

# Guidance on information requirements and chemical safety assessment

## Chapter R.7a: Endpoint specific guidance

**THIRD DRAFT (of update to generate Version 2.0) FOR  
CARACAL**

**August 2012**



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## Guidance on information requirements and chemical safety assessment

### Chapter R.7a: Endpoint specific guidance

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### European Chemicals Agency

Mailing address: P.O. Box 400, FI-00121 Helsinki, Finland

Visiting address: Annankatu 18, Helsinki, Finland

## Preface

This document describes the information requirements under REACH with regard to substance properties, exposure, uses and risk management measures, and the chemical safety assessment. It is part of a series of guidance documents that are aimed to help all stakeholders with their preparation for fulfilling their obligations under the REACH regulation. These documents cover detailed guidance for a range of essential REACH processes as well as for some specific scientific and/or technical methods that industry or authorities need to make use of under REACH.

The guidance documents were drafted and discussed within the REACH Implementation Projects (RIPs) led by the European Commission services, involving stakeholders from Member States, industry and non-governmental organisations. After acceptance by the Member States Competent Authorities the guidance documents had been handed over to ECHA for publication and further maintenance. Any updates of the guidance are drafted by ECHA and are then subject to a consultation procedure, involving stakeholders from Member States, industry and non-governmental organisations. For details of the consultation procedure, please see:

[http://echa.europa.eu/documents/10162/17203/mb\\_14\\_2011\\_consultation\\_procedure\\_guidance\\_en.pdf](http://echa.europa.eu/documents/10162/17203/mb_14_2011_consultation_procedure_guidance_en.pdf)

The guidance documents can be obtained via the website of the European Chemicals Agency

<http://echa.europa.eu/web/guest/guidance-documents/guidance-on-reach>

Further guidance documents will be published on this website when they are finalised or updated.

This document relates to the REACH Regulation (EC) No 1907/2006 of the European Parliament and of the Council of 18 December 2006.

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1 Corrigendum to 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 (REACH), establishing a European Chemicals Agency, amending Directive 1999/45/EC and repealing Council Regulation (EEC) No 793/93 and Commission Regulation (EC) No 1488/94 as well as Council Directive 76/769/EEC and Commission Directives 91/155/EEC, 93/67/EEC, 93/105/EC and 2000/21/EC (OJ L 396, 30.12.2006); amended by amended by: Council Regulation (EC) No 1354/2007 of 15 November 2007 adapting Regulation (EC) No 1907/2006 of the European Parliament and of the Council on the Registration, Evaluation, Authorisation and Restriction of Chemicals (REACH), by reason of the accession of Bulgaria and Romania, Commission Regulation (EC) No 987/2008 of 8 October 2008 as regards Annexes IV and V; Regulation (EC) No 1272/2008 of the European Parliament and of the Council of 16 December 2008 on classification, labelling and packaging of substances and mixtures; Commission regulation No 453/2010 of 20 May 2010 as regards Annex II; Commission Regulation No 252/2011 of 15 March 2011 as regards Annex I; Commission Regulation No 366/2011 of 14 April as regards Annex XVII (Acrylamide), Commission Regulation No 494/2011 of 20 May 2011, as regards Annex XVII (Cadmium).

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## Convention for citing the REACH and the CLP Regulations

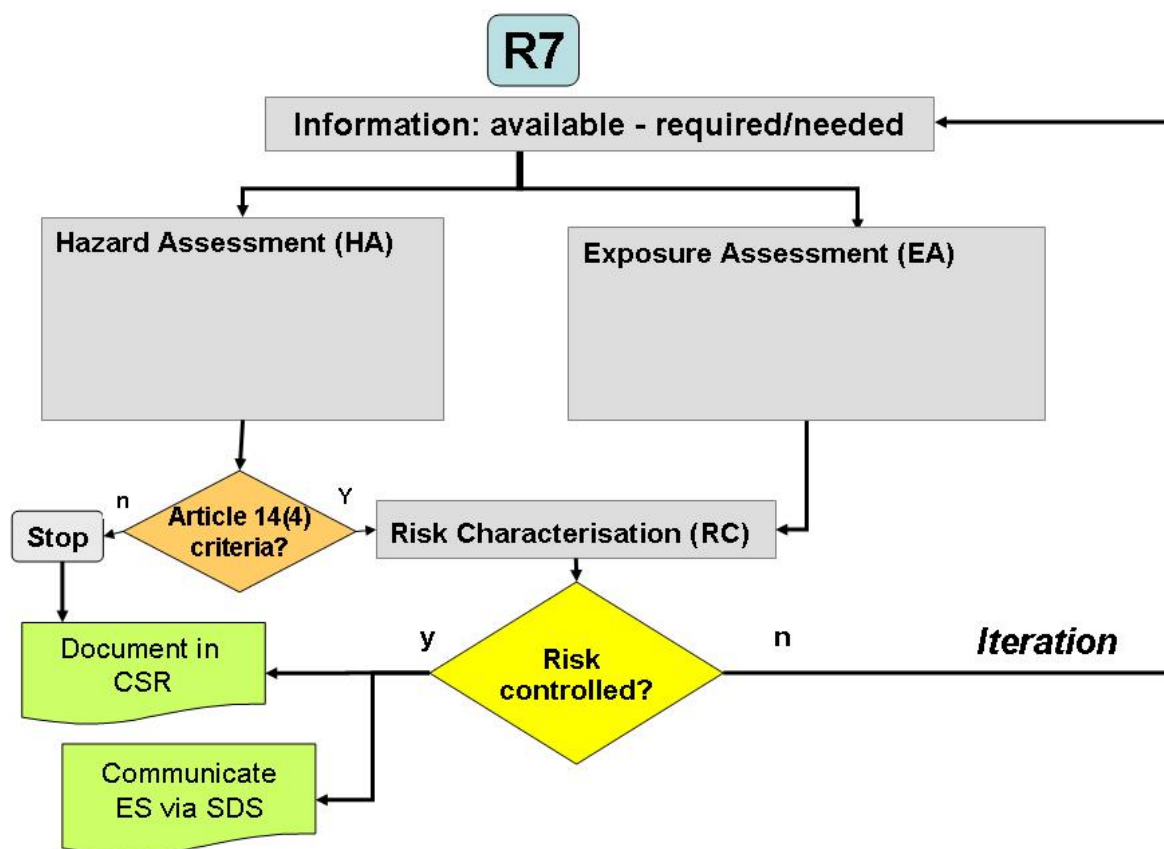
Where the REACH and the CLP Regulations are cited literally, this is indicated by text in italics between quotes.

## Table of Terms and Abbreviations

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## Pathfinder

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## R.7 Endpoint specific guidance

### Introduction

The previous sections of the Guidance on information requirements and chemical safety assessment (IR/CSA) provide advice on the interpretation and application of generic aspects of the Regulation describing the overall process that should be followed in finding, assembling and evaluating all the relevant information that is required for the registration of a chemical under Regulation (EC) No 1907/2006 (the REACH Regulation). The chapters also describe factors that may have an influence on the information requirements and give advice on how the information collected from different sources could be integrated and used in a *weight of evidence* (WoE) approach to allow a conclusion on whether or not the available information is sufficient for regulatory purposes, i.e. hazard assessment and risk assessment.

The guidance given thus far is applicable across the field and comprises the general rules that should be followed.

### Structure of Chapter R.7a

In this chapter, specific guidance on meeting the information requirements set out in Annexes VI to XI to the REACH Regulation is provided. The information requirements relate both to those physicochemical properties that are relevant for exposure and fate considerations as well as to physical hazards, human health hazards and environmental hazards. The guidance for each specified property or hazard has been developed as a specific sub-chapter in this guidance, addressing the aspects of collection, generation and evaluation of information to help registrants provide adequate and relevant information for registration under REACH.

All data sources, including non-testing data, have to be taken into account when doing the chemical safety assessment. Most of the reports follow a logical common format that complements the generic guidance and the general decision making frameworks detailed in first paragraph above. The first chapter, namely the R.7.1 Physicochemical properties, underwent a guidance revision process between 2011 and 2012 and therefore follows a revised chapter structure. The R.7.1 chapter covers both classification and non-classification related properties, where the sub-chapters covering the physicochemical properties have each six or seven sections, depending on the need for information on references and the sub-chapters covering the physical hazards have seven sections. In the physicochemical properties sub-chapters the first section details the type of property, the second section provides the definition of the property, the third lists the preferred test method(s). The fourth section deals with adaptation of the standard testing regime, namely adaptation options that can be explored under each specific physicochemical property. The fifth section deals with impurities and uncertainties and the last section outlines what kind of property-specific information should be given in the registration dossier. Note that sometimes an additional section is added where relevant references are provided. By contrast the physical hazard sub-chapters start with the definition section, followed by a second section on classification criteria and relevant information. The third section explores various adaptation options, namely how the standard testing regime can be adapted. The fourth section outlines the impurities and uncertainties. The fifth section aims to help in concluding on the Directive 67/548/EEC (Dangerous Substances Directive - DSD) classification and the sixth section outlines the physical hazards-specific information to be included in the registration dossier and in IUCLID. The seventh section gives relevant further information and used references.

Chapters tackling human health properties or hazards in R.7a remain unchanged. In those chapters there are six main sections to the guidance on each property or hazard; the introduction section provides an introduction in which the property or hazard is described, further defined and an explanation given as to its importance in the context of human health,

or environmental fate and effect of a given substance. The first section details the specific information requirements for the endpoint of interest; these will depend on the tonnage band of the substance, its usage pattern and other considerations including data on other endpoints and on related substances. Endpoint<sup>2</sup> specific guidance can be thought of as the four logical steps that should be taken to assemble the information that is detailed under the second section; thus, the second section provides an inventory of all the types of data that could potentially provide useful information on the endpoint of interest and, most importantly the sources of that information.

Guidance is given in the third section on how to evaluate the information that might be available for a given substance; this advice focuses on providing the criteria to aid in the judgement and ranking of the available data for their adequacy and completeness. This section also provides an indication of the remaining uncertainty inherent in the different types of data for the given endpoint.

The fourth section describes how conclusions may be drawn for a given substance on the suitability of the available information for regulatory purposes. Guidance is given on how to develop and apply a WoE approach for the endpoint in order to establish whether there is a need for further information and if so, what test should be performed. Chemical safety assessment within REACH is fundamentally dependent on an adequate conclusion on classification and PBT/vPvB assessment since exposure assessment and risk characterisation are triggered by classification and fulfilment of PBT/vPvB criteria. Therefore data need to be adequate for both classification & labelling and for chemical safety assessment if the latter is required.

The fifth section comprises an Integrated Testing Strategy (ITS) for the given endpoint(s), providing guidance on how to define and generate relevant information on substances in order to meet the requirements of REACH. It is noteworthy that all experiments using vertebrate animals shall be designed to avoid distress and unnecessary pain and suffering to experimental animals, in accordance to Article 7.4 of Directive 86/609/EEC.

The proposed testing strategies are guidance for data generation in a stepwise approach. The strategies build on the concept that if the available information is not sufficient to meet the regulatory needs, further gathering of information at a succeeding step in the testing strategies is needed. On the other hand, if the available information is adequate and the standard information requirements are met, no further gathering of information is necessary. In those cases where the available information is judged to be sufficient to meet the regulatory needs even though the standard information requirements are not met, under certain circumstances, in particular for Annexes IX and X to REACH, this might be part of a justification for waiving a certain test that is requested in the standard information requirements.

The final section lists all used references on the given endpoints.

The following additional considerations apply generally to the endpoint specific guidance given in this chapter:

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<sup>2</sup> REACH uses the term “endpoint” both to denote a physicochemical property (example: Annex VII to REACH, Column 1 standard information required: 7.3 Boiling point, and 7.4 Relative density) and to denote hazardous properties (example: Annex VII to REACH, Column 1 standard information required: 7.11 Explosive properties and 7.13 Oxidising properties) which are subject to classification according to the applicable EU legislation. In the following, the wording of Part 7(a) of this guidance document will differentiate between these different types of properties where this appears appropriate, in order to facilitate the identification of properties which serve the regulatory purpose of classification.



## **Information requirements in the light of the applicable classification regime**

The main regulatory purpose of the information requirements set out in Annexes VI to XI to the REACH Regulation is to assess risks related to substances and to develop and recommend appropriate risk management measures, as highlighted in Recital 19 the REACH Regulation. According to Recital 26: *'in order to undertake chemical safety assessment of substances effectively, manufacturer and importers of substances should obtain information on these substances, if necessary by performing new tests'*. The chemical safety assessment (CSA) should be performed in accordance with the provisions set out in Annex I to the REACH Regulation. According to Section 0.6 of Annex I, the first three steps of the CSA require the carrying out of human health hazard assessment, human health hazard assessment of physicochemical properties and environmental hazard assessment, including determining the classification of substances. When the REACH Regulation was adopted, the DSD was the applicable classification regime (see, more in particular, the transitional provisions set out in Article 61 of Regulation (EC) No 1272/2008). Accordingly, many REACH information requirements are inspired by the categories of danger under DSD such as points 7.10, 7.11 and 7.13 in column I of Annex VII to REACH (*i.e* flammability, explosive properties and oxidising properties, respectively).

On 20 January 2009 Regulation (EC) No 1272/2008 (CLP Regulation or CLP) entered into force. The CLP Regulation has amended certain parts of the REACH Regulation (see Article 58 of CLP for amendments applicable from 1<sup>st</sup> December 2010 and Article of 59 CLP for amendments applicable from 1<sup>st</sup> June 2015). Nevertheless, the terminology used in REACH currently still comprises terms which were used under the DSD (for substances) and still apply (for mixtures until 1 June 2015) under Directive 1999/45/EC (Dangerous Preparations Directive - DPD). With respect to the updated physicochemical part of this guidance and the section dealing with the exploration of adaptation possibilities of the standard testing regime, the term 'dangerous' can be interpreted in a broader context (particularly, in certain contexts within this document, to include "hazardous" as defined under CLP) as it does not refer strictly to the DSD.

According to the requirements of Article 10(a)(iv) of the REACH Regulation, the technical dossier required for registration purposes includes the classification and labelling of the substance as specified in section 4 of Annex VI to REACH, resulting from the application of Titles I and II of CLP Regulation. From 1 December 2010 until 1 June 2015 substances must be classified in accordance with both DSD and CLP and they must be labelled and packaged in accordance with CLP (Article 61(3) of CLP). Similarly, until 1 June 2015 Safety Data Sheets (SDSs) must include information on classifications according to both CLP and DSD for substances and component substances in mixtures until 1 June 2015 (see updates to REACH via Commission Regulation (EU) No 453/2010 and the ECHA guidance on the compilation of Safety Data Sheets: [http://echa.europa.eu/documents/10162/13643/sds\\_en.pdf](http://echa.europa.eu/documents/10162/13643/sds_en.pdf)).

## **Use of data derived from EU or other international standardised test methods**

For the purposes of determining whether any of the physical hazards referred to in Part 2 of Annex I of CLP apply to a substance (or a mixture), the manufacturer, importer or downstream user should perform the tests required by the above mentioned Part 2, unless there is adequate and reliable information available (see Article 8(3) of CLP). Further in this guidance for each relevant physical hazard a reference to the corresponding test according to UN Recommendations on the Transport and Dangerous Goods, Manual of Test and Criteria, starting with an UN test method name will be provided.

According to Article 8(5) of CLP, where new tests for physical hazards are carried out for classification and labelling purposes, they must be performed in compliance with a relevant recognised quality system (*e.g.* GLP) or by laboratories complying with a relevant recognised standard (*e.g.* with EN ISO/IEC 17025), at the latest from January 2014.

Further, according to Article 13(3) of REACH, tests for generating information on intrinsic properties of substances should be conducted in accordance with the test methods laid down in Commission Regulation (EC) 440/2008 (Test Method Regulation)<sup>3</sup> or in accordance with other international test methods recognised by the Commission or the Agency as being appropriate, such as European Standards (EN) ([www.cen.eu](http://www.cen.eu)) or the OECD guidelines ([www.oecd.org](http://www.oecd.org)). Regulation (EC) 440/2008 lays down the test methods to be applied for the purposes of REACH. Thus, in the following sections on specific endpoints, references given for each test method will include the OECD Test Guideline (TG) number and, where available, the test method (A) number, as defined in the Test Method Regulation.

According to Recital 37 of the REACH Regulation, if tests are performed, they should comply with the relevant requirements for protection of laboratory animals, as set out in Council Directive 86/609/EEC<sup>4</sup>. Article 13(4) of REACH states that ecotoxicological and toxicological tests and analyses shall be carried out in compliance with the principles of good laboratory practice (GLP) provided for in Directive 2004/10/EC<sup>5</sup> or other international standards recognised as being equivalent by the Commission or the Agency and with the provisions of Council Directive 86/609/EEC, if applicable.

### **Interdependence of endpoints in hazard assessment**

Although guidance is provided for each specific endpoint separately, it should be remembered that different endpoints are related to each other. Information collected within one endpoint may influence hazard/risk assessment of other endpoints, e.g. information on rapid primary degradation of a parent compound may result in including the degradation products in the overall assessment of the toxicity of a substance. Regarding the physicochemical properties of a substance, for example boiling point and flash point are properties used for the classification of flammable liquids, and therefore these properties are important for physical hazard assessment. Similarly, information on toxicity/specific mode of action in one endpoint may indicate possible adverse effects for organisms considered for assessment of other endpoints, e.g. endocrine disrupting mode of action in mammals may indicate the same mode of action in fish. Another example may be when data on toxic effects measured in one group of organisms may be directly used in more than one endpoint, e.g. data from a repeated dose toxicity study may also be used in assessment of risk for secondary poisoning of mammals exposed via the food chains.

### **Adequacy of methods for generating additional information**

Before proposing additional animal testing, use of alternative methods and all other options must be considered. It is important to emphasise that testing on vertebrate animals should only be conducted or proposed as a last resort, when all other data sources have been exhausted (see, Recital 47 of the REACH Regulation, Article 25 of REACH and Step 4 of Annex VI to REACH). Therefore, it is important to first consider all issues that may impact upon this decision to perform the testing, such as:

- testing requirements;
- exposure/use pattern (emissions, yes or no, consumer use etc);

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<sup>3</sup> Council Regulation (EC) No 440/2008 laying down test methods pursuant to Regulation (EC) No 1907/2006 of the European Parliament and of the Council concerning the Registration, Evaluation, Authorisation and Restriction of Chemicals (REACH) [OJ L 142, 31.5.2008, p. 1].

<sup>4</sup> COUNCIL DIRECTIVE of 24 November 1986 on the approximation of laws, regulations and administrative provisions of the Member States regarding the protection of animals used for experimental and other scientific purposes (86/609/EEC).

<sup>5</sup> Directive 2004/10/EC of the European Parliament and of the Council of 11 February 2004 on the harmonisation of laws, regulations and administrative provisions relating to the application of the principles of good laboratory practice and the verification of their applications for tests on chemical substances.



- occurrence (monitoring data);
- indications of the effect/ property based on animal or human data, *in vitro* data and non-testing information;
- any concern e.g. based on toxicokinetics, read-across and (Q)SAR considerations,
- weight of evidence;
- seriousness of the effect;
- other effects of relevance for the endpoint.

All these issues should be considered, not only to design fit for purpose *in vivo* tests, but also for providing evidence for not performing *in vivo* testing under certain circumstances. Animal tests must comply with the provisions laid down in Council Directive 86/609/EEC<sup>6</sup>.

## Degradation products and metabolites

In the context of evaluating substances for their effects, it is important to note that, once released into the environment or taken up by animals, a substance may be transformed through degradation or metabolism. These processes and their outcome may need to be taken into account in the overall assessment.

Degradation products may be formed as a result of transformation processes in the environment, either biotic or abiotic. For distinguishing the substance undergoing degradation from the degradation products, the former is often referred to as the parent compound.

Degradation products may be formed as a result of abiotic environmental processes such as hydrolysis, direct or indirect photolysis or oxidation. They may also be formed as a result of aerobic or anaerobic biodegradation, i.e. due to microbial activity. Degradation products require further investigation if the Chemical Safety Assessment indicates the need, i.e. if stable degradation products are formed in the environment within a relevant time frame, as deduced from the test system, or if they fulfill the PBT/ vPvB criteria. Likewise it may be considered to assess whether degradation products fulfil the environmental hazard classification criteria (see Section R.7.9 in Chapter R.7(b): Endpoint specific guidance).

Metabolites refer to transformation products, which are formed due to biodegradation (and then the term metabolite is synonymous with the term biodegradation product) or formed as a result of biotransformation (metabolism) within exposed organisms after uptake of the parent compound. Metabolic pathways and hence the identity of metabolites may or may not be fully known. The latter is frequently the case. Moreover for the same substances metabolic pathways may or may not differ between various organisms belonging to different phyla and/or trophic levels. However, the toxicity of metabolites formed within the duration of laboratory tests will be reflected by their parent compound, with the exception of delayed effects which are only evident after the observation time of the tests. Knowledge of metabolic pathways and metabolites may increase planning and focussing of toxicity testing and understanding of toxicological findings (see Section R.7.12 in Chapter R.7(c): Endpoint specific guidance). Therefore, in some cases it may be possible to use grouping approaches for structurally closely-related substances, which undergo similar metabolic transformation (see Section R.6.2, Chapter R.6: Guidance on QSARs and grouping of substances).

When biotransformation processes include oxidation, metabolites are often less hydrophobic than the parent compound. This is a very general rule of thumb and may not always apply; however, when it does, often this has implications for the hazard profile of the metabolites. For example more polar metabolites created after oxidation processes have normally a lower

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<sup>6</sup> Council Directive 86/609/EEC regarding the protection of animals used for experimental and other scientific purposes [OJ L 358, 18.12.1986, p. 1].

adsorption potential, and thus the relevance of the metabolites for the soil and sediment compartments is normally lower than that of the parent compound. Such less hydrophobic metabolites also tend to be excreted more rapidly from organisms than the parent compound. Hence both their bioaccumulative potential and narcotic toxicity tend to be lower.

Similarities in metabolic pathways of structurally-related substances may serve as an alert for waiving for further investigation, depending on the case and nature of the metabolites.

It should be noted that metals, and in particular metal compounds, do not degrade in the environment in the same way as organic substances. They transform usually through dissolution to the dissolved form.

## **Selection of the appropriate route of exposure for toxicity testing**

Having established the need for additional toxicity testing to meet the requirements of REACH for a given substance, for certain endpoints, notably acute or repeated dose toxicity but also reproductive toxicity, chronic toxicity and carcinogenicity, a decision must be made on which route/(s) of exposure is/(are) most appropriate. The overall objective of such testing is to determine the potential hazard of the test substance to human beings. Humans may be exposed to substances by one or more of three routes: inhalation, dermal and oral. In general, the final decision on which route of exposure is to be considered in a particular test should be taken in the light of all available information including physicochemical properties of the substance, structure-activity relationships (SAR) or the data from available toxicity tests on the substance itself. Fundamentally, the administration of the substance by inhalation in animal tests should be considered only when human exposure via inhalation is relevant.

Route-to-route extrapolation can be used to assess potential health effects and their threshold in a route other than the one tested. It should be stressed that toxicity data obtained using the appropriate route of exposure are preferred. Route-to-route extrapolation should be considered on a case-by-case basis and may introduce additional uncertainties, especially if the toxicity data were obtained using an administration route that does not correspond to the most relevant route of human exposure. In a subsequent risk assessment the uncertainties introduced through route-to-route extrapolation should be taken into account, for example by adjusting the assessment factor in the determination of the DNEL (see Section R.8.4.3, Chapter R.8: Characterisation of dose [concentration]-response for human health). Further guidance on this strategic approach to toxicity testing is given in Chapter R.8 Characterisation of dose [concentration]-response for human health.

## **Assessment of the environmental impact of a substance**

With regard to the evaluation of the environmental impact of a substance, the interaction of that substance with the environment is an important consideration. The fate and behaviour of a substance are largely governed by its inherent physico-chemical properties. The knowledge of the physicochemical properties of the substance, together with results from multimedia fate and transport models (e.g. Mackay level 3 models), enables the identification of the environmental compartment(s) of primary concern. Such information will also determine the prioritisation of higher tiered tests. More extensive guidance and considerations on this aspect are given in Chapter R.16: Environmental Exposure Estimation.

### **R.7.1 Physicochemical properties**

Advice to registrants with regard to nanomaterials characterisation can be found in "Appendix R7-1 Recommendations for nanomaterials applicable to: Chapter R7a Endpoint specific guidance", section 2 Recommendation for physicochemical properties arising from RIP-oN 2 for nanomaterials.

### R.7.1.1 Introduction on physicochemical properties

According to Article 12 of the REACH Regulation, for registration purposes all physicochemical information that is relevant and available to the registrant must be included in the technical dossier, i.e. information such as:

- Data on intrinsic properties of the substance (e. g. melting point/freezing point, boiling point, vapour pressure, density);
- Data necessary to assess the physical hazards of a substance (e. g. flammability), with the view to determine its classification and labelling according to CLP (and until June 1 2015, according to DPD, see Article 61 of CLP);
- Supplementary data for hazard assessment and health and environmental classification (e. g. viscosity, n-octanol/water partition coefficient).

Some physicochemical properties - notably explosive, flammable and oxidising properties - are intimately linked to physical hazards. The most straight-forward way of assessing these properties is through the classification of the substance for the corresponding physical hazards. There is thus a link between the physical hazards classification and the information requirements on explosive, flammable and oxidising properties. This is further elaborated below (see table R.7.1-1) and in the various chapters addressing these endpoints. For substances manufactured or imported in quantities of 100 tonnes or more per annum, some additional physicochemical data are required; in accordance with Annex IX to REACH (see also Table R.7.1-1).

Further details are given in the sections dedicated to specific endpoints.

#### R.7.1.1.1 Information requirements on physicochemical properties

Commission Regulation (EU) No 252/2011<sup>7</sup> has amended Annex I to REACH in order to adapt the chemical safety assessment provisions to the criteria for classification laid down in the CLP Regulation. The relevant amendments have been applied since 5 May 2011; however, for registrations submitted prior to this date, the chemical safety report shall be updated in accordance with Regulation No 252/2011 by 30 November 2012 at the latest.

The information needed under Article 12, REACH on one hand and according to section 4 of Annex VI to REACH on the other (namely hazard classification according to Title I and II CLP) is often complementary but in some cases may be different. The reason is that the classification criteria and/or test methods under DSD and CLP regimes are different. This is also expressed by the fact that CLP classifications are distributed over a different grid of hazard classes and categories compared to the DSD regime, e.g. substances and mixtures classified as explosive under DSD may be classified as explosives or self-reactives or organic peroxides under CLP, or they may even be classified as flammable solids, oxidizing solids or not at all. A translation table from DSD to CLP classification is provided in Annex VII, CLP and an indication of potential classification outcomes under CLP compared to DSD classifications is provided by Table 1.7.2.1(a) in the 'Guidance on the Application of the CLP Criteria' which can be found on the following ECHA page: <http://echa.europa.eu/web/guest/guidance-documents/guidance-on-the-different-methods-under-reach>.

The CLP classification regime is not explicitly considered in Annex VII to REACH and therefore has to be understood as part of the information requirements under REACH. In particular, certain headlines set out in column 1 of Annex VII to REACH, namely 'explosive properties',

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<sup>7</sup> Commission Regulation (EU) No 252/2011 of 15 March 2011 amending Regulation (EC) No 1907/2006 of the European Parliament and of the Council on the Registration, Evaluation, Authorisation and Restriction of Chemicals (REACH) as regards Annex I.

1 'flammability' and 'oxidizing properties', must be interpreted as covering the CLP hazard  
2 classes that are referred to in Article 58(11) of CLP.

3 The physical hazard classes according to CLP are structured differently from the corresponding  
4 classifications according to DSD. Despite this, most of the CLP physical hazard classes can  
5 unambiguously be assigned to specific heading of the information requirements according to  
6 Annexes VI to XI to REACH,. However, for some CLP physical hazard classes - notably the  
7 hazard class "self-reactive substances and mixtures" and the hazard class "organic peroxides"  
8 - the assignment to a specific heading is not straight-forward, since they may have both  
9 explosive and/or flammable properties. Therefore, some of the hazard classes are listed twice  
10 in Table R.7.1-1 below. It should be noted that this assignment is provided only as example  
11 and is done for the purposes of structuring this guidance document according to CLP but  
12 nevertheless also allowing the assignment to the respective information requirements  
13 according to Annexes VII to XI to REACH.

14 According to Article 1(6) CLP, CLP Regulation does not apply to the transport of dangerous  
15 goods by air, sea, road, rail or inland waterways (save where the specific rules for labelling of  
16 packaging applies under Article 33 of CLP). The transport of dangerous goods is, covered by  
17 the UN Model Regulations for Transport of Dangerous Goods (UN-RTDG) and related legal  
18 instruments (ADR, RID, ADN, IMDG Code and ICAO TI); the criteria listed in these instruments  
19 and in CLP Regulation for classification purposes are intended to be the same. Thus, a  
20 substance (or mixture) classified in a hazard class which is common to both CLP and the  
21 transport legislation will normally be classified the same according to both systems. Therefore  
22 the transport classification of a substance could be a source of information for the classification  
23 and labelling of substance (or a mixture) under CLP for physical hazards. However it should be  
24 kept in mind that the transport classifications do not cover all hazard categories which are  
25 relevant for CLP and it may be based on other considerations than just the test data and  
26 criteria (e.g classifications which are based on experience rather than testing or which apply  
27 only in connections with certain special provisions). As a result, the transport classifications  
28 may be different for the classification according to CLP. Similarly, the absence of a transport  
29 classification does not necessarily mean the substance (or mixture) should not be classified  
30 under CLP. Consequently in the case of a substance which has been tested for the purposes of  
31 the UN-RTDG and for which the same procedure was followed as required by the CLP  
32 Regulation, the same information could be used to comply with the REACH Regulation on a  
33 case-by-case basis. The limitations to the approach described above are described in detail in  
34 the 'Guidance on the Application of the CLP Criteria', Section 1.7.2.1.

35  
36 For the preparation of the registration dossier, registrants are required to submit all the  
37 information listed in Article 10 of REACH. Article 14(1) in conjunction with Annex I and Article  
38 10(a)(vii) of the REACH Regulation, require the provision of a Robust Study Summary (RSS)  
39 for information derived from the application of Annexes VI to XI to REACH. In order to facilitate  
40 the evaluation conducted by the European Chemicals Agency and the Member States, as well  
41 as to save registrant's resources in case of a tonnage update, it **is recommended** that  
42 registrants also use the RSS for covering physicochemical endpoints under section 4 of the  
43 IUCLID file. This guidance includes under each physicochemical property chapter a list of  
44 detailed information to be given for each respective endpoint. Note that no further guidance is  
45 provided on the general aspects related to information common for all endpoints in IUCLID. For  
46 these aspects, further guidance can be found in 'Practical guide 3: How to report robust study  
47 summaries' available on the ECHA Website at:  
48 <http://echa.europa.eu/web/guest/support/documents-library> and in the IUCLID 5 End User  
49 Manual available on the IUCLID Website at:  
50 <http://iuclid.eu/index.php?fuseaction=home.documentation#usermanual>.

51  
52 Those endpoints, such as explosive properties and oxidising properties, which are intimately  
53 linked to classification, should be assessed according to CLP. For these endpoints, the test  
54 methods of CLP should preferably be used, in order to avoid double testing. For endpoints not  
55 linked to classification the preferred test methods are those found in the Test Method  
56 Regulation. For some endpoints (for example flammability), more than one test procedure is

referred to in the standard test method reported in the Test Method Regulation. The one chosen should be suitable for the substance in question and be operating within its validity range.

Note that in the table below in order to distinguish the physicochemical properties that are directly linked to physical hazard classifications from those that are not, the former have been shaded in gray and that in addition the preferred test methods for the different endpoints have been put in bold text.

**Table R.7.1-1 Information requirements as specified in Annexes VII to IX to REACH and corresponding tests methods according to the Test Method Regulation and CLP**

<b>Information requirement according to Art. 10 (a) (vi) of the REACH Regulation (EC) No. 1907/2006</b> (the no. in brackets is the respective no. in the table in Annexes VII to XI to REACH)	<b>Corresponding test method according to The Test Method Regulation 440/2008</b>	<b>Chapter in revised R.7(a) guidance</b>	<b>CLP Regulation (EC) No. 1272/2008</b> (the no. in brackets is the respective chapter no. in Annex I to CLP)	<b>Corresponding test method according to CLP Regulation</b>
Melting/ Freezing point (7.2)	<b>A.1 Melting/Freezing temperature</b>	7.1.2	n.a.	n.a.
Boiling point (7.3)	<b>A.2 Boiling temperature</b>	7.1.3	n.a.	n.a.
Relative density (7.4)	<b>A.3 Relative density</b>	7.1.4	n.a.	n.a.
Vapour pressure (7.5)	<b>A.4 Vapour pressure</b>	7.1.5	n.a.	n.a.
Surface tension (7.6)	<b>A.5 Surface tension</b>	7.1.6	n.a.	n.a.
Water solubility (7.7)	<b>A.6 Water solubility</b>	7.1.7	n.a.	for metals - Transformation/Dissolution Protocol (Annex 10 to UN GHS)
Partition coefficient n-octanol/water (7.8)	<b>A.8 Partition coefficient</b>	7.1.8	n.a.	n.a.
Flash point (7.9)	A.9 Flash-point	7.1.9	n.a.	<b>CLP Annex I chapter 2.6.4.4</b>
Flammability (7.10)	A.11 Flammability (gases)	7.1.10.1	Flammable gases (2.2)*	<b>ISO 10156 EN 1839</b>
	for liquids: see Flash point	7.1.10.2	Flammable liquids (2.6)*	<b>see CLP, Annex I, Chapter 2.6.4.4, Table 2.6.3</b>
	A.10 Flammability (solids)	7.1.10.3	Flammable solids (2.7)*	<b>UN Test N.1</b>
	n.a.	7.1.10.4	Self-reactive substances and mixtures	<b>UN Test series A to H</b>

			(2.8)*	
	A.13 Pyrophoric properties of solids and liquids	7.1.10.5	Pyrophoric liquids (2.9)*	<b>UN Test N.3</b>
		7.1.10.6	Pyrophoric solids (2.10)*	<b>UN Test N.2</b>
	n.a.	7.1.10.7	Self-heating substances and mixtures (2.11)*	<b>UN Test N.4</b>
	A.12 Flammability (Contact with water)	7.1.10.8	Substances and mixtures which in contact with water emit flammable gases (2.12)*	<b>UN Test N.5</b>
	n.a.	7.1.10.9	Organic peroxides (2.15)*	<b>UN Test series A to H</b>
Explosive properties (7.11)	A.14 Explosive properties	7.1.11.1	Explosives (2.1)*	<b>UN Test series 1 to 3 (further test series 4 to 6 are necessary for classification)</b>
	n.a.	R.7.1.11.2 see R.7.1.10.4	Self-reactive substances and mixtures (2.8)*	<b>A.14 (existing data only)</b>
	n.a.	R.7.1.11.3 See R.7.1.10.9	Organic peroxides (2.15)*	<b>A.14 (existing data only)</b>
Self ignition temperature (7.12)	A.15 Auto-ignition temperature (liquids and gases)	7.1.12.1	For gases and liquids*	n.a.
	A.16 Relative self-ignition temperature for solids	7.1.12.2, 7.1.10.7	For solids*  Note: the UN Test N.4 is preferable to generate the information for this endpoint. Refer to R.7.1.10.7.	n.a.
Oxidising properties (7.13)	n.a.	7.1.13.1	Oxidising gases (2.4) *	<b>ISO 10156</b>
	A.21 Oxidising properties (liquids)	7.1.13.2	Oxidising liquids (2.13) *	<b>UN Test O.2</b>



	A.17 Oxidising properties (solids)	7.1.13.3	Oxidising solids (2.14) *	<b>UN Test O.1</b>
Granulometry (7.14)	n.a.	7.1.14	n.a.	n.a.
Adsorption/Desorption (7.15)	n.a.	7.1.15	n.a.	n.a.
Stability in organic solvent and degradation products (7.16)	n.a.	7.1.16	n.a.	n.a.
Dissociation constant (7.17)	n.a.	7.1.17	n.a.	n.a.
Viscosity (7.18)	n.a.	7.1.18	n.a.	n.a.

\* Note that regardless of whether the hazard class or category is listed in Article 14 (4) (a) of REACH, the chemical safety assessment (when required) must be performed in accordance with Article 14 (3) of REACH. Furthermore, according to Article 10 (a) (iv) of REACH the technical dossier of a registration for a substance under the REACH Regulation must include information on classification and labelling of the substance as specified in section 4 of Annex VI to the REACH Regulation.

In addition the CLP Regulation has the following hazard classes for which the REACH Regulation does not require the generation of information (Article 10(a)(vi) and (vii) REACH):

<b>CLP Regulation (EC) No. 1272/2008 (the no. in brackets is the respective chapter no. in Annex I to CLP)</b>	<b>Corresponding test method according to the Test Method Regulation</b>	<b>Chapter in revised R.7(a) guidance</b>	<b>Information requirement according to Art. 10(a)(vi) of the REACH Regulation</b>	<b>Corresponding test method according to CLP Regulation</b>
Flammable aerosols (2.3)	n.a.	7.1.21.1	n.a.	Test methods according to 75/324/EC amended by 2008/47/EC (harmonised with <b>UN Section 31</b> )
Gases under pressure (2.5)	n.a.	7.1.21.2	n.a.	n.a.
Corrosive to metals (2.16)	n.a.	7.1.21.3	n.a.	<b>UN Test C.1 (section 37.4)</b>

In order to comply with the REACH information requirements, registrants have to take due account of specific rules for adaptation according to column 2 of the tables in Annexes VII to XI to REACH, including the provisions given within the individual test methods of the Test Method Regulation, which have to be interpreted and applied in relation to the appropriate CLP hazard class. Further adaptations according to Annex XI to REACH must then be read together with the adaptation possibilities provided for by Article 8(2) of CLP and the CLP criteria themselves, namely those in Part 2 of Annex I to CLP.

Physicochemical data are mostly numeric and should be provided in SI units. Normally a numeric value or range is required. Where relevant, additional information should be provided on test conditions, such as temperature and/or pressure and/or concentration level or range

etc., and estimated uncertainty in the numerical value. Furthermore details of any observations made during testing should be reported, e.g. decomposition during melting or boiling, emulsion formation during partitioning.

#### R.7.1.1.2 Available information on physicochemical properties

There are many published sources of data for basic substance characterisation and of supplementary information for hazard assessment. The relevant references are listed under respective endpoint.

#### R.7.1.1.3 Evaluation of available information on physicochemical properties

Advice to registrants with regard to nanomaterials characterisation can be found in Appendix R7-1 Recommendations for nanomaterials applicable to: Chapter R7a Endpoint specific guidance, section 2.1.3 Evaluation of available information.

### Experimental data

Further, according to Article 13 (3) of the REACH Regulation, tests to generate information on intrinsic properties of substances must be conducted in accordance with the test methods laid down in a Commission Regulation or in accordance with other international test methods recognised by the Commission or the Agency as appropriate, such as European standards ([www.cen.eu](http://www.cen.eu)) or OECD guidelines ([www.oecd.org](http://www.oecd.org)). Data obtained from the tests in accordance with section 1.1.1 of Annex XI to REACH can be considered to be equivalent to data generated by the corresponding test methods referred in Article 13 (3) of REACH. Data for the purpose of physical hazard classification can be obtained using the test methods specified in the Articles 5 (1) and 8 (3) CLP. The test methods for the physicochemical properties are described in Regulation (EC) No 440/2008, whereas preferred tests for the purposes of physical hazard classification are referred to in Part 2 of Annex I to CLP, via references to the UN Manual of Tests and Criteria and to applicable standards. In Table R.7.1-1, the preferred test method for each endpoint is highlighted in bold. The test methods referred to in the CLP Regulation are also used for the transport of dangerous goods. Therefore, available information on physicochemical properties and physical hazards may also originate from tests that were carried out for the purposes of classification for transport. Such test data may be used for the information requirements according to the REACH Regulation. It should, however, be kept in mind that the classification for transport does not cover all hazard categories which are relevant for CLP and it may be based on other considerations than just the test data and criteria (e.g. classifications which are based on experience rather than testing or which apply only in connection with certain special provisions). As a result transport classifications may be different from the classification according to CLP. Such limitations are described in detail in the 'Guidance on the Application of the CLP Criteria', Section 1.7.2.1.

Where relevant recognised standards for testing are applicable, the use of the most recent updates is advised; they are accessible via numerous websites, for example:

1. EN standards;
2. [ISO standards](#);
3. [IEC standards](#).

The national editions of the EN or ISO standards are available via the national standardization organizations accessible via the [CEN Website](#).

Measured values which are evaluated in reviews and assigned recommended values are given precedence over calculated values. The major criteria that characterise the analysis of the available information are:



- **Experimental data.** When assessing physicochemical properties, priority is given to first hand experimental results (primary references) provided that the methods are suitable for the substance under investigation and that they operate within their validity range. Proper documentation on the methods and the inherent uncertainty of the measurements should also be provided.
- **Non-testing information.** If the information described in point (a) is not available, QSPRs, read-across or secondary data sources (e.g. handbook data) can be used in accordance with the limitations described in the individual endpoint chapters (7.2 to 7.19 in this guidance) instead, and within the constraints of Annex XI to REACH.

## Measurement uncertainty

Test data have an uncertainty of measurement. Some test methods include information about their uncertainty, which then may be referred to for test data generated using these test methods. Where the uncertainty of measurement is not specified by the test method, it is recommended to determine uncertainty by generally accepted processes of measurement uncertainty estimation (e.g. according to ISO/IEC Guide 98-3:2008).

## Quality assurance for the determination of physicochemical properties

Test data on physicochemical properties should be of sufficient quality i.e. they must be reliable. Normally this can only be achieved by testing that is carried out in compliance with a relevant recognised quality system (e.g. GLP) or by laboratories complying with a relevant recognised standard (e.g. EN ISO/IEC 17025). Under Article 8 (5) of CLP, where new tests for physical hazards are required for the purposes of CLP they have to be carried out in compliance with a relevant recognised quality system or by laboratories complying with a relevant recognised standard at the latest from January 1st, 2014.

## Non-experimental data

Quantitative Structure Property Relationships (QSPR) models exist for some of the physicochemical endpoints. Where applicable, the details of any specific QSPR models are given under each endpoint.

The majority of QSPR models have been built using training sets of substances. The model will have been optimised to calculate values for the training substances that most closely match measured ones. Therefore, the use of QSPR estimation techniques requires expert judgment. The calculated values need to be checked to ensure that they are reasonable and that the model used is appropriate.

A valid model will give values that are in reasonably close agreement with the measured ones for your chosen analogue substances (i.e. the substance with a data gap should have similar substances in the training set of the model). The models may not predict very well the properties of substances which are too dissimilar to the reference set for the model. Thus, the model can be used to provide a predicted value for your substance without the need for testing. Another check is that the values are realistic. This can be done by cross-referencing the calculated value to measured values for similar substances and related endpoints. If a QSAR method is used as a stand alone method to determine a value to meet the endpoint data requirements, the QSAR model must meet the criteria set out in section 1.3 of Annex XI to REACH.

## Assessing the quality of QSPR models

The European Commission and the OECD member countries adopted five principles for the validation of (Q)SAR/(Q)SPR models in 2004 (OECD<sup>a</sup>, 2004). According to these principles, a valid (Q)SPR model should have 1) a defined endpoint whose experimental conditions are clearly specified; 2) an unambiguous algorithm; 3) a defined domain of applicability that

defines for what kind of substances predictions can be made; 4) appropriate measures of goodness-of-fit, robustness and predictivity; and 5) a mechanistic interpretation if possible. These principles are outlined on the ECB website and more extensively covered in *IR/CSA R.6: QSAR and grouping of substances*, section R.6.1.3. Moreover, a practical overview of these principles is given in the report from the expert group on (Q)SARs (OECD<sup>b</sup> 2004).

## Assessing the quality of read-across predictions

This paragraph reports the basic concepts of a read-across approach. Thorough information on this topic can be found in the guidance on the grouping of substances (see *IR/CSA R.6: QSAR and grouping of chemicals*, Section R.6.2).

A read-across/analogue approach assesses the relevance of a given property on one or more chemical structures and then makes some assessment (qualitative or quantitative) on the relevance of this information for another substance (see Annex XI, REACH). Since a read-across may involve two substances<sup>8</sup> it is of paramount importance to detail the reasoning behind the inference on the substance whose property is unknown. An analogue must:

- contain the same major structural features and the same functional groups as the substance under investigation;
- have a physicochemical profile comparable to that of the substance under examination as far as the known physicochemical properties are concerned;
- have comparable values for the relevant molecular descriptors (i.e. excess molar refractivity and hydrogen bond donor and acceptor abilities for water solubility predictions) generally used for the quantification of the property of interest;
- have approximately the same molecular weight.

The interpretative analysis of a read-across is usually the result of an expert judgement evaluation and detailed documentation should therefore always be provided to support the conclusions. It is important to point out that, in practice, read-across for physicochemical properties is not generally recommended, since reliable data should normally be available or easily obtainable. This is particularly true for physical hazard related physicochemical properties for which reliable test data should be available according to Article 8 (2) of CLP. Therefore, if read-across is used as a stand alone method to generate a value to meet the endpoint data requirements, the criteria given in section 1.5 of Annex XI to REACH must be met.

## Use of secondary and historical data sources for physicochemical properties

The reliability of data must be demonstrated by providing information on the identity and purity of the test substance, the methodology used to make the measurement, and whether or not this was performed in compliance with a relevant recognised quality system (e.g. GLP) (Annex VI, REACH).

Numerical physicochemical data is particularly prone to data recycling (transfer from one database to another, often with loss of the original source and contextual information). Data from secondary and historical sources must be adequate and is especially important where the endpoint is relevant for classification, PBT/vPvB assessment, is the basis of waiving arguments for other endpoints, or has a large influence on the outcome of the risk assessment. The criteria in section 1.1.1 of Annex XI or section 1.2 of Annex XI to REACH must be met.

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<sup>8</sup> A read-across can also involve more than two substance: a) one-to-one (one analogue used to make an estimation for a single substance) b) many-to-one (two or more analogues used to make an estimation for a single substance) c) one-to-many (one analogue used to make estimations for two or more substances) d) many-to-many (two or more analogues used to make estimations for two or more substances).

#### 1 R.7.1.1.4 Overall consistency of the physicochemical information

2 The physicochemical data for a given substance cannot contain incompatible values for two or  
3 more properties (i.e. high boiling point and high vapour pressure at normal temperature). This  
4 consistency check should be always done and it can turn out to be particularly useful when  
5 measured values are significantly at odds with predictions from QSPR models. Indeed, in this  
6 case a wider assessment of the known physicochemical properties should be performed in  
7 order to determine the possible cause of the inconsistencies.

#### 8 **Concluding on classification and labelling and chemical safety assessment**

9 Data on physicochemical properties not only determine the presence or absence of a physical  
10 hazard but also have also an impact on the sections of the chemical safety assessment  
11 concerning the environment and human health. The assessment determines the risk posed to  
12 humans and the environment from all stages of the substance's lifecycle. This includes its  
13 manufacture, transfer, use and disposal. Firstly, the physicochemical data set provides the  
14 input parameters for the purpose of the human and environmental exposure estimation. For  
15 example, the vapour pressure and particle size information are required to estimate the likely  
16 exposure of humans, both in the workplace and in consumer use as well as to estimate the  
17 likelihood of forming flammable/explosive vapour/dust-air mixtures. The volatility (vapour  
18 pressure) or the size and nature of particles are indicators of the potential for inhalation  
19 exposure. Particle size is also important for determining the likely dermal exposure and the  
20 presence of a dust explosion hazard. Viscosity is a key parameter in determining aspiration  
21 hazards. The physical state of a substance at the process temperature is important for  
22 determining possible hazards. Further, physico-chemical data are essential for the correct  
23 planning of (eco)toxicological studies and for the optimisation of the test conditions.

#### 24 R.7.1.1.5 References for introduction of Physicochemical properties

25 Recommendations on the transport of dangerous goods, Manual of Test and Criteria, United  
26 Nations. [http://www.unece.org/trans/danger/publi/manual/manual\\_e.html](http://www.unece.org/trans/danger/publi/manual/manual_e.html)

27 Guidance on the Application of the CLP Criteria, Version 2.0 - 2012, ECHA.  
28 <http://echa.europa.eu/web/guest/support/guidance-on-reach-and-clp-implementation>

29 OECD<sup>a</sup> (2004) Principles for the Validation of (Q)SARs  
30 [http://ecb.jrc.it/QSAR/home.php?CONTENU=/QSAR/background/background\\_oecd\\_principles.php](http://ecb.jrc.it/QSAR/home.php?CONTENU=/QSAR/background/background_oecd_principles.php)  
31

32 OECD<sup>b</sup> (2004) series on testing and assessment Number 49 The report from the expert group  
33 on (quantitative) structure activity relationships [(Q)SARs] on the principles for the validation  
34 of (Q)SARs. 2nd Meeting of the ad hoc Expert Group on QSARs  
35 [http://www.oecd.org/officialdocuments/displaydocumentpdf/?cote=env/jm/mono\(2004\)24&doclanguage=en](http://www.oecd.org/officialdocuments/displaydocumentpdf/?cote=env/jm/mono(2004)24&doclanguage=en)  
36

#### 37 **R.7.1.2 Melting/freezing point**

##### 38 R.7.1.2.1 Type of property

39 The melting point contributes to the identification of a substance and to the designation of its  
40 physical state (liquid or solid<sup>9</sup>) of a substance. A number of physical hazard classes are  
41 distinguished based on the physical state. Therefore the melting point of a substance and the  
42 consequent designation as liquid or solid has also consequences for the assignment of the  
43 correct hazard class. Furthermore, the melting/freezing point together with vapour pressure  
44 serves as an indicator for the physical state (liquid or solid) of a substance under specific

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<sup>9</sup> Definitions of physical states can be found in Section 1.0. of Annex I to the CLP Regulation.

conditions (e.g environmental conditions, manufacturing process conditions). As a result, with regard to environmental relevance the melting point can give an indication of the distribution of the substance within and between the environmental media (water, soil and air).

#### R.7.1.2.2 Definition

The melting temperature is defined as the temperature at which the phase transition from the solid to the liquid state occurs at atmospheric pressure and this temperature ideally corresponds to the freezing temperature. As the phase transition of many substances takes place over a temperature range, it is often described as the melting range. For some substances, the determination of the freezing or solidification point is more appropriate. Where, due to the particular properties of the substance, none of the above parameters can be conveniently measured, a pour point may be appropriate.

#### R.7.1.2.3 Test method(s)

Method A.1 of Regulation (EC) 440/2008 or OECD Test Guideline 102 should be generally used for testing. Any procedure given in A.1 may be used within the scope and applicability specifications. However, it is advisable to use the Differential Scanning Calorimetry (DSC) or Differential Thermo-Analysis (DTA) method since they give additional information about the thermal stability of the substance like decomposition onset and energy. If decomposition occurs during the melting point study, determination of the boiling point need not be carried out. In this case, if DSC has been used, conducting the experiment under inert gas should be considered.

#### R.7.1.2.4 Adaptation of the standard testing regime

##### **Adaptation possibilities according to column 2 of Annex VII to REACH**

Column 2 of REACH Annex VII provides the following specific rules for adaptation of the standard information requirement for melting/freezing point:

*The study does not need to be conducted below a lower limit of - 20 °C.*

Therefore, Annex VII to REACH does not require determination of the melting point below a lower limit of -20 ° C. The lower limit should be confirmed through testing, except where a (Q)SAR indicates a melting point of -50 ° C or lower.

##### **Adaptation possibilities according to Annex XI to REACH**

##### **USE OF EXISTING DATA: DATA ON PHYSICAL-CHEMICAL PROPERTIES FROM EXPERIMENTS NOT CARRIED OUT ACCORDING TO GLP OR THE TEST METHODS REFERRED TO IN ARTICLE 13 (3) OF REACH**

If experimental data are available (study reports or literature data) meeting the criteria of Annex XI, section 1.1.1, these could be used to meet the endpoint data requirements. If an estimation method is used as a source of information according to Column 2 of Annex VII, the QSAR model must meet the criteria set out in Annex XI, section 1.3.

##### **WEIGHT OF EVIDENCE**

Where no single source of existing data (study reports, QSAR, literature data) is considered sufficiently reliable, thus not fully meeting the criteria of Annex XI, section 1.1.1 or where

1 several sources of similar reliability with deviating results exist, a weight-of-evidence approach  
2 may be used. The criteria of Annex XI, section 1.2 must then be met<sup>10</sup>.

### 3 **(Q)SAR**

4 For the determination of the melting point, (Q)SAR approaches are discouraged, because the  
5 accuracy is not sufficient ( $\pm 25$  °C or more) for the purposes of classification / risk  
6 assessment.

### 7 **GROUPING OF SUBSTANCES AND READ-ACROSS APPROACH**

8 For the determination of the melting point read-across is usually not possible. However  
9 interpolation may still be possible within homologous series.

### 10 **TESTING IS TECHNICALLY NOT POSSIBLE**

11 Some substances will decompose or sublime before the melting point is reached.

### 12 **Further adaptation possibilities**

13 Not foreseen.

#### 14 **R.7.1.2.5 Impurities; uncertainties**

15 Impurities can have a significant influence on the melting point, as they will generally lower  
16 the melting point noticeably. Therefore utmost care should be taken in the selection of the key  
17 study(s), or weight-of-evidence approaches, that the data selected is representative of the  
18 substance being registered by the respective companies.

#### 19 **R.7.1.2.6 Endpoint specific information in the registration dossier/ IUCLID**

##### 20 **Materials and methods**

21 • type of method or reference to the standard or the test method applied.

##### 22 **Results and discussion**

23 • melting point value (°C) as measured;

24 • rate of temperature increase if available;

25 • decomposition or sublimation temperature (if applicable);

26 • measurement uncertainty if available;

27 • if testing is waived, the reasons for waiving must be documented in the dossier.

28

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<sup>10</sup> National Institute of Standards and Technology (NIST) have a useful statistical approach which has been used for the evaluation of literature melting point data (ref.: <http://webbook.nist.gov/chemistry/site-cal.html#AVG>).

Any deviation from the guideline method used or any other special consideration should be reported. In cases where there is more than one source of data, the endpoint summary under results and discussion should provide a justification for the selection of the key study chapter.

## Reference to other ECHA Guidance Documents

Further detailed guidance on melting point/freezing point can be found in the following chapters:

IUCLID Section	REACH Annex	Endpoint title	IUCLID 5 End User Manual Chapter	ECHA Practical Guide 3
4.2	VII 7.2	Melting point/freezing point	E.4.3	3.2

### R.7.1.3 Boiling point

#### R.7.1.3.1 Type of property

The boiling point is a property:

- which contributes to the characterisation of a substance and to the designation of its physical state (gas or liquid);
- which is the basis for the assignment of the correct hazard class because a number of physical hazard classes are distinguished based on the physical state;
- which is needed for the classification of flammable liquids into categories;
- which gives an indication of the distribution of the substance within and between the environmental compartments (air, soil and water);
- which have correlations with vapour pressure and therefore gives indications whether a substance may be available for inhalation as a vapour or may form flammable/explosive vapour-air mixtures, too;
- which is important for physical hazard assessment.

#### R.7.1.3.2 Definition

The normal boiling point is the temperature at which the vapour pressure of a liquid equals 101.3 kPa.

Note: If the vapour pressure equals 101.3 kPa or more at a given temperature this means the substance is completely gaseous at that temperature. If this is the case at temperatures  $\leq 20$  °C the substance is a gas also according to the CLP Regulation.

#### R.7.1.3.3 Test method(s)

Method A.2 of Regulation (EC) 440/2008 or OECD Test Guideline 103 should be used for testing. Any determination method may be used within the scope and applicability specifications. DSC allows the determination of the melting and boiling point in a single test. Likewise, for some substances a single test can be used to determine both 'boiling point' and 'vapour pressure', as when the dynamic method is applied.

For high-boiling liquids or liquids which may decompose, auto-oxidize etc. before the boiling point at 101.3 kPa or more is reached, it is recommended to determine the boiling point either under inert gas or at reduced pressures, in order to derive the boiling point at reduced pressures from the vapour pressure curve.

If explosive substances, pyrophoric substances or self-reactive substances are to be characterized, determination of the boiling point is in general not practicable. For pyrophoric substances testing under inert gas or reduced pressures should be considered.

Where standards are applicable, the use of the most recent updates is advised; they are accessible via numerous websites, see above in section R.7.1.1.3.

#### R.7.1.3.4 Adaptation of the standard testing regime

#### **Adaptation possibilities according to column 2 of Annex VII to REACH**

Column 2 of REACH Annex VII provides the following specific rules for adaptation of the standard information requirement for boiling point:

*The study does not need to be conducted:*

- *for gases; or*
- *for solids which either melt above 300 °C or decompose before boiling. In such cases the boiling point under reduced pressure may be estimated or measured; or*
- *for substances which decompose before boiling (e.g. auto-oxidation, rearrangement, degradation, decomposition, etc.).*

Therefore the Annex VII to REACH does not require determination of the boiling point if:

- The substance is a gas;

However, for some gases the boiling point may be relevant. In the CLP Regulation, the boiling point is the main criterion to distinguish gases from liquids (see Annex I, section 1.0: Gas means a substance which (i) at 50 °C has a vapour pressure greater than 300 kPa (absolute); or (ii) is completely gaseous at 20 °C at a standard pressure of 101.3 kPa). Therefore it is important to report the boiling point in borderline cases where the transition from liquid to gas occurs close to 20 °C.

- The melting point of the substance is above 300 °C or when any chemical change occurs during the melting point study;
- The substance decomposes before boiling at ambient pressure;

In such cases the boiling point under reduced pressure (down to 0.2 kPa) should be determined if possible without decomposition.

#### **Adaptation possibilities according to Annex XI to REACH**

#### **USE OF EXISTING DATA: DATA ON PHYSICAL-CHEMICAL PROPERTIES FROM EXPERIMENTS NOT CARRIED OUT ACCORDING TO GLP OR THE TEST METHODS REFERRED TO IN ARTICLE 13 (3) OF REACH**

If experimental data are available (study reports or literature data) meeting the criteria of Annex XI, section 1.1.1, these could be used to meet the endpoint data requirements. If an estimation method is used as a source of information according to Column 2 of Annex VII, the QSAR model must meet the criteria set out in Annex XI, section 1.3.

#### **WEIGHT OF EVIDENCE**

Where no single source of existing data (study reports, QSAR, literature data) is considered sufficiently reliable, thus not fully meeting the criteria of Annex XI, section 1.1.1 or where



several sources of similar reliability with deviating results exist, a weight-of-evidence approach may be used. The criteria of Annex XI, section 1.2 must then be met<sup>11</sup>.

### **(Q)SAR**

For the determination of the boiling point, (Q)SAR approaches are discouraged for the purpose of classification / risk assessment, except when the mean absolute error of the method is lower than 2 K.

### **GROUPING OF SUBSTANCES AND READ-ACROSS APPROACH**

For the determination of the boiling point read-across is usually not possible. However interpolation may still be possible within homologous series.

### **TESTING IS TECHNICALLY NOT POSSIBLE**

Testing is not possible if:

- the substance is an explosive;
- the substance is self-reactive;
- any chemical change occurs during the melting point study;
- the liquid decomposes before the boiling point is reached even at reduced pressures below 0.2 kPa.

In such cases the decomposition temperature in relation to the (reduced) pressure should be reported, in order to allow determination of whether it is the substance itself or its decomposition products that should be considered under environmental conditions for the purpose of risk assessment. The details of the determination method should also be reported.

### **Further adaptation possibilities**

Data generated with the same tests and classification principles as specified in the CLP Regulation on viscosity generated in conjunction with transport classification can be deemed to satisfy the REACH requirements on a case-by-case basis. As stated in Annex IX of the REACH Regulation, when for certain endpoints, it is proposed to not provide information for other reasons than those mentioned in column 2 of that Annex or in Annex XI of REACH, this fact and the reasons must also be clearly stated. Such an approach may then be used.

#### **R.7.1.3.5 Impurities; uncertainties**

Impurities can have a significant influence on the boiling point. Therefore utmost care should be taken in the selection of the key study(s), or weight-of-evidence approaches, that the data selected is representative of the substance being registered by the respective companies.

#### **R.7.1.3.6 Endpoint specific information in the registration dossier / in IUCLID**

##### **Materials and methods**

- type of method or reference to the standard or the test method applied.

##### **Results and discussion**

- boiling point value ( °C) as measured;

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<sup>11</sup> The NIST have a useful statistical approach which has been used for the evaluation of literature boiling point data (ref.: <http://webbook.nist.gov/chemistry/site-cal.html#AVG>).



- 1 • pressure value and unit;
- 2 • rate of temperature increase if available;
- 3 • decomposition (if applicable);
- 4 • measurement uncertainty if available;
- 5 • boiling point value in °C (corrected to standard pressure, except where the boiling point has
- 6 been determined at specified reduced pressures) (as above, but in a separate block of fields);
- 7 • if testing is waived, the reasons for waiving must be documented in the dossier.

8 Note: In case the boiling point is determined at ambient pressure, a determination at reduced  
9 pressure is not possible. A boiling point at standard pressure could then only be derived by  
10 extrapolation of the vapour pressure curve in cases where a vapour pressure curve is known.  
11 Even in such cases this corrected/extrapolated boiling point could only be nominal one and  
12 would be potentially misleading because it is not possible to determine it at ambient pressure.

13 Any deviation from the guideline method used or any other special consideration should be  
14 reported. In cases where there is more than one source of data, the endpoint summary under  
15 results and discussion should provide a justification for the selection of the key study chapter.  
16

## 17 **Reference to other ECHA Guidance Documents**

18 Further detailed guidance on boiling point can be found in the following chapters:

IUCLID Section	REACH Annex	Endpoint title	IUCLID 5 End User Manual Chapter	ECHA Practical Guide 3
4.3	VII 7.3	Boiling point	E.4.4	3.2

### 19 **R.7.1.4 Relative density**

#### 20 **R.7.1.4.1 Type of property**

21 For gaseous materials, relative density is of value in determining the tendency to settle or to  
22 disperse when discharged at high concentrations into the atmosphere. The relative density of  
23 gaseous substances can be calculated from molecular weight using the Ideal Gas Law.

24 For insoluble liquids and solids, (absolute) density will be a determining factor in the settling  
25 of the substance.

#### 26 **R.7.1.4.2 Definition**

27 Density ( $\rho$ ) of a substance is the quotient of the mass  $m$  and its volume  $V$ :

28  $\rho = m/V$  SI units ( $\text{kg}/\text{m}^3$ )

29 The relative density is related to a standard, the density of which is set to 1. It has no  
30 dimension. For gases air is used as standard so that gases with a relative density of less than 1  
31 are lighter than air (and those with a value above 1 heavier).

32 The relative density,  $D_4^{20}$ , of solids or liquids is the ratio between the mass of a volume of  
33 substance to be examined, determined at 20 °C, and the mass of the same volume of water,  
34 determined at 4 °C (at which temperature, water has its maximum density, i.e. 0.999975  
35  $\text{kg}/\text{m}^3$ ).

#### 36 **R.7.1.4.3 Test method(s)**

1 Test methods for determining (absolute) density are applicable to solids and liquids. Table  
2 R.7.1- lists the respective test methods.

3 **Table R.7.1-2 Test methods for determining density**

Method	Applicability	Maximum Dynamic Viscosity (Liquids only)/Pa.S
Hydrometer	Liquids	5
Hydrostatic balance	Solids and Liquids	5
Immersion ball	Liquids	20
Pycnometer	Solids and Liquids	500
Air comparison pycnometer	Solids	-
Oscillating densitometer	Liquids	5

4  
5 EU Test guideline A.3 for relative density Regulation (EC) No 440/2008 includes a list of  
6 standards with technical information about the different methods and actual measuring of  
7 different types of substances.

8 R.7.1.4.4 Adaptation of the standard testing regime

### 9 **Adaptation possibilities according to column 2 of Annex VII to REACH**

10 Column 2 of REACH Annex VII provides the following specific rules for adaptation of the  
11 standard information requirement for relative density:

12 *The study does not need to be conducted if:*

- 13 • *the substance is only stable in solution in a particular solvent and the solution density is*  
14 *similar to that of the solvent. In such cases, an indication of whether the solution*  
15 *density is higher or lower than the solvent density is sufficient; or*
- 16 • *the substance is gaseous at room temperature. In this case, an estimation based on*  
17 *calculation can be made from its molecular weight and the Ideal Gas Laws.*

18 For liquids, it is useful to have some indication of the dynamic viscosity as this can affect the  
19 choice of method. The physical state of test substances should always be homogeneous, this is  
20 particularly relevant for highly viscous substances where internal bubbles can be formed; in  
21 these cases, the test substance should be allowed to rest until all internal bubbles have  
22 disappeared.

23 The summary should include the numerical value for density and temperature at which it was  
24 measured, test material identity, purity of the sample used, physical state, method and  
25 guideline used and reference substance (if any).

### 26 **Adaptation possibilities according to Annex XI to REACH**

#### 27 **USE OF EXISTING DATA: DATA ON PHYSICAL-CHEMICAL PROPERTIES FROM** 28 **EXPERIMENTS NOT CARRIED OUT ACCORDING TO GLP OR THE TEST METHODS** 29 **REFERRED TO IN ARTICLE 13 (3) OF REACH**

30 If experimental data are available (study reports or literature data) meeting the criteria in  
31 section 1.1.1 of Annex XI to REACH, these could be used to meet the endpoint data  
32 requirements. If an estimation method is used as a source of information according to Column

2 of Annex VII, the QSAR model must meet the criteria set out in section 1.3 of Annex XI to REACH.

### **WEIGHT OF EVIDENCE**

Where no single source of existing data (study reports, QSAR, literature data) is considered sufficiently reliable, thus not fully meeting the criteria in section 1.1.1 of Annex XI to REACH, or where several sources of similar reliability with deviating results exist, a weight of evidence approach may be used. The criteria in section 1.2 of Annex XI to REACH must then be met<sup>12</sup>.

### **(Q)SAR**

(Q)SAR is generally not applicable for determination of relative density.

For this endpoint there are often experimental measurements and therefore QSPR models for this property have not received special attention in the environmental literature. Several software programs can be used to calculate the density of a given substance but the documentation and validation of the methods is limited.

### **GROUPING OF SUBSTANCES AND READ-ACROSS APPROACH**

For the determination of the relative density read-across is usually not possible. However interpolation may still be possible within homologous series.

### **TESTING IS TECHNICALLY NOT POSSIBLE**

Testing should always be considered, if none of the waiving possibilities applies. Waiving relative density testing on the basis of not being technically possible is not applicable.

### **Further adaptation possibilities**

Not foreseen.

#### **R.7.1.4.5 Impurities; uncertainties**

Impurities can have a significant influence on the density. This influence depends on the amount and density of the impurity; thus, the higher the amount of impurity and the higher the difference between the densities of the main component and the impurity, the higher the influence. Therefore utmost care should be taken in the selection of the key study(s), or weight-of-evidence approaches, that the data selected is representative of the substance being registered by the respective companies.

Density is temperature dependant. Whenever possible, determinations should be performed at 20 °C.

#### **R.7.1.4.6 Endpoint specific information in the registration dossier / in IUCLID**

### **Materials and methods**

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<sup>12</sup> The NIST have a useful statistical approach which has been used for the evaluation of literature data (ref.: <http://webbook.nist.gov/chemistry/site-cal.html#AVG>).

- type of method or reference to the standard or the test method applied.

## Results and discussion

- temperature (°C);
  - relative (for gases)/ absolute (for liquids and solids) density value (dimensionless);
  - measurement uncertainty if available;
  - if testing is waived, the reasons for waiving must be documented in the dossier.
- Any deviation from the guideline method used or any other special consideration should be reported. In cases where there is more than one source of data, the endpoint summary under results and discussion should provide a justification for the selection of the key study chapter.

## Reference to other ECHA Guidance Documents

Further detailed guidance on relative density can be found in the following chapters:

IUCLID Section	REACH Annex	Endpoint title	IUCLID 5 End User Manual Chapter	ECHA Practical Guide 3
4.4	VII 7.4	Relative density	E.4.5	3.3

## R.7.1.5 Vapour pressure

### R.7.1.5.1 Type of property

Vapour pressure is a property:

- for substance characterisation;
- which serves as a key parameter for assessing some toxicological and environmental hazards;
- which gives indications whether a substance may be available for inhalation as a vapour or may form flammable/explosive vapour-air mixtures;
- which allows determination of the volatility of a substance from an aqueous medium or soil, in terms of the Henry's Law constant (Appendix R.7.1-1) and partition coefficient air/soil, respectively;
- which allows determination of the right container/vessel to ensure safety during storage, transport and use;
- which is important for physical hazard assessment.

### R.7.1.5.2 Definition

The vapour pressure of a substance is defined as the saturation pressure above a solid or a liquid substance at constant temperature. At the thermodynamic equilibrium, the vapour pressure of a pure substance is a function of temperature only.

### R.7.1.5.3 Test method(s)

Method A.4 of Regulation (EC) 440/2008 or OECD Test Guideline 104 (Vapour pressure) should be used for testing. It is useful to have preliminary information on the structure, the melting point and the boiling point of the substance to perform this test.

There is no single measurement procedure applicable to the entire range of vapour pressure values. Therefore, several methods are recommended to be used for the measurement of vapour pressure from  $< 10^{-10}$  to  $10^5$  Pa. For the selection of the test method the scope and applicability specifications have to be taken into account. The results should be checked for consistency with other physical data like boiling point, flash point etc.

It is recommended to determine the vapour pressure at least for two temperatures, for volatile substances (boiling point up to 150 °C) preferably at 20 °C and at 50 °C.

Where standards are applicable, the use of the most recent updates is advised, please check section R.7.1.1.3 for further information.

### R.7.1.5.4 Adaptation of the standard testing regime

#### **Adaptation possibilities according to column 2 of Annex VII to REACH**

Column 2 of REACH Annex VII provides the following specific rules for adaptation of the standard information requirement for vapour pressure:

*The study does not need to be conducted if the melting point is above 300 °C.*

*If the melting point is between 200 °C and 300 °C, a limit value based on measurement or a recognised calculation method is sufficient.*

Vapour pressure testing is also not required for substances with a standard boiling point of  $< 30$  °C, as these substances will have vapour pressures above the limit of measurement (i.e.  $10^5$  Pa).

## 1 **Adaptation possibilities according to Annex XI to REACH**

### 2 **USE OF EXISTING DATA: DATA ON PHYSICAL-CHEMICAL PROPERTIES FROM** 3 **EXPERIMENTS NOT CARRIED OUT ACCORDING TO GLP OR THE TEST METHODS** 4 **REFERRED TO IN ARTICLE 13 (3) OF REACH**

5 If experimental data are available (study reports or literature data) meeting the criteria in  
6 section 1.1.1 of Annex XI to REACH, these could be used to meet the endpoint data  
7 requirements. If an estimation method is used as a source of information according to Column  
8 2 of Annex VII, the QSAR model must meet the criteria set out in section 1.3 of Annex XI to  
9 REACH.

## 10 **WEIGHT OF EVIDENCE**

11 Where no single source of existing data (study reports, QSAR, literature data) is considered  
12 sufficiently reliable, thus not fully meeting the criteria in section 1.1.1 of Annex XI to REACH,  
13 or where several sources of similar reliability with deviating results exist, a weight of evidence  
14 approach may be used. The criteria in section 1.2 of Annex XI to REACH must then be met.

## 15 **(Q)SAR**

16 For the determination of the vapour pressure, (Q)SAR approaches may be used if  
17 determination by experiment is not possible.

18  
19 The vapour pressure depends on the temperature. This dependence was modelled by Grain  
20 (Grain, 1982), based on thermodynamic principles. The estimation methods differ for vapour  
21 pressure that can be applied for compounds that are liquid or gaseous at the temperature of  
22 interest, and for solid and liquid compounds. The former can be estimated by the Antoine  
23 equation, while the latter could be predicted by the Watson correlation, which accounts also for  
24 the heat of vaporisation. Another method, described by Mackay *et al.* (1982), is applicable only  
25 for hydrocarbons and halogenated hydrocarbons. Further, the Grain model was modified to be  
26 applicable for all solids, liquids, and gases. These methods are still in practical use today.

27 The OECD guideline 104 reports that the Watson correlation is applicable over the pressure  
28 range from  $10^5$  Pa to  $10^{-5}$  Pa. It should in any case be pointed out that estimated values for  
29 vapour pressure can be subjected to great uncertainty if the computed pressure is lower than  
30 1 Pa, especially when the boiling point has not been experimentally determined (OECD  
31 monograph 67). The uncertainty is even greater if the estimated value is used together with  
32 water solubility in order to estimate the Henry's Law constant.

33 The environment monograph 67 of the OECD describes all of the above mentioned methods  
34 and the OECD guideline 104 supports the use of the Watson correlation for the calculation of  
35 vapour pressure, but does not specifically reject other calculation methods.

36 The handbook for estimating the physico-chemical properties of organic compounds (Reinhard  
37 and Drefahl, 1999) reports another method based on thermodynamic properties and  
38 elaborated by Mishra and Yalkowsky that discussed the application of the method of Mackay  
39 (Mackay *et al.*, 1982).

40 The equation by Mishra and Yalkowsky gave significantly better estimates than the method of  
41 Mackay on the same data set (Mishra and Yalkowsky, 1991).

42 Another methodology that proved to be effective in estimating vapour pressure relies on group  
43 contribution approaches. Several models using this strategy have been proposed (Reinhard  
44 and Drefahl, 1999; see Table R.7.1-3).

**Table R.7.1-3 Group contribution approach and vapour pressure**

Compounds	Authors	Methodology	Statistics
Alkyl aromatic compounds	Amidon and Anik	Group contribution approach	Standard error ... 1.1 kJ on the estimation for the free energy of vaporisation
Mono-, di-, tri- and tetra substituted	Hoshino et al.	Group contribution approach	Average error 3.7 % Max. Error 30.9 %
Perfluorinated saturated hydrocarbons	Kelly et al.	Group contribution approach	Arithmetic mean deviation < 0.5 %

Numerous other models are available for the estimation of vapour pressure, and Schwarzenbach *et al.* (1993), Delle Site (1996), Sage and Sage (2000) and Dearden (2003) have reviewed many of these. The descriptors used in vapour pressure QSPRs include physico-chemical, structural and topological descriptors, and group contributions. Katritzky *et al.* (1998) used 4 CODESSA descriptors to model the vapour pressure (in atmospheres at 25 °C) of 411 diverse organic chemicals, with  $r^2 = 0.949$  standard error = 0.331 log unit. A number of studies (Andreev *et al.* 1994, Kühne *et al.* 1997, Yaffe & Cohen 2001) allow of the estimation of vapour pressures over a range of temperatures.

## GROUPING OF SUBSTANCES AND READ-ACROSS APPROACH

For the determination of vapour pressure read-across is usually not possible. However interpolation may still be possible within homologous series.

## TESTING IS TECHNICALLY NOT POSSIBLE

Vapour pressure testing is not required for substances with a standard boiling point of < 30 °C, as these substances will have a vapour pressure value above the limit of measurement (i.e.  $10^5$  Pa).

For substances which decompose during measurement or which are unstable or explosive, determination of the vapour pressure may not be technically possible. This also applies to self-reactive substances and organic peroxides.

Pyrophoric substances may be difficult to handle experimentally. If fully inert conditions cannot be maintained during sample preparation and measurement, use of an appropriate calculation method is recommended.

A calculation method should also be applied in the case of some corrosive substances which would destroy essential metallic parts of the measurement apparatus.

## Further adaptation possibilities

Not foreseen.

### R.7.1.5.5 Impurities; uncertainties

Impurities can have a large influence on vapour pressure. The influence depends on the amount of the impurity and the vapour pressure of that impurity. Small amounts of volatile impurities may increase the vapour pressure by several orders of magnitude. This has to be kept in mind when performing the measurements and for the interpretation of results.

Therefore utmost care should be taken in the selection of the key study(s), or weight-of-evidence approaches, that the data selected is representative of the substance being registered by the respective companies.

Where there are volatile impurities in the sample which could affect the result, the substance may be purified. Test method A.4 states that it may also be appropriate to quote the vapour pressure for the technical material. However, in consideration of the large effect that impurities may have (see above), doing so is strongly discouraged.

#### R.7.1.5.6 Endpoint specific information in the registration dossier / in IUCLID

##### **Materials and methods**

- type of method or description of the apparatus or reference to the standard or the test method applied.

##### **Results and discussion**

- if testing is waived, the reasons for waiving must be documented in the dossier;
- measured value of the vapour pressure for at least two temperatures;
- estimate of the vapour pressure at 20 or 25 °C (if not measured at these temperatures);
- if a transition (change of state, decomposition) is observed, the following should be noted:
  - nature of change;
  - temperature at which change occurs.

Any deviation from the guideline method used or any other special consideration should be reported. In cases where there is more than one source of data, the endpoint summary under results and discussion should provide a justification for the selection of the key study chapter.

##### Reference to other ECHA Guidance Documents

Further detailed guidance on vapour pressure can be found in the following chapters:

IUCLID Section	REACH Annex	Endpoint title	IUCLID 5 End User Manual Chapter	ECHA Practical Guide 3
4.6	VII 7.5	Vapour pressure	E.4.7	3.5

#### R.7.1.5.7 References on vapour pressure

OECD Guidelines for the Testing of Chemicals / Section 1: Physical-Chemical properties, Test No. 104: Vapour Pressure, OECD Code: 979910401E1, July 2006.

Andreev N.N, Kuznetsov S.E, Storozhenko S.Y. (1994) Prediction of vapour pressure and boiling points of aliphatic compounds. Mendelev Commun. 173-174.

Grain C.F., (1982) Handbook of chemical property estimation methods. New York, Mc Graw-Hill

Delle Site A. (1996) The vapour pressure of environmentally significant organic chemicals: a review of methods and data at ambient temperature. J. Phys. Chem. Ref. Data 26:157-93.



- 1 Dearden JC. (2003) Quantitative structure-property relationships for prediction of boiling point,  
2 vapour pressure, and melting point. *Environ Toxicol Chem* 22(8):1696-709.
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- 6 Kühne R, Ebert RU, Schüürmann G. (1997) Estimation of vapour pressures for hydrocarbons  
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8 34:671-86.
- 9 Mackay D, Bobra A, Chan W, Shiu WY. (1982) Vapour pressure correlation for Low-Volatility  
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12 *Eng. Chem. Res.* 1991;30:1609-12.
- 13 OECD Guidelines for Testing of Chemicals, Method 104 "Vapour Pressure Curve"
- 14 Reinhard M, Drefahl (1999).A. Handbook for Estimating Physico-Chemical Properties of Organic  
15 Compounds. New York: Wiley.
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17 Raton, FL: Lewis.
- 18 Schwartzenbach, R.P., Gswend, P.M., Imboden, D.M. (1993). Environmental Organic  
19 Chemistry. John Wiley and Sons.
- 20 Yaffe D, Cohen, Y (2001) Neural network based temperature-dependent quantitative structure  
21 property relationships (QSPRs) for predicting vapour pressure of hydrocarbons. *J. Chem. Inf.*  
22 *Comput. Sci.* 41:463-477.
- 23

## 1 R.7.1.6 Surface tension

### 2 R.7.1.6.1 Type of property

3 Surface tension measurements of aqueous solutions are significant since decreasing the  
4 surface tension of water may impact on the properties of the solution and other  
5 physicochemical measurements.

### 6 R.7.1.6.2 Definition

- 7 • Surface tension:

8 *The free surface enthalpy per unit of surface area is referred to as surface tension* (Council  
9 Regulation (EC) No 440/2008).

10 The surface tension is given as: N/m (SI unit) or mN/m (SI sub-unit).  $1 \text{ N/m} = 10^3 \text{ dyne/cm}$   
11 or  $1 \text{ mN/m} = 1 \text{ dyne/cm}$  in the obsolete cgs system.

12 The surface tension of an aqueous solution of a substance can be used to determine whether  
13 the substance is surface active.

- 14 • Surface active substance (surfactant):

15 *'Surfactant' means any organic substance and/or preparation [mixture] used in detergents,*  
16 *which has surface-active properties and which consists of one or more hydrophilic and one or*  
17 *more hydrophobic groups of such a nature and size that it is capable of reducing the surface*  
18 *tension of water, and of forming spreading or adsorption monolayers at the water-air interface,*  
19 *and of forming emulsions and/or microemulsions and/or micelles, and of adsorption at water-*  
20 *solid interfaces* (see Article 2(6) of Council Regulation (EC) No 648/2004).

### 21 R.7.1.6.3 Test method(s)

22 Testing should be done in accordance with one of the methods specified under section A.5 of  
23 Regulation (EC) No 440/2008. These methods are applicable to most chemical substances.

24 It is useful to have preliminary information on the water solubility, the structure, the hydrolysis  
25 properties and the critical concentration for micelles formation of the substance before  
26 performing the test.

27 Surface tension measurements require a test material that is stable against hydrolysis during  
28 the test period and soluble in water at concentrations of  $> 1 \text{ mg/l}$ . Measurements should be  
29 performed on a solution at either 90 % of the solubility limit or  $1 \text{ g/l}$  (where viscosity permits),  
30 whichever is smaller.

### 31 R.7.1.6.4 Adaptation of the standard testing regime

## 32 Adaptation possibilities according to column 2 of Annex VII to REACH

33 Column 2 of REACH Annex VII provides the following specific rules for adaptation of the  
34 standard information requirement for surface tension:

35 *The study need only be conducted if:*

- 36 • *based on structure, surface activity is expected or can be predicted; or*
- 37 • *surface activity is a desired property of the material.*

38 *If the water solubility is below  $1 \text{ mg/l}$  at  $20^\circ \text{C}$  the test does not need to be conducted.*

## **Adaptation possibilities according to Annex XI to REACH**

### **USE OF EXISTING DATA: DATA ON PHYSICAL-CHEMICAL PROPERTIES FROM EXPERIMENTS NOT CARRIED OUT ACCORDING TO GLP OR THE TEST METHODS REFERRED TO IN ARTICLE 13 (3) OF REACH**

If experimental data are available (study reports or literature data) meeting the criteria in section 1.1.1 of Annex XI to REACH, these could be used to meet the endpoint data requirements. If an estimation method is used as a source of information according to Column 2 of Annex VII, the QSAR model must meet the criteria set out in section 1.3 of Annex XI to REACH.

### **WEIGHT OF EVIDENCE**

Where no single source of existing data (study reports, QSAR, literature data) is considered sufficiently reliable, thus not fully meeting the criteria in section 1.1.1 of Annex XI to REACH, or where several sources of similar reliability with deviating results exist, a weight of evidence approach may be used. The criteria in section 1.2 of Annex XI to REACH must then be met.

### **(Q)SAR**

At the time of writing, no reliable (Q)SAR methods exist for sufficiently accurate predictions of surface tension.

### **GROUPING OF SUBSTANCES AND READ-ACROSS APPROACH**

For the determination of the surface tension read-across is usually not possible. However interpolation may still be possible within homologous series.

### **TESTING IS TECHNICALLY NOT POSSIBLE**

Testing should always be considered, if none of the waiving possibilities applies. Testing may not be possible for reactive substances which react with water or air (hydrolyse, are pyrophoric, evolve gas, etc).

### **Further adaptation possibilities**

Not foreseen.

#### **R.7.1.6.5 Impurities; uncertainties**

For the measurement of surface tension the ring or plate tensiometer methods are preferred. The error on the measurement is in the order of 0.1–0.3 mN/m. Use of the standard protocols and GLP procedures are recommended. Surface active impurities in substances may in some cases lead to false-positive surface tension measurements.

#### **R.7.1.6.6 Endpoint specific information in the registration dossier / in IUCLID**

##### **Materials and methods**

- description of the apparatus and dimensions or reference to the standard or the test method applied;

- test material identity: apart from general issues, if surface tension of active impurities affects results, it should be noted.

### Results and discussion

- surface tension value and unit (preferably mN/m or N/m but other units are also acceptable);
- concentration of the solution (as indicated in test A.5. Surface tension described in Council Regulation (EC) No 440/2008);
- age of solution;
- type of water or solution used;
- results from repeated measurements with varied equilibrium time (of the solution);
- several measurement results should be provided to assess the possible timedependency of the measurement. Equilibration times may vary from minutes to hours. Measurements should be sufficient to prove that a constant surface tension was reached;
- if testing is waived, the reasons for waiving must be documented in the dossier.

Any deviation from the guideline method used (and reasons for it) or any other special consideration should be reported. In cases where there is more than one source of data, the endpoint summary under results and discussion should provide a justification for the selection of the key study chapter.

### Reference to other ECHA Guidance Documents

Further detailed guidance on surface tension can be found in the following chapters:

IUCLID Section	REACH Annex	Endpoint title	IUCLID 5 End User Manual Chapter	ECHA Practical Guide 3
4.10	VII 7.6	Surface tension	E.4.11	3.8

### R.7.1.7 Water solubility

Advice to registrants with regard to nanomaterials characterisation of water solubility can be found in Appendix R7-1 Recommendations for nanomaterials applicable to: Chapter R7a Endpoint specific guidance, section 2.2.1 Water solubility.

#### R.7.1.7.1 Type of property

Water solubility is a significant parameter for a number of reasons:

- the mobility of a test substance is largely determined by its solubility in water. In general, highly soluble substances are more likely to be distributed by the hydrological cycle;
- water soluble substances gain access to humans and other living organisms;
- knowledge of the water solubility is a prerequisite for setting up test conditions for a range of fate (e.g. biodegradation, bioaccumulation) and effects studies;
- it is also used to derive other environmental parameters, such as  $K_{ow}$ ,  $K_{oc}$  and Henry's Law Constant (Appendix R.7.1-1). It is also used as input for some QSAR models;
- water solubility is used as a regulatory trigger for waiving certain physicochemical and ecotoxicological endpoints.

#### R.7.1.7.2 Definition

*The solubility of a substance in water is specified by the saturation mass concentration of the substance in water at a given temperature. The solubility in water is specified in units of mass per volume of solution. The SI unit is  $\text{kg/m}^3$  (grams per litre may also be used) (see Regulation (EC) No 440/2008, A.6, section 1.2).*

Mixtures of organic compounds, e.g. petroleum substances, behave differently from their single constituent compounds when brought into contact with water. Petroleum substances are typically hydrophobic and exhibit low solubility in water. However, reflecting the range of structures, constituent hydrocarbons will exhibit a wide range of water solubility. Therefore, water solubility measurements for these substances are loading rate dependent due to their complex composition. This water solubility behaviour impacts on both the conduct and interpretation of aquatic toxicity tests for these complex substances. The complex composition, and generally low water solubility, impact also on the choice and conduct of biodegradation studies.

Consequently, the above definition for solubility of a single substance in water is not applicable to substances which are multi-component, such as multi-constituent or UVCB substances, i.e. complex substances. The usually accepted meaning of 'solubility' in such cases is 'the composition of the aqueous solution formed at equilibrium under a defined set of conditions'. Temperature and the amount of substance added per unit volume of water (i.e. the 'loading') are the main factors to consider. It may not always be possible to establish that equilibrium of all components has been achieved; in these cases, time and type of agitation of the test vessels must also be described.

Similar testing issues also apply to inorganic compounds. Water solubility among compounds of the same metal may differ by several orders of magnitude. Differences in the solubility of metal compounds are related to the metal species and the characteristics of the aqueous medium. Highly soluble inorganic metal compounds can be assessed through the normal procedures. For sparingly soluble metal compounds, a solubility product can be calculated thermodynamically (e.g. by using the Facility for Analysis of Chemical Thermodynamics ("F\*A\*C\*T", FACT-Win version 3.05). Although metals are generally insoluble, metals in the

1 elemental state may react with water or a dilute aqueous electrolyte to form soluble or  
2 sparingly soluble cationic or anionic products. During this process the metal will oxidise, or  
3 transform, from the neutral or zero oxidation state to a higher oxidation state. The OECD Test  
4 Guidance on transformation/dissolution of metals and sparingly soluble metal compounds  
5 (OECD, 2001) can be used to determine the rate and extent to which metals and sparingly  
6 soluble metal compounds can produce soluble bioavailable ionic and other metal-bearing  
7 species in aqueous media under a set of standard laboratory conditions representative of those  
8 generally occurring in the environment. The outcomes of the transformation/dissolution tests  
9 are to be used for aquatic environmental hazard classification purposes.

#### 10 R.7.1.7.3 Test method(s)

11 No single method is available to cover the whole range of solubility values in water, from  
12 relatively soluble to very low soluble substances. General test guidelines (OECD Method 105;  
13 EU Method A.6, Regulation (EC) No 440/2008) include two test methods which cover the whole  
14 range of solubility values but are not applicable to volatile substances. Water solubility  
15 determinations are normally run at 20 °C in distilled water according to standard test  
16 guidelines (OECD Method 105; EU Method A.6). Solubility data determined using these  
17 standard physico-chemical guidelines may differ if the test material is solubilised in either  
18 aqueous solutions containing salts or at different test temperatures (or both) (e.g.  
19 ecotoxicological test media).

20 The methods should be applied to essentially pure substances that are stable in water. Details  
21 of suitable methods are shown in Table R.7.1-4.

22 A number of standardised methods are available for the determination of single substances  
23 and complex mixtures of liquids and solids. For metals and sparingly soluble inorganic metal  
24 compounds a specific water solubility approach was designed to measure transformation to the  
25 dissolved fraction under standard conditions. The test methods are not applicable to volatile  
26 substances. Care should be taken to ensure that the test substances examined are as pure as  
27 possible and their solubility levels are determined analytically using a specific analytical  
28 method wherever possible. Precautions should be taken to minimise degradation of the test  
29 substance, in particular if long periods of equilibration are required (e.g. 'slow stir' methods).

30 Measurement of water solubility does not usually impose excessive demands on chemical  
31 techniques. However, measurement of the solubility of sparingly soluble compounds requires  
32 extreme care to generate saturated solutions of the material without the introduction of  
33 dispersed material; invariably specific methods of analysis are able to determine the low levels  
34 (sub ppb-ppm) in solution. Reported water solubility data for such compounds can often  
35 contain appreciable errors.

1 **Table R.7.1-4 Test methods for the determination of water solubility**

Method details	Applications and requirements	Repeatability and sensitivity
<p>Column elution method</p> <p>Based on elution of the test substance with water from a micro-column which is charged with an inert carrier material such as glass beads, silica gel or sand and an excess of test substance. The water solubility is determined when the mass concentration of the eluate is constant.</p> <p>The mass concentration of the test substance is determined analytically</p>	<p>Applicable to essentially pure substances only</p> <p>Used for low solubilities (<math>&lt; 10^{-2}</math> g/l)</p> <p>Organic substances, but not mobile oils or liquids</p>	<p><math>&lt; 30 \%</math> ; down to <math>1\mu\text{g/l}</math></p>
<p>Flask method</p> <p>The test substance is dissolved in water at a temperature somewhat above the test temperature. When saturation is achieved the mixture is cooled and kept at the test temperature, stirring as long as necessary to reach equilibrium</p> <p>The mass concentration of the test substance is determined analytically</p>	<p>Applicable to essentially pure substances and also complex substances.</p> <p>Use of fast stirring techniques (300-400 rpm) appropriate for higher solubility (<math>&gt; 10^{-2}</math> g/l) test substances.</p> <p>Use of slow-stirring techniques (<math>&lt; 100</math> rpm) appropriate for low solubility (<math>&lt; 10^{-2}</math> g/l) test substances (Letinski et al, 2002)</p> <p>Requires equilibration study to determine the time taken to equilibrate the test substance and water</p>	<p><math>&lt; 15 \%</math> ; down to <math>1\mu\text{g/l}</math></p>
<p>OECD series on Testing and Assessment Number 29 - Guidance Document on Transformation/Dissolution of Metals and Metal Compounds in Aqueous media.</p>	<p>Applicable to all metals and sparingly soluble inorganic metal compounds</p>	<p>/</p>

2

3 R.7.1.7.4 Adaptation of the standard testing regime

4 **Adaptation possibilities according to column 2 of Annex VII to REACH**

5 Column 2 of REACH Annex VII provides the following specific rules for adaptation of the  
6 standard information requirement for water solubility:

7 *The study does not need to be conducted if:*

- 8     • *the substance is hydrolytically unstable at pH 4, 7 and 9 (half-life less than 12 hours);*  
9         *or*
- 10     • *the substance is readily oxidisable in water; or*
- 11     • *the substance is flammable in contact with water.*

12 *If the substance appears 'insoluble' in water, a limit test up to the detection limit of the*  
13 *analytical method shall be performed.*



For ionising substances, the pH-dependence of the water solubility should be known. At least the pH of the test water needs to be identified. In the context of marine risk assessment, when the  $pK_a$  is close to 8 it may be necessary to obtain realistic measurements using seawater.

For volatile compounds, it can be useful to have information on the vapour pressure.

## Adaptation possibilities according to Annex XI to REACH

### USE OF EXISTING DATA: DATA ON PHYSICAL-CHEMICAL PROPERTIES FROM EXPERIMENTS NOT CARRIED OUT ACCORDING TO GLP OR THE TEST METHODS REFERRED TO IN ARTICLE 13 (3) OF REACH

Most physical properties, such as molecular weight, melting point, boiling point, density and water solubility can be obtained from commonly used environmental Handbooks, such as Verschueren's Handbook of Environmental Data on Organic Chemicals (1983), Howard's Handbook of Environmental Fate and Exposure Data, Vol. I and II (1990), Lide's CRC Handbook of Physics and Chemistry, Lange's Handbook of Chemistry, the Merck Index, the Aldrich Catalog, Kirk-Othmer Encyclopaedia of Chemical Technology and other handbook compilations such as Riddick *et al.* (1986).

Alternatively, searching on various environmental databases, such as HSDB (<http://www.toxnet.nlm.nih.gov/cgi-bin/sis/htmlgen?HSDB>), will provide summaries of chemical and physical properties of substances.

It is not unusual to find in the literature a wide range of solubilities for the same product. The oldest literature generally yields the highest solubility values: this is due to the fact that products were originally not as pure as they are nowadays and also non-specific methods were used which would not differentiate between the dissolved product and any impurities. Reported water solubility data for such compounds can often contain appreciable errors. Therefore, the reliability of data must be demonstrated by providing information on the identity and purity of the test substance, the methodology used to make the measurement, and whether or not this was performed to GLP standards.

If experimental data are available (study reports or literature data) meeting the criteria in section 1.1.1 of Annex XI to REACH, these could be used to meet the endpoint data requirements. If an estimation method is used as a source of information according to Column 2 of Annex VII, the QSAR model must meet the criteria set out in section 1.3 of Annex XI to REACH.

### WEIGHT OF EVIDENCE

Secondary data sources can be used in a WoE approach and they can collectively support the choice of a specific value for the water solubility. These secondary sources have to be based on a critical evaluation of peer-reviewed data and a consequent selection of a reliable and representative value for the water solubility. The use of Klimisch codes, can be extended to these secondary sources and a reliability code of (2) valid with restrictions should be assigned when using an authoritative secondary source.

Where no single source of existing data (study reports, QSAR, literature data) is considered sufficiently reliable, thus not fully meeting the criteria in section 1.1.1 of Annex XI to REACH, or where several sources of similar reliability with deviating results exist, a weight of evidence approach may be used. The criteria in section 1.2 of Annex XI to REACH must then be met.

## (Q)SAR

For an organic solute to dissolve in water, firstly, the solute molecules must be separated from one another. Secondly, the solvent molecules must become partially separated from one another to create a cavity large enough to accommodate the solute. Once the solute occupies the cavity, there will be new attractive forces between solute and solvent. Finally, the water molecules in the solvation shell will form extra H-bonds to neighbouring water molecules. Thus, the water solubility depends not only on the affinity of a solute for water, but also on its affinity for its own structure. Molecules that are strongly bound to each other require considerable energy to separate them. This also means that such compounds have high melting points (for solids). Generally, solids with a high-melting temperature have poor solubility in any solvent.

Removal of a molecule from its crystal lattice means an increase in entropy, and this can be difficult to model accurately. For this reason, as well as the fact that the experimental error on solubility measurements can be quite high (generally reckoned to be about 0.5 log unit), the prediction of aqueous solubility is not as accurate as is the prediction of octanol/water partitioning. Nevertheless, many papers (Dearden 2006) and a book (Yalkowsky & Banerjee 1992) have been published on the prediction of aqueous solubility, as well as a number of reviews (Lyman 1990, ECETOC 1998, Reinhard & Drefahl 1999, Mackay 2000, Schwarzenbach *et al.* 2003, Dearden 2006). There are also a number of software programs available for that purpose (ECETOC 2003, Dearden 2006). Livingstone (2003) has discussed the reliability of aqueous solubility predictions from both QSPRs and commercial software.

It should be noted that there are various ways that water solubilities can be reported: in pure water, at a specified pH, at a specified ionic strength, as the undissociated species (intrinsic solubility), or in the presence of other solvents or solutes. Solubilities are also reported in different units, for example g/100 ml, mole/litre, mole fraction. The use of mole/litre is recommended, as this provides a good basis for comparison.

For solids, work has to be done to remove molecules from their crystal lattice, and the simplest way to account for this is to use what Yalkowsky and co-workers have termed the general solubility equation (GSE), which incorporates a melting point term to account for the behaviour of solids (Sanghvi *et al* 2003):

$$\log S_{\text{aq}} = 0.5 - \log K_{\text{ow}} - 0.01(\text{MP} - 25)$$

where MP is the melting point (°C). The melting point term is taken as zero for compounds melting at or below 25°C. Calculated  $\log K_{\text{ow}}$  and MP values can be used in the GSE, although measured values are preferred. Aqueous solubilities of 1026 non-electrolytes, with a  $\log S_{\text{aq}}$  range of -13 to +1 (S in mole L<sup>-1</sup>), calculated with the GSE had a standard error of 0.38 log unit.

Good predictions for a large diverse data set have been obtained by the use of linear solvation energy descriptors (Abraham & Le 1999). These included two terms for polarity/polarisability, the sums of hydrogen bond donor and acceptor abilities of the solute molecule, and an expression of molecular volume

According to the Abraham and Le equation, the main factors controlling aqueous solubility seem to be hydrogen bond acceptor ability and molecular size, both of which are important elements in the molecular mechanisms of solubility.

Solubility can vary considerably with temperature, and it is important that solubility data are reported at a given temperature.

## 1 **GROUPING OF SUBSTANCES AND READ-ACROSS APPROACH**

2 For the determination of the water solubility read-across is usually not possible. However  
3 interpolation may still be possible within homologous series.

## 4 **TESTING IS TECHNICALLY NOT POSSIBLE**

5 For this endpoint, testing should almost always be possible and water solubility should usually  
6 be determined experimentally. Nonetheless, testing by the flask method might be precluded  
7 when the high viscosity of the saturated test solutions prevent from normal stirring. If it is  
8 technically not possible to conduct the study as a consequence of the properties of the  
9 substance (e.g. substances flammable in contact with water or substances readily oxidisable in  
10 water), testing may be omitted according to general rules for adaptation of the standard  
11 testing regime described in REACH Annex XI, Section 2.

### 12 **Further adaptation possibilities**

13 Not foreseen. However, for complex substances the information obtained from such testing is  
14 not relevant or of practical use, and therefore conducting the test may be waived where the  
15 data is irrelevant for subsequent assessments.

#### 16 **R.7.1.7.5 Impurities; uncertainties**

17 The water solubility of the test substance can be considerably affected by the presence of  
18 impurities.

19 For a complex substance, the measured solubility is dependent on the amount of test  
20 substance added. In practical terms, solubility data are generated using at least two loading  
21 rates (e.g. 100 mg/l and 1000 mg/l). Accuracy in determining water solubility decreases as the  
22 water solubility of a test substance is reduced (e.g. as shown for reference substance data in  
23 OECD Method 105). When dealing with test substances with water solubilities of the order of <  
24 10 µg/l, precautions need to be taken to avoid the introduction of dispersed material into the  
25 final extract.

26 Therefore utmost care should be taken in the selection of the key study(s), or weight-of-  
27 evidence approaches, that the data selected is representative of the substance being  
28 registered by the respective companies.

#### 29 **R.7.1.7.6 Endpoint specific information in the registration dossier / in IUCLID**

##### 30 **Materials and methods**

- 31 • description of the apparatus and dimensions or reference to the standard or the test method  
32 applied;
- 33 • results from preliminary test (if any);
- 34 • chemical identity and impurities (preliminary purification step, if any);
- 35 • water temperature during saturation process;
- 36 • analytical method employed;
- 37 • any evidence of chemical instability;
- 38 • all information relevant for the interpretation of the results.

##### 39 **If Column Elution method:**

- concentrations, flow rates and pH for each sample;
- mean and standard deviation of five samples at least;
- average for each of two successive runs at least;
- nature and loading of support material;
- solvent used.

**If Flask method:**

- pH of each sample;
- individual analytical determinations and the average;
- average of the values for different flasks.

**Results and discussion & Applicant's summary and conclusion**

- water solubility in (mg/l) at temperature (°C);
  - pH value and concentration of test substance;
  - description of solubility (if relevant);
  - if testing is waived, the reasons for waiving must be documented in the dossier.
- Any deviation from the guideline method used or any other special consideration should be reported. In cases where there is more than one source of data, the endpoint summary under results and discussion should provide a justification for the selection of the key study chapter.

**Reference to other ECHA Guidance Documents**

Further detailed guidance on water solubility can be found in the following chapters:

IUCLID Section	REACH Annex	Endpoint title	IUCLID 5 End User Manual Chapter	ECHA Practical Guide 3
4.8	VII 7.7	Water solubility	E.4.9	3.7

1 R.7.1.7.7 References on water solubility

- 2 Abraham M.H. and Le J. (1999) The correlation and prediction of the solubility of compounds in  
3 water using an amended solvation energy relationship. *J. Pharm. Sci.* 88, 868-880.
- 4 Dearden J.C. (2006) *In silico* prediction of aqueous solubility. *Expert Opinion on Drug*  
5 *Discovery* 1, 31-52.
- 6 EC Method A6. "Water Solubility", Dir 92/69/EEC, Official Journal of the European  
7 Communities, O.J. L383 A)
- 8 EC Method A7. "Hydrolysis ", Dir 92/69/EEC, Official Journal of the European Communities,  
9 O.J. L383 A)
- 10 ECETOC Technical Report No. 74: *QSARs in the Assessment of the Environmental Fate and*  
11 *Effects of Chemicals*. ECETOC, Brussels, 1998.
- 12 ECETOC Technical Report No. 89: *(Q)SARs: Evaluation of the Commercially Available Software*  
13 *for Human Health and Environmental Endpoints with Respect to Chemical Management*  
14 *Applications*. ECETOC, Brussels, 2003.
- 15 Livingstone D.J. (2003) Theoretical property predictions. *Current Topics in Med. Chem.* 3,  
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- 17 Letinski, D.J., Connolly, M.J., Peterson, D.R. and Parkerton, T.F. (2002) "Slow-stir water  
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- 19 Mackay D. Solubility in water. In Boethling R.S. and Mackay D. (Eds.), *Handbook of Property*  
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21 2000, pp. 125-139.
- 22 OECD Environmental Health and Safety Publications, 2000. Number 23. Guidance document on  
23 aquatic toxicity testing of difficult substances and mixtures.
- 24 OECD Environment, Health and Safety Publications, 2001. Series on Testing and Assessment,  
25 No. 29, Guidance Document on Transformation/Dissolution of Metals and Metal Compounds in  
26 Aqueous Media
- 27 OECD Guidelines for Testing of Chemicals – Method 105 "Water Solubility"
- 28 OECD Guidelines for Testing of Chemicals – Method 111 "Hydrolysis as a Function of pH"
- 29 Reinhard M. and Drefahl A. *Handbook for Estimating Physico-chemical Properties of Organic*  
30 *Compounds*. Wiley, New York, 1999.
- 31 Sanghvi T., Jain N., Yang G. and Yalkowsky S.H. (2003) Estimation of aqueous solubility by the  
32 general solubility equation (GSE) the easy way. *QSAR Comb. Sci.* 22, 258-262.
- 33 Schwarzenbach R.P., Gschwend P.M. and Imboden D.M. (2003) *Environmental Organic*  
34 *Chemistry*, 2<sup>nd</sup> edition, Wiley, Hoboken, NJ.
- 35 Yalkowsky S.H. and Banerjee S. (1992). *Aqueous Solubility: Methods of Estimation for Organic*  
36 *Compounds*. Marcel Dekker, New York.

## 1 **R.7.1.8 Partition coefficient n-octanol/water**

2 Advice to registrants with regard to nanomaterials characterisation of water solubility can be  
3 found in Appendix R7-1 Recommendations for nanomaterials applicable to: Chapter R7a  
4 Endpoint specific guidance, section 2.2.2 Partition coefficient n-octanol/water.

### 5 R.7.1.8.1 Type of property

6 The n-octanol/water partition coefficient ( $K_{ow}$ ) is one of the key physicochemical parameters,  
7 and it is used in numerous estimation models and algorithms for environmental partitioning,  
8 sorption, bioavailability, bioconcentration, bioaccumulation and also human toxicity and  
9 ecotoxicity. As such  $K_{ow}$  is a critical parameter for chemical safety assessment, classification  
10 and labelling, and PBT assessment/screening (where required).

11 The generation of a  $K_{ow}$  value is required at all tonnage bands (i.e. > 1 t/y; information  
12 requirements according to REACH Annexes VII-X).

### 13 R.7.1.8.2 Definition

14 The n-octanol/water partition coefficient ( $K_{ow}$ ) is defined as the ratio of the equilibrium  
15 concentrations of a dissolved substance in a two-phase system consisting of the largely  
16 immiscible solvents n-octanol and water. The property is moderately temperature-dependent  
17 and typically measured at 25 °C. For further information on definition and units please see the  
18 Test Methods Regulation ((EC) No 440/2008), guideline A.8, section 1.2.

### 19 R.7.1.8.3 Test method(s)

20 EU test guideline A.8 of the Test Methods Regulation ((EC) No 440/2008) describes two test  
21 procedures; a direct measurement via the Shake Flask method (OECD Test Guideline 107) and  
22 a correlation approach using the HPLC method (OECD Test Guideline 117). The Shake Flask  
23 method falls within the logKow range -2 to 4 and the HPLC method within the range 0 to 6.  
24 The applicability of the methods differ depending on the substance type and the amount of  
25 impurities in the test substance. Neither of the methods is applicable to surface active  
26 materials, for which an estimated value based on individual solubilities, or a calculated value  
27 along with calculation details should be provided. As with any endpoint and predictive method,  
28 the documentation and training set of the predictive method should be examined carefully to  
29 decide whether it is applicable to special categories of substances, such as zwitterionic or  
30 surface active substances.

31 Regardless of the method used, highly accurate measurements of  $\log K_{ow} > \sim 5$  are  
32 complicated by the fact that small amounts of octanol are entrained in the aqueous phase,  
33 leading to a potential underestimation of the measured  $\log K_{ow}$  values. All of the direct  
34 methods for measuring  $\log K_{ow}$  require quantifying the test material in either octanol or water  
35 and preferably in both matrices.

36 In addition, the OECD test guideline 123, Slow-stirring method, can be used to generate data  
37 for this endpoint.

38

1 **Table R.7.1-5 Methods for determination of partition coefficient n-octanol/water**

Method details	REPEATABILITY	APPLICABILITY RANGE
<p>Shake Flask Method (EU A.8, OECD TG 107)</p> <p>The Shake Flask method is the default procedure. It is considered to give accurate results for low to medium hydrophobic substances. For substances with a high expected log <math>K_{ow}</math>, alternative methods are recommended. A suitable analytical method is needed to determine the concentration of the test material in the octanol and water phases. By applying mass balance considerations, it may be possible to measure the test material in only the less-soluble phase. However, this approach significantly decreases the reliability in the reported value.</p> <p>This technique is not suitable for surface active compounds (surfactants), or compounds that hydrolyse rapidly.</p>	Three replicates should fall within +/- 0.3 log $K_{ow}$	$-2 < \log K_{ow} < 4$
<p>HPLC Method (EU A.8, OECD TG 117)</p> <p>This is a relatively quick way of estimating log <math>K_{ow}</math>. It is not measured directly, but from a correlation between log k (capacity factor) and log <math>K_{ow}</math> for a series of reference substances. It therefore depends on the quality of the log <math>K_{ow}</math> measurement of reference substances (often measured by the shake flask method). A series of reference compounds with similar chemical functionality to the test material should be used to generate the log k: log <math>K_{ow}</math> correlation. In general, the HPLC method is less sensitive to impurities than the shake flask method. The RP-HPLC is not recommended for strong acids and bases, metal complexes or surface active agents, or for measurements across very different classes of substances. The HPLC method is also very suitable for measuring the <math>K_{ow}</math> of mixtures of chemical homologues.</p>	Three replicates should fall within +/- 0.1 log $K_{ow}$	$0 < \log K_{ow} < 6$
<p>Slow-Stirring Method (OECD TG 123)</p> <p>This is a more recent method developed as an alternative to the shake flask procedure (OECD TG 107, EU A.8). The advantage of slow stirring versus shaking is that emulsion formation will be reduced. The method requires a few days to reach equilibrium. The method may be difficult to adapt to a high throughput approach. As with the other direct methods, a suitable analytical method is needed to measure the concentration of the test material in the octanol and water phases.</p> <p>NB: Radiolabelled substances – which may be synthesised for use in other tests – can be very useful for accurate log <math>K_{ow}</math> determination.</p>	Intralaboratory median standard deviation from 0.15 – 0.3 Log $K_{ow}$ (Tolls et al, 2003).	Validation has shown that this method can also be used for very hydrophobic substances, up to Log $K_{ow}$ 8.3 (OECD 2003, Tolls <i>et al</i> , 2003).
<p>Estimation method based on individual solubilities in EU A.8</p> <p>This method enables partition coefficients to be estimated based on the ratio of the solubility of the material in octanol and water. For some</p>		



substances (e.g. some surfactants and pigments) it is technically not feasible (or good practice) to measure an octanol-water partition coefficient by OECD 107. For such substances it may be possible to obtain a ratio of the saturated water solubility (OECD 105) and saturated octanol solubility (no guideline currently available but based on the principles of OECD 105). This method however has the drawback that it does not include the interaction between the water and solvent phase (i.e. a substance with high K <sub>ow</sub> is rather 'pushed out of the water' than 'pulled into octanol'). This explains the poor correlation typically observed between octanol solubility and K <sub>ow</sub> (Dearden, 1990, Sijm <i>et al.</i> , 1999). The ratio was found to be somewhat more representative if one uses octanol/saturated water and water/saturated octanol.		
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As such, a ratio estimation would be a less preferred yet acceptable alternative for the octanol/water partition coefficient (K<sub>ow</sub>), but must be treated with caution as it would not have been derived in the same manner as other K<sub>ow</sub>s (OECD TG 107).

1

## 2 R.7.1.8.4 Adaptation of the standard testing regime

3 **Adaptation possibilities according to column 2 of Annex VII to REACH**

4 Column 2 of REACH Annex VII provides the following specific rules for adaptation of the  
5 standard information requirement for n-octanol water partition coefficient:

6 *The study does not need to be conducted if the substance is inorganic. If the test cannot be*  
7 *performed (e.g. the substance decomposes, has a high surface activity, reacts violently during*  
8 *the performance of the test or does not dissolve in water or in octanol, or it is not possible to*  
9 *obtain a sufficiently pure substance), a calculated value for log P as well as details of the*  
10 *calculation method shall be provided.*

11 If experimental testing including estimation from the individual solubilities is not possible, log  
12 K<sub>ow</sub> must normally be calculated by an appropriate numeric method based on the molecule's  
13 structure.

14  
15 In case of rapid hydrolysis the registrant needs to provide evidence in the form of a hydrolysis  
16 endpoint study record (study summary) and should consider testing for the hydrolysis products  
17 instead, as information on the properties of (environmentally and toxicologically) relevant  
18 degradation products are needed for conducting the risk assessment of the substance to be  
19 registered.

20 **Adaptation possibilities according to Annex XI to REACH**

21 The reporting of the K<sub>ow</sub> information cannot usually be waived (except for inorganic  
22 substances), because it is essential for CSA, classification and labelling and PBT assessments.

## **USE OF EXISTING DATA: DATA ON PHYSICAL-CHEMICAL PROPERTIES FROM EXPERIMENTS NOT CARRIED OUT ACCORDING TO GLP OR THE TEST METHODS REFERRED TO IN ARTICLE 13 (3) OF REACH**

Log  $K_{ow}$  is a commonly documented property in substance databases, such as IUCLID (<http://ecb.jrc.it>). Additional sources are the Canadian National Committee for CODATA (CNC/CODATA) database with evaluated log  $K_{ow}$  values for over 20000 substances (<http://logkow.cisti.nrc.ca/logkow/>) and the QSAR Toolbox (<http://www.qsartoolbox.org>).

If experimental data are available (study reports or literature data) meeting the criteria in section 1.1.1 of Annex XI to REACH, these could be used to meet the endpoint data requirements. If an estimation method is used as a source of information according to Column 2 of Annex VII, the QSAR model must meet the criteria set out in section 1.3 of Annex XI to REACH.

## **WEIGHT OF EVIDENCE**

Measured values are given precedence over calculated values. For organic substances experimentally derived high-quality  $K_{ow}$  values, or values which are evaluated in reviews and assigned *recommended values*, are preferred over other determinations of  $K_{ow}$ . Where no single source of existing data (study reports, QSAR, literature data) is considered sufficiently reliable, thus not fully meeting the criteria in section 1.1.1 of Annex XI to REACH, or where several sources of similar reliability with deviating results exist, a weight of evidence approach may be used. The criteria in section 1.2 of Annex XI to REACH must then be met.

## **(Q)SAR**

When no experimental data of high quality are available, or if experimental methods are known to be unreliable, valid (Q)SARs for log  $K_{ow}$  may be used e.g. in a weight-of-evidence approach. Due to the availability of large number of measured log  $K_{ow}$  values and robust QSAR models for this property, the QSARs can, in some cases, predict the partition coefficient of a molecule with higher accuracy compared to a single test. Such valid QSAR models may be used if they are restricted to substances for which their applicability is well characterised. In order to be used as a stand alone source of values to meet the data requirements of Annex VII, 7.8, the QSARs must meet the criteria set out in Annex XI, 1.3.

## **GROUPING OF SUBSTANCES AND READ-ACROSS APPROACH**

For the determination of the partition coefficient n-octanol/water read-across is usually not possible. However interpolation may still be possible within homologous series.

## **TESTING IS TECHNICALLY NOT POSSIBLE**

Testing should always be considered, if none of the waiving possibilities applies.

## **Further adaptation possibilities**

Not foreseen.

### **R.7.1.8.5 Impurities; uncertainties**

The effect of impurities in the test substance are discussed in the referenced test guidelines.

1 **Difficult to test substances:**

2 There are certain structural or physico-chemical properties that can make the accurate  
3 determination of  $K_{ow}$  or its measurement difficult. Difficult to test substances include poorly  
4 soluble, volatile, surface active, ionisable substances, mixtures of substances, as well as  
5 substances subject to rapid degradation due to such processes as phototransformation,  
6 hydrolysis, oxidation, or biotic degradation.  
7

8 **Guidance on regulatory compliant  $K_{ow}$  determination for ionisable substances and salts:**

9 The  $K_{ow}$  is typically defined as the partition coefficient of the neutral, undissociated form of a  
10 substance. However, the relative extent to which an ionisable substance is likely to be  
11 dissociated in the environment (with pH usually in the range 5-9) can have a marked effect on  
12 its physicochemical properties, especially the octanol-water partition coefficient and water  
13 solubility, which in turn affect fate and behaviour. As log  $K_{ow}$  is routinely used to predict  
14 bioconcentration/bioaccumulation potential, this aspect is especially important in a PBT  
15 context. For substances which dissociate within an environmentally relevant pH range ( $pK_a$  5-  
16 9), values for  $K_{ow}$  must be derived for the neutral form, and preferably also for the dissociated  
17 form. In some cases a factor 4-5 has been recorded between the log  $K_{ow}$  of both species. The  
18 value for the dissociated molecule determined around a pH of 7 (sometimes referred to as  
19 Dow) is considered more realistic for PBT and chemical safety assessment.  
20

21 **Based on practical experience the following guidance is provided:**

22 **Simple acids and bases in the normal pH range:**

- 23
- 24 • The HPLC method is to be applied to acids and bases in their non-ionised forms,  
although the pH should be kept in the range 2 to 9 (however pH 5 to 9 is preferred).
  - 25 • For the shake-flask method, the approach must be followed in which the study is  
26 conducted at a pH where the substance is not ionised, if possible, or at a pH where the  
27 extent of ionisation is minimised.
  - 28 • Validated QSAR estimations may be useful for acids and bases.

29 **Zwitterionic substances:**

- 30
- 31 • For zwitterions, the shake-flask method should be used to develop a valid  $K_{ow}$  value.  
32 Even if the ionic charge pattern of the compound in octanol is not known, the value  
33 represents a practical and useful parameter. It is not justifiable to expect a full  
34 description of all the equilibria in both water and octanol. The pH of such a study should  
35 be 7 or the iso-electric point (pH value at which the molecule has no net electrical  
36 charge), as long as that point is in the range pH 5 to 9, so as to maximise the  
possibility of partition into octanol. There is no need to give both pH values.
  - 37 • The HPLC method must not be used. The usual estimation methods should be valid, but  
38 particular care should be exercised.
  - 39 • QSAR estimations may be useful provided that they are validated.

40 **Salts of organic compounds:**

- 41
- 42 • The shake-flask method should be used, usually at pH 7, or at any pH in the range 5 to  
43 9 which maximises the potential for partition into octanol. For salts, the nature of the  
44 analytical method compared to the chemical composition will have to be considered.  
45 The ideal is to monitor cation and anion\*\* individually in both phases. When only one  
46 half can be analysed, then the result must be understood as partial, even if it is the  
best that is achievable.

- Estimation by HPLC is not valid for the whole salt.
- QSAR methods will be valuable in assessing the properties of each half of the salt. Current estimation methods cannot estimate the  $K_{ow}$  of the ion pair.

#### Guidance on regulatory compliant $K_{ow}$ determination for surfactants:

In many cases a calculated  $K_{ow}$  value based on the octanol and water solubilities will be the first choice for surfactants. It is also useful to compare a calculated with a measured value. For the calculation approaches, one needs to consider the pH of the system (which determines the ionisation of the surfactant – see Section R.7.1.17). None of the experimental methods is very well suited for determining the  $K_{ow}$  of surface active substances. The shake flask method is the least suitable experimental method for surfactants. HPLC methodology may fail due to secondary interactions, and is sensitive to fluctuations of ionic strength. The slow stirring method in theory is the best, but still not demonstrated to be perfect. If using slow stirring, one needs to demonstrate a consistent result when starting with the surfactant in either phase, not just in the octanol. A working approach for surfactants might be the comparison of measured solubilities in octanol and water. However, it would then be prudent to take the critical micelle concentration in water (CMC) as a solubility limit, in order to avoid the artefact of unrealistically low  $K_{ow}$  values.

#### Guidance on regulatory compliant $K_{ow}$ determination for mixtures:

It is possible that different components of mixtures have significantly different behaviour in the physico-chemical tests and therefore also *in vivo* and in the environment. It is therefore important to ensure that the results presented for the physico-chemical tests represent each component rather than the mixture being treated as a single component. For simple mixtures where the components are known and easily identifiable, this may mean presenting individual values for  $K_{ow}$ . For complex mixtures, the HPLC method is ideal for determination of  $K_{ow}$ , and a defined range of values should be presented, with an indication of the proportion of substance within a given range (e.g. > 90 % of components have log  $K_{ow}$  in the range 4-5), to allow the significance of these results to be reflected in the risk assessment. The HPLC method is also recommended for petroleum products, which are typically mixtures.

#### R.7.1.8.6 Endpoint specific information in the registration dossier / in IUCLID

##### **Materials and methods**

##### **Shake-flask method (EU A.8/OECD TG 107):**

- equilibrium concentrations of the test substance in both phases;
- relative volumes of the two phases;
- analytical method(s).

##### **Calculation method (EU A.8):**

- identification of the method;
- working principle of the method;
- reference to the method;
- information on source chosen to justify  $K_{ow}$  values of fragments being manipulated;
- applicability of the method.

##### **HPLC method (EU A.8/OECD TG 117):**

- column(s) used;

- mobile phase (composition, buffer, pH);
  - reference substances with respective Kow values from the literature;
  - concentrations measured.
- Slow-stirring method (OECD TG 123):**
- label purity of labelled substances and molar activity (where appropriate);
  - sampling times;
  - description of the test vessels and stirring conditions;
  - number of replicates;
  - temperature during the experiment;
  - volumes of 1-octanol and water at the beginning, during and remaining after the test;
  - determined concentrations of the test substance in 1-octanol and water as a function of time;
  - description of the test vessels and stirring conditions (geometry of the stirring bar and of the test vessel, vortex height in mm, and when available: stirring rate) used;
  - analytical methods used to determine the test substance (its repeatability and sensitivity) and the method limit of quantification;
  - sampling times;
  - pH of the aqueous phase and of the buffers used, when pH is adjusted for ionisable molecules;
  - number of replicates;
  - demonstration of mass balance;
  - temperature and standard deviation or the range of temperature during the experiment;
  - the regression of concentration ratio against time.
- Results and discussion**
- final value for log Kow;
  - Kow values and their mean;
  - standard deviation of individual Kow values;
  - theoretical value when it has been calculated;
  - temperature of the test solutions (°C);
  - pH value(s) of the aqueous solution(s);
  - composition and concentration of buffers;
  - concentration of the stock solution;
  - if testing is waived, the reasons for waiving must be documented in the dossier.
- Any deviation from the guideline method used and reasons for it or any other special consideration should be reported. In cases where there is more than one source of data, the

- 1 endpoint summary under results and discussion should provide a justification for the selection  
2 of the key study chapter.  
3

4 **Reference to other ECHA Guidance Documents**

- 5 Further detailed guidance on partition coefficient can be found in the following chapters:

IUCLID Section	REACH Annex	Endpoint title	IUCLID 5 End User Manual Chapter	ECHA Practical Guide 3
4.7	VII 7.8	Partition coefficient	E.4.8	3.6

## 1 R.7.1.9 Flash point

### 2 R.7.1.9.1 Type of property

3 The flash point is a property:

- 4 • for substance characterization;
- 5 • for the classification of flammable liquids;
- 6 • which is important for physical hazard assessment.

### 7 R.7.1.9.2 Definition

8 The flash point is the lowest temperature of the liquid (as measured in a prescribed manner) at  
9 a pressure corrected to 101.325 kPa, at which application of an ignition source causes the  
10 vapour of the liquid to ignite momentarily and the flame to propagate across the surface of the  
11 liquid under the specified conditions of test (see section 1.2, Test Method A.9).

### 12 R.7.1.9.3 Test method(s)

13 The Test Method A.9 – Flash point from the Regulation (EC) 440/2008 can be used. Suitable  
14 methods are listed in the CLP Regulation Annex I, 2.6.4.4, Table 2.6.3.

15  
16 The method to be used has to be chosen taking into account the properties of the liquid  
17 (viscosity, halogenated compounds present) and the scope of the standard.

18 For substances with a high decomposition potential, a method using small amounts of liquid  
19 (e.g. EN ISO 3679: Determination of flash point - Rapid equilibrium closed cup method) is  
20 recommended to reduce the amount of substance under test.

21  
22 For classification purposes it is recommended to use the mean of at least two test runs. If the  
23 experimentally determined flashpoint is found to be within  $\pm 2$  °C of the limiting criterion for  
24 classification or assigning a category when using a non-equilibrium method, it is recommended  
25 to repeat the determination with an equilibrium method.

### 26 R.7.1.9.4 Adaptation of the standard testing regime

#### 27 **Adaptation possibilities according to column 2 of Annex VII to REACH**

28 Column 2 of REACH Annex VII provides the following specific rules for adaptation of the  
29 standard information requirement for flash point:

30 *The study does not need to be conducted if:*

- 31 • *the substance is inorganic;*
- 32 • *the substance only contains volatile organic components with flash-points above 100 °C*  
33 *for aqueous solutions; or*
- 34 • *the estimated flash-point is above 200°C; or*
- 35 • *the flash-point can be accurately predicted by interpolation from existing characterised*  
36 *materials.*

37 The first point has to be further specified as:

- 38 • The substance is inorganic except where there are covalent bonds;

39 because some inorganic liquids with covalent bonds are flammable e.g. CS<sub>2</sub>, N<sub>2</sub>H<sub>2</sub>, HCN.

40 The third point should only be applied when a well validated estimation model was used.



1 The fourth point should only be applied when there are enough reliable experimental data from  
2 existing characterised materials to be able to accurately interpolate to estimate the flash point.

### 3 **Adaptation possibilities according to Annex XI to REACH**

#### 4 **USE OF EXISTING DATA: DATA ON PHYSICAL-CHEMICAL PROPERTIES FROM** 5 **EXPERIMENTS NOT CARRIED OUT ACCORDING TO GLP OR THE TEST METHODS** 6 **REFERRED TO IN ARTICLE 13 (3) OF REACH**

7 If experimental data are available (study reports or literature data) which meet the criteria in  
8 section 1.1.1 of Annex XI to REACH, these could be used to meet the endpoint data  
9 requirements. If an estimation method is used as a source of information according to Column  
10 2 of Annex VII, the QSAR model must meet the criteria set out in section 1.3 of Annex XI to  
11 REACH.

#### 12 **WEIGHT OF EVIDENCE**

13 For the determination of the flash point, weight of evidence is not possible.

#### 14 **(Q)SAR**

15 For the determination of the flash point, QSAR approaches are discouraged for the purpose of  
16 classification / risk assessment, except where the mean absolute error of the QSAR is less than  
17 2 °C.

18 For non-halogenated liquids calculation based on the vapour pressure curve and lower  
19 explosion limit of the substance can be used as a screening test and a flashpoint need not be  
20 determined experimentally if the calculated value is at least 5 °C higher than the relevant  
21 classification criterion.

#### 22 **GROUPING OF SUBSTANCES AND READ-ACROSS APPROACH**

23 For the determination of the flash-point read-across is usually not possible. However  
24 interpolation may still be possible within homologous series.

#### 25 **TESTING IS TECHNICALLY NOT POSSIBLE**

26 This applies if:

- 27 • the liquid is an explosive;
- 28 the liquid is pyrophoric or self-reactive;
- 29 • decomposition occurs during the melting point study;
- 30 • some impurities have an impact on the ignition source in such a way as to
- 31 distort/invalidate the results.

32 Testing should always be considered, if none of the waiving possibilities applies.

#### 33 **Further adaptation possibilities**

34 The flash point does not need to be determined experimentally if conclusive and consistent  
35 literature data are available.

36 Data generated with the same tests and classification principles as specified in the CLP  
37 Regulation for flash point generated in conjunction with transport classification can satisfy the  
38 REACH requirements, but this needs to be checked on a case by case basis.

#### R.7.1.9.5 Impurities; uncertainties

Impurities can have a significant influence on the flash point. The influence depends on the amount and the vapour pressure of the impurity. Even if their concentration is below 0.5 %, especially if their boiling point is substantially lower, they may have a strong effect on the flash point. Impurities with a higher boiling point will normally have no effect on the flashpoint. Therefore utmost care should be taken in the selection of the key study(s), or weight-of-evidence approaches, that the data selected is representative of the substance being registered by the respective companies.

#### R.7.1.9.6 Endpoint specific information in the registration dossier / in IUCLID

##### **Materials and methods**

- reference to the standard or the test method applied;
- open cup or closed cup (for classification purposes only the closed cup methods are allowed);
- equilibrium or non-equilibrium method;

##### **Results and discussion**

- corrected flashpoint and unit;
- data on repeatability and reproducibility as given in the method;
- if testing is waived, the reasons for waiving must be documented in the dossier.

Any deviation from the guideline method used (and reasons for it) or any other special consideration should be reported. In cases where there is more than one source of data, the endpoint summary under results and discussion should provide a justification for the selection of the key study chapter.

##### **Reference to other ECHA Guidance Documents**

Further detailed guidance on flash point can be found in the following chapters:

IUCLID Section	REACH Annex	Endpoint title	IUCLID 5 End User Manual Chapter	ECHA Practical Guide 3
4.11	VII 7.9	Flash point	E.4.12	3.9

### 1 **R.7.1.10 Flammability**

2 Some of the information requirements according to REACH Annex VII were phrased in a way  
3 that they correspond to 'indications of danger' as given in Annex II of the DSD. For  
4 substances, classification and labelling according to CLP Regulation has been mandatory since  
5 1<sup>st</sup> December 2010 (and will become mandatory for mixtures (preparations) from 1<sup>st</sup> June  
6 2015, when the DPD will be repealed). Consequently properties associated with flammability  
7 are covered by classification of the substance according to the CLP Regulation. However, the  
8 physical hazards according to the CLP Regulation are structured completely differently from the  
9 physicochemical properties according to the DSD (and therefore also REACH, Annex VII). This  
10 means that for some of the CLP hazard classes an unambiguous assignment to one of the  
11 headlines (information requirements) in Annex VII to REACH is not possible. The assignment of  
12 hazard classes to the headline 'Flammability' as shown in the table below must therefore only  
13 be understood as a means to structure this document in accordance with Annex VII to REACH.  
14 It has to be noted that self-reactive substances and organic peroxides are assigned to the  
15 headline 'Flammability' and only a cross reference is added under the headline 'Explosive  
16 properties' because these two hazard classes can have explosive and/or flammable  
17 properties.

18 **Table R.7.1-6 Assignment of CLP hazard classes to the information requirement 'Flammability'**  
19 **according to REACH, Annex VII and correlation between the Test Method Regulation and the**  
20 **test method according to CLP and supporting link with the Guidance on the Application of the**  
21 **CLP Criteria.**

Information requirement according to Art. 10 (a) (vi) of the REACH Regulation (EC) No. 1907/2006 (the no. in brackets is the respective no. in the table in Annexes VII to XI to REACH)	CLP Regulation (EC) No. 1272/2008 (the no. in brackets is the respective chapter no. in Annex I to CLP)	Chapter in revised R.7(a) guidance	Corresponding test method according to The Test Method Regulation (EC) No 440/2008	Corresponding test method according to CLP Regulation	Chapter in the Guidance on the application of the CLP Criteria (ex RIP 3.6)
Flammability (7.10)	Flammable gases (2.2)*	7.1.10.1	A.11 Flammability (gases)	<b>ISO 10156</b> <b>EN 1839</b>	<b>2.3</b>
	Flammable liquids (2.6)*	7.1.10.2	for liquids: see Flash point	<b>see CLP, Annex I, Chapter 2.6.4.4, Table 2.6.3</b>	<b>2.7</b>
	Flammable solids (2.7)*	7.1.10.3	A.10 Flammability (solids)	<b>UN Test N.1</b>	<b>2.8</b>
	Self-reactive substances and mixtures (2.8)*	7.1.10.4	n.a.	<b>UN Test series A to H</b>	<b>2.9</b>
	Pyrophoric liquids (2.9)*	7.1.10.5	A.13 Pyrophoric properties of solids and liquids	<b>UN Test N.3</b>	<b>2.10</b>
	Pyrophoric solids (2.10)*	7.1.10.6		<b>UN Test N.2</b>	<b>2.10</b>

	Self-heating substances and mixtures (2.11)*	7.1.10.7	n.a.	<b>UN Test N.4</b>	<b>2.11</b>
	Substances and mixtures which in contact with water emit flammable gases (2.12)*	7.1.10.8	A.12 Flammability (Contact with water)	<b>UN Test N.5</b>	<b>2.12</b>
	Organic peroxides (2.15)*	7.1.10.9	n.a.	<b>UN Test series A to H</b>	<b>2.14</b>

\* Note that regardless of whether the hazard class or category is listed in Article 14(4)(a) REACH the chemical safety assessment (where required) must be performed in accordance with Article 14(3) REACH. Furthermore, according to Article 10(a)(iv) of REACH the technical dossier of a registration of a substance under the REACH Regulation must include information on classification and labelling of the substance as specified in section 4 of Annex VI to the REACH Regulation.

In addition, it has to be noted that some substances have flammable properties which do not result in classification. Examples are the following:

- Gases that do not have a flammable range at 20 °C and standard pressure (and therefore are not classified as flammable gases) might have a flammable range at higher temperatures and/or pressure (e.g. ammonia);
- Liquids that do not have a flash point (and therefore are not classified as flammable liquids) might have an explosion range (especially halogenated hydrocarbons).

Information about such properties should also be indicated in the dossier.

#### R.7.1.10.1 Flammable gases

##### **Definition**

*Flammable gas means a gas or gas mixture having a flammable range with air at 20 °C and a standard pressure of 101.3 kPa (Annex I to CLP, Section 2.2.1).*

##### **Classification criteria and relevant information**

Flammable gases are classified into two categories depending on their flammability range (Annex I to CLP, Section 2.2.2. Table 2.2.1).

Detailed guidance on the classification criteria and the test method(s) can be found in the *Guidance on the application of the CLP Criteria*, section 2.3.

##### **Adaptation of the standard testing regime**

#### **ADAPTATION POSSIBILITIES ACCORDING TO COLUMN 2 OF ANNEX VII TO REACH**

Column 2 of REACH Annex VII provides the following specific rules for adaptation of the standard information requirement for flammability:

*The study does not need to be conducted:*

- *if the substance is a solid which possesses explosive or pyrophoric properties. These properties should always be considered before considering flammability; or*

- for gases, if the concentration of the flammable gas in a mixture with inert gases is so low that, when mixed with air, the concentration is all time below the lower limit; or
- for substances which spontaneously ignite when in contact with air.

The relevant points can be paraphrased (first point is not relevant for this chapter), namely the study does not need to be conducted:

- if the concentration of the flammable gas in a mixture when mixed with air is below the lower limit;
- if the gas spontaneously ignites when in contact with air.

Gases that spontaneously ignite in contact with air are pyrophoric and are therefore flammable gases.

## Adaptation possibilities according to Annex XI to REACH

### USE OF EXISTING DATA: DATA ON PHYSICAL-CHEMICAL PROPERTIES FROM EXPERIMENTS NOT CARRIED OUT ACCORDING TO GLP OR THE TEST METHODS REFERRED TO IN ARTICLE 13 (3) OF REACH

If experimental data are available (study reports or literature data) meeting the criteria in section 1.1.1 of Annex XI to REACH, these could be used to meet the endpoint data requirements. If an estimation method is used as a source of information according to Column 2 of Annex VII, the QSAR model must meet the criteria set out in section 1.3 of Annex XI to REACH.

Many gases are classified in Annex VI to CLP either as *Flam. Gas 1* or *Flam. Gas 2*, and additional flammable gases are listed in the UN Recommendations on the Transport of Dangerous Goods, Model Regulations whose classifications correspond to *Flam. Gas 1* according to CLP.

### WEIGHT OF EVIDENCE

Where no single source of existing data (study reports, QSAR, literature data) is considered sufficiently reliable, thus not fully meeting the criteria in section 1.1.1 of Annex XI to REACH, or where several sources of similar reliability with deviating results exist, a weight of evidence approach may be used. The criteria in section 1.2 of Annex XI to REACH must then be met.

For gases that are not classified in Annex VI to the CLP Regulation nor in the UN Recommendations on the Transport of Dangerous Goods, Model Regulations, there is ample scientific literature giving the flammability range for most gases (e.g. IEC 60079-20-1 "Data for flammable gases and vapours, relating to the use of electrical apparatus" – (under revision)).

### (Q)SAR

At present (Q)SAR is generally not applicable for determination of explosion (/flammability) limits of gases.

### GROUPING OF SUBSTANCES AND READ-ACROSS APPROACH

For the determination of the flammable gases read-across is usually not possible. However interpolation may still be possible within homologous series.

## TESTING IS TECHNICALLY NOT POSSIBLE

Testing should always be considered, if none of the waiving possibilities applies.

## FURTHER ADAPTATION POSSIBILITIES

Further adaptation is possible for gases that are known to be non-flammable. Examples are nitrogen, the noble gases (helium, neon, argon, krypton, xenon), carbon dioxide and sulphur hexafluoride. As stated in Annex IX of REACH, when for certain endpoints, it is proposed to not provide information for other reasons than those mentioned in column 2 of that Annex or in Annex XI of REACH, this fact and the reasons must also be clearly stated. Such an approach may then be used.

### Impurities; uncertainties

Tests should be performed with the lowest concentration of impurities in the gas encountered in the normal manufacturing process and the moisture content should be less than or equal to 0.01 mol %. Utmost care should be taken in the selection of the key study(s) and/or use of weight-of-evidence approaches that the data selected is representative of the substance being registered by the respective companies.

### How to conclude on the DSD classification

All gases with a flammability range in air are classified 'Extremely flammable F+ ; R12' according to DSD, unless classified differently according to Annex VI, Table 3.2 of the CLP Regulation. This means that all gases classified as flammable gases according to CLP (either category 1 or 2) are classified as 'Extremely flammable F+ ; R12'.

## Endpoint specific information in the registration dossier/in IUCLID

### Material and methods:

- description of the apparatus and dimensions or reference to the standard or the test method applied;
- test temperature;
- tested concentrations.

### Results and discussion & Applicant's Summary and conclusion (interpretation of results)

- indicate lower and upper explosion limits in % volume;
- if testing is waived, the reasons for waiving must be documented in the dossier.

Any deviation from the guideline method used (and reasons for it) or any other special consideration should be reported. In cases where there is more than one source of data, the endpoint summary under results and discussion should provide a justification for the selection of the key study chapter.

### Reference to other ECHA Guidance Documents

Further detailed guidance on flammability can be found in the following chapters:

IUCLID Section	REACH Annex	Endpoint title	IUCLID 5 End User Manual Chapter	ECHA Practical Guide 3

4.13	VII 7.10	Flammability	E.4.14	3.11
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1

## 2 Further information / references

3 For the testing of flammable gases according to CLP classification requirements, refer also to  
4 the Guidance on the Application of the CLP Criteria, section 2.3, and in Directive 2008/47/CE.

### 5 R.7.1.10.2 Flammable liquids

#### 6 Definition

7 *Flammable liquid means a liquid which may form flammable/explosive vapour-air mixtures*  
8 *(see CLP Annex I, section 2.6.1). Within the CLP Regulation 'Flammable liquid' means a liquid*  
9 *having a flashpoint of not more than 60 °C.*

#### 10 Classification criteria and relevant information

11 Flammable liquids are classified in three categories according to the criteria of the CLP  
12 Regulation (see section 2.6, table 2.6.1) based on their boiling point and their flash point.  
13 Derogation is possible (CLP Annex I, section 2.6.4.5) for Flam. Liquid Cat. 3 having a  
14 flashpoint above 35 °C based on the information on sustained combustibility. Furthermore, gas  
15 oils, diesel and light heating oils having a flash point between  $\geq 55$  °C and  $\leq 75$  °C may be  
16 regarded as Category 3 flammable liquids according to the CLP Regulation (CLP Annex I  
17 Section 2.6, Note to Table 2.6.1).

18

19 In addition EUH018 - *'In use may form flammable/explosive vapour-air mixture'* has to be  
20 assigned to substances classified under the CLP Regulation which may form  
21 flammable/explosive vapour-air mixtures although they do not have a flash point e. g.  $\text{CH}_2\text{Cl}_2$ ,  
22  $\text{C}_2\text{H}_3\text{Cl}_3$ . In such cases it is possible to make the decision on whether flammable/explosive  
23 vapour-air mixture may be formed based on either the determination of explosion limits  
24 according to EN 1839 or the determination of explosion points according to EN 15794. It is  
25 sufficient to determine either the lower explosion limit or the lower explosion point.

26 Detailed guidance on the classification criteria and the test method(s) can be found in the  
27 *Guidance on the application of the CLP Criteria*, section 2.7.

#### 28 Adaptation of the standard testing regime

#### 29 ADAPTATION POSSIBILITIES ACCORDING TO COLUMN 2 OF ANNEX VII TO REACH

30 The entries 'flammability' (7.10), 'boiling point' (7.3) and 'flashpoint' (7.9) are the relevant  
31 ones. For the latter two entries, see their respective relevant sections in this document.

32 Column 2 of REACH Annex VII provides the following specific rules for adaptation of the  
33 standard information requirement for flammability:

34 *The study does not need to be conducted:*

- 35 • *if the substance is a solid which possesses explosive or pyrophoric properties. These*  
36 *properties should always be considered before considering flammability; or*
- 37 • *for gases, if the concentration of the flammable gas in a mixture with inert gases is so*  
38 *low that, when mixed with air, the concentration is all time [i.e. "always"] below the*  
39 *lower limit; or*
- 40 • *for substances which spontaneously ignite when in contact with air.*



The relevant points can be paraphrased (first two points are not relevant for this chapter), namely the 3<sup>rd</sup> point specifies that for flammability, Annex VII to REACH does not require testing for substances which spontaneously ignite when in contact with air.

## **Adaptation possibilities according to Annex XI to REACH**

### **USE OF EXISTING DATA: DATA ON PHYSICAL-CHEMICAL PROPERTIES FROM EXPERIMENTS NOT CARRIED OUT ACCORDING TO GLP OR THE TEST METHODS REFERRED TO IN ARTICLE 13 (3) OF REACH**

If experimental data are available (study reports or literature data) meeting the criteria in section 1.1.1 of Annex XI to REACH, these could be used to meet the endpoint data requirements. If an estimation method is used as a source of information according to Column 2 of Annex VII, the QSAR model must meet the criteria set out in section 1.3 of Annex XI to REACH.

### **WEIGHT OF EVIDENCE**

Where no single source of existing data (study reports, QSAR, literature data) is considered sufficiently reliable, thus not fully meeting the criteria in section 1.1.1 of Annex XI to REACH, or where several sources of similar reliability with deviating results exist, a weight of evidence approach may be used. The criteria in section 1.2 of Annex XI to REACH must then be met.

### **(Q)SAR**

To be used as a stand alone value to meet the data requirements of Annex VII, 7.8, QSAR models must meet the criteria set out in Annex XI, 1.3. The entries 'boiling point' (7.3) and 'flashpoint' (7.9) are also the relevant ones, therefore please check under each respective QSAR sub-section for more information.

#### **Sustained Combustibility:**

No (Q)SAR exists currently.

For further reference see also *the Guidance on the Application of the CLP Criteria*, section 2.7.

### **GROUPING OF SUBSTANCES AND READ-ACROSS APPROACH**

The entries 'boiling point' (7.3) and 'flashpoint' (7.9) are again the relevant ones. For both these entries, see their respective sections in this document.

#### **Sustained Combustibility:**

For the determination of the sustained combustibility read-across is usually not possible. However interpolation may still be possible within homologous series.

### **TESTING IS TECHNICALLY NOT POSSIBLE**

Testing is not possible if:

- the liquid is an explosive;
- the liquid is pyrophoric or self-reactive.

Testing should always be considered if none of the waiving possibilities applies.

## 1 FURTHER ADAPTATION POSSIBILITIES

2 Use of data on boiling point, flashpoint when determined with a closed cup method, explosion  
3 limits or lower explosion point from validated literature (see below chapter Further  
4 information/ references) is possible. Data on boiling point generated in relation to transport  
5 classification may also satisfy the Annex XI requirements. Data on flashpoint generated in  
6 relation to with transport classification may satisfy the Annex XI requirements if closed cup  
7 methods have been used. However care has to be taken in cases where there is no transport  
8 classification as "flammable liquid", because certain substances can form flammable/explosive  
9 vapour-air mixtures although they do not have a flash point.

10

11 As stated in Annex IX of REACH, when for certain endpoints, it is proposed to not provide  
12 information for other reasons than those mentioned in column 2 of that Annex or in Annex XI  
13 of REACH, this fact and the reasons must also be clearly stated. Such an approach may then  
14 be used.

## 15 Impurities; uncertainties

### 16 Boiling point:

17 Impurities will influence the boiling point of the main component. The influence depends on the  
18 amount and boiling point of the impurity. The higher the amount and the higher the difference  
19 between the boiling points of the main component and the impurity, the higher the influence.

20

### 21 Flashpoint:

22 Special care has to be taken when a sample contains impurities with a lower boiling point than  
23 the main component. Even if their concentration is below 0.5 %, especially if their boiling point  
24 is substantially lower, they may have a strong effect on the test result. Impurities with a  
25 higher boiling point will normally have no effect on the flashpoint.

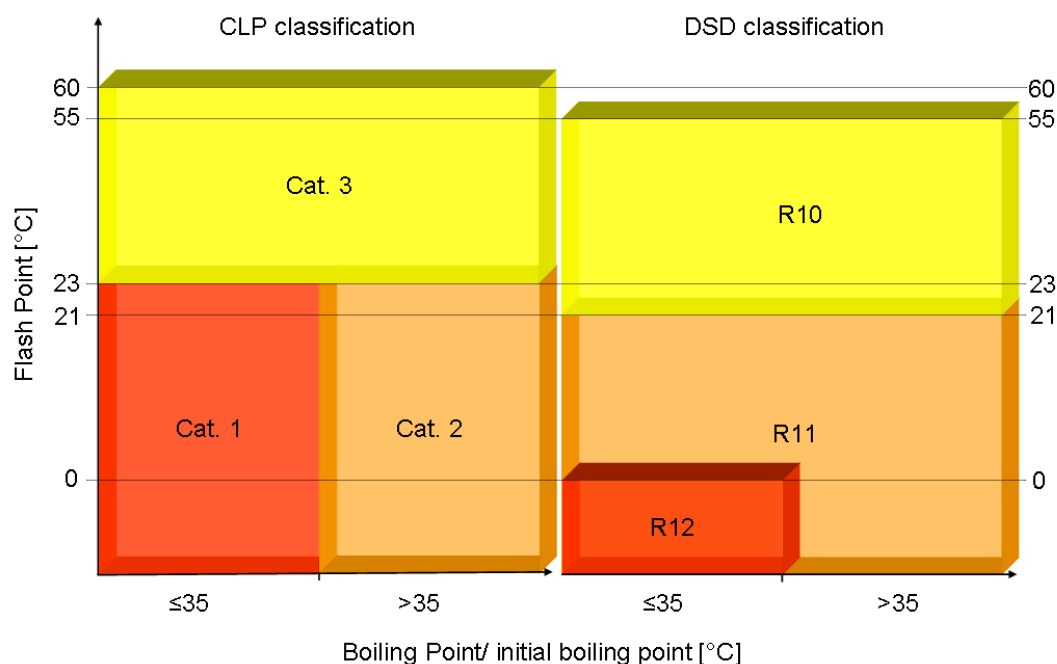
26

### 27 Sustained combustibility:

28 Impurities with lower boiling point may influence the ability to sustain combustion. However it  
29 is not yet possible to quantify the influence of impurities.

## 30 How to conclude on the DSD classification

31 Based on the data on boiling point and flashpoint the DSD classification according to the  
32 respective DSD criteria is possible. Simplified direct translation between CLP classification and  
33 DSD classification is not possible, see figure below.



**Figure R.7.1-1 Comparison of the DSD and the CLP classification**

Substances exempted from classification in Cat. 3 because of their flashpoint and behaviour when tested for sustained combustibility can be exempted from being classified under DSD as R10, if they don't show additional dangerous properties relevant for classification.

## Endpoint specific information in the registration dossier/in IUCLID

The physicochemical studies reporting data relevant for classification as a flammable liquid (flashpoint and boiling point) are to be reported in the relevant IUCLID endpoint records.

### Material and methods

See chapter flash point R.7.1.9 and boiling point R.7.1.3.

### Results and discussion

- corrected flashpoint and unit;
- data on repeatability and reproducibility as given in the method;
- boiling point value (°C) as measured;
- pressure value and unit;
- rate of temperature increase;
- decomposition (if applicable);
- measurement uncertainty if available;
- boiling point value in °C (corrected to standard pressure, except where the boiling point was determined at reduced pressures) (as above, but in a separate block of fields);

• if available explosion limits;

• if testing is waived, the reasons for waiving must be documented in the dossier.

Any deviation from the guideline method used (and reasons for it) or any other special consideration should be reported. In cases where there is more than one source of data, the endpoint summary under results and discussion should provide a justification for the selection of the key study chapter.

## Reference to other ECHA Guidance Documents

Further detailed guidance on flammability can be found in the following chapters:

IUCLID Section	REACH Annex	Endpoint title	IUCLID 5 End User Manual Chapter	ECHA Practical Guide 3
4.13	VII 7.10	Flammability	E.4.14	3.11

## Further information / references

See also R.7.1.3 Boiling point and R.7.1.9 Flash point. For testing of flammable liquids according to CLP classification requirements refer also to the Guidance on the Application of the CLP Criteria, section 2.7.

### R.7.1.10.3 Flammable solids

#### Definition

*A flammable solid means a solid which is readily combustible, or may cause or contribute to fire through friction. Readily combustible solids are powdered, granular, or pasty substances or mixtures which are dangerous if they can be easily ignited by brief contact with an ignition source, such as a burning match, and if the flame spreads rapidly (see CLP Regulation, Annex I, section 2.7.1).*

#### Classification criteria and relevant information

Solid substances and mixtures are classified as flammable in two categories according to their burning behaviour (see the CLP Regulation, Annex I, section 2.7) using UN Test N.1 as described in section 33.2.1 of the UN Recommendations on the Transport of Dangerous Goods, Manual of Tests and Criteria.

Chapter 2.8 of the *Guidance on the Application of the CLP Criteria* gives detailed information on the CLP classification of flammable solids, the UN Test N.1 and the relation to the DSD and the Transport of Dangerous Goods regulations.

#### Adaptation of the standard testing regime

#### ADAPTATION POSSIBILITIES ACCORDING TO COLUMN 2 OF ANNEX VII TO REACH

Column 2 of REACH Annex VII provides the following specific rules for adaptation of the standard information requirement for flammable solids:

*The study does not need to be conducted:*

- if the substance is a solid which possesses explosive or pyrophoric properties. These properties should always be considered before considering flammability; or*

- *for gases, if the concentration of the flammable gas in a mixture with inert gases is so low that, when mixed with air, the concentration is all time [i.e. always] below the lower limit; or*
- *for substances which spontaneously ignite when in contact with air.*

Concerning the first indent, testing for flammability of a solid is a part of classification in CLP Regulation. Refer also to the Guidance on the Application of the CLP Criteria, section 2.8 on classification requirements. For substances having explosive properties, testing for a classification as a flammable solid may be waived. This applies to substances and mixtures classified as explosives, organic peroxides and self-reactive substances and mixtures.

Second indent is not applicable for this endpoint.

With regards to the third indent, substances which spontaneously ignite when in contact with air are pyrophoric substances as defined by the CLP Regulation (see the Guidance on the Application of the CLP Criteria, section 2.10). Such substances are not classified as flammable solids but as pyrophoric solids under the CLP Regulation.

## **Adaptation possibilities according to Annex XI to REACH**

### **USE OF EXISTING DATA: DATA ON PHYSICAL-CHEMICAL PROPERTIES FROM EXPERIMENTS NOT CARRIED OUT ACCORDING TO GLP OR THE TEST METHODS REFERRED TO IN ARTICLE 13 (3) OF REACH**

Literature data – even if available – should not be used since flammability strongly depends on particle size, surface treatment and other parameters.

If experimental data are available (study reports or literature data) meeting the criteria in section 1.1.1 of Annex XI to REACH, these could be used to meet the endpoint data requirements. If an estimation method is used as a source of information according to Column 2 of Annex VII, the QSAR model must meet the criteria set out in section 1.3 of Annex XI to REACH.

If available data from an A.10 test indicate that a classification as a flammable solid does not apply (result: not highly flammable), no more testing is necessary. However, if the A.10 test has come to the conclusion 'highly flammable', it will be necessary to also determine the influence of the wetted zone as described in the UN Test N.1.

### **WEIGHT OF EVIDENCE**

Where no single source of existing data (study reports, QSAR, literature data) is considered sufficiently reliable, thus not fully meeting the criteria in section 1.1.1 of Annex XI to REACH, or where several sources of similar reliability with deviating results exist, a weight of evidence approach may be used. The criteria in section 1.2 of Annex XI to REACH must then be met.

### **(Q)SAR**

At present (Q)SAR is generally not applicable for flammable solids. Application of (Q)SAR is not possible.

### **GROUPING OF SUBSTANCES AND READ-ACROSS APPROACH**

At present, grouping and read across are not applicable.

## 1 TESTING IS TECHNICALLY NOT POSSIBLE

2 Testing should always be considered if none of the waiving possibilities applies.

## 3 FURTHER ADAPTATION POSSIBILITIES

4 As stated in Annex IX of REACH, when for certain endpoints, it is proposed to not provide  
5 information for other reasons than those mentioned in column 2 of that Annex or in Annex XI  
6 of REACH, this fact and the reasons must also be clearly stated. Such an approach may then  
7 be used.

8 If a suitable screening test clearly shows that the substance is not flammable, further testing is  
9 not necessary (see also the *Guidance on the application of the CLP Criteria*, section 2.8.4.2).  
10 An example for a suitable screening test is the burning index as described in VDI guideline  
11 (VDI Guideline, 1990) if a burning index of 3 or less is found, the substance should not be  
12 classified as a flammable solid and no further testing is required.

13 Substances and mixtures classified according to the CLP Regulation as explosives, organic  
14 peroxides, self-reactive substances and mixtures as well as pyrophoric or oxidising solids  
15 should not be considered for classification as flammable solids (see the *Guidance on the*  
16 *Application of the CLP criteria*, section 2.8.3).

17 However, if a substance gives a positive result in UN Test Series 1 or 2 as described in the UN  
18 Manual of Tests and Criteria, but is exempted from classification as an explosive on the basis  
19 of UN Test Series 6, a test for classification as a flammable solid should be performed.

## 20 Impurities; uncertainties

21 Impurities do not tend to have a large effect on the flammability of a solid. However, if a solid  
22 which is not flammable in the pure state contains flammable organic liquids or organometallic  
23 impurities it may burn more rapidly and thus become flammable. Therefore utmost care should  
24 be taken in the selection of the key study(s) and during use of weight-of-evidence approaches,  
25 that the data selected is representative of the substance being registered by the respective  
26 companies.

## 27 How to conclude on the DSD classification

28 Any substance found to be a flammable solid according to CLP Regulation has to be classified  
29 as 'F; R11' according to the DSD.

## 30 Endpoint specific information in the registration dossier/in IUCLID

### 31 Material and methods

32 • description of the apparatus and dimensions or reference to the standard or the test method  
33 applied;

### 34 Solid flammability:

- 35 • indicate if preliminary and/or main test performed;  
36 • moisture content;  
37 • particle size and distribution (if available) (see R.7.1.14.1. Granulometry).

### 38 Results and discussion

- 39 • indicate burning time;  
40 • pass/non pass of the wetted zone (in the case of the UN Test N.1);

- if testing is waived, the reasons for waiving must be documented in the dossier.

Any deviation from the guideline method used or any other special consideration should be reported. In cases where there is more than one source of data, the endpoint summary under results and discussion should provide a justification for the selection of the key study chapter.

## Reference to other ECHA Guidance Documents

Further detailed guidance on flammability can be found in the following chapters:

IUCLID Section	REACH Annex	Endpoint title	IUCLID 5 End User Manual Chapter	ECHA Practical Guide 3
4.13	VII 7.10	Flammability	E.4.14	3.11

## Further information / references

VDI guideline 2263, part 1, (1990): "Test methods for the Determination of the Safety Characteristics of Dusts".

For testing of flammable solids according to CLP classification requirements, refer also to the *Guidance on the Application of the CLP Criteria*, section 2.8.

### R.7.1.10.4 Self-reactive substances and mixtures

In the CLP Regulation self-reactive substances are a distinct hazard class. Self-reactive substances are classified into one of the seven categories of "Types A to G" according to the classification criteria given in section 2.8.2.3 of Annex I of CLP. In the Dangerous Substances Directive (67/548/EEC) no hazard class for 'self-reactive substances' is defined. Nevertheless, self-reactive substances were also classified as dangerous according to the DSD, e.g. as flammable or as substances with explosive properties.

As mentioned below under the sub-section "Definition", self-reactive substances are excluded from testing as explosives according to Test Series 1 to 8 in Part I of the UN Recommendations on the Transport of Dangerous Goods, Manual of Tests and Criteria (see R.7.1.11.1 Explosives). In Test Series A to H however, no tests on sensitivity to impact (solids and liquids) and friction (solids only) are included. For the risk assessment and the safe use and handling, data according to the EU test method A.14 as described in Regulation (EC) No 440/2008, if available, or UN Test 3 (a) (ii) BAM Fallhammer and Test 3 (b) (i) BAM friction apparatus (see R.7.1.11) should be part of the hazard communication in the registration dossier (REACH Annex VII, 7.11) and the safety data sheet.

## Definition

The definition of a self-reactive substance is given in section 2.8.1 of Annex I to CLP Regulation:

*Self-reactive substances or mixtures are thermally unstable liquid or solid substances or mixtures liable to undergo a strongly exothermic decomposition even without participation of oxygen (air). This definition excludes substances and mixtures classified according to this Part as explosives, organic peroxides or as oxidising. A self-reactive substance or mixture is regarded as possessing explosive properties when in laboratory testing the formulation is liable to detonate, to deflagrate rapidly or to show a violent effect when heated under confinement.*

Background information and guidance on the definition is given in the *Guidance on the Application of the CLP Criteria*, sections 2.9.1 and 2.9.2.



## 1 **Classification criteria and relevant information**

2 Classification principles are given in CLP Regulation Annex I, sections 2.8.2 and 2.8.4.  
3 Background information and guidance on relevant aspects regarding the classification is given  
4 in the *Guidance on the Application of the CLP Criteria*, sections 2.9.3, 2.9.5 and 2.9.6.

## 5 **Adaptation of the standard testing regime**

### 6 **ADAPTATION POSSIBILITIES ACCORDING TO COLUMN 2 OF ANNEX VII TO REACH**

7 Only self-reactive substances, as defined in the section definition, have to be tested according  
8 to the United Nations Manual of Tests and Criteria, Part II test series A - H.

9 CLP Annex I provides the following specific rules for adaptation of the standard information  
10 requirement for self-reactive substances and mixtures.

11 *Any self-reactive substance or mixture shall be considered for classification in this class as a*  
12 *self-reactive substance or mixture unless:*

13 *(a) they are explosives, according to the criteria given in 2.1;*

14 *(b) they are oxidising liquids or solids, according to the criteria given in 2.13 or 2.14, except*  
15 *that mixtures of oxidising substances, which contain 5 % or more of combustible organic*  
16 *substances shall be classified as self-reactive substances according to the procedure defined in*  
17 *2.8.2.2;*

18 *(c) they are organic peroxides, according to the criteria given in 2.15;*

19 *(d) their heat of decomposition is less than 300 J/g; or*

20 *(e) their self-accelerating decomposition temperature (SADT) is greater than 75 °C for a 50*  
21 *kg package.*

### 22 **ADAPTATION POSSIBILITIES ACCORDING TO ANNEX XI TO REACH**

#### 23 **USE OF EXISTING DATA: DATA ON PHYSICAL-CHEMICAL PROPERTIES FROM** 24 **EXPERIMENTS NOT CARRIED OUT ACCORDING TO GLP OR THE TEST METHODS** 25 **REFERRED TO IN ARTICLE 13 (3) OF REACH**

26 A number of already tested and classified substances and mixtures are listed in United Nations  
27 Recommendations on the Transport of Dangerous Goods, section 2.4.2.3.2.3. Available  
28 information may originate from the classification for transport. More details are given in the  
29 *Guidance on the Application of the CLP Criteria*, sections 1.7.2.1 and 2.9.5.

30 If experimental data are available (study reports or literature data) meeting the criteria in  
31 section 1.1.1 of Annex XI to REACH, these could be used to meet the endpoint data  
32 requirements. If an estimation method is used as a source of information according to Column  
33 2 of Annex VII, the QSAR model must meet the criteria set out in section 1.3 of Annex XI to  
34 REACH.

## 35 **WEIGHT OF EVIDENCE**

36 For the determination of the self-reactive substances and mixtures, weight of evidence is not  
37 possible.

## **(Q)SAR**

At present (Q)SAR is generally not applicable for determination of self-reactive substances. Application of (Q)SAR is not possible.

## **GROUPING OF SUBSTANCES AND READ-ACROSS APPROACH**

At present grouping and read-across are not applicable.

## **TESTING IS TECHNICALLY NOT POSSIBLE**

A few of substances can, for safety reasons, only be handled and tested in diluted form, see the substances and mixtures listed in UN Recommendations on the Transport of Dangerous Goods, Model Regulations, section 2.4.2.3.2.3.

## **FURTHER ADAPTATION POSSIBILITIES**

Not foreseen.

## **Impurities; uncertainties**

Minor impurities can have an influence on thermal stability. Background information and guidance on these aspects is given in the *Guidance on the Application of the CLP Criteria*, section 2.9.3.3.

## **How to conclude on the DSD classification**

In the DSD self-reactive substances are not covered. They may be classified in other DSD classes (e.g. explosive substance, flammable solid or liquid). See also the *Guidance on the Application of the CLP Criteria*, section 2.9.5.

## **What information is required in the registration dossier in IUCLID**

### **Material and methods**

- See UN MTC, Part II, classification procedures and test series A-H.

### **Results and discussion**

- The following data on self-reactive substances should be submitted:
- type of self-reactive substance;
- decomposition energy (value and method of determination);
- SADT (Self accelerating decomposition temperature) together with the volume the SADT relates to;
- detonation properties (Yes/Partial/No);
- deflagration properties (Yes rapidly/Yes slowly/No);
- effect of heating under confinement (Violent/Medium/Low/No);
- explosive power if applicable (Not low/Low/None).

For assigning the type of self-reactive substance, the list of currently assigned self-reactive substances according to the 2.4.2.3.2.3 of the UN Recommendations on the Transport of

Dangerous Goods, Model Regulations can be used, in cases where the assignment was based on test(s) according to the UN Manual of Tests and Criteria. The relevant underlying test data may be collected from the respective UN documents from the UN committee of experts on the transport of dangerous goods, from test reports produced by competent authorities or industry, or from other reliable sources.

Any deviation from the guideline method used (and reasons for it) or any other special consideration should be reported. In cases where there is more than one source of data, the endpoint summary under results and discussion should provide a justification for the selection of the key study chapter.

The following example shows how the data mentioned above could be documented in the chemical safety report (CSR):

**Figure R.7.1-2 Example 2,2'-Azodi (isobutyronitrile)**

UN Test Series A to H	Test method	Results + Evaluation	Remarks
Propagation of detonation	A.5	"yes"	Apparent density (kg/m <sup>3</sup> ): 366 Fragmented length (cm): 40
Propagation of deflagration #1	C.1	"yes, slowly"	68 ms
Propagation of deflagration #2	C.2	"no"	
Effect of heating under defined confinement #1	Koenen E.1	"violent"	Limiting diameter 3.0 mm Type of fragmentation: F
Effect of heating under defined confinement #2	DPVT E.2	"medium"	Limiting diameter 5.5 mm
Explosive power	F.4	"not Low"	Average net expansion (cm <sup>3</sup> ): 18
SADT	H.4	50 °C	500 ml Dewar vessel
Competent Authority approval number	<i>Example from UN Recommendations on the Transport of Dangerous Goods, Manual of Tests and Criteria</i>		

## Reference to other ECHA Guidance Documents

A template data set does not currently exist in IUCLID for the hazard class "self-reactive substances". As long as there is no specific section available in IUCLID the test results in IUCLID section 4.23 "Additional physico-chemical information" under the endpoint title "Self-reactive substances" should be inserted. In the CSR the information should be included under flammability.

## Further information / references

Background information and guidance on classification testing, additional testing and available information is given in the *Guidance on the Application of the CLP Criteria*, section 2.9.

### R.7.1.10.5 Pyrophoric liquids

#### Definition

The definition of a pyrophoric liquid is given in the section 2.9.1 of Annex I to CLP Regulation:

*Pyrophoric liquid means a liquid substance or mixture which, even in small quantities, is liable to ignite within five minutes after coming into contact with air.*

Background information and guidance on the definition is given in the *Guidance on the Application of the CLP Criteria*, sections 2.10.1 and 2.10.2.

### **Classification criteria and relevant information**

Classification principles are given in CLP Regulation Annex I, section 2.9.2.

The criterion for a pyrophoric liquid is as follows: *The liquid ignites within 5 min when added to an inert carrier and exposed to air, or it ignites or chars a filter paper on contact with air within 5 min.*

Background information and guidance on relevant aspects regarding the classification is given in the *Guidance on the Application of the CLP Criteria*, sections 2.10.1, 2.10.2, 2.10.3 and 2.10.4.

### **Adaptation of the standard testing regime**

#### **ADAPTATION POSSIBILITIES ACCORDING TO COLUMN 2 OF ANNEX VII TO REACH**

Other flammability tests do not have to be performed as well as the determination of the self-ignition temperature, if the substance is a pyrophoric substance. However, flammability in contact with water may be relevant.

#### **ADAPTATION POSSIBILITIES ACCORDING TO ANNEX XI TO REACH**

#### **USE OF EXISTING DATA: DATA ON PHYSICAL-CHEMICAL PROPERTIES FROM EXPERIMENTS NOT CARRIED OUT ACCORDING TO GLP OR THE TEST METHODS REFERRED TO IN ARTICLE 13 (3) OF REACH**

The UN Test N.3 of the UN Manual of Tests and Criteria is also used for classification according to the regulations on the transport of dangerous goods (ADR and RID). If the liquid in question has been classified as belonging to Class 4.2, packing group I of the ADR/RID on the basis of UN Test N.3 results, it is a pyrophoric liquid according to CLP criteria. Packing group I of the ADR/RID directly corresponds to Category 1 of the CLP.

According to the DSD, the A.13 method of Regulation (EC) 440/2008 is used for the assessment of pyrophoric properties for liquids and liquids. This method is identical to the UN Test N.3.

If experimental data are available (study reports or literature data) meeting the criteria in section 1.1.1 of Annex XI to REACH, these could be used to meet the endpoint data requirements. If an estimation method is used as a source of information according to Column 2 of Annex VII, the QSAR model must meet the criteria set out in section 1.3 of Annex XI to REACH.

#### **WEIGHT OF EVIDENCE**

Where no single source of existing data (study reports, QSAR, literature data) is considered sufficiently reliable, thus not fully meeting the criteria in section 1.1.1 of Annex XI to REACH, or where several sources of similar reliability with deviating results exist, a weight of evidence approach may be used. The criteria in section 1.2 of Annex XI to REACH must then be met.

**(Q)SAR**

Application of (Q)SAR is not possible, however assessment of the chemical structure may be used to exclude pyrophoric properties of a substance. Such an assessment of chemical structure, in conjunction with experience in manufacture and handling, could also formally form part of a weight-of-evidence argument.

**GROUPING OF SUBSTANCES AND READ-ACROSS APPROACH**

Assessment of the chemical structure may be used to anticipate pyrophoric properties of a substance.

**TESTING IS TECHNICALLY NOT POSSIBLE**

Testing should always be considered if none of the waiving possibilities applies. Due to pyrophoric properties a number of other tests on physicochemical, toxicological and ecotoxicological endpoints cannot be conducted.

**FURTHER ADAPTATION POSSIBILITIES**

Not foreseen.

**Impurities; uncertainties**

More background information and guidance on this and other aspects is given in the *Guidance on the Application of the CLP Criteria*, section 2.10.

**How to conclude on the DSD classification**

Because the test methods of DSD and CLP are identical for this endpoint there is no difference in classification, see also the *Guidance on the Application of the CLP Criteria*, section 2.10.6.

**Endpoint specific information in the registration dossier IUCLID****Material and methods**

Description of the apparatus and dimensions or reference to the standard or the test method applied. Note that in this case the experience in handling may be sufficient.

**Results and discussion**

- whether ignition occurs when poured or whether the filter paper is charred;
- if testing is waived, the reasons for waiving must be documented in the dossier.

Any deviation from the guideline method used (and reasons for it) or any other special consideration should be reported. In cases where there is more than one source of data, the endpoint summary under results and discussion should provide a justification for the selection of the key study chapter.

**Reference to other ECHA Guidance Documents**

Further detailed guidance on flammability can be found in the following chapters:

IUCLID	REACH	Endpoint	IUCLID 5 End User Manual	ECHA Practical Guide
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Section	Annex	title	Chapter	3
4.13	VII 7.10	Flammability	E.4.14	3.11

## Further information / references

Background information and guidance on classification testing, additional testing and available information is given in the *Guidance on the Application of the CLP Criteria*, section 2.10.

### R.7.1.10.6 Pyrophoric solids

#### Definition

The definition of a pyrophoric solid is given in CLP Regulation Annex I, section 2.10.1.

*Pyrophoric solid means a solid substance or mixture which, even in small quantities, is liable to ignite within five minutes after coming into contact with air.*

Background information and guidance on the definition is given in the *Guidance on the Application of the CLP Criteria*, sections 2.10.1 and 2.10.2.

#### Classification criteria and relevant information

Classification principles are given in CLP Regulation Annex I, section 2.10.2.

The criterion for a pyrophoric solid is as follows: *The solid ignites within 5 minutes of coming into contact with air.*

Background information and guidance on relevant aspects regarding the classification is given in the *Guidance on the Application of the CLP Criteria*, sections 2.10.1, 2.10.2, 2.10.3 and 2.10.4.

#### Adaptation of the standard testing regime

#### ADAPTATION POSSIBILITIES ACCORDING TO COLUMN 2 OF ANNEX VII TO REACH

Other flammability tests do not have to be performed in addition to the determination of the self-ignition temperature, if the substance is a pyrophoric substance. However, flammability in contact with water may be relevant.

#### ADAPTATION POSSIBILITIES ACCORDING TO ANNEX XI TO REACH

#### USE OF EXISTING DATA: DATA ON PHYSICAL-CHEMICAL PROPERTIES FROM EXPERIMENTS NOT CARRIED OUT ACCORDING TO GLP OR THE TEST METHODS REFERRED TO IN ARTICLE 13 (3) OF REACH

The UN Test method N.2 of the UN Manual of Tests and Criteria is also used for classification according to the regulations on the transport of dangerous goods (ADR and RID). If the solid in question has been classified as belonging to Class 4.2, packing group I of the ADR/RID on the basis of UN Test N.2 results, it is a pyrophoric solid according to CLP Regulation criteria. Packing group I of the ADR/RID directly corresponds to Category 1 of CLP.

1 If experimental data are available (study reports or literature data) meeting the criteria in  
2 section 1.1.1 of Annex XI to REACH, these could be used to meet the endpoint data  
3 requirements. If an estimation method is used as a source of information according to Column  
4 2 of Annex VII, the QSAR model must meet the criteria set out in section 1.3 of Annex XI to  
5 REACH.

6 According to the DSD, the A.13 method of Regulation (EC) 440/2008 is used for the  
7 assessment of pyrophoric properties for solids and liquids. This method is identical to the N.2  
8 test method.

## 9 **WEIGHT OF EVIDENCE**

10 Where no single source of existing data (study reports, QSAR, literature data) is considered  
11 sufficiently reliable, thus not fully meeting the criteria in section 1.1.1 of Annex XI to REACH,  
12 or where several sources of similar reliability with deviating results exist, a weight of evidence  
13 approach may be used. The criteria in section 1.2 of Annex XI to REACH must then be met.

## 14 **(Q)SAR**

15 Application of (Q)SAR is not possible, however assessment of the chemical structure may be  
16 used to exclude pyrophoric properties of a substance. Such an assessment of chemical  
17 structure, in conjunction with experience in manufacture and handling, could also formally  
18 form part of a weight-of-evidence argument.

## 19 **GROUPING OF SUBSTANCES AND READ-ACROSS APPROACH**

20 Assessment of the chemical structure may be used to anticipate pyrophoric properties of a  
21 substance.

## 22 **TESTING IS TECHNICALLY NOT POSSIBLE**

23 Testing should always be considered if none of the waiving possibilities applies. Due to  
24 pyrophoric properties a number of other tests on physicochemical, toxicological and  
25 ecotoxicological endpoints cannot be conducted.

## 26 **FURTHER ADAPTATION POSSIBILITIES**

27 Not foreseen.

## 28 **Impurities; uncertainties**

29 Particle size may play an important role. More background information and guidance on this  
30 and other aspects is given in the *Guidance on the Application of the CLP Criteria*, section 2.10.

## 31 **How to conclude on the DSD classification**

32 Because the test methods of DSD and CLP Regulation are identical for this endpoint there is no  
33 difference in classification, see also the *Guidance on the Application of the CLP Criteria*, section  
34 2.10.6.

## 35 **Endpoint specific information in the registration dossier/in IUCLID**

### 36 **Material and methods**



• description of the apparatus and dimensions or reference to the standard or the test method applied;

• particle size and distribution (if practicable);

Note that in this case experience in handling may be sufficient.

## Results and discussion

• whether ignition occurs when poured;

• if testing is waived, the reasons for waiving must be documented in the dossier.

Any deviation from the guideline method used (and reasons for it) or any other special consideration should be reported. In cases where there is more than one source of data, the endpoint summary under results and discussion should provide a justification for the selection of the key study chapter.

## Reference to other ECHA Guidance Documents

Further detailed guidance on flammability can be found in the following chapters:

IUCLID Section	REACH Annex	Endpoint title	IUCLID 5 End User Manual Chapter	ECHA Practical Guide 3
4.13	VII 7.10	Flammability	E.4.14	3.11

## Further information / references

Background information and guidance on classification testing, additional testing and available information is given in the *Guidance on the Application of the CLP Criteria*, section 2.10.

### R.7.1.10.7 Self-heating substances and mixtures

#### Definition

For solids and liquids absorbed onto a large surface, self-heating may occur by reaction with air with subsequent ignition. According to the section 2.11.1.1 of Annex I to CLP Regulation:

*A self-heating substance or mixture is a liquid or solid substance or mixture, other than a pyrophoric liquid or solid, which, by reaction with air and without energy supply, is liable to self-heat; this substance or mixture differs from a pyrophoric liquid or solid in that it will ignite only when in large amounts (kilograms) and after long periods of time (hours or days).*

#### Classification criteria and relevant information

Self-heating substances and mixtures are classified in two categories according to the criteria of the CLP Regulation (see section 2.11, table 2.11.1). In general, self-heating occurs only for solids in contact with air. The *Guidance on the Application of the CLP Criteria*, section 2.11 gives detailed background information about this phenomenon.

## 1 **Adaptation of the standard testing regime**

### 2 **ADAPTATION POSSIBILITIES ACCORDING TO COLUMN 2 OF ANNEX VII TO REACH**

3 Column 2 of the REACH Annex VII provides the following specific rules for adaptation of the  
4 standard information requirement for self-ignition temperature.

5 *The study does not need to be conducted:*

- 6 • *if the substance is explosive or ignites spontaneously with air at room temperature; or*
- 7 • *for liquids non flammable in air, e.g. no flash point up to 200 °C, or*
- 8 • *for gases having no flammable range, or*
- 9 • *for solids, if the substance has a melting point < 160 °C, or if preliminary results*  
10 *exclude self-heating of the substance up to 400 °C.*

11  
12 The first indent specifies that no data is required for substances which is explosive or ignites  
13 spontaneously with air at room temperature.

14 Second and third indent are not applicable for this endpoint.

15 With regards to fourth indent, for the purposes of REACH, no data are required for solids  
16 classified as:

- 17 • pyrophoric; or
- 18 • explosive, unstable or division 1.1 to 1.6; or
- 19 • organic peroxide; or
- 20 • self-reactive substance.

21  
22 Further, no data are required for substances with a melting point below 160 °C. This means  
23 also that liquids do not have to be tested for this endpoint for the purposes of this regulation.  
24 Annex VII of REACH also allows waiving "*if preliminary results exclude self-heating of the*  
25 *substance up to 400 °C.*" This refers to Test Method Regulation 440/2008, method A.16.  
26 However, the criteria are not very clear, and therefore it is recommended to instead refer to  
27 the CLP Regulation classification criteria, if applicable, and to waive otherwise.

### 28 **ADAPTATION POSSIBILITIES ACCORDING TO ANNEX XI TO REACH**

#### 29 **USE OF EXISTING DATA: DATA ON PHYSICAL-CHEMICAL PROPERTIES FROM** 30 **EXPERIMENTS NOT CARRIED OUT ACCORDING TO GLP OR THE TEST METHODS** 31 **REFERRED TO IN ARTICLE 13 (3) OF REACH**

32 Literature data – even if available – should not be used since self-heating strongly depends on  
33 particle size, surface treatment and other parameters.

34 The use of existing data is possible provided that the test has been carried out by a qualified  
35 institution. If available data from a test according to method A.16 indicate that a classification  
36 as a self-heating substance does not apply, no more testing is necessary. However, the  
37 interpretation of the A.16 test method data in terms of the CLP criteria requires appropriate  
38 expert knowledge.

## **WEIGHT OF EVIDENCE**

For the determination of the self-heating substances and mixtures, weight of evidence is not possible.

## **(Q)SAR**

At present (Q)SAR is generally not applicable for self-heating substances and mixtures. Application of QSAR is not possible.

## **GROUPING OF SUBSTANCES AND READ-ACROSS APPROACH**

At present grouping and read-across are not applicable.

## **TESTING IS TECHNICALLY NOT POSSIBLE**

In some cases, exothermic decomposition may occur when performing the test, and special care will be necessary with respect to performing the tests and interpreting the results; see the *Guidance on the Application of the CLP criteria*, section 2.11.4.4.3. In such cases, it may not be possible to determine these properties.

## **FURTHER ADAPTATION POSSIBILITIES**

According to the UN Manual of Tests and Criteria, the classification procedure for self-heating substances or mixtures need not be applied if the results of a screening test can be adequately correlated with the classification test and an appropriate safety margin is applied. Examples of screening tests are:

(a) The Greiner Oven test (VDI guideline, 1990) with an onset temperature 80 K above the reference temperature for a volume of 1 litre;

(b) The Bulk Powder Screening Test (Gibson *et al.*, 1985) with an onset temperature 60 K above the reference temperature for a volume of 1 litre.

As stated in Annex IX of REACH, when for certain endpoints, it is proposed to not provide information for other reasons than those mentioned in column 2 of that Annex or in Annex XI of REACH, this fact and the reasons must also be clearly stated. Such an approach may then be used. The *Guidance on the Application of the CLP Criteria*, section 2.11.4.2 should be consulted for details about waiving and screening criteria.

## **Impurities; uncertainties**

Particle size may play an important role. More background information and guidance on this and other aspects is given in the *Guidance on the Application of the CLP Criteria*, section 2.11.

## **How to conclude on the DSD classification**

This hazard class is not defined in DSD, therefore translation is not possible.

## **Endpoint specific information in the registration dossier/in IUCLID**

### **Material and methods**

- description of the apparatus and dimensions or reference to the standard or the test method applied;

- indicate if preliminary and/or main test performed;
- moisture content;
- particle size and distribution (if available).

## Results and discussion

- indicate temperature rise obtained for the individual tests and classification result.

Any deviation from the guideline method used or any other special consideration should be reported. In cases where there is more than one source of data, the endpoint summary under results and discussion should provide a justification for the selection of the key study chapter.

## Reference to other ECHA Guidance Documents

Further detailed guidance on flammability can be found in the following chapters:

IUCLID Section	REACH Annex	Endpoint title	IUCLID 5 End User Manual Chapter	ECHA Practical Guide 3
4.13	VII 7.10	Flammability	E.4.14	3.11

## Further information / references

ECHA guidance document *the Guidance on the Application of the CLP Criteria* gives in section 2.11 detailed information on the self-heating property, the CLP-classification, the relevant test method and the relation to the DSD and the transport of dangerous goods regulations.

VDI guideline 2263, part 1 (1990): "Test methods for the Determination of the Safety Characteristics of Dusts".

Gibson, N. Harper, D.J. Rogers (1985): "Evaluation of the fire and explosion risks in drying powders", *Plant Operations Progress*, 4 (3), 181-189.

### R.7.1.10.8 Substances which in contact with water emit flammable gases

#### Definition

The CLP Regulation, Annex I, section 2.12.1 provides the following definition:

*Substances or mixtures which, in contact with water, emit flammable gases means solid or liquid substances or mixtures which, by interaction with water, are liable to become spontaneously flammable or to give off flammable gases in dangerous quantities.*

#### Classification criteria and relevant information

Classification according to the CLP Regulation is required if the gas produced upon contact with water ignites spontaneously and/or if the reaction rate with which the flammable gas is produced is  $\geq 1$  l/kg h.

If the gas produced ignites spontaneously, this does not necessarily imply that the gas produced is pyrophoric but this generally is the case if the heat of reaction is sufficient to result in ignition of the gas.

The test method for classification of substances and mixtures which in contact with water emit flammable gases is described in the UN Manual of Tests and Criteria (UN Test N.5, see Section 33.4). This method is referred to in Annex I, Part 2 of the CLP Regulation and it is strongly recommended to use this method and not to apply test method A.12 of the Test Methods

Regulation if new testing is carried out. UN Test N.5 foresees dividing into three categories depending on the violence and rate of the reaction whereas test method A.12 does not allow any further dividing of the substances. Furthermore, the results of both methods might differ slightly due to some differences in the testing procedure (for these differences see the *Guidance on the Application of the CLP Criteria*, Section 2.12.5). Therefore unnecessary testing can be avoided by applying only UN Test N.5 because it leads to more detailed information (and has in any case to be applied for other purposes such as classification and transport).

Data which is based on the classification according to DSD may be available. There are, however, differences between the methods UN Test N.5 and A.12 which should be considered. They are described in detail in the *Guidance on the Application of the CLP Criteria*, section 2.12.5.

Detailed guidance on the test method itself can be found in the *Guidance on the Application of the CLP Criteria*, section 2.12.3.4.1.

## **Adaptation of the standard testing regime**

### **ADAPTATION POSSIBILITIES ACCORDING TO COLUMN 2 OF ANNEX VII TO REACH**

Column 2 of REACH Annex VII provides the following specific rules for adaptation of the standard information requirement for flammability.

*The study does not need to be conducted:*

- if the substance is a solid which possesses explosive or pyrophoric properties. These properties should always be considered before considering flammability; or*
- for gases, if the concentration of the flammable gas in a mixture with inert gases is so low that, when mixed with air, the concentration is all time below the lower limit; or*
- for substances which spontaneously ignite when in contact with air.*

The first point is valid with regard to explosive substances because they are not classified as substances which in contact with water emit flammable gases. In that case testing can be waived.

The other waiving possibilities are not applicable with regard to substances which in contact with water emit flammable gases.

The first point is not correct with regard to pyrophoric substances because pyrophoric substances can be classified as substances which in contact with water emit flammable gases based on UN Test N.5 which is referred to by CLP. UN Test N.5 explicitly requires testing of pyrophoric substances under nitrogen (see UN Manual of Tests and Criteria, section 33.4.1.3.1).

The second point is not applicable because gases do not fall under the hazard class of substances which in contact with water emit flammable gases.

For the same reasons, the last point (waiving would be possible for substances which spontaneously ignite when in contact with air) is also not valid in this case.

## 1 ADAPTATION POSSIBILITIES ACCORDING TO ANNEX XI TO REACH

### 2 USE OF EXISTING DATA: DATA ON PHYSICAL-CHEMICAL PROPERTIES FROM 3 EXPERIMENTS NOT CARRIED OUT ACCORDING TO GLP OR THE TEST METHODS 4 REFERRED TO IN ARTICLE 13 (3) OF REACH

5 If experimental data are available (study reports or literature data) meeting the criteria in  
6 section 1.1.1 of Annex XI to REACH, these could be used to meet the endpoint data  
7 requirements. If an estimation method is used as a source of information according to Column  
8 2 of Annex VII, the QSAR model must meet the criteria set out in section 1.3 of Annex XI to  
9 REACH.

## 10 WEIGHT OF EVIDENCE

11 Where no single source of existing data (study reports, QSAR, literature data) is considered  
12 sufficiently reliable, thus not fully meeting the criteria in section 1.1.1 of Annex XI to REACH,  
13 or where several sources of similar reliability with deviating results exist, a weight of evidence  
14 approach may be used. The criteria in section 1.2 of Annex XI to REACH must then be met.

## 15 (Q)SAR

16 There are currently no QSPR models for predicting whether a substance in contact with water  
17 emits flammable gases and if so what the gas evolution rate is.

## 18 GROUPING OF SUBSTANCES AND READ-ACROSS APPROACH

19 At present grouping and read-across are not applicable.

## 20 TESTING IS TECHNICALLY NOT POSSIBLE

21 Testing should always be possible if none of the waiving possibilities applies. If the substance  
22 is known to be soluble in water to form a stable solution, or if it is clearly known that it does  
23 not react with water, e.g. because it is manufactured or washed with water, testing is not  
24 necessary.

## 25 FURTHER ADAPTATION POSSIBILITIES

26 Classification in certain hazard classes do not foresee the assignment of further physical  
27 hazard classes or at least normally do not match with classification in this hazard class:

- 28 • Substances that are classified as explosives, self-reactives or organic peroxides are not  
29 classified in this hazard class (or any other physical hazard class). For explosives this is  
30 considered through the first point of the adaptation possibilities according to REACH Annex  
31 VII, column 2 (see above).
- 32 • Oxidizing substances are generally not considered for flammability and therefore are also  
33 not classified in this hazard class (there may be some exceptions, however).

35 As stated in Annex IX of REACH, when for certain endpoints, it is proposed to not provide  
36 information for other reasons than those mentioned in column 2 of that Annex or in Annex XI  
37 of REACH, this fact and the reasons must also be clearly stated. Such an approach may then  
38 be used.

## Impurities; uncertainties

The descriptions of the methods UN Test N.5 and A.12 are not very detailed and therefore allow for technical variations such as with regard to the apparatus used or the procedure. In particular, the testing protocol does not prescribe a specific method for measuring the gas evolution rate. An interlaboratory comparison for this test method has shown that laboratories - based on the freedom the description of the test methods gives - apply different approaches when performing this test. Furthermore, the interlaboratory comparison showed that the test results vary in a rather wide range. It therefore has to be kept in mind that this test method has a non-negligible uncertainty with regard to trueness and precision. Therefore utmost care should be taken in the selection of the key study(s), or weight-of-evidence approaches, that the data selected is representative of the substance being registered by the respective companies.

Sea water may be a particular case of interest (in case of maritime transport).

## How to conclude on the DSD classification

Substances which in contact with water emit flammable gases would be classified as 'F; R15' under DSD (the sum of categories 1 to 3 corresponds to 'F; R15').

## Endpoint specific information in the registration dossier/in IUCLID

### Material and methods

- description of the apparatus and dimensions or reference to the standard or the test method applied;
- particle size and distribution.

### Results and discussion

- indicate whether full test was performed or whether it was terminated at a particular step/stage;
- substance identity of evolved gas;
- indicate whether the gas evolved ignites spontaneously;
- rate of gas evolution (unless the test has been terminated);
- if testing is waived, the reasons for waiving must be documented in the dossier.

Any deviation from the guideline method used (and reasons for it) or any other special consideration should be reported. In cases where there is more than one source of data, the endpoint summary under results and discussion should provide a justification for the selection of the key study chapter.

## Reference to other ECHA Guidance Documents

Further detailed guidance on flammability is found in the following chapters:

IUCLID Section	REACH Annex	Endpoint title	IUCLID 5 End User Manual Chapter	ECHA Practical Guide 3
4.13	VII 7.10	Flammability	E.4.14	3.11

## Further information / references

The ECHA document *Guidance on the Application of the CLP Criteria* gives in its section 2.12 detailed information on substances and mixtures which, in contact with water, emit flammable



gases, their CLP-classification, the relevant test method and the relation to the DSD and the transport of dangerous goods regulations.

Janès *et al.*, "Towards the improvement of UN N.5 test method intended to the characterization of substances which in contact with water emit Flammable Gases", submitted in revised form to the Journal of Loss Prevention in the Process Industries.

Interlaboratory test on the method UN Test N.5 / EC A.12 "Substances which, in contact with water, emit flammable gases" 2007, Kunath, K., Lüth, P., Uhlig, S., ISBN 978-3-9814634-1-5, [http://www.bam.de/de/service/publikationen/publikationen\\_medien/short\\_report\\_rv\\_un\\_n\\_5.pdf](http://www.bam.de/de/service/publikationen/publikationen_medien/short_report_rv_un_n_5.pdf).

#### R.7.1.10.9 Organic peroxides

In the Dangerous Substances Directive (DSD) (67/548/EEC) organic peroxides were classified on the basis of their chemical structure either as explosive or as oxidising. In general, organic peroxides have only weak oxidising properties or do not show oxidizing properties at all. In the CLP Regulation organic peroxides are a distinct hazard class. Organic peroxides are classified in one of the seven categories of "Types A to G" according to the classification criteria given in Section 2.15.2 of Annex I, of CLP.

As mentioned below under sub-section Definition, organic peroxides are excluded from testing as explosives according to Test Series 1 to 8 in Part I of the UN Recommendations on the Transport of Dangerous Goods, Manual of Tests and Criteria (see R.7.1.11.1 Explosives). In Test Series A to H however, no tests on sensitivity to impact (solids and liquids) and friction (solids only) are included. For the risk assessment and the safe use and handling, data according to the EU test method A.14 as described in Regulation (EC) No 440/2008, if available, or UN Test 3 (a) (ii) BAM Fallhammer and Test 3 (b) (i) BAM friction apparatus (see R.7.1.11) should be part of the hazard communication in the registration dossier (REACH Annex VII, 7.11) and in the safety data sheet.

#### Definition

The definition of an organic peroxide is given in CLP Annex I, section 2.15.1:

*Organic peroxide means a liquid or solid organic substance which contains the bivalent -O-O- structure and as such is considered a derivative of hydrogen peroxide, where one or both of the hydrogen atoms have been replaced by organic radicals. The term organic peroxide includes organic peroxide mixtures (formulations) containing at least one organic peroxide. Organic peroxides are thermally unstable substances or mixtures, which can undergo exothermic self-accelerating decomposition. In addition, they can have one or more of the following properties:*

- (i) Be liable to explosive decomposition;*
- (ii) Burn rapidly;*
- (iii) Be sensitive to impact or friction;*
- (iv) React dangerously with other substances.*

*An organic peroxide is regarded as possessing explosive properties when in laboratory testing the mixture (formulation) is liable to detonate, to deflagrate rapidly or to show a violent effect when heated under confinement.*

Background information and guidance on the definition is given in *Guidance on the Application of the CLP Criteria*, sections 2.14.1 and 2.14.2.

## Classification criteria and relevant information

The Classification principles are given in CLP Annex I, sections 2.15.2 and 2.15.4. Background information and guidance on relevant aspects regarding the classification is given in *Guidance on the Application of the CLP Criteria*, sections 2.14.3, 2.14.4, 2.14.6, 2.14.7 and 2.14.8.

## Adaptation of the standard testing regime

### ADAPTATION POSSIBILITIES ACCORDING TO COLUMN 2 OF ANNEX VII TO REACH

Only organic peroxides, as defined in CLP, Annex I, section 2.15.1 definition, have to be tested according to the United Nations Manual of Tests and Criteria, Part II test series A - H.

*The study does not need to be conducted if:*

*- the substance or mixture contains:*

*(a) not more than 1.0% available oxygen from the organic peroxides when containing not more than 1.0% hydrogen peroxide; or*

*(b) not more than 0.5% available oxygen from the organic peroxides when containing more than 1.0% but not more than 7.0% hydrogen peroxide.*

**NOTE:**

*The available oxygen content (%) of an organic peroxide mixture is given by the formula:*

$$16 \times \sum_i^n \left( \frac{n_i \times c_i}{m_i} \right)$$

*where:*

*$n_i$  = number of peroxygen groups per molecule of organic peroxide  $i$ ;*

*$c_i$  = concentration (mass %) of organic peroxide  $i$ ;*

*$m_i$  = molecular mass of organic peroxide  $i$ .*

### ADAPTATION POSSIBILITIES ACCORDING TO ANNEX XI TO REACH

#### USE OF EXISTING DATA: DATA ON PHYSICAL-CHEMICAL PROPERTIES FROM EXPERIMENTS NOT CARRIED OUT ACCORDING TO GLP OR THE TEST METHODS REFERRED TO IN ARTICLE 13 (3) OF REACH

A number of already tested and classified substances and mixtures are listed in the United Nations Recommendations on the Transport of Dangerous Goods, 2.5.3.2.4.

Available information may especially originate from the classification for transport. In the DSD organic peroxides were classified as oxidizing substances, by definition. More details are described in the *Guidance on the Application of the CLP Criteria*, sections 1.7.2.1 and 2.14.6. If experimental data are available (study reports or literature data) meeting the criteria in section 1.1.1 of Annex XI to REACH, these could be used to meet the endpoint data requirements. If an estimation method is used as a source of information according to Column 2 of Annex VII, the QSAR model must meet the criteria set out in section 1.3 of Annex XI to REACH.

## 1 **WEIGHT OF EVIDENCE**

2 For the determination of the organic peroxides, weight of evidence is not possible. Where no  
3 single source of existing data (study reports, QSAR, literature data) is considered sufficiently  
4 reliable, thus not fully meeting the criteria in section 1.1.1 of Annex XI to REACH, or where  
5 several sources of similar reliability with deviating results exist, a weight of evidence approach  
6 may be used. The criteria in section 1.2 of Annex XI to REACH must then be met.

## 7 **(Q)SAR**

8 At present QSAR is generally not applicable for organic peroxides. Application of (Q)SAR is not  
9 possible.

## 10 **GROUPING OF SUBSTANCES AND READ-ACROSS APPROACH**

11 At present grouping and read across are not applicable.

## 12 **TESTING IS TECHNICALLY NOT POSSIBLE**

13 A number of substances can, for safety reasons, only be handled and tested in diluted form,  
14 see the substances and mixtures listed in UN TDG, 2.5.3.2.4. Testing should always be  
15 considered if none of the waiving possibilities applies.

## 16 **FURTHER ADAPTATION POSSIBILITIES**

17 Not foreseen.

## 18 **Impurities; uncertainties**

19 Minor impurities can have an influence on thermal stability. Background information and  
20 guidance on these aspects is given in *Guidance on the Application of the CLP Criteria*, section  
21 2.14.4.

## 22 **How to conclude on the DSD classification**

23 In the DSD organic peroxides are classified as oxidizing substances and a few of them as  
24 having explosive properties.

## 25 **Endpoint specific information in the registration dossier/in IUCLID**

### 26 **Material and methods**

- 27 • See UN MTC, Part II, classification procedures and test series A-H.

### 28 **Results and discussion**

29 The following data on organic peroxides should be submitted:

- 30 • if testing is waived, the reasons for waiving must be documented in the dossier;  
31 • type of organic peroxide;  
32 • SADT (Self accelerating decomposition temperature) together with the volume the SADT  
33 related to;

- detonation properties (Yes/Partial/No);
  - deflagration properties (Yes rapidly/Yes slowly/No);
  - effect of heating under confinement (Violent/Medium/Low/No);
  - explosive power, if applicable (Not low/Low/None).
- The following example shows how data mentioned above could be documented in the CSR:

**Figure R.7.1-3 Example: Di-tert-butyl peroxide**

UN Test Series A to H	Test method	Results + Evaluation	Remarks
Propagation of detonation	A.1	"No"	Fragmented length (cm): 16
Propagation of deflagration #1	C.1	"Yes, slowly "	Maximum pressure (kPa): > 2070 Time for a pressure rise from 690 to 2070 kPa (ms): 100
Propagation of deflagration #2	C.2	"No"	deflagration rate (mm/s): 0.27
Effect of heating under defined confinement #1	Koenen E.1	"No"	Limiting diameter (mm): < 1.0 Type of fragmentation (and pieces): 0
Effect of heating under defined confinement #2	DPVT E.2	"Medium"	Limiting diameter (mm): 3.5
Explosive power	F.3	"Not Low"	Expansion (cm <sup>3</sup> /10 g test sample): 28
Explosive power	F.4	"Not Low"	Average net expansion (cm <sup>3</sup> ): 12
SADT	H.4	80 °C	500 ml Dewar vessel
Competent Authority approval number	<i>Example from UN Recommendations on the Transport of Dangerous Goods, Manual of Tests and Criteria</i>		

For assigning the Type of organic peroxide, the list of currently assigned organic peroxides according 2.5.3.2.4 of the UN RTDG can be used, in case the assignment was based on a test according to the UN MTC. The relevant underlying test data may be collected from the respective UN documents from the UN committee of experts on the transport of dangerous goods, from test reports produced by either competent authorities or industry, or from other reliable sources (such as e.g. the dedicated database 'DATATOP').

Any deviation from the guideline method used (and reasons for it) or any other special consideration should be reported. In cases where there is more than one source of data, the endpoint summary under results and discussion should provide a justification for the selection of the key study chapter.

## Reference to other ECHA Guidance Documents

A Template data set in IUCLID does not exist for the hazard class "organic peroxides". As long as there is no specific section in IUCLID the test results in section 4.23 "Additional physico-chemical information" should be inserted under the endpoint title "organic peroxides". The information on organic peroxides should not be included in IUCLID section 4.15 "Oxidising properties". In the registration dossier the information should be included under flammability.

## Further information / references

Background information and guidance on classification testing, additional testing and available information is given in *Guidance on the Application of the CLP Criteria*, section 2.14.

Data from the 'DATATOP' database can be obtained from the gatekeeper of this database TNO, Department Energetic Materials, Lange Kleiweg 137, 2288GJ, Rijswijk The Netherlands.

Various national guidelines which provide guidance and outline safe standards for handling and storage of organic peroxides for the assignment of organic peroxides to storage groups are available e.g. Netherlands Directive: Publication Series on Dangerous Substances 8 (PGS 8) Storage of Organic Peroxides, UK HSE: The storage and handling of organic peroxides - Guidance Note CS21 or German guideline: BGV B4.

### R.7.1.11 Explosive properties

Some of the information requirements according to the REACH Regulation, Annex VII were phrased such that they correspond to 'indications of danger' as given in Annex II of DSD. For substances, classification and labelling according to the CLP Regulation has been mandatory since December 1, 2010 (and will become mandatory for mixtures (preparations) from June 1, 2015, when the DSD and DPD will be repealed). Consequently, explosive properties are covered by classification of the substance according to the CLP Regulation. However, the physical hazards according to CLP are structured completely differently from the physico-chemical properties according to the DSD (and therefore also REACH, Annex VII). This means that for some of the CLP hazard classes an unambiguous assignment to one of the headlines (information requirements) in Annex VII to REACH is not possible. The assignment of hazard classes to the headline 'Explosive properties' as shown in Table below must therefore only be understood as a means to structure this document in accordance with Annex VII to REACH. It has to be noted that self-reactive substances and organic peroxides are primarily assigned to the headline 'Flammability' and only a cross reference to corresponding sub-chapter under heading 'Flammability' is included in the sub-chapters on 'Explosive properties' below because these two hazard classes can have explosive and/or flammable properties.

**Table R.7.1-7 Assignment of CLP hazard classes to the information requirement 'Explosive properties' according to REACH, Annex VII and correlation between the Test method Regulation and the test method according to CLP and supporting link with the Guidance on the Application of the CLP Criteria.**

Information requirement according to Art. 10 (a) (vi) of the REACH Regulation (EC) No. 1907/2006 (the no. in brackets is the respective no. in the table in Annexes VII to XI to REACH)	CLP Regulation (EC) No. 1272/2008 (the no. in brackets is the respective chapter no. in Annex I to CLP)	Chapter in revised R.7(a) guidance	Corresponding test method according to the Test Method Regulation, Regulation (EC) 440/2008	Corresponding test method according to the CLP Regulation	Chapter in the Guidance on the Application of the CLP Criteria (ex RIP 3.6)
Explosive properties (7.11)	Explosives (2.1)*	R.7.1.11.1	A.14 Explosive properties	<b>UN Test series 1 to 3 (further test series 4 to 6)</b>	2.2

				are necessary for classification)	
	Self-reactive substances and mixtures (2.8)*	R.7.1.11.2 see R.7.1.10.4	n.a.	<b>A.14 (existing data only)</b>	2.9
	Organic peroxides (2.15)*	R.7.1.11.3 See R.7.1.10.9	n.a.	<b>A.14 (existing data only)</b>	2.14

\* Note that regardless of whether the hazard class or category is listed in Article 14(4)(a) REACH the chemical safety assessment must be performed in accordance with Article 14 (3) of REACH. Furthermore, according to Article 10(a)(iv) of REACH the technical dossier of a registration of a substance under the REACH Regulation must include information on classification and labelling of the substance as specified in section 4 of Annex VI to the REACH Regulation.

In addition, it has to be noted that some substances have explosive properties which do not result in classification. Examples are the following:

- Substances with a positive result in UN Test Series 1 or 2 but which are exempted from the classification as explosives based on their packaging in UN Test Series 6;
- Substances which are mechanically sensitive only. These are substances with a sensitiveness to impact (determined by UN Test Series 3 (a) (ii)) of 40 J or less and/or a sensitiveness to friction (determined by Test Series 3 (b) (i)) of 360 N or less for substances and mixtures which may have explosive properties based on the screening procedure according to Appendix 6, Part 3 of the UN RTDG Manual of Tests and Criteria and which are not classified as explosives, self-reactive or organic peroxide.

Such substances may be classified in other hazard classes (e.g. as flammable solids, oxidizing solids, corrosive to metals) or even not at all. Information about such explosive properties should be indicated in the dossier as well.

#### R.7.1.11.1 Explosives

Please note that explosive atmospheres as, for example, created by flammable liquids and by powders are not the subject of this chapter.

#### Definition

The following definitions are provided in CLP Annex I, section 2.1.1:

*An explosive substance or mixture is a solid or liquid substance or mixture of substances which is in itself capable by chemical reaction of producing gas at such a temperature and pressure and at such a speed as to cause damage to the surroundings. Pyrotechnic substances are included even when they do not evolve gases.*

*A pyrotechnic substance or mixture is a substance or mixture of substances designed to produce an effect by heat, light, sound, gas or smoke or a combination of these as the result of non-detonative self-sustaining exothermic chemical reactions.*

*An unstable explosive is an explosive substance or mixture which is thermally unstable and/or too sensitive for normal handling, transport and use.*

*An explosive article is an article containing one or more explosive substances or mixtures.*

1 *A pyrotechnic article is an article containing one or more pyrotechnic substances or mixtures.*

2 *An intentional explosive is a substance, mixture or article which is manufactured with a view to*  
3 *producing a practical, explosive or pyrotechnic effect.*

4 Organic Peroxides and Self Reactive Substances may also have explosive properties and should  
5 be screened. See chapter R.7.1.11.3 for Organic peroxides and chapter R.7.1.11.2 for Self  
6 Reactive Substances and Mixtures.

#### 7 **Intentional explosive**

8 Council Directive 93/15/EEC of 5 April 1993 lays down rules for the harmonisation of the  
9 provisions relating to the placing on the market and supervision of explosives for civil uses.

10 Directive 2007/23/ EC on the placing on the market of pyrotechnic articles establishes rules  
11 designed to achieve the free movement of pyrotechnic articles in the internal market while, at  
12 the same time, ensuring a high level of protection of human health and public security and the  
13 protection and safety of consumers and taking into account the relevant aspects related to  
14 environmental protection. Pyrotechnic articles (CLP, Annex I, Section 2.1.1.2) are classified as  
15 explosives for CLP and as class 1 for transport (see UN-RTDG). According to Article 9 and Annex  
16 II of Directive 2007/23/EC the conformity assessment procedures are carried out by notified  
17 bodies, which have to issue an EC type-examination certificate to the applicant. All data  
18 included in the EC type-examination certificate are sufficient for the information requirements  
19 under the REACH Regulation.

#### 20 **Classification criteria and relevant information**

21 Substances, mixtures and articles of this class are classified as an unstable explosive on the  
22 basis of the flowchart in Annex I to CLP Regulation, Figure 2.1.2. The test methods are  
23 described in Part I of the UN Recommendations on the Transport of Dangerous Goods, Manual  
24 of Tests and Criteria.

25 Explosives, which are not classified as an unstable explosive, must be classified in one of the  
26 six Divisions referred to in paragraph 2.1.2.2 of Annex 2.1 to the CLP Regulation, based on the  
27 results of the tests laid down in Table 2.1.1 on Test Series 2 to 8 in Part I of the UN  
28 Recommendations on the Transport of Dangerous Goods, Manual of Tests and Criteria. If  
29 explosives are unpackaged or repacked in packaging other than the original or similar  
30 packaging, they must be retested. If a substance gives a positive result in any of the test  
31 series 1 or 2 this should be mentioned in the REACH registration dossier for the substance,  
32 even if it would not be classified as an 'Explosive' in Test Series 6.

33 The test methods used for deciding on provisional acceptance into the class of explosives are  
34 grouped into four series, numbered 1 to 4 (see CLP Annex I, Figure 2.1.2).

35 It may be important for the safety of testers that certain tests, using small amounts of  
36 material, be conducted first before proceeding to test with larger quantities. Therefore it is  
37 highly recommended to start the testing procedure with Test Series 3, because these tests  
38 involve relatively small sample sizes, which reduces the risk to personnel.

#### 39 **Adaptation of the standard testing regime**

#### 40 **ADAPTATION POSSIBILITIES ACCORDING TO COLUMN 2 OF ANNEX VII TO REACH**

41 Column 2 of REACH Annex VII provides the following specific rules for adaptation of the  
42 standard information requirement for explosive properties.

43 *The study does not need to be conducted if:*

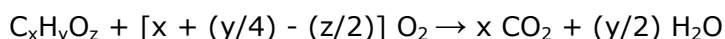


- *there are no chemical groups associated with explosive properties present in the molecule, or*
- *the substance contains chemical groups associated with explosive properties which include oxygen and the calculated oxygen balance is less than -200, or*
- *the organic substance or a homogenous mixture of organic substances contains chemical groups associated with explosive properties, but the exothermic decomposition energy is less than 500 J/g and the onset of exothermic decomposition is below 500 °C, or*
- *for mixtures of inorganic oxidising substances (UN Division 5.1) with organic materials, the concentration of the inorganic oxidising substance is:*
  - *less than 15 %, by mass, if assigned to UN Packaging Group I (high hazard) or II (medium hazard),*
  - *less than 30 %, by mass, if assigned to UN Packaging Group III (low hazard).*

*Note: Neither a test for propagation of detonation nor a test for sensitivity to detonative shock is required if the exothermic decomposition energy of organic materials is less than 800 J/g.*

#### Note on the use of the Oxygen Balance:

The oxygen balance is calculated for the chemical reaction:



Using the formula:

$$\text{Oxygen balance} = -1600 [2x + (y/2) - z] / \text{molecular weight};$$

The oxygen balance was developed for compounds containing only nitrate groups and it applies only to organic substances. Extending its use to molecules with other oxygen containing groups should be done with care. As an example the presence of hydroxyl-groups will strongly affect the oxygen balance towards higher values, whereas this group does not contribute to explosive properties. In addition the presence of for instance halogens tends to decrease the flammability and explosivity but this is not accounted for.

Please also check Appendix 6, Section 3 of the UN Recommendations on the Transport of Dangerous Goods, Manual of Tests and Criteria.

## ADAPTATION POSSIBILITIES ACCORDING TO ANNEX XI TO REACH

### USE OF EXISTING DATA: DATA ON PHYSICAL-CHEMICAL PROPERTIES FROM EXPERIMENTS NOT CARRIED OUT ACCORDING TO GLP OR THE TEST METHODS REFERRED TO IN ARTICLE 13 (3) OF REACH

If experimental data are available (study reports or literature data) meeting the criteria in section 1.1.1 of Annex XI to REACH, these could be used to meet the endpoint data requirements. If an estimation method is used as a source of information according to Column 2 of Annex VII, the QSAR model must meet the criteria set out in section 1.3 of Annex XI to REACH.

## 1 **WEIGHT OF EVIDENCE**

2 Where no single source of existing data (study reports, QSAR, literature data) is considered  
3 sufficiently reliable, thus not fully meeting the criteria in section 1.1.1 of Annex XI to REACH,  
4 or where several sources of similar reliability with deviating results exist, a weight of evidence  
5 approach may be used. The criteria in section 1.2 of Annex XI to REACH must then be met.  
6 Application of weight of evidence is possible with substances where explosive properties can  
7 clearly be excluded. Weight of evidence should be accompanied with extensive and reliable  
8 literature references.

## 9 **(Q)SAR**

10 There is currently no QSPR/(Q)SAR software known with sufficient accuracy and reliability to  
11 assist in assessing (potential) explosive properties. DSC testing is cheap and fast and is  
12 strongly recommended to identify potential hazards connected with the substance.

## 13 **GROUPING OF SUBSTANCES AND READ-ACROSS APPROACH**

14 An assessment of chemical structure would formally form part of a column 2 waiver. For  
15 further information please refer to the Guidance on the Application of the CLP Criteria, Part 2:  
16 Physical Hazards, Section 2.2 Explosives.

## 17 **TESTING IS TECHNICALLY NOT POSSIBLE**

18 Testing should always be considered if none of the waiving possibilities applies. Testing for  
19 explosives may be omitted if it is technically not possible to conduct the study as a  
20 consequence of the properties of the substance.

## 21 **FURTHER ADAPTATION POSSIBILITIES**

22 Testing may be waived if there are no chemical groups associated with explosive properties  
23 present in the molecule. The potential generation of explosive atmospheres by flammable  
24 gases/liquids or combustible solids is not considered an explosive property and should  
25 therefore not be reported under this heading.

26 As stated in Annex IX of REACH, when for certain endpoints, it is proposed to not provide  
27 information for other reasons than those mentioned in column 2 of that Annex or in Annex XI  
28 of REACH, this fact and the reasons must also be clearly stated. Such an approach may then  
29 be used.

## 30 **Impurities; uncertainties**

31 Small amounts of other compounds may enhance or suppress the chemical reaction that gives  
32 the explosive property to a substance. Therefore impurities may considerably influence the  
33 explosive properties of a substance. Therefore utmost care should be taken in the selection of  
34 the key study(s), or weight-of-evidence approaches, that the data selected is representative of  
35 the substance being registered by the respective companies.

## 36 **How to conclude on the DSD classification**

37 For DSD explosives are substances and preparations which may explode under the effect of  
38 flame or which are more sensitive to shocks or friction than dinitrobenzene.

Reclassification of substances classified as explosive according to DSD:

Under the regime of the old DSD, testing of explosive properties was achieved by performing test A.14. For classification purposes under the CLP Regulation this test is not adequate in the case of a negative result for thermal sensitivity. The method A.14 stops with a limiting diameter of 2 mm, while UN Test E.1 proceeds to down to a 1 mm orifice. Testing according to the CLP Regulation is the same as that described in Part I of the UN Recommendations on the Transport of Dangerous Goods, Manual of Tests and Criteria. This is why the translation table of Annex VII of the CLP Regulation states that there is no direct translation possible for classification from (E, R2) and (E, R3) to CLP criteria.

Therefore, if the screening procedure of section 2.1.4.2 of the CLP Regulation identifies a substance or mixture to be a potential explosive, appropriate data are required for classification.

Moreover, if data from performing test A.14 or the UN Test series 3 tests 3a or 3b indicate that a substance is sensitive to impact or friction such information should be provided in the REACH registration dossier.

## Endpoint specific information in the registration dossier/in IUCLID

### Material and methods

- reference to the standard and the test method applied;
- description of the substance that was tested.

### Results and discussion

- if testing is waived, the reasons for waiving must be documented in the dossier;
- if testing is not waived then the tests done according to the UN Test Manual and the outcome (explosive or not explosive) must be documented in the dossier. The mechanical sensitivity test according to UN Test Series 3a and 3b must be done and documented if UN Test Series 1 or 2 give a positive result. If data according to test A.14 are available, then the results can be used instead of UN Test series 3a and 3b.

An example is given below of how summarised results from the application of the class 1 procedure for the hypothetical substance "New explosive substance" could be presented.

**Figure R.7.1-4 Results from application of the class 1 acceptance procedure**

<b>1. Name of substance</b>	New explosive substance
<b>2. General data</b>	2.1 Composition : technically pure 2.2 Physical form : Fine crystalline powder 2.3 Colour : Yellow
<b>3. Box 2</b>	Is the substance manufactured with the view to producing a practical explosive or pyrotechnic effect? 3.1 Answer : <b>No</b>
<b>4. Box 3</b>	4.1 Propagation of Detonation : UN-Test A.1 Result : "-", no propagation of detonation 4.2 Effect of heating under confinement: 4.2.1 Koenen test (test 1(b)) Result : "+"

	<p>4.2.2 Time/pressure test (test 1(c)(i))</p> <p>Result : “-”, no effect on ignition under confinement</p> <p>4.5 Exit : Go to Box 4</p>
<b>5. Box 4</b>	<p>Is it an explosive substance?</p> <p>5.1 Answer from Test Series 1 : <b>Yes</b></p> <p>5.2 Exit : Go to box 5</p>
<b>6. Box 5</b>	<p>6.1 Sensitivity to shock : based on the test result of UN-Test A.1</p> <p>Result “-”</p> <p>6.2 Effect of heating under confinement:</p> <p>Koenen test (test 2(b)): limiting diameter 2,5 mm</p> <p>Result: “+”</p> <p>6.3 Exit : Go to Box 6</p>
<b>7. Box 6 :</b>	<p>Is the substance too insensitive for acceptance into Class 1?</p> <p>7.1 Answer from Test Series 2 : <b>No</b></p> <p>7.2 Conclusion : Substance to be considered for Class 1 (box 8)</p> <p>7.3 Exit : Go to Box 9</p>
<b>8. Box 9</b>	<p>Test Series 3</p> <p>8.1 Thermal Stability: based on the DSC measurement data</p> <p>Result: thermally stable</p> <p>8.2 Impact sensitivity : BAM fallhammer test (test 3(a)(ii))</p> <p>Result : “-”, not too dangerous to transport in form tested</p> <p>8.3 Friction sensitivity : BAM friction test (test 3(b)(i))</p> <p>Result : “-”, not too dangerous to transport in form tested</p> <p>8.4 Exit : Go to box 10</p>
<b>9. Box 10</b>	<p>Is the substance thermally stable?</p> <p>9.1 Answer from DSC data : <b>Yes</b></p> <p>9.2 Exit : Go to box 11</p>
<b>10. Box 11</b>	<p>Is the substance too dangerous for transport in the form in which it was tested?</p> <p>10.1 Answer from Test Series 3 (a)(ii) and 3 (b)(i): <b>No</b></p> <p>10.2 Exit : Go to box 18</p>
<b>11. Conclusion</b>	<p><b>PROVISIONALLY ACCEPT INTO CLASS 1</b></p> <p>11.1 Exit : Apply the Class 1 assignment procedure</p>

1 **Figure R.7.1-5 Results from the application of the class 1 assignment procedure**

<b>1. Box 19</b>	Is the substance a candidate for Division 1.5?  1.1 Answer : <b>No</b>  1.2 Exit : Go to box 25
<b>2. Box 25</b>	2.1 UN-Tests 6(a) and 6(c) were not conducted because the substance showed no propagation of detonation in the UN-Test A.1 and also no propagation of deflagration in the UN-test 1(c)(ii). 2.2 UN-Test 6 (c) Sample conditions: 1 × 30 kg fibre drum Observations: Only slow burning with black smoke and soot occurred.  2.3 Exit : Go to box 26
<b>3. Box 26</b>	Is the result a mass explosion?  3.1 Answer from Test Series 6 : <b>No</b>  3.2 Exit : Go to box 28
<b>4. Box 28</b>	Is the major hazard that from dangerous projections?  4.1 Answer from Test Series 6 : <b>No</b>  4.2 Exit : Go to box 30
<b>5. Box 30</b>	Is the major hazard radiant heat and/or violent burning but with no dangerous blast or projection hazard?  5.1 Answer from Test Series 6 : <b>No</b>  5.2 Exit : Go to box 32
<b>6. Box 32</b>	Is there nevertheless a small hazard in the event of ignition or initiation?  6.1 Answer from Test Series 6 : <b>No</b>  6.2 Exit : Go to box 35
<b>7. Box 35</b>	Is the substance or article manufactured with the view to producing a practical explosive or pyrotechnic effect?  7.1 Answer : <b>No</b>  7.2 Exit : Go to box 38
<b>8. Conclusion</b>	<b>NOT CLASS 1</b>  8.1 Exit : Consider for another class/division

2  
3 Any deviation from the guideline method used (and reasons for it) or any other special  
4 consideration should be reported. In cases where there is more than one source of data, the  
5 endpoint summary under results and discussion should provide a justification for the selection  
6 of the key study chapter.

7  
8 **Reference to other ECHA Guidance Documents**

9 Further detailed guidance on explosiveness can be found in the following chapters:

IUCLID Section	REACH Annex	Endpoint title	IUCLID 5 End User Manual Chapter	ECHA Practical Guide 3
4.14	VII 7.11	Explosiveness	E.4.15	3.12

1

## 2 Further information / references

3 Further information about classification and testing for explosives can be found in the *Guidance*  
4 *on the application of CLP criteria*, section 2.2.

5 Gharagheizi F. Quantitative structure-property relationship for prediction of the lower  
6 flammability limit of pure compounds. *Energy & Fuels* 22 (2008) 3037-3039.

7 Gharagheizi F. A new group contribution-based model for estimation of lower flammability limit  
8 of pure compounds. *J. Haz. Mat.* 170 (2009a) 595-604.

### 9 R.7.1.11.2 Self-reactive substances and mixtures

10 Self-reactive substances are primarily assigned to the headline 'Flammability' therefore please  
11 also refer to chapter R.7.1.10.4.

12 The sensitivity of self-reactive substances to impact (solids and liquids) and friction (solids  
13 only) may be of importance for the safe handling of the substances, in the event that these  
14 substances have pronounced explosive properties. If data according to EU test method A.14 as  
15 described in Regulation (EC) No 440/ 2008 are available, then this information should be part  
16 of the hazard communication in the registration dossier (REACH Annex VII, 7.11).

### 17 R.7.1.11.3 Organic peroxides

18 Organic peroxides are primarily assigned to the headline 'Flammability' therefore please also  
19 refer to chapter R.7.1.10.9.

20 The sensitivity of organic peroxides to impact (solids and liquids) and friction (solids only) may  
21 be of importance for the safe handling of the substances, in the event that these substances  
22 have pronounced explosive properties. If data according to EU test method A.14 as described  
23 in Regulation (EC) No 440/ 2008 are available, then this information should be part of the  
24 hazard communication in the registration dossier (REACH Annex VII, 7.11).

## 25 R.7.1.12 Self-ignition temperature

26 The terminology used in Annex VII of REACH is not very precise. Therefore, some guidance in  
27 interpretation appears necessary:

- 28
- 29 • For liquids and gases, the term '**auto-ignition**' instead of 'self-ignition' is generally used.  
30 Auto-ignitability is of high importance for the assignment of temperature classes in  
31 explosion protection (i. e. ATEX in Europe) of plants and equipment.
- 32
- 33 • For solids and liquids absorbed on a large surface, **self-heating** may occur by reaction  
34 with air with subsequent ignition. According to the CLP Regulation, Annex I, section 2.11, a  
35 self-heating substance or mixture is a liquid or solid substance or mixture, other than a  
36 pyrophoric liquid or solid, which, by reaction with air and without energy supply, is liable to  
37 self-heat; this substance or mixture differs from a pyrophoric liquid or solid in that it will  
38 ignite only when in large amounts (kilograms) and after long periods of time (hours or  
39 days). Therefore solids are considered under self heating substances in the chapter below.

**Table R.7.1-8 Assignment of CLP hazard classes to the information requirement 'Self ignition temperature' according to REACH, Annex VII and the Test Method Regulation.**

Information requirement according to Art. 10 (a) (vi) of the REACH Regulation (EC) No. 1907/2006 (the no. in brackets is the respective no. in the table in Annexes VII to XI to REACH)	CLP Regulation (EC) No. 1272/2008 (the no. in brackets is the respective chapter no. in Annex I to CLP)	Chapter in revised R.7(a) guidance	Corresponding test method according to The Test Method Regulation 440/2008	Corresponding test method according to CLP Regulation	Chapter in the Guidance on the Application of the CLP Criteria (ex RIP 3.6)
Self ignition temperature (7.12)	For gases and liquids*	7.1.12.1	A.15 Auto-ignition temperature (liquids and gases)	n.a.	n.a.
	For solids * Note: the UN Test N.4 is preferable to generate the information for this endpoint. Refer to R.7.1.10.7.	7.1.12.2, 7.1.10.7	A.16 Relative self-ignition temperature for solids	n.a.	Section 2.11

\* Note that regardless of whether the hazard class or category is listed in Article 14 (4) (a) of REACH, the chemical safety assessment (when required) must be performed in accordance with Article 14 (3) of REACH. Furthermore, according to Article 10 (a) (iv) of REACH the technical dossier of a registration for a substance under the REACH Regulation must include information on classification and labelling of the substance as specified in section 4 of Annex VI to the REACH Regulation.

#### R.7.1.12.1 Auto-ignition

##### Type of property

For liquids and gases, the term '**auto-ignition**' instead of 'self-ignition' is generally used. Auto-ignitability is of high importance for the assignment of temperature classes in explosion protection (i. e. ATEX in Europe) of plants and equipment. In this chapter, only the **auto-ignition** phenomena will be discussed.

##### Definition

The degree of auto-ignitability is expressed in terms of the auto-ignition temperature. The auto-ignition temperature is the lowest temperature at which the test substance will ignite when mixed with air under the conditions defined in the test method.



## 1 **Test method(s)**

2 For testing Auto-ignition temperature, method A.15 of Regulation (EC) 440/2008 should be  
3 used, which references several national and international standards (e.g. EN 14522, etc.). The  
4 test procedure is applicable to gases, liquids and vapours which, in the presence of air, can be  
5 ignited by a hot surface.

## 6 **Adaptation of the standard testing regime**

### 7 **ADAPTATION POSSIBILITIES ACCORDING TO COLUMN 2 OF ANNEX VII TO REACH**

8 Column 2 of REACH Annex VII provides the following specific rules for adaptation of the  
9 standard information requirement for self-ignition temperature.

10 *The study does not need to be conducted:*

- 11 • *if the substance is explosive or ignites spontaneously with air at room*  
12 *temperature; or*
- 13 • *for liquids non flammable in air, e.g. no flash point up to 200 °C; or*
- 14 • *for gases having no flammable range, or*
- 15 • *for solids, if the substance has a melting point  $\leq 160$  °C, or if preliminary results*  
16 *exclude self-heating of the substance up to 400 °C.*

17 This means:

#### 18 **For gases:**

19 Only gases classified as flammable according to the CLP Regulation have to be considered.

#### 20 **For liquids:**

21 The auto-ignition temperature should be determined according to Directive EC 440/2008,  
22 method A.15. No data are required for liquids classified as:

- 23 • pyrophoric; or
- 24 • explosive, unstable or division 1.1 to 1.6; or
- 25 • organic peroxide; or
- 26 • self-reactive substance.

27 Further, the auto-ignition temperature does not have to be determined for liquids having no  
28 flash point up to 200 °C. In practice, liquids with a boiling point above 350 °C will not have a  
29 flash point below 200 °C. Therefore, determination of the auto-ignition temperature is not  
30 necessary in such cases if the flash point is not known.

### 31 **ADAPTATION POSSIBILITIES ACCORDING TO ANNEX XI TO REACH**

#### 32 **USE OF EXISTING DATA: DATA ON PHYSICAL-CHEMICAL PROPERTIES FROM** 33 **EXPERIMENTS NOT CARRIED OUT ACCORDING TO GLP OR THE TEST METHODS** 34 **REFERRED TO IN ARTICLE 13 (3) OF REACH**

35 If experimental data are available (study reports or literature data) meeting the criteria in  
36 section 1.1.1 of Annex XI to REACH, these could be used to meet the endpoint data  
37 requirements. If an estimation method is used as a source of information according to Column  
38 2 of Annex VII, the QSAR model must meet the criteria set out in section 1.3 of Annex XI to  
39 REACH.

## **WEIGHT OF EVIDENCE**

For the determination of the auto-ignition temperature, the weight of evidence approach is not possible. Where no single source of existing data (study reports, QSAR, literature data) is considered sufficiently reliable, thus not fully meeting the criteria in section 1.1.1 of Annex XI to REACH, or where several sources of similar reliability with deviating results exist, a weight of evidence approach may be used. The criteria in section 1.2 of Annex XI to REACH must then be met.

## **(Q)SAR**

For the determination of the auto-ignition temperature, (Q)SAR approaches are strongly discouraged for the purpose of classification/ risk assessment.

## **GROUPING OF SUBSTANCES AND READ-ACROSS APPROACH**

For the determination of the auto-ignition temperature read-across is usually not possible. However interpolation may still be possible within homologous series.

However, it is not possible to read across from methyl compounds to ethyl and propyl compounds and vice versa.

## **TESTING IS TECHNICALLY NOT POSSIBLE**

Testing should always be considered if none of the waiving possibilities applies. Substances which decompose below room temperature or which react vigorously with moisture may be difficult to test. In such cases, the test may be waived due to technical reasons.

## **FURTHER ADAPTATION POSSIBILITIES**

Not foreseen.

## **Impurities; uncertainties**

The auto-ignition temperature can be considerably reduced by the presence of catalytic impurities. Therefore utmost care should be taken in the selection of the key study(s), or weight-of-evidence approaches, that the data selected is representative of the substance being registered by the respective companies.

## **Endpoint specific information in the registration dossier / in IUCLID**

### **Material and methods**

- description of the apparatus or reference to the standard or the test method applied;
- quantity of sample used.

### **Results and discussion**

- The value or the range of the auto-ignition temperature;
- if testing is waived, the reasons for waiving must be documented in the dossier.

For liquids/gases: observations (e.g decomposition with air, reactions with moisture, etc.)

For solids see the below chapter R.7.1.12.2.

Any deviation from the guideline method used (and reasons for it) or any other special consideration should be reported. In cases where there is more than one source of data, the endpoint summary under results and discussion should provide a justification for the selection of the key study chapter.

## Reference to other ECHA Guidance Documents

Further detailed guidance on auto flammability can be found in the following chapters:

IUCLID Section	REACH Annex	Endpoint title	IUCLID 5 End User Manual Chapter	ECHA Practical Guide 3
4.12	VII 7.12	Auto flammability	E.4.13	3.10

### R.7.1.12.2 Self-heating substances

For solids and liquids absorbed on a large surface, **self-heating** may occur by reaction with air with subsequent ignition. According to the CLP Regulation Annex I, section 2.11 the following definition is provided:

*A self-heating substance or mixture is a liquid or solid substance or mixture, other than a pyrophoric liquid or solid, which, by reaction with air and without energy supply, is liable to self-heat; this substance or mixture differs from a pyrophoric liquid or solid in that it will ignite only when in large amounts (kilograms) and after long periods of time (hours or days).*

The ECHA *Guidance on the Application of the CLP Criteria* gives in Chapter 2.11 detailed information on the self-heating property, the CLP-classification, the relevant test method and the relation to the DSD and the transport of dangerous goods regulations.

See section R.7.1.10.7 of this guidance document for further details and information.

### R.7.1.13 Oxidising properties

Some of the information requirements according to REACH Annex VII were phrased such that they correspond to 'indications of danger' as given in Annex II of DSD. For substances, classification and labelling according to the CLP Regulation has been mandatory since December 1<sup>st</sup>, 2010 (and will become mandatory for mixtures (preparations) from June 1 2015, when the DSD and DPD will be repealed). Consequently, information requirements on oxidising properties are inherently covered by classification of the substance according to the CLP Regulation. However, the physical hazards according to CLP Regulation are structured completely differently from the physicochemical properties according to DSD (and therefore also REACH, Annex VII). This means that for some of the CLP hazard classes an unambiguous assignment to one of the headlines (information requirements) in Annex VII to REACH is not possible. The assignment of hazard classes to the headline 'oxidising properties' as shown in table below must therefore only be understood as a means to structure this document in accordance with Annex VII to REACH.

**Table R.7.1-9 Assignment of CLP hazard classes to the information requirement 'Oxidising properties' according to REACH, Annex VII and correlation between the Test method Regulation and the test method according to CLP and supporting link with the Guidance on the application of the CLP Criteria.**

Information requirement according to Art. 10 (a)	CLP Regulation (EC) No. 1272/2008	Chapter in revised R.7(a) guidance	Corresponding test method according to The Test	Corresponding test method according to CLP Regulation	Chapter in the Guidance on the Application of the CLP Criteria
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(vi) of the REACH Regulation (EC) No. 1907/2006 (the no. in brackets is the respective no. in the table in Annexes VII to XI to REACH)	(the no. in brackets is the respective chapter no. in Annex I to CLP)		Method Regulation , Regulation (EC) 440/2008		(ex RIP 3.6)
Oxidising properties (7.13)	Oxidising gases (2.4) *	7.1.13.1	n.a.	<b>ISO 10156</b>	<b>2.5</b>
	Oxidising liquids (2.13) *	7.1.13.2	A.21 Oxidising properties (liquids)	<b>UN Test O.2</b>	<b>2.13</b>
	Oxidising solids (2.14) *	7.1.13.3	A.17 Oxidising properties (solids)	<b>UN Test O.1</b>	<b>2.13</b>

\* Note that regardless of whether the hazard class or category is listed in Article 14 (4)(a) of REACH the chemical safety assessment (when required) must be performed in accordance with Article 14 (3) REACH. Furthermore, according to Article 10(a)(iv) of REACH the technical dossier of a registration of a substance under the REACH Regulation must include information on classification and labelling of the substance as specified in section 4 of Annex VI to the REACH Regulation.

#### R.7.1.13.1 Oxidising gases

##### Definition

The following definition of oxidising gases is provided in CLP Annex I, section 2.4.1.:

*Oxidising gas means any gas or gas mixture which may, generally by providing oxygen, cause or contribute to the combustion of other material more than air does.*

The criteria 'more than air does' is further defined in a Note under Table 2.4.1 in Section 2.4.1 as 'having an oxidising power greater than 23.5 % as determined by a method specified in ISO 10156 as amended'.

##### Classification criteria and relevant information

All oxidising gases are classified as oxidising gas, Category 1 (Ox. Gas 1, H270). Detailed guidance on the classification criteria and the test method(s) can be found in the Guidance on the application of the CLP Criteria, section 2.5.

##### Adaptation of the standard testing regime

#### ADAPTATION POSSIBILITIES ACCORDING TO COLUMN 2 OF ANNEX VII TO REACH

Column 2 of REACH Annex VII provides the following specific rules for adaptation of the standard information requirement for oxidising properties.

*The study does not need to be conducted if:*

- the substance is explosive, or

- the substance is highly flammable, or
- the substance is an organic peroxide, or
- the substance is incapable of reacting exothermically with combustible materials, for example on the basis of the chemical structure (e.g. organic substances not containing oxygen or halogen atoms and these elements are not chemically bonded to nitrogen or oxygen, or inorganic substances not containing oxygen or halogen atoms).

The full test does not need to be conducted for solids if the preliminary test clearly indicates that the test substance has oxidising properties.

Note that as there is no test method to determine the oxidising properties of gaseous mixtures, the evaluation of these properties must be realised by an estimation method based on the comparison of the oxidising potential of gases in a mixture with that of the oxidising potential of oxygen in air.

According to above indents, the study therefore does not need to be conducted if the gas:

- is classified as highly flammable; or
- does not contain oxygen, fluorine and/or chlorine which are chemically bonded to elements other than carbon or hydrogen.

The other above cited indents are not relevant for this endpoint.

## ADAPTATION POSSIBILITIES ACCORDING TO ANNEX XI TO REACH

### USE OF EXISTING DATA: DATA ON PHYSICAL-CHEMICAL PROPERTIES FROM EXPERIMENTS NOT CARRIED OUT ACCORDING TO GLP OR THE TEST METHODS REFERRED TO IN ARTICLE 13 (3) OF REACH

There are not many gases that are oxidising. Most oxidising gases are identified as such in the UN-RTDG and in ISO 10156: 2010 *Gas cylinders - Gases and gas mixtures: - Determination of fire potential and oxidizing ability for the selection of cylinder valve outlets*.

If experimental data are available (study reports or literature data) meeting the criteria in section 1.1.1 of Annex XI to REACH, these could be used to meet the endpoint data requirements. If an estimation method is used as a source of information according to Column 2 of Annex VII, the QSAR model must meet the criteria set out in section 1.3 of Annex XI to REACH.

### WEIGHT OF EVIDENCE

There is no known scientific literature that refers to test results for gases that are not classified in ISO 10156 nor in the UN-RTDG.

Where no single source of existing data (study reports, QSAR, literature data) is considered sufficiently reliable, thus not fully meeting the criteria in section 1.1.1 of Annex XI to REACH, or where several sources of similar reliability with deviating results exist, a weight of evidence approach may be used. The criteria in section 1.2 of Annex XI to REACH must then be met.

## 1 (Q)SAR

2 At present (Q)SAR is generally not applicable for the determination of oxidising limits of gases.  
3 Application of (Q)SAR is not possible. However, assessment of the chemical structure may be  
4 used to exclude oxidising behaviour of a substance. Possibly, this relation could be exploited in  
5 the development of future QSPR methods.

## 6 GROUPING OF SUBSTANCES AND READ-ACROSS APPROACH

7 For the determination of the oxidising gases read-across is usually not possible. However  
8 interpolation may still be possible within homologous series.

## 9 TESTING IS TECHNICALLY NOT POSSIBLE

10 Testing should always be considered if none of the waiving possibilities applies.

## 11 FURTHER ADAPTATION POSSIBILITIES

12 Not foreseen.  
13

## 14 Impurities; uncertainties

15 The normal level of impurities in the technical grade of oxidising gases does not impact the  
16 result of the test. Tests should be performed with the lowest concentration of impurities in the  
17 gas encountered in the normal manufacturing process and the moisture content should be less  
18 than or equal to 0.01 mol%. Therefore utmost care should be taken in the selection of the key  
19 study(ies), or weight-of-evidence approaches, that the data selected is representative of the  
20 substance being registered by the respective companies.

## 21 How to conclude on the DSD classification

22 All gases with a positive test result according to the test method described in ISO 10156 are  
23 classified 'Oxidising O, R8'.

## 24 Endpoint specific information in the registration dossier/in IUCLID

### 25 Material and methods

- 26 • reference to the standard applied.

### 27 Results and discussion

- 28 • if the test is positive indicate that the gas is 'oxidising'.  
29 • if testing is waived, the reasons for waiving must be documented in the dossier.

30 Any deviation from the guideline method used (and reasons for it) or any other special  
31 consideration should be reported. In cases where there is more than one source of data, the  
32 endpoint summary under results and discussion should provide a justification for the selection  
33 of the key study chapter.  
34

## 35 Reference to other ECHA Guidance Documents

36 Further detailed guidance on oxidising properties can be found in the following chapters:

IUCLID Section	REACH Annex	Endpoint title	IUCLID 5 End User Manual Chapter	ECHA Practical Guide 3
4.15	VII 7.13	Oxidising properties	E.4.16	3.13

1

2 **Further information / references**

3 Further information about classification and testing for oxidising gases can be found in the  
4 ECHA *Guidance on the application of CLP criteria*, section 2.5.

5 The test method is described in ISO 10156. The test is qualitative. If reaction is observed  
6 during the test, the gas to be evaluated is oxidizing.

7 For several gases, a 'coefficient of oxygen equivalency' (Ci) has been deduced from the  
8 explosion ranges observed during the tests. The Ci factors are listed in ISO 10156 along with  
9 the list of oxidising gases.

## 10 R.7.1.13.2 Oxidising liquids

11 **Definition**

12 The following definition of oxidising liquids is provided in CLP Annex I, section 2.13.1.:

13 *Oxidising liquid means a liquid substance or mixture which, while in itself not necessarily*  
14 *combustible, may, generally by yielding oxygen, cause, or contribute to, the combustion of*  
15 *other material.*

16 **Classification criteria and relevant information**

17 According to the CLP Regulation, a liquid is classified as an oxidising liquid if, in testing  
18 according to the UN Test O.2 of the UN Manual of Tests and Criteria (Part III, Section 34) it is  
19 at least as oxidising as a 65 % aqueous solution of nitric acid. The CLP Regulation has three  
20 categories for Oxidising Liquids. The category is also determined through the UN Test O.2, by  
21 comparison to various reference oxidisers.

22 **Adaptation of the standard testing regime**23 **ADAPTATION POSSIBILITIES ACCORDING TO COLUMN 2 OF ANNEX VII TO REACH**

24 Column 2 of REACH Annex VII provides the following specific rules for adaptation of the  
25 standard information requirement for oxidising properties.

26 *The study does not need to be conducted if:*

- 27 • *the substance is explosive, or*
- 28 • *the substance is highly flammable, or*
- 29 • *the substance is an organic peroxide, or*
- 30 • *the substance is incapable of reacting exothermically with combustible materials, for*  
31 *example on the basis of the chemical structure (e.g. organic substances not containing*  
32 *oxygen or halogen atoms and these elements are not chemically bonded to nitrogen or*  
33 *oxygen, or inorganic substances not containing oxygen or halogen atoms).*

34 *The full test does not need to be conducted for solids if the preliminary test clearly indicates*  
35 *that the test substance has oxidising properties.*



*Note that as there is no test method to determine the oxidising properties of gaseous mixtures, the evaluation of these properties must be realised by an estimation method based on the comparison of the oxidising potential of gases in a mixture with that of the oxidising potential of oxygen in air.*

The first indent states that explosive substances should not be tested for oxidising properties. For instance, organic substances with oxidising functional groups may be explosive and should first undergo the screening procedures for explosive properties in Annex 6 of the UN Manual of Tests and Criteria to rule out possible explosive behaviour. Such substances may also be thermally unstable and show self-reactive behaviour. Substances that have been classified as Explosives according to the CLP Regulation or have been assigned risk phrases R2 or R3 according to the DSD, should normally not be tested for oxidising properties, since they are known to be explosive.

The second indent states that highly flammable substances do not have to be tested for oxidising properties. While it is not very clear what 'highly flammable' means in this case (whether it is or is not intended to mean 'extremely flammable' and 'flammable'), liquids that have a low flash point, or which are pyrophoric, are rarely oxidising. This implies that liquids classified as Flammable Liquids category 1 or 2, or as Pyrophoric Liquids, according to the CLP Regulation, normally do not need to be tested for oxidising properties. This corresponds to classification with risk phrases R12, R11 or R17 according to the DSD. If they contain oxidising functional groups, such substances may instead show self-reactive or explosive behaviour.

The third indent states that organic peroxides should not be tested for oxidising properties. Organic peroxides are distinguished by their chemical structure, and should be treated according to the procedures for the hazard class Organic Peroxides of the CLP Regulation, see Section R.7.1.10.9 of this document.

Waiving according to the fourth indent relies on the absence of particular molecular structural features. The wording is more precise in section 2.13.4 of Annex I to the CLP Regulation, which is in principle the same as the wording as in section 6 of Appendix 6 to the UN Manual of Tests and Criteria.

The last two paragraphs above quoted from Column 2 Specific rules for adaptation from Column 1 are not applicable for this endpoint.

According to 2.13.4.1 of Annex I to the CLP Regulation, an organic liquid does not have to be assessed for oxidising properties if:

- (i) *the substance does not contain oxygen, fluorine or chlorine; or*
- (ii) *the substance contains oxygen, fluorine or chlorine and these elements are chemically bonded only to carbon or hydrogen.*

*For inorganic liquids, assessment of oxidising properties does not have to be done if the substance does not contain any oxygen or halogen atoms, according to section 2.13.4.2 of Annex I to the CLP Regulation.*

## **ADAPTATION POSSIBILITIES ACCORDING TO ANNEX XI TO REACH**

### **USE OF EXISTING DATA: DATA ON PHYSICAL-CHEMICAL PROPERTIES FROM EXPERIMENTS NOT CARRIED OUT ACCORDING TO GLP OR THE TEST METHODS REFERRED TO IN ARTICLE 13 (3) OF REACH**

The UN Test O.2 of the UN Manual of Tests and Criteria is also used for classification according to the UN Model Regulations on the Transport of Dangerous Goods, and consequently also in the various regulations on transport of dangerous goods e.g. ADR and RID. A liquid that has

been classified as belonging to Division 5.1 according to the regulations on transport of dangerous goods on the basis of results from the UN Test O.2, is an Oxidising Liquid according to the criteria of the CLP Regulation.

If experimental data are available (study reports or literature data) meeting the criteria in section 1.1.1 of Annex XI to REACH, these could be used to meet the endpoint data requirements. If an estimation method is used as a source of information according to Column 2 of Annex VII, the QSAR model must meet the criteria set out in section 1.3 of Annex XI to REACH.

## WEIGHT OF EVIDENCE

For the determination of whether a liquid is an oxidising liquid, weight of evidence is not possible. Where no single source of existing data (study reports, QSAR, literature data) is considered sufficiently reliable, thus not fully meeting the criteria in section 1.1.1 of Annex XI to REACH, or where several sources of similar reliability with deviating results exist, a weight of evidence approach may be used. The criteria in section 1.2 of Annex XI to REACH must then be met.

## (Q)SAR

At the time of writing, no reliable (Q)SAR-methods exist for sufficiently accurate predictions of oxidising properties. As explained above, however, assessment of the chemical structure may be used to exclude oxidising behaviour of a substance. Possibly, this relation could be exploited in the development of future QSPR-methods. Such an assessment of chemical structure would formally form part of a Column 2 adaptation justification.

## GROUPING OF SUBSTANCES AND READ-ACROSS APPROACH

For the determination of the whether a liquid is an oxidising liquid, read-across is usually not possible. However interpolation may still be possible within homologous series.

## TESTING IS TECHNICALLY NOT POSSIBLE

Testing should always be considered, if none of the waiving possibilities applies. Some oxidising substances may decompose when heated. Substances may occasionally react with cellulose in other ways than through oxidation of the cellulose (e.g. through breaking chemical bonds within the cellulose). See also section 2.13.4.4 of Annex I to the CLP Regulation.

## FURTHER ADAPTATION POSSIBILITIES

Not foreseen.

## Impurities; uncertainties

Minor impurities will usually not influence the test, unless they are very strong oxidisers. Expert judgement should be used to determine whether impurities may have an effect. Therefore utmost care should be taken in the selection of the key study(s), or weight-of-evidence approaches, that the data selected is representative of the substance being registered by the respective companies.

A few substances may show other reactions than pure oxidation of the cellulose, or may decompose. If this is suspected, expert judgement should be sought. See also section 2.13.4.4 of Annex I to the CLP Regulation.

## How to conclude on the DSD classification

Any substance classified as an oxidising liquid according to the CLP-criteria should normally be classified with risk phrase R8 or R9 according to the DSD. The DSD-criteria for classification with risk phrase R9 are not very precise, but if the CLP classification is Category 1, the substance should be classified with risk phrase R9 if the reaction with cellulose is violent, e.g. if spontaneous ignition occurs in the test.

In the DSD, the A.21 test method of Regulation (EC) 440/2008 is used for the assessment of oxidising properties of liquids. This method is in principle identical to the UN Test O.2 of the UN Manual of Tests and Criteria used in the CLP Regulation. However, the DSD does not make any division corresponding to the categories of the CLP, and therefore only one reference substance is used in the A.21 test method. Since the CLP Regulation method is used for classification of substances, it is strongly advisable to use the UN Test O.2 instead of the A.21 test method. This is because the O.2 test method will also give more detailed information on the oxidising behaviour of a substance (or mixture), since more reference mixtures are used.

## Endpoint specific information in the registration dossier/in IUCLID

### Material and methods

- description of the apparatus and dimensions or reference to the standard or the test method applied;

### Results and discussion

- indicate the results of the spontaneous ignition test;
- indicate the mean pressure rise time for the test substance;
- indicate the mean pressure rise time for the reference substance(s);
- interpretation of results;
- if testing is waived, the reasons for waiving must be documented in the dossier.

Any deviation from the guideline method used (and reasons for it) or any other special consideration should be reported. In cases where there is more than one source of data, the endpoint summary under results and discussion should provide a justification for the selection of the key study chapter.

## Reference to other ECHA Guidance Documents

Further detailed guidance on oxidising properties can be found in the following chapters:

IUCLID Section	REACH Annex	Endpoint title	IUCLID 5 End User Manual Chapter	ECHA Practical Guide 3
4.15	VII 7.13	Oxidising properties	E.4.16	3.13

## Further information / references

The ECHA guidance document Guidance on the Application of the CLP Criteria gives in Chapter 2.13 detailed information on the oxidising property, the CLP-classification, the UN test O.2 and the relation to the DSD and the transport of dangerous goods regulations.

### R.7.1.13.3 Oxidising solids

#### Definition

The following definition of oxidising solids is provided in CLP Annex I, section 2.14.1:

*An oxidising solid means a solid substance or mixture which, while in itself not necessarily combustible, may, generally by yielding oxygen, cause, or contribute to, the combustion of other material.*

#### Classification criteria and relevant information

According to the CLP Regulation, a solid is classified as an oxidising solid if in testing according to the UN Test O.1 of the UN Manual of Tests and Criteria (Part III, Section 34), it is at least as oxidising as potassium bromate in a 3:7 mixture with cellulose. The test is based on the burning behaviour of a mixture of cellulose and the tested solid. The CLP Regulation has three categories for oxidising solids. The category is also determined through the UN Test O.1 in the UN Manual of Tests and Criteria, by comparison to reference mixtures of cellulose and potassium bromate<sup>13</sup>.

#### Adaptation of the standard testing regime

#### ADAPTATION POSSIBILITIES ACCORDING TO COLUMN 2 OF ANNEX VII TO REACH

Column 2 of REACH Annex VII provides the following specific rules for adaptation of the standard information requirement for oxidising properties.

*The study does not need to be conducted if:*

- the substance is explosive, or*
- the substance is highly flammable, or*
- the substance is an organic peroxide, or*
- the substance is incapable of reacting exothermically with combustible materials, for example on the basis of the chemical structure (e.g. organic substances not containing oxygen or halogen atoms and these elements are not chemically bonded to nitrogen or oxygen, or inorganic substances not containing oxygen or halogen atoms).*

*The full test does not need to be conducted for solids if the preliminary test clearly indicates that the test substance has oxidising properties.*

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<sup>13</sup> At the time of writing, work is in progress at the UN-level to modify Test O.1: Test for oxidising solids. This includes changing the reference substance and introducing a gravimetric method for the measurement. For further information, see document UN/SCEGHS/23/INF.17 available at the following link: <http://www.unece.org/fileadmin/DAM/trans/doc/2012/dgac10c4/UN-SCEGHS-23-INF17.doc-UN-SCETDG-41-INF.43e.pdf>.

*Note that as there is no test method to determine the oxidising properties of gaseous mixtures, the evaluation of these properties must be realised by an estimation method based on the comparison of the oxidising potential of gases in a mixture with that of the oxidising potential of oxygen in air.*

The first indent states that explosive substances should not be tested for oxidising properties. For instance, organic substances with oxidising functional groups may be explosive and should first undergo the screening procedures for explosive properties in Annex 6 of the UN Manual of Tests and Criteria to rule out possible explosive behaviour. Such substances may also be thermally unstable and show self-reactive behaviour. Substances that have been classified as Explosives according to the CLP-regulation or have been assigned risk phrases R2 or R3 according to the DSD, should normally not be tested for oxidising properties, since they are known to be explosive.

The second indent states that highly flammable substances do not have to be tested for oxidising properties. While it is not very clear what 'highly flammable' means in this case (whether it is or is not intended to mean 'extremely flammable' and 'flammable'), solids classified as Flammable Solids or as Pyrophoric Solids according to the CLP-regulation are rarely oxidising. This corresponds to classification with risk phrases R11 or R17 according to the DSD. If they contain oxidising functional groups, such substances may instead show self-reactive or explosive behaviour.

The third indent states that organic peroxides should not be tested for oxidising properties. Organic peroxides are distinguished by their chemical structure, and should be treated according to the procedures for the hazard class Organic Peroxides of the CLP-regulation, see Section R.7.1.10.9 of this document.

Waiving according to the fourth indent relies on the absence of particular molecular structural features. The wording is more precise in section 2.14.4 of Annex I to the CLP-regulation, which is in principle the same as the wording as in Section 6 of Appendix 6 to the UN Manual of Tests and Criteria.

The first note under last indent from REACH Annex VII, which allows waiving of further testing, namely "[...] *if the preliminary test clearly indicates that the test substance has oxidising properties*" is relevant only when using the A.17 test method of Regulation (EC) 440/2008, which is **not** the preferred test method since it belongs to the DSD classification system. The UN Test O.1 used for classification according to the CLP Regulation does not include any preliminary test.

The last note taken from Column 2 "Specific rules for adaptation from Column 1" is not applicable for this endpoint. For inorganic solids, assessment of oxidising properties does not have to be done if the substance does not contain any oxygen or halogen atoms, according to section 2.14.4.2 of Annex I to the CLP Regulation.

According to section 2.14.4.1 of Annex I to the CLP-regulation, an organic solid does not have to be assessed for oxidising properties if:

- the substance does not contain oxygen, fluorine or chlorine; or*
- the substance contains oxygen, fluorine or chlorine and these elements are chemically bonded only to carbon or hydrogen.*

## 1 ADAPTATION POSSIBILITIES ACCORDING TO ANNEX XI TO REACH

### 2 USE OF EXISTING DATA: DATA ON PHYSICAL-CHEMICAL PROPERTIES FROM 3 EXPERIMENTS NOT CARRIED OUT ACCORDING TO GLP OR THE TEST METHODS 4 REFERRED TO IN ARTICLE 13 (3) OF REACH

5 The UN Test O.1 of the UN Manual of Tests and Criteria is also used for classification according  
6 to the UN Model Regulations on the Transport of Dangerous Goods, and consequently also in  
7 the various regulations on transport of dangerous goods e.g. ADR and RID. A solid that has  
8 been classified as belonging to Division 5.1 according to the regulations on transport of  
9 dangerous goods on the basis of results from the UN Test O.1, is an oxidising solid according  
10 to the criteria of the CLP Regulation.

11 If experimental data are available (study reports or literature data) meeting the criteria in  
12 section 1.1.1 of Annex XI to REACH, these could be used to meet the endpoint data  
13 requirements. If an estimation method is used as a source of information according to Column  
14 2 of Annex VII, the QSAR model must meet the criteria set out in section 1.3 of Annex XI to  
15 REACH.

### 16 WEIGHT OF EVIDENCE

17 Where no single source of existing data (study reports, QSAR, literature data) is considered  
18 sufficiently reliable, thus not fully meeting the criteria in section 1.1.1 of Annex XI to REACH,  
19 or where several sources of similar reliability with deviating results exist, a weight of evidence  
20 approach may be used. The criteria in section 1.2 of Annex XI to REACH must then be met.

### 21 (Q)SAR

22 At the time of writing, no reliable (Q)SAR-methods exist for sufficiently accurate predictions of  
23 oxidising properties. As explained above, however, assessment of the chemical structure may  
24 be used to exclude oxidising behaviour of a substance. Possibly, this relation could be exploited  
25 in the development of future (Q)SPR-methods. Such an assessment of chemical structure  
26 would formally form part of a Column 2 adaptation argument.

### 27 GROUPING OF SUBSTANCES AND READ-ACROSS APPROACH

28 For the determination of the oxidising solids read-across is usually not possible. However  
29 interpolation may still be possible within homologous series.

### 30 TESTING IS TECHNICALLY NOT POSSIBLE

31 Testing should always be considered if none of the waiving possibilities applies. Some  
32 substances may decompose upon heating. Substances may occasionally react with cellulose in  
33 other ways than through oxidation of the cellulose.

### 34 FURTHER ADAPTATION POSSIBILITIES

35 Not foreseen.

## **Impurities; uncertainties**

The UN Test O.1 is (currently) performed using the unaided eye as measuring instrument. Only by expert judgement and thorough experience can the result of the test be correctly judged, and even then uncertainties may arise.

Minor impurities will usually not influence the test, unless they are very strong oxidisers. Expert judgement should be used to determine whether impurities may have an effect.

A few substances may show other reactions than pure oxidation of the cellulose, or may decompose. If this is suspected, expert judgement should be sought. Particle size and size distribution can have an influence on the test results.

## **How to conclude on the DSD classification**

Any substance classified as an oxidising solid according to the CLP Regulation criteria should normally be classified with risk phrase R8 or R9 according to the DSD. The DSD-criteria for classification with risk phrase R9 are not very precise, but if the CLP Regulation classification is Category 1, the substance should be classified with risk phrase R9 if the reaction with cellulose is violent.

In the DSD, the A.17 test method of Regulation (EC) 440/2008 is used for the assessment of oxidising properties of solids. Although the principle of this method is to a large extent the same as that of the UN Test O.1 of the UN Manual of Tests and Criteria, the experimental set-up, reference substance (barium nitrate) and measured quantity differ. Furthermore, the DSD does not make any division corresponding to the categories of the CLP. Since the CLP Regulation is used for classification of substances, it is not advisable to use the A.17 method (which belongs to the DSD classification system). Instead, the UN Test O.1 should be used, which will also give more detailed information on the oxidising behaviour of a substance (or mixture), since more reference mixtures are used.

## **Endpoints specific information in the registration dossier/in IUCLID**

### **Material and methods**

- description of the apparatus and dimensions or reference to the standard or the test method applied;
- particle size and distribution.

### **Results and discussion**

- if testing is waived, the reasons for waiving must be documented in the dossier;

#### **If the O.1 test method was used:**

- indicate if a vigorous reaction was observed;
- indicate the maximum burning time for the test mixture;
- indicate the maximum burning time for the reference mixtures;
- interpretation of results, including any relevant special observations;
- estimated accuracy of the result (including bias and precision).

#### **If A.17 test method was used:**

- indicate if in the preliminary test, a vigorous reaction was observed;
- indicate the maximum burning rate for the test mixture;
- indicate the maximum burning rate for the reference mixture;
- interpretation of results, including any relevant special observations;
- estimated accuracy of the result (including bias and precision).



Any deviation from the guideline method used (and reasons for it) or any other special consideration should be reported. In cases where there is more than one source of data, the endpoint summary under results and discussion should provide a justification for the selection of the key study chapter.

## Reference to other ECHA Guidance Documents

Further detailed guidance on oxidising properties can be found in the following chapters:

IUCLID Section	REACH Annex	Endpoint title	IUCLID 5 End User Manual Chapter	ECHA Practical Guide 3
4.15	VII 7.13	Oxidising properties	E.4.16	3.13

## Further information / references

The ECHA Guidance on the Application of the CLP Criteria gives in Chapter 2.13 detailed information on the oxidising property, the CLP-classification, the UN Test O.1 and the relation to the DSD and the transport of dangerous goods regulations.

## R.7.1.14 Granulometry

Advice to registrants with regard to nanomaterials characterisation of granulometry can be found in Appendix R7-1 Recommendations for nanomaterials applicable to: Chapter R7a Endpoint specific guidance, section 2.2.3 Granulometry.

### R.7.1.14.1 Type of property

Granulometry is not a specific physico-chemical property of a substance. The original particle size distribution is highly dependent on the industrial processing methods used and can also be affected by subsequent environmental or human transformations. Particle size is usually measured in micrometers ( $= 10^{-6}$  m;  $\mu\text{m}$ ; "microns").

Granulometry is of considerable importance for the toxic properties of a substance as it influences aspects such as:

- the route of exposure of humans and toxicity by inhalation;
- the choice of route of administration for animal testing;
- the efficiency of uptake in an organism;
- the distribution in the environment.

Granulometry is of importance for combustible dusts as it influences aspects such as the likelihood to form combustible/explosive dust - air mixtures.

In general all powder materials have a range of particle sizes (particle size distribution), a presentation of the particle size distribution (e.g. using a histogram of the particle size vs. mass, particle size vs. number of particles, etc.) is therefore necessary to interpret the data.

For inhalation exposure it is well known that the human toxicity will be related with the place of deposition into the respiratory tract. The location of deposition mainly depends on the properties of the particle (size, shape, density etc) that are commonly taken into account considering the aerodynamic diameter of the particle (see definition below). Thus, the general approach has been to use mass fractions (e.g. health related fractions as defined by EN 481 or the EPA PM Fractions). For instance, in Europe, from the publication of the EN 481 the OELs for powder materials have been defined for one or several fractions (inhalable, thoracic or respirable).

**Photocentrifuge method** - the method of determining the particle size distribution, which is described in ISO 13318-2:2007, is applicable to powders that can be dispersed in liquids, powders that are present in slurry form and some emulsions. Typical particle size range for analysis is from about 0.1  $\mu\text{m}$  to 5  $\mu\text{m}$ . The method is applicable to powders in which all particles have the same density and comparable shapes and do not undergo chemical or physical change in the suspension liquid. It is usually necessary that the particles have a density higher than that of the liquid.

**Light extinction liquid-borne particle counter** - in ISO 21501-3:2007 a calibration and verification method for a light extinction liquid-borne particle counter (LSLPC) is described, which is used to measure the size and particle number concentration of particles suspended in liquid. The light extinction method is based on single particle measurements and the typical size range of particles measured by this method is between 1  $\mu\text{m}$  and 100  $\mu\text{m}$ .

**Light scattering liquid-borne particle counter** - in ISO 21501-2:2007 a calibration and verification method for a light scattering liquid-borne particle counter (LSLPC) is described, which is used to measure the size and particle number concentration of particles suspended in liquid. The light scattering method is based on single particle measurements and the typical size range of particles measured by this method is between 0.1  $\mu\text{m}$  and 10  $\mu\text{m}$ .

**Centrifugal X-ray method** - the method of determining the particles size distribution described in ISO 13318-3:2004 is applicable to powders which can be dispersed in liquids or powders which are present in slurry form. The typical particle size range for analysis is from 0.1 µm to 5 µm. The method is applicable to powders in which all particles have the same effective density, chemical composition and comparable shapes.

The CEN document, EN 481 "Workplace Atmospheres – size fraction definitions for measurement of airborne particles" (CEN 1993) provides definitions of the inhalable, thoracic and respirable size fractions, and target specifications (conventions) for sampling instruments to measure these fractions. The current standard defines sampling conventions for particle size fractions which are to be used in assessing the possible health effects resulting from inhalation of airborne particles in the workplace. The different particle sizes defined in EN 481 are:

- inhalable fraction (the mass fraction of particles that can be inhaled by nose and mouth. Particles >100 µm are not included in the inhalable convention;
- thoracic fraction (the mass fraction of the inhaled particles that passes the larynx). The convention for thoracic fraction sets that 50% of the particles in air with an aerodynamic diameter of 10 µm belong to the thoracic fraction;
- respirable fraction (the mass fraction of the inhaled particles that reaches the alveoli) The convention for respirable fraction sets that 50% of particles with an aerodynamic diameter of 4 µm belong to the respirable fraction.

#### R.7.1.14.2 Definitions

**Aerodynamic diameter:** the diameter of a sphere of density 1 g cm<sup>-3</sup> with the same terminal velocity (falling speed) due to gravitational force in calm air as the particle under the prevailing conditions of temperature, pressure and relative humidity (CEN, 1993). The aerodynamic diameter is used to compare particles of different sizes, shapes and densities and it is a useful parameter to predict where in the respiratory tract such particles may be deposited. It is used in contrast to "optical", "measured" or "geometric" diameters which are representations of actual diameters which in themselves cannot be related with the deposition within the respiratory tract.

**Particle diffusion diameter:** for particles of aerodynamic diameter less than 0.5 µm, the particle diffusion diameter should be used instead of the particle aerodynamic diameter. For diffusion, the appropriate *equivalent diameter* is the diffusion (mobility) diameter. This is defined as the diameter of a sphere with the same diffusion coefficient as the particle under the prevailing conditions of temperature, pressure and relative humidity.

The parameter of interest is the effective hydrodynamic radius, or effective Stoke's radius  $R_s$ . Particle size distribution (effective hydrodynamic radius) requires information on water insolubility. Fibre length and diameter distributions require information on the fibrous nature of the product and on stability of the fibrous shape under electron microscope conditions.

**A fibre:** is a water insoluble particle with an aspect ratio (length/diameter > 3) and diameter < 100 µm. Fibres of length < 5 µm need not be considered.

**Particle:** Minute piece of matter with defined physical boundaries. (ISO/TS 27687:2008)

**Agglomerate:** A collection of weakly bound particles of aggregates or mixtures of the two where the resulting external surface area is similar to the sum of the surface areas of the individual components (ISO/TS 27687:2008).

**Aggregate:** Particle comprising strongly bonded or fused particles where the resulting external surface area may be significantly smaller than the sum of calculated surface areas of the individual components (ISO/TS 27687:2008).

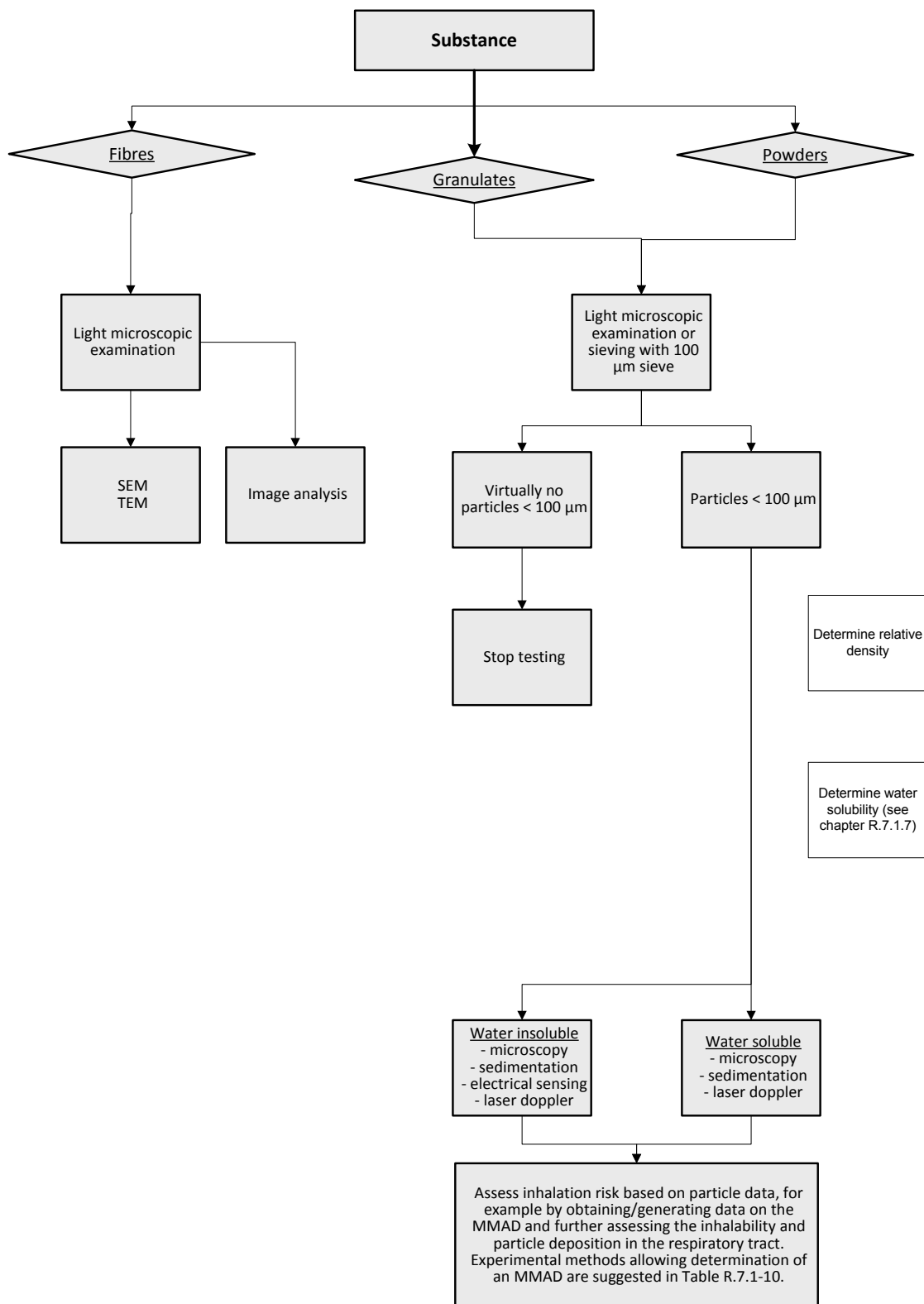
1 R.7.1.14.3 Test methods

2 Many methods are available for particle size measurements, but none of them is applicable to  
3 the entire size range (see [Table R.7.1-10](#)). Sieving, microscopic sedimentation and elutriation  
4 techniques are most commonly employed. Methods for determining particle size distribution  
5 are designed to provide information on the transportation and sedimentation of insoluble  
6 particles in water and air. An integrated testing strategy (ITS) detailing the appropriate  
7 methods for determination of particle size distribution of respirable and inhalable particles is  
8 shown in [Figure R.7.1-6](#).

9 Details of methods for determining particle size distribution and for fibre length and diameter  
10 distributions are outlined in OECD TG 110 and in the "Guidance Document on the  
11 Determination of Particle Size Distribution, Fibre Length and Diameter Distribution of Chemical  
12 Substances" (JRC, 2002).

13 The particle size distribution is carried out on the material under investigation and not as  
14 airborne dust.

15 The measurement principle of the method used will determine what kind of diameter of the  
16 particle can be determined: for instance, optical diameter when using light scattering or  
17 aerodynamic diameter when using impactors. Methods which determine the mass median  
18 aerodynamic diameter (MMAD) need the generation of representative test atmospheres using  
19 suitable generation equipment and correct sampling techniques. They can be used in case of  
20 airborne particles (dusts, smokes, fumes), nebulised particles (wet aerosol) or dispersed  
21 particles (dry aerosol).

1 **Figure R.7.1-6 Integrated testing strategy for granulometry**

1 **Table R.7.1-10 Methods to determine particle size distribution of a material**

Method and details	Material and size range	MMAD
<p>Microscopic examination</p> <p>It is preferable to prepare samples directly in order not to influence shape and size of the particles.</p> <p>This method determines size distribution of particles.</p>	<p>Particles of all kinds</p> <p>Size range: 0.5–5000 microns (light microscope) and &lt;0.1–10 microns (SEM/TEM)</p>	MMAD cannot be determined
<p>Sieving</p> <p>Sieving using wire-mesh sieves and perforated sheet metal sieves is not suitable to determine the distribution of particles of respirable and inhalable size since their range is only 100–10,000 microns. Micro mesh sieves (range 5–100 micron) may give better results. However, since these sieves are generally operated in combination with mechanical or ultrasonic vibration, modification of median size and form may result.</p> <p>Sieving not suitable to determine distribution of particles of respirable size, but might be suitable to determine bigger particles.</p>	<p>Dry powders/granulates</p> <p>Size range: 100–10,000 microns (wire mesh/metal sieves) and 5–100 (micromesh)</p>	MMAD cannot be determined
<p>Sedimentation (gravitational settling)</p> <p>Method is based on gravitational settling of particles in liquid and the effective hydrodynamic radius is determined. Effective hydrodynamic radius distribution should be measured 3x with no two values differing by &gt;20%. Requires sufficient numbers of radius intervals be used to resolve the radius distribution curve. Binary or ternary mixtures of latex spheres (2–100 microns) are recommended as calibration material.</p> <p>Method might be suitable to determine the distribution of particles of respirable and inhalable size.</p>	<p>Dry powders/granulates</p> <p>Size range: 2–200 microns</p>	MMAD cannot be determined
<p>Electrical Sensing Zone (e.g. Coulter) method</p> <p>Samples are suspended in an electrolytic solution. As the particle is drawn through an aperture, the change in conductance gives a measure of particle size. The important parameter is the settling velocity in the liquid phase, which depends on both density and diameter. Particles having a density of several g/cm<sup>3</sup> can be determined.</p> <p>Applicable to particles that are complete electrical isolators in the fluid. Difference in density between particles and fluid must not be too large.</p> <p>Method might be suitable to determine the distribution of particles of respirable and inhalable size.</p>	<p>Dry powders/granulates (non-conducting)</p> <p>Size range: 1–1000 microns</p>	MMAD cannot be determined
<p>Phase Doppler Anemometry</p> <p>Expensive technique. Particle size distribution can be measured either in air or in liquid. The method presupposes that the particles are spherical with known refractive index.</p> <p>Method might be suitable to determine the distribution of particles of respirable and inhalable</p>	<p>Dry powders/granulates</p> <p>Size range: 0.5–80 microns (in air); 0.5–1000 microns (in liquid)</p>	MMAD cannot be determined

size.		
<p>Determination of fibre length and diameter distributions</p> <p>Light microscopy used to examine likelihood of fibres present by comparing similarities to known fibrous or fibre releasing substances or other data. Extreme care required during sample preparation to avoid fibre breaking and clumping. Care should also be taken to avoid contamination by airborne fibres. Samples might be prepared by (a) producing suspensions in water by gentle hand agitation or vortex mixing or (b) transfer of dry material onto copper tape either directly or by spraying of the dry fibres by use of atomiser or pipette.</p> <p>Length and diameter distributions should be measured independently at least twice and at least 70 fibres counted. No two values in a given histogram interval should differ by &gt; 50% or 3 fibres, whichever is larger. The presence of long thin fibres would indicate a need for further, more precise measurements.</p>	<p>Fibrous products</p> <p>Size range: diameters as small as 0.1 micron and as large as 100 micron and lengths as small as 5 micron and as large as 300 micron</p>	

1

2 It is advantageous to have accurate information about the propensity of materials to produce  
3 airborne dust (the *dustiness* of the material). No single method of dustiness testing is likely to  
4 represent and reproduce the various types of processing and handling used in industry. The  
5 measurement of dustiness depends on the test apparatus used, the properties of the dust and  
6 various environmental variables (i.e the dustiness is not a measurement of the "dust as it is") .  
7 There are a number of methods for measuring the dustiness of bulk materials, based on the  
8 health related aerosol fractions defined in EN 481. Two methods (the rotating drum method  
9 and the continuous drop method) are detailed in EN 15051 "Workplace atmospheres –  
10 Measurement of the dustiness of bulk materials – Requirements and reference test methods"  
11 (CEN, 2006).

12 Dustiness is a relative term (derived from the amount of dust emitted during a standard test  
13 procedure). This is dependent on the method chosen, the condition and properties of the  
14 tested bulk material, and various environmental variables in which the tests are carried out.  
15 Thus, the two methods in EN 15051 may provide different results (the methods are intended  
16 to simulate handling processes) The standard is currently under revision (draft of European  
17 standard available) and the final publication is expected for 2013. The standard has been  
18 divided in 3 parts (a general part and one part for each of the methods). The methods ([Table](#)  
19 [R.7.1-11](#)) as described in the standard are used to determine dustiness in terms of the health  
20 related fractions defined by EN 481. Further analyse (e.g. analysing the contents on the dust  
21 collection stages ) can be used to obtain the particle sizedistribution. These methods require  
22 the generation of representative test atmospheres using suitable generation equipment and  
23 correct sampling techniques.  
24



1 **Table R.7.1-11 Methods to generate/sample airborne dispersed or nebulised particles**

Method and details	Material and size range	MMAD
<p><b>Cascade impaction</b></p> <p>Cascade impactors can be used to obtain the size distribution of an aerosol (i.e in this context a dust cloud). Air samples are drawn through a device which consists of several stages on which particles are deposited on an impactation substrate. Particles will impact on a certain stage depending on their aerodynamic diameter . The cut- off size can be calculated from the jet velocities at each stage by weighing each stage before and after sampling and the MMAD derived from these calculations.</p> <p>This is a well established technique to measure the size distribution of particles (allowing calculating any mass fraction). Some models are specifically designed to give the 3 health related fractions defined by the EN 481.</p> <p>Please also check ISO/TR 27628:2007, which contains specific information on methods for bulk aerosol characterization and single particle analysis while using cascade impaction method.</p>	<p>Particles in an aerosol</p> <p>Size range: 0.1-20 and 0.5-80 microns</p>	<p>MMAD can be determined via an appropriate coupled analytical technique.</p>
<p><b>Laser scattering/diffraction</b></p> <p>In general, the scattering of the incident light gives distinct pattern which are measured by a detector. This technique is particle property dependent – i.e. material has unique scattering and diffraction properties which are also particle size dependent. It is important to calibrate the instrument with similar material (of the same size range as the material to be measured). Laser scattering techniques are suitable for geometric particles, viz spheres, cubes and monocrystals. Particle size will be established optically. The MMAD can be calculated by means of a calculation correction.</p> <p>Further information about corrections and limitations of the methods can be found in CEN/TR 16013-1 and CEN/TR 16013-2.</p> <p>Please also check ISO 13320:2009 Particle size analysis – Laser diffraction methods taking into account the possible limitations of the method, a the technique assumes a spherical particle shape in its optical model. The resulting particle size distribution is different from that obtained by methods based on other physical principles (e.g. sedimentation, sieving).</p>	<p>Particles of all kind</p> <p>Size range: 0.1 um to 3 mm (with special instrumentation and conditions, the size range can be extended above 3 mm and below 0.1 mm)</p>	<p>MMAD can be determined.</p>
<p><b>Rotating drum method (prEN 15051-2)</b></p> <p>This method is based on size selective sampling of an airborne dust cloud produced by the repeated lifting and dropping of a material in a rotating drum. Air drawn through the drum passes through a specially designed outlet and a 3-stage fractionating system consisting of two porous polyurethane foams and a membrane filter. The mass of dust collected on each</p>	<p>Dry powders/granulates/friable products</p> <p>Size range: 0.5-10,000 microns</p>	<p>MMAD cannot be determined.</p>

<p>collection stage is determined gravimetrically to give a direct measure of the biologically relevant size fractions. This method simulates a wide range of material handling processes in industry and determines the biologically relevant size functions of a material in the airborne state.</p> <p>This method is suitable to determine the respirable thoracic or inhalable fractions.</p>		
<p>Continuous drop method (prEN 15051-3)</p> <p>This method is based on the size selective sampling of an airborne dust cloud produced by the continuous single dropping of material in a slow vertical air current. The dust released by dropping material is conducted by the airflow to a sampling section where it is separated into the inhalable and respirable fractions.</p> <p>This method is suitable to determine the respirable and inhalable fractions.</p>	<p>Dry powders/granulates/friable products</p> <p>Size range: 0.5-10,000 microns</p>	<p>MMAD can be determined.</p>

**Table R.7.1-12 Methods that measure inhalable fractions only or that give no detailed distributions**

Method and details	Material and size range	MMAD
<p>Elutriation</p> <p>Particles are drawn out on a column at varying velocity. The velocity is used to calculate particle size and the weight of the remaining sample at a particular velocity is used to calculate the distribution. The method is limited to particles &gt;15 microns.</p> <p>The method is not suitable to determine the distribution of particles of respirable size, but might be suitable to determine the distribution of particles of inhalable size</p>	<p>Dry powders/granulates</p> <p>Size range: 15-115 microns</p>	<p>MMAD cannot be determined.</p>
<p>Air jet sieve</p> <p>Air is aspirated through a weighted sample on a fine sieve and the weight loss measured. The method is capable of estimating the non-floatable fraction of the material under investigation. Aggregation of the particles will result in unreliable values. In addition, since the lower detection limit is only 10 micron, this method is not suitable to determine the distribution of particles of respirable size.</p> <p>The method is not suitable to determine the distribution of particles of the respirable fraction, but might be suitable to determine the distribution of particles between 10 and 10,000 microns..</p>	<p>Particles of all kind</p> <p>Size range: 10-10,000 microns</p>	<p>MMAD cannot be determined.</p>
<p>Cyclons</p> <p>The use of a cyclone is a simple approach to determining whether respirable and/or inhalable particles are present in the test atmospheres by constructing the cyclone cut off points at 4.25 and 100 microns. By measuring the weight of particles which pass through the cyclone it can be decided whether more sophisticated methods have to be applied to determine the size distribution of the particles smaller than 10 micron.</p> <p>This method is suitable to determine the respirable, thoracic or inhalable fraction.</p>	<p>Particles of all kind</p> <p>Size range: 0.1-200 microns</p>	<p>MMAD cannot be determined.</p>

#### R.7.1.14.4 Adaptation of the standard testing regime

#### **Adaptation possibilities according to column 2 of Annex VII to REACH**

Column 2 of REACH Annex VII provides the following specific rules for adaptation of the standard information requirement for granulometry.

*The study does not need to be conducted if the substance is marketed or used in a non solid or granular form.*

## 1 **Adaptation possibilities according to Annex XI to REACH**

### 2 **USE OF EXISTING DATA: DATA ON PHYSICAL-CHEMICAL PROPERTIES FROM** 3 **EXPERIMENTS NOT CARRIED OUT ACCORDING TO GLP OR THE TEST METHODS** 4 **REFERRED TO IN ARTICLE 13 (3) OF REACH**

5 As the granulometry of a substance is highly dependent on the industrial processing methods  
6 and possibly also on handling of the material, any published data on granulometry will be  
7 pertinent only to the particular sample or process.

8 There are a number of web sites and electronic databases that include compilations of and  
9 evaluations of data on particle properties. However, there appear to be a limited number of  
10 reference books that provide particle size data.

11 The equivalence of the various national and international standard methods for particle size  
12 distribution has not been tested and is not known.

13 If experimental data are available (study reports or literature data) meeting the criteria in  
14 section 1.1.1 of Annex XI to REACH, these could be used to meet the endpoint data  
15 requirements. If an estimation method is used as a source of information according to Column  
16 2 of Annex VII, the QSAR model must meet the criteria set out in section 1.3 of Annex XI to  
17 REACH.

### 18 **WEIGHT OF EVIDENCE**

19 In some situations where data is available from multiple sources (e.g. information on particle  
20 size distribution of different batches, or information from different methods), a weight of  
21 evidence approach may be used. Where no single source of existing data (study reports,  
22 QSAR, literature data) is considered sufficiently reliable, thus not fully meeting the criteria in  
23 section 1.1.1 of Annex XI to REACH, or where several sources of similar reliability with  
24 deviating results exist, a weight of evidence approach may be used. The criteria in section 1.2  
25 of Annex XI to REACH must then be met.

### 26 **(Q)SAR**

27 There are no QSPR/(Q)SAR tools available for predicting particle size and the data will  
28 therefore need to be experimentally determined. Application of (Q)SAR is not possible.

### 29 **GROUPING OF SUBSTANCES AND READ-ACROSS APPROACH**

30 At present grouping and read across are not applicable.

### 31 **TESTING IS TECHNICALLY NOT POSSIBLE**

32 Testing should always be considered, if none of the waiving possibilities applies. Testing should  
33 always be possible for solids or granular substances.

### 34 **Further adaptation possibilities**

35 Not foreseen.

36 R.7.1.14.5 Impurities; uncertainties

There is a particular problem in relation to sedimentation and Coulter counter measurements. The effect of impurities on particle shape should be considered when measuring fibre length and diameter distributions.

The small quantities used as samples must be representative of product batches comprising many kilograms; therefore sampling and sample handling require great care.

Great care should also be taken due to the fact that non-conducting particles in a non-conducting liquid may be electrically charged resulting in non-representative settling of particles of a certain size. In addition, in the process of particle size distribution determination, it is very important to take the electrostatic charge of the particles into account. Electrostatically charged particles behave differently and may influence sampling.

It is useful to distinguish between aggregates and agglomerates. While an aggregate is held together by strong forces and may be considered to be permanent, agglomerates are held together with weak forces and may break up under certain circumstances. As small particles often form agglomerates, sample pre-treatment (e.g. the addition of dispersing agents, agitation or low-level ultrasonic treatment) may be required before the primary particle size can be determined. However, great care must be taken to avoid changing the particle size distribution.

#### R.7.1.14.6 Endpoint specific information in the registration dossier / in IUCLID

##### **Material and methods**

- sample preparation, such as any sonication, grinding, or addition of dispersion agents (if any);
- if a suspending medium is used (e.g. sedimentation test): indicate type of medium, temperature pH, concentration and solubility of the substance in the suspending medium;
- The type of method used.

##### **Results and discussion**

- in the particle size field: mean and standard deviation;
- in the particle size distribution at different passages field: size and distribution;
- Approximate information on particle shape (e.g. spherical, platelike, needle shaped) if available;
- for fibres: indicate both length and diameter of fibres.

Any deviation from the guideline method used or any other special consideration should be reported. In cases where there is more than one source of data, the endpoint summary under results and discussion should provide a justification for the selection of the key study chapter.

##### **Reference to other ECHA Guidance Documents**

Further detailed guidance on particle size distribution (Granulometry) can be found in the following chapters:

IUCLID Section	REACH Annex	Endpoint title	IUCLID 5 End User Manual Chapter	ECHA Practical Guide 3
4.5	VII 7.14	Particle size distribution (Granulometry)	E.4.6	3.4

## 1 R.7.1.14.7 Further information / references

- CEN 1993 EN 481: Workplace atmospheres. Size fraction definitions for measurement of airborne particles
- CEN 2006 EN 15051: Workplace atmospheres. Measurement of the dustiness of bulk materials – Requirements and reference test methods
- JRC (2002) "Guidance Document on the Determination of Particle Size Distribution, Fibre Length and Diameter Distribution of Chemical Substances", ISBN 92-894-3704-9, EUR 20268 EN, <http://publications.jrc.ec.europa.eu/repository/handle/111111111/5555>
- OECD TG 110 Test No. 110: Particle size distribution/fibre length and diameter distributions
- prEN 15051-1 rev Workplace exposure - Measurement of dustiness of bulk materials - Part 1: Requirements and choice of test methods
- prEN 15051-2 Workplace exposure - Measurement of the dustiness of bulk materials - Part 2: Rotating drum method
- prEN 15051-3 Workplace exposure - Measurement of the dustiness of bulk materials - Part 3: Continuous drop method
- (ISO/TS 27687:2008) Nanotechnologies-Terminology and definitions for nano-objects- Nanoparticle, nanofibre, and nanoplate
- CEN/TR 16013-1:2010 Workplace exposure. Guide for the use of direct-reading instruments for aerosol monitoring. Choice of monitor for specific applications
- CEN/TR 16013-2:2010 Workplace exposure. Guide for the use of direct-reading instruments for aerosol monitoring. Evaluation of airborne particle concentrations using optical particle counters

## 1 **R.7.1.15 Adsorption/Desorption**

2 Advice to registrants with regard to nanomaterials characterisation of adsorption/desorption  
3 can be found in Appendix R7-1 Recommendations for nanomaterials applicable to: Chapter R7a  
4 Endpoint specific guidance, section 2.2.4 Adsorption/desorption.

### 5 **R.7.1.15.1 Type of property**

6 Adsorption/desorption is not a specific physicochemical property of a substance. This property  
7 indicates the binding capacity (or “stickiness”) of a substance to solid surfaces, and so is  
8 essential for understanding environmental partitioning behaviour.

9 Information on adsorption/desorption is an essential input to environmental exposure models,  
10 because:

- 11 • adsorption to suspended matter can be an important physical elimination process from  
12 water in sewage treatment plants (STPs). This in turn may mean that sewage sludge, if  
13 spread to land, is a major source of the substance in soil;
- 14 • adsorption to suspended matter in receiving waters affects both the concentration in  
15 surface water and the concentration in sediment;
- 16 • desorption of a substance from soil directly influences its mobility and potential to reach  
17 surface or groundwaters.

18 Consequently, information on adsorption/desorption is also an important factor in test  
19 strategies for assessing toxicity to sediment- or soil-dwelling organisms.

20 Substances that adsorb strongly to biological surfaces (e.g., gills, skin, etc.) may lead to toxic  
21 effects in higher organisms after biomagnification.

22 The information is also relevant for assessing environmental persistence. For example:  
23 degradation rates in sediment and soil are also assumed to be reduced by default if a  
24 substance is highly sorptive (since it is less bioavailable to microorganisms). This may lead to  
25 consideration of soil/sediment simulation testing in some cases.

26 Finally, there may be practical implications for test performance: Substances that adsorb  
27 strongly to surfaces can be difficult to test in aquatic systems.

### 28 **R.7.1.15.2 Definition**

29 Adsorption is caused by temporary (reversible) or permanent bonding between the substance  
30 and a surface (e.g. due to van der Waals interactions, hydrogen bonding to hydroxyl groups,  
31 ionic interactions, covalent bonding, etc.). The OECD guidances offer further information  
32 (OECD 2000a, OECD 2000b, OECD 2001, OECD 2002).

33 The organic carbon normalized adsorption coefficient ( $K_{oc}$ ) is the ratio of a substance  
34 concentration sorbed in the organic matter component of soil or sediment to that in the  
35 aqueous phase at equilibrium. In other words,  $K_{oc} = K_d/f_{oc}$ , where  $K_d$  is the distribution  
36 coefficient for adsorption, and  $f_{oc}$  the organic carbon content – the fraction organic carbon  
37 present in the soil or sediment. In turn,  $K_d$  is the experimental ratio of a substance's  
38 concentration in the soil ( $C_s$ ) to that in the aqueous phase ( $C_{aq}$ ) at equilibrium; namely  
39  $K_d = C_s/C_{aq}$ . The organic matter normalized distribution coefficient ( $K_{om}$ ) is similarly defined,  
40 but refers to the organic matter content of soil rather than the organic carbon content (OECD,  
41 2000a).

### 42 **R.7.1.15.3 Test method(s)**



- 1 The adsorption of a substance to sewage sludge, sediment and/or soil can be measured or
- 2 estimated using a variety of methods, which are tabulated in Table R.7.1-13 in order of
- 3 increasing complexity. The dissociation constant (if appropriate) should be known before
- 4 testing. Information on vapour pressure, solubility in water and organic solvents, octanol-water
- 5 partition coefficient and stability/degradability is also useful.

1 **Table R.7.1-13 Methods for the measurement of adsorption**

Method and Description	Applicability/Notes
<p>Adsorption control within an inherent biodegradability test (OECD TG 302B)</p> <p>Estimate of the extent of adsorption to STP sludge made from the elimination level in a Zahn-Wellens inherent biodegradation test. (e.g. OECD TG 302B).</p> <p>3-hour value recommended. Values beyond 24 hours not normally used. Where data are not available for adsorption up to 24 hours, data from time scales beyond this can only be used if adsorption is the only removal mechanism, with an upper limit of 7 days.</p>	<p>Highly adsorptive substances that are water soluble</p>
<p>HPLC method: OECD TG 121; EU C.19: Estimation of the Adsorption Coefficient (<math>K_{oc}</math>) on Soil and on Sewage Sludge using High Performance Liquid Chromatography (HPLC) (Original Guideline, adopted 22 January 2001)</p> <p>Calibration with reference substances (preferably structurally related to the test substance) of known <math>K_{oc}</math> allows the <math>K_{oc}</math> of the test substance to be estimated. Test substance <math>K_{oc}</math> value should lie within the calibration range of the reference substances.</p>	<p>Measurement of log <math>K_{oc}</math> in the range 1.5 to 5.0.</p> <p>Validated for several chemical types, see test guideline for details.</p> <p>Poorly soluble and volatile substances as well as mixtures.</p> <p>Ionisable substances: test both ionised and unionised forms in appropriate buffer solutions where at least 10 % of the test compound will be dissociated within pH range 5.5 to 7.5.</p> <p>May not be suitable for: substances that react with the column, solvent or other test system components; surface active substances; substances that interact in a specific way with inorganic soil components such as clay minerals; inorganic compounds; moderate to strong acids and bases.</p>
<p>Batch test of adsorption of substances on activated sludge (ISO 18749)</p> <p>Screening method to determine the degree of adsorption of substances on activated or primary sludge in sewage treatment plants (ISO, 2004). The method does not differentiate between adsorption and other elimination methods (such as complex formation, flocculation, precipitation, sedimentation or biodegradation).</p>	<p>Suitable for substances that:</p> <ul style="list-style-type: none"> <li>are water soluble, or allow for stable suspensions/dispersions/emulsions,</li> <li>are not significantly removed by abiotic processes (e.g. stripping/foaming),</li> <li>do not de-flocculate activated sludge,</li> <li>are not readily biodegradable, and</li> <li>have a sufficiently sensitive analytical method.</li> </ul>
<p>Sediment and soil adsorption/desorption isotherm (OPPTS 835.1220)</p> <p>Screening method according to US-EPA guideline (OPPTS, 1996) using three soil types.</p>	
<p>Batch equilibrium method (OECD TG 106; EU C.18: Absorption – Desorption Using a Batch Equilibrium Method (Updated Guideline, adopted 21 January 2000)</p> <p>Test uses a range of actual soils and so represents a more realistic scenario than the HPLC (OECD 121) method.</p>	<p>Used for substances with <math>K_{oc}</math> values that cannot be reliably determined using other techniques (e.g. surfactants).</p> <p>Requires a quantitative analytical method for the substance, reliable over the range of test concentrations.</p> <p>For ionisable substances, soil types should cover a wide range of pH.</p> <p>Adjustments for poorly soluble substances given in the test guideline.</p>

OECD TG 312: Leaching in Soil Columns (Original Guideline, adopted 13 April 2004) $K_d$ values can be derived from column leaching studies.	Appropriate study design to estimate $K_d$ values particularly for unstable test substances that degrade significantly during the equilibrium time of 'shake flask' sorption studies
Simulation tests and direct field measurement: including OECD guidance document no. 22 (OECD, 2000b).  Monolith lysimeters can be used to study the fate and behaviour of substances in an undisturbed soil profile under outdoor conditions. They allow for monitoring of the volume of leaching/drainage water as well as the concentrations of a substance and its transformation products. They are mainly used in pesticide studies. Field leaching studies can also be carried out where hydrodynamically isolated soil layers are analysed <i>in situ</i> . Although such studies are the most realistic, their reproducibility and representativity may be limited (e.g. due to the effects of large-scale soil structure, weather events, the soil conditions at the time of application, etc.). Since data from these methods are unlikely to be encountered for the vast majority of industrial substances, they are not considered further here. Further information can be found in guidance for pesticide registration.	

1

## 2 R.7.1.15.4 Adaptation of the standard testing regime

3 **Adaptation possibilities according to column 2 of Annex VIII and IX to REACH**

4 Screening information on adsorption (and desorption) is required for substances manufactured  
5 or imported in quantities of 10 t/y or more. Depending on the results, further information (for  
6 example, a test) may be required for substances manufactured or imported in quantities of  
7 100 t/y or more.

8 Column 2 of REACH Annexes VIII and IX provides two exemptions.

9 *The study does not need to be conducted if:*

- 10 • *based on the physicochemical properties the substance can be expected to have a low*  
11 *potential for adsorption (e.g. the substance has a low octanol water partition*  
12 *coefficient), or*
- 13 • *the substance and its relevant degradation products decompose rapidly.*

14 Or in other words, the substance and its relevant degradation products decompose rapidly.  
15 Therefore, if a substance hydrolyses, it might be more appropriate to also determine the  
16 degree of adsorption of the hydrolysis products.

17 In practice, a cutoff value of  $\log K_{ow} = 3$  can be applied for adsorption potential. However,  
18 caution should be exercised in using this criterion, as substances that are water soluble and  
19 have a low octanol-water partition coefficient do not necessarily always have a low adsorption  
20 potential. A *measured* adsorption coefficient is usually needed for ionising substances, since it  
21 is important to have information on pH-dependence (cationic substances in particular generally  
22 adsorb strongly). Similarly, measured values will normally be needed for surface active  
23 substances (e.g. surfactants), because  $K_{ow}$  values (predicted or measured) are likely to be poor  
24 predictors of adsorption for these types of substance. For ionisable substances, partition  
25 coefficients should also be corrected according to the pH of the environment being assessed  
26 (see Annex 2). For complex mixtures (e.g. UVCBs), a single value of  $K_{oc}$  will not be definitive.  
27 In such cases a range of values or a representative value can be given, depending on the  
28 substance.

## Adaptation possibilities according to Annex XI to REACH

### USE OF EXISTING DATA: DATA ON PHYSICAL-CHEMICAL PROPERTIES FROM EXPERIMENTS NOT CARRIED OUT ACCORDING TO GLP OR THE TEST METHODS REFERRED TO IN ARTICLE 13 (3) OF REACH

For all organic substances manufactured or supplied in quantities of 10 tonnes per year or more, the  $K_{oc}$  should be estimated using read-across or QSPR methods as a first step. If the property is likely to be a significant determinant in the calculation of risk (e.g. following a sensitivity analysis), then a test should be conducted to provide a more reliable value for substances manufactured or supplied in quantities of 100 t/y or more. In general, confirmatory testing would not be expected for non-ionising substances with a  $\log K_{ow}$  value below 3, or for substances that degrade rapidly (in which case the degradation products may be more relevant). The HPLC method may be used as a first step in testing, with the batch equilibrium method being considered only if more definitive data become necessary for the Chemical Safety Assessment. Column leaching studies might be an option under some circumstances (e.g. for unstable test substances that degrade significantly during the equilibrium time of shake flask sorption studies).

If estimation methods are not appropriate (e.g. because the substance is a surfactant or ionisable at environmentally-relevant pH), then a batch equilibrium test may need to be considered at the 10 tonnes per year band, and would be essential at the 100 tonnes per year band.

If experimental data are available (study reports or literature data) meeting the criteria in section 1.1.1 of Annex XI to REACH, these could be used to meet the endpoint data requirements. If an estimation method is used as a source of information according to Column 2 of Annex VII, the QSAR model must meet the criteria set out in section 1.3 of Annex XI to REACH.

### WEIGHT OF EVIDENCE

Where no single source of existing data (study reports, QSAR, literature data) is considered sufficiently reliable, thus not fully meeting the criteria in section 1.1.1 of Annex XI to REACH, or where several sources of similar reliability with deviating results exist, a weight of evidence approach may be used. The criteria in section 1.2 of Annex XI to REACH must then be met.

### (Q)SAR

Soil sorption ( $K_{oc}$ ) of organic non-ionic substances can often be estimated from their octanol-water partition coefficient ( $K_{ow}$ ), as well as from other properties such as aqueous solubility. Such methods, including QSPR, are useful in the first instance to indicate the qualitative/quantitative adsorption coefficient of a substance. In some instances an estimated value may be sufficient for this endpoint. In all such cases the estimated method must be proven to be valid for the type of substance considered (see the general guidance for use and applicability of QSPR), and if possible a sensitivity analysis should be conducted with values generated from different models. Using a range of values in the CSA will help to highlight if the adsorption coefficient is an important factor for environmental behaviour of the substance. In general an estimated value will be sufficient if it is indicated that the adsorption coefficient will not affect the CSA, i.e. no risk is identified for the sediment/soil compartments. Estimated values are essential for substances for which experimental measurement is not feasible i.e. for *difficult substances*. Estimated values are also useful for comparing screening tests [e.g. HPLC method (OECD 121; EC C19)]. A number of reviews of  $K_{oc}$  prediction have been published recently (Lyman 1990, Reinhard & Drefahl 1999, Doucette 2000, Delle Site 2001, Doucette

2003, Dearden 2004). That of Doucette (2000) contains a number of worked examples of the estimation of log  $K_{oc}$  values. Additional information on the  $K_{oc}$  can be found in Gerstl (1990), Briggs (1981) and Nendza (1998).

#### **GROUPING OF SUBSTANCES AND READ-ACROSS APPROACH**

Read-across and/or QSPR prediction for  $K_{oc}$  are important predictive tools and should be the first method used to predict  $K_{oc}$  if reliable measured data do not exist and the model is valid for the substance. However if these options do not give meaningful and valid information or if  $K_{oc}$  is an important factor in the CSA (i.e. risks are indicated for sediment/soil compartments based on a predicted value and  $\log K_{ow} > 3$ ), then an experimental value should be measured.

#### **TESTING IS TECHNICALLY NOT POSSIBLE**

Testing should always be considered, if none of the waiving possibilities applies. In general, partition coefficients that are measured with a suitable standard method are preferred (and they are usually essential for surfactants and ionic substances that dissociate at environmentally relevant pH).

#### **Further adaptation possibilities**

Not foreseen. The  $K_{oc}$  is not directly relevant for environmental classification or the PBT assessment. However, it is a key property for exposure assessment so the information requirement should not be waived.

#### **R.7.1.15.5 Impurities; uncertainties**

Impurities can have an impact on the measurement of adsorption/desorption. Expert judgement should be used when considering whether impurities may affect the determination of the adsorption/desorption. Therefore utmost care should be taken in the selection of the key study(s), or weight-of-evidence approaches, that the data selected is representative of the substance being registered by the respective companies.

#### **R.7.1.15.6 Endpoint specific information in the registration dossier/ in IUCLID**

#### **HPLC method (OECD TG 121, EU C.19)**

##### **Materials and methods**

- description of the HPLC equipment and operating conditions (column, mobile phase, means of detection, temperature);
- dead time and method used for its determination;
- reference substances (identity, purity,  $K_{oc}$ , retention times) with results of at least 6 measurements with at least one of them above and one below the expected value for the test substance;
- quantities of test and reference substances introduced in the column.

##### **Results and discussion**

- average retention data and estimated  $\log K_{oc}$  value for test compound;
- all values of  $\log K_{oc}$  derived from individual measurements.

#### **Batch equilibrium method (OECD TG 106, EU C.18)**

## 1 **Materials and methods**

- 2 • details on soil types (nature and sampling site(s), organic C, clay content and soil texture,  
3 and pH, if relevant Cation Exchange Capacity);
- 4 • information on the test substance (nominal and analytical test concentrations, stability and  
5 adsorption on the surface of the test vessel, solubilising agent if relevant (and justification for  
6 its use), radiochemical purity if relevant);
- 7 • details on test conditions (e.g. soil/solution ratio, number of replicates and controls, sterility,  
8 test temperature, and pH of the aqueous phase before and after contact with the soil);
- 9 • details on sampling (e.g. frequency, method);
- 10 • details on the analytical methods used for determination of the substance (detection limit,  
11 recovery %);

## 12 **Results and discussion**

- 13 • soil dry mass, total volume of aqueous phase, concentration of test substance in solution  
14 and/or soil after agitation and centrifugation, equilibration time, Koc, if appropriate mass  
15 balance;
- 16 • explanations of corrections made in the calculations, if relevant (e.g. blank run);

## 17 **Leaching in soil columns (OECD TG 312)**

### 18 **Materials and methods**

- 19 • details on soil types (nature and sampling site(s), organic C, clay content and soil texture,  
20 Cation Exchange Capacity, bulk density (for disturbed soil), water holding capacity and pH;
- 21 • information on the test substance (amount of test substance and, if appropriate, reference  
22 substance applied, solubilising agent if relevant (and justification for its use), radiochemical  
23 purity if relevant);
- 24 • details on test conditions (number of replicates and controls, test temperature, amount,  
25 frequency and duration of application of artificial rain);
- 26 • details on the analytical methods used for determination of the substance (detection limit,  
27 recovery %);
- 28 • reference substance used.

### 29 **Results and discussion**

- 30 • Koc, tables of results expressed as concentrations and as % of applied dose for soil segments  
31 and leachates;
- 32 • mass balance, if appropriate;
- 33 • leachate volumes;
- 34 • leaching distances and, where appropriate, relative mobility factors.

## 35 **Adsorption control within an inherent biodegradability test (OECD TG 302B)**

### 36 **Materials and methods**

- 37 • details on inoculum;
- 38 • Information on the test substance (toxicity to bacteria, test concentration);
- 39 • details on test conditions (blank controls used, inoculum and test compound ratio (as DOC));

• details on sampling (frequency);

• details on the analytical methods used for determination of the DOC or COD;

• reference substance.

#### **Results and discussion**

• estimate of the extent of adsorption to STP sludge made from the elimination level in this Zahn-Wellens inherent biodegradation test, based on the 3-hour value if possible;

• values beyond 24 hours should not normally be used but where data is not available for adsorption up to 24 hours, data from time scales beyond this can only be used if adsorption is the only removal mechanism, with an upper limit of 7 days;

• if relevant results of testing of inhibition of biodegradation.

#### **Simulation test/field measurement (OECD TG 22)**

##### **Materials and methods**

• details on soil types (nature and sampling site(s); if relevant: organic C, clay content and soil texture, Cation Exchange Capacity and pH;

• details on lysimeter;

• information on the test substance (nominal and analytical test concentrations, solubilising agent if relevant (and justification for its use), radiochemical purity if relevant);

• details on test climate conditions (e.g. air temperature, solar radiation, humidity, potential evaporation or rate of artificial rainfall), soil temperature and soil moisture and duration of the study;

• details on sampling (frequency, method);

• details on the analytical methods used for determination of the test substance (detection limit, recovery %).

##### **Results and discussion**

• concentration of test substance in soil layers; Koc, if appropriate mass balance and concentrations and as % of applied dose for soil segments and leachates;

• explanations of corrections made in the calculations, if relevant (e.g. blank run).

#### **Distribution modelling**

##### **Materials and methods**

• model name and version;

• date of the model development;

• model type description e.g. steady-state, dynamic, fugacity, Gaussian, Level I-IV, etc.;

• environmental compartments which the model covers;

• information on model segmentation and environmental properties;

• input parameters (minimum information required for assessing the partitioning and degradation behaviour):

• vapour pressure;



- 1 • water solubility;
- 2 • molecular weight;
- 3 • octanol-water partition coefficient;
- 4 • information on ready biodegradability;
- 5 • for inorganic substances: it is recommended to have information on the partition coefficients
- 6 and possible abiotic transformation products;
- 7 • temperature effect.

## 8 **Results and discussion**

- 9 • key exposure routes and distribution of the substance among them.

10 Any deviation from the guideline method used (and reasons for it) or any other special  
11 consideration should be reported. In cases where there is more than one source of data, the  
12 endpoint summary under results and discussion should provide a justification for the selection  
13 of the key study chapter.  
14

## 15 **Reference to other ECHA Guidance Documents**

16 Further detailed guidance on adsorption/desorption can be found in the following chapters:

IUCLID Section	REACH Annex	Endpoint title	IUCLID 5 End User Manual Chapter	ECHA Practical Guide 3
5.4.1	VIII 9.3.1	Adsorption / desorption	E.5.5.2	4.1.4
5.4.2	/	Henry's Law constant	E.5.5.3	4.1.4
5.4.3	X 9.3.4	Distribution modelling	E.5.5.4	4.1.4
5.4.4	X 9.3.4	Other distribution data	E.5.5.5	4.1.4

17

### 18 R.7.1.15.7 Further information/references

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- 25 OECD (2002) Guidelines for Testing of Chemicals (Draft): Leaching in Soil Columns.  
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#### 38 **R.7.1.16 Stability in organic solvents and identity of relevant degradation** 39 **products**

##### 40 R.7.1.16.1 Type of property

41 The stability in organic solvents is required for substances manufactured or imported in  
42 quantities of  $\geq 100$  t/a only if their stability in organic solvent is considered critical (REACH  
43 Annex IX, section 7.15).

There are rare occasions when it is important to have information on the stability of a compound in an organic solvent, to ensure confidence in the test results. However, for many substances, the stability in organic solvents will not be critical and testing need not be conducted.

Examples of when stability in organic solvents could be important are:

- for certain solubility measurements (e.g. octanol–water partition coefficient);
- to check on the stability of reagent solutions, fortification standards or calibration standards;
- when a test substance is dosed as a solution in an organic solvent (e.g. ecotoxicity studies);
- when a test substance is extracted from an environmental sample, plant or animal tissue or diet matrix (arising from a variety of physicochemical property, ecotoxicity and animal toxicity studies) into an organic solvent and stored pending analytical measurement.

#### R.7.1.16.2 Definition

A study of the stability of a test compound in an organic solvent is normally undertaken for a specific time period to confirm whether the test compound is stable under these conditions for the duration of the storage of the organic solvent or extract containing the test substance. Often several time periods are selected to check whether there is any particular downward trend in stability over time.

The stability of the test substance at a particular time period during the study is normally expressed as a percentage of the concentration of the test substance in the solvent extract, at that time period compared with the initial starting concentration of the test substance at  $t = 0$ , namely:

$$\frac{C_t}{C_0} \times 100 \%$$

where  $C_t$  is the concentration of test substance in solvent extract at  $t = t_1, t_2, t_3, \dots, t_n$ ; and  $C_0$  is the concentration of test substance in solvent extract at  $t = 0$ .

#### R.7.1.16.3 Test method(s)

A number of physical, chemical and biological processes can result in a decline in the actual concentration of a test substance in an organic solvent over time. Information on the stability of a test substance in a solvent is desirable, particularly when samples are to be stored. However, there does not appear to be any generally accepted methodology for performing such stability studies. Factors affecting the rate of degradation include rates of hydrolysis, of photolysis and of oxidation.

Typically, one or more concentrations of the test substance in the solvent are made up and analysed immediately after preparation (i.e.  $t = 0$ ). They are then stored in appropriate vessels under the required test conditions (e.g. temperature, absence of light) and analysed, along with a freshly prepared solution of the test substance at the original test concentration(s), at regular intervals during the period of interest.

At each time of analysis, a sample is withdrawn from storage and mixed thoroughly before taking any aliquot for analysis. The analysis is carried out using the recommended method to determine whether any significant loss of the test substance has occurred during storage. It is important to analyse freshly made standards of the test substance in the organic solvent at the

same time as analysing stored samples, so that any losses that may occur of the test substance during sampling, sample treatment and analysis are taken into consideration.

It is important to be able to have a check on the temperature to ensure that the temperature regime has been maintained throughout the period of the stability study.

Unlabelled reference material of suitable known purity may be used where a reliable method of analysis is available. Where an analytical method is still under development or is unlikely to be sufficiently sensitive, radio-labelled compounds should be used if available. Use of radio-labelled compounds can shorten the analysis time and help facilitate identification of any degradation products, should the test substance not be stable in the organic solvent.

Recovery or spiking experiments should normally be run. The number of spiking levels or the range of concentrations tested within a project should be left to the judgement of the analyst.

Further information should be obtained by checks on the stability of standards of the test substance in organic solvents as part of routine analytical protocols, to confirm whether the test substance is unstable under normal storage conditions.

Further tests may be necessary to identify storage conditions which minimise any degradation of the test substance not only in organic solvents, but also during the conducting of other tests, such as water solubility, surface tension and in the preparation of test media for ecotoxicity studies (OECD, 2000). Identification of the degradation product(s) will allow an assessment of whether they are likely to be more toxic than the parent material in subsequent ecotoxicity studies.

#### R.7.1.16.4 Adaptation of the standard testing regime

##### **Adaptation possibilities according to column 2 of Annex IX to REACH**

Column 2 of REACH Annex IX provides the following specific rules for adaptation of the standard information requirement for stability in organic solvents and identity of relevant degradation products:

*The study does not need to be conducted if the substance is inorganic.*

##### **Adaptation possibilities according to Annex XI to REACH**

##### **USE OF EXISTING DATA: DATA ON PHYSICAL-CHEMICAL PROPERTIES FROM EXPERIMENTS NOT CARRIED OUT ACCORDING TO GLP OR THE TEST METHODS REFERRED TO IN ARTICLE 13 (3) OF REACH**

Stability data of substances in organic solvents are not normally reported in standard published sources of physicochemical data. Relevant sources of basic information regarding stability and storage conditions of substances are the Hazardous Substances Data Base (HSDB) and Sax's 'Dangerous Properties of Industrial Materials'.

If experimental data are available (study reports or literature data) meeting the criteria in section 1.1.1 of Annex XI to REACH, these could be used to meet the endpoint data requirements. If an estimation method is used as a source of information according to Column 2 of Annex VII, the QSAR model must meet the criteria set out in section 1.3 of Annex XI to REACH.

## WEIGHT OF EVIDENCE

Where no single source of existing data (study reports, QSAR, literature data) is considered sufficiently reliable, thus not fully meeting the criteria in section 1.1.1 of Annex XI to REACH, or where several sources of similar reliability with deviating results exist, a weight of evidence approach may be used. The criteria in section 1.2 of Annex XI to REACH must then be met.

## (Q)SAR

At present (Q)SAR is generally not applicable for determination of stability in organic solvent and degradation products. Application of (Q)SAR is not possible.

## GROUPING OF SUBSTANCES AND READ-ACROSS APPROACH

At present grouping and read across are not applicable.

## TESTING IS TECHNICALLY NOT POSSIBLE

Testing should always be considered, if none of the waiving possibilities applies.

## Further adaptation possibilities

Not foreseen.

### R.7.1.16.5 Impurities; uncertainties

Impurities can have an impact on the measurement of stability in organic solvent and degradation products. Expert judgment should be used when considering whether impurities may affect the determination of the stability in organic solvent and degradation products. Therefore utmost care should be taken in the selection of the key study(s), or weight-of-evidence approaches, that the data selected is representative of the substance being registered by the respective companies.

### R.7.1.16.6 Endpoint specific information in the registration dossier / in IUCLID

This endpoint needs to be fulfilled on a case by case basis. As several different methods can be used to document this intrinsic property, we recommend the same strategy for drafting robust study summaries as described for the other endpoints. The general aspects described in section 2 should also be applied for this endpoint. All endpoint specific characteristics should be described in such a way that the RSS allows an independent assessment of the endpoints reliability and completeness. The objectives, methods, results and conclusions of the full study report should be reported in a transparent manner as described for all other endpoints in this practical guide.

Any deviation from the guideline method used or any other special consideration should be reported. In cases where there is more than one source of data, the endpoint summary under results and discussion should provide a justification for the selection of the key study chapter.

## REFERENCE TO OTHER ECHA GUIDANCE DOCUMENTS

Further detailed guidance on stability in organic solvents can be found in:

IUCLID	REACH	Endpoint title	IUCLID 5 End User Manual	ECHA Practical
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Section	Annex		Chapter	Guide 3
4.17	VII 7.15	Stability in organic solvents and identity of relevant degradation products	E.4.18	3.14

#### 1 R.7.1.16.7 Further information / references

2 OECD Series on Testing and Assessment Number 23 Guidance Document on Aquatic Toxicity  
3 Testing of Difficult Substances and Mixtures, ENV/JM/MONO(2000)6  
4 ([http://www.oecd.org/officialdocuments/displaydocumentpdf?cote=env/jm/mono\(2000\)6&doclanguage=en](http://www.oecd.org/officialdocuments/displaydocumentpdf?cote=env/jm/mono(2000)6&doclanguage=en))  
5

### 6 R.7.1.17 Dissociation constant

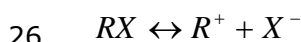
#### 7 R.7.1.17.1 Type of property

8 Information on the dissociation constant is **supplementary data** for hazard assessment  
9 (OECD TG 112, 1981). The dissociation of a substance in water is of importance in assessing  
10 its impact upon the environment and may also influence the ADME of a substance and  
11 consequently its effects on human health. It governs the form of the substance which in turn  
12 determines its behaviour and transport. It may affect the adsorption of the substance on soils  
13 and sediments and absorption into biological cells.

14 The dissociation constant may also be an important factor in deciding which method or  
15 conditions should be used to determine the octanol-water partition coefficient ( $K_{ow}$ ) and soil  
16 adsorption partition coefficient ( $K_{oc}$ ). Slight changes in pH can considerably affect the form in  
17 which the substance is present in solution, especially if the  $pK_a$  value is within the  
18 environmentally-relevant pH range<sup>14</sup>. The dissociated and non-dissociated species may have  
19 significantly different water solubilities and partition coefficients. Therefore, significantly  
20 different bioavailability and toxicity may result. It is important to note that the dissolution of  
21 salts from their crystal lattice into individual ions is not intended to be covered by the endpoint  
22 dissociation constant. Therefore this section refers only to acid dissociation ( $pK_a$ ).

#### 23 R.7.1.17.2 Definition

24 Dissociation is the reversible splitting of a substance into two or more chemical species, which  
25 may be ionic (OECD TG 112, 1981). The process can be represented as:



27 The dissociation constant (K) for this process is expressed as the ratio of concentrations of the  
28 species on either side of the equation in water at equilibrium:

$$29 \quad K = \frac{[R^+][X^-]}{[RX]}$$

30 Where the cation  $R^+$  is hydrogen, the substance can be considered an acid, and so this  
31 constant becomes an acid dissociation constant ( $K_a$ ).

---

14 Fresh surface waters have pH values in the range 4-9, whereas marine environments have a stable pH of about 8. pH normally varies between 5.5 and 7.5 for agricultural soils and sewage treatment plant tanks.

$$K_a = \frac{[H^+][X^-]}{[HX]}$$

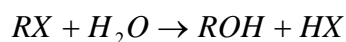
A substance can have more than one acidic (or basic<sup>15</sup>) group, and the dissociation constant can be derived for each dissociation step in a similar way.

The  $K_a$  is related to pH as follows (where p is  $-\log_{10}$ ):

$$pK_a = pH - \log_{10} \left( \frac{[X^-]}{[HX]} \right)$$

In practice for a simple substance having one dissociating group, the  $pK_a$  is equivalent to the pH at which the ionised and non-ionised forms are present in equal concentration (i.e. the substance has undergone 50% dissociation).

It is important to differentiate between dissociation and hydrolysis as hydrolysis is a separate standard information requirement according to Annex VIII of the REACH regulation. Hydrolysis is defined as reaction of a substance RX with water, with the net exchange of the group X with OH at the reaction centre (OECD TG 111, 2004).



#### R.7.1.17.3 Test method(s)

OECD test guideline 112 (Dissociation constants in water, adopted May 1981) describes three laboratory methods to determine the  $pK_a$  of a substance. The three methods are appropriate for particular types of substances as described in the test guideline<sup>16</sup>.

#### R.7.1.17.4 Adaptation of the standard testing regime

### **Adaptation possibilities according to column 2 of Annex IX to REACH**

Column 2 of REACH Annex IX provides the following specific rules for adaptation of the standard information requirement for dissociation constant:

*A study does not need to be conducted if:*

- *the substance is hydrolytically unstable (half-life less than 12 hours) or is readily oxidisable in water; or*
- *it is scientifically not possible to perform the test (e.g. because the analytical method is not sensitive enough).*

In all cases where the above specific rules for adaptation are used to waive testing, evidence demonstrating the existence of that property of the substance which triggers the adaptation rule should be provided in the IUCLID dossier, e.g. if the test is not performed because the substance is hydrolytically unstable (half life < 12 hours) then the dossier must contain valid data on the hydrolysis clearly indicating a half life < 12 hours.

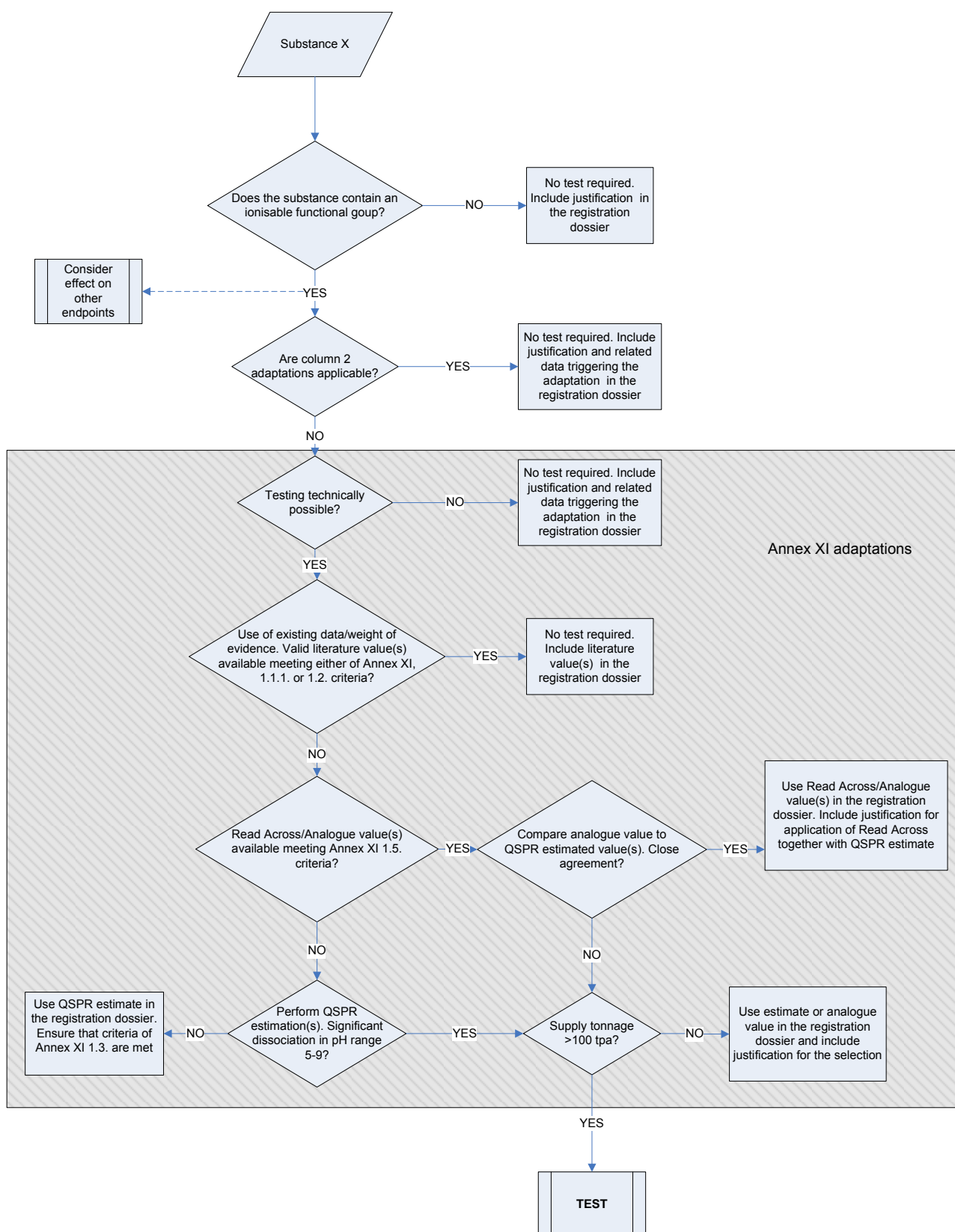
<sup>15</sup> Base strength is expressed as the acidity of the conjugate acid. The term  $pK_b$  was once used to express basicity so that the same scale could be used alongside acidity – care should be taken when citing older sources to check which term has been used. For consistency, dissociation of bases should preferably be expressed using the  $pK_a$  of the conjugate acid.

<sup>16</sup> The test method is available at the following link: [http://www.oecd-ilibrary.org/environment/test-no-112-dissociation-constants-in-water\\_9789264069725-en](http://www.oecd-ilibrary.org/environment/test-no-112-dissociation-constants-in-water_9789264069725-en)



- 1 It is important to note that OECD TG 112 allows the use of a small amount of a water-miscible
- 2 solvent to aid dissolution of sparingly soluble substances. Therefore low solubility will only
- 3 prevent performance of the test in the context of the column 2 rules above for substances
- 4 which remain highly insoluble and undetectable by analytical techniques in the presence of
- 5 water miscible solvents.

1 **Figure R.7.1-7 Integrated testing strategy for dissociation constant**



## 1 **Adaptation possibilities according to Annex XI to REACH**

### 2 **USE OF EXISTING DATA: DATA ON PHYSICAL-CHEMICAL PROPERTIES FROM** 3 **EXPERIMENTS NOT CARRIED OUT ACCORDING TO GLP OR THE TEST METHODS** 4 **REFERRED TO IN ARTICLE 13 (3) OF REACH**

5 Many literature sources for dissociation constant exist; some reference textbooks and on-line  
6 sources are listed in section R.7.1.1.2. These should be searched for published, valid data. As  
7 mentioned in section 1.1.1 of Annex XI to REACH a number of conditions need to be met  
8 before any such data can be used. Namely:

- 9 • *adequacy for the purpose of classification and/or risk assessment;*
- 10 • *sufficient documentation is provided to assess the adequacy of the study; and*
- 11 • *the data are valid for the endpoint being investigated and the study is performed using*  
12 *an acceptable level of quality assurance.*

13 If experimental data are available (study reports or literature data) meeting the criteria in  
14 section 1.1.1 of Annex XI to REACH, these could be used to meet the endpoint data  
15 requirements. If an estimation method is used as a source of information according to Column  
16 2 of Annex VII, the QSAR model must meet the criteria set out in section 1.3 of Annex XI to  
17 REACH.

## 18 **WEIGHT OF EVIDENCE**

19 Where no single source of existing data (study reports, QSAR, literature data) is considered  
20 sufficiently reliable, thus not fully meeting the criteria in section 1.1.1 of Annex XI to REACH,  
21 or where several sources of similar reliability with deviating results exist, a weight of evidence  
22 approach may be used provided that data from a number of distinct sources indicate a similar  
23 value for the dissociation constant which is supported by one or more relevant QSPR  
24 predictions. The criteria in section 1.2 of Annex XI to REACH must then be met.

## 25 **(Q)SAR**

26 Estimated  $pK_a$  data can be generated by valid QSPR methods. In general,  $pK_a$  values that are  
27 measured with a suitable method are preferred to QSPR predictions. If an estimated  $pK_a$  value  
28 suggests that the substance will dissociate significantly at environmentally relevant pH, a test  
29 may be required to confirm the result.

30 There have been a few attempts to model  $pK_a$  values of diverse sets of substances. Klopman  
31 and Fercu (1994) used their MCASE methodology to model the  $pK_a$  values of a set of 2464  
32 organic acids, and obtained good predictions; a test set of about 600 organic acids yielded a  
33 standard error of 0.5  $pK_a$  unit. Klamt *et al.* (2003) employed their COSMO-RS methodology to  
34 predict  $pK_a$  values of 64 organic and inorganic acids, with a standard error of 0.49  $pK_a$  unit. A  
35 comparison of commercially available software for the prediction of  $pK_a$  was done by Dearden  
36 *et al.* (2007).

## 37 **GROUPING OF SUBSTANCES AND READ-ACROSS APPROACH**

38 For most ionisable substances supplied at greater than 100 t/y that are predicted to dissociate  
39 at environmentally relevant pHs, a test will typically be required for dissociation constant.  
40 Similar substances (analogues) for which measured  $pK_a$  data according to a reliable method  
41 are available may be considered for read-across. Such values should be reinforced by

estimated methods for  $pK_a$  (e.g. the result of a QSPR prediction; see section above). In some instances it may be acceptable to read-across dissociation constant from an analogue. However if there is significant variation between the analogue read-across and the predicted  $pK_a$  then a test should be conducted.

## TESTING IS TECHNICALLY NOT POSSIBLE

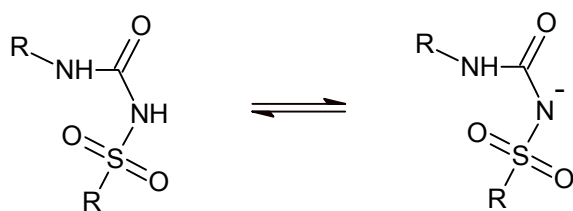
Testing should always be considered if none of the waiving possibilities applies. Instances where testing is technically not possible as a consequence of the properties of the substance are expected to be limited to highly reactive or unstable substances, and substances which in contact with water emit flammable gases.

## Further adaptation possibilities

As stated in Annex IX of REACH, when for certain endpoints, it is proposed to not provide information for other reasons than those mentioned in column 2 of that Annex or in Annex XI of REACH, this fact and the reasons must also be clearly stated. Such an approach may then be used.

### No dissociating groups

If the substance cannot dissociate due to a lack of relevant functional groups, the dissociation constant is irrelevant and testing information does not need to be provided. However, ionisable groups might not always be obvious (e.g. in sulphonyl urea herbicides, which contain the function  $-S(=O)_2NH.C(=O)NH-$ , the acid group is  $S(=O)_2NH$ ).



If a substance is much more soluble in water than expected, this may be an indication that dissociation has occurred.

### UVCBs

For complex mixtures (e.g. UVCBs) containing ionisable components the assessment of  $pK_a$  is clearly complicated. Estimation of the representative constituent's  $pK_a$  values, if appropriate, should be considered.

#### R.7.1.17.5 Impurities; uncertainties

Impurities can have an impact on the measurement of dissociation constant. Expert judgement should be used when considering whether impurities may affect the determination of the dissociation constant. Therefore utmost care should be taken in the selection of the key study(s), or weight-of-evidence approaches, that the data selected is representative of the substance being registered by the respective companies.

The presence of multiple dissociation/equilibrium reactions can complicate determination of the dissociation constant(s). In cases where multiple dissociation reactions can take place due to the presence of numerous dissociating groups and/or the presence of tautomerisation and/or zwitterionic forms, care should be taken in the interpretation of experimental results. QSPR predictions for such substances should also be carefully analysed as the models may not account for concurrent equilibria/dissociations. Additionally QSPR predictions may not account for intramolecular hydrogen bonding effects which can have a pronounced effect on the

observed dissociation constant. In some cases, formation of intramolecular hydrogen bonding depends on the cis/trans isomerism of the substance, as is the case for the isomers fumaric and maleic acid. Care should be taken when using QSPR predictions for such molecules, as cis/trans isomerism is typically not taken into account.

The extent of ionisation may vary according to pH, ionic strength and/or the level of common ions in the test medium (common ion effect), and relatively small changes may significantly alter the equilibrium between dissociated and non-dissociated species.

#### R.7.1.17.6 Endpoint specific information in the registration dossier / in IUCLID

Knowledge of an ionisable substance's  $pK_a$  is important for all such substances. For substances supplied at levels below 100 tonnes per annum dissociation constant is not a testing requirement. Ideally however, a literature value, analogue value and/or QSPR prediction can be obtained and provided for such substances, especially if dissociation is relevant for interpreting the results of other physicochemical or fate and (eco)toxicological tests and for chemical safety assessment. For ionisable substances supplied at tonnages greater than 100 t/y, dissociation constant is a standard information requirement.

For substances which contain multiple ionisable functionalities, all measured macro  $pK_a$  values should be reported and preferably assigned to specific micro-reactions.

With regard to study summaries of experimental data, the IUCLID dossier should contain all relevant information regarding the endpoint and as a minimum the items listed below:

#### **Materials and methods**

- type of method;
- test guideline followed.

#### **Test Materials**

- test material identity;

#### **Results and discussion**

- concentration of the substance;
- test results as  $pK_a$ -value(s);
- temperature of the test medium ( °C);
- If testing is waived, the reasons for waiving must be documented in the dossier.

Any deviation from the guideline method used (and reasons for it) or any other special consideration should be reported. In cases where there is more than one source of data, the endpoint summary under results and discussion should provide a justification for the selection of the key study chapter.

#### **Reference to other ECHA Guidance Documents**

Further detailed guidance on dissociation constant can be found in:

IUCLID Section	REACH Annex	Endpoint title	IUCLID 5 End User Manual Chapter	ECHA Practical Guide 3
4.21	IX 7.16	Dissociation constant	E.4.22	3.15

1    R.7.1.17.7    References on dissociation constant

- 2    Balogh G.T., Gyarmati B., Nagy B., Molnar L. and Keseru G.M. Comparative evaluation of in  
3    silico pK<sub>a</sub> prediction tools on the Gold Standard dataset. *QSAR Comb Sci* (2009) 28:1148-1155.
- 4    Dearden J.C., Cronin M.T.D., and Lappin D.C. A comparison of commercially available software  
5    for the prediction of pK<sub>a</sub>. *J. Pharm. Pharmacol.* (2007) 59, Suppl. 1, A-7.
- 6    Klamt A., Eckert F., Diedenhofen M. and Beck M.E. (2003) First principles calculations of  
7    aqueous pK(a) values for organic and inorganic acids using COSMO-RS reveal an inconsistency  
8    in the slope of the pK(a) scale. *J. Phys. Chem. A* 107, 9380-9386.
- 9    Klopman G. and Fercu D. (1994) Application of the multiple computer automated structure  
10    evaluation methodology to a quantitative structure-activity relationship study of acidity. *J.*  
11    *Comput. Chem.* (1994) 15, 1041-1050.
- 12    Liao C. and Nicklaus M.C. Comparison of nine programs predicting pK<sub>a</sub> values of  
13    pharmaceutical substances. *J. Chem. Inf. Model.* (2009) 49, 2801-2812.
- 14    Manchester J, Walkup G, Rivin O. and You Z.P. Evaluation of pK<sub>a</sub> estimation methods on 211  
15    druglike compounds. *J Chem Inf Model* (2010) 50, 565-571.
- 16    Meloun M. and Bordovská S. Benchmarking and validating algorithms that estimate pK<sub>a</sub> values  
17    of drugs based on their molecular structure. *Anal. Bioanal. Chem.* (2007) 389, 1267-1281.
- 18    OECD (1981). Dissociation constants in water (titration method – spectrophotometric method  
19    – conductometric method). Organisation for Economic Co-operation and Development (OECD)  
20    Guideline for the testing of chemicals no 112.

21

### 1 R.7.1.18 Viscosity

#### 2 R.7.1.18.1 Type of property

3 Viscosity is a property:

- 4 • needed for substance characterization;
- 5 • needed for the classification of aspiration hazard of liquids;
- 6 • which gives an indication of the penetration of the substance within soil.

#### 7 R.7.1.18.2 Definition

8 Viscosity: viscosity is the (inner) resistance of a substance (gas, liquid) to a shift caused by  
9 laminar flow.

10 Dynamic viscosity (= dynamic viscosity coefficient)  $\eta$ :

11 Quantifies the property 'viscosity' by the quotient shear stress  $\tau$  / shear rate  $\dot{\gamma}$  ( $\eta = \tau / \dot{\gamma}$ )

12 Kinematic viscosity (= kinematic viscosity coefficient)  $\nu$ :

13 is given by the quotient dynamic viscosity to density ( $\nu = \eta / \rho$ ).

#### 14 R.7.1.18.3 Test method(s)

15 Five different types of test methods are standardized for liquid substances:

- 16 • capillary viscometer;
- 17 • flow cup;
- 18 • rotational viscometer;
- 19 • rolling ball viscometer;
- 20 • drawn-shear viscometer.

21  
22 There exist a lot of standardized determination methods with sometimes very specialised  
23 application ranges with respect to products, especially mixtures. For substances (within the  
24 scope of the REACH Regulation) the following standardised determination methods are  
25 recommended:

- 26 • Capillary viscometer:
  - 27 ○ EN ISO 3104, EN ISO 3105, DIN 51562, BS 188, NF 60-100, ASTM D445, ASTM
  - 28 D4486;
- 29 • Flowcup:
  - 30 ○ EN ISO 2431;
- 31 • Rotational viscometer:
  - 32 ○ EN ISO 3219, DIN 53019;
- 33 • Rolling ball viscometer:
  - 34 ○ DIN 53015.

35  
36 For newtonian liquids (liquids for which the viscosity is independent of the shear stress and  
37 shear rate) any determination method may be used within the scope and applicability  
38 specifications. For non-newtonian liquids (liquids for which the viscosity depends on the shear  
39 rate) only the use of rotational viscometers is possible. Because the viscosity is remarkably  
40 temperature dependent each determination must be accompanied by the temperature at which  
41 the measurement was made. It is recommended to use the mean of two test runs. It is also  
42 recommended to determine the viscosity at at least two different temperatures. The  
43 classification criteria for aspiration hazard refer to kinematic viscosity at 40 °C.

44



If explosives, pyrophorics or self-reactives are to be characterized, determination of the viscosity may not be practicable. For pyrophorics and self-reactives testing under inert gas should be considered. In any case the determination method has to be chosen carefully. The use of the most recent update of the standard is advised; they are accessible via numerous websites, see R.7.1.1.3.

#### R.7.1.18.4 Adaptation of the standart testing regime

Within the REACH Regulation requirements testing of viscosity is only of interest for liquid substances.

### **Adaptation possibilities according to column 2 of Annex IX to REACH**

Column 2 of REACH Annex IX does not provide any specific rules for adaptation from column 1.

### **Adaptation possibilities according to Annex XI to REACH**

#### **USE OF EXISTING DATA: DATA ON PHYSICAL-CHEMICAL PROPERTIES FROM EXPERIMENTS NOT CARRIED OUT ACCORDING TO GLP OR THE TEST METHODS REFERRED TO IN ARTICLE 13 (3) OF REACH**

If experimental data are available (study reports or literature data) meeting the criteria in section 1.1.1 of Annex XI to REACH, these could be used to meet the endpoint data requirements. If an estimation method is used as a source of information according to Column 2 of Annex VII, the QSAR model must meet the criteria set out in section 1.3 of Annex XI to REACH.

#### **WEIGHT OF EVIDENCE**

Where no single source of existing data (study reports, QSAR, literature data) is considered sufficiently reliable, thus not fully meeting the criteria in section 1.1.1 of Annex XI to REACH, or where several sources of similar reliability with deviating results exist, a weight of evidence approach may be used. The criteria in section 1.2 of Annex XI to REACH must then be met.

#### **(Q)SAR**

For the determination of the viscosity, (Q)SAR approaches are discouraged for the purpose of classification / risk assessment, except when the mean absolute error of the (Q)SAR is less than 5 %.

#### **GROUPING OF SUBSTANCES AND READ-ACROSS APPROACH**

For the determination of the viscosity read across is not possible.

#### **TESTING IS TECHNICALLY NOT POSSIBLE**

Testing should always be considered if none of the waiving possibilities applies. But the testing is technically not possible:

- if the substance is a solid;
- if liquid explosives, pyrophorics or self-reactives are to be characterized, determination of the viscosity may not be practicable (see above section Test method(s)).

**Further adaptation possibilities**

- the viscosity does not have to be determined experimentally if conclusive and consistent literature data are available;
- data for viscosity generated with the same tests and classification principles as specified in the CLP Regulation generated in conjunction with transport classification can satisfy the REACH requirements on a case-by-case basis.

As stated in Annex IX of REACH, when for certain endpoints, it is proposed to not provide information for other reasons than those mentioned in column 2 of that Annex or in Annex XI of REACH, this fact and the reasons must also be clearly stated. Such an approach may then be used.

**R.7.1.18.5 Impurities; uncertainties**

The influence of impurities is negligible if their concentration is below 1 %. The influence of higher concentrations may be significant. There exists no generalised tendency of the influence on the viscosity. Therefore utmost care should be taken in the selection of the key study(s), or weight-of-evidence approaches, that the data selected is representative of the substance being registered by the respective companies.

**R.7.1.18.6 Endpoint specific information in the registration dossier / in IUCLID****Results and discussion**

- viscosity value and unit according to the used test method;
- preferred units are m Pa s (for dynamic viscosity) and mm<sup>2</sup>/s (for static viscosity) but other units are also accepted;
- each measured value should be accompanied with temperature (in °C). Usually two values are needed. Preferably one value is measured at approximately 20 °C and another at an approximately 20 °C higher temperature. Two determinations of viscosity should be measured for each temperature;
- for non-Newtonian liquids, the results obtained are preferably in the form of flow curves, which should be interpreted;
- individual and mean values should be provided at each temperature (from OECD Guideline 114 'Viscosity of liquids').

Any deviation from the guideline method used or any other special consideration should be reported. In cases where there is more than one source of data, the endpoint summary under results and discussion should provide a justification for the selection of the key study chapter.

**Reference to other ECHA Guidance Documents**

Further detailed guidance on viscosity can be found in the following chapters:

IUCLID Section	REACH Annex	Endpoint title	IUCLID 5 End User Manual Chapter	ECHA Practical Guide 3
4.22	VII 7.17	Viscosity	E.4.23	3.16

**R.7.1.19 Shape**

Please check Appendix R7-1 Recommendations for nanomaterials applicable to: Chapter R7a Endpoint specific guidance, section 2.2.3.3 Recommendations for shape.

**R.7.1.20 Surface area**

Please check Appendix R7-1 Recommendations for nanomaterials applicable to: Chapter R7a Endpoint specific guidance, section 2.2.3.4 Recommendations for surface area.

### R.7.1.21 Further information to be submitted for classification and labelling in hazard classes of the substance in accordance with article 10 (a) (iv) REACH

The criteria listed in the table below should be provided for general registration purposes according to Article 10 (a) (iv) and section 4 of Annex VI to REACH. The assignment of hazard classes to relevant subchapters in R.7.1.21.1 to R.R.7.21.3 should therefore only be understood as a means to structure this document in accordance with Annexes VII to XI to the REACH Regulation.

**Table R.7.1-14 Information to be submitted for general registration purposes according to Article 10 (a) (iv) REACH, CLP hazards classes and corresponding tests methods according to the Test Method Regulation and CLP<sup>17</sup>**

CLP Regulation (EC) No. 1272/2008 (the no. in brackets is the respective chapter no. in Annex I to CLP)	Corresponding test method according to the Test Method Regulation, Regulation (EC) No. 440/2008	Chapter in revised R.7(a) guidance	Information requirement according to REACH Regulation (EC) No. 1907/2006	Corresponding test method according to CLP Regulation
Flammable aerosols (2.3)	n.a.	7.1.21.1	See Article 10 (a) (iv) REACH requirements	Test methods according to 75/324/EC amended by 2008/47/EC (harmonised with <b>UN Section 31</b> )
Gases under pressure (2.5)	n.a.	7.1.21.2	See Article 10 (a) (iv) REACH requirements	n.a.
Corrosive to metals (2.16)	n.a.	7.1.21.3	See Article 10 (a) (iv) REACH requirements	<b>UN Test C.1 (section 37.4)</b>

#### R.7.1.21.1 Flammable aerosols

For further guidance on these please check the Guidance on the application of the CLP Criteria, chapter 2.4.

#### R.7.1.21.2 Gases under pressure

For further guidance please check the *Guidance on the Application of the CLP Criteria* chapter 2.6.

#### R.7.1.21.3 Corrosive to metals

For further guidance please check the *Guidance on the Application of the CLP Criteria* chapter 2.15.

<sup>17</sup> Please note that REACH information requirements regarding classification and labelling in accordance with Article 10(a)(iv) of the REACH Regulation are not limited to the items listed in this table. This table stresses that, while the REACH Regulation does not require the generation of information regarding the following hazard classes (Article 10(a)(vi) of the REACH Regulation, see Table R.7.1-1), any information available on these hazard classes shall be included in a REACH registration dossier for a substance pursuant to Article 10(a)(iv) of the REACH Regulation.

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## **Appendix 1 to Section R.7.1**

## Appendix R.7.1-1 Henry's law constant and evaporation rate

The Henry's law constant (HLC) is one of the most important factors in determining the environmental fate of chemicals. Henry's law states that the mass of gas dissolved by a given volume of solvent is proportional to the pressure of the gas with which it is in equilibrium. HLC is the ratio of the equilibrium concentration of the chemical in the gas phase ( $C_G$ ) and that in the liquid phase ( $C_L$ ):

$$\text{HLC} = \frac{C_G}{C_L}$$

Therefore, HLC quantifies the partitioning of substances between the aqueous phase and the gas phase such as rivers, lakes and seas with respect to the atmosphere (gas phase). Indeed, this constant is a fundamental input for fugacity models that estimate the multimedia partitioning of chemicals (Mackay, 1991). As HLC is a ratio of two concentrations, it is without unit if both concentrations are expressed in the same unit. Some prefer to express the gas concentration in pascals and the liquid concentration in mol/m<sup>3</sup>, thus giving the unit Pa·m<sup>3</sup>/mol for the HLC.

For many chemicals, volatilisation can be an extremely important removal process, with half lives as low as several hours. HLCs can give qualitative indications of the importance of volatilisation. For substances with HLC values less than 0.01 Pa·m<sup>3</sup>/mol, the substance is less volatile than water and as water evaporates the concentration of the substance in the aqueous phase will increase; for substances with HLC values around 100 Pa·m<sup>3</sup>/mol, volatilisation will be rapid.

However, the degree of volatilisation of substances from the aquatic environment is highly dependent on the environmental parameters for the specific water bodies in question, such as the depth and the gas exchange coefficient (influenced e.g. by wind speed and water flow rate). The HLC cannot be used for evaluation of the removal of a substance from the water phase without considering these factors. As the n-octanol/water partition coefficient ( $K_{ow}$ ) is used to predict bioaccumulation potential in air-breathing organisms, this aspect is especially important in a PBT context.

For example, where a substance has both a low vapour pressure and low water solubility, HLC can be relatively large if calculated using the ratio of vapour pressure and water solubility, which might imply that volatilisation is an important fate process. In practice, adsorption to dissolved organic carbon is likely to be much more relevant, and volatilisation will be lower than the HLC value suggests.

### EXPERIMENTAL DETERMINATION OF HENRY'S LAW CONSTANT

The experimental approaches can be classified into two major groups: dynamic equilibration approach (often referred to as the *gas purge* approach) and the static equilibration approach. The following table briefly summarises the reviewing work done by Staudinger and Roberts (1996).

1 **Table R.7.1-15 Experimental approaches for the determination of HLC**

Approach	Average Relative Standard Deviations (RSDs)/Notes
<b>Dynamic approach</b>	
<p><i>Batch air stripping (bubble column)</i></p> <p>Henry's law constant (HLC) values are determined by measuring the rate of loss of the substance of interest from water by isothermally stripping with a gas (typically air) in a suitable bubble column apparatus.</p>	Average RSDs determined from different literature sources ranged from 2.8 to 21
<p><i>Concurrent flow (wetted wall column)</i></p> <p>Values are determined based on the use of a wetted wall (desorption) column. The wetted wall column equilibrates an organic solute between a thin film of water and a concurrent flow of gas. Substance-laden water is introduced into the wetted wall column where it comes in contact with a substance-free gas stream flowing concurrently. HLC: The knowledge of flow rates and compound masses present in the separated phase streams enables the direct calculation of HLC.</p>	<p>Average RSDs determined from different literature sources ranged from 19 to 52</p> <p>Preliminary work must be performed to ensure that phase equilibrium is reached.</p>
<b>Static approach</b>	
<p><i>Single equilibration</i></p> <p>A known mass of a substance is introduced into an air-tight vessel with a known volume of water and air. When the equilibrium is attained the substance concentration is determined in one or both phases.</p>	Average RSDs determined from different literature sources ranged from 2.8 to 30
<p><i>Multiple Equilibration</i></p> <p>A liquid sample containing a known quantity of solute is allowed to equilibrate with a known volume of solute-free air. The air is expelled and a new equilibration with the same amount of solute-free air is started. This process can be repeated until the number of equilibrations exhausts the mass of solute remaining in the system.</p>	<p>RSDs ranged from 0.7 to 3.5</p> <p>This method is applicable for substances with <math>0.1 \leq \text{HLC} \leq 2</math></p> <p>The experimental error is reduced with a larger number of equilibrations.</p>
<p><i>EPICS Technique</i></p> <p>HLC is determined by measuring the gas headspace concentration ratios from pairs of sealed bottles. Relative rather than absolute air-phase concentrations are required.</p>	Average RSDs determined from different literature sources ranged from 2.9 to 19
<p><i>Variable Headspace</i></p> <p>The method is based upon the measurement of the relative equilibrium air-phase concentration (gas chromatography peak areas) from aliquots of the same solution in multiple containers having different headspace-to-liquid volume ratios.</p>	Average RSDs determined from different literature sources ranged from 0.5 to 7.9

2

3 A data-analysis of reviewed experimental studies for HLC can be found in Staudinger and  
 4 Roberts (1996). HLC values can also be found in one or more of the following references:

1 Sander (1999), CRC Handbook of Chemistry and Physics (2000), the NIST Chemistry WebBook  
2 (1998), and "The Handbook of Environmental Data on Organic Chemicals" (Verschuere K,  
3 2001).

#### 4 MAIN FACTORS AFFECTING HENRY'S LAW CONSTANT VALUES

5 Staudinger and Roberts (1996) thoroughly explain all the factors affecting HLC values and  
6 report equations that quantify the effect of temperature and pH. According to their work, in a  
7 majority of cases temperature is the main parameter affecting HLC values for natural waters  
8 with moderate contamination (1 mg/ml or less). Other conditions that have influence on HLC  
9 values are listed in Table R.7.1-16 (Staudinger and Roberts, 1996):

10 **Table R.7.1-16 Conditions that have influence on HLC values**

pH	Important for compound (substance) classes that dissociate to a significant extent in water because only nondissociated species undergo air-water exchange. For most natural waters ( $6 < \text{pH} < 8$ ) the apparent HLC will be significantly less than the intrinsic HLC.
Compound Hydration	Important for aldehydes, which hydrate nearly completely in water, resulting in HLC apparent being several orders of magnitude lower than the intrinsic constant.
Compound concentration/ Complex mixtures effects	If a solution cannot be regarded as diluted (e.g. concentration approaching 10.0 mg/ml) HLC apparent will be lower than HLC values determined at lower concentrations.
Dissolved salts	If the ionic strength of a solution is high (e.g. seawater) the apparent HLC will be higher than the HLC determined in pure water.
Suspended solids /Dissolved Organic Matter (DOM)	If a compound is easily adsorbed (e.g. pesticides) the apparent HLC will be higher than the HLC determined in pure water.
Surfactants	Compounds with high $K_{ow}$ are expected to have an effect on HLC by lowering its value. Recorded effects increase in direct proportion with $K_{ow}$ .

11

12 It is worth noting that because of the complex nature of the water matrix the net effect of a  
13 possible combination of the parameters listed above may be more than the simple sum of  
14 individual effects (Staudinger and Roberts, 1996).

#### 15 QSPR PREDICTION OF HENRY'S LAW CONSTANT

16 The prediction of HLC has been reviewed by Schwarzenbach *et al.* (1993), Reinhard and  
17 Drefahl (1999), Mackay *et al.* (2000) and Dearden and Schüürmann (2003). The most  
18 important approaches are:

- 19
- Ratio of water solubility ( $c_w$ ) to vapour pressure (vp);
  - 20 • Estimation using connectivity indices;
  - 21 • Estimation using group and bond contribution methods.

22 The first method for estimating HLC is not strictly a QSAR method as it uses the water  
23 solubility ( $c_w$ ) and vapour pressure (vp). It is not a highly accurate method, but neither is the  
24 measurement of HLC, especially for substances with very high or very low HLC values.  $\text{vp}/c_w$   
25 can be converted to the dimensionless form of HLC (ratio of concentrations in air and water,  
26  $c_a/c_w$ ) or  $K_{aw}$  by the following equation, which is valid at 25 °C:



1  $c_a/c_w = 40.874 \text{ vp}/c_w$

2 Since both water solubility and vapour pressure can be calculated by QSAR methods, then this  
3 approach might in some circumstances be a QSAR based method. The method is limited to  
4 substances of low water solubility ( $< 1.0 \text{ mol/L}$ ). If QSAR calculated values are used for vp  
5 and/or  $c_w$ , then the respective uncertainties must be considered. For miscible compounds or  
6 compounds with water solubility  $> 1 \text{ mol/L}$  the  $\text{vp}/c_w$  method is not valid.

7 The second method is based on a combination of connectivity indices and calculated  
8 polarisability (Nirmalakhandan and Speece, 1988). A relatively narrow range of chemical types  
9 was used to develop the model, so it is not widely applicable. Moreover, Schüürmann and  
10 Rothenbacher [1992] found it to have poor predictive power.

11 Most prediction methods for HLC use a group or bond contribution approach, although some  
12 have used physicochemical properties [Dearden *et al.* 2000]. The group and bond contribution  
13 methods were first used by Hine and Mookerjee [1974], who obtained, for a set of 263 diverse  
14 simple organic chemicals, a standard deviation of 0.41 log unit for the group contribution  
15 method and one of 0.42 for the bond contribution method. Cabani *et al.* [1981] claimed an  
16 improvement in the group contribution method over that of Hine and Mookerjee, whilst Meylan  
17 and Howard [1991] extended the bond contribution method and obtained, for a set of 345  
18 diverse chemicals, a standard error of 0.34 log unit.

## 19 EVAPORATION RATE

20 Evaporation rates generally have an inverse relationship to boiling points, i.e. the higher the  
21 boiling point, the lower the rate of evaporation. Knowledge of the evaporation rate of spills of  
22 volatile liquids can be useful in several respects. If it is known that a spill of a high vapour  
23 pressure liquid will evaporate completely in a short period of time, it may be preferable to  
24 isolate the area and avoid any intervention or clean-up. The evaporation rate also controls the  
25 atmospheric concentration of the vapour and hence the threat of explosion or fire. Data on the  
26 volatility properties of the liquid, its temperature, the wind speed, and the spill dimensions are  
27 used to calculate the evaporation rate and hence the fraction evaporated at any time.

28 The substance's tendency to partition into the atmosphere is controlled by the vapour  
29 pressure, which is essentially the maximum vapour pressure that a pure substance can exert  
30 in the atmosphere. This can be viewed as a kind of *solubility* of the chemical in the  
31 atmosphere. Using the ideal gas law ( $PV=nRT$ ), the vapour pressure  $P$  in the pressure unit  
32 pascal (Pa) can be converted into a solubility ( $\text{mol/m}^3$ ), where the gas constant  $R$  is  $8.314$   
33  $\text{Pa}\cdot\text{m}^3/\text{mol}\cdot\text{K}$  and  $T$  is absolute temperature (K).

34 Conversion from vapour pressure into concentration in air under ambient temperature:

35  $\% \text{ volume} = \text{vapour pressure (Pa)} / 101\,325 \times 100$

36  $\text{or ppm} = \text{vapour pressure (Pa)} / 101\,325 \times 1\,000\,000$

37 Since the molar volume is the same for all ideal gases (equal volumes of all gases under the  
38 same conditions of temperature and pressure contain the same number of molecule)  $\text{ppm} \equiv$   
39 volume (i.e.  $\text{ml/m}^3$ ). To convert to weight per unit volume:

40  $X \text{ ppm} = X \times \text{MW} / 24.041 \text{ mg/m}^3, 1 \text{ mg/m}^3 = 24.041 / \text{MW ppm}$

41 In the formulation of paints and related products, solvents are chosen based on their  
42 evaporation characteristics appropriate to the application technique and the curing  
43 temperature. To a large extent the evaporation rate of a solvent determines where and how it  
44 can be used. In determining the evaporation rate of solvents, n-butyl acetate is used as the  
45 standard and is assigned an evaporation rate value of 1. Other solvents are assigned  
46 evaporation rate values that indicate how fast they evaporate in relation to n-butyl acetate.  
47 For instance, a solvent that evaporates three times as fast as n-butyl acetate would be

assigned a value of 3, whereas a solvent that evaporates half as fast as n-butyl acetate would be assigned a value of 0.5.

The rate of evaporation is determined using ASTM D3539-87. A known volume of liquid is spread on a known area of filter paper that is suspended from a sensitive balance in a cabinet. Dry air or nitrogen at 25 °C is passed through the cabinet at a known rate. The loss of weight is determined and plotted against time.

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