

Committee for Risk Assessment

RAC

Opinion

proposing harmonised classification and labelling
at EU level of

Lead

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

CLH-O-0000001412-86-260/F

Adopted

30 November 2018

OPINION OF THE COMMITTEE FOR RISK ASSESSMENT ON A DOSSIER PROPOSING HARMONISED CLASSIFICATION AND LABELLING AT EU LEVEL

In accordance with Article 37 (4) of Regulation (EC) No 1272/2008, the Classification, Labelling and Packaging (CLP) Regulation, the Committee for Risk Assessment (RAC) has adopted an opinion on the proposal for harmonised classification and labelling (CLH) of:

Chemical name: **Lead**
EC Number: **231-100-4**
CAS Number: **7439-92-1**

The proposal was submitted by **Denmark** and received by RAC on **7 August 2017**.

In this opinion, all classification and labelling elements are given in accordance with the CLP Regulation.

PROCESS FOR ADOPTION OF THE OPINION

Denmark has submitted a CLH dossier containing a proposal together with the justification and background information documented in a CLH report. The CLH report was made publicly available in accordance with the requirements of the CLP Regulation at <http://echa.europa.eu/harmonised-classification-and-labelling-consultation/> on **13 september 2017**. Concerned parties and Member State Competent Authorities (MSCA) were invited to submit comments and contributions by **30 October 2017**.

ADOPTION OF THE OPINION OF RAC

Rapporteur, appointed by RAC: **Marian Rucki**

Co-Rapporteur, appointed by RAC: **Michael Neumann**

The opinion takes into account the comments provided by MSCAs and concerned parties in accordance with Article 37(4) of the CLP Regulation and the comments received are compiled in Annex 2.

The RAC opinion on the proposed harmonised classification and labelling was adopted on **30 November 2018** by **consensus**.

Classification and labelling in accordance with the CLP Regulation (Regulation (EC) 1272/2008)

	Index No	International Chemical Identification	EC No	CAS No	Classification		Labelling			Specific Conc. Limits, M-factors and ATE	Notes
					Hazard Class and Category Code(s)	Hazard statement Code(s)	Pictogram, Signal Word Code(s)	Hazard statement Code(s)	Suppl. Hazard statement Code(s)		
Current Annex VI entry	[1] 082-013-0 0-1 [2] 082-014-0 0-7	[1] lead powder; [particle diameter < 1 mm] [2] lead massive: [particle diameter ≥ 1 mm]	[1,2] 231-10 0-4	[1,2] 7439-92- 1	Repr. 1A Lact.	H360FD H362	GHS08 Dgr	H360FD H362		[1] Repr. 1A; H360D: C ≥ 0,03 %	
Dossier submitters proposal	[1] 082-013-0 0-1 [2] 082-014-0 0-7	[1] lead powder; [particle diameter < 1 mm] [2] lead massive: [particle diameter ≥ 1 mm]	[1,2] 231-10 0-4	[1,2] 7439-92- 1	Retain Repr. 1A Lact. Add Aquatic Acute 1 Aquatic Chronic 1	Retain H360FD H362 Add H400 H410	Retain GHS08 Dgr Add GHS09	Retain H360FD H362 Add H410		Retain [1] Repr. 1A; H360D: C ≥ 0,03 % Add M = 10 M = 10	
RAC opinion	[1] 082-013-0 0-1 [2] 082-014-0 0-7	[1] lead powder; [particle diameter < 1 mm] [2] lead massive: [particle diameter ≥ 1 mm]	[1,2] 231-10 0-4	[1,2] 7439-92- 1	Retain Repr. 1A Lact. Add Aquatic Acute 1 Aquatic Chronic 1	Retain H360FD H362 Add H400 H410	Retain GHS08 Dgr Add GHS09	Retain H360FD H362 Add H410		Retain [1] Repr. 1A; H360D: C ≥ 0,03 % Add M = 1 M = 10	
Resulting Annex VI entry if agreed by COM	[1] 082-013-0 0-1 [2] 082-014-0 0-7	[1] lead powder; [particle diameter < 1 mm] [2] lead massive: [particle diameter ≥ 1 mm]	231-10 0-4	7439-92- 1	Repr. 1A Lact. Aquatic Acute 1 Aquatic Chronic 1	H360FD H362 H400 H410	GHS08 GHS09 Dgr	H360FD H362 H410		[1] Repr. 1A; H360D: C ≥ 0,03 % M = 1 M = 10	

GROUNDS FOR ADOPTION OF THE OPINION

RAC general comment

The current CLH proposal addresses only the environmental classification of lead.

As agreed at RAC 47, the opinion of RAC on the environmental classification of lead as a whole (powder and massive forms) is presented here. The view of a minority of members that a separate classification of lead powder and massive lead was possible is also reflected here. Even though the latter was not ultimately supported by RAC, these alternative classifications are given in the interests of balance (See Appendix 1).

ENVIRONMENTAL HAZARD EVALUATION

RAC evaluation of aquatic hazards (acute and chronic)

Summary of the Dossier Submitter's proposal

The Dossier Submitter (DS) proposed to classify lead (powder and massive forms) as Aquatic Acute 1 - H400 and Aquatic Chronic 1 - H410, both with an M-factor of 10, given that the concentrations in the Transformation/Dissolution protocol tests (T/Dp) reached the levels of the Ecotoxicity Reference Values (ERVs) at the appropriate loadings and with the consideration that there is no evidence of rapid environmental transformation.

Degradation

The substance is a natural element and so is not degradable by definition. It is therefore not relevant to assess degradation rate as is usually done for organic compounds. Furthermore, the results from a full Transformation/Dissolution protocol test (T/Dp) (loading of 1mg/L for 28 days at pH 6) demonstrate an increase in the dissolved metal ion concentrations from 5.1 µg/L (at day 7) to 14.2 µg/L (at day 28). Consequently, the DS concludes that there is no evidence of rapid environmental transformation of lead from soluble to insoluble forms.

Bioaccumulation

There is quite a large database of whole-body bioaccumulation and bioconcentration factors (BCF and BAF respectively). The DS states that variation in BAF and BCF values is very high and that a large part of that variation is due to the fact that there is no correlation between either BAF or BCF and the water concentration (VRAR, 2008). There is also a substantial variation between species, not only in the degree of accumulation, but also in the slope of the relationship. At environmentally relevant concentrations, the degree of bioaccumulation will generally be at the higher area of the range. The DS therefore concludes that lead has a clear potential to bioaccumulate at environmentally relevant concentrations for the purposes of classification and labelling.

Aquatic toxicity

The available database for lead (II) cations is large because several soluble lead compounds (mainly Pb(NO₃)₂ and PbCl₂) have been tested under a wide range of conditions involving a variety of species.

The DS used the joint REACH registration dossier as the primary source of data. Data is also extracted from the VRAR (2008). The CLH report presents data from aquatic toxicity tests with 13 (acute) and 38 (chronic) standard and non-standard species. The DS proposes to use the lowest value for each species by stating that the studies have been performed under very varying conditions (pH, hardness and DOC). In addition, splitting the data into pH bands is not considered appropriate by the DS, as no correlation could be established between toxicity and pH for the two most sensitive species (toxicity vs pH (1) *Lymnaea stagnalis*: $r_s = -0.075$ (2) *Ceriodaphnia dubia*: $r_s = 0.237$).

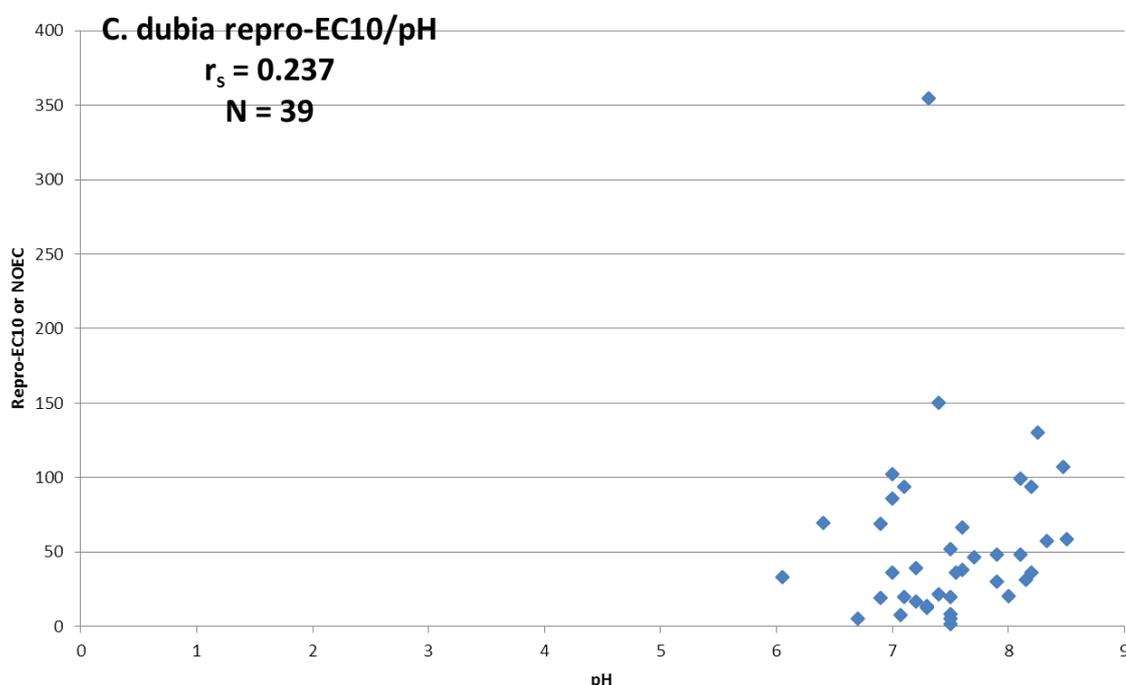


Figure: Correlation between toxicity and pH for *C. dubia*.

Furthermore, the DS is of the opinion that the normalisation techniques (such as the Biotic Ligand Model (BLM)) applied by industry on the data points does not provide any reduction in the variability as the span between the minimum and maximum values is not reduced.

The following description presents the available toxicity information for each trophic level, with the lowest values highlighted in bold.

Acute fish toxicity

Acute data is reported for five fish species, all with standard 96 h LC₅₀ values. The lowest acute fish LC₅₀ is **40.8 µg/L** for *Pimephales promelas* (pH 5.7, H 15.9, 26°C).

Long-term fish toxicity

There is data for ten fish species with a lowest long-term NOEC of **0.9 µg/L** for *Pimephales promelas*. The study (Grosell *et al.*, 2006a) was rated with Reliability 3 in the REACH registration because the buffer MOPS (3-(N-morpholino)propanesulfonic acid) was used. The DS states in the CLH report that by disregarding the value of 0.9 µg/L the lowest long-term fish EC₁₀ is 9 µg/L for *O. mykiss* (Davies *et al.*, 1976). This study is rated as Reliability 2 by the registrant although it does not follow a guideline; it is well described including water chemistry and had a control mortality between 0% and 0.6% (Pb(NO₃)₂; pH 6.7-7.3; H 28, Dissolved Oxygen, 8.3 mg/L, 11.1 °C).

Short-term toxicity to aquatic invertebrates

There are several studies available with the crustacean *Ceriodaphnia dubia* with EC₅₀ values in the range of 26 to 74 µg/L, the lowest reliable EC₅₀ reported in the CLH report being **26 µg/L** from Diamond *et al.* (1997).

Long-term invertebrate toxicity

There is data for several different invertebrate species. The lowest long-term EC₁₀ and NOEC value identified by the DS is **1.7 µg/L** for both the snail *Lymnaea stagnalis* and for the crustacean *Ceriodaphnia dubia*.

Algal/aquatic plants toxicity

The lowest acute and long-term toxicity values reported by the DS are a 72 h EC₅₀ of **20.5** and an EC₁₀ of **6.1 µg/L**, respectively, for the green algae *Pseudokirchneriella subcapitata*. The study (ECHA, 2016) included 13 individual tests with varying pH, hardness and DOC. Two types of buffers were applied, 5 tests with MES (2-(N-morpholino)ethanesulfonic acid) and 8 with MOPS. The analysis performed by the DS does not show any statistically significant difference between the EC₁₀s of the MES and MOPS tests (Mann-Whitney U = 21, P > 0.2, two-tailed) whereas a significant correlation between pH and EC₁₀ when the hardness level is held constant (H = 24 mg/L, Spearman rank r_s = -0.846, n = 8, 0.01 < P < 0,02) is calculated. Thus, the DS concludes that there is no indication of a significant effect related to the choice of buffer.

ERV and M-factor derivation

The CLH report lists the acute and chronic ERVs chosen by the REACH registrant for classification, *i.e.* 73.6 and 17.8 µg dissolved Pb/L (studies conducted at the acidic pH of 5.5 – 6.5), respectively, as well as the acute and chronic ERVs, *i.e.* 20.5 and 6.1¹ µg dissolved Pb/L (studies conducted at the alkaline pH band of 7.5 – 8.5) selected for deriving the M-factors. The lowest acute and chronic ecotoxicity reference values (ERVs) selected by the DS are summarised in the following table. Following the methodology applied by the DS, these values represent the lowest available results from relevant acute and chronic aquatic toxicity studies.

Table: ERVs used by the Dossier Submitter (µg/L)

Acute ERV	EC₅₀	20.5 (<i>P. subcapitata</i> , lowest value; growth rate)
Chronic ERV	EC₁₀/NOEC	1.7 (<i>L. stagnalis</i> , lowest value; growth and <i>C. dubia</i> , lowest value; reproduction)

T/Dp data

Based on the CLP Guidance (Guidance on the Application of the CLP criteria, version 5.0, 2017), the classification of metals is based on a comparison of acute and chronic ERVs (derived from

¹ In the CLH report the DS refers to the lowest EC₁₀ or NOEC of 1.7 µg/L for both *L. stagnalis* and *C. dubia*. While these two studies are indeed reported in the REACH registration dossier, the chronic ERV chosen by the registrant for hazard classification purposes is the EC₁₀ of 6.1 µg/L conducted with *P. subcapitata* (growth rate).

soluble metal species) with the concentration of metal ions in solution after a period of 7 days (short-term test) and 28 days (long-term test), respectively, at different loadings following the T/D protocol. Several studies with different particle size of lead (75 µm and 1 mm) are available.

For lead with a particle diameter of 1 mm, the CLH report lists results of **5.1 µg Pb/L** and **14.2 µg Pb/L** from 7 and 28 day full T/Dp tests at pH 6 and at a loading of 1 mg/L.

For lead with a particle diameter of 75 µm, only the result from a 24 hour screening T/Dp test at pH 6 and a loading of 100 mg/L is included in the CLH report, indicating a dissolved Pb ion concentration of **3211 µg/L**. The DS emphasised that 24 hour screening T/Dp tests are normally conducted for metal compounds only (CLP guidance, Annex IV.2.2.2). However, the REACH registration does not include 7 and 28 days full T/Dp tests using lead with a particle diameter of 75 µm. Based on this supposed lack of information, the DS furthermore assumed that a 100 times decrease in loading will also decrease the concentration of metal in solution a hundred times, hence a 24 hour T/Dp test using lead with a particle diameter of 75 µm at a loading of 1 mg/L would result in a dissolved concentration of 32 µg/L. This concentration (32 µg/L) is expected to increase substantially in a 7 days T/Dp test.

Proposed classification

The DS concludes that it cannot be excluded that under 'reasonably expected use' the massive form of lead could result in lead particles in powder form which can enter the environment (e.g. by casting of bullets and fishing weights in the home or by grinding it into smaller pieces or polishing). Consequently, splitting the classification is not justified for lead and the proposed classification should apply to both forms as they currently appear in annex VI to CLP, massive and powder.

The acute and chronic ERVs are both far below the concentration obtained in the 24 hour screening T/D test and it is unlikely that the concentration achieved in a 28 days full T/Dp test will be lower than the ERV. This conclusion is further supported by the result of the 28 day full T/Dp test with 1 mm particles. This resulted in a concentration at a loading of 1 mg/L (14.2 and 2 µg Pb/L at pH 6 and 7, respectively) that was found to be greater than the chronic ERV selected by the DS (1.7 µg/L for *L. stagnalis* and *C. dubia*).

Based on the above considerations and by concluding that there was no evidence for rapid environmental transformation, the DS proposes to classify lead (powder and massive forms) as **Aquatic Acute 1 - H400** and **Chronic 1 - H410**.

In addition, the DS proposes an **M-factor of 10** for both acute and chronic aquatic hazards following the approach as applied for organic substances, as the M-factor should reflect how much a substance contributes to the toxicity of a mixture.

Comments received during public consultation

Four Member State Competent Authorities (MSCAs) supported the proposal to classify lead as Aquatic Acute 1 and Chronic 1, one MSCA asked for clarification of the impurity content, while another MSCA asked for clarification as to whether the acute and chronic endpoints reported in table 23 of the CLH report (at least the key ones used for classification) are based on mean measured test concentrations, and asked for clarification that the acute and chronic endpoints for algae (at least the key ones in relation to classification) are based on growth rate. All other comments were submitted by industry and individual. The comments cover a range of issues, which can be summarised as follows:

- a) *Concerns regarding the classification of alloys, mainly aluminium ones. Comments on the melting process of scrap and the manufacturing of recycled aluminium alloys, does not produce particles. Information on the solubility of alloys (test with Al-alloy and massive*

lead, lower migration from alloy), the elimination of lead from the aluminium recycling stream is not yet technically practicable.

RAC notes that it is possible to test Al-alloys according to the T/D protocol in order to assess their potential classification. Furthermore, alloys need not be labelled if they do not pose a risk to human health or the environment.

- b) *Concerns regarding one single classification proposal for lead, industry proposes not to classify the massive form for acute and chronic environmental hazards.*

Lead has two separate entries (one for the massive and one for the powder form; distinguished by a particle diameter of 1 mm) in Annex VI of the CLP Regulation for health hazards with the only difference that an SCL was specified for the powder form. This is addressed at the end of this opinion.

- c) *Concerning the use of the C. dubia 7-days test for long-term hazard classification.*

This test is clearly a chronic test as three broods are produced within the test duration, which compares to three broods in a 21 days *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.

- d) One MSCA questioned whether nano-scale forms of lead (i.e. nanoparticles and nanoforms) currently being manufactured are also covered by the proposed environmental classification. In his response, the DS clarified that the proposal does not cover nanoforms of lead. RAC has included an explicit statement in the opinion that nanoforms should be considered separately.

- e) *Preference to use data from "standard test species".*

RAC is of the opinion that, in principle, it is preferable to base classification decisions on data from standard test guideline studies, since these methods have been ring-tested and approved for regulatory purposes. However, where valid data are available from non-standard species and from non-standard 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. In general, both freshwater and marine species toxicity data are considered suitable for use in classification provided the test methods used are equivalent.

- f) *Concerning the use of the Lymnaea stagnalis study (growth rate, chronic ERV).*

According to the CLP Guidance (Annex IV.4.1.3.1.2): "*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*". RAC notes that the quality of the Parametrix (2007) study with *Lymnaea* sp. was rated by the REACH registrants as reliable without restriction (as also pointed out by the DS). It has also been used in the species sensitivity distribution used to set the PNEC in the joint REACH registration dossier, which indicates the level of hazard for risk assessment purposes.

- g) *Question regarding the setting of M-factors.*

The comments received suggest to follow the CLP Guidance for metals and poorly soluble metal compounds and not to use alternative ways for deriving of M-factors. However, the DS clarified during RAC discussions that they have applied the CLP Guidance method for calculating the M-factors as well, although this was hampered by lack of the suitable data.

Consequently, extrapolation was necessary to a concentration after 24 hours at a loading of 100 mg/L instead of the concentration after 28 days at a loading of 1 mg/L according to the CLP Guidance (Annex IV.5.4).

In addition, the DS also applied the GHS method given the same result in this specific case. RAC confirms that the approach by the DS likely underestimates the concentration after 28 days.

h) *Supplemental industry comments of the International Lead Association (ILA), submitted in August 22, 2018, on normalising the test results to comparable conditions by two methods, Biotic Ligand Modelling (BLM), and selection of data generated under "similar conditions".*

In response to these supplemental comments, the DS noted that from the lists of chronic ERVs for the two most sensitive species, *C. dubia* and *L. stagnalis*, it is not possible to find, within each pH-band (5.5-6.5, 6.5-7.5, 7.5-8.5), four values that have been derived from studies performed under equal conditions.

From the normalisation results presented (using BLM), the DS concluded that the normalisation generally does not reduce the variability in the data compared to the non-normalised data. There is no general reduction in the span between the minimum and maximum values. In conclusion and as the normalisation procedures do not reduce the overall variability in the differing test conditions, the DS therefore re-enforced their opinion that geometric means cannot be used.

The DS also stated that for the two most sensitive species *C. dubia* and *L. stagnalis*, there is no correlation between toxicity and pH ($r_s = -0.075$ and $r_s = 0.237$ respectively). Therefore, grouping according to pH is not appropriate.

RAC agrees with the DS and considers that splitting the data to reflect pH bands defined for T/Dp testing is not appropriate in this case. RAC also recognises that there are no clear trends in the data driven by the water quality parameters and therefore does not see a need to normalise the data. However, RAC does not agree with the DS on the selection of the lowest value in every case. The latter is addressed in the next section of this opinion.

Assessment and comparison with the classification criteria

Degradation

RAC agrees with the conclusions of the DS that lead is not rapidly transformed by normal environmental processes.

Aquatic Bioaccumulation

According to the CLP Guidance (Annex IV.4), assessing bioconcentration factors for non-essential metals, should preferably be done from BCF studies using environmentally relevant concentrations in the test media (NOEC). The BCF value for fish is the preferred information for classification purposes.

There are several BCF values for different fish species:

Species	Water Pb (µg/L)	BCF (ww)
<i>Poecilia reticulata</i> (guppy)	3.1	1,322
<i>Salvelinus fontinalis</i> (brook trout)	0.9	80
<i>Perca flavescens</i> (yellow perch)	0.5	405

There are three different values available, one above the classification cut-off value for BCF of ≥ 500 and two below. If high-quality BCF values for different fish species are available, generally the highest valid value should be used as the basis for classification.

RAC agrees with the DS that lead can be considered as bioaccumulative under CLP at environmentally relevant concentrations.

Aquatic toxicity

Following the CLP Guidance (Annex IV.2), the classification of metals is based on a comparison of acute and chronic ERVs (derived from soluble metal species) with the concentration of metal ions in solution after a period of 7 days (short-term test) and 28 days (long-term test), respectively, at different loadings and at the pH that maximises the concentration of dissolved metal ions in solution and expresses the highest toxicity.

There is acute and chronic aquatic toxicity data available for all three trophic levels. Based on the information provided in the CLH report, as well as clarifications provided by the DS and industry experts during RAC discussions (see Background document), RAC considers that the following ERVs are most appropriate:

Acute ERV	EC₅₀	20.5 µg Pb/L (<i>P. subcapitata</i> , lowest value; growth rate)
Chronic ERV	EC₁₀	1.7 µg Pb/L (<i>L. stagnalis</i> , lowest value; growth)

The CLP Guidance (Annex IV.4.1.3.2.4.3) allows the calculation of geometric mean values as the representative toxicity value for that species, where four or more data points are available for the same species. In estimating a mean value, the CLP Guidance further specifies that it is not advisable to combine tests of different species within a different taxonomic group or in different life stages or tested under different conditions or duration. In addition, the CLP Guidance (Annex IV.2.3) also allows splitting the acute and chronic ERVs according to the pH used during the T/Dp test in case a more extensive toxicity/dissolution dataset is available. The geometric means split in the pH bands are used in the REACH registration dossier. RAC agrees with the DS that splitting into pH bands is not appropriate, given that no clear trend could be seen in any of the three water quality parameters (pH, DOC, hardness). This was supported by the information in the CLH report and additional clarifications provided by the DS following RAC discussions, demonstrating that for the two most sensitive species, no correlation between toxicity and pH could be established (toxicity vs pH (1) *Lymnaea stagnalis*: $r_s = -0.075$ (2) *Ceriodaphnia dubia*: $r_s = 0.237$).

RAC disagrees with the DS on the selection of the lowest NOEC of 1.7 µg/L for *Ceriodaphnia dubia* (reproduction) for chronic ERV derivation and used the geometric mean instead, finding that it appropriately summed up the available data.

Independent of the above, the highest toxicity of dissolved lead ions was observed in a study conducted with *L. stagnalis* larvae resulting in a chronic EC₁₀ of 1.7 µg/L (measured; growth (wet weight)). This study was conducted according to GLP standards with newly hatched snails under static renewal conditions using standard synthetic freshwater and natural water collected from South Platte river. After 30 days of exposure growth (wet weight) was measured. While there is no

existing standard OECD TG for testing with larvae of this species, the available OECD TG 243¹ on adult *L. stagnalis*, allows the use of individual growth of the reproducing snails and the number of eggs produced per snail as additional test endpoints. Acknowledging that the study was not conducted according to a standardised test guideline, RAC agrees with the DS to consider the study as reliable and valid for classification purposes and to use the EC₁₀ of 1.7 µ/L for chronic ERV derivation. This value was also used for PNEC (Predicted-No-Effect-Concentration) derivation and employed in the Species Sensitivity Distribution (SSD) in the REACH registration dossier by the registrants, which further supports its use in hazard classification.

Solubility of Pb

In accordance with the CLP Guidance (Annex IV.2.2.3), full T/Dp tests (7 days for short-term and 28 days for long-term hazards) should be carried out at the pH that maximises the concentration of dissolved metal ions in solution and that expresses the highest toxicity. Based on the data from these tests, it is possible to generate a concentration of the metal ions in solution after 7 days (short-term test) for each of the three loadings (*i.e.* 1 mg/L as 'low', 10 mg/L as 'medium' and 100 mg/L as 'high loading') used in the tests. If the purpose of the test is to assess the long-term hazard of the substance, then the loadings should be 0.01 mg/L, 0.1 mg/L or 1 mg/L depending on the transformation rate and the duration of the test should be extended to 28 days (long-term test).

For lead, 24 hour T/Dp test data with 75 µm particles at a loading of 100 mg/L and pH 6 are available, resulting in dissolved lead ion concentrations of **3211 µg Pb/L**.

There is also 7 and 28 day full T/Dp test data available with 1 mm particles at a loading of 1 mg/L at pH 6, which resulted in dissolved lead ion concentrations of **5.1 µg/L** and **14.2 µg/L**, respectively.

The T/Dp results indicate different dissolution kinetics for both tested forms of lead which can be attributed to the different particle size tested. RAC acknowledges the differences in the dissolution rate between the tested particles 75µm and 1mm, but shares the opinion of the DS that the solubility may not be considered as an argument in its own right which would justify a split classification. It is furthermore concluded that the CLP Regulation addresses this substance property with regards to the derogation from labelling requirements of massive forms (CLP Regulation, Article 23), acknowledging that larger particles (*i.e.* massive) are less soluble than smaller particles.

The CLP Guidance (Annex IV.5.5²) specifies that "*normally the classification data generated would have used the smallest particle size marketed to determine the extent of transformation. There*

¹ OECD TG 243... this Test Guideline is designed to assess effects of prolonged exposure to chemicals on the reproduction and survival of the hermaphrodite freshwater snail *Lymnaea stagnalis* (the Great Pond Snail).

² CLP Guidance, Annex IV.5.5 (version 5.0, July 2017): Normally, the classification data generated would have used the smallest particle size marketed to determine the extent of transformation. 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.

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)." RAC notes that the massive and the powder forms of lead do not exhibit different crystallographic structures and thus, deviation from the default classification approach according to the CLP Guidance was not considered warranted.

A further exemption from the default approach listed in the CLP Guidance - allowing for a separate classification of the massive form based on testing of a more representative particle size or surface area - refers to the manufacturing of the powder form by a special process and that it is not generally generated from the massive metal. One previous metal case with an existing entry in Annex VI to CLP for which a split classification was considered justified is nickel, taking into consideration that the fine powder is produced by a special process, the so-called Nickel Carbonyl Gas process. In the case of nickel, RAC concludes that a split classification between the two forms seems warranted. However, in the case of lead RAC is of the opinion that it is unclear whether the manufacturing of lead powder constitutes a special process, taking into consideration that for lead and other metals, powder is intentionally and commonly produced by melting and dispersing the molten metal in an air jet-stream to form small particles.

In agreement with the DS view, RAC concludes that evidence was not provided to show that lead massive cannot form fine particles spontaneously which can then enter the environment.

In addition to the conditions specified in the CLP Guidance, the DS refers to previous metal classification cases where a split classification was warranted based on specific physico-chemical properties between the two forms, *i.e.* they are flammable or even explosive. For example, for aluminium powder both existing entries in Annex VI to CLP are classified for physical hazards, while no classification was concluded for the massive form of Aluminium. Similar conditions apply to the entries for zinc powder (pyrophoric and stabilised), where the pyrophoric entry is also classified for physical hazards. RAC is of the opinion, that a split in classifications between the powder and massive form must be justified by a significant difference in physico-chemical properties, which is not the case for lead.

Overall, RAC shares the view of the DS that the conditions of the CLP Guidance for an exemption are not met in the case of lead. RAC therefore proposes to classify lead on the basis of the available T/Dp data with the smallest particle size marketed (*i.e.* 75 µm), comparing that with the acute and chronic ERVs to decide on the appropriate classification.

Acute toxicity

The available 24 hour Screening T/Dp data with 75 µm particles at a loading of 100 mg/L and pH 6 resulted in **3211 µg Pb/L** in solution. According to the CLP Guidance, classification as Aquatic Acute 1 shall apply, if the dissolved metal ion concentration at a loading rate of 1 mg/L after 7 days exceeds the acute ERV. There is no full T/Dp test available for lead powder and as a consequence, the DS assumed that a 100 times decrease in loading will also decrease the concentration of metal in solution a hundred times. Consequently, a 24 hour Screening T/Dp test with the 75 µm particle diameter at a loading of 1 mg/L would result in a dissolved concentration of 32 µg/L. (the concentration at a loading of 1 mg/L is likely to be greater, as the dissolved concentration will reach saturation with increased loading). The DS concluded that this concentration (32 µg/L) would be expected to increase substantially in a 7 day full T/Dp test. RAC agrees with the DS conclusion, noting that the approach is rather conservative and also assumes a linear kinetic model for the dissolution of lead.

Table: Dissolved lead concentration ($\mu\text{g Pb/L}$) achieved in available T/Dp test data

Particle diameter	T/Dp test duration	Result ($\mu\text{g Pb/L}$)
1 mm	7 days T/Dp test with a loading of 1 mg/L (pH 6)	5.1 $\mu\text{g/L}$
75 μm	7 days full T/Dp test with a loading of 1 mg/L	not available
	24 hours Screening T/D test with a loading of 100 mg/L (pH 6)	3211 $\mu\text{g/L}$
	24 hours Screening T/D test with a loading of 1 mg/L (extrapolated)	> 32 $\mu\text{g/L}$

RAC concludes that a 7 day T/Dp test with 75 μm particles is likely to result in a dissolved concentration well above the acute ERV of 20.5 $\mu\text{g Pb/L}$ for *P. subcapitata*, resulting in a classification as **Aquatic Acute 1 – H400**.

The CLP Guidance specifies the setting of M-factors for metals and poorly soluble metal compounds by dividing the concentration achieved in the T/Dp test (7 days for acute and 28 days for chronic with a loading of 1 mg/L) with the appropriate ERV and to set the appropriate M-factor(s) based on the resulting calculated ratio(s). In the DS's opinion, approach is problematic because the M-factor should reflect how much a substance contributes to the toxicity of a mixture. As the T/Dp test has shown that the toxicity of the ion in fact is being expressed, the right thing to do would be to set the M-factor directly in relation to the ERV as for all other kinds of substances. With lead, there is no 7 or 28 days T/Dp data available for the 75 μm particle diameter. Whether the 7 day T/Dp concentration at a loading of 1 mg/L will be more than 10 times greater than the acute ERV is difficult to assess, but is not unlikely and for this reason the DS assigned an acute M-factor of 10. RAC does not support this view and prefers the methodology for metals as specified in the CLP Guidance.

The acute M-factor calculated from the ratio of the soluble metal ion concentrations obtained from the available T/Dp test (in this case > 32 $\mu\text{g/L}$ for an estimated loading of 1 mg/L) and the acute ERV of the dissolved metal ion (20.5 $\mu\text{g Pb/L}$): **> 1.6**. Based on the resulting ratio, the corresponding **M-factor is 1**.

Chronic toxicity

For classification purposes (CLP Guidance, Annex IV.5.2.2.1) the dissolved metal ion concentration obtained from a 28 day T/Dp test in comparison with the chronic ERV should be used to derive the relevant chronic hazard category.

For 75 μm particles, only the result of the 24 hour Screening T/Dp test is available (*i.e.* 3211 $\mu\text{g Pb/L}$). From this result it is difficult to estimate a 28 day T/Dp value. However, during RAC discussions the industry representative confirmed that the 24 hour screening results mean that the 75 μm particle size should effectively be considered equivalent to a soluble lead salt. It is assumed that a 1000 times decrease in loading will also decrease the concentration of metal in solution a thousand times, then a 28 day full T/Dp test at a loading of 0.1 mg Pb/L would result in a dissolved concentration of 3.2 $\mu\text{g/L}$. This concentration (3.2 $\mu\text{g/L}$) would be expected to increase substantially in a 28 days test and is furthermore exceeding the chronic ERV of 1.7 $\mu\text{g Pb/L}$ for *L. stagnalis*. Given there is no evidence of rapid environmental transformation, this results in a classification of lead as Aquatic Chronic 1 – H410.

The corresponding ratio of the soluble metal ion concentrations and chronic ERV is:
ratio (*L. stagnalis*): $>32/1.7 = >19$

From the above calculated ratios with the lowest NOEC the corresponding M-factor for lead is 10.

Resulting classification of lead agreed by RAC:

Acute (short-term) aquatic hazard: **Aquatic Acute 1 - H40), M-factor = 1.**

Long-term aquatic hazard: **Aquatic Chronic 1 - H410, M-factor = 10.**

Although not agreed by RAC, there was considerable discussion whether the intentional production of lead powder is considered as sufficiently special to meet the conditions of the CLP Guidance and the possibility of an alternative classification for 1mm Pb particles. In the interests of balance, this view is presented in Appendix 1.

The opinion does not address nanoforms of lead. These should be considered separately.

Additional references

Mager, E.M., Esbaugh, A.J., Brix, K.V., Ryan, A.C., Grosell, M., 2011. Influences of water chemistry on the acute toxicity of lead to *Pimephales promelas* and *Ceriodaphnia dubia*. *Comparative Biochemistry and Physiology C: Comparative Pharmacology* 153, 82–90.

ANNEXES:

- Annex 1 The Background Document (BD) gives the detailed scientific grounds for the opinion. The BD is based on the CLH report prepared by the Dossier Submitter; the evaluation performed by RAC is contained in 'RAC boxes'.
- Annex 2 Comments received on the CLH report, response to comments provided by the Dossier Submitter and RAC (excluding confidential information).

Appendix 1

Considerations on the classification of lead with a particle diameter ≥ 1 mm

This alternative view was not ultimately supported by RAC and is provided in the interests of balance.

In the view of some RAC members the intentional production of lead powder is considered as sufficiently 'special' to meet the conditions of the CLP Guidance (exemptions). In particular, the production techniques applied for powders by gas atomisation of molten lead using a jet of high temperature gas allows for the conclusion that these are different compared to those applied for the massive form (such as rolling, extrusion, pressing, stamping, etc.). Moreover, due to its malleability, a spontaneous generation of relevant amounts of fine particles from the massive lead and in a timeframe relevant for hazard classification is considered unlikely. As a consequence, the environmental classification of lead should reflect the differences in the dissolution kinetics, as demonstrated in available full 7 and 28 days T/Dp test data performed with 1 mm lead particles.

In addition, considerations were made as regards consistency with existing metal entries, however, the information available to RAC was not considered conclusive in all cases. In some cases the massive and powder forms were assessed separately (*i.e.* separate T/Dp data were looked at), based on which the classification decision was taken – either to apply the same classification (*e.g.* cadmium), due to the high release rates for both forms resulting in a classification in the most severe hazard category or, both forms did not show any relevant release and were thus not classifiable, such as aluminium.

Overall and by taking into consideration the malleable structure of lead, which will not allow 'fines' to be produced when *e.g.* drilling, there is no reason to neglect the T/Dp data for the massive form and as a consequence there is no justification to not split the classification.

The available full T/Dp data performed at pH 6 with lead particles of 1 mm (default diameter value for T/Dp testing of massive metal) at a loading rate of 1 mg/L indicate a dissolved lead ion concentration after a period of 7 days of 5.1 $\mu\text{g Pb/L}$. As this concentration does not exceed the acute ERV of 20.5 $\mu\text{g Pb/L}$, lead particles with a diameter of ≥ 1 mm would not meet the classification criteria for acute aquatic hazard.

Following the CLP Guidance, classification as Aquatic Chronic 1 shall apply, if the dissolved metal ion concentration obtained at a loading rate of 0.1 mg/L is greater than the chronic ERV. In absence of measured T/Dp data at a loading rate of 0.1 mg Pb/L and by assuming a linear relationship between the different loadings, the extrapolated metal ion concentration at a loading rate of 0.1 mg/L after a period of 28 days is 1.42 $\mu\text{g Pb/L}$. This value does not exceed the chronic ERV of 1.7 for *L. stagnalis*, hence a classification as Aquatic Chronic 1 for particles ≥ 1 mm is not warranted. The next step in the classification scheme for metals is to consider if the dissolved metal ion concentration obtained at a loading rate of 1 mg/L is greater than or equal to the chronic ERV. There is data for a 28 day full T/Dp test at pH 6 with 1 mm particles at a loading rate of 1 mg/L, resulting in a dissolved lead ion concentration of 14.2 $\mu\text{g/L}$. This value exceeds the chronic ERV of 1.7 $\mu\text{g/L}$ and there is no evidence of rapid environmental transformation and would have resulted in a classification for lead particles with a diameter of ≥ 1 mm as Aquatic Chronic 2 - H411.