Brussels, 4 October 2010

Dear Madam,
Dear Sir,

REACH GLASS DOSSIER

Please find attached a report written by three independent experts on the exemption from registration of glass. Also attached are two papers by the International Commission on Glass TC13 on leaching of selenium from tinted flat glass and of antimony from rolled plate glass, conducted in support of the methodology developed for the glass exemption from REACH.

This report was prepared, in order to create a practical modus operandi that could easily be used by each glass producer to test whether the glass types he manufactures can benefit from the exemption from registration under REACH.

Indeed, in the framework of the REACH Regulation (1907/2006/EC) glass is exempted from registration under Annex V item 11 (Commission Regulation 987/2008/EC of 8 October 2008 amending Regulation 1907/2006/EC).

CPIV, as the European association of glass industries, has mandated three independent experts to study the exemption from registration of glass. This approach has been undertaken in the framework of the philosophy which underpins the REACH Regulation (reversal of the burden of proof) and in agreement with recommendations received from the Commission, ECHA, as well as from several member states consulted.

The experts are:

- **Prof. Dr. med. Helmut GREIM**, Director of the Institute of Toxicology and Environmental Hygiene, Technische Universität München; Chairman of the Scientific Committee on Health and Environmental Risks (SCHER) of the General Directorate Health and Consumer Protection of the European Commission.
- **Prof. Dr. rer. nat. Helmut Schaeffer**, former Managing Director of the Research Association of the German Glass Industry (HVG) and the German Society of Glass Technology (DGG) and former Chairman of the International Commission on Glass (ICG).
- **Dr. Nicola FAVARO**, Responsable of the Environmental and Chemical department of the Italian Glass Institute, Stazione Sperimentale del Vetro (Murano, Italy) and member of the PEG on Waste and Recovered Substances with ECHA.

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REACH DOSSIER

Exemption from registration for glass under REACH regulation n. 1907/2006/EC

Prof. Helmut Greim
Prof. Helmut Schaeffer
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Brussels, 12 November 2009

1) REACH regulation and glass industry

REACH is the EU Regulation on Registration, Evaluation, Authorization and Restriction of Chemicals. It entered into force on 1st June 2007. It streamlines and improves the former legislative framework on chemicals of the European Union (EU).

REACH makes industry responsible for assessing and managing the risks posed by chemicals and for providing appropriate safety information to their users. In parallel, the European Union can take additional measures on highly dangerous substances, where there is a need for complementary action at EU level.

As far as glass is concerned, in June 2008 the EU Commission and the Member States have come to a political compromise defining glass in the following way: “Glass is a state of a substance, rather than a substance as such”. On this basis, glass should be treated as a UVCB substance, which means a substance of “unknown or variable composition, complex reaction products or biological materials”[1].

Based on the previous definition and the specific generic characteristic of inertness of glass, in October 2008, the EU Commission added glass to the list of substances exempted from certain provisions under REACH (ANNEX V of the REACH Regulation)[2], in particular Title II (registration of the substances), Title V (downstream users) and Title VI (evaluation).

However, to be exempted the glass has to fulfil the following requirement:

“The following substances unless they meet the criteria for classification as dangerous according to Directive 67/548/EEC and provided that they do not contain constituents meeting the criteria as dangerous in accordance with Directive 67/548/EEC present in concentrations above the lowest of the applicable concentration limits set out in Directive 1999/45/EC or concentration limit set out in Annex 1 to Directive 67/548/EEC, unless conclusive scientific experimental data show that these constituents are not available throughout the life-cycle of the substance and those data have been ascertained to be adequate and reliable: Glass, ceramic frits”.

The aim of the present document is to provide guidelines to glass manufacturers to check if their glass is exempted according to the previous requirements as stated in Annex V item 11 of the REACH regulation.

Moreover, some real examples of applications of the guidelines are reported at the end of the present document.

[1)] 4th Meeting of the Competent Authorities for the implementation of Regulation (EC) 1907/2006 (REACH) 16-17 June 2008
[2)] Commission Regulation (EC) No 987/2008 of 8 October 2008 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 Annexes IV and V (1)

2) Overview of the glass industry

The glass industry can be divided into the following sub sectors:

- container glass
- flat glass
- continuous filament glass fibre
- domestic glass
- specialty glass
- glass wool

Glass industry raw materials are largely solid inorganic compounds, either naturally occurring minerals or synthetic products. They vary from very coarse materials to finely ground powders. The main raw materials used in the glass production are listed in Table 3.1 of the DRAFT GLASS BREF[3]

Glass is a substance of variable composition, which for simplicity is expressed by convention in terms of oxide of the constituents' elements (SiO_2, Na_2O, CaO, B_2O_3, etc). Although conventionally, glass compositions are expressed as oxides of the different components, glass is a non-crystalline or vitreous inorganic macromolecular structure, which does not contain the chemical components of the different raw materials.

The glass classification is normally made considering the chemical composition. The DRAFT GLASS BREF gives four main categories:

- soda-lime-silica glass
- borosilicate glass
- lead crystal glass
- specialty glass.

The principle glass compositions for each common type of glass are shown below. As stated glass may contain minor constituents but these are normally below 1%. This dossier should not be interpreted as covering glasses where such minor constituents exceed 1% unless supported by test evidence.

<u>Soda-lime-silica glass for container, flat glass and domestic glass</u>

This category includes more than 95 % of the glass produced in Europe. A typical soda-lime-silica glass composition is normally included between the following percentages:

- 71-75 % silicon dioxide (derived mainly from quartz sand)
- 12-16 % sodium oxide (mainly from soda ash)
- 10-15 % calcium oxide (mainly from limestone)
- 0.5-3 % aluminium oxide (mainly from feldspar or oxides of aluminium)

Low levels (normally below 1 % w/w) of other components can be present to impart specific properties to the glass, like colouring dopants (example Cr, Fe, Co, Se), fining dopants (example Sb) reducing dopants (example graphite).

Opal glass (domestic glass or flaconnage) can contain also some percentage points of fluoride or phosphate.

In some compositions a portion of calcium oxide or sodium oxide can be replaced by magnesium oxide or potassium oxide respectively.

<u>Borosilicate glass for continuous filament fibres, glass wool, domestic glass (cookware) and special applications</u>

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[3] table 3.1“Draft Reference Document on Best Available Techniques in the Glass manufacturing” July 2009 (http://eippcb.jrc.es/reference/_download.cfm?twg=gls&file=gls_d2_07-2009.pdf)

The typical composition of borosilicate glass is included between the following percentages:

- 50-80 % silicon dioxide (derived mainly from quartz sand)
- 5-20 % boron trioxide (derived mainly from borax, boric acid or colemanite)
- 16-25 % calcium or magnesium oxide (mainly from limestone or dolomite)
- 0-16 % aluminium oxide (mainly from feldspar or oxides of aluminium)
- 1-15 % sodium oxide or potassium oxide
- Other components (normally below 1 %) can be present to impart specific properties to the glass, like Ti, Fe and fluoride

Lead crystal for domestic glass

A typical composition for lead crystal glass is:

- 54-65 % silicon dioxide
- 25-30 % lead oxide
- 13-15 % sodium or potassium oxide
- Other components (normally below 1 %) can be present to impart specific properties to the glass

Specialty glass

Specialty glass covers a wide variety of glass. It is generally based on a soda-lime-silica composition, borosilicate or aluminosilicate composition, plus others compounds designed to impart specific properties to the glass.

The chemical composition of the main products is reported in Table 2.8 of the DRAFT GLASS BREF[4]

[4)] table 2.8 “Draft Reference Document on Best Available Techniques in the Glass manufacturing” July 2009 (http://eippcb.jrc.es/reference/_download.cfm?twg=gls&file=gls_d2_07-2009.pdf)

3) Interpretation of the exemption text

The text in item 11 of Annex V to the REACH Regulation that excludes glass from the obligation to register is as follow:

"The following substances unless they meet the criteria for classification as dangerous according to Directive 67/548/EEC and provided that they do not contain constituents meeting the criteria as dangerous in accordance with Directive 67/548/EEC present in concentrations above the lowest of the applicable concentration limits set out in Directive 1999/45/EC or concentration limit set out in Annex 1 to Directive 67/548/EEC, unless conclusive scientific experimental data show that these constituents are not available throughout the life-cycle of the substance and those data have been ascertained to be adequate and reliable: Glass, ceramic frits".

The EU Directives on classification, labelling and packaging of substances (Directive 67/548/EEC) and preparations (Directive 1999/45/EC) are being replaced by the EU Regulation 1272/2008/EC (called "CLP Regulation") which implements the "Globally Harmonized System of Classification and Labelling of Chemicals". For the purpose of this document we will consider only Directive 67/548/EEC, as this is the directive mentioned in REACH ANNEX V.

Considering the CPIV interpretation of the exemption and the Partner Expert Group (PEG) draft guidelines for Annex V currently present on the ECHA webpage[5] the glass exemption wording can be interpreted following three steps:

1) glass is not exempted if it (as a substance as such) meets the criteria for classification as dangerous according to Directive 67/548/EEC
2) glass is exempted if it is not dangerous (as substance as such) and it does not contain constituents meeting the criteria as dangerous in accordance with Directive 67/548/EEC in concentrations above the lowest of the applicable concentration limits set out in Directive 1999/45/EC or concentration limit set out in Annex 1 to Directive 67/548/EEC
3) glass is exempted if it is not dangerous (as substance as such) and conclusive scientific experimental data show that the constituents, meeting the criteria as dangerous in accordance with Directive 67/548/EEC present in concentrations above the lowest of the applicable concentration limits set out in Directive 1999/45/EC or concentration limit set out in Annex 1 to Directive 67/548/EEC (see previous point 2), are not available throughout the life-cycle of the substance. Those data have to be ascertained to be adequate and reliable.

It is the responsibility of the producer to prove that his glass is exempted.

[5] http://guidance.echa.europa.eu/guidance4_en.htm

4) Explanation of the items to check

4.1) STEP ONE: Glass is not exempted if it (as a substance as such) does meet the criteria for classification as dangerous according to Directive 67/548/EEC

Based on the last updated version of the Annex 1 of the Directive 67/548/EEC (CLP Regulation) only certain categories of Man Made Vitreous Fibres (MMVF) [6] [7] are listed as a dangerous substance.

> *Assessment step one*
>
> *Those categories of Man Made Vitreous Fibres included in Annex 1 of Directive 67/548/EEC are not exempted and need to be registered. For all the other kinds of glass the following criterion needs to be checked.*

4.2) STEP TWO: Glass is exempted if it does not contain constituents meeting the criteria as dangerous in accordance with Directive 67/548/EEC in concentrations above the lowest of the applicable concentration limits set out in Directive 1999/45/EC or concentration limit set out in Annex 1 to Directive 67/548/EEC

To understand the previous sentence it is important to clarify the meaning of "constituents".

Definition of constituents

- Constituents are not defined in the REACH Regulation. However, a definition can be found in the RIP 3.10 Guidance Document: "Any single species present in a substance that can be characterized by its unique chemical identity".
- Glass is obtained by a mineralogical process, resulting in a chemical network (matrix). Its constituents are closely linked together and are in a specific chemical environment, totally different from the initial state (raw materials), and from that does not occur in simple compounds such as metals or oxides. The toxicological effect of the constituents present in the glass are different from that foreseeable for the raw materials. Under normal conditions, glass never releases metal or oxide as direct dissociation products, but rather dissolved ionic metal compounds as Na^+, K^+, Ca^{2+}, Mg^{2+}, etc or soluble organic complexes[8]

On this basis it is possible to state that:

- no single species present in the glass can be characterized by its unique chemical identity and form;
- no single species present in the glass has maintained the form and the chemical characteristics of the raw materials;
- no single species eventually released from the glass has maintained the form and the chemical characteristics of the raw materials.

Therefore it seems appropriate to interpret "dangerous constituents" in the glass as elements meeting the criteria for classification as dangerous in all their chemical forms according to Directive 67/548/EEC (example "lead compounds with the exception of those specified elsewhere in this annex").

Constituents classified as dangerous according to Annex 1 of Directive 67/548/EEC

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6) CPIV is representing the "Continuous Filament Glass Fibre sector". However, it is not the intention of this dossier to make any representation concerning other categories of Man Made Vitreous Fibres and their regulatory status under REACH, as e.g. mineral wool (glass, wool, stone wool, slag wool) which is represented by EURIMA.

7) "Man-made vitreous (silicate) fibres with random orientation with alkaline oxide and alkali earth oxide ($Na_2O+K_2O+CaO+MgO+BaO$) content greater than 18 % by weight" (cod. 650-016-00-2): 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, amending and repealing Directives 67/548/EEC and 1999/45/EC, and amending Regulation (EC) No 1907/2006)

8) Ahmed, A.A. and Youssof, I.M. Dissolution of lead crystal glass (24% PbO) in aqueous solutions of ethyl and methyl alcohols, Glass Technol. 37(1), 21-28 (1996)

Based on the last updated version of Directive 1999/45/EC and Annex 1 of Directive 67/548/EEC and the previous considerations, the following substances of those normally used to produce glass (see Chapter 2), are classified as dangerous:

Table 1: update classification of substances according to Annex 1 of Directive 67/548/EEC and Directive 1999/45/EC[9]

Substance	Classification	Concentration limit set out in annex 1 Directive 67/548/EEC	concentration limit set out in Directive 1999/45/EC
antimony compounds, with the exception of the tetroxide (Sb_2O_4), pentoxide (Sb_2O_5), trisulphide (Sb_2S_3), pentasulphide (Sb_2S_5) and those specified elsewhere in this Annex	Xn: R20/22 N: R51/53	Xn: R20/22: C ≥ 0.25 %	Xn: C ≥ 1 % N: C ≥ 0.1 %
arsenic compounds, with the exception of those specified elsewhere in this annex	T: R23/25 N: R50-53	T: R23/25, R33: C ≥ 0.2 % Xn: R20/22: 0.1 % ≤ C < 0.2 %	T: C ≥ 0.1 % N: C ≥ 0.1 %
cadmium compounds, with the exception of sulphoselenide (xCdS.yCdSe) reaction mass of cadmium sulphide with zinc sulphide (xCdS.yZnS), reaction mass of cadmium sulphide with mercury sulphide (xCdS.yHgS), and those specified elsewhere in this Annex	Xn: R20/21/22 N: R50-53	Xn: R20/21/22: C ≥ 0.1 %	Xn: C ≥ 1 % N: C ≥ 0.1 %
chromium (VI) compounds, with the exception of barium chromate and of compounds specified elsewhere in this annex	Canc. Cat.2: R49, R43 N: R50-53		Carc. cat 2: C ≥ 0.1 % N: C ≥ 0.1 %
lead compounds with the exception of those specified elsewhere in this annex	Repr. cat. 1: R61 Repr. cat. 3: R62 Xn: R20/22, R33 N: R50-53	Repr. cat. 3: R62: C ≥ 2.5 % Xn: R20/22: C ≥ 1 % R33: C ≥ 0.5 %	Repr. cat 1: C ≥ 0.1 % Xn: C ≥ 1 % N: C ≥ 0.1 %
selenium compounds except cadmium sulphoselenide	T: R23/R25, R33 N: R50-53		T: C ≥ 0.1 % N: C ≥ 0.1 %

Others substances are listed as dangerous compounds in all their forms in Annex 1 of Directive 67/548/EEC (beryllium, mercury, thallium and uranium) but they are not normally present in the glass composition (see Chapter 2) and for this reason they are not taken into consideration in this document.

For constituents not still listed as "general compounds" in Annex 1 of Directive 67/548/EEC, it is responsibility of the producer to evaluate their possible dangerousness according to the criteria set out in Directive 67/548/EEC in concentrations above the lowest of the applicable concentration limits set out in Directive 1999/45/EC. In this case table 1 has to be integrated with those constituents and with the relative concentration limits.

Assessment step two

If all constituents in the glass listed in the previous Table 1 are below the lowest concentration limits set out in Directive 1999/45/EC or Annex 1 of the Directive 67/548/EEC, the glass can be considered as exempted. In this case no further steps are needed.

If the glass has at least one constituent listed in Table 1 above the concentration limit, the requirement is not respected. In this case the next step has to be taken.

9) until ATP 31 of Directives 67/548/EEC and ATP 1 of Regulation No. 1272/2008

4.3) STEP THREE: Glass is exempted if conclusive scientific experimental data show that its constituents, meeting the criteria as dangerous in accordance with Directive 67/548/EEC in concentrations above the lowest of the applicable concentration limits set out in Directive 1999/45/EC or concentration limit set out in Annex 1 to Directive 67/548/EEC, are not available throughout the life-cycle of the substance. Those data have been ascertained to be adequate and reliable.

It is clear that the requirement applies only to the constituents of Table 1, or other constituents that are added for specific purposes and meeting the criteria as dangerous.

There is much evidence that non-soluble glass is inert and does not pose any danger to human health and the environment along the lifecycle:

- Glass is characterized by a continuous and essentially non-crystalline or vitreous inorganic macromolecular structure, which is highly insoluble and inert.
- Glass is also considered as inert and has been granted derogations or exemptions in European legislation (Landfill Directive, Packaging Directive, RoHS Directive…).
- Glass vessels (and most often borosilicate glass vessels) are extensively used in "in vitro" tests, also under REACH, clearly illustrating that glass is one of the most chemically and biologically inert materials known[10].

However, to verify the requirement "constituents are not available throughout the life-cycle of the substance" in a adequate and reliable way, the glass industry needs to apply a unequivocal "pass or fail" test.

Availability throughout the life-cycle

Considering the life-cycle of glass it is possible to identify four main ways in which glass can release substances:

- Release of dust in the workplace
  due to cutting, grinding, etc. especially during the preparation or secondary processing of an article inside the glass industry;
- Release of metals into foods, beverages, cosmetics and drugs
  due to leaching from glass container, tableware or flaconnage in the specific matrices
- Release of metals into the environment;
  due to leaching from windows, car glass, etc in specific environmental matrices, such as water, rain, etc.;
- Release of metals on landfill
  due to leaching of metal from glass after the disposal in landfill

In the table below are reported the main ways of release of substances for some important kinds of glass articles.

	Release of dust in the workplace	**Release of metals into foods, beverage, cosmetics, drugs**	**Release of metals into the environment**	**Release of metals on landfill**
Building glass (windows, solar panels, etc.)	X		X	X
Glazing for automotive	X		X	X
Container glass, tableware and flaconnage	X	X		X
Glass articles for domestic use (tableware, cooking, vases, mirrors, etc)	X			X
Continuous filament glass fibre	X			X

10) Regulation n°440/2008 of 30th of may 2008 laying down tests methods pursuant to REACH regulation

<u>Analysis of each individual way of release</u>

<u>Release of dust in the workplace</u>

Based on currently available information, there are no specific occupational exposure limits for "glass dust" in the workplace (only generic dust exposure levels i.e. "inert dust"). Moreover, all possible exposure sources related to the glass manufacturing process are well known and controlled and well covered by existing regulations. BATs (best available techniques) are defined in the glass manufacturing "IPPC BREF (BAT Reference Document)" since 2001. For instance, for downstream processes, emissions of particulates from "dusty processes" are strongly reduced by cutting, grinding, polishing under liquid or applying a bag filter system.

Compliance with the specific regulations regarding dust in the workplace and dust emissions ensures that availability of dangerous substances is well controlled and far below any potential dangerous situations. For this reason it is not taken into consideration.

<u>Release of metals into food, beverage, cosmetics, drugs</u>

The release of metals from containers, tableware, flaconnage, etc. in these specific matrices is in general very low. The characteristics of inertness of the glass, compared with others materials, are well known. Moreover, there are many specific national regulations that fix specific leaching test and limits in the context of the <u>food contact regulations</u>. The update list of European and national legislation regarding food contact is reported in EU report "References of the European and national legislations working document"[11]. The international standard ISO 7086:2000 defines the test method (Part 1) and the permissible limits (Part 2) to verify the release of lead and cadmium from glass hollowware in contact with food. Lead crystal glass is regulated by a specific European agreement which defines the test methods and limit values for lead leaching from glass tableware into foodstuffs[12]. The reference for drugs and cosmetics is the European Pharmacopoeia[13]

Compliance with those specific regulations ensures that availability of dangerous substances is well controlled and far below any potentially dangerous levels. For this reason it is not necessary to be taken into consideration.

<u>Release of metals into the environment</u>

The release of metals from glass in contact with specific environmental matrices, such as water, rain, etc. (windows, automotive glass, etc.) is irrelevant, considering the very well known inertness of glass and its long life time without loss of weight. As reported in "Annex H - Guidance in testing for environmental impact assessment, treatment evaluation and regulatory compliance aspects example: glass and glass products – M/135"of the report "Evaluation of a horizontal approach to assess the possible release of dangerous substances from construction products in support of requirements from the construction products Directive (tr2)"[14] leaching of metals from common glass types to soil & groundwater can be considered non-critical

In the rare cases where the constituents listed in Table 1 are above the limit concentrations the leaching test performed according to the standard for testing of waste EN 12457-2 is, according the same report[14], the most appropriate.

<u>Release of metals on landfill</u>

[11] European Commission - Health & consumer protection directorate-general, "references of the European and National legislations working document (version updated 28 March 2008)" http://eur-lex.europa.eu

[12] Council of Europe, Partial Agreement in the Social and Public Health Field, "Lead leaching from glass tableware and food stuff – version1", 22.09.2004: http://www.coe.int/t/e/social_cohesion/soc-sp/public_health/food_contact/presentation.asp#TopOfPage

[13] European Pharmacopoeia 6° edition: http://www.edqm.eu/en/Ph_Eur_Reference_Standards-627.html

[14] Energy Research Centre of the Netherlands, "Evaluation of a horizontal approach to assess the possible release of dangerous substances from construction products in support of requirements from the construction products directive (tr2)"; Hans van der Sloot (ECN), Joris Dijkstra (ECN), Ole Hjelmar (DHI), Gerd Spanka (VDZ), Philo Bluyssen (TNO), Sara Giselsson (Boverket); november 2008

Release of metals during the disposal of glass in landfill is the only way that should be taken into consideration for all types of glass (end of life).

Consistent with the position adopted by the Commission (e.g. Toys Directive), the meeting of adequately and reliably set limits of a leaching test is an accepted methodology to demonstrate the non-availability of the constituents.

In the specific case where the constituents listed in Table 1 are above the concentration limits, the most appropriate and applicable leaching test to evaluate the release of constituents in environmental liquid matrices and/or during the disposal in landfill, seems to be the test laid down in Chapter 2.2 “Criteria for landfills for non-hazardous waste” in Council Decision 2003/33/EC. It introduces criteria for acceptance of non-hazardous waste at landfills and leaching thresholds that have been set to ensure that no contamination of the environment occurs.

The limit values for acceptance of a waste as non-hazardous material according to Council Decision 2003/33/EC (L/S = 10 l/kg) are given in Table 2 for the relevant elements used in a glass formulation and meeting the criteria for classification as dangerous in all their chemical forms according to Directive 67/548/EEC (see Table 1).

Table 2: leaching limit values according to Council Decision 2003/33/EC

Element	Leaching limit (mg/kg dry)
As	2
Cd	1
Cr (total)	10
Sb	0.7
Pb	10
Se	0.5

Assessment step three

If the leaching test for the specific constituents listed in Table 1 above and the concentration limits set out in the same table, shows that the concentrations are below the limits set for non-hazardous waste the glass can be considered exempted.

Otherwise it has to be registered.

5 Description of the scientific methodology for testing glass

According to Council Decision 2003/33/EC glass is accepted at landfills without further testing. Therefore, a specific protocol for testing glass in landfill does not exist.

The standard for testing of waste EN 12457-2 appears to be a good starting point for defining a methodology, but needs to be adapted to glass.

Work by TC13 (Environmental Technical Committee) of the ICG (International Commission on Glass)[15] has lead to a new test focusing on glass, which presents a clear protocol, including thresholds for the measured elements.

The work was carried out by ten European labs on two samples, one black soda-lime-silica glass and one glass-ceramic cooktop plate. The TC13 report “Preliminary report on a method to determine the availability of glass constituents with regard to the European regulation Reach” is appended to the present document.

The protocol proposed by the TC13 Committee for a leaching test on glass is reported below.

TC13 protocol

TC13 considers that the following procedure is appropriate to test the glass products put on the market in the EU:

- follow the reduction size procedure for the leaching test according to standard EN 12457-2 or equivalent
- remove pieces less than 0.5 mm by sieving[16]
- put the glass sample in contact with distilled water (20^{o}C, 24 h) , using a liquid/solid ratio = 10 l/kg and agitate
- remove glass sample from the resulting suspension by filtration (filter 0.45µm) or centrifugation
- quantify the elements in the leachate by methods used for trace analysis in water
- compare with the limits given for "non hazardous" waste in the Council Decision 2003/33/EC
- check the procedure with a blank analysis on the used liquid (distilled water)

The Committee considers that this is an appropriate approach for obtaining "conclusive scientific experimental data" and to demonstrate that the relevant glass qualifies for the exemption, in the case of an official request from national authorities.

Typical values for repeatability and reproducibility limits as reported in chapter ”8.4 Summary of the performance characteristics evaluation” of the European Standard EN 12457-2 are:

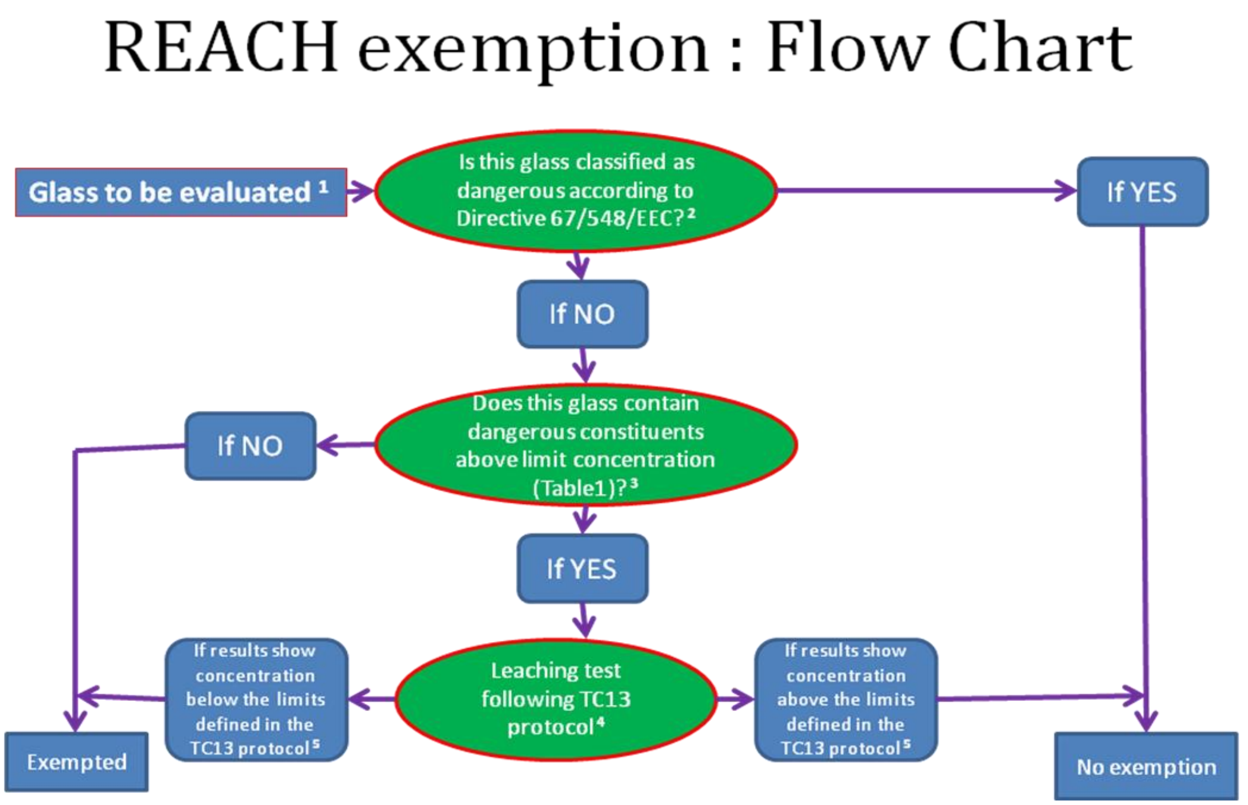
	Typical value	Observed range
Repeatability limit r	40 %	15 % - 95 %
Reproducibility limit R	100 %	65 % - 120 %

6) Decisional flow chart

15) ICG is a non-profit international GLASS SOCIETY consisting of 31 national organisations in glass science and technology. Webpage www.icglass.org

16) The European Standard EN 12457-2 or equivalent specifies in Chapter 4.3.2 “Particle size reduction” that “On no account shall the material be finely ground”. In Annex A is explained that the material shall not be finely ground and plans to limit the particle size reduction.

The following flow chart provides a visualization of the decision steps:



Note about flowchart:

1) Glass to be evaluated.: glass manufactured or imported above 1 tonne per year per legal entity
2) Directive 67/548/EEC: "Directive on the approximation of laws, regulations and administrative provisions relating to the classification, packaging and labelling of dangerous substances"
3) Table 1: updated classification of substances and limit value according to Annex 1 of Directive 67/548/EEC and Directive 1999/45/EC
4) Leaching test following TC13 protocol: reduction size procedure for the leaching test according to standard EN 12457-2 or equivalent; remove pieces less than 0.5 mm by sieving; put the glass sample in contact with distilled water (20 °C, 24 h) , using a liquid/solid ratio = 10 l/kg and agitate; remove glass sample from the resulting suspension by filtration (filter 0.45µm) or centrifugation; quantify the elements in the leachate by methods used for trace analysis in water
5) limits defined in the TC13 protocol: limits given for "nonhazardous" waste in the Council Decision 2003/33/EC

ANNEX 1: EXAMPLES

In the following chapter four examples are given to illustrate the methodology:

- Flat glass for building: clear soda-lime-silica glass
- Container glass for food: coloured (green) soda-lime-silica glass
- Tableware: coloured (black) soda-lime-silica glass
- Speciality glass: Glass-ceramic

The examples should not be taken as evidence for the exclusion of the entire sector. It is the responsibility of the producer to prove that his glass is exempted.

1) Flat glass for building: clear soda-lime-silica glass

Assessment

First step: Soda-lime-silica glass can be classified as "Glass, oxide, chemicals".

The substance is not listed in Annex 1 of the Directive 67/548/EEC. In this case it is necessary to evaluate the following step.

Second step: Based on analytical results obtained from SSV (Stazione Sperimentale del Vetro – Italy) by wet chemical analysis on a single sample, the constituents reported in Table 1 are present in the glass composition in the following concentration:

Constituent	Concentration % w/w	Concentration limits %w/w as set out in Table 1
antimony	< 0.0002	0.25
arsenic	< 0.0002	0.1
cadmium	< 0.00007	0.1
chromium (VI)	< 0.0005	0.1
lead	0.005 ± 0.001	0.5
selenium	< 0.0002	0.1

Beryllium, mercury, thallium and uranium are not present in the composition.

All constituents are below the lower concentration limits set out in Table 1.

The glass can be considered as ***exempted***.

In this case no additional steps are needed.

2) Container glass for food: coloured (green) soda-lime-silica glass

Assessment

First step: Soda-lime-silica glass can be classified as “Glass, oxide, chemicals”.

The substance is not listed in Annex 1 of the Directive 67/548/EEC. In this case it is necessary to evaluate the following step.

Second step: Based on analytical results obtained from SSV (Stazione Sperimentale del Vetro – Italy) by wet chemical analysis on a single sample, the constituents reported in Table 1 are present in the glass composition in the following concentration:

Constituent	Concentration % w/w	Concentration limits %w/w as set out in Table 1
antimony	< 0.0002	0.25
arsenic	< 0.0002	0.1
cadmium	< 0.00007	0.1
chromium (VI)	< 0.0005	0.1
lead	0.021 ± 0.0042	0.5
selenium	< 0.0002	0.1

Beryllium, mercury, thallium and uranium are not present in the composition.

All constituents are below the lower concentration limits set out in Table 1.

The glass can be considered as ***exempted***.

In this case no additional steps are needed.

3) Tableware items made of black soda-lime-silica glass

Assessment

First step: Soda-lime-silica glass can be classified as “Glass, oxide, chemicals”

The substance is not listed in Annex 1 of Directive 67/548/EEC. In this case it is necessary to evaluate the following step.

Second step: Based on the TC13 protocol work, the constituents reported in Table 1 are present in the glass composition in the following concentration:

Constituent	Concentration % w/w*	Concentration limits % w/w (wt %) as set out in Table 1
antimony	< 0.0001	0.25
arsenic	< 0.0001	0.1
cadmium	< 0.0001	0.1
chromium VI	0.25**	0.1
lead	0.02	0.5
selenium	< 0.0001	0.1

* data provided to TC13 by the producer of glass
** total chromium

Beryllium, mercury, thallium and uranium are not present in the composition.

Total chromium is above the concentration limits for chromium VI set out in Table 1. Since it is difficult to distinguish chromium VI and III in the analysis, it is prudent to evaluate the following step.

Third step: The leaching test carried out according to the TC13 protocol for the constituents reported in Table 1 with a concentration above the limits defined in the same table, shows the following result:

Constituent	Concentration mg/kg	limit value for non-hazardous waste mg/kg
chromium	0.06 ± 0.003*	10

* average value obtained from the nine labs involved in the TC13 work

All elements are below the limit values set out in Chapter 2.2 “Criteria for landfills for non-hazardous waste” in Council Decision 2003/33/EC”.

Therefore, the glass can be considered as ***exempted***.

<u>4) Cooktop plate made of specialty glass (glass-ceramic)</u>

<u>Assessment</u>

First step: Glass ceramic can be classified as “Glass, oxide, chemicals”

The substance is not listed in Annex 1 of Directive 67/548/EEC. In this case it is necessary to evaluate the following step.

Second step: Based on the TC13 protocol work, the constituents reported in Table 1 are present in the glass composition in the following concentration:

Constituent	Concentration % w/w*	Concentration limits % w/w (wt %) as set out in Table 1
antimony	< 0.003	0.25
arsenic	< 0.008	0.1
chromium VI	< 0.004**	0.1
lead	< 0.005	0.5

* data provided to TC13 by the producer of glass
** total chromium

Beryllium, mercury, thallium and uranium are not present in the composition.

All constituents are below the lower concentration limits set out in Table 1.

The glass can be considered as ***exempted***.

In this case no additional steps are needed.

Note to the document

The document was based on the information and the legislations in force during the drawing up of the document. It should be updated in case of introduction of new interpretations or legislations, particularly regarding the EU Directives on classification, labelling and packaging of substances (Directive 67/548/EEC) and preparations (Directive 1999/45/EC).

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TECHNICAL ANNEX

International Commission on Glass – Technical Committee 13 on Environment

PRELIMINARY REPORT ON A METHOD TO DETERMINE THE AVAILABILITY OF GLASS CONSTITUENTS WITH REGARD TO THE EUROPEAN REGULATION REACH

Abstract

Abstract

For the application of REACH, glass has the status of a substance, and is therefore subject to the requirements of this new European Regulation. However, glass has been exempted from registration even if it contains dangerous constituents, when conclusive scientific experimental data show that these constituents are not available throughout the life-cycle of the substance. This criterion has been interpreted in the glass industry as equivalent to fulfilling the rules for acceptance for landfilling as a non-hazardous waste. The current standard EN 12457-2 or equivalent is appropriate to test the criterion for this purpose, but it requires clarification for an adequate application of the procedure to glass. The TC13 has performed a detailed investigation on two glass samples, the first representing (in terms of containing constituents of concern) a worse than typical soda-lime formulation (black glass with atypically high levels of transition metals), and the other a typical special glass (glass ceramics). The results obtained by ten different laboratories lead to practical recommendations for the application of the standard to sample preparation for the glass under test. It is observed that the release of metals is very small compared to concentrations in the glass (for example, the ratio of the amount of lead (Pb) released in the leachate to the initial concentration is less than 100 $x10^{-6}$). The two glass samples comply with the proposed criterion based on Council Decision 2003/33/EC which covers the criteria for acceptance to landfill.

1°/ Context

The EC Regulation No 1907/2006 on chemicals and their safe use, known as REACH (Registration, Evaluation, Authorisation and Restriction of Chemicals), aims to improve the protection of human health and the environment and to improve the safe handling and use of substances across all sectors of industry, including the glass industry and downstream users. This law has been in force since the 1st of June 2007.

It has been decided by Member States in the Competent Authorities Committee[17] dealing with REACH that glass is similar to a UVCB substance (Chemical Substance of Unknown or Variable Composition) and is therefore subject to the REACH Regulation. However, Article 2(7)(b) in combination with Annex V of the REACH Regulation exempts certain substances from registration, downstream user obligations and evaluation requirements. In Commission regulation N°987/2008, an exemption under this Annex V is given to glass (and ceramic frits) *"unless they meet the criteria for classification as dangerous according to Directive 67/548/EEC and provided that they do not contain constituents meeting the criteria as dangerous in accordance with Directive 67/548/EEC present in concentrations above the lowest of the applicable concentration limits set out in Directive 1999/45/EC or concentration limit set out in Annex I to Directive 67/548/EEC, unless conclusive scientific experimental data show that these constituents are not available throughout the life-cycle of the substance and those data have been ascertained to be adequate and reliable"*. In this text, the concepts of "dangerous constituents" and of "availability throughout the lifecycle" are introduced, but not defined. The European Chemicals Agency (ECHA) is preparing guidance with the intention to give more clarification for when an exemption could or could not be applied. But again, at least in the current version of the draft document, no explanation of "availability" has been given.

The responsibility to demonstrate compliance is put on the supplier/producer of the substances. The glass industry therefore needs to have timely and clear indications of the status of different glass types with respect to the REACH exemptions.

The Standing Committee of the European Glass Industries (CPIV in Brussels), representing the large majority of the European glass industry and national glass federations has developed its own interpretation of the exemption text [CPIV Position Paper on the interpretation of the Commission's proposal to include certain glasses in Annex V of REACH , July 2008)].

– *CPIV interprets that "dangerous constituents" in the glass are elements meeting the criteria for classification as dangerous in all their chemical forms according to Directive 67/548/EEC*

The CPIV Position Paper also states:

*Regarding the concept of "availability throughout the lifecycle", CPIV fully shares the preoccupation of the legislator not to exempt glasses that might during their lifetime release dangerous substances in quantities that could endanger human health or the environment. Because the lifecycle can be deemed to include the disposal of the product, leaching tests on waste glass may be an appropriate way to prove that no significant leaching occurs. Particularly, CPIV suggests referring to the **"Criteria for landfills for non-hazardous waste"** in Chapter 2.2 of the Council Decision 2003/33/EC as the criteria for the "availability throughout the lifecycle".*

This interpretation has been shared with the EU Commission and to date no adverse comment/response or arguments have been received.

[17] *"Glass is the state of a substance rather than a substance as such. For legislative purposes, it can best be defined through its starting materials and production process, similar to many other UVCB substances*" in Doc: CA/24/2008 rev.3 Follow-up to 5th Meeting of the Competent Authorities for the implementation of Regulation (EC) 1907/2006 (REACH) 25-26 September 2008

The expert group within ICG, TC13 (Technical Committee on Environment of the International Commission of Glass) has the mission to exchange information on reducing the environmental impact of the glass industry and to determine the best practice for measuring the main pollutants released during the glass manufacturing process. Given the importance of REACH TC13 decided to review the situation.

2°/ Quick Review of existing standards

Council Decision 2003/33/EC [*COUNCIL DECISION of 19 December 2002 establishing criteria and procedures for the acceptance of waste at landfills pursuant to Article 16 of and Annex II to Directive 1999/31/EC*] introduces criteria for the acceptance of non-hazardous waste at landfill sites and sets leaching thresholds for individual elements of chemical compounds to ensure that no contamination of the environment occurs. These are based on the application of leaching tests in water, with a liquid to solid ratio (L/S) of 10 l/kg or 2 l/kg, with particles (cullet) sizes reduced to below 4 mm (standard EN 12457/1 and /2) or below 10 mm (standard EN 12457/3 and /4).

These standards were reviewed. In the four standards, the test conditions are very severe compared to real life. In testing, all the material is broken into small pieces, has intimate contact with water and undergoes agitation giving further abrasion. The procedure used is intended to represent a worse case rather than to accurately represent the landfill situation.

Based on the experience of the TC13 members, it quickly appeared that there were specific issues for glass with the application of the leaching test. In the standard certain potentially significant parameters were missing:

- Crushing conditions,
- Sieving method,
- Agitation method conditions during the leaching stage,
- Filtration method,
- Analytical technique(s) for the leachate,

A particularly critical issue appears to be the particle size.

The procedure requires the technician to reduce the (glass) particles size to less than 4 mm and to pass them on a sieve with 4 mm openings in the mesh. But the standard EN 12457-2 states also that *"on no account shall the material be finely ground"*.
These two requirements seem contradictory for a material like glass. Due to its intrinsic, brittle nature, the crushing of glass samples results in inhomogeneous fragmentation, with many very fine, powdery fragments in the sample. The committee realised that, due to this phenomenon, the current standard technique for the leaching assessment, EN 12457-2 might give erroneously high (and in practice, unrealistic) values, especially when placed in the context of the conditions in the environment for which the test was developed.

3°/ Testing schedule

The committee agreed that an urgent Round Robin assessment of the technique and an improved version was needed and that it should be tested on appropriate glass compositions. Two glass samples were chosen for the investigation: a soda-lime-silica glass (this category represents more than 90 % of the production of the glass industry in Europe) and a glass ceramics type (representative of a large group of the special glass category). However as will be seen from the descriptions below, the tests have been geared towards those compositions which are likely to contain higher than normal levels of constituents/elements of concern.

a) Tableware items made of black soda-lime glass

A soda-lime-silica glass type with black coloration was chosen, as it represents the case with the highest level of transition metals in the formulation. The composition of the studied sample was determined by X-ray Fluorescence (XRF) analysis.
The results were obtained on cullet from annealed (stress free) samples.

Table 1: Relevant metal content of the black soda-lime glass

Element	Analysis in mass-%
As	< 0.0001 %
Ba	0.08 %
Co	0.03 %
Cu	0.03 %
Cr (total)	0.25 %
Mo	< 0.0001 %
Mn	3.90 %
Ni	0.03%
Pb	0.02 %
Sb	< 0.0001 %
Zn	0.01 %

b) Glass-ceramic tabletop plates

The glass ceramic type studied here belongs to the glass system Li_2O-Al_2O_3-SiO_2 in which the SiO_2- mass concentration can reach up to 70% and the Al_2O_3 content up to 20%.

Table 2: Relevant metal content of the glass-ceramic type

Element	Analysis in mass-%
As	< 0.008 %
Ba	2.3 %
Cu	< 0.004 %
Cr (total)	< 0.004 %
Mo	< 0.004 %
Ni	< 0.004 %
Pb	< 0.005 %
Sb	< 0.003 %
Zn	0.01 %

For these two types of glass, the trial was performed by ten laboratories, following the standard EN 12457-2, except that two samples were prepared: one with the glass crushed to sizes less than 4 mm and containing all grain sizes below 4 mm, and a second with the glass crushed to less than 4 mm and with pieces less 0.5 mm removed by consecutive sieving.

The concentrations of the relevant metals in the leachates were analysed.

Table 3: List of laboratories and experts involved in the study

AGC Flat Glass Europe	Anne Rasneur	Moustier s/Sambre, Belgium
Arc International	Etienne Sénéchal	Arques, France
GTS / BGMC	John Stockdale	Sheffield , UK
Pilkington Technical Centre	Simon Slade	Ormskirk, UK
Saint-Gobain Recherche	Maria Malheiro	Aubervilliers, France
Saint-Gobain Sekurit	Dr. Andreas Kasper	Herzogenrath , Germany
Schott AG	Ralf Eiden	Mainz , Germany
Şişecam	Dilek Bolcan	Istanbul , Turkey
Stazione Sperimentale del Vetro	Nicola Favaro	Murano , Italia
TNO Glass Group	Hans van Limpt	Eindhoven, The Netherlands

Note: some of the participants involved are also members of ICG TC2 (Technical Committee of the ICG on: Analysis and durability of glasses).

4°/ Operating conditions

Table 4: review of the operating conditions used in the different laboratories

			A	B	C	D	E	F	G	H	I	J
1	Test Method	EN 12457 Part 2	X	X	X	X	X	X	X	X	X	X
2	Crushing device	jaw crusher		X		X				X		
		cutting device										
		mortar with hammer			X		X		X		X	
		grinding mill										X
		Steel mortar						X				
		other	X									
3	Sieving method	manual	X	X	X	X	X		X	X	X	X
		automatic device						X				
4	Agitation device	and-over-and tumbler	X			X		X		X	X	
		roller-table		X	X		X					X
		horizontal tumbler							X			
		other										
5	Filtration device	filter 0.45µm	X	X	X	X	X	X		X	X	
		centrifuging										X
		solid-liquid separation										
6	Method of analysis	ENV 12506		X							X	
		ENV 13370		X								
		ISO 11885	X							X		
		other			X	X	X	X				X
7	Analytical technique	ICP-OES	X	X		X	X	X	X	X	X	
		ICP-MS		X	X							X
		GF-AAS									X	
		AAS			X							
		HG-AAS									X	
		Ionic Chromatography					X					
		HG-CVAAS							X	X		
8	Blank Analysis	yes	X	X	X	X	X	X	X	X	X	X
		no										

Note:
ICP-OES: inductively coupled plasma optical emission spectrometry
ICP-MS: inductively coupled plasma-mass spectrometry
GF-AAS: graphite furnace atomic absorption spectrometry
AAS: atomic absorption spectrometry
HG-CVAAS: hydride generation cold vapour atomic absorption spectrometry

5°/ Results

The complete data file is given in the annex.

Some of the main results are extracted and commented on below.

A general observation is that the range of results tends to be rather high.
This is in line with the description in the standard, which mentions a limit of reproducibility of 72% (with a range from 20 to 160%).

In most cases, the leaching values are very low; below the detection limits usually used in the laboratories concerned. These detection limits are dependent on the analytical procedure chosen by the different laboratories. This prevents us from performing a detailed statistical analysis in every case.

5.1/ Leaching Test – **Lead** (Pb) (mg/kg) for black soda-lime-silica glass

Lead is not included in the formulation of this glass, but it was decided to analyse for the element, as it might be a possible contaminant.
The analyzed initial concentration in the glass was circa 0.02 % = 200 mg/kg

Table 5: results of leaching test from black soda-lime sample, Pb release in mg/kg of dry mass

Laboratory	sample with size distribution below 4 mm	sample with size distribution between 0.5 and 4 mm
A	<0.1	<0.1
B	<0.1	<0.1
C	0.009	0.015
D	<0.1	<0.1
E	<1	<1
F	<0.01	<0.01
G	<0.1	<0.1
H	<0.5	<0.5
I	< 0.01	< 0.01
J	0.004	<0.001

There is no disagreement between the results of the different laboratories.

There are no major differences in the results between those obtained with or without the small particle size below 0.5 mm.

In all cases, the leaching level is very low: the ratio between the amount of lead released in the leachate and the initial concentration is in the range (0.5 to 5) x10^{-6}.

5.2/ Leaching Test – **Barium (Ba)** (mg/kg) for black soda-lime-silica glass

The analyzed initial concentration in the glass was circa 0.08 % = 800 mg/kg

Table 6: results of leaching test from black soda-lime sample, Ba release in mg/kg of dry mass

Laboratory	sample with size distribution below 4 mm	sample with size distribution between 0.5 and 4 mm
A	0.2	<0.1
B	<1	<1
C	0.07	0.05
D	<0.1	<0.1
E	0.6	0.35
G	0.06	0.03
H	0.2	0.17
I	< 0.2	< 0.2
J	0.19	0.041

The exclusion of the fine particles gives a lower range of results.
In all cases, the leaching level is very low: the ratio between the amount of Barium released in the leachate and the initial concentration is in the range (4 to 40) $x10^{-5}$

5.3/ Leaching Test – **Zinc (Zn)** (mg/kg) for Glass ceramics

The inclusion of the fine particulates gives at least one result which deviates significantly from the others. Even without considering this outlier, the range of the results obtained is relatively high.

5.4/ Leaching Test – **Barium (Ba)** (mg/kg) for Glass ceramics

The analyzed initial concentration in the glass was circa 2.3 % = 23 000 mg/kg

Table 7: results of leaching test from the glass-ceramic sample, Ba release in mg/kg in dry mass

Laboratory	sample with size distribution below 4 mm	sample with size distribution between 0.5 and 4 mm
A	0.4	<0.1
B	1.27	<1
C	1.5	0.69
D	<0.1	<0.1
E	1.1	1.25
F	0.03	0.04
G	0.53	0.09
H	2.7	1.0
I	< 0.2	< 0.2

In all cases, the leaching level is very low: the ratio between the amount of barium released in the leachate and the initial concentration is in the range of 2 to 54 $x10^{-6}$.

6°/ Conclusions

– The leaching test described in the standard EN 12457-2 is generally appropriate for investigating the criteria of the "non availability" of glass constituents, provided that the following condition is fulfilled:

  - the fine particles below 0.5 mm should be removed before contact with the distilled water. This is required to avoid the risk of results which are inconsistent and unrepresentative of the conditions for which the test is being carried out. It is in line with the statement in the standard EN 12457-2: "*on no account shall the material be finely ground*".

Others options in the standard methodology e.g. different methods of crushing and agitation or chemical analysis of the solution) do not seem to have a significant impact. However closer investigations of these conditions has still to be done.

– The observed releases of relevant metals are very small compared to the concentrations of the metals in the original glass samples; less than 0.01% of the metallic elements contained in the glass were released. This is seen as clear evidence of the "non availability" of glass constituents at the critical step of the "end of life" of the glass products investigated in this study.

7°/ Recommended methodology

TC13 consider that the following procedure is appropriate to test the glass products put on the market in the EU:

- follow the reduction size procedure for the leaching test according to standard EN 12457-2 or equivalent
- remove particles less than 0.5mm by sieving
- put the glass sample in contact with distilled water, using a liquid/solid ratio = 10 l/kg and agitate for 24hrs +or- 0.5 hrs at 20^{o}C +or- 5^{o}C
- remove the glass sample from the resulting suspension by filtration (e.g. filter 0.45µm) or centrifugation
- quantify the elements in the leachate by methods used for trace analysis in water
- compare with the limits given in Council Decision 2003/33/EC on landfill

The limit values for acceptance of a waste as non-hazardous material according to Council Decision 2003/33/EC (L/S = 10 l/kg) are given in table 8 for the relevant elements used in the glass formulations and meeting the criteria for classification as dangerous in all their chemical forms according to Directive 67/548/EEC.

Table 8: some leaching limit values according to Council Decision 2003/33/EC

Element	Leaching limit (mg/kg dry)
As	2
Cd	1
Cr (total)	10
Pb	10
Se	0.5

The Committee considers that this is an appropriate approach for obtaining "*conclusive scientific experimental data"* and to demonstrate that the relevant glass qualifies for the exemption, in the case of an official request from national authorities or any other body.

Current list of TC 13 members:

- Mr Hugues Abensour, Saint-Gobain Conceptions Verrières (France)
- Prof. Dr.Ir. Ruud G. C. Beerkens, TNO Science and Industry (Netherlands)
- Mr Lucien Belmonte, Abividro (Brazil)
- Ing. Petr Beranek, Glass Service Inc. (Czech Republic)
- Mrs Dilek Bolcan, Şişecam (Turkey)
- Mr Nicola Favaro, Stazione Sperimentale del Vetro (Italy)
- Mr Karlheinz Gitzhofer, HVG (Germany)
- Dr Thomas Huenlich, Schott AG (Germany)
- Dr Andreas Kasper, Saint-Gobain Sekurit (Germany)
- Mr Denis Lalart, Arc International (France)
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- Mr C. Philip Ross, GICI (USA)
- Mr Etienne Sénéchal, Arc International (France)
- Dr Simon Slade, NSG (UK)
- Mr John Stockdale, British Glass Manufacturers' Confederation (UK)
- Mr H. van Limpt, TNO Science and Industry (The Netherlands)
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ANNEX part 1

Black Soda Lime Glass
Particle Size 0 to 4 mm

	Laboratory									
	A	B	C	D	E	F	G	H	I	J
As (mg/kg)	<0.1	<0.5	<0.0075	<0.1	<0.5		<0.1	<0.1	0.018	0.001
Ba (mg/kg)	0.2	<1	0.07	<0.1	0.6		0.06	0.02	< 0.2	0.19
Cd (mg/kg)	<0.1	<0.02	<0.01	<0.1	<0.1	<0.01	<0.01	<0.1	< 0.01	<0.001
Cr (total) (mg/kg)	0.1	<0.1	0.14	<0.1	<0.1	<0.06	0.16	0.016		0.09
Cu (mg/kg)	<0.1	<0.1	0.21	<0.1	<0.1	<0.07	<0.2	<0.1	< 0.2	0.014
Hg (mg/kg)	<0.1	<0.005	<0.0001	<0.1	<0.2		< 0.005		< 0.005	<0.0003
Mo (mg/kg)	<0.1	<0.5	<0.01	<0.1	<0.2		<0.05	<0.1	< 0.05	<0.01
Ni (mg/kg)	<0.1	<0.1	0.023	<0.1	<0.5	<0.06	<0.02	0.030	< 0.01	0.011
Pb (mg/kg)	<0.1	<0.1	0.009	<0.1	<1	<0.01	<0.1	<0.5	< 0.01	0.004
Sb (mg/kg)	<0.1	<0.01	<0.01	<0.1	<0.2		<0.2		0.053	
Se (mg/kg)	<0.1	<0.05	<0.01	<0.1	<1		<0.2		< 0.02	
Zn (mg/kg)	<0.1	0.130	<0.05	<0.1	<0.05	0.13	<0.1	<0.2	< 0.2	<0.01
pH	9.8	9.9	10.2		8.9		6.5	9.5		

ANNEX part 2

Black Soda Lime Glass
Particle Size 0.5 to 4 mm

	Laboratory									
	A	B	C	D	E	F	G	H	I	J
As (mg/kg)	<0.1	<0.5	<0.0075	<0.1	<0.5		<0.1	<0.1	0.015	< 0.001
Ba (mg/kg)	<0.1	<1	0.05	<0.1	0.350		0.03	0.017	< 0.2	0.04
Cd (mg/kg)	<0.1	<0.02	<0.01	<0.1	<0.1	<0.01	<0.01	<0.1	< 0.01	<0.001
Cr (total) (mg/kg)	<0.1	0.13	0.14	<0.1	<0.1	<0.06	0.07	0.02		0.02
Cu (mg/kg)	<0.1	<0.1	0.15	<0.1	<0.1	<0.07	<0.2	<0.1	< 0.2	0.006
Hg (mg/kg)	<0.1	<0.005	<0.0001	<0.1	<0.2		< 0.005		< 0.005	<0.00025
Mo (mg/kg)	<0.1	<0.5	<0.01	<0.1	<0.2		<0.05	<0.1	< 0.05	<0.001
Ni (mg/kg)	<0.1	<0.1	0.02	<0.1	<0.5	<0.06	<0.02	0.01	< 0.01	0.004
Pb (mg/kg)	<0.1	<0.1	0.015	<0.1	<1	<0.01	<0.1	<0.5.	< 0.01	<0.001
Sb (mg/kg)	<0.1	<0.01	<0.01	<0.1	<0.2		<0.2		0.029	
Se (mg/kg)	<0.1	<0.05	<0.01	<0.1	<1		<0.2		< 0.02	
Zn (mg/kg)	<0.1	<0.1	<0.05	<0.1	<0.05	0.11	<0.1	<0.2	< 0.2	<0.01
pH	9.6	9.5	9.8		8.7		6.3	8.7		

ANNEX part 3

Glass Ceramics
Particle Size 0 to 4 mm

	Laboratory								
	A	B	C	D	E	F	G	H	I
As (mg/kg)	<0.1	<0.5	<0.0075	<0.1	<0.5		<0.1	<0.1	0.005
Ba (mg/kg)	0.4	1.27	1.5	<0.1	1.1	0.034	0.53	2.7	< 0.2
Cd (mg/kg)	<0.1	<0.02	<0.01	<0.1	<0.1	<0.01	<0.01	<0.1	< 0.01
Cr (total (mg/kg)	<0.1	<0.1	<0.01	<0.1	<0.1	<0.15	<0.02	<0.1	
Cu (mg/kg)	<0.1	<0.1	0.62	<0.1	<0.1	<0.1	<0.2	0.2	< 0.2
Hg (mg/kg)	<0.1	<0.005	<0.0001	<0.1	<0.2		< 0.005		< 0.005
Mo (mg/kg)	<0.1	<0.5	0.02	<0.1	<0.2		<0.05	<0.1	< 0.05
Ni (mg/kg)	<0.1	<0.1	<0.02	<0.1	<0.5	<0.06	<0.02	<0.1	< 0.01
Pb (mg/kg)	0.1	<0.1	0.02	<0.1	<1	<0.01	<0.1	<0.5	< 0.01
Sb (mg/kg)	<0.1	<0.01	0.09	0.050	<0.2		<0.2		0.033
Se (mg/kg)	<0.1	<0.05	<0.01	<0.1	<1		<0.2		< 0.02
Zn (mg/kg)	<0.1	0.5	0.72	<0.1	15.	<0.05	0.4	1.4	< 0.2
pH	8.5	8.2	8.8		8.5		6.7	4.8	

ANNEX part 4

Glass Ceramics
Size Particle 0.5 to 4 mm

	Laboratory								
	A	B	C	D	E	F	G	H	I
As (mg/kg)	<0.1	<0.5	<0.0075	<0.1	<0.5		<0.1	<0.1	0.003
Ba (mg/kg)	<0.1	<1	0.69	<0.1	1.25	0.04	0.09	1.0	< 0.2
Cd (mg/kg)	<0.1	<0.02	<0.01	<0.1	<0.1	<0.01	<0.01	<1	< 0.01
Cr (total) (mg/kg)	<0.1	<0.1	<0.01	<0.1	<0.1	<0.17	<0.02	<1	
Cu (mg/kg)	<0.1	<0.1	0.2	<0.1	<0.1	<0.07	<0.2	<0.1	< 0.2
Hg (mg/kg)	<0.1	<0.005	<0.0001	<0.1	<0.2		< 0.005		< 0.005
Mo (mg/kg)	<0.1	<0.5	<0.01	<0.1	<0.2		<0.05	<0.1	< 0.05
Ni (mg/kg)	<0.1	<0.1	<0.015	<0.1	<0.5	<0.06	<0.02	0.2	< 0.01
Pb (mg/kg)	<0.1	<0.1	0.008	<0.1	<1	<0.01	<0.1	<0.5	< 0.01
Sb (mg/kg)	<0.1	<0.01	0.02	<0.1	<0.2		<0.2		0.028
Se (mg/kg)	<0.1	<0.05	<0.01	<0.1	<1		<0.2		< 0.02
Zn (mg/kg)	<0.1	0.450	0.36	<0.1	<0.05	<0.05	0.2	0.6	< 0.2
pH	8.5	7.0	8.5		7.6		6.5	6.5	