EUROPEAN CHEMICALS AGENCY

Decision number: CCH-D-2114292251-54-01/F Helsinki, 20 March 2015

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

For sulphuric acid, compound with graphite, CAS RN 12777-87-6 (EC No 235-819-4), 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 sulphuric acid, compound with graphite, CAS RN 12777-87-6 (EC No 235-819-4), submitted by [redacted] (Registrant).

This decision is based on the registration as submitted with submission number [redacted], for the tonnage band of 100 to 1000 tonnes 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 4 November 2013.

On 17 December 2013 ECHA sent the draft decision to the Registrant and invited them to provide comments in accordance with Article 50(1) of the REACH Regulation on the draft decision. That draft decision was based on submission number [redacted].

After interaction, the Registrant was provided an additional deadline to submit an update of their registration dossier by 31 March 2014.

On 31 January 2014 ECHA received comments from the Registrant on the draft decision.

On 31 March 2014 the Registrant updated their registration dossier, with the submission number [redacted].

The ECHA Secretariat considered the Registrant's comments and update. The information is reflected in the Statement of Reasons (Section III) whereas no amendment to the Information Required (Section II) was made.

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 them 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 did not amend 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. The Member State Committee took the comments of the Registrant on the proposals for amendment into account.

A unanimous agreement of the Member State Committee on the draft decision was reached on 24 November 2014 in a written procedure launched on 13 November 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), 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. Name, molecular and structural formula or other identifier of the substance (Annex VI, 2.1. and 2.2.): information which is suitable and necessary to allow ECHA to establish and verify the name and the identity of the registered substance, as specified under section III.A.1 below;
2. Composition of the substance (Annex VI, 2.3.): Information which is suitable and necessary to allow ECHA to establish and verify the composition and the identity of the registered substance, as specified under section III.A.2 below;
3. Description of the analytical methods (Annex VI, 2.3.7.), as specified under section III.A.3 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)(d), 13 and Annexes VIII and IX 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. *In vitro* gene mutation study in mammalian cells (Annex VIII, 8.4.3.; test method: EU B.17./OECD 476);
2. Sub-chronic toxicity study (90-day), oral route (Annex IX, 8.6.2.; test method: EU B.26./OECD 408) in rats.

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.

Pursuant to Article 41(4) of the REACH Regulation the Registrant shall submit the information, including, where relevant, an update of the Chemical Safety Report, in the form of an updated registration to ECHA by **2 January 2017**.

III. Statement of reasons

Based on the examination of the technical dossier, ECHA concludes that the information therein does not comply with the requirements of Article 10 of the REACH Regulation and Annex VI thereof. Consequently, the Registrant is requested to submit the information mentioned above that is 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. Name, molecular and structural formula or other identifiers of the substance

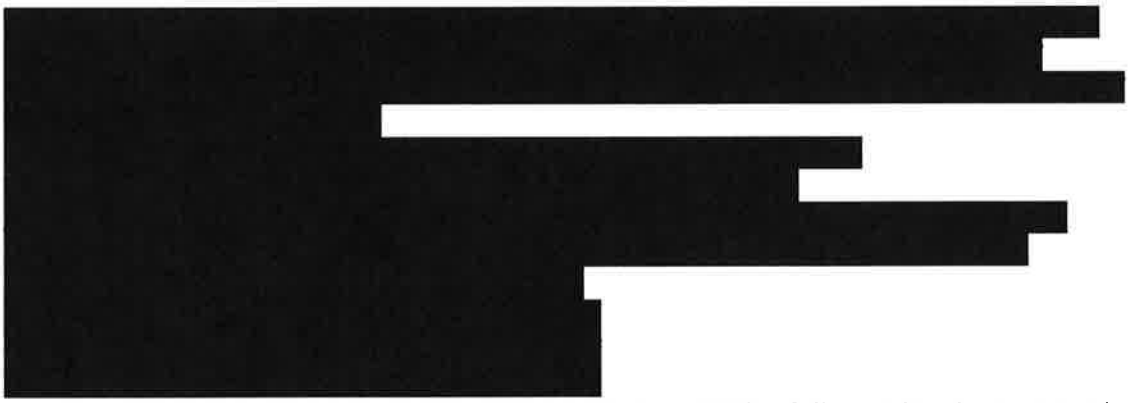
ECHA notes that the Registrant has not provided sufficient information to identify the substance, as required by Annex VI, Section 2.1. and 2.2. of the REACH Regulation. Based on the information included in Section 1.1 of the technical dossier, it is not possible to unambiguously establish the identity of the substance registered.

More specifically, the Registrant identified the registered substance as a well-defined mono-constituent substance. According to the Guidance for identification and naming of substances under REACH (Version 1.2, March 2012)[1], thereinafter referred to as "the Guidance", a mono-constituent substance is identified by the chemical name and other identifiers (including the molecular and structural formula) of the main constituent and the chemical identity of the impurities and/or additives, and their typical concentration(s) and concentration range(s), which is proven by the spectral and analytical information. Normally, for a mono-constituent substance the main constituent is present with concentrations at or above 80% (w/w) and should be specified completely by all the parameters.

However, in Section 1.1 of the registration dossier only generic EC (235-819-4) and CAS (12777-87-6) entries have been used to identify the substance. These entries refer to the

[1] The Guidance for identification and naming of substances under REACH (Version: 1.2, March 2012) is published on the ECHA website at: http://echa.europa.eu/documents/10162/13643/substance_id_en.pdf

generic chemical name "sulphuric acid, compound with graphite", which does not relate to a specific substance but covers an unspecific compound of graphite (including all phases) with sulfuric acid. The molecular formula reported "*Cm+ HSO4-* n H2SO4*" is not specific. The structural formula and text included in the remarks field of Section 1.1, namely



For the calculation of the molecular weight range only the fully oxidized compound (stage 1) has been taken into account."

are more specific than the EC and CAS entries but they also do not meet the requirements to describe a well-defined mono-constituent substance, as the extent of reaction between graphite and sulfuric acid is not defined and no one main constituent identified.

Furthermore, the following description of the registered substance provided in Section 3.1 of the registration dossier indicates that the registration refers to the reaction products of graphite and sulfuric acid in the presence of an oxidising agent: "*The graphite flakes are treated with highly concentrated sulphuric acid in the presence of an oxidizing agent under controlled temperature conditions. After completion of the reaction, to remove the non-reacted sulphuric acid, the resulting graphite salt is filtered off, subsequently washed thoroughly with water, and finally dried. During this treatment remnants of the oxidizing agent are removed and are therefore not part of the final substance.*"

However, this description does not report the extent of reaction between acid and graphite nor define the product(s) that are the outcome of the reaction. The description of the substance and the process is thus not consistent with the identification of the substance as a well-defined substance.

Based on the name and other identifiers and the description of the manufacturing process given in Section 3.1, the substance shall be rather identified as a UVCB substance (a substance of Unknown or Variable composition, Complex reaction products or Biological materials). Consequently, further information is required to appropriately identify the registered substance, in line with Annex VI, Section 2.1. of the REACH Regulation. More specifically, the naming of a UVCB substance consists of two parts: the chemical name and the more detailed description of the manufacturing process.

ECHA notes that the Registrant provided additional justification in the comments provided to the draft decision and the updated dossier as to why they consider the substance as a well-defined mono-constituent substance. In the updated dossier, the Registrant included a detailed justification in a report [redacted] attached to section 1.4 of the dossier. Specifically, the Registrant contends that: "[redacted] *is an inorganic substance with different acid equivalent / carbon ratios depending on the manufacturing process and its main constituent is (graphite salt) present at a concentration of at least* [redacted] *% in all products available on the market."*

ECHA notes that the substance cannot be regarded as well-defined as its stoichiometry is variable and as a consequence, does not have a unique molecular formula. ECHA reminds the Registrant that Section 4.1 of the Guidance states the following: "*well-defined substances: Substances with a defined qualitative and quantitative composition*". A well-defined substance therefore has constituents of specific stoichiometry and its identity is solely defined by these specific constituents.

The Registrant additionally states in the same report that sulphuric acid, compound with graphite ([redacted] graphite) follows the EINECS entry identity 235-819-4. However, based on the EINECS reporting rules, substances that cannot be represented by a complete chemical structure diagram and specific molecular formulae are regarded as substance of unknown or variable composition, complex reaction products or biological materials (UVCB). Consequently the EINECS entry "graphite compound with sulfuric acid" refers to a UVCB substance.

The Registrant also included additional information on the nature of the intercalation products formed when graphite reacts with sulfuric acid. The Registrant states that [redacted] graphite can be considered as one compound with a concentration range of intercalated bisulfate. ECHA notes that this description would not refer to a well-defined salt but rather a complex substance where there is variability in stoichiometry depending on the reaction conditions.

The Registrant also included IUPAC nomenclature for graphite intercalation compounds as attachment with the comments sent on 31 March 2014. ECHA notes that the IUPAC nomenclature is specific in terms of intercalation species, the order of stacking, the identity of the guest, and whether it is a donor or acceptor compound. The name "graphite compound with sulfuric acid" is therefore not consistent with the IUPAC nomenclature for graphite intercalation compounds. The Registrant also included two scientific papers on the properties of graphite bisulfate. ECHA notes that the description of graphite bisulfate in these papers demonstrate that the compound is complex and is not a simple salt.

In accordance with Article 41(1) and (3) of the REACH Regulation, the Registrant is required to identify the registered substance as a UVCB substance by reference to a name that allows for an accurate and complete identification of the substance and to provide a detailed description of the manufacturing process, including the chemical identity of the source and information on the most relevant steps of the manufacturing process. In accordance with the Guidance, the name of a UVCB substance shall adequately reflect the source materials and the manufacturing process. The information shall be consistent with the molecular and structural formula and other identifiers reported in the dossier.

The description of the manufacturing process shall be sufficiently detailed to allow ECHA to understand which starting materials are used, and how any other steps and process parameters may affect the substance composition and therefore its identity. It shall include, as appropriate:

- Identity and ratio of starting materials/reactants, including also all auxiliary agents (e.g. source and type(s) of graphite, identity of the oxidising agent, concentration and grade(s) of sulfuric acid);
- Typical reaction parameters such as temperature, pressure and time;
- Nature of the intercalation compound formed (e.g. graphite bisulfate) and its carbon to sulfate ratio;
- Any post-treatment carried out; and
- A description of any other relevant operating parameters or process.

Regarding how to report the requested information in IUCLID the following applies:

- The revised chemical name shall be included in the IUPAC name field in Section 1.1 of the IUCLID dossier;
- The description of the manufacturing process of the UVCB substance shall be included in the description field in Section 1.1 of the IUCLID dossier;
- Where different grades are manufactured, details of the key process parameters for each grade shall be reported in the description field.

Further information on how to report the description of the manufacturing process is available in "Data Submission Manual Part 18 - How to report the substance identity in IUCLID 5 for registration under REACH".

2. Composition of the substance

The substance composition corresponds to the chemical representation of what the substance consists of and is therefore an essential part of substance identification and the cornerstone of all the REACH obligations. ECHA notes that the Registrant has not included sufficient information on the composition of the substance to enable the identity of the registered substance to be verified, as required under Annex VI, Section 2.3. of the REACH Regulation.

Specifically, the Registrant has reported two compositions in Section 1.2 of the IUCLID dossier. These compositions include their main constituent with the same generic reference substance reported in Section 1.1 as "sulphuric acid, compound with graphite". From this limited information and due to the inconsistencies in the identifiers, described in Section III.A.1 of this decision, the composition of the substance manufactured/ imported cannot be established.

In accordance with section 4.2 of the Guidance, the composition shall normally be described up to 100% and each constituent requires a complete chemical specification, including its structural information. For UVCB substances, section 4.3 of the Guidance recognizes that they either cannot be fully specified with the IUPAC name of the constituents, as not all the constituents can be identified, or that they may be described with a lower specificity due to variability of the exact composition. However, also for UVCB substances the chemical composition and the identity of the constituents should still be reported as far as known.

In the updated dossier submitted by the Registrant in response to the draft decision, the Registrant did not update the composition reported in section 1.2 of the dossier. ECHA notes that the Registrant indicated in the document "[redacted]" attached to section 1.4, that the composition is not unknown, and that only the main constituent exists in different technical stages (amount of intercalated sulfate and order of intercalation) in different products on the market, that the amount of intercalated is not variable but is correlated with the production parameters and post-treatment and can be determined by analytical methods. ECHA notes that the composition reported in section 1.2 of the dossier does not include any information on the intercalation compounds or their variability depending on production parameters.

The Registrant is, therefore, required to further specify the identity of each specific constituent reported in Section 1.2. The name and other identifiers for each constituent shall describe the specific graphite intercalation compound and its carbon to sulfate ratio, as relevant. This information shall be sufficient to enable the specific constituents of the substance registered by this legal entity to be identified and shall be consistent with the information included in Section 1.1 on the "name and other identifiers" for the substance.

Further technical details on how to report details on the constituents of a substance in IUCLID are available in the "Data Submission Manual – Part 18: How to report the substance identity in IUCLID 5 for registration under REACH."

If the Registrant covers different grades of the same substance in a registration, the Registrant shall report separately the compositional information of each grade. This means that if the substance covered by the registration has two (or more) different compositions, then these compositions must be presented separately. The corresponding analytical data to enable the identity and composition of each grade listed in Section 1.2 to be verified shall be included in Section 1.4.

Information on how to report several compositions in IUCLID is specified in paragraph 2.3, Q&A (8), of the "Data Submission Manual – Part 18: How to report the substance identity in IUCLID 5 for registration under REACH."

ECHA highlights that failure to report separately the compositional information of each grade of a substance may result in one or more grades not being covered by this registration.

The Registrant should also note that multiple compositions may indicate multiple substances and consequently the possible requirement for multiple registrations per each individual substance.

3. Description of the analytical methods

ECHA notes that the Registrant has not provided sufficient information on the methods used to determine the identity and composition of the substance registered by his legal entity, as required by Annex VI, 2.3.7. of the REACH Regulation.

In the updated dossier, the Registrant has included certificates of analysis for eight representative samples where the sulfur content, the volatile content, the impurity content (elements, ash, moisture) are reported. However a description of the method used to determine the sulfur content was not provided.

In the updated dossier the Registrant did not provide either the requested qualitative data on specific constituents in terms of intercalation compounds (i.e. intercalation compound content, graphite content etc.). In the absence of this information, the identity and composition of the substance cannot be verified. A description of the methods used to identify and quantify the impurities reported in Section 1.2 of the dossier ([redacted], other impurities) was not included. In addition, the "volatiles", reported in the analytical report in Section 1.4, are present in a significant concentration ([redacted] %) but they are not further identified and they are not included in the composition in Section 1.2 of the dossier. This information is not sufficient to determine the composition of the substance constituents.

In line with Annex VI, 2.3.7., the Registrant shall include information on the methods used to quantify all substance constituents in terms of graphite to sulfate ratio and phase (e.g. [redacted]) where relevant. This information shall be sufficient to enable the substance identified in Section 1.1 of the dossier and all constituents reported in Section 1.2 to be verified. Where grades of the substance are registered, sufficient data that will enable the identity and composition of each grade to be verified shall be included. The Registrant may use any method or combination of methods to do this (e.g. elemental analysis, gravimetry, quantitative XRD, etc. The Registrant shall note

that a description of each method used shall be included in such detail that the method may be reproduced.

ECHA received Proposals for Amendment (PfAs) from a Competent Authority on 06 October 2014, related to the three above-mentioned requests for information on substance identity, proposing a termination of the current compliance check process and initiation of a new compliance check targeting the substance identity requests given in Section II.A. The PfAs considered that the requests in Section II.B could not be undertaken prior to clarification of the identity of the substance subject to the present decision.

In response to the PfAs, the Registrant agreed that "*the substance identity of the registered substance needs to be clarified first (ideally in direct communication between ECHA and the registrants' technical experts) before further information [is] requested from the registrant*". The Registrant also claimed that the clarification of the "*current approach will result in considerable efforts for discussion between the Registrants.*"

Taking the PfAs and the respective comment of the Registrant into account, ECHA confirms that, while there is uncertainty on the scope of the registered substance in terms of the specific graphitic salt covered (grades of the substances in terms of specific stoichiometric ratios or ranges), there is sufficient information in Section 3.1 of the registration dossier to infer that the substance refers to a graphitic sulphate manufactured by a specific process. From the information provided by the Registrant "[redacted] *is an inorganic substance with different acid equivalent / carbon ratios depending on the manufacturing process and its main constituent is (graphite salt) present at a concentration of at least* [redacted]% *in all products available on the market*", it is possible to determine the intention of the Registrant in terms of the substance registered and to conclude that the substance may have many possible grades. Therefore this does not prevent ECHA from requesting the necessary experimental testing.

Finally, to allow for clarification of the substance identity prior to conducting the tests required under Section II.B., ECHA has extended the deadline of the decision from 18 to 21 months to submit all requested information.

B. Information in the technical dossier related to the manufacture and use(s) of the substance

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.

Pursuant to Articles 10(a)(vi) and/or (vii), 12(1)(d) of the REACH Regulation, a technical dossier for a substance manufactured or imported by the Registrant in quantities of 100 to 1000 tonnes per year shall contain as a minimum the information specified in Annexes VII to IX of the REACH Regulation.

1. *In vitro* gene mutation study in mammalian cells

An "in vitro *gene mutation study in mammalian cells*" is an information requirement as laid down in Annex VIII, Section 8.4.3. of the REACH Regulation, "if a negative result in Annex VII, Section 8.4.1. and Annex VIII, Section 8.4.2." is obtained. ECHA notes that the registration dossier contains negative results for the latter two information requirements. Therefore, adequate information on this endpoint needs to be present in the technical dossier for the registered substance to meet this information requirement.

The Registrant has not provided any study record of an *in vitro* gene mutation study in mammalian cells in the dossier that would meet the information requirement of Annex VIII, Section 8.4.3.

The Registrant has sought to adapt this information requirement. The justification of the adaptation given by the Registrant is that "*conducting of the* in vitro *gene mutation test in mammalian cells with sulphuric acid, compound with graphite is considered as unnecessary.*" Furthermore, the Registrant indicates that "*sulphuric acid, compound with graphite is an inorganic solid, consisting of natural graphite and variable amounts of intercalated sulphuric acid. Graphite is a naturally occurring solid substance, which is insoluble in water. It is not systemically available, does not cross biological membranes and is not bioavailable.*" In addition, the Registrant assumes that "*the intercalated sulphuric acid is partially leached out by water and dissociated into the biologically ubiquitous sulphate and hydronium ions, and that due to their ionic character they do not cross biological membranes. Sulphate is a normal biological constituent of the body, a normal biological metabolite and a degradation product of sulphur-containing amino-acids. Due to its physico-chemical characteristics the registered substance is neither systemically available nor bioavailable and can therefore not reach critical organs and tissues. The substance does not enter the blood stream and can therefore not permeate cells to the sub-cellular level.*"

ECHA notes that the Registrant has not explicitly claimed a specific adaptation possibility. ECHA concludes that the justification provided does not meet the specific rules for adaptation of Annex VIII, 8.4.3., column 2 because no *in vivo* gene mutation test is available.

In addition, ECHA evaluated the suggested adaptation as weight of evidence approach according to Annex XI, Section 1.2.

In the updated dossier (submission number [redacted]), the Registrant has provided a structured weight of evidence approach, relying on the insoluble properties of the registered substance and on the content of the leachate which is expected to be tested as a result of metabolisation.

In line with the OECD test guideline 476 the Registrant stated that: "*solid test substances should be dissolved or suspended in appropriate solvents or vehicles and diluted if appropriate prior to treatment of the cells. In case of water or polar extracts (i.e. aqueous NaCl-solution) as vehicle the resulting aqueous solution will include the same metals and ions as already detected during the performance of the water solubility test.*" The Registrant subsequently reviewed the genotoxicity potential of each component of the leachate analysed in section 4.8 of their IUCLID dossier, namely sodium sulphate, Al^{3+}-ions, Si-ions, iron oxides (such as Fe_2O_3), and the insoluble component, namely graphite.

ECHA considers that the Registrant has addressed satisfactorily with several independent sources of information how they come to a conclusion on the intrinsic properties corresponding to the information requirement for gene mutation. In addition, ECHA notes that the presented documentation does explain why or how the individual elements interact, to give a sufficient weight of evidence for the particular intrinsic property related to this endpoint. On this basis ECHA considers the weight of evidence approach for this endpoint, according to Annex XI, 1.2., to be plausible.

A weight of evidence approach requires always an "*adequate and reliable documentation*" (Annex XI, 1.2., last sentence of the REACH Regulation). However, the Registrant has only provided a robust study summary (RSS) for the OECD TG 476 studies for the components silicon dioxide and $AlCl_3$. Although the Registrant makes reference to other Registrants'

data, the Registrant failed to provide, in the updated dossier, adequate RSSs of each of the cited studies for sodium sulphate, synthetic graphite, $AlCl_3$ and $Al(OH)_3$, or Fe_3O_4. Hence ECHA considers that due to a lack of documentation of the weight of evidence approach the necessary information to meet the endpoint is not present in the Registrant's dossier, and cannot be assessed.

ECHA concludes that the justification provided does not meet the specific rules for adaptation of Annex XI, Section 1.2. and cannot be accepted.

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: *in vitro* mammalian cell gene mutation test (test method: EU B.17./OECD 476).

2. Sub-chronic toxicity study (90-day), oral route

A "*sub-chronic toxicity study (90 day)*" is a standard information requirement as laid down in Annex IX, Section 8.6.2. 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 has not provided any study record of a sub-chronic toxicity study (90-day) in the dossier with the registered substance that would meet the information requirement of Annex IX, Section 8.6.2.

The Registrant has sought to adapt this information requirement. The justification of the adaptation given by the Registrant is that, in addition to the arguments reported above (under III.1, i.e. the registered substance is not systemically and biologically available, does not cross biological membranes, can be considered as an inert substance, is dissociated into the biologically ubiquitous sulphate and hydronium ions, is unlikely to be taken up into the blood stream), there is a lack of uptake and absence of systematic availability of the registered substance.

The Registrant argues also that the registered substance "*exhibited no acute oral toxic characteristics and no acute dermal toxic characteristics when applied to rats at a concentration of 2,000 mg/kg body weight. In addition, all toxicological studies available for sulphuric acid, compound with graphite showed no toxic characteristics of the substance indicating lack of uptake and absence of systemically availability.*" The Registrant further explains that "*risk reduction measures are in place limiting the exposure of workers. Personal protective clothing and protective glasses as well as mechanical ventilation to reduce airborne concentrations are recommended to minimize exposure. Exposure of workers during production, handling and use of sulphuric acid, compound with graphite in an amount of toxicological relevance is considered to be highly unlikely under normal or reasonably foreseeable conditions of use.*" As a consequence the Registrant considers it "*unnecessary to perform laboratory studies on oral repeated dose toxicity with sulphuric acid, compound with graphite.*"

ECHA notes that the Registrant has not explicitly claimed a specific adaptation possibility. However, Annex IX, 8.6.2. provides a column2 adaptation possibility to omit the sub-chronic toxicity study (90-day) if "*the substance is unreactive, insoluble and not inhalable and there*

is no evidence of absorption and no evidence of toxicity in a 28-day 'limit test', particularly if such a pattern is coupled with limited human exposure" According to the technical dossier, components of the substance are soluble ([redacted]% of the components are iron, aluminium, silicium and sulphur), and the substance is inhalable (particle size less than 100 microns, but more than 10 microns), and so ECHA concludes that the justification provided does not meet this specific rules for adaptation of Annex IX, 8.6.2., column 2, nor other of the column 2 adaptations.

In the updated dossier, the Registrant has provided a structured weight of evidence approach, based in part upon the intrinsic properties of the constituents of the registered substance. ECHA evaluated the proposed adaption using a weight of evidence approach according to Annex XI, Section 1.2.

However, ECHA considers that (i) the failure to provide a robust study summary (RSS) for one constituent, synthetic graphite, and (ii) the inclusion of RSSs for sodium sulphate and SiO2 which are given a reliability score of 3 and 4, respectively (and there are no other studies covering these substances), constitute a failure of adequate and reliable documentation for these constituents to justify any weight of evidence as required by Annex XI, 1.2. Furthermore the Registrant did not clarify how the information and the related identified shortcomings can be used in the subsequent hazard assessment.

Additionally, ECHA considers that there is not sufficient weight of evidence from several independent sources of information leading to the assumption/conclusion that a substance has or has not a particular dangerous property, in this case for a sub-chronic toxicity study, as required for Annex XI, 1.2. ECHA particularly notes that some of the studies relied upon are of either poor quality, of insufficient duration, do not cover key parameters (e.g. the Registrant does not discuss the different durations of studies proposed and how they can be relevant to address the current information requirements) or have multiple of these deficiencies.

Consequently, ECHA concludes that the justification provided does not meet the specific rules for adaptation of Annex XI, Section 1.2.

Therefore, the adaptation of the information requirement suggested by the Registrant cannot be accepted.

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.

Since the substance is a solid with particle size ranging from 50 to 1000 µm and therefore not respirable, ECHA considers that testing by the oral route is most appropriate.

In the updated dossier, the Registrant discussed the route of exposure proposed by ECHA, claiming that (i) the inhalation route would be most relevant. However they subsequently indicate that an acute inhalation toxicity study could not be performed as the Registrant was not able to generate the requested "*stable atmosphere*" (due to the hygroscopic property of the substance), and that (ii) dermal testing would not be appropriate. ECHA considers that its original considerations on route of exposure reflect this information.

According to the test method EU B.26/OECD 408 the rat is the preferred species. ECHA considers this species as being appropriate and testing should be performed with the rat.

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: Repeated dose 90-day oral toxicity study (test method: EU B.26./OECD 408) in rats.

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]
Leena Ylä-Mononen
Director of Evaluation