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[1] 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

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[1] *"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

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.

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 $x10^{-6}$.

5°/ 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.

6°/ 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)
- Mr Gyorgy Liptak, GE Consuler and Industrial (Hungary)
- 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)
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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	