Committee for Risk Assessment
RAC

OPINION

on the harmonised classification of lead (environment)

Pursuant to Article 77(3)(c) of Regulation (EC) No 1907/2006 of the European Parliament and of the Council of 18 December 2006 concerning the Registration, Evaluation, Authorisation and Restriction of Chemicals

EC Number: 231-100-4
CAS Number: 7439-92-1

A77-O-0000007042-85-01/F

Adopted
16 September 2021

A77-O-0000007042-85-01/F
16 September 2021

OPINION OF THE COMMITTEE FOR RISK ASSESSMENT

A reassessment at the Request of the European Commission to review the harmonised classification of lead (environment) as adopted by RAC in its opinion of 30 November 2018

Pursuant to Article 77(3)(c) of Regulation (EC) No 1907/2006 of the European Parliament and of the Council of 18 December 2006 concerning the Registration, Evaluation, Authorisation and Restriction of Chemicals (the REACH Regulation), the Committee for Risk Assessment (RAC) has adopted an opinion on the harmonised classification of lead (environment).

I   PROCESS FOR ADOPTION OF THE OPINION

Following a request from The European Commission dated 13 May 2020, the Executive Director of ECHA in the mandate of 24 June 2020, requested RAC to prepare an opinion in relation to the harmonised classification of lead (environment).

Rapporteurs, appointed by RAC: **Michal Martínek (sequentially)**
                                            Michael Neumann

Members of the ad hoc working group appointed by RAC:
                                            Ignacio de la Flor Tejero
                                            Laure Geoffroy
                                            Raili Moldov
                                            Marián Rucki

The opinion was discussed at:

- **RAC 55**, December 2020
- **RAC 56**, March; **RAC 57**, June (and at its preparatory working group); **RAC 58**, September 2021.

During the development of this opinion, the appointed ad hoc working group met on nine occasions:

- 9 October and 18 November 2020 and on
- 14 January, 19 and 25 February, 31 March, 8 and 21 April, 20 May 2021.

The RAC opinion was adopted **on 16 September 2021 by consensus**.

II   OPINION OF RAC

Request from the European Commission to the Executive Director of ECHA

Following the Commission's request under Art. 77(3c) of REACH, RAC has reassessed the chronic Ecotoxicity Reference Value (ERV) for lead as well as its scientific opinion on how many entries are appropriate to appear in Annex VI to CLP, with regards to the massive (≥ 1 mm) and powder forms of lead (< 1 mm).

The reassessment took into account all information in the original CLH dossier as well as additional information received during this assessment that addressed:

- chronic aquatic toxicity,
- the effect of water chemistry on lead toxicity,
- the solubility of lead from the relevant forms, and
- the forms of lead themselves, in particular the potential for massive lead to produce particles <1mm during reasonable handling and use.

For completeness, RAC has also made a brief reassessment of rapid transformation, bioaccumulation, and the acute ERV (Sections III.6, III.7, and III.8.7).

CLP guidance on metals

RAC conducted an extensive review of the relevant CLP guidance in order to provide an interpretation for classifying metals with regards to the forms encountered (CLP guidance IV.5.5) (Sections III.4.1 – 4.6). It concluded that the potential for and relevance of particles generated from reasonable handling and use of the massive form should be determined when assessing the number of entries any given metal warrants. RAC concluded that the guidance intends that any amount of particles < 1 mm generated from reasonable handling and use is relevant and that as these will fall into the established general category of powder (particle diameter < 1 mm), generation of such particles would justify classifying the massive metal based on data from the powder, where T/Dp data on the powder derives a more stringent classification. RAC also concluded that the term 'special process' as applied to the production of metal powders in the CLP guidance had little relevance to the above.

Chronic Aquatic Toxicity

RAC concludes that pH has no significant influence on the toxicity of lead in the aquatic environment and as such does not find that pH banding is warranted for lead (Section III.8.1 – 8.6). As a consequence, the lowest ERV value is compared with the T/Dp data at the pH that gives the highest dissolution. RAC further concludes that for lead this occurs at pH 5.5 for chronic aquatic hazards. Hardness also has no influence on toxicity and although DOC has an influence on lead toxicity, the range of data available covers a wide range of water chemistry eventualities and is representative of wide range of natural scenarios. Therefore, no normalisation is required for any water chemistry parameters. As an extension of this conclusion, it was not found appropriate to use the (Biotic Ligand Model) BLM for lead ERVs under CLP.

RAC concludes that the freshwater snail *Lymnaea stagnalis* is the most sensitive organism under chronic testing with newly hatched juveniles being more sensitive than adults

following exposure to lead in aqueous solution. Furthermore, juvenile growth and reproductive output beyond 28 days appear to be the most sensitive endpoints. Regarding the new study by Fox (2020) submitted by Industry, this followed the latest OECD TG (OECD TG 243) and was found to be reliable and relevant for classification although it used adult snails at test initiation, did not include the most sensitive endpoints (growth of juveniles or reproductive output past 28 days), and did not provide the lowest chronic ERV available in the dataset. Although carried out to an Internationally standardised test Guidelines, RAC did not consider that this new study could overrule or was more relevant than the earlier studies on the growth of the more sensitive juvenile forms of the pond snail. In this regard, RAC points out that a single new study is only rarely able to significantly influence the outcome, when all the evidence in a large data base is weighed together.

Consequently, the chosen chronic ERV for lead is an EC_{10} of 0.48 µg/L for *L. stagnalis* from Munley *et al*. (2013).

Assessment of the forms of lead

RAC assessed a wide range of information provided by Industry before and during the development of this opinion, on the uses of lead and the generation of particles < 1 mm in diameter from the reasonably expected use. RAC concludes that particles < 1 mm are generated from industrial processing of lead sheets, the main evidence originates from an example of cutting lead sheets[1] elaborated by Industry. As the quantity of lead particles < 1 mm is considered irrelevant (there is no scientific evidence as to where a threshold quantity might lie) and a significant proportion of the particles are below 1 mm in diameter, these are considered relevant for the classification of massive lead.

Furthermore, lead powder is not a structurally different material (despite lead powder being produced via a dedicated process) and derives a more stringent classification than the massive form (based on T/Dp data from 1 mm particle or equivalent surface area). Finally, as particles < 1 mm generated from the documented use of massive lead fall in the general category of powers (i.e., < 1 mm), RAC considers that lead powder is suitable for classifying massive lead and massive lead should be classified based on the T/Dp data from lead powder (Sections III.5.1 – 5.7).

Comparison with the CLP criteria

RAC concludes that the following ERVs should be used for classification:

- Acute aquatic toxicity: 20.5 µg/L *P. subcapitata*
- Chronic aquatic toxicity: 0.48 µg/L *L. stagnalis*

RAC concludes that lead powder is suitable for the classification of massive lead and that T/Dp data for the powder should be used. As a consequence:

- Dissolution of lead powder over 7 days at pH 6 and 1mg/L loading (390 µg Pb/L)

---

[1] Whether the lead sheets involved in the example technically qualify as a substance, as articles or the swarf produced by cutting the sheets qualifies as a by-product or a waste is not considered by RAC as relevant to hazard assessment; they all concern metallic lead.

- is larger than the acute ERV (20.5 µg Pb/L) so classification as Aquatic Acute 1 – H400 is warranted.
- The ratio between the acute ERV and the dissolution rate is 19, so an acute M-factor of 10 is warranted
- Dissolution of lead powder over 28 days at pH 5.5 at 0.1 mg/L loading (94.28 µg Pb/L) is larger than the chronic ERV (0.48 µg Pb/L) so classification as Aquatic Chronic 1 – H410 is warranted.
- The ratio between the chronic ERV and T/Dp value over 28 days at pH 5.5 at 0.1 mg/L loading (94.28 µg Pb/L) is 196.4, so a chronic M-factor of 100 is warranted.

Consequently, lead warrants classification as:

Aquatic Acute 1 (H400), M = 10

Aquatic Chronic 1 (H410), M = 100

RAC notes the changes in M-factors when compared to the classification of lead powder from its previous opinion of 2018. The provision of new T/Dp data for lead powder, as well as the reassessment of the chronic ERV value, have contributed to these changes.

III SCIENTIFIC GROUNDS AND BACKGROUND INFORMATION

1 Background, mandate, and process

1.1 Current Annex VI entries for lead

Lead metal currently has two Annex VI entries:

- Lead powder [particle diameter < 1 mm]: Repr. 1A; H360FD (H360D: C ≥ 0,03%), Lact; H362, Aquatic Acute 1, M=1, Aquatic Chronic 1, M=10
- Lead massive [particle diameter ≥ 1 mm]: Repr. 1A; H360FD, Lact; H362

The two entries only differ in the concentration limit for Repr. 1A; H360D. The specific concentration limit (SCL) of 0,03% applies only to the powder form.

1.2 RAC opinion from 2013

In 2013, RAC concluded on a single human health classification of Repro 1A; H360D with and SCL of 0,03% for lead, covering the human health concerns of both lead powder and massive lead. The main argument for human health hazard assessment was that during reasonably expected use such as e.g., grinding, filing, sawing, melting, or soldering of massive lead, small and potent particles/fumes that are ingestible and/or inhalable can be produced.

Following the intervention of industry, the Meeting of Competent Authorities for REACH and CLP (CARACAL) decided to split the harmonised classification into two entries in Annex VI due to "the lack of certainty regarding the bioavailability of lead in the massive form" (9th ATP to CLP, Reg. (EU) 2016/1179).

1.3 RAC opinion from 2018

In 2018, RAC concluded on a single environmental classification of Aquatic Acute 1; H400, M=1, and Aquatic Chronic 1; H410, M=10 for lead, covering the aquatic hazard concerns of both lead powder and lead massive.

RAC concluded, that the conditions of the CLP regulation and Guidance on the Application of the CLP Criteria (hereafter CLP guidance) for a split environmental classification of a metal were not met in the case of lead and that the transformation/dissolution (T/Dp) data from the powder form were also representative of the massive form. This was in line with the proposal by the original dossier of the Dossier Submitter (DS) Denmark.

The RAC opinion from 2018 discussed under which conditions of the CLP regulation and guidance it would be justified to use T/Dp data from particles ≥ 1 mm which would result in a separate environmental classification for the massive form. However, these alternative considerations were ultimately not supported by RAC.

In May 2020, the above classification from RAC's 2018 opinion on the aquatic hazards of lead was added to part 3 of Annex VI to CLP via ATP 15 (Commission Delegated Regulation (EU) 2020/1182 of 19 May 2020) for lead powder only.

1.4 Current mandate

Following the intervention of industry, the robustness of the RAC opinion from 2018 was questioned at the CARACAL-32 and CARACAL-33 meetings. In addition, industry conducted a new test using the new OECD TG 243 with the hermaphrodite freshwater snail *Lymnaea stagnalis* (Fox, 2020).

RAC subsequently received a request from the Commission through the Executive Director of ECHA in accordance with Article 77(3)(c) of the REACH Regulation (EC) No 1907/2006 to prepare an opinion on the environmental classification of lead. The opinion should focus on (1.) a reassessment of the ERV values for lead, using the existing dataset from the CLH dossier of the original Dossier Submitter (DS) Denmark and taking into account the new chronic toxicity study for lead in *L. stagnalis* (Fox, 2020), study number 1077.00101) and (2.) A re-examination of whether the powder and massive forms of lead warrant the same classification for hazards to the aquatic environment was also requested.

1.5 Targeted consultation held from 3 August to 7 September 2020

Comments were received from 5 Member State Competent Authorities (MSCA), from 1 non-EU National Authority, and from 4 industry organisations. Comments were sought on both aspects of the current mandate. Besides the comments, industry submitted a new full T/Dp test with lead powder (<75 µm) at pH 6 and 8 (Table 1).

2 Results of the general consultation

2.1 Comments on the new OECD TG 243 study in *Lymnaea stagnalis*

All 5 commenting MSCAs and one National Authority clearly indicated that in general a new ecotoxicity study does not automatically devalue or override the existing data that have been evaluated as valid, reliable, and relevant during a previous assessment.

The new Fox (2020) study with *L. stagnalis* showed a 17% reduction of the number of egg clutches per individual-day at the highest concentration. The observed effects on mortality, growth, and reproduction up to the highest test concentration were statistically not significant. Among all the available chronic studies on *L. stagnalis*,

Fox (2020) is the least sensitive study available for this species. One comment explained this by indicating that the new study was only conducted with the less sensitive adult life-stage (measured by shell growth) and that other results clearly show that growth in the larval stage (measured as blot-dried wet weight) is a more sensitive endpoint.

Consequently, all 5 commenting MSCAs and one National Authority argue in their comments that a re-evaluation of the environmental classification of lead is not warranted and not necessary.

In contrast, comments received from industry expressed the opinion that a re-evaluation of the environmental classification is needed and justified. The main argument is based on the view that the new guideline study overrides the non-guideline study that provided the ERV in the 2018 RAC opinion (Parametrix, 2007).

The commenting National Authority considers that the new study may not be valid according to the OECD TG 243 validity criteria because there appear to be less than 4 egg clutches per individual-day in the control. RAC recalculated the data and concluded that the number of "egg-clutches per individual-day" was 13.5 and well above the validity criterion of 4.

2.2   Comments on a single classification vs two classifications

None of the commenting 5 MSCAs or 1 non-EU NA were in favour of a split classification of lead. Two MSCAs preferred a single classification, one of them noting that the powder and massive form of lead are structurally identical, and that higher dissolution rate of the powder compared to the massive form is not surprising given that this parameter depends on particle size.

Another MSCA was of the view that normally there would not be a split entry for the same metal according to the CLP guidance, and that a crucial question is whether significant amounts of particles with a specific surface area (mm^2/mg) larger than that of 1 mm spheres will be produced during reasonably expected use.

The Industry commentators generally considered the conditions for separate classifications of the powder and the massive form to be met. Although the powder and the massive form are not structurally different materials, the powder is manufactured by a distinct process (atomisation, compared to casting of the massive form). Lead powder is claimed not to be released from massive lead under foreseeable use (e.g., cutting, drilling) due to the softness of lead and comparison of the full T/Dp data showed a marked difference in dissolution rate between the two forms according to industry. Industry also called for consistency with previous cases such as zinc, cadmium, or nickel where T/Dp data for the two forms were considered separately for environmental classification. Finally, they objected to using the existence of lead films or lead shot as an argument against a split entry, pointing out that these are alloys and/or articles rather than substances.

3   Introduction and aim of the re-evaluation

In addition to evaluating the submission from the current Art. 77 3(c) consultation, RAC has also re-evaluated all comments received after the RAC opinion from 2013, during the development of the RAC opinion from 2018 and thereafter. This includes

e.g., comments received by MSCAs, by the original DS (Denmark) and by RAC members during RAC discussions at that time.

From industry, comments were received from e.g., Eurometaux (2013, 2017, 2020), the International Lead Association (ILA) (2017, 2018, 2020) and the Lead REACH Consortium (2017) and by P. C. Frost (2017) on the Manufacturing Methods, Products and Properties of Lead Metal. Arche Consulting derived an Acute/Chronic Ecotoxicity Reference Value (ERV) for lead in 2017.

The impact and consequences to Downstream Users (DU) such as the non-ferrous metals industry were highlighted and addressed by comments received from The European Copper Institute (2017), European Aluminium (2017), the *Fachverbandes der Nichteisenmetallindustrie* and *the Fachverbandes Bergbau-Stahl* (2017) as well from the *WirtschaftsVereinigung Metalle* (WVMetalle), the German Non-Ferrous Metals Association (2017, 2020).

RAC has assessed the harmonised classification of lead in line with the CLP regulation and guidance including the specific environmental classification guidance for metals (CLP guidance, Annex IV). A significant part of this new RAC opinion is based on an intensive analysis and discussion of the meaning of relevant sections in the CLP regulation (CLP 1.3.4 and 4.1.2.10) and CLP guidance (IV.2.3 and IV.5.5). In this analysis, RAC has also taken into account the environmental classification of metals in the past following either CLP and the CLP guidance or the previous legislation: Dangerous Substance Directive (Dir. 67/548/EEC) (DSD).

The aim was to provide a scientifically robust assessment resulting in a well justified harmonised environmental classification for lead.

3.1 Important terms for metal forms

Powder – Refers to a metal form deliberately produced by a dedicated production method with a diameter < 1 mm and/or a specific surface area (e.g., m^2/g) greater than the specific surface area of a spherical particle of 1 mm.

Massive form – Refers to metals in any form with a diameter ≥ 1 mm and/or a specific surface area (e.g., m^2/g) equal to or less than the specific surface area of a spherical particle of 1 mm diameter.

Generated particles < 1 mm – Refers to particles with a diameter < 1 mm or specific surface area (e.g., m^2/g) greater than the specific surface area of a spherical particle of 1 mm, unintentionally generated from reasonably expected use of the massive form.

3.2 The assessment of the intrinsic hazard of metals under CLP

For each metal an unlimited number of environmental classifications and entries in Annex VI would be theoretically created if each specific form of a metal would be tested in transformation/dissolution (T/Dp) test systems in the form it is placed on the market and the concentrations of released ions compared with the ERVs. The scientific reason is explained in the part of the CLP guidance available for metals in Annex IV.5.5:

Surface area is a crucial parameter in that variation in surface area tested may cause a significant change in the levels of metals ions released in a given time-window. However, the intrinsic hazard of the diverse massive forms of a metal is not caused

by the specific form which is marketed but by small particles which may be generally generated by reasonably expected use. This fact is reflected in the OECD guidance document on transformation/dissolution of metals and metal compounds in aqueous media (No 29), where it states:

> *9. As in addition the surface area of the particles in the test sample has an important influence on the rate and extent of transformation/dissolution, powders are tested at the smallest representative particle size as placed on the market, while massives are tested at a particle size representative of normal handling and use.*

Consequently, for the testing of powders, the smallest particle on the market shall be tested. On the other hand, the intrinsic hazard for most metals is not caused by the massive form itself but rather by small particles of the metal generated by reasonably expected use (< 1 mm).

The intention to classify the massive form based on the generation of particles < 1 mm during reasonably expected use is also indicated by the labelling derogation of metals in massive form given in section 1.3.4 of Annex I to the CLP-regulation. Particularly, CLP 1.3.4.2 says:

> *Instead, the supplier shall provide the information to downstream users or distributors by means of the SDS.*

Hence, the massive form may still need to be classified in order to inform the professional user that subsequent transformation of the substance may produce the hazardous form.

In the case that the representative size and surface area of these generated particles < 1 mm causes the same intrinsic hazard as the powder form on the market, a single classification of the metal would be scientifically justified. Annex IV.5.5 further defines:

> *Thus, particle size or surface area is fixed for the purposes of the transformation test, allowing the comparative classifications to be based solely on the loading level. Normally, the classification data generated would have used the smallest particle size marketed to determine the extent of transformation.*

Here, the CLP guidance suggests that that the smallest particle size marketed may be representative of a metal and form the basis of classification. This is justified either under worst case considerations or if the massive form of the metal generates small particles under reasonably expected use.

Both cases would be in line with CLP guidance Section 1.2.3.3:

> *The system of classification is designed to ensure that a single classification applies to a substance. In general, it takes no account of the specific form since this can vary and is not intrinsic to the substance.*

4   Hazard assessment of the different forms of metals

4.1   Hazard assessment of the powder form (< 1 mm) of metals

If the powder form (< 1 mm) is to be classified under CLP for aquatic hazards the hazard assessment according to the CLP guidance Annex IV.5.5 is as follows:

> *Metals with a particle size smaller than the default diameter value of 1 mm can be tested on a case-by-case basis. One example of this is where metal powders are produced by a different production technique or where the powders give rise to a higher dissolution (or reaction) rate than the massive form leading to a more stringent classification.*

This is in line with the above-mentioned OECD Doc 29. Both indicate that the intrinsic hazard of powders of metals should be tested and assessed based on the smallest form on the market. This seems to be necessary if the produced powders are different than the small particles (< 1 mm) generated by reasonably expected use of the massive forms, for example, if they are produced by a dedicated process.

4.2   Considerations on the default diameter value of 1 mm in T/Dp testing

Both the CLP guidance and the OECD Doc 29 request that the intrinsic hazard of the massive form of metals is assessed with small particles (< 1 mm) generated by reasonably expected use. However, a diameter value of 1 mm is mentioned as a default. In OECD Doc 29 it is stated:

> *[…] while massives are tested at a particle size representative of normal handling and use. A default diameter value of 1 mm should be used in absence of this information. For massive metals, this default may only be exceeded when sufficiently justified.*

Consequently, a default diameter value of 1 mm should only be used for assessing the intrinsic hazard of the massive form of a metal if the information on any generated particles < 1 mm representative of reasonably expected use of the metal is absent.

The 1 mm default would also be used where particles < 1 mm are not generated from reasonably expected use.

In the interest of understanding the significance of the "*default diameter value of 1 mm*" mentioned in OECD Doc 29 and the CLP guidance for the environmental classification of the massive forms of a metal, RAC decided to examine documentation from the discussions around the development of guidance held under the Technical Committee for Classification and Labelling (TCC&L), which were held from 1995 until the early 2000s.

In these documents, it can be seen that the background to the discussions indicates that the particle size used for testing of the massive form of a metal should be representative in size and surface area of normal (reasonably expected) handling and use (ECBI/61/95 add 46) and these considerations appear in the T/D protocol text (OECD Doc 29). It becomes apparent that 1 mm was adopted as a default diameter value for the massive form of a metal if reasonably expected use does not generate particles < 1 mm or in case information on the representative size and surface area of such generated particles is not available.

Documentation submitted to RAC by the Swedish CA (SE Doc binder) and industry (ILA Feb 2021) during February 2021 confirm that this was the intention behind the design of the classification system under Directive 67/548/EEC (DSD).

4.3 Previous cases under the Dangerous Substances Directive: Directive 67/548/EEC (DSD)

Under DSD, a number of metals were classified until the introduction of CLP in 2008. These are summarised below. The classifications of cadmium, zinc and nickel were agreed at a time when the GHS guidance (2001) was already available. Still, the TC C&L appears to have followed the DSD guidance from 1997, according to which a separate classification of the powder and massive form based on T/Dp data was considered more or less automatically and only for few metals was a topic of considerable discussion.

Cadmium

Cadmium was the first metal whose environmental classification was agreed after the introduction of the DSD guidance. Cadmium powder was classified with N; R50-53 (very toxic to aquatic organisms, may cause long-term adverse effects in the aquatic environment). In 2000, TC C&L agreed to request a T/Dp test with cadmium in massive form (default particle size: 1 mm). The results of the test with massive, supplied in 2002, resulted in the same classification outcome as the powder, i.e., N; R50-53. Consequently, all forms of cadmium now have a harmonized classification as Aquatic Acute 1 and Aquatic Chronic 1.

Zinc

Zinc powder was provisionally classified with N; R50-53 in 1997 based on a T/Dp test. The discussions on the massive form were reopened in 2000. Despite the previous agreement on a default value of 1 mm, there was a considerable controversy about the cut-off value between massive and powder for zinc. The situation was further complicated by the lack of T/Dp data at pH 6 (only data for pH 8 was available). At a certain point, R53 for the massive without specification of the cut-off value appeared to be a viable compromise, but then industry and some MSs returned to the idea of a cut-off value. The cut-off limits discussed were in the order of 1 cm, but no agreement could be reached.

Massive zinc forms are melted and used by first line users such as galvanizers, zinc alloy producers and rolled zinc manufactures. Massive zinc is not polished, grounded, machined or handled in such a way that may give rise to small particles. This was concluded also from data submitted by the galvanizing, rolled zinc and zinc alloy zinc industry (See table 2 in Annex 1) (Galvanizing Industry, 1999; Rolled Zinc Industry, 2000; Zinc Alloy Industry, 2001).'

In September 2000, NL argued that a classification of zinc in massive form was currently not considered relevant as no particle size of < 1 mm was to be expected but this should be confirmed by industry (doc 4800a1_II). Only a small portion (< 0.01%) on the market of produced massive zinc concerns particles around 1 mm. RAC notes that the proposal by the NL was never accepted by the ECB C&L WG and no conclusion for zinc in massive form ever drawn (ECBI/48/00 add 1).

Due to disagreement within the ECB C&L WG two compromises on zinc massive were sent to the European Commission after the November 2002 meeting: (1) R53 for

whole zinc massive; (2) N; R50-53 for particles up to a certain size and no classification above this size (cut-off value to be further defined). RAC notes that the discussions were stopped and never resumed. Although there was no T/Dp-test data for all pH-bands available, the data at hand showed that based on T/D-testing of 1 mm particle size zinc should have been classified as N; R50-53. The outcome was that zinc powder/dust (size limit not specified) has a harmonized classification as Aquatic Acute 1 and Aquatic Chronic 1, zinc massive does not have an Annex VI entry.

Nickel

The classification of nickel was agreed in TC C&L in 2006. The powder was classified with R52-53 based on a T/Dp test with the smallest representative particle size while the T/Dp test for the massive, conducted with particles around 0.8 mm, did not lead to classification. Nickel has two Annex VI entries: 'nickel' is not classified for environmental hazards while 'nickel powder [particle diameter < 1 mm]' has a harmonised classification as Aquatic Chronic 3.

4.4   Previous cases under CLP

Copper

There are two harmonised entries for copper, both agreed under CLP: copper flakes (RAC opinion 2014) and granulated copper (RAC opinion 2018). Both substances were classified based on T/Dp data for that particular form: copper flakes as Aquatic Acute 1 (M=10) and Aquatic Chronic 1 (M=10), granulated copper as Aquatic Chronic 2. A CLH dossier for granulated copper was submitted as this form in particular is used a biocidal active substance and there was no generic entry for 'copper' on which to base a hazard assessment. As of November 2021, a CLH dossier with proposals for 'copper' has been submitted to ECHA by the Swedish CA.

4.5   Small particles < 1 mm generated from reasonably expected use

In section 1.2.2, the CLP guidance defines the term 'reasonably expected use' as all physical forms and states of a substance or mixture that may occur during intended use or reasonably foreseeable conditions of misuse. It includes production, handling, disposal, any technical operations (e.g., sawing, drilling, grinding, etc), any professional and non-professional uses as well as reasonably foreseeable accidental exposure but not abuse such as criminal or suicidal uses.

As mentioned before, concerning the intrinsic environmental hazard, particles < 1mm generated from reasonably expected use are more hazardous for most metals, because of the larger specific surface area and therefore increased transformation/dissolution. Therefore, generated particles < 1 mm need to be taken into account when assessing the intrinsic hazard of the massive form of a metal.

Based on the available guidance and information on previous assessments, the considerations and arguments for the relevance of particles < 1 mm generated under reasonably expected use for the hazard assessment of the massive form could be based on:

- whether such particles are generally generated by reasonably expected uses

- here, the amount in which particles < 1mm are generated from massive could be a relevant aspect, although there is no guidance or criteria by which to assess a relevant quantity
- the fact that such generated particles < 1 mm present a hazard greater than that of the specific massive form to be classified or that of default 1 mm particles tested
- size, shape, surface area of the small particles
- the produced powder and the generated small particles are of the same crystallographic structure

If generated particles < 1 mm are considered relevant for the hazard assessment of the massive form, then they need to be T/Dp tested. Alternatively, particles with representative particle size or surface area may be T/Dp tested.

Therefore, RAC considers that if particles < 1 mm can be generated through reasonably expected use of the massive metal and, if such particles are relevant for classification of the massive form, the massive form should be classified based on T/Dp testing of such representative generated particles < 1 mm.

4.6 Hazard assessment of the massive form of metals

If the massive form (< 1 mm) is to be classified under CLP for aquatic hazards the hazard assessment follows some key considerations as follows.

- If under reasonably expected use particles < 1 mm are generated either intentionally (e.g., via atomisation) or unintentionally (e.g., during casting cutting, milling, grinding etc.).

- if such generated particles< 1 mm are relevant for the environmental classification based on available information.

Due to the generation of relevant particles < 1 mm, the massive form may be classified by using the T/Dp test data for a produced powder. This can be done if the data from the produced powder are also suitable for classification of the massive form.

The CLP guidance, section IV.5.5 (corresponding to GHS, Annex 9, A9.7.5.4) states:

There may be cases where data generated for a particular metal powder are not considered as suitable for classification of the massive forms. For example, where it can be shown that the tested powder is structurally a different material (e.g., different crystallographic structure) and/or it has been produced by a special process and is not generally generated from the massive metal, classification of the massive can be based on testing of a more representative particle size or surface area, if such data are available. The powder may be classified separately based on the data generated on the powder. However, in normal circumstances it is not anticipated that more than two classification proposals would be made for the same metal.

This passage determines that when the powder form is used for classifying the massive form it should be suitable for doing so and should not be, for example:

(1) structurally different from the massive (e.g., crystallographically)

and/or

(2) produced via a dedicated process, which means it is not (generally) generated from [the use of] the massive form

Taking all of the above into account, RAC has developed a scheme (Fig. 1) to visualise the key questions in environmental hazard assessment of the massive form (≥ 1 mm) of a metal. The aim of the scheme is to help understand in which cases the default diameter value of 1 mm and in which cases the small particles < 1 mm generated by reasonably expected use represent the intrinsic hazard of the massive form. The scheme also visualises in which cases the classification of the massive form should be based on test results from produced powders.

Flow chart for aquatic hazard assessment of the massive form > 1 mm of a metal
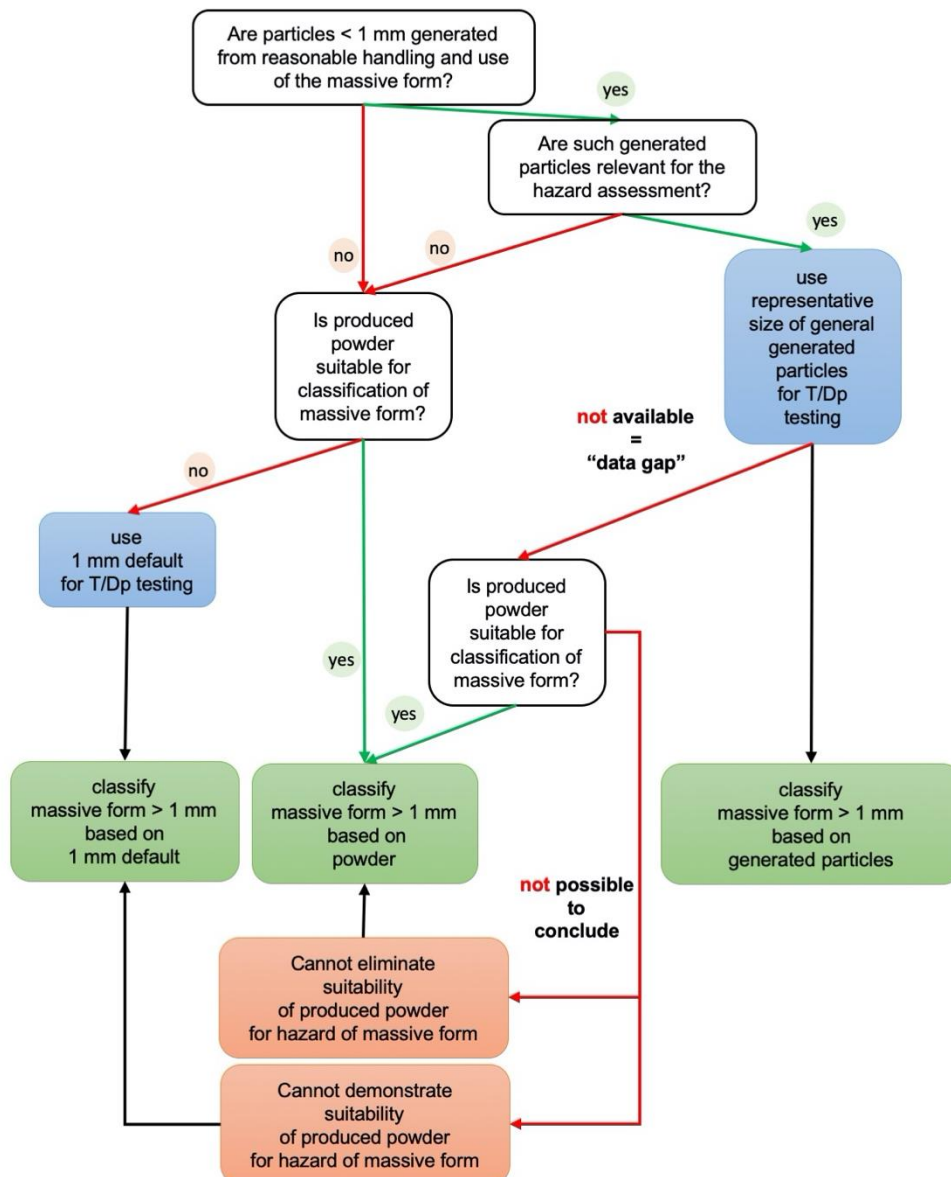

Figure 1: Flow chart for the aquatic hazard assessment of the massive form > 1 mm of a metal under CLP.

RAC considers, that under CLP, a default diameter value of 1 mm should only be used for environmental classification of the massive form if (1) reasonably expected use

conclusively does not generate particles < 1 mm, or if (2) particles < 1 mm generated by reasonably expected use are not considered relevant for classification of the massive form (as outlined in section III.4.5), or if (3) no information is available on which to assess the relevance of such particles.

5 Assessment for lead and determination of whether a single or separate classification for the powder and the massive forms are warranted

Having examined the CLP guidance and arrived at an interpretation of its underlying intention for metals in general, the information available for lead can be appropriately assessed using the above scheme.

5.1 Solubility of lead

Measured T/Dp data at a loading of 1 mg/L are available for particles with a size < 75 µm (mean diameter 0.03 mm, specific surface area 0.05 m^2/g, 2012BJ_Kr_EN) in the form of powders only at pH 6 and 8 (Brouwers, 2020) and for the default diameter value of 1 mm with a specific surface area of 0.000529 m^2/g only at pH 6, 7 and 8 (Rodríguez *et al.*, 2009 and 2012; Arbildua *et al.*, 2017). Missing measured data at pH 5.5 (for both particle sizes) and pH 7 (for particles with a size < 75 µm) were interpolated applying linear or polynomial linear regression (calculated by RAC). The results are presented in table 1 below. Hereafter, these forms shall be referred to as 'powder' and 'massive' for lead in this opinion.

Table 1: Available data of the concentration of dissolved lead in solution [µg/L] resulting from measurements at a loading of 1 mg/L following OECD Doc 29 and calculation based on linear or polynomial linear regression. For the loading of 0.1 mg/l the calculations assume a linear relationship between particles size/surface area and dissolution rate since these values are needed for comparison with CLP criteria.

Time	Loading	concentration of dissolved lead in solution [µg/l]			
		pH 5.5	pH 6	pH 7	pH 8
		powder < 75 µm			
7 days	1 mg/L	No data	390 (measured)	163 (calculated)	68.5 (measured)
28 days	1 mg/L	942.8 (calculated)	639 (measured)	292 (calculated)	134 (measured)
	0.1 mg/L	94.28 (calculated)	63.9 (calculated)	29.2 (calculated)	13.4 (calculated)
		Massive (default diameter value of 1 mm or equivalent specific surface area)			
7 days	1 mg/L	No data	5.1 (measured)	2 (measured)	0.28 (measured)
28 days	1 mg/L	52.1 (calculated)	14.2 (measured)	2 (measured)	0.66 (measured)
	0.1 mg/L	5.21 (calculated)	1.42 (calculated)	0.2 (calculated)	0.066 (calculated)

RAC notes, that neither particles < 1 mm generated from the reasonably expected use of the massive form, nor particles which are representative in size and surface area of such generated particles < 1 mm have been T/Dp tested. Table 2 presents the relative higher dissolution of the powder compared to the default diameter value of 1 mm or equivalent specific surface area. RAC notes that the relative difference in the concentration of dissolved lead between powder and massive [µg/L] is decreasing with decreasing pH and reaches its minimum after 28 days at pH 5.5.

Table 2: Relative higher dissolution of lead tested in the form of powder (< 75 µm, mean diameter 0.03 mm) compared to the massive (1 mm)

Time	pH 5.5	pH 6	pH 7	pH 8
7 days		76×	81x	240×
28 days	18x	45×	146x	200×

While it can be stated that the T/Dp data indicate different dissolution for the powder compared to the massive it needs to be evaluated if the T/Dp data from the powder or from the massive are more representative for the particles < 1 mm generated by reasonably expected use of the massive form.

The OECD guidance document on transformation/dissolution of metals and metal compounds in aqueous media (No 29) clarifies:

> *8. As pH has a significant influence on transformation/dissolution both the screening test and the full test should in principle be carried out at a pH that maximises the concentration of the dissolved metal ions in solution. With reference to the conditions generally found in the environment a pH range of 6 to 8.5 must be used, except for the 28-day full test where the pH range of 5.5 to 8.5 should be used in order to take into consideration possible long term effects on acidic lakes.*

Also, the CLP guidance under IV.2.2.3 requests:

> *The Full Transformation Dissolution test should be carried out at the pH (footnote 92) that maximises the concentration of dissolved metal ions in solution and that expresses the highest toxicity.*

In Footnote 92 it confirms:

> *The UN GHS transformation/dissolution protocol specifies a pH range of 6-8.5 for the 7 days test and 5.5 to 8.5 for the 28 days test. Considering the difficulty in carrying out transformation/dissolution tests at pH 5.5, the OECD only validated the test in the pH range of 6-to 8.5.*

No scientific explanation is given as to why it might be difficult to maintain the pH at 5.5 in T/Dp testing. However, RAC notes that the CLP guidance does not remove the obligation to assess the dissolution at the pH that maximises the concentration of dissolved lead in solution. In the case of lead, this is at the pH of 5.5 as the concentration of lead in the solution increased with decreasing pH. The available T/Dp test results at pH 6 may underestimate chronic toxicity in acidic water bodies, a concern outlined in the T/Dp.

According to the study Vuorenmaa and Forsius (2008), more than 55% of lakes in Finland have a pH lower than 6.0 and 25 % a pH even lower than 5.5. A similar

situation is known in Norway and Sweden. Problems with acidification of lakes and rivers in Europe persists even after successful SO_2 emission reduction measures and this can be seen in, for example, Czech Republic, Germany, Slovakia, Italy, Austria, Switzerland, Denmark, France, and the UK where acidification of lakes and rivers continues.

There is also a phenomenon called stratification, usually caused by temperature differences within a body of water, where each layer of water does not mix with the layers above or below and which can maintain layers of high pH water[2].

On this basis, RAC concludes that the concentration of the dissolved lead ions in solution at pH 5.5 should be used for the classification of lead for chronic aquatic hazards.

5.2 Small particles < 1mm generated from reasonably expected use of the massive form of lead

Lead is a relatively soft material with Moh number of 1.5 with massive lead being used in a broad range of applications, which includes blocks and sheets. Its range of uses in the EU, is summarised in Table 3 along with the estimated tonnages in 2021, taken from industry documentation (First uses of lead metal-2021).

Table 3: Summary of lead uses and estimates quantities in the EU in 2021.

Use	Tonnes (2021 ILA estimate)	Production	Notes
Automotive batteries	809000	Lead metal cast into plates/grids	The overwhelming majority of batteries use lead alloys. Information provided by Industry indicates that no appreciable amounts of particles < 1 mm are generated from modern battery production methods.
Industrial batteries	460000	Lead metal cast into plates/grids	The overwhelming majority of batteries use lead alloys. Information provided by Industry indicates that no appreciable amounts of particles < 1 mm are generated from modern battery production methods.
Rolled extruded products	50000	Rolling or extruding from lead ingots	47500 tonnes are in the form of lead sheet mentioned elsewhere in this text

---

[2] These layers are separated by thermoclines (temperature divides) or chemoclines (chemistry gradients). Chemoclines can be based on oxygen, carbon dioxide or salinity, or other chemical factors that do not cross the cline. Differences in pH levels between water strata are due to increased CO_2 from respiration and decomposition below the thermocline. In some cases, the pH can rapidly drop from a surface level around pH 7 down to pH 5.5. This significant drop comes from the saturated CO_2 that is stored up in the lower strata of the lake.

Use	Tonnes (2021 ILA estimate)	Production	Notes
Shot and ammunition	57000	Ingots melted in sot towers to produce spheres > 1 mm	Hardness often controlled by alloying with Tin or Antimony
Cable sheathing	20000	Ingots extruded into sheathing	
Lead compounds manufacture	20000	Processed melted lead ingots	
Alloys	5000	Alloyed to brass, aluminium etc.	
Total tonnage 2021	1421000		

In the Industry document summarised in Table 3, the largest use is in battery manufacture (automotive and industrial) which are either stamped or cast from molten lead. RAC was informed that no cutting as with lead sheets/rolls takes place. Information from casting of lead ingots indicates that casting does not produce small particles in appreciable quantities (Information request from RAC on manufacture process for lead batteries). Information provided by Industry indicates that no appreciable amounts of particles < 1 mm are generated from modern battery production methods. Furthermore, it also appears that pure lead use in batteries is a minor part of the battery market and is not the industry standard, the overwhelming majority of batteries use lead alloys. As a consequence, generation of particles < 1 mm of pure lead from battery manufacture does not appear to be relevant. However, other information from industry clearly indicates that small particles < 1 mm can be generated (ILA Feb 2021, K. Uses of Lead in the EU and particles can be generated from the massive) amongst the swarf generated from cutting lead sheet in the form of unprocessed lead rolls, although this does not leave the factory (Lead Metal ENV Classification_April 2021). Such information for the other lead uses included in table 3 are not available. Unprocessed rolls constitute approximately 3% of annual massive lead production in the EU, which amounts to 47500 tonnes of sheet. Although these sheets are considered as articles (Regulation (EC) No 1907/2006, Art. 3(3)) due to their shape (Lead Sheet Process Flow; REACH – recycling – authorisation – helpdesk ref2304DXG13-0234), the swarf generated is itself considered a substance according to Directive 2008/98/EC Art. 5 on by-products, as the swarf is:

a) certain to be reused,
b) not subject to further processing other than that required by manufacture of the lead sheet,
c) produced as an integral part of production of lead sheet,
d) produced in a lawful manner.

Consequently, RAC considers that generation of particles < 1 mm is not only likely from the broad uses of lead but is demonstrated by the evidence presented and that these particles are within the scope of hazard assessment under CLP. However, it is then essential to assess whether these particles are relevant for the hazard assessment of the massive form of lead (See section III.5.6 and 5.7).

Based on information submitted by clarifying the tonnage of lead sheet manufactured, the mass of swarf generated and the particle size distribution of the swarf, RAC has calculated the mass of particles fractions in swarf from cutting unprocessed lead sheet (Table 4).

Table 4: Fractions of lead roll cutting swarf by weight/year (based on 47500 tonnes lead sheet estimate for 2021).

Swarf fractions (mm)	% of total swarf	kg
All swarf	100	332500
8 - 4	27.8	92435
4 - 2	61.4	204155
2 - 1	9	29925
1 - 0.5	1.600	5321
0.5 - 0.250	0.166	550.62
0.250 - 0.125	0.031	102
< 0.125	0.002	5.4

Industry has provided particle size analysis data of the swarf indicated above (Appendix 3-BLM British Lead Particle Size Distribution (003); Characterisation of Lead sheet Swarf-April 2021), from which is it possible to calculate specific surface areas for the particles in the swarf (Table 5) as well as dissolution values from particles as part of the swarf (Table 6).

Table 5: Specific surface area of particles as a fraction of 1g swarf sample provided by Industry

Size Range	calculated specific surface area per size fraction (m²/g)
8mm-4mm	3.05E-06
4mm-2mm	6.63E-05
2mm-1mm	1.69E-04
1mm-500µm	6.96E-05
500µm-250µm	1.44E-05
250µm-125µm	5.36E-06
<125µm	5.63E-07
Total calculated specific surface area of Swarf (m²/g)	0.000328

Table 6: Table of estimated standardised dissolution values from particles sizes found in swarf at 1 mg/L loading. Calculation of dissolution based on regression using provided T/Dp data and particle specific surface areas for powder and 1 mm particles used in the T/Dp.

Size Range	SA (m^2/g)	SA (m^2/mg)	Number of particles in each size fraction (per gram of swarf)	T/D data at 7d pH 6, 1 mg/L loading (µg/L)	T/D data at 28d pH 6, 1 mg/L loading (µg/L)
8mm-4mm	8.8183E-05	8.82E-08	2.70E-02	1.69	8.40
4mm-2mm	0.00017637	1.76E-07	2.34E+00	2.40	9.28
2mm-1mm	0.00035273	3.53E-07	2.39E+01	3.81	11.05
1mm-500µm	0.00060469	6.05E-07	3.94E+01	5.82	13.57
500µm-250µm	0.00141093	1.41E-06	3.26E+01	12.27	21.63
250µm-125µm	0.00282187	2.82E-06	4.85E+01	23.56	35.74
<125µm	0.00562873	5.63E-06	2.03E+01	46.01	63.81

As would be expected, dissolution increases as particle size decreases when standardised to m^2/g material at each particle size (Table 6). However, dissolution from the various particles found in a sample of swarf indicate that surface area and therefore dissolution is reasonably consistent across the particle sizes due to the numbers of particles in each size category.

5.3 Smallest form of lead available on the market and representative particle size

The powder tested was a −200 mesh (< 75 µm) powder. The sieve analysis of the material showed the following (Brouwers, 2020):

- +200 mesh (> 75 µm) 5%
- +325 mesh (> 45 µm) 27%
- −325 mesh (< 45 µm) 69%

RAC inquired as to whether this powder represents the smallest particle size on the market. According to industry, most powders on the market are −100 mesh (< 150 µm) as in the majority of applications there is no requirement to produce smaller particle sizes. Industry further explained that it is technically possible to produce particle sizes such as −325 mesh (<45 µm) but these would be custom made for specialist R&D laboratory purposes and are not representative of typical lead powders on the market.

RAC concludes, that the information available indicates that the lead powder used in the T/Dp test is representative of lead powders as the smallest form available on the market. Consequently, lead in powder form < 1 mm should be classified based on the available T/Dp test result with the powder, which in this case means lead powder with a mean particle diameter of 0.03 mm (2012BJ_GrainsSize).

5.4 Crystal structure

RAC notes that both powder and massive lead have the same crystalline structure (face centred cubic) and there is no evidence of a structural difference between the two forms.

5.5 Special process

As mentioned previously, the CLP guidance uses the term 'special process' to determine if produced powders of lead are suitable for classifying the massive form of lead.

It may be assumed that the condition of a 'special process' mentioned in the CLP guidance might need to involve chemical transformation or structure or crystallographic transformation. This is not the case for lead.

Lead powder is produced by a dedicated process (gas atomisation), which is distinct from that used to produce the massive metal. As industry confirmed, the process via atomisation where lead is melted and then disintegrated in a stream of gas to droplets that solidify upon subsequent cooling appears to be a standard process to produce powders of many metals.

Consequently, RAC considers that the CLP guidance intends that any dedicated process to produce powders is in general meant to be 'special' and that these powders need to have their suitability for classification of the massive form assessed.

5.6 Assessment of relevance of the particles < 1 mm

Lead is a relatively soft material with Moh number of 1.5 and massive lead is used in a range of applications, including in blocks or in sheets. Information from industry clearly indicates that particles < 1 mm can be generated amongst the swarf generated from sawing lead sheets and that particles < 1 mm are produced at approximately 6000kg/year (ILA Feb 2021, K. Uses of Lead in the EU and can powders be generated from the massive). Consequently, RAC considers that generation of particles < 1 mm is demonstrated by the evidence presented.

In order to assess the relevance of particles < 1 mm generated by reasonably expected use of massive for the hazard assessment of massive lead, RAC used the considerations outlined in section III.4.5 to assess potential relevance. To aid in this assessment, Table 7 demonstrates the general relationship between specific surface area and release of lead in relation to particle diameter using available measured data.

Table 7: Regression analysis of dissolution based on experimental data from particles of known specific surface area and dissolution

	Default	**Powder form**
Particle size diameter (mm)	1	< 0.075 (mean 0.03)
Specific surface area m^2/g	0.000529	0.05
Specific surface area m^2/mg	0.000000529	0.00005
dissolution under OECD protocol 29 at pH 6 and 1 mg/L loading (µg/L)	5.1	390
Regression equation	y = 8e+06x + 0.9842	y = 1e+07x + 7.5189

Although lead powder is produced by gas atomisation of molten lead, not by any chemical process, lead in massive and powder forms both have the same crystal structure and, consequently, lead can be considered the same material in both forms.

Taking these factors into account, RAC considers that two possible options need to be considered.

Option 1: Particles < 1 mm are relevant for the classification of massive lead

Lead in massive and powder forms both have the same crystal structure and lead powder is produced by gas atomisation of molten lead, not by any chemical process. Consequently, lead can be considered the same material in both forms.

RAC considers that documented evidence indicting production of particles < 1 mm from a widely used manufacturing method for the production of lead sheets (ILA Feb 2021, K. Uses of Lead in the EU and can powders be generated from the massive) demonstrates that particles of lead < 1 mm are generally generated from reasonably expected use of lead. As such, the quantity of particles < 1 mm produced is not relevant for assessing the relevance of particles < 1 mm.

Table 8: Specific surface area of minimum size in each particle size range based on particles as sphere.

Particle *d* mm	Calculated specific surface area (m^2/g), based on sphere
1	0.00053
0.5	0.00106
0.25	0.00212
0.125	0.00423
0.063	0.00840

RAC further considers that any production of particles < 1 mm would present an intrinsic hazard greater than that of the massive form (≥ 1 mm) as is indicated by the specific surface areas of particles < 1 mm indicated in Table 8, which are greater

than that of 1 mm particles or equivalent surface area. It is further considered that the actual swarf particles will not be spherical, so the data in Table 8 is likely to underestimate the actual specific surface area of such particles. In conclusion, particles < 1 mm must be taken into account for hazard assessment of massive lead regardless of the quantity produced.

Possible conclusions from option 1

- **Conclusion 1**: Use T/Dp test with powder because it is suitable to classify the massive form, because:
  - o the nature (e.g., size shape, surface area) or properties (e.g., dissolution) of particles < 1 mm generated from massive lead cannot be separated from lead powder.
- **Conclusion 2**: Classify massive lead based on particles < 1 mm generated from massive lead. Compare classification outcome with that for powder to determine whether separate classifications for massive and powder lead are warranted. RAC notes that no data are available for such particles and that attempts to model that data are unreliable as they rely on too many assumptions.

Option 2: Particles < 1 mm are not relevant for the hazard assessment of the massive form

Particles below 1 mm consist of only 1.8% of swarf generated from the cutting of unprocessed lead rolls, which amounts to approximately 6000kg per year, a relatively small amount. Indeed, the most representative particle size in the swarf appear to be in the 2 - 4 mm range. Although dissolution from particles < 1 mm is higher when standardised to m^2/g, the relatively low number of these in the swarf does not result in significant release of lead. Using the regression presented above (Table 7), calculated dissolution from the total specific surface area of swarf (0.000328 m^2/g) result in dissolution of 3.61 ug/L after 7 days and 10.8 ug/L after 28 days, at 1 mg/L loading. Both values for the whole swarf are lower than that obtained from the T/Dp test based on specific surface area equivalent to a 1 mm sphere. The particle size (as a perfect sphere) that would be needed to derive the same dissolution as lead powder (*d* 0.011 mm, SA 0.05 m^2/g) cannot be demonstrated to appear in the swarf. Furthermore, none of the particle sizes found in the powder can be reliably identified in the cutting swarf.

RAC could conclude that although evidence has been provided by industry that particles < 1 mm are generated from reasonably expected use of massive lead, they are not relevant for the hazard assessment of massive lead.

The particles generated from reasonably expected use of lead are not relevant for classification of massive lead as there is not enough evidence to justify using a representative particle size < 1 mm.

Possible conclusions from option 2

- **Conclusion 3**: Use T/Dp test with default of 1 mm because it is not possible to demonstrate the suitability of powder for hazard assessment of the massive form. Compare classification outcome for massive with that for powder to

determine whether separate classifications for massive and powder lead are warranted.

5.7 Conclusion of RAC

RAC concluded that option 1 above is preferred. Furthermore, as the produced particles < 1 mm fall into the general categories of powders, lead powder is suitable for classifying the massive form and lead should be classified based on data for lead powder, resulting in one entry in Annex VI to CLP.

Despite RAC clearly recommending one outcome, the other options have been retained to clearly explain the other potential outcomes and their consequences (See Annex II). This follows interpretation of Figure 1 for this eventuality, which can be seen in Figure 2.

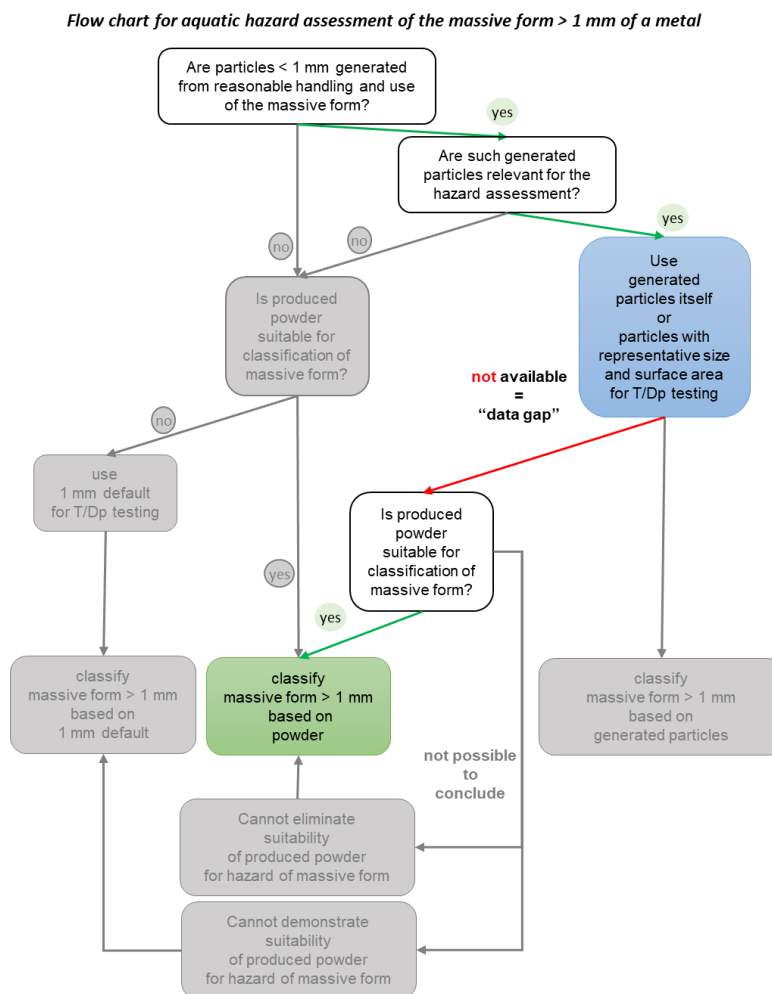*Flow chart for aquatic hazard assessment of the massive form > 1 mm of a metal*

Figure 2: Interpretation of decision scheme for assessing basis for hazard assessment of massive metal where massive should be classified based on powder (deliberately produced metal form < 1 mm).

6   Rapid transformation of lead

The results from the available T/Dp tests demonstrate an increase in dissolved lead concentrations with test duration. RAC received several comments from industry to evaluate and discuss if the concept of rapid removal can and should be applied in the case of lead. During a workshop in 2012 it was discussed if lead (together with e.g., Cu, Ni and Zn) could be one of those metals for which the key question is 'irreversibility' (i.e., binding of metals to oxides or sulphides and natural organic matter forming a non-bioavailable form under a range of environmental conditions) ("group 3 metals") within the concept of rapid removal.

The Rapid Removal Workshop held at ECHA in Helsinki on June 11th, 2019 concluded that "*the fundamental issue of irreversibility has not yet been demonstrated with the method developed so far. Moreover,* […]*, the WS participants (except industry) cannot recommend the concept of rapid removal being applied for chronic environmental hazard classification purposes*" (final workshop report 2019). At the 32nd Meeting of Competent Authorities for REACH and CLP (CARACAL) from 6-7 November 2019 COM and the MSCAs decided that "*The workshop concluded that the model presented was not suitable for hazard assessment and that the concept was also not suitable for use under CLP.*" (document CA/68/2019).

RAC recapitulates the conclusion from the RAC opinion of 2018 and the proposal of the original DS (Denmark) in 2017, that based on T/Dp testing there is no evidence of rapid environmental transformation of lead to non-bioavailable forms.

7   Aquatic bioaccumulation of lead

During this re-evaluation, RAC was made aware of three new studies with BCF values for lead in fish from the open literature. In Filho *et al*. (2017), the fish *Phalloceros caudimaculatus* (Guaru) was used for determination of bioconcentration factor (BCF) of lead. The resulting value of BFC was 1192.3. In Chengxinag Xu *et al*. (2021), the BCF was determined in several fish species living in the cave systems. The concentration of Pb^{2+} the water was rather low, but the results of BCF are high, in all cases more than several thousand. In Milošković (2016), several fish species and several locations were evaluated, the BFC values are in the range 20 to 1080, based on the location. In addition, there were already several BCF values for different fish species available to RAC (Table 9).

Table 9: Measured BCF value (ww) in fish species

Species	BFC	Reference
Phalloceros caudimaculatus (Guaru)	1,192	Filho *et al.* (2017)
Poecilia reticulata (guppy)	1,322	Vighi, M. (1981)
Salvelinus fontanilis (brook trout)	80	Holcombe *et al.* (1976)

In addition, the original CLH report (Table 3.2.4-1) also provided measured BCF values for other freshwater organisms. Within the typical environmental concentration range, the gathered BCFs for molluscs ranged between 180 and 2,500 l/kgww, for insects between 896 and 1,875 l/kgww and for crustaceans between 250 and 1,153 l/kgww.

Overall, there are several different measured BCF values available above the cut-off BCF value of ≥ 500. If reliable high-quality measured BCF values for different fish species are available, generally the highest valid value should be used for the purpose of classification.

RAC recapitulates the conclusion from the RAC opinion of 2018 and the proposal of the original DS (Denmark) in 2017 that at environmentally relevant concentrations lead is bioaccumulative for the purposes of classification.

8 Ecotoxicity of lead

8.1 Impact of water chemistry on lead toxicity and implications for data grouping

Physico-chemical water characteristics, such as dissolved organic carbon (DOC), pH and hardness can potentially modify the toxicity of lead. The VRAR, the SCHER Opinion, the lead EQS dossier of 2011 and the CLH dossier acknowledge the important influence of water chemistry on the toxicological effects of lead in the aquatic environment. Dissolved lead occurs in natural waters in several forms. The most relevant species in terms of toxicity is free Pb^{2+}. Speciation of lead can be estimated by speciation models. RAC has taken into account the dependency on DOC, pH, and hardness of lead speciation.

To date, the relationship between the toxicity of lead and water chemistry has been investigated for only a few aquatic species e.g., *Pimephales promelas* (fish) by Mager *et al*. (2011a), *Lymnaea stagnalis* (invertebrate) by Esbaugh *et al.* (2012) and De Forest *et al*. (2017), *Ceriodaphnia dubia* (invertebrate) by Nys *et al*. (2014), and *Pseudokirchneriella subcapitata* (algae) by De Schamphelaere *et al*. (2014). It is unclear if the effect of water chemistry on the toxicity of lead is valid for all species and across all taxonomic groups.

Section 4.1.3.2.4.3. of the CLP Guidance advises:

> *When larger data sets (four or more values) are available for the same species, the geometric mean of toxicity values may be used as the representative toxicity value for that species. In estimating a mean value, it is not advisable to combine tests of different species within a taxonomic group or in different life stages or tested under different conditions or duration.*

To investigate the potential effects of water chemistry parameters on the toxicity of lead via statistical approaches like Multilinear Regression (MLR) analysis and Species Sensitivity Distribution (SSD) analysis for the whole data set (while taking into account that it has been shown, that different water chemistry parameters effect the toxicity of lead for each species in different ways and to different degrees), RAC proposes to group only those data performed with the same species within a taxonomic group, the same life stage, the same test duration and the same endpoint that have been tested under similar conditions, with regards to DOC, pH and hardness. The approach is presented in Table 10.

Table 10: Grouping based on DOC, pH and hardness used by RAC.

pH	DOC	hardness
-	lowest DOC:< 1	-
low pH: < 6.5	low DOC:>=1 - <2	low hardness: < 50
medium pH: 6.5 – 7.5	medium DOC:>=2 - <3	-
high pH: >7.5	high DOC:>=3	high hardness: > 50

Where four or more aquatic toxicity data points are found for the same species, the same life stage, the same test duration and the same endpoint, the data are grouped, and the geometric mean used as a representative toxicity value for that species.

8.2 Biotic Ligand Model (BLM) for lead

The toxicity of lead to biota is not the same in all freshwater ecosystems due to differences in the chemistry of the water body. The biotic ligand model (BLM) is a computer model that estimates the toxic effects of metals on aquatic freshwater species, such as fish, crustaceans, and algae, vary with changing water conditions.

The lead industry states that the lead 'BLM SSD' normalisation tool is designed for deriving site specific ERVs and may be considered acceptable for use in deriving site-specific Environmental Quality Standard (EQS) values (EQS BLM SCHEER opinion 2017). There is no evidence available to RAC that the Lead BLM SSD normalisation tool is validated for more generic hazard assessment, as is the case under CLP.

In 2018 and again in this review, RAC has assessed the applicability and usefulness of the lead BLM SSD normalisation tool for the purpose of aquatic hazard classification of lead. The lead industry suggests using it to normalise (modify) all the measured ERV values for lead to estimated ERV values that would occur under water quality conditions used in the T/Dp tests for lead. In order to assist with its assessment, RAC sent a number of questions to the lead industry and answers were provided (RAC BLM Questions).

RAC considers that great care needs to be taken to include all valid, reliable, and relevant study results in the database underpinning such tools. RAC notes that the standard (default) raw effect database in the BLM does not include some of the most sensitive effect data (e.g., Munley *et al*., 2013; AquaTox 2012a; Wang *et al*., 2010; Hariharan *et al*., 2016) or that divergent from values selected by the original DS, less sensitive effect data were selected from the same studies (e.g., Esbaugh *et al*., 2012; Besser *et al*., 2005; De Schamphelaere *et al*., 2014; Davies *et al*., 1976; Davies, 1973; and Goettl, 1974). It seems that before introducing this tool into the harmonised classification assessment of lead, the standard (default) raw effect database in the background of the lead BLM SSD normalisation tool would need to be quality checked.

In the view of RAC, it must be ensured that the harmonised classification of lead is based upon the best available science and all valid, reliable, and relevant studies. As presented at the end of this opinion, RAC has invested a high amount of effort to take for each species the most sensitive effect data into consideration for the harmonised classification of lead. Applying the lead BLM SSD normalisation tool with

the current standard (default) raw effect database with values selected by the lead industry itself would mean that sensitive data is lost.

RAC notes that the CLP guidance IV.2.1.1 only refers to the BLM with reference to pH and not to other water quality parameters like DOC and hardness. In the case of lead, several investigations of the available aquatic toxicity data set show that DOC more significantly influences lead toxicity than pH. However, it is important to note that lead is a data rich substance, and that the data set represents an extremely wide range of water chemistry parameters and a combination of these parameters. Consequently, from a scientific (statistical) point of view it is very likely that the ERV values as generated under experimental conditions, without further modification best represent the aquatic hazards posed by lead.

For all these reasons, RAC finds it not to be justified to normalise (modify) all the measured ERV values for lead to estimated ERV values that would occur under the water quality conditions used under T/Dp testing. The water quality conditions under which the original studies were carried out are diverse and the impact of the water quality parameters DOC, pH, and hardness on comparable sets of results are not clear enough to warrant normalisation (see further below), including with the BLM tool.

8.3 Data selection

The dossier of the original DS (Denmark) used the joint REACH registration (ECHA, 2016) and the VRAR (2008) as the primary sources of data. The CLH report presents data from aquatic toxicity tests with 13 (acute) and 38 (chronic) standard and non-standard species. RAC has further included in the data set information from the Registration Dossier and from recent relevant peer-reviewed publications.

As a first step in the data analysis, RAC evaluated which studies are reliable and relevant for deriving the aquatic classification of lead[3].

The dataset comprises standard and non-standard tests and species and RAC looked at their reliability and relevance for the purpose of harmonised classification. RAC paid particular attention to the following aspects:

- test set-up: GLP, guidance, validity criteria, presence of control
- test species: well described (age, gender, appropriate, gender)
- exposure conditions (appropriate spacing between concentrations, proper chemical analysis, exposure duration, temperature, Ph)
- statistics: sufficient number of replicates, concentration-response observed, appropriate statistical method

Studies were rejected if they had:

---

[3] According to the CLP 4.1.1.2.2: "*Preferably data shall be derived using the standardised test methods referred to in Article 8(3). In practice data from other standardised test methods such as national methods shall also be used where they are considered as equivalent. Where valid data are available from non-standard testing and from non-testing methods, these shall be considered in classification provided they fulfil the requirements specified in section 1 of Annex XI to Regulation (EC) No 1907/2006.*"

- an insufficiently described methodology (e.g., number of replicates, number of test concentrations, type of test medium used)
- high control mortality
- endpoints reported with nominal concentrations
- lack of dose-response relationship
- otherwise inadequate test conditions

Non-standard tests have been considered appropriate when exposure conditions can be verified and biological relevance determinable. For example, a test done with a species at a particular life stage different to guideline requirements was accepted if evidence convincingly shows that this particular life stage is relevant/more sensitive, as it is the case with *L. Stagnalis*.

Using MOPS buffer is a common method for pH buffering but seems not to be optimal. Detailed scientific considerations by RAC can be found in the Annex of this opinion. There might be species or taxa for which MOPS can have a toxic effect. During data selection, RAC discussed studies with MOPS at each taxa/species and made a case-by-case decision if the study result is reliable.

At the end of the data selection process, an aquatic toxicity data set for 47 species including marine fish and saltwater invertebrates with a total number of 199 valid data points was taken into account by RAC. Although it is often said that lead is overall a data rich metal, for 30 species only one data point is available. Only for 13 species are 4 or more data points available.

Table 11: List of species with the most data points for the aquatic toxicity of lead. Please note that the number of data points may include studies with e.g., different life stages, test duration and endpoint.

Taxonomic group	Species	Number of data points
Invertebrate	*Ceriodaphnia dubia*	44
Fish	*Pimephales promelas*	22
Invertebrate	*Brachionus calyciflorus*	19
Invertebrate	*Lymnaea stagnalis*	13
Algae	*Pseudokirchneriella subcapitata*	13
Saltwater mollusc	*Mytilus trossolus*	9
Fish	*Oncorhynchus mykiss*	8
Algae	*Lemna minor*	7
Saltwater mollusc	*Mytilus galloprovincialis*	7
Invertebrate	*Philodina rapida*	6
Invertebrate	*Chironomus riparius*	5
Invertebrate	*Daphnia magna*	4
Saltwater echinoderm	*Strongylocentrotus purpuratus*	4

8.4 Comparison of aquatic toxicity data and water solubility data

RAC has assessed the two approaches laid down in section IV.2.3 of the CLP guidance on how to compare aquatic toxicity data and solubility data. In the case of lead, it is well known that several physico-chemical water characteristics, such as dissolved organic carbon (DOC), pH and hardness can influence toxicity. However, it can also be shown that the toxicity of lead in different species is affected by the different water quality parameters (e.g., pH, DOC, hardness) in varying and inconsistent ways. This is why RAC has taken the effect of water chemistry into account when grouping (geometric mean) effect data on the level of a single species. However, the effect has been investigated only for a few species. When investigating the whole available data set, RAC was not able to identify any clear effect of pH on the toxicity of lead.
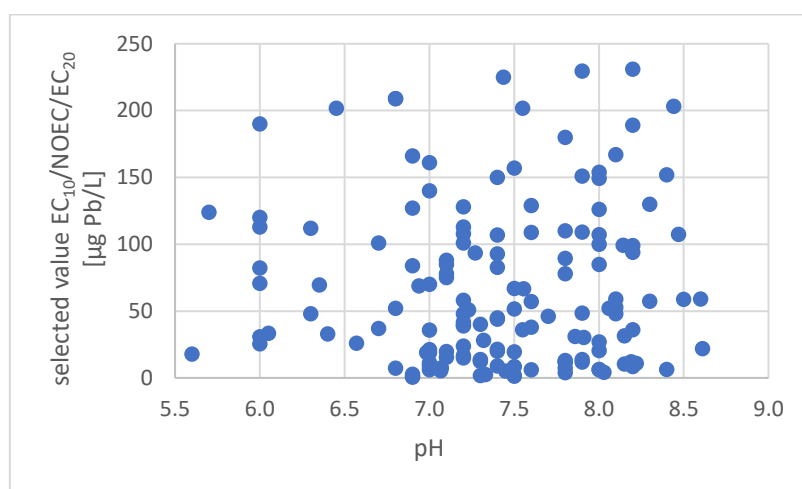

Figure 3: Effect of the pH value on the resulting toxicity of lead. Please note only effect values below 250 µg Pb/L are shown.

Figure 3 demonstrates that no effect of pH can be observed in the whole data set relevant for the classification of lead. The seven most sensitive values result from testing at medium pH between 6.8 and 7.5. This finding by RAC is in line with the finding of LDAI (2010) that "*water hardness and pH have been shown to have a significantly less dramatic effect on Pb availability than DOC.*" And the finding of the EQS dossier (2011) that "*in summary, there is considerable evidence that DOC reduces the chemical availability of Pb but insufficient evidence to propose a biotic ligand-based model that includes other physicochemical variables*."

In the case of lead, RAC was unable to find any scientific evidence that for the purpose of classification banding of the acute and chronic ERVs according to their pH used during T/Dp test is justified.

Consequently, RAC agreed to apply the lowest toxicity data point compared with the highest T/Dp testing result each derived at different pH levels. Following the CLP guidance, as pH has a significant influence on transformation/dissolution of lead, the pH must be selected which maximises the concentration of the dissolved metal ions in solution.

8.5 Multilinear Regression Analysis

RAC conducted a multi-linear regression (MLR) analysis to identify the relative importance of individual water chemistry components, DOC, pH, and hardness in predicting chronic lead toxicity for each species.

Results, also presented in Fig. 4, with normal variables and natural logarithm transformed variables showed that DOC was the only significant variable explaining toxicity. Yet, the model was poor and only explained a small percentage of toxicity across all available species ($R^2 = 0.074$).
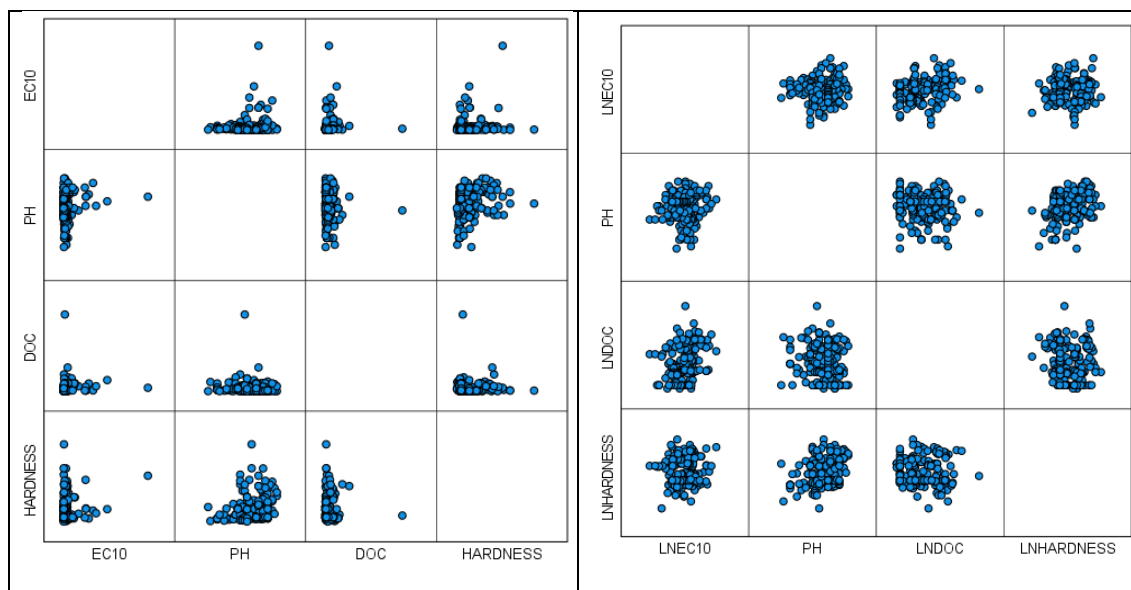

Figure 4: Outcome of the multi-linear regression (MLR) approach with original data (left side) and log transformed data (right side).

This generally poor correlation changes when species are individually analysed. For instance, in the lead WFD EQS dossier (2011) a correlation factor between DOC and the EC_{10}/NOEC of 0.47 was found for 31 data points with *C. Dubia*. In the current data set, the model with non-transformed values results in $R^2 = 0.559$, whereas log transformed variables resulted in a lower $R^2 = 0.25$. In the case of *L. stagnalis*, DOC was the only factor affecting toxicity (see also De Forest *et al.*, 2017) and a correlation $R^2 > 0.7$ was found. Further, for *P. promelas*, all three variables (DOC, hardness, and pH) were found to significantly affect toxicity with the model explaining up to a 67% of the EC_{10}.

Based on the scatter matrix data and the model adjustment, RAC considers that different species are affected by water parameters (e.g., pH, DOC, hardness) in different ways. This is while RAC takes these effects into account when grouping and building geometric means on the species level. However, for the purpose of aquatic hazard classification and evaluating the whole available data set, RAC was only able to find an effect of DOC on the toxicity of lead. This has a direct impact on considerations regarding pH banding for aquatic hazard classification since pH is a water quality parameter which seems to impact the toxicity of lead less significantly compared to DOC. No significant correlation e.g., for pH has been shown when all the available reliable data are considered together.

Given this and taking into account the large available data set with a wide range of different water chemistry parameters, RAC concludes that selecting the lowest endpoint is the most appropriate way to take all available data into account and to classify lead. The main scientific reason is that the lowest ERV available is a surrogate for all the different species at different life stages in all the different natural ecosystems within Europe.

8.6 Species Sensitivity Distribution (SSD) for lead

RAC applied the SSD approach[4] to support the chronic hazard classification of lead. With the ETX software program (version 2.1) by RIVM, several HC_5 values were calculated for lead. This exercise was done to investigate the effect of water chemistry parameters like DOC, pH, and hardness on the toxicity of lead. Saltwater invertebrates were excluded from the SSD approach because they had a negative effect on the quality of the SSD in some cases. Results for each data grouping are presented in Table 12 and may be compared with the HC_5 value based on the whole data set. For each value the number of species involved is given in brackets. Each distribution was tested for statistical criteria on normality.

Table 12: HC_5 values resulting from selecting the lowest value for each species from all available data points or after grouping into DOC, or pH or hardness groups. Saltwater invertebrates are not included.

whole data set		DOC	pH	hardness
	high	0.67 µg Pb/L (n=10)	1.96 µg Pb/L (n=24)	1.15 µg Pb/L (n=17)
1.88 µg Pb/L (n=35)	medium	5.24 µg Pb/L (n=8; not normal distributed)	1.34 µg Pb/L (n=18)	
	low	2.53 µg Pb/L (n=17; not normal distributed)	15.0 µg Pb/L (n=9)	3.09 µg Pb/L (n=22)
	lowest	2.52 µg Pb/L (n=18)		

The highest toxicity compared to the whole data set was observed when grouping for high DOC (> 3), or high hardness (> 50) or medium pH was applied. While the grouping for low pH confirmed that toxicity of lead decreases for low pH, this theoretical effect could not be observed in the highest pH grouping, where toxicity is not maximised. RAC concludes that this is another strong scientific argument against pH banding for the aquatic hazard classification. Instead, compared to the whole data

---

[4] The CLP guidance in section 4.1.3.2.4.3 states: "In case of very large data sets meeting the criteria for applying the Species Sensitivity Distribution (SSD) approach (see IR&CSA, Chapter R.10), statistical techniques (e.g., HC5 derivation) can be considered to estimate the aquatic toxicity reference value for classification (equivalent to using the lowest EC50 or NOEC), in a weight of evidence approach".

set DOC has a significant effect on toxicity reducing the HC$_5$ from 1.88 µg Pb/L to 0.67 µg Pb/L. This value was confirmed after grouping high DOC and medium pH together (n=9), which further reduced the HC$_5$ to 0.487 µg Pb/L, confirming the significant effect of high DOC under a fixed pH regime. This SSD fit fulfils all quality and goodness-of-fit criteria at the best level and can be used without any restrictions.

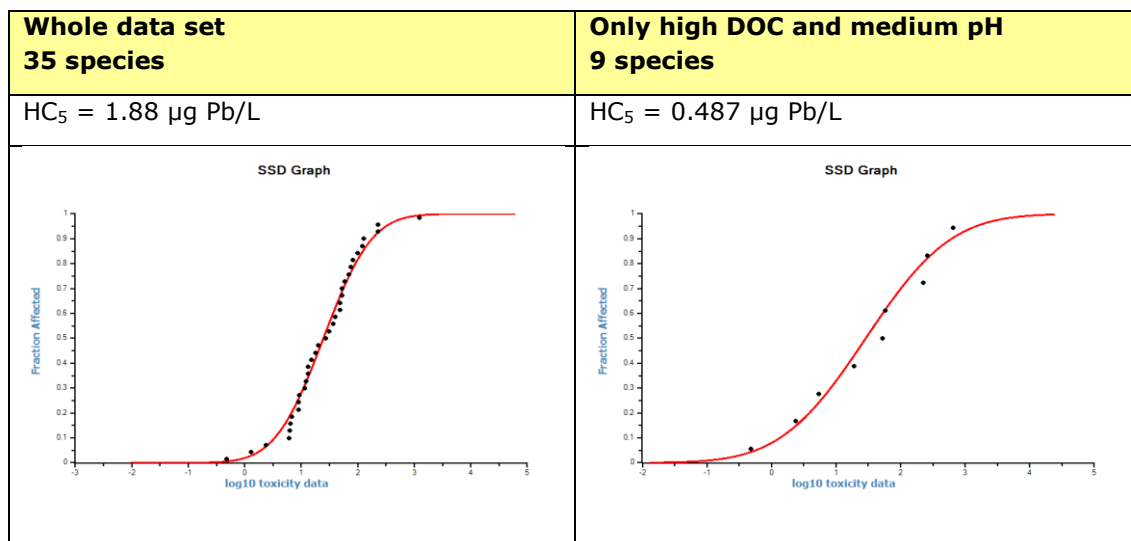| Whole data set 35 species	Only high DOC and medium pH 9 species
HC$_5$ = 1.88 µg Pb/L	HC$_5$ = 0.487 µg Pb/L

Figure 5: Species Sensitivity Distribution Graph for the whole data set (left side) and after grouping for high DOC and medium pH together (right side).

RAC concludes that in the case of lead the available data set is large enough to successfully apply an SSD approach and to estimate the aquatic toxicity reference value for classification (equivalent to using the lowest EC$_{50}$ or NOEC), in a weight of evidence approach. The HC$_5$ value of 0.487 µg Pb/L based on an SSD with 9 species (5 invertebrate, 2 fish and 2 algae species) after grouping for high DOC and medium pH should be used as supporting information for the chronic classification of lead.

8.7 Acute Aquatic toxicity

RAC has not reassessed the available acute aquatic toxicity data set for lead as no new data or comments were provided. Additionally, no further acute toxicity data has been received and no comments have been received on the acute ERV or resulting classification in the 2018 RAC opinion (Aquatic Acute 1, M=1). Therefore, as in 2018 the most sensitive endpoint relevant for the acute classification of lead is the acute ERV of 20.5 µg Pb/L for *P. subcapitata*.

However, in contrast to 2018 measured 7-day T/Dp data at a loading of 1 mg/L became available for particles with a size <75 µm in the form of produced powders at pH 6 and 8 (Brouwers, 2020). Consequently, although the acute ERV remains the same, the resulting classification may alter because of the new T/Dp data when applying the data to the CLP criteria.

8.8 Chronic Aquatic toxicity

8.8.1 Fish

Table 13: Valid chronic ERVs for fish (ELS: Early Life Stage, LC: Life cycle, JG: Juvenile Growth)

Method	Endpoint (EC$_{10}$/NOEC µg/L)	pH	Hardness	DOC (mg/L)	References	Remarks
pH < 6.5						
ELS *Cyprinus carpio*	EC$_{10}$ = 17.5	5,6	100	0.5	Stouthart *et al*. (1994)	CLH report
ELS *Salmo salar*	NOEC = 48	6.3	11	1.6	Grande and Andersen (1983)	CLH report
ELS *Pimephales promelas*	EC$_{10}$ = 33	6.4	26	1.4	Mager *et al*. (2011)	CSR
pH 6.5-7.5						
ELS *Oncorhynchus mykiss*	EC$_{10}$ = 26	6.6	20	1	Mebane *et al*. (2008)	CLH report
ELS *Pimephales promelas*	EC$_{10}$ = 44	7.4	26	1.3	Mager *et al*. (2011)	CSR
ELS *Oncorhynchus mykiss*	EC$_{10}$ = 108	7.2	29	2	Mebane *et al*. 2008	CLH report
ELS *Pimephales promelas*	EC$_{10}$ = 39.6	7.2	47	1.2	Grosell *et al*. (2006)	CLH report
ELS *Pimephales promelas*	EC$_{10}$ = 41.8	7.2	104	1.2	Grosell *et al*. (2006)	CLH report
ELS *Salvelinus namaycush*	EC$_{10}$ = 78	7.2	33	0.5	Sauter *et al*. (1976)	CLH report
ELS *Oncorhynchus mykiss*	EC$_{10}$ = 113	7.2	35	0.5	Sauter *et al*. (1976)	CLH report
LC Yearling, *Salmo fontinalis*	NOEC = 39.4	7.2	44	101	Holcombe *et al*. (1976)	CLH report
ELS *Ictalurus punctatus*	NOEC = 70.5	7.1	36	0.5	Sauter *et al*. (1976)	CLH report
ELS *Lepomis macrochirus*	NOEC = 70	7	41	0.5	Sauter *et al*. (1976)	CLH report
ELS *Oncorhynchus mykiss*	EC$_{10}$ = 18.9	7.0	28	1.6	Davies *et al*. (1976)	CLH report
pH > 7.5						
ELS *Pimephales promelas*	EC$_{10}$ = 99	8.2	24	1.5	Mager *et al*. (2011)	CSR

ELS *Pimephales promelas*	EC_{10} = 149.3	8.0	22	2	Grosell *et al.* (2006)	CLH report
ELS *Pimephales promelas*	EC_{10} = 255.3	7.9	21	2.6	Grosell *et al.* (2006)	CLH report
ELS *Pimephales promelas*	EC_{10} = 448.9	8.0	25	7.3	Grosell *et al.* (2006)	CLH report
ELS *Pimephales promelas*	EC_{10} = 1102.2	7.9	25	10.5	Grosell *et al.* (2006)	CLH report
JG *Oncorhynchus mykiss*	EC_{10} = 55.6	7.9	140	2	Alsop *et al.* (2016)	CSR
ELS *Atherinops affinis* (m)	EC_{10} = 6.8	7.9	105	1.7	Reynolds *et al.* (2018)	CSR
ELS *Oncorhynchus mykiss*	EC_{10} = 18.2	8.0	353	1.6	Davies *et al.* (1976)	CLH report
ELS *Cyprinodon variegatus* (m)	EC_{10} = 229.6	7.9			Parametrix (2010h)	CLH report
JG *Mugil cephalus* (m)	EC_{10} = 11	7.8			Hariharan *et al.* (2016)	CSR
JG *Terapon jarbua* (m)	EC_{10} = 21	7.8			Hariharan *et al.* (2016)	CSR
ELS *Acipenser sinensis*	NOEC = 129	7.6	64	1	Hou *et al.* (2011)	CLH report
JG *Oncorhynchus mykiss*	NOEC = 108.8	7.6	145	0.7	Burden *et al.* (1998)	CLH report
ELS *Pimephales promelas*	EC_{10} = 20	7.5	19	1.2	Grosell *et al.* (2006)	CLH report

For fish, 18 studies are available in the CLH dossier of the original DS, the REACH registration dossier and in relevant publications. These studies reported lead toxicity in 14 species, mainly freshwater species. The main represented species are *Pimephales promelas* (fathead minnow) and *Oncorhynchus mykiss* (rainbow trout) with 5 and 6 studies, respectively. Three species are tested in marine or brackish salinity conditions. Most of them are performed during the early life stage of fish (according or close to OECD TG 210) nevertheless, some of them are performed with fish juvenile growth or during a specific period of the life cycle (Holcombe *et al.*, 1976; Burden *et al.*, 1998; Alsop *et al.*, 2016; Hariharan *et al.*, 2016).

Chronic toxicity studies are assessed according to the general criteria set out for test validity, with three studies rejected. In Wang *et al.* (2012), the data were not thoroughly described to permit a calculation of the EC_{10} or NOEC, only the EC_{20} is shortly reported. In Grosell *et al.* (2006), the buffer MOPS was used to maintain two different conditions. As noted in the CLH report, Esbaugh *et al.* (2013) investigated the effects on toxicity in *P. promelas* of different pH manipulation methods, concluded that the use of MOPS had a significant effect, increasing the toxicity. In their

conclusion, they recommend not to use buffers as they affect ionoregulatory processes. There are, on the other hand, quite some uncertainties connected to the study of Esbaugh *et al*. (2013) with respect to the water chemistry of the different series treated with different kinds of pH regulators, which varied significantly between series. Nevertheless, regarding results in Grosell *et al.* (2006), toxicity observed with MOPS seems to be different to those observed in almost similar conditions without MOPS and since a toxicity of MOPS by itself cannot be excluded, these results are rejected.

Among the valid studies used for RAC's assessment, some of them were not presented in the CLH report. This is the case for Mager *et al.* (2011) where the toxicity of lead was examined for fish and daphnids in waters modified for hardness, DOC, alkalinity and for parameterisation of the freshwater BLM. The exposure of 8-day old fathead minnow larvae was conducted in a flow-through system for 30 days. Harihan *et al.* (2016) conducted acute and chronic bioassay toxicity test of lead in grey mullet (*Mugil cephalus*), and tiger perch (*Terapon jarbua*). Calculated chronic values were 11 µg Pb/L for *M. cephalus* and 21 µg Pb/L for *T. jarbua*. Biochemical and histological alterations were observed during the exposure time. Reynolds *et al.* (2018) is another valid study performed to assess lead toxicity towards marine fish. A 28-day chronic lead toxicity test with *A. affinis* was performed in a flow-through test system with synthetic saltwater using less than 3-day old larvae and 2.5-month-old juveniles, based on the ASTM International 2004 standard guidelines for conducting early life-stage tests. The measured endpoints were survival and growth data (fish length and dry weight). An EC_{10} of 6.81 µg Pb/L is obtained for high salinity waters with the larval dry weight as the endpoint. This EC_{10} is the lowest valid value reported for fish.

Rainbow trout and fathead minnow are the most tested species. For rainbow trout, the EC_{10} or NOEC ranged from 18.9 µg Pb/L to 113 µg Pb/L for pHs from 6.6 to 8.0, Hardness from 20 to 353 mg/L, and DOC from 0.5 to 2.5 mg/L. For fathead minnow, the EC_{10} ranged from 20 to 1102 µg Pb/L for pH from 6.4 to 8.2, hardness from 19 to 104 mg/L, and DOC from 1.2 to 10.5 mg/L. In accordance with section 4.1.3.2.4.3 of the CLP guidance and the guidance for grouping studies, RAC is of the view that a geomean cannot be applied for these species because tests were done under different conditions. Thus, as RAC does not consider that banding for water quality parameters is warranted for lead toxicity, hazard assessment should be based on the data available the for the lowest ERV for fish: EC_{10} = 6.8 µg Pb/L from Reynolds *et al*. (2018).

8.8.2 Invertebrates

<u>*Lymnaea stagnalis*</u>

Table 14: Available studies on the ecotoxic effect of lead on Lymnaea stagnalis. Studies not in bold indicate studies that RAC did not take into account in harmonised classification of lead. The "*" indicates studies with high control mortality (60% was observed at day 28) and the "**" indicates that the growth rate was unaffected in the tested Lead concentration range (3.2, 10, 32, 100 and 320 µg/L).

Age, size, life stage at initiation	Method	Endpoint	EC$_{10}$ (µg/L)	NOEC diss. (µg/L)	pH	Hardness (mg/L)	DOC/TOC (mg/L)	Reference
pH 5.5-6.5								
7-10 d	ELS	14d survival and growth		3.8	5.8	15	0.4	Esbaugh *et al*. (2012)
pH > 6.5-7.5								
7-10 d	ELS	14d survival and growth		5.9	6.8	4.7	1.4	Esbaugh *et al*. (2012)
Hatchlings (< 24-hr)	**FLC**	**56d survival, growth, and reproduction**	**0.48**	**< 1 LOEC=1**	**6.9**	**87**	**4**	**Munley *et al*. (2013)**
7-10 d	ELS	14d survival and growth	15.4	24	7.1	12	6.6	Esbaugh *et al*. (2012)
Hatchlings (< 24-hr)	**ELS**	**30d survival and growth**	**18.4**	**28.9**	**7.2**	**247**	**7.1**	**Parametrix (2007)**
7-10 d	**ELS**	**14d survival and growth**	**93.6**	**234.3**	**7.3**	**23**	**15.8**	**Esbaugh *et al*. (2012)**
Hatchlings (< 24-hr)	**ELS**	**30d survival and growth**	**1.7**	**2.3**	**7.3**	**85**	**< 0.5**	**Parametrix (2007)**
pH > 7.5-8.5								
10-14 d	**ELS**	**16d survival and growth**	**7.4**	**10**	**7.8**	**60**	**2.4**	**Brix *et al*. (2012)**
3 weeks		**14d survival and growth**	**4.0**		**7.8**	**116**	**0.8**	**Crémazy *et al*. (2018)**
Adults (2.5-3.0 cm)	**OECD TG 243**	**28d survival and reproduction**		**110**	**7.8**	**237**	**< 3.2**	**Fox (2020)**
7-10 d	**ELS**	**14d survival and growth**	**4.1**	**10.9**	**8.0**	**72**	**6.3**	**Esbaugh *et al*. (2012)**
3-4 days	**ELS**	**28d Growth (length)**	**11**		**8**	**138.8**	**3.9**	**Nys *et al*. 2013**
3-4 days	**ELS**	**28d Growth (length)**	**73**		**7.55**	**138.8**	**4.8**	**Nys *et al*. 2013**
3-4 days	ELS	28d Growth (length)	-*		7.85	138.8	3.8	Nys *et al*. 2013
3-4 days	ELS	28d Growth (length)	71		8	138.8	3.9	Nys *et al*. 2013
3-4 days	ELS	28d Growth (length)	-*		7.4	138.8	3.7	Nys *et al*. 2013
3-4 days	**ELS**	**28d Growth (length)**	**-****		**7.9**	**138.8**	**3.9**	**Nys *et al*. 2013**
3-4 days	**ELS**	**28d Growth (length)**	**84**		**7.9**	**138.8**	**4.7**	**Nys *et al*. 2013**
Hatchlings (< 24-hr)	ELS	30d survival and growth		12	8.2	102	1.2	Grosell *et al*. (2006)
7-10 d	ELS	14d survival and growth		5.8	8.3	142	1.2	Esbaugh *et al*. (2012)
7-10 d	ELS	14d survival and growth	21.9	46.4	8.6	196	6.9	Esbaugh *et al*. (2012)

For *L. stagnalis*, there are 21 available studies found in the CLH dossier of the original DS, the REACH registration dossier and in relevant publications, of which 12 are considered valid (Table 14 table in bold). According to the general criteria set out for test validity nine studies were rejected (Table 14 not in bold). In Esbaugh *et al.* (2012), the test done at low pH (5.5), was considered stressful for the snails as it presents challenges to shell formation and uptake. In this test, there was 70% survival of control. In the other tests rejected by Esbaugh *et al.* (2012), low control growth was observed, below 4.73mg. Further, in several of these tests the EC_{10} could not be reliably estimated and the NOEC value was higher than 20% of effect. In Grosell *et al.* (2006), control performance was good with survival higher than 80% and a growth above 60mg. However, as growth in the lowest exposure treatment was significantly different than the control, the study resulted in an unbounded NOEC, with the NOEC > 20% of effect. However, the study offers good information on the sensitiveness of this species to lead. Finally, three of the studies by Nys *et al.* (2013) were not considered adequate. In two of them, control mortality was around 60% as indicated in the table. In the other, an EC_{10} = 71 µg/L (13-374). This confidence interval is considered quite wide, also, the fit of the curve to the data was not good, although it improved at the EC_{10}.

Valid tests were done under different water chemistry regimes (pH 6.9-8.6 and DOC range < 0.5-15.8 mg/L) and at different life stages varying from newly hatched snails, to 7-10 days age, up to adults. Test duration also varied from 14 to 56 days. Parameters measured were growth, reproduction, and survival. Most of the tests available were done at an early life stage. Results show that this freshwater pulmonate snail is either the most-sensitive or the second-most-sensitive freshwater organism in chronic exposures to lead. Juvenile snail growth is a particular sensitive life stage.

Test validity was checked against OECD TG 243 principles in the case of Fox (2020). For the early life-stage non-standard tests, no guideline is available to check validity and therefore reliability and relevance was checked against the general principles described before. Of particular interest for classification are the two tests reporting the lowest endpoints: Parametrix (2007) and Munley *et al.* (2013).

RAC notes that the quality of the Parametrix (2007) study with *L. stagnalis* was rated by the REACH registrants as reliable without restriction (as also pointed out by the original DS). It was also used in the SSD to set the PNEC in the joint REACH registration dossier, which indicates the level of hazard for risk assessment purposes. In this test, control performance is appropriate with survival > 80%. A clear dose response curve and a reliable EC_{10} of 1.7 µg Pb/L for the endpoint growth (measured as weight) was reported.

Munley *et al.* (2013) tested the sensitivity of *L. stagnalis* to lead in a 56d full life cycle test evaluating survival, growth, reproductive and embryonic growth endpoints and compared the estimated effect levels to those established using the 28d ELS test design. Snails were exposed to a control and three concentrations, 1.0 ± 0.16, 2.7 ± 0.38, and 8.4 ± 1.05 µg Pb/L (measured). The test included 4 replicates each with 5 snails. The control snails showed a survival rate higher than 80%. The most sensitive results in this study were 28d growth and 56d egg mass production, both with a NOEC of < 1.0 µg Pb/L and a LOEC of 1.0 µg Pb/L, showing that the early life stage growth endpoint is predictive of the 56d reproduction endpoint. This test was considered reliable in the REACH Registration Dossier, RI: 2. Furthermore, RAC

recalculated an EC_{10} = 0.48 µg Pb/L (0.303 - 0.564) for 56 days for the reproduction endpoint.

RAC notes that to provide an estimate of ECx, the primary demand on the study design is to have a sufficient number of concentration groups[5] as the more concentrations tested, the more robust the EC_{10}.

The quality requirement by the guidance is clearly fulfilled and consequently, RAC considers the EC_{10} calculated from the study by Munley *et al*. (2013) to be reliable and appropriate for use in aquatic hazard classification.

A further point analysed in non-standard studies has been the suitability of the type and quality of diet provided to the snails in these tests since the nutritional status may have an impact on lead toxicity and control performance. In the case of *L. stagnalis*, various publications show how snails fed only with lettuce grew less than snails fed with a mix of lettuce and pellets or snails fed just with turtle pellets or fish flakes (Berteloot *et al.,* 2015; Fidder *et al.,* 2017; Reátegui-Zirena and Salice, 2018), although the environmental relevance of snails only (super) fed with high caloric pellets might be questionable since food in nature will vary in quality and quantity.

However, the literature also shows how snails fed with lettuce or a mix of lettuce, potatoes, and carrots have similar growth rates to snails fed with pellets. Brix *et al.* (2011) showed mean ± SEM control weight of 66.9 ± 9.90 mg after 30 days in a test where new hatch snails were fed *ad libitum* a mixture of lettuce and sweet potato during Cu exposure. In Grosell *et al.* (2006), newly hatched snails fed with carrots, sweet potato, and lettuce reached a mean body mass of more than 60mg at the end of the 30d exposure period whereas Berteloot *et al.* (2015) found lower growth rates 50.3 ± 10.6 mg (n=7) after 28 days with newly hatch snails fed with fish flakes.

Data related to the toxicity of chemicals and the diet quality is diverse. Berteloot *et al.* (2015) found lettuce fed *L. stagnalis* to be more sensitive for copper toxicity at size related endpoints than Tetramin fed animals. Fidder *et al.* (2017) found that juveniles and adult *L. stagnalis* reared on a diet of romaine lettuce were more sensitive to pyraclostrobin compared to snails reared on diets with higher overall macronutrient content, *i.e.*: turtle pellets or a combination of both. Contrary to these results, Reategui-Zirena *et al*. (2016), showed that the freshwater snail, *L. stagnalis*, was more sensitive to cadmium when fed a diet higher in nutritional content. In this study it was also found that egg masses from parental snails fed with pellets did not hatch. This data suggests that responses might be contaminant specific.

RAC also notes that OECD TG 243 recommends lettuce as a diet during exposure conditions. This seems to be appropriate for juvenile and adult life stages as can be seen in good control performances. For rearing snails, the OECD TG 243 indicates lettuce as the main food source and gives the option of adding a secondary food source if needed (e.g.: commercial fish flakes). The suitability of lettuce for

---

[5] According to OECD number 54: "concentration-response modelling can only be applied if the data contain sufficient information on the shape of the concentration-response relationship. Although this condition should be judged in each individual situation, experience teaches that at least four different response levels are needed (including the control group) in the case of continuous data".

maintaining a healthy culture is confirmed by the diet used in the studies mentioned above. For example, snail cultures were fed with lettuce in Berteloot *et al.* (2015) and Fidder *et al.* (2017), Reategui-Zirena and Salice (2016), or with a richer nutritional diet based on lettuce and sweet potatoes in Munley *et al.* (2013) or with lettuce, sweet potatoes, and carrots in Parametrix (2007).

WCA environment Ltd. report to ILA from May 2021

ILA requested WCA environment Ltd. (hereafter WCA) to review all available evidence on this theme for several metals and to consider whether there is any evidence of high sensitivity of *L. stagnalis* to trace metals *in-situ* (2105010 ILA Snails Testing report). This study aimed to assess variability in laboratory tests with the pond snail *L. stagnalis*, to understand whether differences between the procedures used could be an important contributor to the differences observed between different studies. WCA is a well-known UK based advisory group on ecotoxicity reviews for the EU water framework activities.

In response to such comments, RAC considered the evidence on the impact of feeding regime on *L. stagnalis* presented in the WCA report from May 2021.

RAC notes that the parameters as recommended in OECD TG 243 reduce the variability during reproduction tests with *L. stagnalis*. However, even when the identical Renilys® strain of snails was used by all laboratories in the international validation studies, EC_{10} values for fecundity endpoints differed by a factor up to 5 among laboratories. This variability is in the same range found in validation studies for OECD reproduction tests with other taxonomic groups (daphnids, chironomids, lumbriculids, fish).

The Detailed Review Paper (DRP) on Molluscs Life-Cycle Toxicity Testing by the OECD (2010) on page 60 states:

> *"It is important to recognize that molluscs are characterised by a high inherent variability for many of the potential endpoints to be considered in […] tests, including reproductive and developmental parameters. […] It is, however, […] not a disturbing factor <u>per se</u> although it clearly affects the statistical power when analysing ecotoxicity data. This characteristic has to be taken into account rather than attempting to reduce the natural variability by choosing test species with less inherent variability […]."*

RAC concludes, that the slightly higher variability in growth parameters in the study of Nys *et al*. (2013) as commented on in the WCA report (May 2021) is not surprising, since the authors compared the responses of snails from three different lab cultures and four field/pond populations, the latter with an unclear exposure and health history.

L. stagnalis is an opportunistic herbivore with low mobility and consequently, an active predation of small fish and amphibians as quoted in the WCA report with reference to Kuroda & Abe (2020) is unlikely and not reported in the literature elsewhere. During the research programme for OECD TG 243 development, a purely plant-based food turned out to be best suited to maintain healthy and self-sustaining cultures of *L. stagnalis*. More protein-rich diets, such as commercial fish flakes, may be added as secondary food sources to accelerate growth if needed. However, there are clear indications that while this can increase growth and reproduction for a short

time, it is by no means sustainable and can lead to rapid metabolic exhaustion and a shortened lifespan of the snails.

RAC questions the statement in the WCA report that "*high protein content of the fish flakes (49% given by manufacturer) appear to make Tetramin fish flakes a better food source for juvenile snails compared to lettuce*" (p. 18; similar on p. 19 for TetraPhyll® as food source). Furthermore, and as pointed out before, RAC see no indication for any "*food limitation* [which] *might exaggerate the response of toxic stress, especially at lower effect levels*" (p. 18) as claimed in the WCA report.

Hardness and temperature

RAC was requested by industry to consider if low hardness or high temperature has a negative effect on *L. stagnalis*.

The recommended range of the OECD TG 243 of 140-250 mg/L as $CaCO_3$ (= 1.4 to 2.5 mM Ca), and preferably close to 250 mg/L as $CaCO_3$. The OECD TG set this value extremely high to support long term cultivation. Although *L. stagnalis* certainly has a high calcium demand, the study by Greenaway (1971) demonstrated that *L. stagnalis* reaches its maximal Ca uptake rate at 0.5 mM Ca (50 mg/L as $CaCO_3$).

The hardness of the test medium in Parametrix (2007) and Munley *et al*. (2013) studies was criticised with only 85 mg/L as $CaCO_3$ (0.5 mM CA) and 70 to 87 mg/L as $CaCO_3$ (0.7 to 0.85 mM CA) to be too low. However, RAC notes that (1) both studies are well above the value from the study by Greenaway (1971), so maximum uptake would be occurring, (2) the high specific growth rates in the control of both studies support that there was no Ca limitation during testing and (3) *L. stagnalis* is found in natural water bodies with much lower Ca values as stable long-term populations.

In addition, it can also be confirmed that a temperature of 25°C is not harmful or stressful for *L. stagnalis*. The Univ. of Miami has continuously maintained *L. stagnalis* cultures at this temperature for 18 years. Obviously, at 25°C snails will have a higher metabolic rate than at 20°C, which will lead to higher metal uptake rates and higher growth rates and this may lead to higher sensitivity to lead. However, this is not a reason to disregard a study and the good control performance indicates that there was no temperature stress during the testing.

Conclusion Lymnaea stagnalis

In Munley *et al*. (2013), the snails displayed less than 5% mortality (vs. up to 20% as validity criterion in OECD TG 243 for only 28 days) and normal growth (2.5g wet wait per snail) in the control group over 56 days. Reproductive output in the control group was also completely normal with a mean of 840 eggs per parent over a period of 24 days. This is equivalent to 7 egg clutches per snail if the upper limit of the clutch size of 50 to 120 eggs in *L. stagnalis* is considered (OECD TG 243, Annex 2, section 4) and thus well above the validity criterion of 4 egg clutches per snail over the 28 days test period.

RAC concludes, that the good control performance in the Munley *et al.* (2013) and Parametrix (2007) tests and in the other tests used for classification indicates that food quality and low hardness of the test media are not reasons to discard these studies, as suggested by industry. In fact, both studies are considered reliable in the REACH Registration Dossier. Furthermore, the *L. stagnalis* used in the tests are

laboratory strains that have been kept under constant conditions and feeding regime' that reproduced and grew well over the years, being fed on lettuce or a mix of lettuce, carrot, and potatoes. Additionally, OECD TG 243 considers lettuce appropriate for rearing snails. Therefore, RAC concludes both studies are valid and relevant for classification. RAC considers the new study by Fox (2020) following OECD TG 243 to be fully valid but notes that it does not provide the most sensitive endpoints and the endpoint value is not the lowest in the dataset (110 µg/L).

Hence, based on the data available the two lowest endpoints for *L. stagnalis* are:

- EC_{10} = 1.7 µg Pb/L by Parametrix (2007)
- EC_{10} = 0.48 µg Pb/L from Munley *et al.* (2013)

In accordance with section 4.1.3.2.4.3 of CLP Guidance and the criteria set for grouping studies across pH, hardness, and DOC bands, RAC is of the view that the geomean cannot be applied for this species because tests were done under different conditions with different life-stages and durations.

Based on the above, RAC concludes that it is appropriate to use for *L. stagnalis* the lowest endpoint for classification purposes: EC_{10} = 0.48 µg Pb/L from Munley *et al.* (2013).

Ceriodaphnia dubia

Table 15: Available (valid) studies on the ecotoxic effect of lead on Ceriodaphnia Dubia.

Age, size, life stage at initiation	Endpoint(s)	EC$_{10}$ (μg/L) from reg. Dossier (reproduction), where the EC$_{10}$ is not available the NOEC is included.	NOEC diss. (μg/L)	pH	Hardness (mg/L)	DOC (mg/L)	Reference
pH < 6.5							
Neonates	7d survival and reproduction	33.3	36.2	6.1	50	<0.5	Parametrix (2010b)
Neonates	7d survival and reproduction	69.5	50	6.4	90	3.3	Nys *et al.* (2014)
pH 6.5-7.5							
Neonates	7d survival and reproduction	68.8	46	6.9	93	3.3	Nys *et al.* (2014)
Neonates	6d survival and reproduction	19	34.5	7.0	13	7.2	AquaTox (2012a)
Neonates	7d survival and reproduction	35.9	19.4	7.0	50	<0.5	Parametrix (2010b)
Neonates	6d survival and reproduction	5.4	40.3	7.1	14	5.9	AquaTox (2012a)
Neonates	7d survival and reproduction		7.6	7.1	16	8.2	Esbaugh *et al.* (2012)
Neonates	7d survival and reproduction	19.8	-	7.1	26	1.2	Mager *et al.* (2011)
Neonates	7d survival and reproduction	85		7.1	97	5.2	Nys *et al.* (2016)
Neonates	7d survival and reproduction	78		7.1	101	6.1	Nys *et al.* (2016)
Neonates	7d survival and reproduction	38.9	-	7.2	35	2.2	Mager *et al.* (2011)
Neonates	7d survival and reproduction	16.6	-	7.2	23	1.2	Mager *et al.* (2011)
Neonates	7d survival and reproduction	128		7.2	205	5.9	Nys *et al.* (2016)
Neonates	7d survival and reproduction	101		7.2	101	5.1	Nys *et al.* (2016)
Neonates	7d survival and reproduction	12.4	-	7.3	168	1.2	Mager *et al.* (2011)
Neonates	7d survival and reproduction	13.8	-	7.3	23	1.2	Mager *et al.* (2011)
Neonates	7d survival and reproduction	354.9	110.6	7.3	28	17.3	Esbaugh *et al.* 2012
Neonates	7d survival and reproduction		150	7.4	29	5.4	Mager *et al.* (2011)
Neonates	7d survival and reproduction	21.4	-	7.4	135	1.2	Mager *et al.* (2011)
Neonates	6d survival and reproduction	5.2	8.0	7.5	78	0.4	AquaTox (2012a)
Neonates	7d survival and reproduction	1.7	2.6	7.5	82	0.5	Cooper *et al.* (2009)
Neonates	7d survival and reproduction		8.4	7.5	354	1.2	Mager *et al.* (2011)
Neonates	7d survival and reproduction	19.6	-	7.5	511	1.2	Mager *et al.* (2011)
Neonates	7d survival and reproduction		51.7	7.5	30	2.5	Mager *et al.* (2011)
pH >7.5							
Neonates	7d survival and reproduction	36	40.9	7.6	44	0.7	Parametrix (2010b)
Neonates	7d survival and reproduction	66.6	44	7.6	89	3.3	Nys *et al.* (2014)

Neonates	7d survival and reproduction		38	7.6	51	1.2	Mager *et al.* (2011)
Neonates	7d survival and reproduction		46.2	7.7	25	1.2	Mager *et al.* (2011)
Neonates	7d survival and reproduction		48.6	7.9	25	1.2	Mager *et al.* (2011)
Neonates	7d survival and reproduction	30.2	47.5	7.9	33	2.7	Esbaugh *et al.* (2012)
Neonates	7d survival and reproduction	20.4	>22.4	8.0	49	0.51	Parametrix (2010b)
Neonates	7d survival and reproduction	270		8.0	162	11.2	Nys *et al.* (2016)
Neonates	7d survival and reproduction	48.1	42.2	8.1	272	<0.5	Parametrix (2010b)
Neonates	7d survival and reproduction	167		8.1	100	5.4	Nys *et al.* (2016)
Neonates	7d survival and reproduction	99.2	164	8.1	90	3.2	Nys *et al.* (2014)
Neonates	7d survival and reproduction	31.4	42.1	8.2	133	<0.5	Parametrix (2010b)
Neonates	7d survival and reproduction		36	8.2	100	6.1	Spehar and Fiandt (1985)
Neonates	7d survival and reproduction	94	-	8.2	26	1.2	Mager *et al.* (2011)
Neonates	7d survival and reproduction	130	128	8.3	231	<0.5	Parametrix (2010b)
Neonates	7d survival and reproduction		57.4	8.3	247	1.7	Esbaugh *et al.* (2012)
Neonates	7d survival and reproduction	107.4	26.7	8.5	224	9.6	Esbaugh *et al.* (2012)
Neonates	7d survival and reproduction	58.8	27.4	8.5	55	<0.5	Parametrix (2010b)
Neonates	7d survival and reproduction	67		7.5	75		Okamoto *et al* (2020)

For *C. dubia* there are 43 valid chronic endpoints found in the CLH Dossier of the original DS, the Registration Dossier and in relevant public literature (i.e.: Nys *et al.,* 2017). All tests were done with neonates and have a duration of 7 days. Test water conditions vary across studies with a pH span from 6.1 to 8.5 and a DOC range from < 0.5 to 17.3 mg/L.

Tests with MOP buffer were rejected since results suggest a potential problem with using this buffer for *C. dubia* bioassays as the daphnids from the control pH 7.2 MOPS tests demonstrated elevated lead sensitivity (Mager *et al.,* 2011). Furthermore, van Sprang *et al.* (2016) also indicated that bioavailability modelling suggests an exacerbation of lead toxicity in the presence of this buffer in the case of *C. dubia* and *P. promelas*. This potential MOPS effect is supported by a previous reports of fathead minnows exposed to lead in MOPS-buffered water adjusted to pH 8.3.

In addition, test media to which Aldrich humic acid was added were not included in the database because a previous study suggests an overestimation of the toxicity due to an underestimation of the lead binding to Aldrich humic acid (van Sprang *et al.,* 2016; Nys *et al.,* 2014).

The lowest long-term EC_{10} and NOEC value identified corresponds to an EC_{10} of 1.7 µg/L. RAC considers the use of the *C. dubia* 7-days test for long-term hazard classification appropriate. This test is clearly a chronic test as three broods are produced within the test duration, which compares to three broods in a 21-day *Daphnia magna* reproduction test. In the CLP Guidance (Annex I.2) it is stated that: "Chronic testing involves an exposure that covers a significant period of time when compared to the organism´s life cycle. The term can signify periods from days to a year, or more depending on the reproductive cycle of the aquatic organism." On this basis, RAC concludes the use of long-term test results from tests using *C. dubia* is justified. This is also consistent with previous cases, such as granulated copper.

In accordance with section 4.1.3.2.4.3 of CLP Guidance and the criteria set for grouping studies across pH, hardness and DOC bands, a geometric mean can be applied for this data rich species. This is the case for all values in the pH range 6.5-7.5 that have a DOC value between 1-2mg/L and a hardness higher than 50mg/L or values within that pH range with a DOC > 3 mg/L that fall within the same hardness range. However, this will not affect the most sensitive endpoint since for a DOC range < 1 mg/L at the medium pH only three studies are available. Based on that RAC concludes that the relevant endpoint to be used for classification purposes for this species is an EC_{10} of 1.7 µg/L.

Other Invertebrates

Besides the data rich invertebrate species *L. stagnalis* and *C. dubia* the whole data set comprises effect data on 11 further freshwater invertebrate species and 12 saltwater invertebrate species. RAC selected the three most sensitive (with the lowest NOEC/EC_{10} value) as these values are generally used for classification.

Table 16: The three the most sensitive (lowest NOEC/EC$_{10}$) effect values for freshwater or saltwater invertebrate species other than the data rich freshwater invertebrate species Lymnaea stagnalis and Ceriodaphnia dubia

Taxa	Species	Endpoint	pH	DOC	Hardness	µg/L		Study
Rotifer	*Philodina rapida*	4d population growth	7.3	3.4	5	2.40	EC$_{10}$	Esbaugh *et al.* (2012)
Amphipoda	*Hyalella azteca*	42d survival, growth, and reproduction	8.4	1.1	138	6.30	NOEC	Besser *et al.* (2005)
Bivalvia	*Lampsilis siliquoidea*	28d survival and growth	8.0	0.5	46	6.40	EC$_{10}$	Wang *et al.* (2010)

8.8.3 Algae and plant species

Table 17: Valid chronic values for algae and plant species

Endpoint	EC$_{10}$ (µg/L)	pH	Hardness (mg/L)	DOC (mg/L)	Remarks	Reference
Chlamydomonas reinhardtii						
3d growth rate	82.3	6.0	24	2.1	Value in CLH report	De Schamphelaere *et al.* (2014)
Chlorella kesslerii						
3d growth rate	120	6.0	24	2.1		De Schamphelaere *et al.* (2014)
2d growth rate	99	6.0	25	2.1	Value in CLH report, validity not met	De Schamphelaere *et al.* (2014)
Lemna minor						
7d root growth rate	655	7.2	56	6.9	UPEN = River	Antunes and Kreager (2014)
7d root growth rate	31	7.9	29	0.7	SANT = River	Antunes and Kreager (2014)
7d root growth rate	152	8.4	266	4.9	TEX = Lake	Antunes and Kreager (2014)
7d root growth rate	59	8.6	172	0.5	CAL = River	Antunes and Kreager (2014)
7d root growth rate	1289	8.4	77	1.4	SOAP = Creek	Antunes and Kreager (2014)
7d root growth rate	116	5.7	8	12.5	PINE = Stream	Antunes and Kreager (2014)
7d root growth rate	52	8.1	33	3.6	McKAY = Creek	Antunes and Kreager (2014)
Dunaliella tertiolecta						
96h growth rate	1232	8.2	30	1.3	In CLH report	Parametrix (2010a)
Phaeodactylum tricornutum						
48h growth rate	100	8.0	33	1.3	1234 µg Pb/L in CLH report, recalculated value used	Beiras *et al.* (2012)
Skeletonema costatum						
96h growth rate	52.9	8.1	38	1.3	In CLH report	Parametrix (2010c)

Champia parvula						
14d growth		9.1		30	MATC value	Steele and Thursby (1983)
48h reproduction	11.9	17.7	7.9	30	In CLH report	AquaTox (2012b)

There are total of 7 valid chronic algae and plant studies available on growth and reproduction endpoints for 8 different fresh and saltwater algae and plant species in the CLH dossier of the original DS, the REACH registration dossier and in other relevant publications.

For the species *C. parvula*, *C. reinhardtii*, *D. tertiolecta*, and *S. costatum* data presented in the CLH report has been used for RAC assessment. For *C. kessleri* an EC_{10} of 99 µ/l was used in the CLH report, but probably not all validity criteria were met during the study. Therefore, RAC used a valid study result for the assessment purposes for this species from the same study. In the CLH report, the EC_{10} of 1234 µg/L for *P. tricornutum* is regarded as unreliable and a recalculated value of 100 µg/L has been presented which RAC considers valid for assessment purposes. For *L. minor*, a lowest value of 19.3 µg Pb/L (dry weight) has been presented in the CLH report from the study of Antunes and Kreager (2014). As the study is considered valid, all calculated IC_{10} values expressed as a function of the total dissolved lead concentration have been used for RAC's assessment.

The above aquatic algae and plant study results cannot be grouped as they were done under very different conditions and do not comply with the developed grouping scenarios. RAC considers it is not possible to determine whether pH, hardness and DOC have effect on lead toxicity to other algae and plant species.

RAC notes that the quality of the De Schamphelaere *et al.* (2014) study investigating chronic exposures to lead with the most sensitive species *P. subcapitata* was rated by the REACH registrants as being of high quality and reliability (as also pointed out by the original DS). The lowest 72h EC_{10} value of 6.1 µg/L value was used in the original CLH report for deriving the chronic ERV. As the study is considered valid, all values from the same study are used for RAC's assessment.

Table 18: Valid chronic values for the green algae P. subcapitata

Endpoint	EC_{10} (µg/L)	pH	Hardness (mg/L)	DOC (mg/L)	MOPS (mmol/L)	MES (mmol/L)	Remarks	Reference
pH > 5,5-6,5								
72h growth inhibition	30.8	6.0	24	2.1	0	10	FA 4 mg/L	De Schamphelaere *et al.* (2014)
72h growth inhibition	70.7	6.0	24	5.3	0	10	FA 10 mg/L	De Schamphelaere *et al.* (2014)
72h growth inhibition	113	6.0	24	10.6	0	10	FA 20 mg/L	De Schamphelaere *et al.* (2014)
72h growth inhibition	25.5	6.0	24	2.1	0	10	-	De Schamphelaere *et al.* (2014)
72h growth inhibition	190	6.0	19	6.5	0	10	Bihain	De Schamphelaere *et al.* (2014)
3d growth rate	32	6.0	24	2.1	0	10	OECD medium	De Schamphelaere *et al.* (2014)
pH > 6.5-7.5								
72h growth inhibition	6.1	7.0	24	2.1	3.6	0	Ca 0.12 mM, Mg 0.12 mM	De Schamphelaere *et al.* (2014)
72h growth inhibition	11.4	7.0	112	2.1	3.6	0	Ca 1 mM, Mg 0.12 mM	De Schamphelaere *et al.* (2014)

Endpoint	EC$_{10}$ (µg/L)	pH	Hardness (mg/L)	DOC (mg/L)	MOPS (mmol/L)	MES (mmol/L)	Remarks	Reference
72h growth inhibition	9.8	7.0	312	2.1	3.6	0	Ca 3 mM, Mg 0.12 mM	De Schamphelaere *et al.* (2014)
72h growth inhibition	6.7	7.0	112	2.1	3.6	0	Ca 0.12 mM, Mg 1 mM	De Schamphelaere *et al.* (2014)
72h growth inhibition	7.4	6.8	24	2.1	3.6	0	-	De Schamphelaere *et al.* (2014)
72h growth inhibition	18.9	7.0	249	22.4	3.6	0	Ankeveen	De Schamphelaere *et al.* (2014)
pH > 7.5-8.5								
72h growth inhibition	6.2	7.6	24	2.1	3.6	0	pH 7.6	De Schamphelaere *et al.* (2014)
72h growth inhibition	6.1	8.0	191	6.5	0	0	Markermeer	De Schamphelaere *et al.* (2014)

De Schamphelaere *et al.* (2014) comprises of 13 test results. At different steps lead toxicity was compared among three algal species, i.e., *P. subcapitata*, *C. kesslerii*, and *C. reinhardtii* in a modified OECD standard test medium at pH 6 with a nominal addition of 4 mg/L of Suwanneer River Fulvic Acid (SRFA), followed by a series of univariate experiments in synthetic media to investigate how modifications of pH, and Ca, Mg and DOC concentrations affected lead toxicity to *P. subcapitata*. Two different types of pH buffers were added: 7 tests were performed with MOPS (3-N-morpholino-propane-sulfonic acid) and 5 tests were performed with MES (2-(N-morpholino)-ethane-sulfonic acid) buffers, in one study no buffer was used.

A bioavailability model was developed as part of the study and validated independently with a field water dataset with four lead spiked surface waters providing aquatic toxicity data of lead to *P. subcapitata*. The bioavailability model was also used to compare the sensitivity of *P. subcapitata* to the reported chronic sensitivity of two chronically lead-sensitive invertebrates (*C. dubia* and *L. stagnalis*). The developed bioavailability model had low mean and maximum prediction error (1.6-fold and 3.7-fold; 85% of the data < 2-fold) and allowed for computing complexation of metal ions with natural organic matter (NOM). The REACH registrants also used the same predicted algal BLM (*P. subcapitata*) to describe chronic lead toxicity to freshwater organisms. It has also been indicated in the REACH registration dossier that a more recent validation study has extended the pH boundary for the *P. subcapitata* BLM to 6.0 – 8.4 (Nys and Schamphelaere, 2017).

De Schamphelaere *et al.* (2014) showed that pH is the main factor affecting green algae toxicity to (filtered) lead, also at constant hardness levels (24 mg/L as indicated by the original DS). When toxicity is expressed on a free Pb^{2+} ion activity basis, a log-linear 260-fold increase of toxicity was observed between pH 6.0 and 7.6. Higher DOC concentrations protected against toxicity of (filtered) lead to *P. subcapitata*, except at the highest pH band where the pH effects were prominent. The hardness parameter seems overall not to affect lead toxicity to green algae due to reduced algal performance in controls at high Ca or Mg levels. There are no indications of a difference between the EC$_{10}$ values of the MES and MOPS tests and the tests where no buffer has been used. FA effects were also considered insignificant when speciation is based on a lead-SRFA binding constant that was recalibrated on the basis of measured speciation, suggesting that the bioavailability of lead-FA complexes (for contributing to toxicity) is limited.

De Schamphelaere *et al.* (2014) concluded that the green algae *P. subcapitata* shows higher chronic sensitivity to lead toxicity than the compared invertebrate species. The model-based comparison with other species indicated that the sensitivity difference between *P. subcapitata* and two chronically lead sensitive invertebrates (*C. dubia* and *L. stagnalis*) is pH dependent, with *P. subcapitata* becoming the most sensitive at pH > 7.4.

RAC concludes that a geomean cannot be applied for this species because tests were done under different conditions across DOC, pH, and hardness. This is in accordance with section 4.1.3.2.4.3 of the CLP Guidance. Additionally, as stated above RAC is of the view that pH banding is not warranted for lead ERV values. For algae, although there is a more pronounced influence of pH than for other species, the differences in DOC across available tests and the influence this can have on toxicity results means that pH banding is further unwarranted. Consequently, the lowest endpoint value for *P. subcapitata*, **EC_{10} = 6.1 μg Pb/L**, from De Schamphelaere *et al*. (2014) should be used as the algal chronic ERV.

9 Comparison with the CLP criteria

Dissolution data for lead at various loadings for comparison with CLP criteria are taken from Table 1. RAC notes that as the proposed classification is based on measured T/Dp data, the proposed use of the Critical Surface Area approach (CSA) for massive lead (Lead metal classification_CSA_Draft Final) is not deemed appropriate.

Furthermore, RAC has concluded that pH banding is not appropriate in the case of lead nor is any correction/normalisation for other water quality parameters (e.g., DOC, hardness). As such, the lowest ERV value will be compared with the T/Dp data at the pH that produces the highest dissolution. RAC has also concluded that lead is both not rapidly transformed to non-bioavailable forms and is bioaccumulative.

RAC concludes that the following ERVs should be used for classification:

- Acute aquatic toxicity: 20.5 μg/L *P. subcapitata*
- Chronic aquatic toxicity: 0.48 μg/L *L. stagnalis*

RAC has also concluded that particles < 1 mm generated from the use of massive lead are relevant and that the powder best represents the massive form of lead for hazard assessment. Consequently, classification is then based on dissolution data from lead powder, resulting in 1 entry in Annex VI to CLP. This approach is known as 'Conclusion 1' (Section III.5.6). The outcomes of alternative approaches from the other potential conclusion options regarding the forms of lead (conclusions 2 and 3, section III.5.6) are presented in Annex II to RACs opinion.

RAC concludes that lead powder is suitable for the classification of massive lead and that T/Dp data for the powder should be used to classify lead. As a consequence:

- Dissolution of lead powder over 7 days at pH 6 and 1mg/L loading (390 μg Pb/L) is larger than the acute ERV (20.5 μg Pb/L) so classification as Aquatic Acute 1 – H400 is warranted.
- The ratio between the acute ERV and the dissolution rate is 19, so an acute M-factor of 10 is warranted
- Dissolution of lead powder over 28 days at pH 5.5 at 0.1 mg/L loading (94.28 μg Pb/L) is larger than the chronic ERV (0.48 μg Pb/L) so classification as Aquatic Chronic 1 – H410 is warranted.

- The ratio between the chronic ERV and T/Dp value over 28 days at pH 5.5 at 1 mg/L loading (942.8 µg Pb/L) is 1964, so a chronic M-factor of 1000 is warranted.

In conclusion, **lead warrants classification as:**

> **Aquatic Acute 1, M=10**
>
> **Aquatic Chronic 1, M=1000**

The other potential outcomes presented in section III.5.6 that RAC did not support are given in Annex II for completeness.

9.1 Clarification on apparent CLP guidance error

RAC notes that the CLP guidance (Annex IV.5.4) appears to contain an error relating to the derivation of chronic M-factors for metals and poorly soluble metal compounds. Whereas the classification stated above in section 9.0 follows the CLP guidance as stated in IV.5.4 (Nov 2021), the error appears to relate to the loading rate used to generate the dissolution data used to derive the chronic M-factor, i.e., the CLP guidance Annex IV.5.4 should indicate the loading rate used to derive a classification as Aquatic Chronic 1 (0.1 mg/L or 0.01 mg/L for not rapidly and rapidly transformed metals or poorly soluble metal compounds, repectively), rather than 1 mg/L. In the case of lead, this would mean a loading rate of 0.1 mg/L (for a non-rapidly transformed metal). As such, the chronic M-factor would be derived:

Dissolution at 0.1 mg/L = 94.28 µg Pb/L, with a chronic ERV of 0.48 µg Pb/L.

Therefore, 94.28/0.48 = 196.4

As the ratio is between 100 and 1000, an M-factor of 100 would be warranted.

Consequently, following this method to derive a chronic M-factor, **lead warrants classification as:**

> **Aquatic Acute 1, M=10**
>
> **Aquatic Chronic 1, M=100**

10 References

Antunes and Kreager (2014) Lead toxicity to *Lemna minor* predicted using a metal speciation chemistry approach. Environ Toxicol Chem. Oct;33(10):2225-33. doi: 10.1002/etc.2688. Epub 2014 Aug 28. PMID: 25044009

AquaTox (2012a) Report on the toxicity of lead to the freshwater invertebrate, *Ceriodaphnia dubia*. Prepared for the International Lead Zinc Research Organization

AquaTox (2012b) Report on the toxicity of lead to the marine macroalgae, *Champia Parvula*. Prepared for International Lead Zinc Research Organization

Appendix 3-BLM British Lead Particle Size Distribution (003)

Beiras, Trigo, Durán, and Tato (2012) Toxicity of Pb to early life stages of the bivalve *Mytilus galloprovincialis* and to the microalgae *Phaeodactylum tricornutum*.

Report Prepared by the University of Vigo for International Lead Zinc Research Organization, Durham, NC, USA

Berteloot, Van Regenmortel, Janssen, and De Schamphelaere (2015) Evaluating and improving the ecological relevance of *Lymnaea stagnalis* chronic metal toxicity estimates. Ghent University, Environmental Toxicology. Submitted to the International Copper Association.

Brix, Esbaugh, Munley, and Grosell (2012) Investigations into the mechanism of lead toxicity to the freshwater pulmonate snail, *Lymnaea stagnalis*. Aquatic Toxicology 106– 107, 147– 156.

Brix, Esbaugh, Grosell (2011) The toxicity and physiological effects of copper on the freshwater pulmonate snail, *Lymnaea stagnalis*. Comparative Biochemistry and Physiology, Part C 154, 261–267.

Characterisation of Lead sheet Swarf-April 2021

Comment to WCA report to ILA – 2021-05-20

Cooper, Bidwell, and Kumar (2009) Toxicity of copper, lead, and zinc mixtures to *Ceriodaphnia dubia* and *Daphnia carinata*. Ecotoxicology and Environmental Safety 72:1523-1528

Crémazy, Brix, Wood (2018) Chronic Toxicity of Binary Mixtures of Six Metals (Ag, Cd, Cu, Ni, Pb, and Zn) to the Great Pond Snail *Lymnaea stagnalis*. Environ. Sci. Technol. 52, 10, 5979–5988

Davies, Goettl, Sinley, and Smith (1976) Acute and chronic toxicity of lead to rainbow trout *Salmo gairdneri*, in hard and soft water. *WaterRes*.10, 199–206.

De Forest, Santore, Ryan, Church, Chowdhury, and Brix (2017) Development of biotic ligand model-based freshwater aquatic life criteria for lead following US Environmental Protection Agency guidelines. Environmental Toxicology and Chemistry 36:2965-2973

De Schamphelaere, Heijerick, and Janssen (2004) Comparison of the effect of different pH buffering techniques on the toxicity of copper and zinc to *Daphnia magna* and *Pseudokirchneriella subcapitata*. Ecotoxicology 13, 697–705.

De Schamphelaere, Nys, and Janssen (2014) Toxicity of lead (Pb) to freshwater green algae: development and validation of a bioavailability model and inter-species sensitivity comparison. Aquat Toxicol. Oct;155:348-59. doi: 10.1016/j.aquatox.2014.07.008. Epub 2014 Jul 15. PMID: 25089923

doc 4800a1_II

ECBI/48/00 add 1

ILA Feb 2021:

- A. ECB-61-95 add 51
- B. ECB-TM-9(97)
- C. ECB-61-95 Add 46
- D. ECB-61-95 Add 50
- E. ENV-JM-MONO-(2001)-9 OECD 29
- F. ENV Classification of Zinc in massive and powder generic classification

- G. Environment classification of Nickel - Draft November 2002 – Danish EPA
- H. ENV_JM_HCL_2004_4
- Historic perspective on Env classification of metals in massive form_final
- J. Overview of metal environmental classification entries including some history

EQS BLM SCHEER opinion 2017

Esbaugh, Brix, Mager, De Schamphelaere, and Grosell (2012) Multi-linear regression analysis, preliminary biotic ligand modeling, and cross species comparison of the effects of water chemistry on chronic lead toxicity in invertebrates. Comparative Biochemistry and Physiology, Part C 155 423–431.

Esbaugh, Mager, Brix, Santore, and Grosell (2013) Implications of pH manipulation methods for metal toxicity: Not all acidic environments are created equal. *Aquatic Toxicology* 130– 131, 27– 30.

Fidder, Reátegui-Zirena, and Salice (2017) Diet quality affects chemical tolerance in the freshwater snail *Lymnaea stagnalis.* Environmental Toxicology and Chemistry. Volume 37, Issue 4.

First uses of lead metal-2021)

Fox (2020) Lead nitrate: Determination of effects on reproduction to *Lymnaea stagnalis* (OECD TG 243). Scymaris.

Greenaway, (1971) Calcium regulation in the freshwater mollusc, *Lymnaea stagnalis* (L.) (Gastropoda: Pulmonata). I. The effect of internal and external calcium concentration. J. exp. Biol. 54, 199-214

Grosell (2010) Final report for Pb BLM development studies - 24 February 2010. Testing laboratory: University of Miami, USA

Grosell, Gerdes, and Brix (2006). Influence of Ca, humic acid and pH on lead accumulation and toxicity in the fathead minnow during prolonged water borne lead exposure. *Comp.Biochem.Physiol.C:Toxicol.Pharmacol.*143, 473–483.

ILA Feb 2021, K. Uses of Lead in the EU and can powders be generated from the massive

Information request from RAC on manufacture process for lead batteries 25/05/2021

Vuorenmaa and Forsius (2008) Recovery of acidified Finnish lakes: trends, patterns and dependence of catchment characteristics. Hydrology and Earth System Sciences Discussions, European Geosciences Union, 12 (2), pp.465-478

Kuroda and Abe (2020) The pond snail *Lymnaea stagnalis*. EvoDevo 11:24.

Lead EQS Dossier 2011
file:///C:/Users/usuario/Downloads/Lead%20and%20its%20compounds%20EQS%20dossier%202011.pdf

Lead metal classification_CSA_Draft Final

Lead Metal ENV Classification_April 2021

Lead Sheet Process Flow

Mager, Brix, Gerdes, Ryan, and Grosell (2011) Effects of water chemistry on the chronic toxicity of lead to the cladoceran, *Ceriodaphnia dubia*. Ecotoxicology and Environmental Safety74 (2011) 238–243.

Munley, Brix, Panlilio, De forest, and Grosell (2013) Growth inhibition in early life-stage tests predicts full life-cycle toxicity effects of lead in the freshwater pulmonate snail, *Lymnaea stagnalis*. Aquatic Toxicology 128– 129 60– 66.

Nys, Janssen, Mager, Esbaugh, Brix, Grosell, Stubblefield, Holtze, and De Schamphelaere (2014) Development and validation of a biotic ligand model for predicting chronic toxicity of lead to *Ceriodaphnia dubia*. Environmental Toxicology and Chemistry 33:394-403

Nys, Janssen, and De Schamphelaere (2016) Development and validation of a metal mixture bioavailability model (MMBM) to predict chronic toxicity of Ni-Zn-Pb mixtures to *Ceriodaphnia dubia*. Environmental Pollution 220:1271-1281

Nys, Janssen, De Schamphelaere (2013) A comparison of chronic Pb sensitivity A comparison of chronic Pb sensitivity A comparison of chronic Pb sensitivity between laboratory and field populations of *Lymnaea stagnalis*. Laboratory of aquatic toxicologyt and aquatic ecology (Ghent University). Unpublished Report.

Okamoto, Masunaga, and Tatarazaco (2020) Chronic toxicity of 50 metals to *Ceriodaphnia dubia*. Journal of Applied Toxicology, doi: 10.1002/jat.4049

Parametrix (2010a) Toxicity of Lead to the Marine Flagellate, *Dunaliella tertiolecta*. Report Prepared for International Lead Zinc Research Organization, Durham, NC, USA

Parametrix (2010b) Chronic toxicity of lead to the cladoceran, *Ceriodaphnia dubia*, under varying calcium and pH water quality conditions. Prepared for the International Lead Zinc Research Organization. Albany, OR, USA

Parametrix (2010c) Toxicity of Lead to the Marine Diatom, *Skeletonema costatum*. Report Prepared for International Lead Zinc Research Organization, Durham, NC, USA

Parametrix (2007) Lead Development Association International. Evaluation of chronic lead toxicity to the Great Pond Snail, *Lymnaea stagnalis*. Prepared by Parametrix, Albany, Oregon. November 28, 2007.

RAC BLM Questions

REACH – recycling – authorisation – helpdesk ref2304DXG13-0234

Steele and Thursby (1983) "A Toxicity Test Using Life Stages of *Champia parvula* (Rhodophyta)," in Aquatic Toxicology and Hazard Assessment: Sixth Symposium, ed. W. Bishop, R. Cardwell, and B. Heidolph (West Conshohocken, PA: ASTM International, 1983), 73-89

Reátegui-Zirena, Fidder, and Salice (2016) A cost or a benefit? Counterintuitive effects of diet quality and cadmium in *Lymnaea stagnalis*. Ecotoxicology volume 25, pages1771–1781.

Reátegui-Zirena and Salice (2018). Parental Diet Affects Embryogenesis of the Great Pond Snail, *Lymnaea stagnalis* Exposed to Cadmium, Pyraclostrobin and

Tributyltin: Parental effects of diet in *Lymnea stagnalis.* Environmental Toxicology and Chemistry;37(9):2428-2438

SE Doc binder:

- 6195a51r4_ECB_metal_classification
- 8904a8_S_Metal_entries_Annex_I
- ECBI_61_95_Add46
- ECBI_61_95_Add57
- KemI reply to ECHA Jan 2021
- Env-jm-mono(2001)9

Spehar and Fiandt (1985) Acute and chronic effects of water quality criteria based metal mixtures on three aquatic species. EPA/600/3-85/074. Testing laboratory: Environmental research lab. -Duluth, MN. Report no.: PB86-122579

Van Sprang, Nys, Blust, Chowdhury, Gustafsson, Janssen, and De Schamphelaere (2016) The derivation of effects threshold concentrations of lead for European freshwater ecosystems. Environmental Toxicology and Chemistry 35:1310-1320

2105010 ILA Snails Testing report

2012BJ_GrainsSize

2012BJ_Kr_EN

IV   ANNEXES

Annex I to the opinion on the aquatic hazards of lead – Proposed harmonised classification and labelling according to the CLP criteria – CLH Tables

Annex II to the opinion on the aquatic hazards of lead – Alternative outcomes regarding the relevance of particles > 1 mm generated from use of massive lead and the subsequent alternative classification outcomes

V   Appendices

Appendix 1 – Further analysis of swarf generated form cutting lead sheets

Using a regression constructed from experimental T/Dp data of the massive and powder, calculated dissolution from the total specific surface area of swarf (0.000328 m^2/g) result in dissolution of 3.61 ug/L after 7 days and 10.8 ug/L after 28 days, at 1 mg/L loading (regression calculated using m^2/mg to closely follow the loading rate used in the T/Dp test. Elsewhere specific surface area is indicated as m^2/g, the standard unit).

In addition, although particles of lead that would give rise to the same dissolution as lead powder cannot immediately be demonstrated in the swarf, it is unlikely that the lead powder particles (labelled as < 0.075 mm, average particle d 0.030 mm) are perfect spheres with perfect surfaces. This is evidenced by the measured surface of the lead powder (0.05 m^2/g, 2012BJ_Kr_EN) which does not correspond to the calculated specific surface area of spheres of either 0.075- or 0.030 mm diameter (0.007 and 0.018 m^2/g, respectively). Furthermore, it is highly unlikely that the particles in the swarf are spherical in nature. Consequently, the above presented lead dissolution for mathematical spheres is likely to underestimate lead dissolution from these particles but is indicative of relative dissolution across a size range.

Indeed, comparing the measured specific surface area of lead powder and the calculated specific surface area based on 0.03 mm spheres indicates that the measured surface area of the powder is 2.8 times higher than the sphere-based modal indicates. This is likely due to the powder particles not being spherical or having perfect surfaces. The powder also contains particles below 0.03 mm in diameter.

It should also be noted that the T/Dp data for massive lead is not based on data from 1 mm spheres but was derived using a piece of lead secured in a resin block with an exposed area equivalent to that of a 1mm sphere. It may therefore be possible that dissolution kinetics may be rather different with 1 mm spheres. Table 6 shows the specific surface area for the minimum particle sizes in each particle size range below 1 mm in diameter in the swarf (including 1 mm for comparison) and the result of correct each value by the difference between the measured value for powder and the theoretical value for particles of 0.03 mm (mean particle diameter of lead powder). This larger surface area would lead to higher dissolution, including what could potentially occur if 1 mm T/Dp testing was based on 1 mm particles rather than idealised surface area.

Furthermore, the regressions that derived the line equations used to calculate the dissolutions above is only based on two particle sizes and is unlikely to provide an accurate representation at very low surface areas. Using calculated spheres combined with the uncertainty in the regression analysis indicates that the calculated dissolution values may underestimate the dissolution from particles < 1 mm in the swarf, meaning that the swarf particles < 1 mm may present a much higher hazard than calculated dissolution values can demonstrate.

If suitable conditions apply (section III.5.6), the powder may not be suitable for classifying the massive form. In such cases the 'classification of the massive can be based on testing of a more representative particle size or surface area'. In normal circumstances the T/Dp testing for the massive form would been perform with particles that are "*generally generated from the massive metal*".

If this has not been done, it is up to expert judgement if the particles in the T/Dp testing are suitable for classifying the massive form.

It is reasonable to assume that the condition of a 'special process' might need to involve or result in chemical transformation or structure or crystallographic transformation.

The process of atomisation where the massive form of a metal is melted and then disintegrated in a stream of inert gas to droplets that solidify upon subsequent cooling is the most common standard process to produce metal powders. It could be assumed that particles produced in such a standard process may be suitable for the classification of the massive form.

As a reciprocal consideration, the CLP guidance implies that if the powder is structurally not a different material (e.g., both massive and powder are face-centred cubic structures) and/or has not been produced by a special process the powder could be suitable for classifying the massive from. It may be assumed that the process via atomisation where the massive form of a metal is melted and then disintegrated in a stream of gas to droplets that solidify upon subsequent cooling does imply that the produced particles are suitable for the classification of the massive form. The fact that powder is produced from the massive form could be one reason that powder and massive should be classified with the same hazard classification based on powder.

However, it is not considered that the development of the guidance anticipated interpretation of the term 'special process' as meaning anything other than that the powder is produced differently from the massive and that the process may be dedicated to the production of powders. On the other hand, any powder, regardless of the production method might then be produced by a special process and it could be questioned if this is the intention of the CLP guidance, since powder *per se* cannot be produce with the same process as the massive form.