EUROPEAN CHEMICALS AGENCY

Decision number: CCH-D-0000005497-64-01/F Helsinki, 28 August 2014

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

For phenol, 2,4-dinitro-, sulfurized, leuco derivatives, CAS No 66241-11-0 (EC No 266-273-5), registration number: [redacted]

Addressee: [redacted]

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

I. Procedure

Pursuant to Article 41(1) of the REACH Regulation ECHA has performed a compliance check of the registration for phenol, 2,4-dinitro-, sulfurized, leuco derivatives, CAS No 66241-11-0 (EC No 266-273-5, submitted by [redacted] (Registrant). The scope of this compliance check is limited to the information requirements of Annex VI, Section 2 of the REACH Regulation. ECHA stresses that it has not checked the information provided by the other joint registrants for compliance with requirements regarding the identification of the substance (Annex VI, Section 2).

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 12 June 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 14 May 2013.

On 30 September 2013 ECHA sent the draft decision to the Registrant ([redacted]) and invited him to provide comments within 30 days of the receipt of the draft decision. That draft decision was based on submission number [redacted].

On 11 October 2013 the Registrant updated his registration dossier due to a change of legal entity. The registration number for the substance subject to the present decision has been transferred to [redacted] (submission number [redacted]). In this dossier update the substance identity information did not change compared to the previous submission.

On 29 October 2013 ECHA received comments from the Registrant.

The ECHA Secretariat considered the Registrant's comments and update regarding the legal entity change. The information is reflected in the Statement of Reasons (Section III) whereas no amendments to the Information Required (Section II) were made.

On 12 June 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.

As no proposal for amendment was submitted, ECHA took the decision pursuant to Article 51(3) of the REACH Regulation.

II. Information required

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

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

1. Name or other identifier of the substance (Annex VI, Section 2.1), as specified under section III.1 below;
2. Composition (Annex VI, Section 2.3), as specified under section III.2 below;
3. Spectral data (Annex VI, Section 2.3.5), as specified under section III.3 below;
4. Description of the analytical methods or the appropriate bibliographical references for the identification of the substance (Annex VI, Section 2.3.7), as described under section III.4 below.

Pursuant to Article 41(4) of the REACH Regulation the Registrant shall submit the information in the form of an updated registration to ECHA by **5 December 2014**.

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.

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 or other identifier of the substance (Annex VI, Section 2.1.)

ECHA notes that the Registrant identified the registered substance as of Unknown or Variable composition, Complex reaction products or Biological materials (UVCB). Information required to be provided according to Annex VI, Section 2.1. of the REACH Regulation on the naming of UVCB substances such as the registered substance shall consist of two parts: (1) the chemical name and (2) a more detailed description of the manufacturing process, as indicated in chapter 4.3 of the Guidance for identification and naming of substances under REACH and CLP (Version: 1.2, March 2012) - referred to as "the Guidance" hereinafter. ECHA observes that the Registrant did not include for the registered substance a chemical name in the "IUPAC name" field and a complete manufacturing process (as explained under points (i) and (ii) hereinafter).

(i) The Registrant did not provide in the "IUPAC name" field a chemical name for the registered substance. Therefore the Registrant shall include in the above specified

field a chemical name which shall be representative for the registered substance and consistent with the manufacturing process.

In his comments on the draft decision, the Registrant suggested the following chemical name for the registered substance: "*Reaction product of 2,4-dinitrophenol with polysulfide, leuco derivatives*".

ECHA has considered the information provided by the Registrant. ECHA notes that the theoretical structure of the registered substance also included in the Registrant's comment indicates that the registered substance refers to sodium salts. However, the chemical name proposed does not make any reference to the source for this sodium cation. ECHA also observes that the reactant used in the thionation step is referred to as "polysulfide", and therefore only as the anionic part of a salt, in the proposed name. In line with the naming conventions for UVCB substance where the source is chemical and the process is a synthesis, such as the registered substance, the name should refer to "reaction products" rather than "reaction product". ECHA therefore considers that the proposed chemical name does not precisely define or reflect the chemical nature, including the cationic part, of the registered substance.

The Registrant is accordingly requested to revise the proposed chemical name of the registered substance. The Registrant shall ensure that the chemical name which he assigns is representative of the specific substance which is the subject of this registration.

(ii) ECHA observes that the description of the manufacturing process is not sufficiently detailed for the identification of the registered UVCB substance. In particular, ECHA notes that the Registrant did not provide the ratio of starting materials used, solvent(s) used, a description of the chemical reactions involved in terms of representative chemical reaction scheme and the values for the relevant parameters applied (temperature, time and pressure).

In addition, the Registrant specified different reducing agents used in the manufacturing process (such as sodium sulfhydrate, sodium sulphide and sulfur) but it is unclear whether these reducing agents are all used in the same reaction or separately and whether the identity of the substance is affected when different reducing agents are used.

In line with the observations under point (ii), the Registrant shall in addition provide the missing information on the manufacturing process description. This information shall include:

1. The ratio of starting materials used: 2,4-dinitrochlorobenzene, inorganic alkalis and reducing agent;
2. Any solvent used at different stages of the manufacturing process;
3. A description of the chemical reactions involved in terms of representative chemical reaction scheme;
4. The values for the relevant parameters applied: temperature, time, pressure;
5. The identity of the inorganic alkalis and the concentration of the solution used to synthesise the 2,4-dinitrophenolate;
6. Clarification on whether the identity of the substance is affected when different reducing agents are used.

In his comments on the draft decision, the Registrant provided detailed information on the manufacturing process (including the name and ratio of the starting materials, the solvent used, the description of the chemical reactions involved and the values for the relevant parameters applied). ECHA considers this information is sufficient to fulfil the information

requirements specified in point (ii) above, if it is reported in the registration dossier However, as the Registrant has not yet updated his registration dossier with this information, ECHA maintains the abovementioned requirement on the manufacturing process description in its decision.

As for the reporting of the information in IUCLID, the chemical name and the manufacturing process description for the substance shall be specified in the "IUPAC name" field and the "Description" field in IUCLID section 1.1, respectively. The Registrant shall note that where the use of different reducing agents affects the identity of the substance, separate registration obligations may pertain.

The Registrant shall ensure that the correct identifiers are used throughout the registration whenever reference to the specific substance which is the subject of this registration is made.

2. Composition (Annex VI, Section 2.3.)

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 registration does not contain sufficient information for establishing the composition of the registered substance and therefore its identity, as required under Annex VI, section 2.3 of the REACH Regulation.

More specifically, ECHA notes that the registered substance was reported with a high content of water (typically [redacted]% content). Pursuant to Article 3(1) of the REACH Regulation, any solvent which may be separated without affecting the stability of the substance or changing its composition is not part of the substance.
In addition, the Registrant did not report any structural representation of the constituents generically described as "phenol, 2,4-dinitro-, sulfurized, leuco derivatives".

According to chapter 4.3 of the Guidance, the Registrant should note that, for UVCB substances consisting exclusively of unknown constituents, such as the registered substance, a generic description of their chemical nature shall be reported. The identification of these unknown constituents must be provided for ECHA to generically establish the composition of the substance as manufactured and to use the compositional information as one identifier for the registered substance. For each constituent or group of constituents, the typical, minimum and maximum concentration levels shall be specified.

In his comments to the draft decision, the Registrant clarified that the water present in the composition of the registered substance acts as a stabiliser for the registered substance since it prevents its oxidation with air. ECHA considers that the stabilising function of water for the registered substance is appropriate based on this justification. In this situation, ECHA would like to underline that only the quantity of water that is necessary to stabilise the substance shall be included in the mass balance, in line with the definition of substance in Article 3(1) of the REACH Regulation. However, based on the concentration values specified for water in the registration dossier, it remains unclear whether such a high concentration of water as up to [redacted] %(w/w) is necessary for that purpose.

In line with the further comments made by the Registrant on the draft decision, ECHA takes note of the theoretical structure provided for constituents of substance and the Registrant's statement that currently no chemical structure for "Leuco Sulphur Black 1" is accepted by the dye industry. ECHA acknowledges the complexity of the registered substance and considers that the theoretical considerations on the structure of the constituents are

elements to be taken into account when specifying as far as possible the composition of the substance. The Registrant furthermore explained in his comments that the laboratory who performed the analytics has been contacted for assistance and that, although they cannot yet respond in the commenting period to all requested information, they will do it by a registration update of the IUCLID dossier. ECHA therefore takes note of the commitment from the Registrant to provide as far as possible compositional information of the "phenol, 2,4-dinitro-, sulfurized, leuco derivatives" in the registered substance.

Accordingly, the Registrant is requested to report water as a stabiliser for the registered substance, in accordance with his own statement on its function. The Registrant shall ensure that the information provided on the composition includes the explanation on the stabilising function of water with regards to oxidation. The Registrant shall also specify the concentration values corresponding to the minimum quantity of water necessary to preserve the stability and exclude from the composition any excess water that is not necessary for the stability. The Registrant shall ensure that these concentration values are verifiable and therefore supported by the description of the analytical method used for the determination of the minimum quantity of water necessary for the stabilisation of the substance.
As for the description of the constituents currently covered by the generic entry "phenol, 2,4-dinitro-, sulfurized, leuco derivatives", taking into account that the Registrant has not updated the registration dossier with any new information, the Registrant is still requested to revise the information on these unknown constituents by providing as far as possible a generic description of their chemical nature.

The Registrant is accordingly requested to revise the reported composition by providing: further information on the generic constituents reported in the original dossier, excluding also water as far as it does not affect the stability of the substance or change its composition.

Regarding how to report the composition in IUCLID, the following applies: the Registrant should indicate the composition of the registered substance in IUCLID section 1.2. For each group of generic constituents, a generic chemical name describing the group of constituents, generic molecular and structural information (if applicable), as well as the minimum, maximum and typical concentration, shall be reported in the appropriate fields in IUCLID. The additive added to stabilise the substance shall be reported under the "Additives" header of the composition. The minimal lower, upper and typical concentration values of the stabiliser shall be reported in the appropriate fields in IUCLID. The function of water shall be selected from the dropdown list of the "Function" field in the repeatable block. The justification for the stabilising function of the quantity of water reported in the composition shall be reported in the Remarks field of that repeatable block.

Further technical details on how to report the composition of UVCB substances in IUCLID are available in paragraphs 2.1 and 2.2.2 of the Data Submission Manual – Part 18: How to report the substance identity in IUCLID 5 for registration under REACH (version: 2.0, July 2012) on the ECHA website.

3. Spectral data (Annex VI, Section 2.3.5.)

ECHA observes that the Registrant did not provide complete spectral data used for the identification of the registered substance, as requested according to Annex VI, Section 2.3.5.

More specifically ECHA notes that the Registrant provided infrared and mass information that present the following deficiencies:

(i) the infrared spectrum was recorded on a wet sample and therefore no useful

information can be retrieved above the wave number 3000 cm^{-1};
(ii) the mass spectrum was not attached in section 1.4 of the dossier.

Regarding the mass spectrum the Registrant stated that "*the test substance is a mixture of relative high molecular compounds with no definitive structure*" but any additional information is useful for a generic identification of certain constituents of the registered substance.

ECHA therefore concludes that the registration does not include sufficient spectral data required for the identification of the registered substance.

In his comments on the draft decision the Registrant underlined that the sample used for the infrared spectroscopic analysis was dried overnight at 40°C under vacuum and that the mass spectra only detected fragments on the range 100 – 500 da. The Registrant furthermore asked for confirmation on whether the spectra are still required despite the new information included in his comments on the manufacturing process and the structure of the substance.

ECHA has considered the new information provided. In line with the explanations provided above on the role of the spectra as fingerprint and for the identification of chemical functionalities, ECHA considers that the phenothiazine moiety alone presented in the structural information would indicate that the infrared spectrum normally is relevant for the identification of the potentially primary amino groups attached to the aromatic ring. In order to record a useful infrared spectrum the Registrant shall define the most appropriate recording conditions, *e.g.* using the transmittance mode instead of extinction mode.
As for the copy of the mass spectrum, taking into account that fragments were detected by this method, the mass spectrum constitutes *a priori* a fingerprint of the substance and shall as such be included in the dossier. Alternatively, a robust scientific justification demonstrating that mass spectroscopic technique cannot be used to derive any structural information or meaningful fingerprint must be provided.

ECHA notes that the technical dossier has not been updated with new information regarding the spectral data.

The Registrant is accordingly requested to provide a new infrared spectrum recorded on a sample that contains the minimum content of water and attach the mass spectrum. The Registrant shall note that both spectra are necessary as fingerprints used for the identification of different functional groups and generic constituents of the registered substance.

As for the reporting of the data in the registration dossier, the information should be attached in IUCLID section 1.4.

4. Description of the analytical methods or the appropriate bibliographical references for the identification of the substance (Annex VI, Section 2.3.7.)

ECHA observes that the Registrant did not provide sufficient description of the analytical methods used for the identification and quantification of the generic groups of constituents required to be reported in the composition of the registered substance, as requested according to Annex VI, Section 2.3.7.

More specifically ECHA notes that the Registrant reported [redacted]. For the quantification of anions the appropriate ISO methods were specified but the description of the analytical methods used to quantify the organic

constituents and Na^+ were not included (for sodium cation only the name of the method was given). The Registrant also reported the molecular weight range of the registered substance however the description of the analytical method used to derive this information is missing from the dossier.

ECHA therefore concludes that the registration does not include sufficient description of the analytical methods required for the identification of the registered substance.

The Registrant is accordingly requested to:

(i) provide a description of the analytical techniques used for the quantification of [redacted];
(ii) provide the chromatogram(s) for the identification of the above organic impurities alongside with the peak table(s) including retention times, area/area % and allocation of each peak;
(iii) provide a description of the analytical method(s) used for the identification and quantification of any other constituent and group of constituents required to be reported including the analytical method(s) used for determining the molecular weight range of the "phenol, 2,4-dinitro-, sulfurized, leuco derivatives" and any other method enabling to resolve structurally and quantitatively this group of constituents or any of its subgroups.

In his comments on the draft decision the Registrant committed to provide additional information on the analytical methods in a dossier update. ECHA notes that the technical dossier has not been updated with new information regarding this requirement. Therefore, the draft decision has not been amended for this information requirement.

As for the reporting of the data in the registration dossier, the information should be attached in IUCLID section 1.4.

The Registrant shall ensure that the composition reported in the dossier is consistent with the analytical results obtained.

IV. Information on right to appeal

An appeal may be brought against this decision to the Board of Appeal of ECHA under Article 51(8) of the REACH Regulation. Such an appeal shall be lodged within three months of receiving notification of this decision. Further information on the appeal procedure can be found on ECHA's internet page at http://echa.europa.eu/appeals/app_procedure_en.asp. The notice of appeal will be deemed to be filed only when the appeal fee has been paid.

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