ECHA
EUROPEAN CHEMICALS AGENCY

Decision number: CCH-D-0000002579-63-03/F Helsinki, 19 October 2012

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

For Phenol, dodecyl-, sulfurized, carbonates, calcium salts, overbased, CAS No 68784-26-9 (EC No 272-234-3), 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 concerning standard information requirements relating to substance identity (Annex VI, Section 2 of the REACH Regulation) of the registration dossier for Phenol, dodecyl-, sulfurized, carbonates, calcium salts, overbased, CAS No 68784-26-9 (EC No 272-234-3) submitted by [redacted] (Registrant).

This decision is based on the registration dossier 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 after 19 July 2012, 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 to initiate further compliance checks on the present dossier at a later stage.

The compliance check was initiated on 19 April 2012.

On 31 May 2012 ECHA sent the draft decision to the Registrant and invited him to provide comments within 30 days of the receipt of the draft decision.

By 2 July 2012 the Registrant did not provide any comments on the draft decision to ECHA.

On 19 July 2012 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 to amend the draft decision within 30 days of the receipt of the notification.

Subsequently, Competent Authorities of the Member States did not propose amendments to the draft decision and ECHA took the decision pursuant to Article 51(3) of the REACH Regulation.

II. Information required

Pursuant to Articles 41(1)(a), 41(3) and 10(a)(ii) as well as Annex VI , section 2 of the REACH Regulation the Registrant shall submit for the registered substance:

a. Name or other identifier of the substance (Annex VI, 2.1.), as specified under section III.(a) below;
b. Composition of the substance (Annex VI, 2.3.), as specified under section III.(b) below;
c. Description of the analytical methods or the appropriate bibliographical references for the identification of the substance (Annex VI, 2.3.7.), as described under section III.(c) below.

Pursuant to Article 41(4) of the REACH Regulation the Registrant shall submit the information in the form of an updated IUCLID dossier to ECHA by **19 December 2012.**

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 requirement. The scope of the present decision covers information requirements relating to substance identity (Section 2 of Annex VI of the REACH Regulation). In accordance with Article 10(a)(ii) of the REACH Regulation, any registration made pursuant to Chapter 1 of Title II of the REACH Regulation shall contain this information.

Missing information related to substance identity

Pursuant to Article 10(a)(ii) and Annex VI, section 2 of the REACH Regulation, the technical dossier of the registration shall include information on the identity of the substance. Annex VI, section 2 lists information requirements that shall be sufficient to identify the registered substance.

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

ECHA notes that the Registrant identified the registered substance as of Unknown or Variable composition, Complex reaction products or Biological materials (UVCB). According to the ECHA "Guidance for the identification and naming of substances under REACH and CLP" (Version: 1.1, November 2011; referred to as "the Guidance" thereinafter), the naming of UVCB substances shall consist of two parts: the chemical name and a more detailed description of the manufacturing process. ECHA observes that the Registrant did not provide sufficient and appropriate information for the naming of the registered substance, as required under Annex VI Section 2.1 of the REACH Regulation.

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More specifically, the chemical name associated with the EC entry (EC No 272-234-3) and CAS entry (CAS No 272-234-3) assigned in the dossier indicates that the registered substance is a derivative of "phenol, dodecyl-", i.e. of a phenol including a linear C12 alkyl substituent at an undefined position on the aromatic ring. However, the compositional information reported in section 1.2 of the IUCLID dossier specifies that the substance is in fact a derivative of tetrapropenylphenol. ECHA points out that a tetrapropenyl substituent originates from the tetramerisation of propene and refers therefore to a specific branched C12 backbone structure. Such substituent is different than a dodecyl substituent.
In addition, ECHA observes that the structural information of the unreacted alkyl phenol would indicate that the alkyl substituent is in para- position relative to the hydroxyl group rather than in every possible position (ortho-, meta- and para) as suggested by the chemical name assigned to the registered substance.

Furthermore, the compositional information highlights also the presence of [redacted] concentration levels ([redacted] of an oil identified as "Distillates (petroleum), hydrotreated heavy paraffinic" with EC number 265-157-1 which is not reflected in the EC and CAS identifiers assigned to the registered substance. ECHA points out that, unless the information required to be provided in the dossier demonstrates that the oil can be excluded to a significant extent from the composition, the identity of the oil shall be quoted in the name of the registered substance since contributes to a [redacted] level [redacted] to its composition.

ECHA therefore concludes that the EC and CAS identifiers currently assigned by the Registrant to the registered substance are inappropriate for its identification.
Moreover, Registrant did not specify the chemical name of the UVCB substance which shall be specified in the "IUPAC name" field, as indicated in chapter 8.2.4 of the Guidance.

Elements of the manufacturing process description which are essential for the identification of the registered substance are also missing from the dossier. In particular, the identity of the alkyl phenol starting material has not been identified to a sufficient level of detail. ECHA points out that UVCB substances such as this starting material cannot be sufficiently identified by a chemical name only. As the composition of such starting material is to a significant extent known and is one of the factors determining the composition of the registered substance, compositional information of that starting material (in terms of overall ratio of the ortho-/ meta- and para- isomers and identity and upper and lower concentration levels of the groups of alkylphenol constituents presenting the same carbon number, including details of the branching type) is a necessary element for its identification and therefore for the identification of the registered substance itself. ECHA points that the qualitative information provided by the registrant on the presence of a carbon number distribution of the alkyl substituent of the phenol starting material ranging from C10 to C15 is not sufficient as such to derive the identity of the specific starting material as it provides neither complete structural information nor quantitative information on its composition.
Other essential elements of the process description have not been reported. The Registrant did not specify the exact ratio of the different reactants used for the manufacturing. Moreover, the Registrant did not define the parameters and the corresponding specifications (as values) used to control the composition of the registered substance, including the degree of sulfurisation and oligomerisation of the alkyl phenolate as well as the alkalinity of the manufactured substance. Furthermore no details on the collection and purification steps were provided.

In line with the above, the Registrant is accordingly requested to specify a chemical name

that is representative of the registered substance. The Registrant shall ensure that the chemical name reflects the exact identity of the alkylphenol starting material used and the presence of the specific oil in the composition of the registered substance.
The Registrant shall also delete from the dossier the CAS entry currently assigned to the substance and provide instead any available CAS information specifically corresponding to the substance.
The Registrant shall not remove or modify at this stage the EC entry currently assigned to this registration for technical reasons, the registration being linked to that EC entry in REACH-IT. To ensure unambiguous identification of the registered substance, the Registrant shall however specify in the dossier that the EC entry currently assigned does not specifically correspond to the registered substance.
Moreover, the Registrant shall provide the missing information on the manufacturing process description mentioned above (i.e. detailed compositional information of the alkylphenol starting material, ratio of reactants, details of the parameters used to control the composition of the manufactured substance and description of the purification/isolation steps).

As for the reporting of the information in IULCID, the chemical name and the manufacturing process description shall be specified in the "IUPAC name" and "Description" fields in IUCLID section 1.1, respectively. Any available CAS information shall be reported under the CAS information header of the reference substance in IUCLID section 1.1. The Registrant may report the CAS entry with CAS number 68784-26-9 under the "Related CAS information" header of the reference substance in IUCLID section 1.1. The Registrant shall also specify, in the "Remarks" field of the reference substance in IUCLID section 1.1, the following: "The EC entry 272-234-3 currently assigned does not specifically correspond to the registered substance. This identifier can technically not be modified or deleted at this stage in the present registration update".

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.

(b) Composition of the substance (Annex VI, 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 corner stone of all the REACH obligations.

ECHA notes that the registration does not contain sufficient and appropriate 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, the Registrant reported the constituents formed in the manufacturing of the registered substance under one generic entry for "Phenol, tetrapropenyl-, sulfurized, carbonates, calcium salts, overbased" and described them as "Phenol, alkylation products with $C_{10\text{-}15}$ branched olefins derived from propene oligomerization, calcium salts, sulfurized, carbonates, overbased" with a representative molecular formula of $C_{36}H_{58}Ca_2O_4S_{x\ (x=\ 1\text{-}3)}$. However, no further information has been provided on the identity of the constituents covered by this entry. Furthermore, the representative molecular formula includes one calcium and three oxygen atoms in addition to a formula fitting with a (poly)sulfanyl-bridged calcium bis-(C12-alkyl)-phenolate. It is unclear how the three oxygens and the calcium elements are bound to the (poly)sulfanyl-bridged calcium bis-

(C12-alkyl)-phenolate. ECHA notes that, in IUCLID Section 1.4, the Registrant stated that "this phenate composition is dominated by dimeric and trimeric alkylphenol oligomers with evidence that even more complicated multiply charged structures may also be present". However, this information is insufficient for ECHA to have a structural representation of the different constituents and groups of constituents covered by this entry.

In addition, the registrant specified, in the manufacturing process description, that carbon dioxide is added to increase the calcium incorporation as carbonate in the registered substance. However, the qualitative and quantitative information on the presence of calcium in the form of calcium carbonate has not been reported in the composition. ECHA therefore concludes that the identity and concentration of the constituents formed in the manufacturing of the registered substance have not been reported to a sufficient level of detail in the composition.

Furthermore, while the registered substance includes a [redacted] amount ([redacted]) of a highly refined mineral oil identified as "Distillates (petroleum), hydrotreated heavy paraffinic" with EC number 265-157-1, the identity and concentration of the different constituents and groups of hydrocarbon constituents present in the composition of the registered substance have also in this case not been specified to a sufficient level of detail. The description of this oil indicates that it consists of hydrocarbons having carbon numbers predominantly in the C20-C50 range and contains a relatively large proportion of saturated hydrocarbons. However, the dossier does not include any qualitative and quantitative information on the different hydrocarbon classes (including linear alkanes, branched alkanes, cycloalkanes, their unsaturated counterparts and the aromatic constituents presenting different number of aromatic cycles (mono-, di-, tri-,...) and on the carbon number distribution within each of these classes. ECHA points out that the identification of the constituents originating from the specific mineral oil used in the process is necessary for ECHA to have a chemical representation of the contribution of these constituents in the registered substance.

ECHA also notes that the Registrant reported in the composition the presence of residual "Phenol, dodecyl-, branched". However, the IUPAC name and molecular and structural information refer to a specific C10-branched-alkylphenol rather than alkylphenols including C12-alkylphenols expected to be present. Structural information on the branching type and quantitative information on the carbon number distribution within the residual alkylphenol constituents is also missing. ECHA therefore considers the compositional information on the residual alkylphenols insufficient and inappropriate.

According to chapter 4.3 of the Guidance, the Registrant should note that, for UVCB substances presenting a large number of constituents such as the registered substance, the following applies:

- All constituents present in the substance with a concentration of ≥ 10 % shall be identified and reported individually;
- All known constituents and the constituents relevant for the classification and/or PBT assessment of the registered substance shall be identified and reported individually; and
- Other constituents shall be identified as far as possible by a generic description of their chemical nature. The identification of these unknown constituent must here again be provided for ECHA to establish the composition of the substance as manufactured and to use the compositional information as one identifier for the registered substance.

- For the hydrocarbon constituents originating from the "Distillates (petroleum), hydrotreated heavy paraffinic", the reporting of the different hydrocarbon classes (including linear alkanes, branched alkanes, cycloalkanes, their unsaturated counterparts and the aromatic constituents presenting the same number of aromatic cycles in their structure (mono-aromatic, di-aromatic, ...) is necessary as a baseline for ECHA to establish the composition of the substance. For each group of constituents, quantitative information on the carbon number distribution shall also be specified to conclude on the compositional profile of the constituents within the group;
- The unknown residual alkylphenols shall be reported under one generic entry. Quantitative information on the carbon number distribution within this group shall also be specified.
- The unknown calcium phenate constituents shall be reported under at least one generic entry specifying clear structural information on the constituents covered (including compositional information, relative position and relative content of the different building blocks, such as the alkylphenols, calcium and sulfide groups which these calcium phenates consist of).

For each constituent and group of constituents, the typical, minimum and maximum concentration levels shall be specified.

The Registrant is accordingly requested to complete and correct the abovementioned information on the composition of the registered substance and on the identity of the individual constituents and groups of constituents present, for ECHA to have a precise chemical representation of what the substance consists of.

Regarding how to report the information in IUCLID, the following applies: For each constituent required to be reported individually, the IUPAC name, CAS name and CAS number (if available), molecular and structural formula, as well as the minimum, maximum and typical concentration, shall be reported in the appropriate fields in IUCLID. For the other constituents to be reported under a generic description, 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. Information on the carbon number distribution within the relevant groups of constituents shall be specified in the Description field of the reference substance for that group.
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: 1.0, June 2010) on the ECHA website.

The Registrant shall ensure that the reported composition is verifiable and therefore supported by the description of the analytical methods used for the identification and quantification of the constituents required to be identified and quantified, in line with Annex VI section 2.3.7. The description shall be sufficient for the methods to be reproduced and therefore include details of the experimental protocols followed, any calculation made and the results obtained.

(c) The description of the analytical methods (Annex VI, 2.3.7.)

ECHA notes that the Registrant has not provided any description of the analytical method used for the quantification of the registered substance including its constituents as required by Annex VI, 2.3.7. of the REACH Regulation.

ECHA notes that the Registrant has attached a copy of an HPLC chromatogram. However, this information is not part of any quantitative analysis of constituents and groups of constituents. Furthermore, the analytical information reported in the dossier does not provide any description of the analytical methods used for the identification and quantification of the constituents required to be reported in the composition, in line with the requirements specified in section III.(b) above.

Regarding the high concentration level of the mineral oil ([redacted]) reported in the composition, this oil is presented by the Registrant as acting (also) as a solvent in IULCLID section 1.2. In line with Article 3(1) of the REACH Regulation, the quantity of oil which can be removed without affecting its stability or changing the composition of the registered substance shall be excluded. However, the extent to which the oil can be removed is ambiguous. In particular, it is indicated in IUCLID section 4.1 of the dossier that "attempts to remove this base oil from the substance result in changes to the other components' original structures and the physicochemical characteristics of the substance as a whole. With this in mind, the substance is being registered to include the base oil as a component of the substance and thus the base oil is present in the test material". As already highlighted above in section III.(a), the presence of significant quantities of highly refined mineral oil as a group of constituents of the registered substance has not been reflected in the EC (No 272-234-3) and CAS (No 68784-26-9) identifiers reported in IUCLID section 1.1. Additionally, the fact that the base oil cannot be removed from the substance without affecting its properties and stability is in contradiction with the test material used to characterize some of the hazard endpoint data in sections 4-6 of the IUCLID dossier. Tests have been performed either with the constituent "Phenol, tetrapropenyl-, sulfurized, carbonates, calcium salts, overbased" in [redacted]% purity or with a commercial sample of "Phenol, tetrapropenyl-, sulfurized, carbonates, calcium salts, overbased". Typical purity of this material as distributed in commerce is [redacted]% alkyl phenol sulphide and [redacted]% highly refined lubricant base oil.

As the presence of high overall concentration of the constituents from the highly refined oil contributes to the identification of the registered substance, the description of the analytical methods used to determine the extent to which oil can be removed from the substance composition is also necessary to verify the identity of the registered substance. However this information is missing from the dossier.

The Registrant is accordingly requested to provide a description of the analytical methods used for the identification and quantification of the constituents and groups of constituents required to be reported in the composition of the registered substance. The description shall be sufficient for the methods to be reproduced and shall therefore include details of the experimental protocol followed, any calculation made and the results obtained. The information shall be sufficient for ECHA to verify both qualitatively and quantitatively the compositional information required to be specified in the dossier. ECHA reminds the Registrant that, for the characterisation of the cationic moieties, quantification of calcium element is necessary.

The Registrant shall also provide the description of the analytical methods used to determine the overall quantity of the highly refined mineral oil which can not be removed without affecting the stability of the substance or changing its composition. The information shall be sufficient for ECHA to conclude that all the relevant physical and chemical methods have been evaluated to extract the constituents from the oil.

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

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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