RAAF, 2017)
Supporting information on the ECHA website (https://echa.europa.eu/support/registration/how-to-avoid-unnecessary-testing-on-animals/grouping-of-substances-and-read-across)

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.

[5] https://echa.europa.eu/guidance-documents/guidance-on-information-requirements-and-chemical-safety-assessment

Appendix G: List of the registrants to which the decision is addressed and the corresponding information requirements applicable to them

Registrant Name	Registration number	(Highest) Data requirements to be fufilled
[redacted]	[redacted]	[redacted]