

FINAL REPORT

“Investigation Of The Significant Factors In Elemental Migration From Glass In Contact With Food”

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1 Summary

The aim of the work presented in this report was to develop a comprehensive overview of the potential for elemental migration from the different glass types used in food contact applications in a range of conditions of use. Whilst there is a large body of historical research on glass durability and migration, there was a perceived need to update this data with further experimentation, to account for modern day manufacturing processes and the influences of factors such as increased rates of recycling.

It was decided however that significant work had been undertaken in recent years specifically on the topic of lead migration from lead crystal ware. As a result, this study was not targeted to carry out detailed experimental investigations on this specific aspect of glass elemental migration.

The work carried out has shown that generally glass is an inert food contact material with limited potential for migration of elements of toxic significance in the compositions commonly used. The most durable compositions were found to be borosilicate ("pyrex" type) glass and glass ceramics which are widely used in cookware/oven to table ware. Where elemental release did occur it was generally at ppm or sub ppm level and limited to the major glass making elements when tested with a range of solutions under a variety of test conditions.

One area of potential interest, however, was the relative lack of inertness (when tested using acetic acid) of some ceramic/inorganic decorations used on glass that had the potential to be in contact with food. As many foodstuffs are acidic this may warrant further investigation.

2 Introduction

The work undertaken in this study consisted of a programme of experimental work to increase understanding of migration issues related to glass materials in contact with foodstuffs. Although there was data available on most commonly used glass types, most of this work was historical and only lead migration from lead crystal had been given extensive attention from the scientific community over recent years. As manufacturing methods have developed with time, and issues such as greater recycling rates and higher rates of imported glassware occur it was felt necessary to update this data.

Some useful aids for the reader included in the appendices at the rear of this report are:

- A descriptive list of the sample types collected and their identification numbers and sample codes used (which appears in the results tables and figures presented).
- A glossary of commonly used terms.
- A list of the analytical reporting limits used throughout this report for the elements of interest.
- An indication of the degree of error / uncertainty associated with the data.
- A list of current drinking water concentration limits for a range of elements for comparative purposes to the analytical data.

2.1 Scientific Objectives

The primary scientific objective of the work programme was to determine which, if any, are the significant leachable elements from the range of glass compositions in common use for food contact purposes and define under what conditions this leaching is most likely to occur. The conditions used, wherever possible, simulated likely, real conditions of use that the material may experience, as well as simulating extremes of conditions of use.

Although glass is generally considered a relatively chemically inert material, it is used in a wide range of applications and several, distinctly different, chemical compositions are in contact with a number of foodstuffs and beverages under different conditions. Further, certain types of glassware are often treated, coated, washed, surface damaged, cooked in, microwaved in and pasteurised in during their normal life cycle. These variables are also complicated by the widely differing rates of recycling used in different countries.

In short the scientific objectives were to:

- Provide quantified information on the migration behaviour of common glass compositions in simulated food contact situations and to develop a testing regime to assess the effects of processing, handling and final application on elemental migration.
- Identify elements of possible toxicological interest in glass and determine at what proportion, if any, of the overall bulk composition they led to measurable migration using common analytical techniques. (ICP-OES, AAS FES).

2.2 Scientific Approach

The approach adopted in the study was relatively simple. Initially it was necessary to identify what types of glassware were in use in the UK for food contact application and in what proportion each type of glassware was used. Once this was established it was necessary to collect a wide range of samples to ensure sufficient testing of all key glass compositions in conditions that would simulate common applications. The experiments would focus most effort on the most widely used applications.

Accelerated migration testing would be used to test commercially available glassware to establish a “baseline” behaviour for that particular type of ware. Further testing would then be carried out under a range of conditions (for example after surface damage, during microwaving etc.) to establish their effect relative to “baseline” migration behaviour of the articles. For this reason it was necessary to have several specimens of each type of glassware to be tested. Testing would involve the use of food simulants and some real foodstuffs.

A further investigative step would look at the ability of glass as a material to retain elements of possible toxicological interest, without releasing them in to the test solution. For this stage of work “spiked” melts of the common glass compositions would be developed to determine the level of interest at which elements were released.

The elements analysed for in the range of test solutions were as follows:-

For Soda Lime Silica Glasses (all colours):

- Na, Si, Ca, Mg, K, Al, Cr, Mn, Co, Cu, Fe, Zn, Se, Ni, Zr, Ti, V, Pb, Cd, Bi

For Borosilicate Glasses

- Na, Si, Ca, B, Mg, K, Cr, Mn, Co, Cu, Fe, Ni, Zr, V, Ba

For Lead Crystal Glasses

- Na, Si, B, Pb, K, Ba, Sr, Bi, Cr, Fe, Ni, Zr, As, Sb, V, Ca, Ba

For Glass Ceramic Glasses

- Si, K, Al, Zr, Na, Mg, Li, Ti, Cr, Mn, Co, Cu, Fe, Zn, Se, Ni, V, Ba

For Decorated Glassware

- Na, K, Si, Pb, Cd, Cr, Fe, Ni, Co, Cu, Zn, Mg, Al, Ca

These elements were selected as they are common glass constituents, colourants or potential contaminants.

Analysis of test solutions was carried out by ICP-OES analysis with reporting limits ranging from 50ppb to 100ppb (0.05 to 0.1ppm) for most elements. Na and K were analysed using AAS, FES with a reporting limit of approx 10ppb to 20ppb (0.01-0.02ppm) as the sensitivity of AAS, FES for Na and K analysis is greater than ICP-OES for these elements.

3 Review Of Literature On Elemental Migration From Glass

3.1 General Glass Migration / Durability

A striking feature of the literature available on the subject of migration of elements from glass ware is that a large body of the published work was actually produced in the 1940's and only a limited number of studies appear to have been undertaken since then. The early leaders in this field were A.K.Lyle ⁽¹⁾, F.R.Bacon and O.G.Burch ^(2,3) who discussed the theoretical aspects of chemical attack on glasses, and in particular the role of time and temperature in accelerating this attack.

Work by the British Glass Industry Research Association has been extensive in the area of elemental migration from lead crystal glass ware, and this work has shown that lead extraction becomes more severe with decreasing pH, with a 4% acetic acid solution being a particularly effective test medium. In simulant migration tests on silicate glass compositions, water has in general been found to be a more effective medium, resulting in a higher level of elemental migration. In one case a migration test comparing 3% acetic acid to water on soda lime silica container glasses showed that water led to approximately 10 times more total elemental extraction than 3% acetic acid at 121⁰C for 60minutes ⁽⁴⁾.

L. Holland in his book "*The Properties of Glass Surfaces*"⁽⁵⁾, describes in detail the effects of alkali, neutral (aqueous) and acidic mediums on silicate glasses, and discusses the findings of a number of researchers in this area. This work has shown that irrespective of attacking medium an Arrhenius equation for the velocity/rate of "attack" can be developed, although the rate of attack does depend on pH and temperature. This is consistent with the findings of Lyle⁽¹⁾ and Bacon and Burch^(2,3). It is generally believed that at <pH9.5, metal ion migration becomes significant, whereas at >pH9.5, attack and breakdown of the silicate network becomes more significant. It is also the case that as temperature increases then so does migration/attack rate.

A particularly useful reference has been BGIRA technical note 195 dated February 1975⁽⁴⁾, in which there is significant discussion on why certain types of migration tests are more suitable than others and which attacking mediums are most relevant to glasses. This technical note also references work on the pH values of food. From a list of 250 foodstuffs and preparations only 15 had pH's above pH7, with the most acidic foodstuffs being listed as lime fruits at pH 1.8-2.0 and the most alkaline being frozen eggs at pH 8.5-9.5⁽⁶⁾

A more recent study of high consumer volume food container glass elemental migration (1990) was carried out by Tingle⁽⁷⁾ in which soda lime silica bottles from eight different UK manufacturers were tested using the ISO 4802 hydrolytic resistance test (121⁰C-1 hour). Both total and individual elemental extraction was presented and compared to typical values obtained from analyses of public authority water supplies. The results showed that even under these severe conditions of test, elemental levels were comparable and in many cases lower than the levels found in the public water supply at that time. Total extraction was also found to be below the overall migration limit of 10mg of solid extract per dm² of contact surface area in the proposed EC directive on plastic materials and articles intended to come in to contact with foodstuffs, as the paper tried to draw a comparison between the two types of material. It should be stated however that glass is very different as a material to plastics and as a result will release chemically different migrants in to test solutions.

Migration tests and theoretical work previously published tend to recommend either:

- Acidic tests, predominantly using 4% acetic acid or
- Accelerated hydrolytic tests using water at elevated temperature (and / or pressure) to simulate prolonged exposure.

Literature has also shown that pH, exposure time and exposure temperature are all significant factors in migration rate and that where food contact is concerned neutral or acidic media are more relevant as test simulants. ^(1,2,3,4,5,6)

In terms of relative exposure it is possible to inter-relate by interpolation different time temperate conditions to derive equivalent exposure under differing conditions.

This is possible because an Arrhenius type relationship can be plotted on a log time versus temperature plot to give a straight line relationship. It is then possible to predict what the equivalent exposure time at one temperature would be relative to a set time at another. Figure 1 shows a graph derived from points given by Bacon and Burch ^(2,3) and Lyle ⁽¹⁾.

For example we know that from published data, attack for one year at room temperature is equivalent to three weeks at 50⁰C, two days at 75⁰C or seven hours at 95⁰C. From this the straight line relationship for figure 1 is derived.

Thus if we wish to know what the relative exposure of for example 2 hours at 85⁰C is, this can be done by simple interpolation. To fit on the line, 85⁰C would give a time equivalence of approximately 1100 mins, to be comparable to a year at 20⁰C. Therefore the equivalence to days at room temperature of a two hour test at this temperature would be:

$$120 \div 1100 = 0.11$$

$$365 \times 0.11 = 40.15$$

Therefore 2 hours at 85⁰C would equate to approximately 40days at 20⁰C

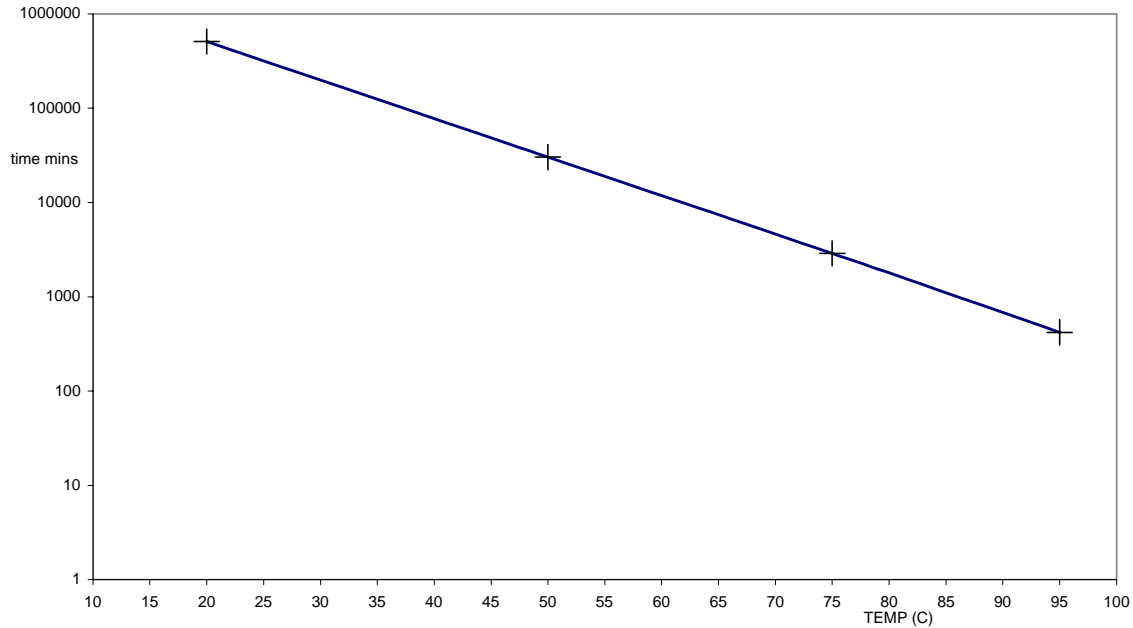


Figure 1: Equivalent Time Temperature Exposure based on Information Derived by A.K. Lyle

3.2 Lead Crystal Migration

It was recognized in the development of the proposal for glass migration studies that over recent years the issue of lead migration from lead crystal glass had been studied in more detail than the other glass compositions in common usage. As a result it was decided that a discussion of Pb migration (from available literature) should be included in the work as this may have some relevance to the work carried out on other glass compositions.

In recent years significant study of lead crystal ware has been undertaken. A recent paper by A A Ahmed and I M Youssoff ⁽⁸⁾ discussed the effects of repeated leaching of commercially available lead crystal glass by acid solutions and water. Lead crystal glass in tumbler and grain form on repeated leaching by acetic, citric, hydrochloric, sulphuric, and nitric acid solutions and de-ionised water at 22⁰C and 95⁰C was studied by solution analysis and weight loss.

This was compared with that obtained during continuous leaching. It was observed that repeated leaching lead to general decrease of Na, K, Pb and Si extraction with increasing number of leach tests with acidic simulants. A reduction of >50% was observed for acidic simulants on the first repeat test for both 1 hour and 6 hour tests at 95⁰C. This was not the pattern observed for water, with migration being similar regardless of number of tests. The quantities of Pb released in an initial 1 hour test at 95⁰C by the different acids could be arranged in the order

Acetic acid>nitric acid=hydrochloric acid=sulphuric acid>citric acid.

with Pb release in the range 0.9 to 1.7ppm. Water extracted much lower amounts of lead under the same conditions (approximately 0.1ppm). On the second leach test for this glass, release was found to drop for acidic simulants to 0.4 to 0.7ppm. When water was used as a simulant release was similar in the first and second test runs (approximately 0.1ppm).

The order of this series changed slightly for a continuous 6 hour test at 95⁰C such that:

hydrochloric acid=nitric acid>sulphuric acid>acetic acid>citric acid

with lead released in the range of 1.5 to 4.5 ppm dependant on simulant. The release into water under similar conditions was approximately 0.5ppm. This testing also found that acidic solutions were generally more effective at causing lead migration than deionised water.

Further papers by A.A Ahmed and I.M Youseff ^(9,10), look at the effect of aqueous solutions of ethanol and methanol on lead crystal glass and the interaction between lead crystal glass and acetic acid. The studies with alcohol solutions were carried out at 75⁰C for between 0.5-6 hours. The release of Pb was found to increase with increasing alcohol solution strength, whilst this simultaneously decreased the release of Na, K and Si. However Pb release was significantly lower (about one tenth) than that of Na, K and Si (<0.5ppm after 6hours exposure at 75⁰C) and also lower than when a water test solution was used.

The relationship :

water>ethanol solution(40% v/v)>methanol solution(40%v/v) was found.

The studies of acetic acid test solutions concluded that for solutions of varying concentrations of acetic acid (0.6, 1, 2, 4, 8 and 12% v/v) at different temperatures (22 to 95⁰C), for different times (30min to 300 days):

- a) Pb²⁺ was released from the glassware tested under a range of standard testing conditions (e.g. ISO 7086, 24 hours 4% acetic acid 21⁰C) in concentrations that were smaller than those specified as being acceptable in the various standards.
- b) The extraction of the cations was severely affected by the form of the sample; glass grains released more cations than bulk glass.
- c) Extraction of Pb²⁺, K⁺ and Na⁺ followed a straight line relationship when plotted versus t^{1/2} (square root of time) for short periods of time up to 6 hours at 95⁰C, or up to 20 days at 22⁰C. Deviation from linearity was observed at greater than 20 days, which it was suggested was related to the leached layer becoming sufficiently thick that cation leaching slowed, such that the leaching and silica network hydrolysis/dissolution proceeded at equal rates.
- d) The extraction of Pb²⁺, K⁺, Si⁴⁺ and Na⁺ was effected by concentration of acetic acid, which was correlated with the corresponding change in pH.
- e) The concentrations of Pb²⁺ released by 4% acetic acid were several orders of magnitude greater than that released by 40% v/v ethanol indicating that the standard 4% acetic acid test is suitable for Pb release safety testing.

A good review of the issues around lead crystal ware for food contact usage was given as an issue paper to the O.E.C.D workshop, Canada 13/14 September 1994 ⁽¹¹⁾ by Dr J Kennedy on behalf of the International Crystal Federation (ICF), Committee Permanent des Industries du Verre (CPIV) and the European Domestic Glass Committee.

In this paper the subject of lead release from lead crystal is covered in some detail: ICF voluntary release limits; ISO 7086 testing; methods of reducing lead migration; and the contribution of lead leached from crystalware to blood lead concentrations are discussed.

3.3 Current Migration Test Methods

There are a wide array of standard migration test methods available for assessing the migration of glass. These are all generally based on an attacking medium (usually water or acetic acid solution) at a given temperature for a given time period. These tests generally use the principles developed by Lyle and Bacon and Burch to effect an attack equivalent to prolonged exposure. Temperatures selected appear to be due to convenience (i.e. autoclave temperature 121⁰C, boiling temperature in lab 98-100⁰C and room temperature 20⁰C). Testing is usually based on either ground grains of glass or a filled article. Determinations can be either weight loss based, titration based or specific elemental analysis based.

The commonly used tests generally fall into three categories.

1) tests at 121⁰C using a water simulant

1.1 United States Pharmacopoeia

30 mins 121⁰C powdered glass test or 60mins 121⁰C whole article test. Titration of the final test solution against standard acid to determine alkali release.

1.2 European/ British Pharmacopoeia

60mins 121⁰C whole article test. Titration of the final test solution against standard acid to determine alkali release.

1.3 ISO 4802

60mins 121⁰C whole article test. FAAS on final test solution

2) tests at 98⁰C using a water simulant

2.1 ISO 719

1 hour 98⁰C powdered glass test using a water simulant. Determination of sodium expressed as sodium oxide in the final test solution.

2.2 ISO 3585

1 hour 98⁰C powdered glass test using a water simulant. Determination of sodium expressed as sodium oxide in the final test solution.

3) tests at 20⁰C using an acetic acid simulant

3.1 ASTM AOAC Method 972.2

24 hours, 20⁰C whole article test using 4% acetic acid. FAAS of lead and cadmium.

3.2 ISO 7086

24 hours, 20⁰C whole article test using 4% acetic acid. FAAS of lead and cadmium.

4 Glass Usage in Food Contact Applications (UK)

4.1 Statistics on Proportions of Use (UK)

Prior to collecting any samples it was necessary to determine in what proportions each of the types of glassware identified in the proposal were in use in the UK. This was to ensure that the most significant compositions and applications were sufficiently represented in the subsequent sampling and testing.

The glass types of interest were as follows:

- soda lime silica containers (bottles, jars etc. including all colours commonly in use)
- soda lime silica table ware/domestic ware (drinking glasses, dishes etc. including lip and rim decoration)
- lead crystal domestic ware (wine glasses, decanters etc.)
- borosilicate table ware (oven dishes, cafettieres etc)
- glass ceramic ware (such as saucepans etc.)

Statistics on proportion of use were derived from data available for the year 1998 and predictions from quarter 1- quarter 3 figures available for the year 1999. This data was gathered from information published by the Office for National Statistics PRA26 Glass Products (excluding flat glass) and figures provided by the British Glass Manufacturers Confederation. The British Glass Manufacturers Confederation has helped to compile and interpret these statistics.

Table 1 shows an approximation of the proportions of use of the various glass types. This data is also shown graphically in Figure 2. This is an approximation as many of the official statistics are reported in tonnage, and we are interested in the number of articles that are produced for use in contact with food. For this reason where conversion from tonnage has been necessary typical weights for each type of ware have been chosen. The weights used are shown in Table 2.

Glass Type	Million Articles (using assumed masses in table 2)	Percentage Breakdown (%)
White Flint SLS Container (WFC)	5578	59.07
Green SLS Container (GC)	1330	14.09
Amber SLS Container (AC)	1500	15.89
Other SLS Colour container (OC)	87	0.92
SLS Tableware (TW-SLS)	914	9.68
Lead Crystal Tableware (TW-LC)	15	0.16
Borosilicate Oven/Tableware (OTW-BS)	16	0.17
Glass Ceramic (GC)	2	0.02

Table 1: Proportions of Use Millions of Units

Glass Type	Assumed Mass (typical for type of ware) (g)
Soda Lime Silica Containers (all colours)	250
Soda Lime Silica Tableware	350
Lead Crystals Tableware	500
Borosilicate Ovenware	1000
Glass Ceramic Ovenware	1000

Table 2: Assumed Article Masses

As can be seen, soda lime silica containers (bottles, jars) and soda lime silica tableware (drinking glasses, dishes) account for almost all of the food contact ware (in terms of units) in the UK (99.65%). The remaining 0.35% consists of lead crystal ware (0.16%), borosilicate ware (0.17%), and glass ceramic ware (0.02%). Even this 0.02% represents 2 million units supplied in 1998-1999 and so with existing household stocks also present, this represents a significant amount of ware in circulation.

Proportions of use - units

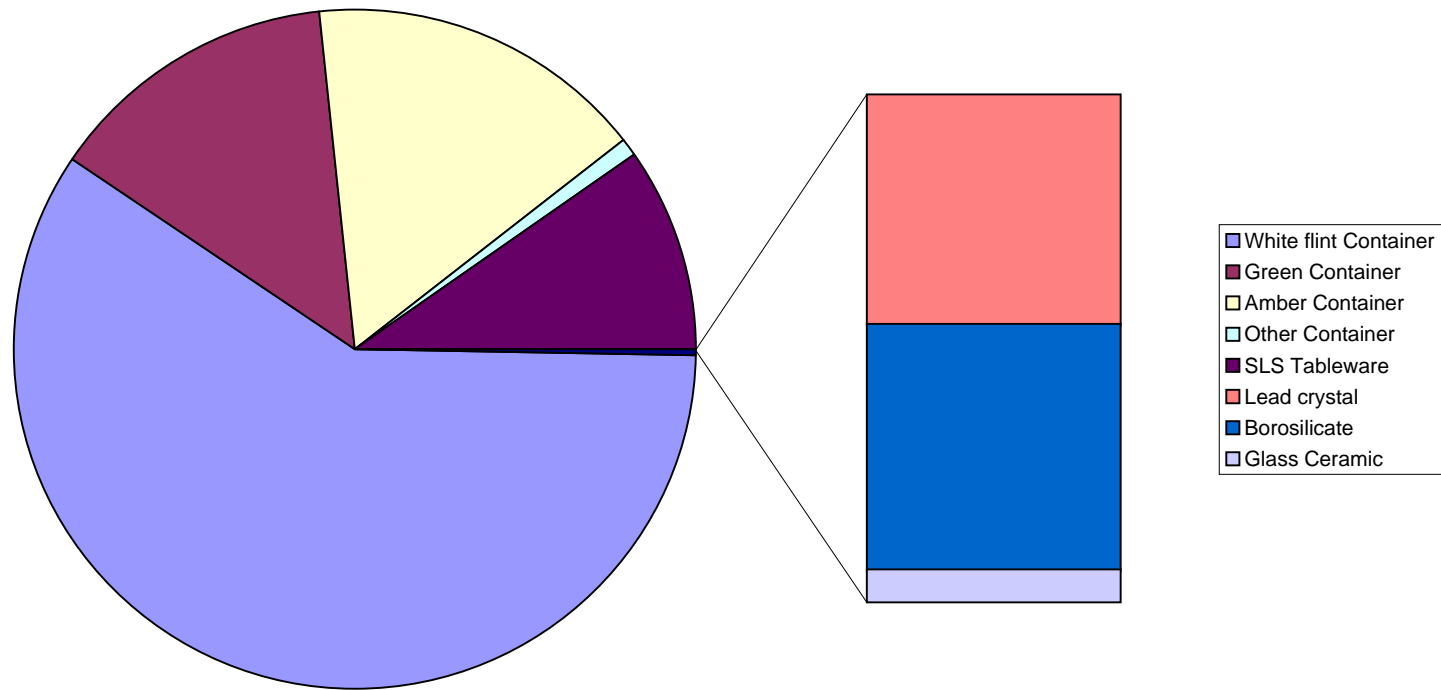


Figure 2 : Proportions of Use

4.1.1 Effect of recycling

Recycling is only commercially significant for soda lime silica containers and typical recycling levels are as follows for the three key colours

- UK Flint/clear glass is typically made up of 16% recycled glass
- UK Amber glass is typically made up of 27% recycled glass
- UK green glass is typically made up of 67% recycled glass
- Continental glass recycling rates are generally higher with green glass manufacture typically consisting of 80-90% recycled glass.

It is for this reason that we would expect green glass, and especially continental green glass to have a greater potential for containing contaminant elements such as lead, which can enter the glass through the recycling chain. Testing was planned to identify if these elevated recycling rates relate in anyway to increased migration.

4.1.2 Homogeneity

Glasses melted industrially on a large scale, such as container glass, are well-known for their high quality and excellent homogeneity. Industrial glass melting furnaces are specifically engineered to produce this high quality, by utilising convection currents within the molten glass. These convection currents occur as a result of the presence of carefully-placed hotter and cooler regions within the glass.

Glass compositions are tightly controlled with regular compositional analysis of the batch materials made to produce the glass, and the glass itself to ensure batch to batch consistency (typically of the order of 0.1weight% concentration) and regular density checks are also carried out to ensure this consistency.

It is in the manufacturers interest to maintain high levels of compositional control as even slight variations in glass composition can have deleterious effects on the forming properties of the glass, which in turn can effect production rates and profitability. High volume glass forming equipment relies on a high level of material consistency to function efficiently.

Glasses melted on a small laboratory scale, such as the spiked melts in this project, are more difficult to homogenise. Mixing of molten glass within a small crucible is more difficult to achieve and therefore generally poorer due to the absence of significant convection currents, since the glass tends to take in heat relatively evenly. This is a well-known phenomenon. Melting conditions were optimised to minimise this problem by initially melting, then breaking up the solidified melt and re-melting. This step tends to greatly improve batch to batch homogeneity (approximately an order of magnitude) but is not completely infallible and can lead to intermittent problems. If however a problem with homogeneity does occur it can have a significant effect on migration behaviour due to the formation of less stable compositions within the glass.

4.1.3 Colourants

The following elements are commonly used in glass compositions to either colour or de-colourise the glass (usually at low levels of the overall composition). The levels of colorant stated are only approximate. A de-colourising addition is an addition whose absorbance in the visible spectrum can deceive the human eye into not seeing the effects of other colouring elements such as Fe

- Cr used as a green glass colorant at typically 0.3 weight% as Cr_2O_3
- Fe present in green and amber glasses (0.3-0.5 weight% as Fe_2O_3), lower levels in white flint soda lime silica glass (<0.05%) and lower still in lead crystal glasses (<0.01%).

- Co is used as a colourant in blue glasses (and some dark amber glasses) at the 0.01-0.02 weight % level as CoO
- Ni can be, but is rarely used as a colourant in some glasses (purple/blue tint) (<0.5 weight % NiO) and is more commonly used as a de-colouriser (often in lead crystal) (<0.001 weight % NiO)
- Cu has been used in amber bottles at <0.01 weight% CuO and has been used as a blue/green colourant at higher (<0.5weight% CuO) levels.
- Se can be used as a de-colouriser, usually in white flint container glasses (low ppm level)

4.2 Articles Collected

A full list of articles collected is given in Appendix I. In terms of proportions of use in the UK market soda lime silica glass dominates (See section 4.1), with glasses of lead crystal, borosilicate and glass ceramic compositions making up only a small proportion of the overall market. It was decided that for the purposes of testing this distribution should be skewed to ensure sufficient testing of articles of compositions with lower market volumes, whilst still carrying out a significant number of tests on the high market volume glass compositions.

Collection of samples was in some cases relatively straightforward. For example, cookware and tableware could be purchased directly. Unfortunately the most commonly used glass food contact articles (soda lime silica glass containers) could not be directly purchased as they appear on the market filled with product. As a result a direct appeal was put out to manufacturers to provide glassware. This appeal was very successful. In total 91 different article types were collected. Table 3 and Figure 3 shows the breakdown of the types of article collected.

Article Type	No Of Sample Types	Percentage Breakdown
White Flint SLS Container (WFC)	23	25.1
Green SLS Container (GC)	6	6.6
Amber SLS Container (AC)	6	6.6
Other SLS Colour container (OC)	3	3.3
SLS Tableware (TW-SLS)	22	24.2
Lead Crystal Tableware (TW-LC)	9	9.9
Borosilicate Oven/Tableware (OTW-BS)	4	4.4
Glass Ceramic (GC)	2	2.2
Decorated	16	17.6

Table 3: Breakdown of Article Types Collected

please note that the figures given indicate sets of articles of the same type, typically each sample set contains between 10 and 20 samples.

No Of Sample Types Collected

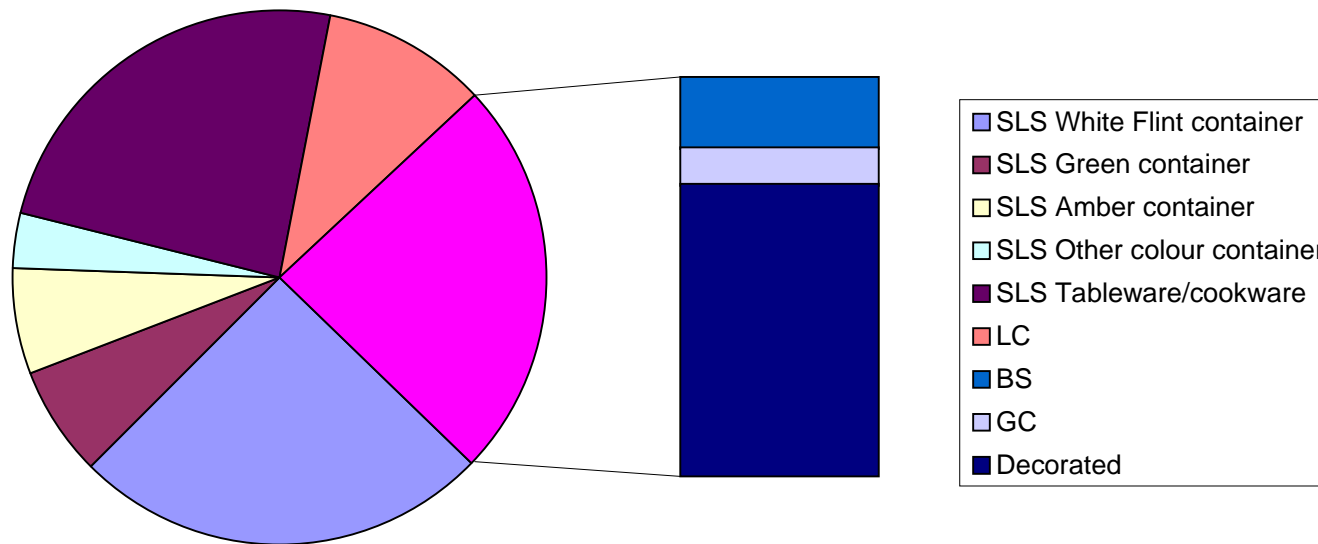


Figure 3: Breakdown by Type of Articles Collected.

There is an additional category of decorated articles in the samples collected data. It was not possible to determine which proportion of articles in the original PRA 26 data (section 4.1) were decorated when establishing the proportions of use, as the statistics did not cover this and only discussed tonnes of glass produced. A relatively large number of articles that had decoration, which could come in to contact with food were collected for testing. These included bowls and dishes that whilst intended perhaps as an ornamental item, may be used by consumers to hold foodstuffs.

The sampling plan was designed in order to expand the proportion of the lesser used glass types whilst still collecting significant numbers of the more widely used glass types such as soda lime silica containers. Article numbers quoted in test results can be cross referenced to the article types from the list in appendix 1 at the rear of the report.

5 Experimental Methods

Throughout this work all analytical results express a reporting limit. This reporting limit is derived as follows.

For ICP-OES work carried out by London and Scandinavian Metallurgical (LSM) Analytical Services, the reporting limit is based on 5x Limit Of Detection (LOD). This limit of detection is expressed by LSM as 2 standard deviations for the element in question, meaning that the reporting limit is effectively 10 standard deviations for the element in question based on blank results.

AAS (FES) work carried out by GTS adopts a similar 5 x limit of detection approach, but LOD under GTS quality systems is expressed as 4 x standard deviations, effectively giving a reporting limit based upon 20 standard deviations.

Analytical determinations of sodium and potassium were carried out by GTS, except in the case of olive oil testing, as the AAS FES instrument at GTS was considered more sensitive for the analysis of these elements. All other results were carried out by LSM using ICP-OES. Where there are specific exceptions to this protocol, this is highlighted in the relevant section of the report. All analytical results are the mean of duplicate determinations and a blank solution was run with every test to ensure reagent quality.

5.1 Baseline Testing

The baseline test adopted has been used as a standard test for all ware to determine the typical behaviour of each type of glassware in aqueous conditions. This typical behaviour can then be compared against behaviour under differing conditions such as different simulants, the effects of surface damage etc. on overall elemental migration. As the baseline test was devised to be a universal test it was decided that it should be simple and easy to set up and be based on the attack of water on the glassware at elevated temperature to simulate a long exposure period at ambient temperatures.

For simplicity, it was decided that baseline testing should be carried out in a standard laboratory oven at normal atmospheric pressure, as the use of an autoclave can be problematic when a switch is made to different simulants such as acetic acid. Problems that may arise from use of an autoclave when using simulants include exposure of the operator to excessive fumes on opening and the accelerated corrosion of the autoclave itself.

A blank test was included in all analytical batches, to check that no elemental “pick up” was occurring from reagents or contact materials such as foil and the plastic bottles used to submit the final test solutions for ICP-OES. Blank solutions were tested in aged borosilicate glassware. Aged borosilicate glassware is glassware that has undergone repeated autoclave cycles and as a result, its elemental release is considered negligible. Its use is widely specified in many glass durability standards including the US, European and British pharmacopoeias. The method adopted was as follows:-

- All baseline tests were carried out on duplicate articles for greater experimental consistency.
- The samples were initially rinsed thoroughly with de-ionised water prior to any testing to remove any surface contaminants such as dust that may contribute to the migration recorded.
- Articles were filled to 1cm of the overflow capacity with grade A de-ionised water. The samples (and the blank) were covered in foil to reduce evaporation losses and placed in the laboratory oven.
- Using a calibrated thermocouple for control, the articles and the blank samples were exposed to the time temperature regime shown in figure 4.
- The test solutions and the blank check solutions were then decanted in to opaque plastic bottles and submitted for ICP-OES analysis.
- Temperature was measured using a calibrated thermocouple in a simulant filled borosilicate conical flask within the test oven.

The time temperature regime chosen is shown in Figure 4 for a typical article and using the equations developed by Lyle based on the Arrhenius expression it is possible to calculate that this exposure equates to approximately 51 - 52 days at 20⁰C. Start temperature did vary for this testing in the range 20⁰C to 30⁰C dependent on ambient conditions, however a similar time (approx. 70 minutes) was taken to reach 95⁰C peak temperature.

This variation equates to a small difference in overall exposure time resulting in a range of 51 - 52 days equivalent exposure for the entire test (with 51 days equating to a 20⁰C start temperature and 52 days being equivalent to a 30⁰C start temperature). The time temperature relationship was kept constant for a range of articles and larger articles were heated at a similar rate to smaller articles by adjustments in power input to the oven to maintain the time temperature profile.

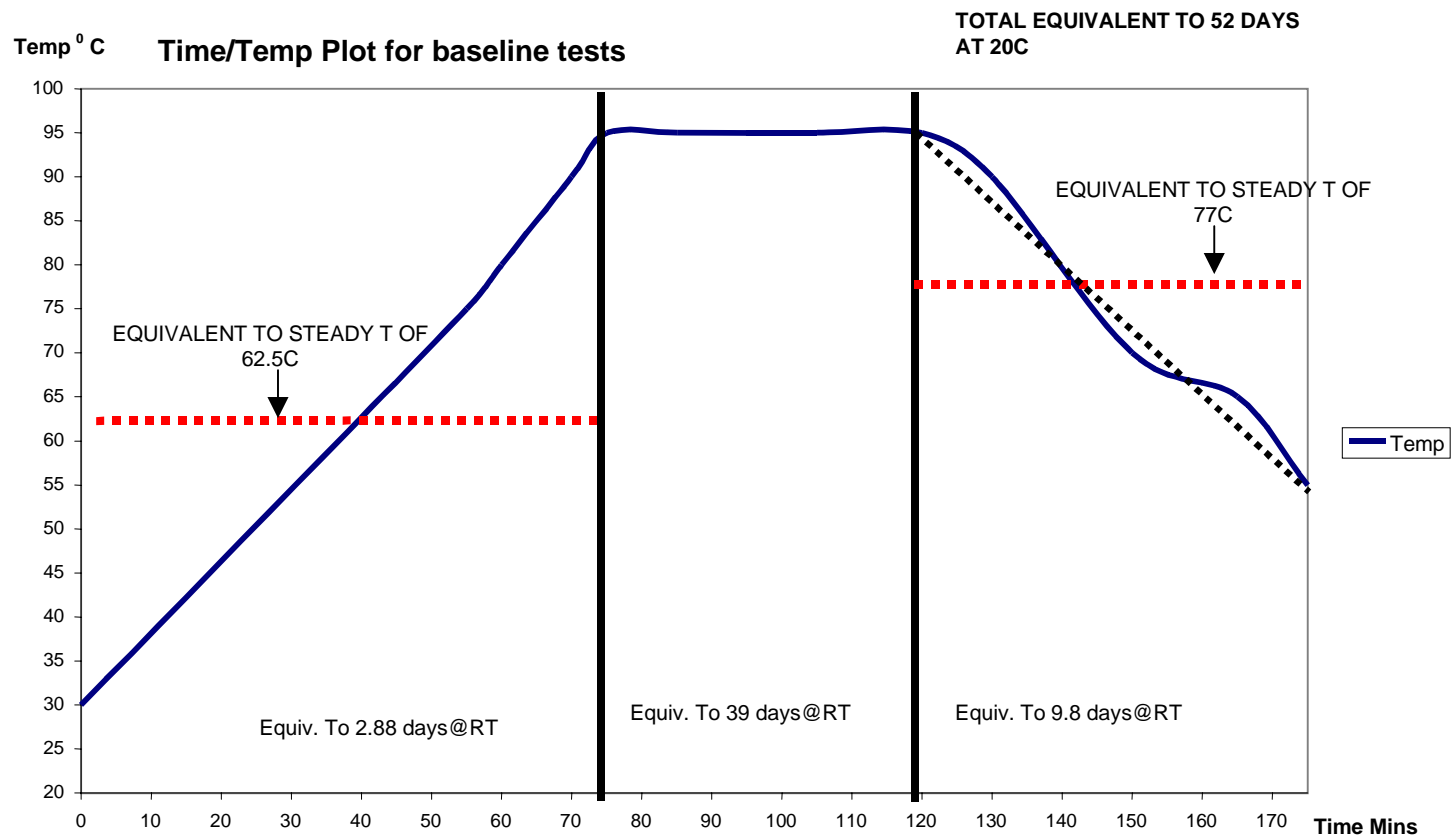
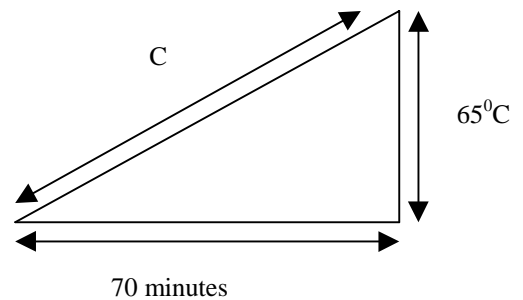
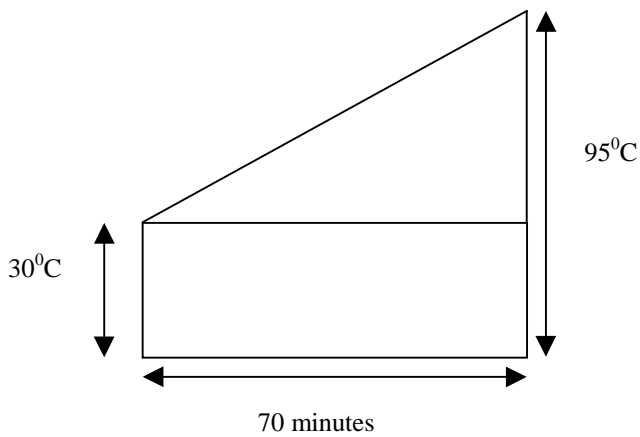


Figure 4: Typical Time Temperature Plot For Baseline Testing (Aqueous Solution).

Calculation Of Equivalent Steady State Temperature

The temperature ramp up area (0-70 minutes) in Figure 4 can be broken down in to a triangular and a rectangular area such that:

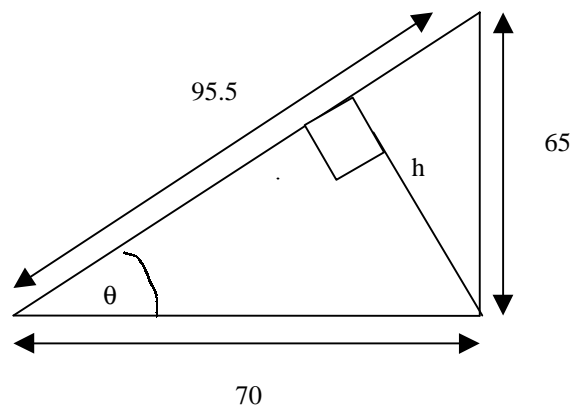


1) Length of Side C

$$A^2 + B^2 = C^2$$
$$65^2 + 70^2 = C^2 = 9125$$
$$C = 95.5$$

2) AREA OF TRIANGLE

$$\text{Area} = \frac{1}{2} \text{ base} \times \text{Perpendicular height}$$
$$\text{Area} = \frac{1}{2} (95.5) \times h$$



3) TO FIND “h” WE MUST FIRST FIND ANGLE θ

$\tan\theta = \text{opp} / \text{adj} = 65 / 70 = 0.9286$ thus $\theta = 42.88^\circ$

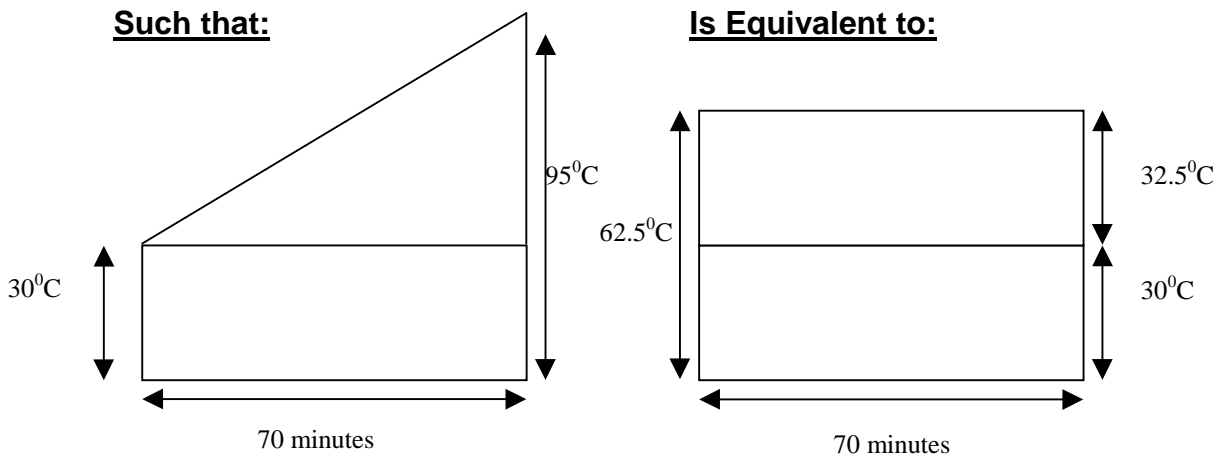
$\sin 42.88 = \text{opp} / \text{hyp} = h / 70$ thus $70 \cdot \sin 42.88 = h$

4) AREA OF TRIANGLE

Area = $\frac{1}{2}(95.5) \times 47.63$
Area = 2274

5) AREA OF EQUIVALENT RECTANGLE WITH 1 DIMENSION = 70 mins

Area = $l \times b$
 $2274 = 70 \times b$
 $2274 / 70 = b = 32.5(^\circ\text{C})$



Therefore the equivalent steady state temperature for the ramp up step would be 62.5°C for 70 minutes. A similar approach can be taken for the ramp down step of the cycle. By placing the figures derived into the relationship given by Lyle (See section 3.1) it is possible to derive an equivalent exposure at room temperature for this section of the test. By combining all portions of the test it is then possible to calculate the overall relative exposure at room temperature in days for the entire test. An example of such calculations is shown in section 3.1 (page 11).

Although the baseline tests were covered with foil some evaporation losses were observed. This issue is discussed in sections 5.7 and 6.8.

5.2 Surface Damage / Caustic Washing / Pasteurisation

5.2.1 Surface Damage

Further samples from the batch of articles that had originally had a baseline behaviour derived were damaged by breaking one of the articles and using a fragment of that article to extensively surface damage a subsequent article. This glass on glass scratching prevents contamination of the surface, which could have occurred were metallic tools used. The scratched articles were then rinsed using de-ionised water prior to being baseline tested. Blank samples were also prepared to check reagent quality.

The samples used for surface damage testing were “fresh” samples i.e. they had not been previously baseline tested but were from the same sample batch. Inhomogeneity of commercial glass melt compositions is very low and manufacturers make great efforts to check that they are producing a chemically consistent composition by a series of quality control measures including regular compositional analysis, regular density determination and tight specification of raw materials. The tight control of chemical composition is essential to the forming characteristics of the glass. The scale of glass production also means that inhomogeneity is very low as a typical commercial glass furnace will have a capacity of approximately 300 tonnes per day, meaning that variations article to article in the same production batch are generally negligible (typically of the order of 0.1 weight%).

5.2.2 Caustic Washing

This test procedure was used on soda lime silica containers to mimic the washing process experienced by returnable bottles such as milk bottles. Alkali solutions are known to attack the surface of bottles and this test was designed to investigate whether the exposure to alkali would make the surface of the glass more susceptible to migration.

The procedure was based on advice given to GTS in discussions with staff at a large industrial bottle filler who wash a large number of returnable bottles annually. The test involved the rinsing of the bottle with de-ionised water, followed by filling the bottle with a 2.5% caustic soda solution which was left to stand at 70⁰C for 20 minutes. This prolonged exposure was designed to mimic repeated washing cycles that a returnable bottle may experience.

The bottles were then thoroughly rinsed and tested as per the baseline method. The results were then compared to those obtained with identical articles that had not been subjected to caustic washing. Blank samples were also prepared to check reagent quality.

The samples used for caustic wash testing were “fresh” samples i.e. they had not been previously baseline tested but were from the same sample batch as articles that had been baseline tested.

5.2.3 Pasteurisation

The pasteurisation cycle was developed after discussions with glass manufacturers about real use, in-container pasteurisation treatments. The treatment was to heat, the previously untested, filled ware rapidly up to 65⁰C and then hold at this temperature for one hour. This was devised as an extreme example of the real use treatment, which is carried out at a similar temperature but for a shorter time period (several minutes). The ware was filled in a similar manner to the baseline test. Blank samples were also prepared to check reagent quality.

5.3 Food Simulants and Foodstuffs

5.3.1 Food Simulants

The food simulants used in this work were as follows:

- 3% acetic acid solution (72 Hours at 50⁰C)
- Water (See baseline time temp regime – max temp 95⁰C)
- Olive Oil (See baseline time temp regime – max temp 95⁰C)
- 15% Ethanol solution (72 Hours at 50⁰C)

The time temperature exposures have been calculated as broadly equivalent with the lower temperature and longer exposure time selected for ethanol and acetic acid solutions due to their relative volatility. The exposure chosen in all cases equates to approx 50 days at room temperature.

5.3.1.1 Acetic Acid Testing

The use of acetic acid solutions in glass migration studies is well documented with numerous international durability/migration standards specifying acetic acid solutions as specific test simulants. Details of these standards is given in section 3.3. For this work a 3% acetic acid solution was selected. This solution concentration is common in migration studies. Test articles were filled to within 1cm of the rim/overflow and covered with foil prior to test.

The time temperature conditions were selected to be broadly equivalent in terms of exposure to that of a baseline test (i.e both tests to be approximately equivalent to 50 days exposure at 20⁰C). Relative to the baseline testing conditions (peak temperature 95⁰C -see section 5.1 for exact time temperature regime), a lower temperature (50⁰C) was selected for a longer time period (72 hours) for the acetic acid testing because of the potential for developing noxious vapours at elevated temperatures when using acetic acid as a simulant. The subsequent tests solutions were submitted for ICP-OES and AAS FES analysis. Blank samples were also prepared to check reagent quality.

5.3.1.2 Water Testing

Water is a commonly used simulant for glass migration studies and many migration tests specify its use (eg United States and European Pharmacopoeia tests). Water migration testing was carried out under the conditions outlined in section 5.1 – Baseline Testing. Water was also used as a simulant in other tests such as microwave testing, surface damage testing etc. The exact conditions for each of these procedures is given in the relevant sections.

5.3.1.3 Olive Oil Testing

Pure rectified olive oil was used for this testing. Test articles were filled to within 1cm of the rim/overflow and covered with foil as per the baseline test method. Blank samples were also prepared using aged borosilicate beakers.

The time temperature conditions were as per the baseline testing regime outlined in section 5.1

1g of oil was diluted with 4ml 4-methyl pentan-2-one (common name MIBK methyl isobutyl ketone). Standards were made up in MIBK and matrix matched with a blank rectified olive oil. The calibration standards were prepared by spiking organometallic standard materials into xylene or mineral oil. Due to the limited availability of these standard materials, only a limited number of elements could be analysed in the final solutions. The standards and samples were run on an ICP-OES. The method reporting limits for the elements available for analysis were (in ppm in the oil) Al<0.5, Ca<0.1, K<1, Mg<0.1, Na<0.5, Pb<2 and Si<0.5. These reporting limits are based on 5x Limit Of Detection (Limit of detection for this work is 2 times the standard deviation of the analytical technique for that element for that element). Effectively these reporting limits equate to 10 the elemental standard deviation based on analysis of matrix matched blank solutions. In the case of olive oil testing Na and K determinations were carried out by London & Scandanavian Ltd.

5.3.1.4 15% Ethanol Testing

The use of 15% ethanol solutions was intended to simulate the use of glassware for alcoholic beverages. Test articles were filled to within 1cm of the rim and covered with foil as per the baseline test method.

The time temperature conditions were selected to be broadly equivalent in terms of exposure to that of a baseline test (i.e both tests to be approximately equivalent to 50 days exposure at 20⁰C. Relative to the baseline testing conditions (peak temperature 95⁰C -see section 5.1 for exact time temperature regime), a lower temperature (50⁰C) was selected for a longer time period (72 hours) for the aqueous ethanol solution testing because of the potential for developing noxious vapours at elevated temperatures when using aqueous ethanol solution as a simulant.

The subsequent tests solutions were submitted for ICP-OES and AAS FES analysis. Blank samples were also prepared to check reagent quality.

5.3.2 Real Foodstuffs

Real food stuffs were selected for testing after discussions with Dr Emma Bradley of CSL. A discussion of the significance of pH to migration is discussed in section 3.1. Test articles were rinsed with de-ionised water prior to testing to remove any dust or contaminants that could affect analytical results, as described in the baseline method in section 5.1. The food stuffs tested were as follows:-

- Wine
- Orange Juice
- Grapefruit Juice
- Seeded Jam
- “Squeezy” Jam (A free flowing jam formulated to be squeezed from flexible packaging)
- Mushroom Soup
- Chicken Soup
- Pasta Sauce
- Sweet and Sour Sauce
- Apple Sauce
- Lemon Juice

Various treatments were used to reflect cooking and general useage conditions of the food stuffs in question.

The wine, orange juice and grapefruit juice samples were tested after standing at room temperature in drinking glasses for 30 minutes to represent typical usage.

The jams were oven baked at 225⁰C for 30 minutes in a jar to represent use in cooked foodstuffs.

The mushroom and chicken soups were micro-waved for 3 minutes in a Pyrex mixing bowl.

The pasta sauce and sweet and sour sauce were oven baked at 225⁰C for 30 minutes in Pyrex and glass ceramic cookware to represent the cooking of a casserole.

The apple sauce and lemon juice were heated in borosilicate oven ware at 225⁰C for 30 minutes to simulate the baking of a dessert.

After testing, portions (approximately 2.5g) of each solution, stirred for homogenisation were digested in 10ml of conc. HNO₃ and 5ml ~30% H₂O₂ and diluted to 50 ml with de-ionised water for analysis by ICP-OES. A blank sample was prepared to check reagent quality and also a sample of food was taken pre-contact with any glass test article, to allow a before and after comparison. As sample weights taken varied slightly from sample to sample and there was a subsequent dilution, the reporting limits cannot just be expressed as those for analysis of the final solution. Mass of sample and dilution must be accounted for also. Table 4 indicates reporting limits derived for the testing of foodstuffs of major glass constituent elements (B, Na, Si, K, Ca, Mg, Al) to give a feel for the effect of the digestion steps on reporting limits compared to other (e.g. baseline) testing where the analytical determination was carried out directly on the test solution.

	B	Na	Si	K	Ca	Mg	Al
Pasta Sauce	0.55	0.36	1.82	0.18	0.18	0.18	0.91
Sweet+Sour Sauce	0.53	0.35	1.77	0.18	0.18	0.18	0.88
Apple Sauce	0.57	0.38	1.89	0.19	0.19	0.19	0.94
Squeezy Jam	0.50	0.34	1.68	0.17	0.17	0.17	0.84
Seeded Jam	0.58	0.38	1.92	0.19	0.19	0.19	0.96
Chicken Soup	0.50	0.33	1.67	0.17	0.17	0.17	0.84
Mushroom Soup	0.55	0.36	1.82	0.18	0.18	0.18	0.91
Wine	0.51	0.34	1.70	0.17	0.17	0.17	0.85
Lemon Juice	0.56	0.37	1.86	0.19	0.19	0.19	0.93
Grapefruit Juice	0.57	0.38	1.90	0.19	0.19	0.19	0.95
Orange Juice	0.55	0.36	1.82	0.18	0.18	0.18	0.91

Table 4: Reporting Limits For Real Foodstuff Testing (Units ppm)

5.4 Lip and Rim Testing / Microwave Testing

5.4.1 Lip and Rim Testing

Articles that are decorated in the lip/rim area have the potential for direct contact of that decoration with foodstuffs.

Lip and rim decoration testing was carried out following the method prescribed in ASTM method C927.80 (1999) ⁽¹²⁾. The test involves the immersion of the inverted pre-rinsed article in a 4% acetic acid solution up to a level 2cm above the rim of the upturned article. The articles are then left in a darkened room / container for 24 hours at 20⁰C to 24⁰C.

The darkened room is specified in the method due to the photosensitivity of cadmium over this time period in low concentration solutions. In other testing during this work the darkened room effect was achieved by testing in a sealed laboratory oven or autoclave and immediate transfer of test solutions in to opaque plastic containers post test. The solutions were then tested using ICP-OES to determine the elemental release from the lip/rim decoration. Blank samples were also prepared to check reagent quality.

5.4.2 Microwave Testing

Microwave testing was carried out using a standard domestic microwave manufactured by Matsui Model No 170TC.

Articles were rinsed and filled in a similar manner to baseline testing but were not foil covered. Articles with their own lids could be heated on full power (650W) for ten minutes (such as casserole dishes) as losses from spitting etc were prevented. Articles without lids were cooked for 20 minutes on medium (325W) power to prevent spitting. Blank samples were also prepared to check reagent quality.

After testing solutions were analysed by ICP-OES analysis and AAS FES analysis.

5.5 Elevated Pressure/Temperature Tests

The test regime selected for elevated temperature/elevated pressure testing was the one prescribed in the European Pharmacopoeia hydrolytic resistance test which is one hour in an autoclave at 121⁰C, which generates a peak pressure of approximately 18 psi, with a prescribed temperature ramp up and ramp down rate using de-ionised water as the simulant solution. Articles were filled and treated as per the baseline migration tests. This exposure is equivalent to six to twelve months exposure at room temperature.

The tests were focused on articles which may hold their contents for long periods of time such as soda lime silica glass containers (bottles, jars etc.) with limited testing of other glass types and articles included for comparative purposes. Blank samples were also prepared to check reagent quality.

5.6 Spiked Melts

The purpose of spiked melts was to assess if certain elements of possible toxicological interest were released from the common glass compositions at typical and elevated levels. Although lead migration from lead crystal compositions had been considered to be well documented in existing literature (see section 3.2) there was interest in the potential for lead to migrate from soda lime silica container compositions, due to the potential for lead content in this glass type from contamination of the recycling stream. The elements of interest were selected in consultation with FSA as follows.

For Soda Lime Silica and Borosilicate glasses

Ni, Cr, Pb and Cd.

Ni and Cr are used as glass colourants and Pb and Cd have the potential to enter the recycling chain through decoration and also mixing of glass streams such as TV tubes and lead crystal. The potential for entry of these elements into borosilicate glass through recycling is low, however the glass was doped at similar levels to the doping levels for soda lime silica glass to allow direct comparison of “migration potential” of the two compositions. Cr and Ni colourants have been used in borosilicate glasses.

As there is a large body of work and international standards covering the release of lead from lead crystal glass it was decided in discussion with the FSA that the elements of interest in lead crystal glass should be Sb and As. Both these elements are used as refining agents and can appear in lead crystal glass compositions at approximately 0.2 – 0.3 percentage weight (wt.%) levels as oxides.

Melts were developed by taking common commercial glass compositions melted from high purity reagent grade raw materials and then introducing dopants by proportionally reducing all other constituents. A simplified example of this proportionate doping is given below.

If a 1kg batch of a simplified soda lime silica glass is prepared with the following composition

SiO ₂	700g
Na ₂ O	150g
CaO	120g
Al ₂ O ₃	20g
K ₂ O	10g

Total weight = 1000g

To dope 1000ppm of Lead (or 0.1 weight % or 1g per Kg) would require an addition of lead as its equivalent oxide such that 1g of lead added to the glass is equivalent to approximately 1.2g of lead oxide (PbO).

We would reduce the mass of SiO₂ by 0.840g (70% of 1.2g) in the batch, Na₂O by 0.180g (15% of 1.2g) in the batch, CaO by 0.144g (12% of 1.2g) in the batch, Al₂O₃ by 0.024g (2% of 1.2g) in the batch and K₂O by 0.012g (1% of 1.2g) in the batch to maintain the overall batch mass (i.e a proportionate reduction of the batch constituents)

The levels chosen were based on typical content levels of commercial glasses. Lead content can reach figures of 100ppm in soda lime silica container glasses with a high proportion of recycling. Cadmium is rare at these levels in such glasses, however a starting point of 50ppm (50% of lead dopant) has been arbitrarily chosen which is more extreme than typical real world levels.

For comparison, Table 5 shows the analysis of ten soda lime silica containers submitted to the project for lead and cadmium content using an X-Ray Fluorescence analysis programme developed for the analysis of glass containers in compliance with the European Commission Directive on Packaging and Packaging Waste – Essential Requirements 94/62/EC regarding Pb, Cd, Hg and Cr(VI) content.

SAMPLE I.D.	Pb (ppm)	Cd (ppm)
8-1	129	< 15
2-3	149	< 15
21-1	94	< 15
2-1	105	< 15
43-2	119	< 15
15-2	43	< 15
43-4	129	< 15
15-4	13	< 15
15-1	83	< 15
43-1	109	< 15

Table 5: Pb and Cd Content of Containers Submitted To Project Determined By X-Ray Fluorescence Analysis.

The melts were prepared in the following proportions.

In soda lime silica glass:-

Blank white flint soda lime silica

	Weight%
SiO ₂	72.60
Al ₂ O ₃	1.80
Fe ₂ O ₃	0.04
Na ₂ O	13.25
CaO	11.12
MgO	0.27
K ₂ O	0.66
TiO ₂	0.06
SO ₃	0.24

Spiked SLS

	<u>Typical</u>	<u>2x</u>	<u>5x</u>	<u>10x</u>
Lead	100ppm	200ppm	500ppm	1000ppm
Cadmium	50ppm	100ppm	250ppm	500ppm
Chromium	1197ppm	2394ppm	5985ppm	11970ppm
Nickel	94ppm	189ppm	471ppm	943ppm

In borosilicate glass.

Blank borosilicate glass

	Weight%
SiO ₂	81.90
B ₂ O ₃	10.70
Al ₂ O ₃	2.70
Na ₂ O	3.69
K ₂ O	1.00

Borosilicate spikes

	<u>Typical</u>	<u>5x</u>
Lead	100ppm	500ppm
Cadmium	50ppm	250ppm
Chromium	1197ppm	5985ppm
Nickel	94ppm	471ppm

In lead crystal glass

Blank lead crystal glass

	Weight%
SiO ₂	55.0
PbO	31.9
K ₂ O	12.3
Na ₂ O	0.2
B ₂ O ₃	0.5
Al ₂ O ₃	0.1

Lead crystal spikes

	Typical	5x
Arsenic	1363ppm	6816ppm
Antimony	1504ppm	7518ppm

Once the melts were prepared they were ground into powder using the method prescribed in the European Pharmacopoeia (EP) powdered glass test. Portions of the spiked grains were then dried as per the EP procedure.

A 20g portion of each spike sample was placed into an aged borosilicate flask and either tested via the baseline method test conditions (see section 5.1) with water, or via the acetic acid test conditions (see section 5.3). The resulting test solution was analysed along with blank samples by ICP-OES and FAAS.

As an additional piece of work funded as a continuation project to the original study the effect of spiked melts pressed into articles against common moulds was also investigated. This was intended to give more of a real use indication of release from spiked articles, as grain tests expose a large surface area to volume ratio to the test solution. There is, therefore, the potential to exaggerate any potential risk for glassware that may be manufactured for food contact purposes. For this work glass melts were prepared at the typical and 5x typical level for the elements and glass compositions described earlier in this section. The glass melts were then pressed using cast iron moulds (which is a common practice in glass manufacture) to form small cups. The mould dimensions were as follows:

The outer mould dimension was

Base OD	64 mm
Top OD	72 mm
Mould height	31mm
Approx. 8° taper	

The plunger dimension was

Base OD	54mm (2 mm radius at the edge)
Top OD	60mm (2mm radius at the joint)
Plunger height	25mm
Approx. 8° taper	

This yielded round cups of the following internal dimensions (± 1 mm)

Depth	=	25.5mm
Diameter top	=	55.0mm
Diameter Bottom	=	48.5mm

The forming conditions were as follows

The mould piece and plunger temperature prior to forming was controlled to 560⁰C. As each glass type is of a different chemical composition and therefore has different viscosity-temperature properties exact forming conditions differ. This is also the case in commercial glass production. The conditions used were as follows.

Lead crystal glasses

Pouring temperature (into mould) 1050⁰C

Pressing time 10 – 15 seconds

Annealing schedule 520⁰C overnight to relieve stresses

Soda Lime Silica Glasses

Pouring temperature (into mould) 1160⁰C

Pressing time 20 – 35 seconds

Annealing schedule 570⁰C overnight to relieve stresses

Borosilicate Glasses

Pouring temperature (into mould) 1350⁰C

Pressing time 30-40 seconds

Annealing schedule 600⁰C overnight to relieve stresses.

The pressed cups were subsequently migration tested with 30ml of test solution, using water as per the baseline test method and acetic acid solution as set out in sections 5.1 and 5.3.. The test solutions were “topped up” to account for evaporation losses (typically 10-15%) at the end of the test cycle. The test solutions were then analysed along with a blank solution using ICP-OES for the elements of interest.

5.7 Surface Area To Volume Ratio

The effect of surface area to volume ratio on release was assessed using baseline migration results on flint soda lime silica containers. The release of Si, Na and Ca (the main glass constituents) were plotted against a factor derived from the calculated contact surface area (cm³) over the fill volume (cm³).

5.8 Evaporation Losses

Evaporation losses were assessed by measuring the test solution volume before and after baseline tests for a number of articles ranging from wide, shallow bowls/dishes to narrow necked wine bottles. The results of this assessment are shown in Table 6.

Subsequent tests with other simulants were “made up” to their original volume with simulant at the end of the test. This was also undertaken on a number of baseline water test articles with a range of diameters (bowls, wine bottles, dishes etc.) and these results compared to test results where the solution had not been “made up” to the original volume. Table 7 shows the effect of evaporation losses on a limited subset of baseline tested articles. The table shows the comparison between test results for articles which under baseline test conditions, have not had the original test volume “made up” at the end of the test, and test results for articles from the same batch after being “made up”. The samples selected for this check were chosen because of their widely differing shapes.

The samples selected for testing were sample 18-1, a shallow borosilicate dish, 12-4 a wide mouth soda lime silica jar, 2-1 a small narrow necked soda lime silica bottle, 17-6 a lead crystal bowl and sample 6-1 another larger narrow neck bottle.

sample	sample weight (g)	sample+ water before (g)	sample + water after (g)	Diameter of Sample Neck / Opening (cm)	Percentage loss (%)
4-2 (Jar)	189.8	443.3	425.1	4.8	7.2
17-1 (Tumbler)	492.3	834.5	817.9	8.5	4.9
13-2 (Large Wine Glass)	211.0	500.2	486.0	7.4	4.9
24 (Small Wine Glass)	203.0	372.1	364.0	4.9	4.8
19-1 (Mug)	402.2	642.1	627.0	7.2	6.3
50 (medium wine Glass)	146.1	351.5	338.8	6.0	6.2
38 (tumbler)	209.3	514.0	488.3	5.5	8.4
13-3 (Tumbler)	281.4	715.1	692.8	6.3	5.1
25-3 (Tumbler)	334.4	727.9	713.1	7.7	3.7
13-1(Large Wine Glass)	213.4	488.1	476.8	7.4	4.1
51 (mug)	216.8	435.0	415.7	6.7	8.9
17-4 (Tumbler)	258.5	525.5	507.0	5.6	6.9
28 (Dish with own lid)	681.3	2241.3	2201.3	20.8	2.6
31-2 (Large shallow Rectangular dish)	1202.2	2882.2	2642.2	29.0 x 20.5	14.4
14-1 (Blue Mineral Water Bottle)	207.9	455.9	441.8	1.9	5.7
43-1 (Green Wine Bottle)	593.6	1338.5	1322.1	1.8	2.2
Mean					6.02

Table 6: Evaporation Losses On Baseline Tests

	2-1-a	2-1-b	6-1-a	6-1-b	18-1-a	18-1-b	17-6-a	17-6-b
Si	1.99	1.42	0.36	0.32	<0.10	<0.10	1.78	1.98
Mg	0.08	0.06	<0.01	0.02	<0.01	<0.01	<0.01	<0.01
Ca	0.47	0.25	0.05	0.06	0.07	<0.01	<0.01	<0.01
Na	0.99	0.56	0.19	0.25	<0.02	<0.02	0.56	0.62
K	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	1.10	1.21
Ba	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.00	0.00
B	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	0.00	0.00
Pb	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	0.14	0.24
Sb	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.07	0.08

Table 7: Difference In Migration Result For non “Made Up” (a) and “Made Up” (b) test solutions.

As can be seen there appears little difference in overall migration result between the two test methods with no general trend being observed. The difference in results between the sets of conditions are generally within the %uncertainty established in the appendices at the rear of this report which, suggests that the uncertainty quoted is of the right order.

6 Results

6.1 Articles Tested

6.1.1 Samples Baseline Tested

Baseline testing was applied to the full range of glass articles. The breakdown of articles baseline tested is shown in Table 8 and Figure 5. As the further tests such as simulant tests, controlled damage etc. were carried out on smaller subsets of the baseline tested samples the sample details for these particular sets of conditions can be found in the relevant results section.

Article Type	No Of Sample Types	Percentage Breakdown
White Flint SLS Container (WFC)	21	25.3
Green SLS Container (GC)	6	7.2
Amber SLS Container (AC)	6	7.2
Other SLS Colour container (OC)	3	3.6
SLS Tableware (TW-SLS)	19	22.9
Lead Crystal Tableware (TW-LC)	10	12.0
Borosilicate Oven/Tableware (OTW-BS)	4	4.8
Glass Ceramic (GC)	2	2.4
Decorated	12	14.5

Table 8: Breakdown of Article Types Baseline Tested

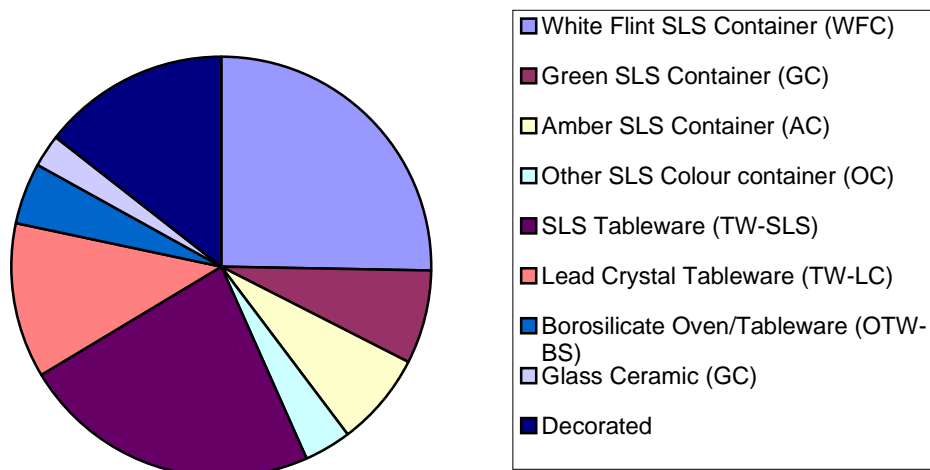


Figure 5: Breakdown of Sample Types Baseline Tested

Table 9 and Figure 6, show the country of origin of the baseline tested articles.

Article Type	Countries of Origin
White Flint SLS Container (WFC)	16 UK, 1 Finland, 2 Netherlands, 2 France
Green SLS Container (GC)	4 UK, 1 Germany, 1 Netherlands
Amber SLS Container (AC)	3 UK, 1 Germany, 1 Eire, 1 France
Other SLS Colour container (OC)	1 (Blue) UK, 2 (Dead Leaf Green) France, 2 (Half white) Germany
SLS Tableware (TW-SLS)	5 UK, 2 Italy, 8 France, 1 China, 3 Unknown
Lead Crystal Tableware (TW-LC)	1 Eire, 2 Italy, 1 Slovakia, 5 UK, 1 Unknown
Borosilicate Oven/Tableware (OTW-BS)	1 UK, 1 France, 1 USA, 1 EU – Unknown Country
Glass Ceramic (GC)	1 France, 1 USA
Decorated	7 UK, 1 Italy, 1 Greece, 1 Unknown, 1 France

Table 9: Country Of Origin Of Baseline Tested Articles.

Country Of Origin Of Samples Baseline Tested

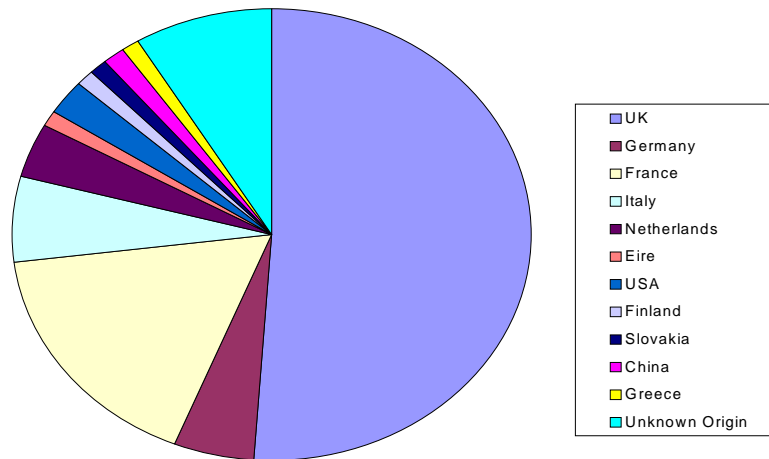


Figure 6: Country Of Origin Of Baseline Tested Articles

6.1.2 Articles Tested Under Surface Damage Conditions

21 sets of articles were surface damaged (see section 5.2.1 for test details), tested and compared to their original baseline test results. The surface damage experiments were carried out as per the method described in Chapter 5. The percentage breakdown of the types of articles tested for the effect of surface damage is shown in Table 10.

Article Type	Number of Sample Types	Percentage Breakdown
White Flint SLS Container (WFC)	6	28.6
Green SLS Container (GC)	1	4.8
Amber SLS Container (AC)	1	4.8
SLS Tableware (TW-SLS)	5	23.8
Lead Crystal Tableware (TW-LC)	3	14.3
Borosilicate Oven/Tableware (OTW-BS)	1	4.8
Glass Ceramic (GC)	1	4.8
Decorated	3	14.3

Table 10: Articles Tested After Surface Damage.

6.1.3 Articles Tested Under Caustic Washing Conditions

12 sets of articles were subjected to caustic wash conditions (see section 5.2.2 for test conditions) then tested under baseline migration conditions and the results compared to the original baseline test results. As caustic washing is a practice normally carried out on returnable soda lime silica containers this testing was limited to containers of soda lime silica composition. Table 11 shows the breakdown of articles that have been subjected to caustic wash conditions.

Article Type	Number of Sample Types	Percentage Breakdown
White Flint SLS Container (WFC)	3	25.0
Green SLS Container (GC)	3	25.0
Amber SLS Container (AC)	4	33.3
Other SLS Container	2	16.7

Table 11: Articles Tested After Caustic Washing.

6.1.4 Articles Tested Under Pasteurisation Conditions

18 sets of articles were subjected to pasteurisation conditions (see section 5.2.3 for test conditions) and the results compared to their original baseline test results. As pasteurisation is a practice normally carried out on returnable soda lime silica containers this testing was limited to containers of soda lime silica composition. Table 12 shows the breakdown of articles that have been subjected to pasteurisation conditions.

Article Type	Number of Sample Types	Percentage Breakdown
White Flint SLS Container (WFC)	9	50.0
Green SLS Container (GC)	5	27.7
Amber SLS Container (AC)	3	16.7
Other SLS Container	1	5.5

Table 12: Articles Tested Under Pasteurisation Conditions.

6.1.5 Articles Tested With Food Simulants

6.1.5.1 Acetic Acid

Table 13 shows the number and types of samples tested using acetic acid. (see section 5.3.1.2 for test conditions) Particular attention was paid to green soda lime silica containers as these containers tend to have the highest recycling rates and therefore a potential to contain lead. Decorated ware was also investigated in more detail as some enamel decorations are known to contain lead. Work on lead crystal glass was limited as significant work has been carried out by other researchers ^(8,9,10,11) on the release of lead into acetic acid simulants from lead crystal glassware. As discussed in section 3.2, acetic acid is an effective solution for lead extraction.

Article Type	Number of Sample Types	Percentage Breakdown
White Flint SLS Container (WFC)	1	7.1
Green SLS Container (GC)	4	28.6
Amber SLS Container (AC)	1	7.1
Other SLS Container (OC)	1	7.1
SLS Tableware (TW-SLS)	1	7.1
Lead Crystal Tableware (TW-LC)	1	7.1
Borosilicate Oven/Tableware (OTW-BS)	1	7.1
Glass Ceramic (GC)	1	7.1
Decorated	3	21.4

Table 13: Articles Tested With Acetic Acid.

6.1.5.2 Olive Oil

Table 14 shows the number and types of samples tested using olive oil. (see section 5.3.1.4 for details). Due to the time consuming nature of olive oil testing and subsequent difficulty in setting up ICP-OES analysis of olive oil test solutions, testing was limited. Olive oil is usually supplied/stored in white flint soda lime silica bottles and also used in cooking and salad dressings. Test samples were thus selected to cover table / cookware and a typical white flint soda lime silica container.

Article Type	Number of Sample Types	Percentage Breakdown
White Flint SLS Container (WFC)	1	16.7
SLS Tableware (TW-SLS)	2	33.3
Lead Crystal Tableware (TW-LC)	1	16.7
Borosilicate Oven/Tableware (OTW-BS)	1	16.7
Glass Ceramic (GC)	1	16.7

Table 14: Articles Tested With Olive Oil

6.1.5.3 15% Ethanol

Table 15 shows the number and types of samples tested using 15% ethanol (see section 5.3.1.5 for details).

Article Type	Number of Sample Types	Percentage Breakdown
White Flint SLS Container (WFC)	2	11.8
Green SLS Container (GC)	4	23.5
Amber SLS Container (AC)	2	11.8
Other SLS Container (OC)	1	5.9
SLS Tableware (TW-SLS)	4	23.5
Lead Crystal Tableware (TW-LC)	1	5.9
Decorated	3	17.6

Table 15: Articles Tested With 15% Ethanol

The focus of this testing was largely on soda lime silica containers that may be used as bottles for storing alcohol and soda lime silica tableware which is most commonly used for drinking glasses with alcoholic beverages. No borosilicate or glass ceramic ware was tested as this is generally used as cookware.

6.1.6 Articles Tested With Real Food Stuffs

Articles tested with real food stuffs are described in section 6.4

6.1.7 Articles Tested Under Lip And Rim Test Conditions

All articles tested under lip and rim test conditions were decorated articles with decoration in the lip/rim area. A total of ten articles were tested.

6.1.8 Articles Tested Under Microwave Conditions

Table 16 shows the number and types of samples tested under microwave conditions. Articles that may be used in microwave heating / cooking were selected. These included two white flint jars as well as oven/tableware, as there is a possibility of baby food being warmed in a microwave by consumers.

Article Type	Number of Sample Types	Percentage Breakdown
White Flint SLS Container (WFC)	2	25.0
SLS Tableware (TW-SLS)	2	25.0
Borosilicate Oven/Tableware (OTW-BS)	2	25.0
Glass Ceramic (GC)	2	25.0

Table 16: Articles Tested Under Microwave Conditions

6.1.9 Articles Tested Under Elevated Temperature / Elevated Pressure Conditions

Tests at 121⁰C (and approximately 18psi pressure) were mainly targeted at SLS containers as these were selected as generally being used in long term storage situations. Table 17 shows the breakdown of samples tested under these conditions.

Article Type	Number of Sample Types	Percentage Breakdown
White Flint SLS Container (WFC)	18	48.6
Green SLS Container (GC)	6	16.2
Amber SLS Container (AC)	6	16.2
Other SLS Container (OC)	3	8.1
SLS Tableware (TW-SLS)	1	2.7
Lead Crystal Tableware (TW-LC)	1	2.7
Borosilicate Oven/Tableware (OTW-BS)	1	2.7
Glass Ceramic (GC)	1	2.7

Table 17: Articles Tested at 121⁰C

6.1.10 Spiked Melts

Samples prepared for spike melt trials are detailed in section 5.6

6.1.11 Surface Area To Volume Trials

Five white flint soda lime silica articles were selected to investigate the effect of surface area to volume ratio on migration.

6.2 Baseline Test Results

Baseline testing was carried out as per the method outlined in section 5.1. Please note that all results are expressed in ppm of the final test solution.

6.2.1 White Flint SLS Containers (Baseline Tests)

The results of migration testing for flint soda lime silica containers are shown graphically in Figure 7. Only elements detected above their relevant reporting limit during testing are listed as migrants. Figure 7 shows that migration of only eight elements was detected under baseline conditions at levels above quoted reporting limits for elemental determination in the final test solution. The results are the average of two independent determinations, carried out by testing each of a matched pair of articles respectively. For the purpose of statistical and graphical treatment values below reporting limit were treated as zero values to generate average figures. The results of the testing are also shown in Table 18

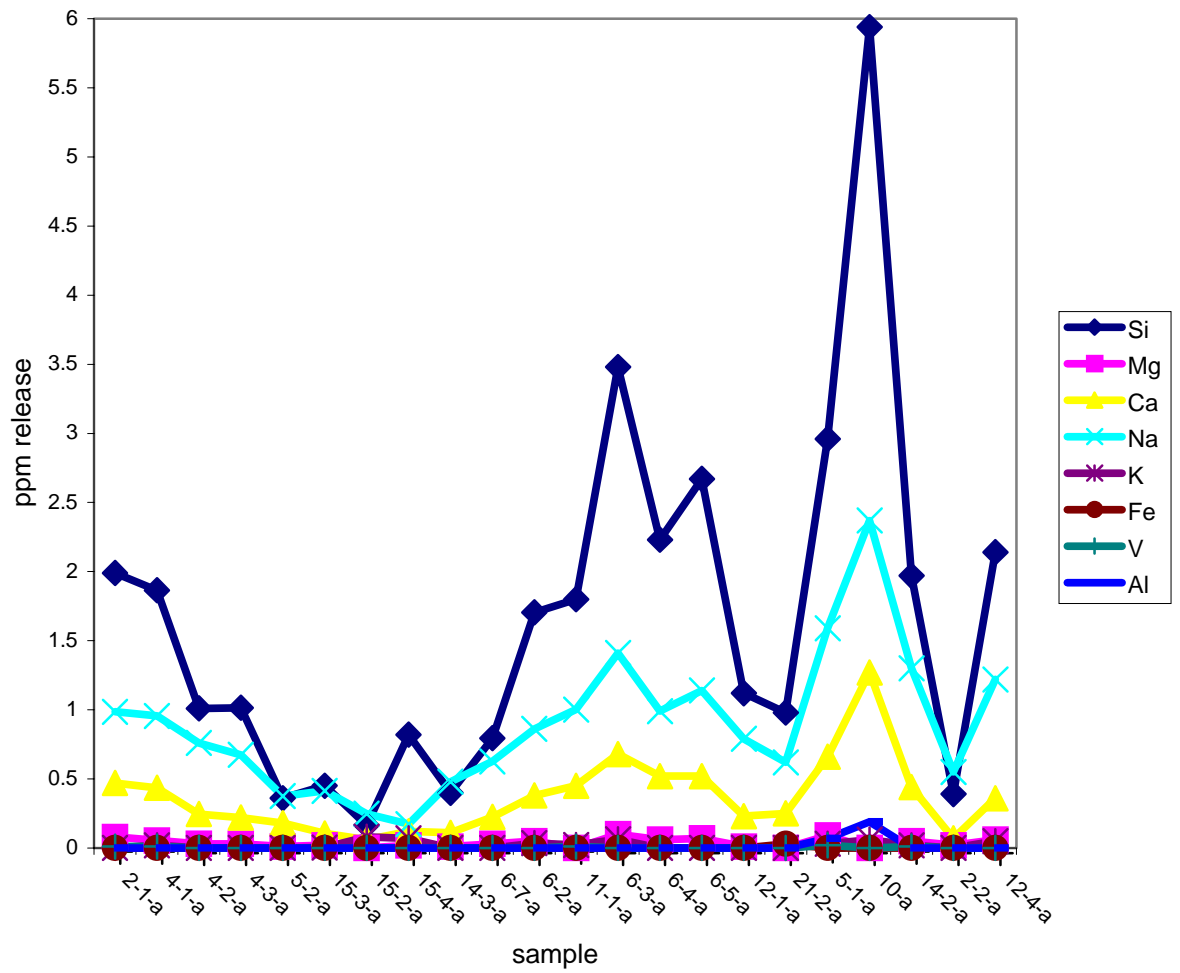


Figure 7: Baseline Migration Results for White Flint SLS Containers

Element	No Of Samples Tested	Number of samples with result equal to or greater than reporting limit	Reporting Limit (ppm)	Min (ppm)	Max (ppm)	Mean (ppm)
Si	21	21	0.1	0.4	5.9	1.7
Mg	21	18	0.01	<0.01	0.10	0.04
Ca	21	21	0.01	0.07	1.27	0.37
Na	21	21	0.02	0.17	2.37	0.92
K	21	9	0.01	<0.01	0.07	0.02
Fe	21	1	0.02	<0.01	0.03	<0.02
V	21	5	0.01	<0.01	0.02	<0.01
Al	21	2	0.05	<0.05	0.19	<0.05

Table 18: Results of baseline Migration Tests On White Flint Soda lime Silica Containers

The sample with overall highest release in this category was sample 10, which was a small container with a very high surface to volume ratio manufactured in the UK. The sample with overall lowest release in this category appears to be sample 5-2, which was a large SLS whiskey bottle manufactured in the UK. The release figures for both of these samples are given in Figure 8.

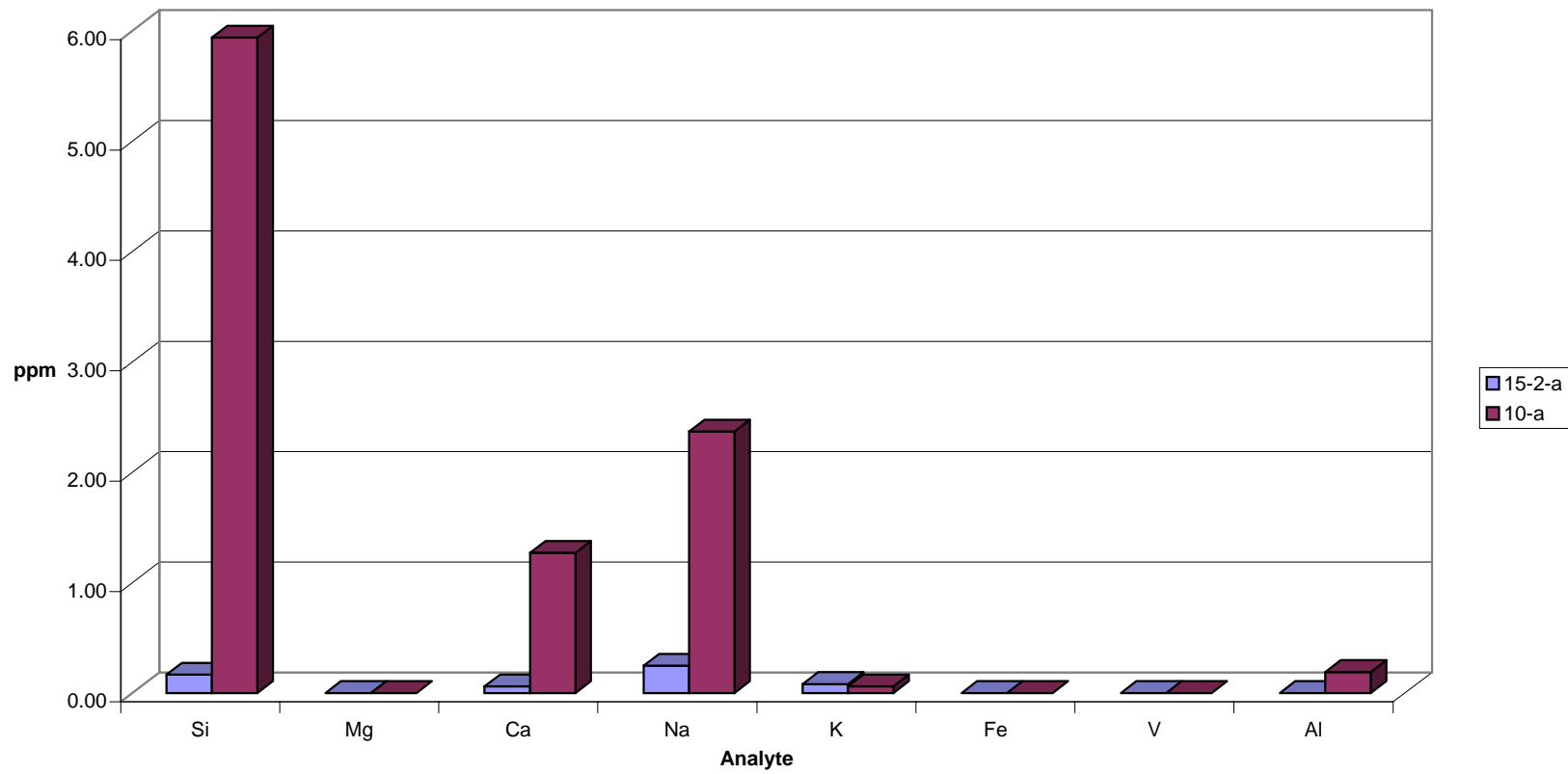


Figure 8: Highest Vs Lowest Overall Release Samples - Baseline Results – White Flint Soda Lime Silica (SLS) containers

6.2.2 Coloured SLS Containers (Baseline Tests)

The results of migration testing for coloured soda lime silica containers are shown graphically in Figure 9. Only elements detected above their reporting limit during testing are listed as migrants. For the purpose of graphical and statistical (averages etc.) treatment results below reporting limit for particular samples are treated as a zero result.

Figure 9 shows that only five elements were found to migrate under baseline conditions at levels above the reporting limits. The results are the average of two independent determinations, carried out by testing each of a matched pair of articles respectively.

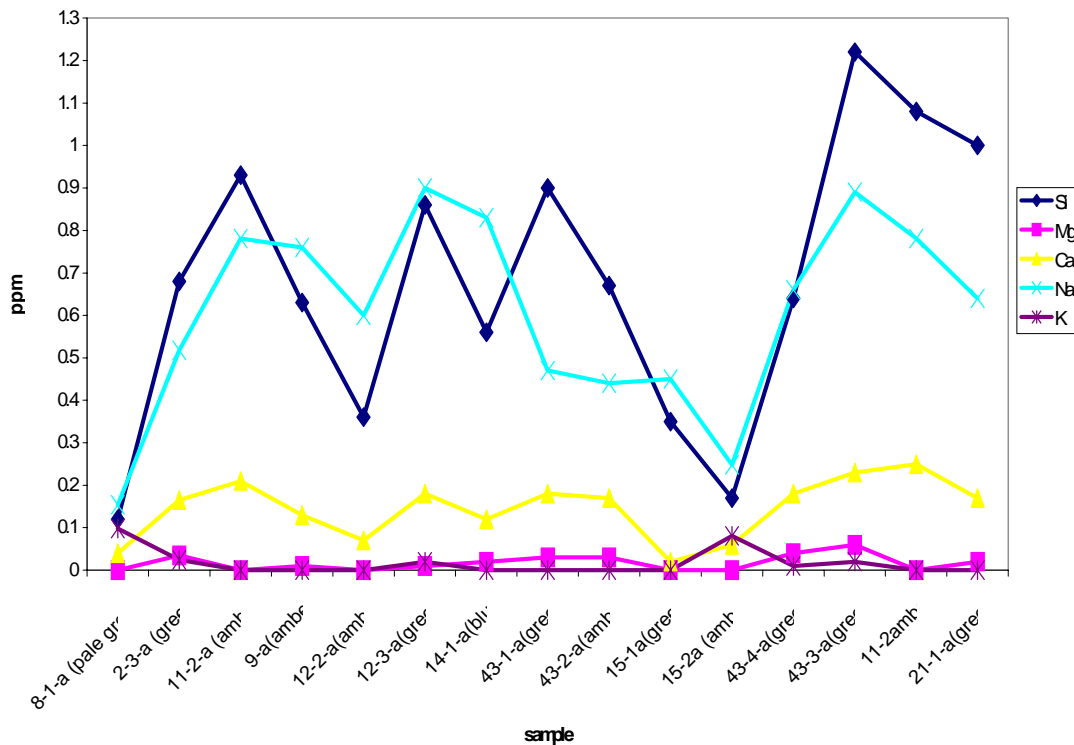


Figure 9: Baseline Migration Results for Coloured SLS Containers

The results of the testing are shown in Table 19

Element	No Of Samples Tested	Number of samples with result equal to or greater than reporting limit	Reporting Limit (ppm)	Min (ppm)	Max (ppm)	Mean (ppm)
Si	15	15	0.1	0.1	1.2	0.7
Mg	15	9	0.01	<0.01	0.06	0.02
Ca	15	15	0.01	0.02	0.25	0.15
Na	15	15	0.02	0.15	0.90	0.61
K	15	6	0.01	<0.01	0.10	0.02

Table 19: Results of Baseline Migration Tests On Coloured Soda Lime Silica Containers

The sample with overall highest release in this category was sample 12-3 which was a green “stubby” 250ml beer bottle produced in the UK. The sample with overall lowest release in this category appears to be sample 8-1 which was a pale green wine bottle produced in France. The release figures for both of these sample are given in Figure 10.

There does not appear to be any evidence of increased migration or migration of contaminant constituents such as Pb when testing coloured articles relative to white flint despite the fact that recycling rates are generally much higher in such ware and as a result, the potential for contamination is greater. Relative recycling levels are discussed in section 4.1.

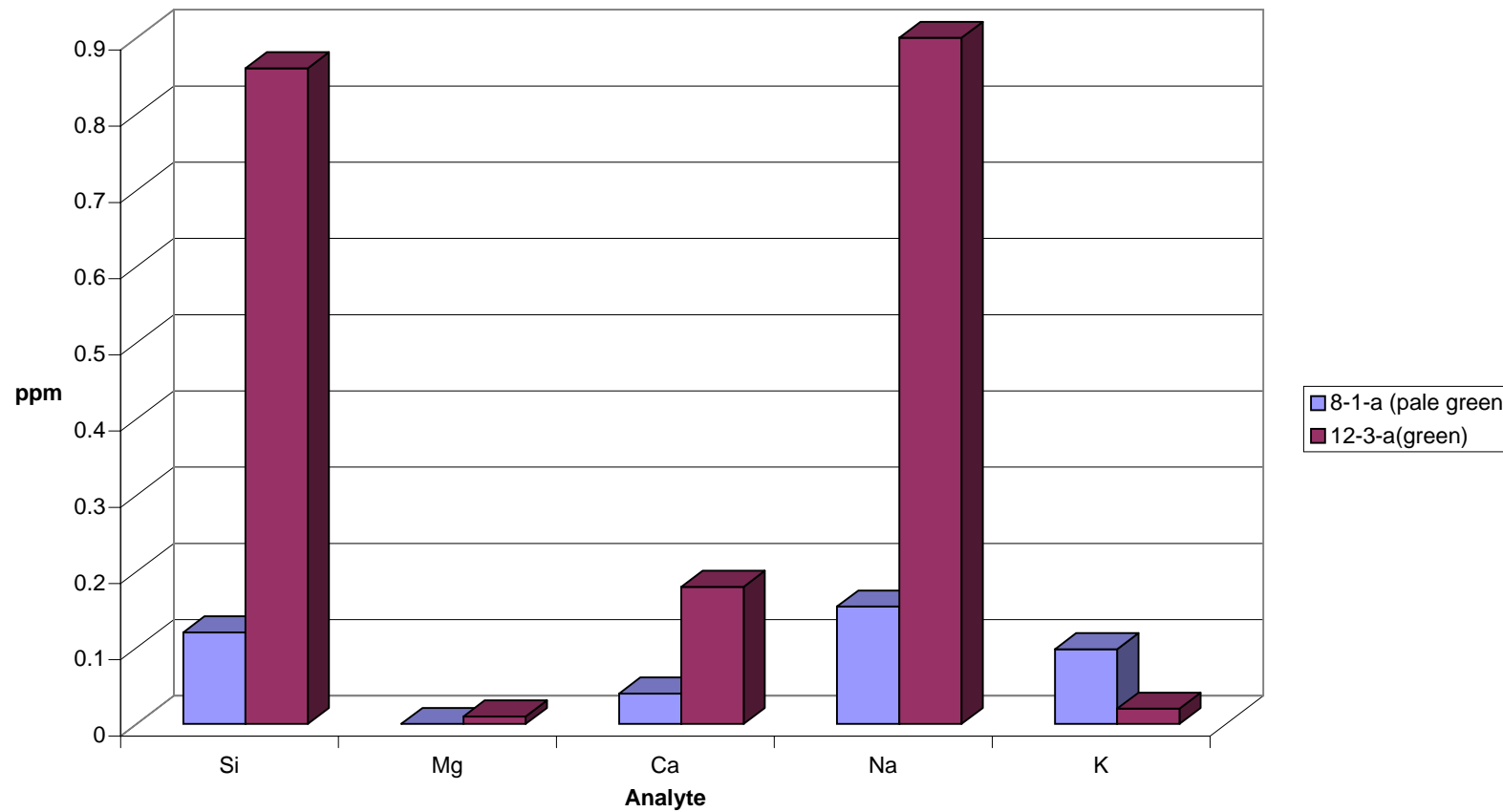


Figure 10: Highest Vs Lowest Overall Release Samples - Baseline Results – Coloured Soda Lime Silica (SLS) Containers.

6.2.3 SLS Tableware/Ovenware (Baseline Tests)

The results of migration testing for soda lime silica tableware/ovenware are shown graphically in Figure 11. Only elements detected above their relevant reporting limit during testing are listed as migrants. For the purpose of graphical treatment results below reporting limit for particular samples are treated as a zero result.

Figure 11 shows that migration of only seven elements were detected under baseline conditions above reporting limits. The results are the average of two independent determinations, carried out by testing each of a matched pair of articles respectively.

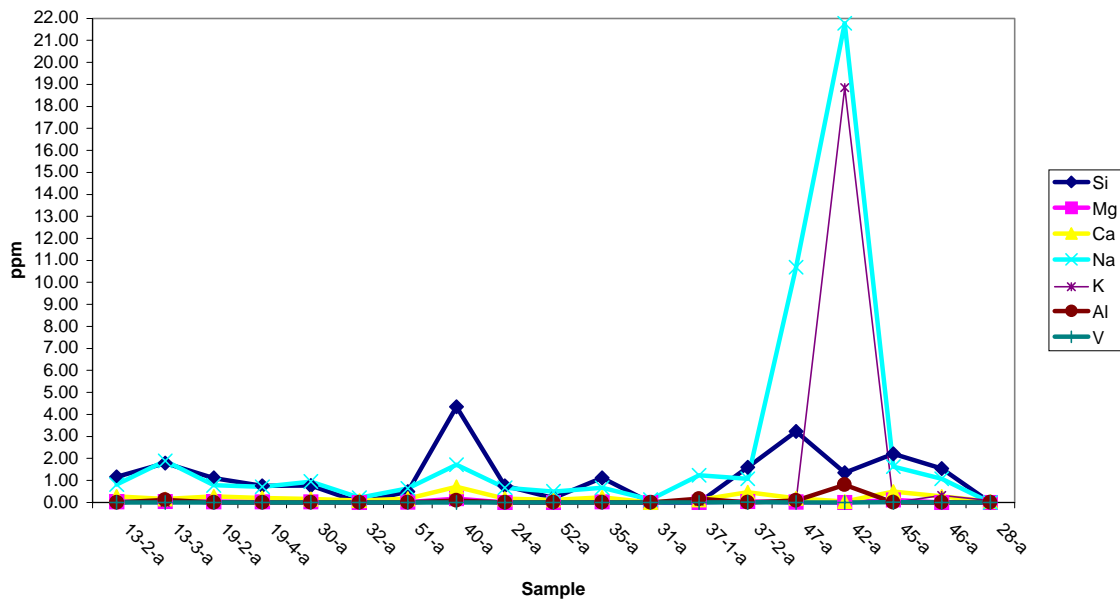


Figure 11: Baseline Migration Results for SLS Tableware/Oven ware

The results of testing are shown in Table 20

Element	No Of Samples Tested	Number of samples with result equal to or greater than reporting limit	Reporting Limit (ppm)	Min (ppm)	Max (ppm)	Mean (ppm)
Si	19	15	0.1	<0.1	4.4	1.2
Mg	19	12	0.01	<0.01	0.18	0.03
Ca	19	16	0.01	<0.01	0.71	0.22
Na	19	18	0.02	<0.02	21.78	2.48
K	19	12	0.01	<0.01	18.85	1.03
V	19	4	0.01	<0.01	0.01	<0.01
Al	19	4	0.05	<0.05	0.80	0.07

Table 20: Results of Baseline Migration Tests On Soda lime Silica

Tableware

The sample with overall highest release in this category was sample 42, which was a shop purchased green glass bowl of unknown origin. The sample with overall lowest release in this category was sample 32 which was a toughened/tempered salad bowl produced in France. The release figures for both of these samples are given in Figure 12.

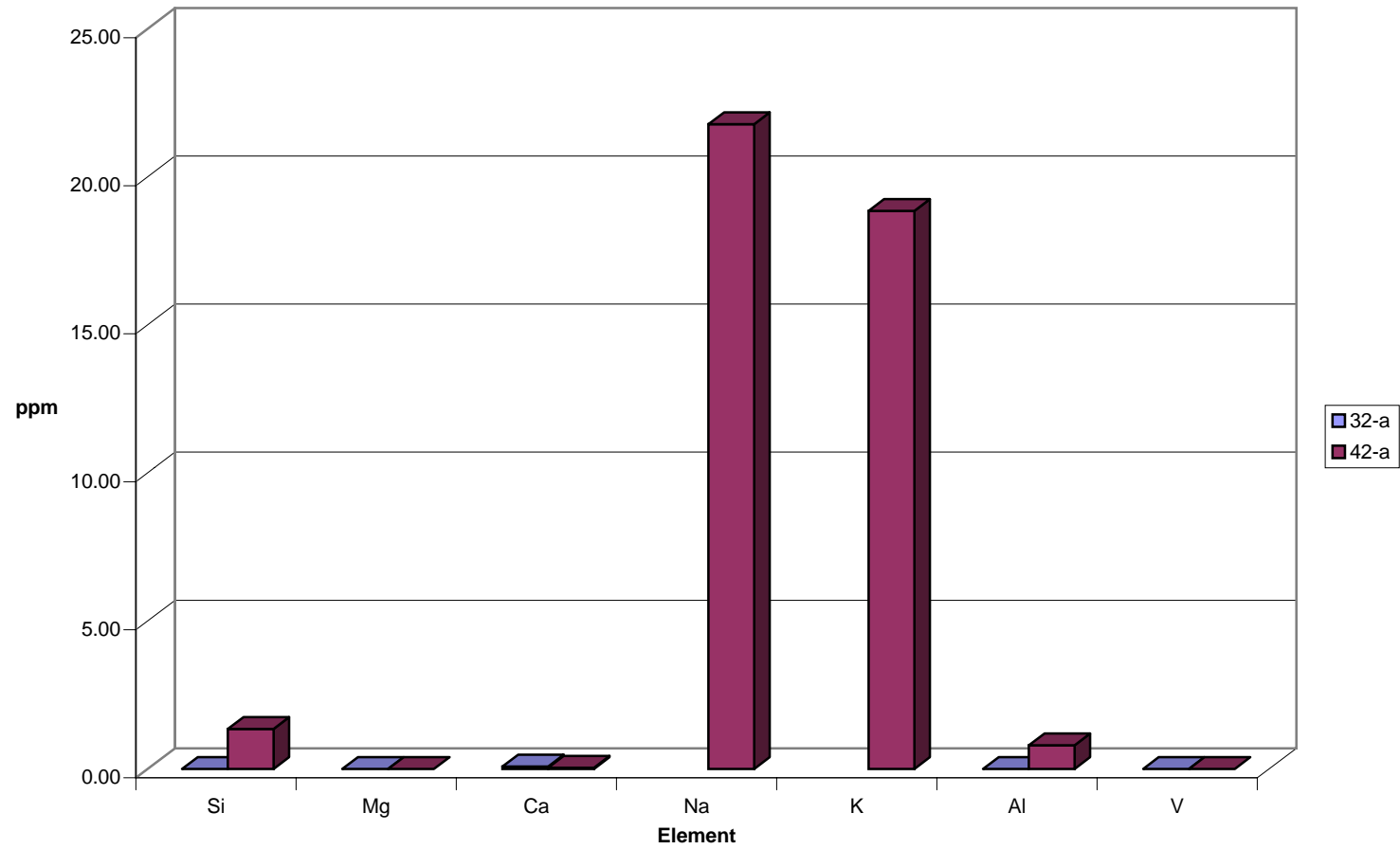


Figure 12: Highest Vs Lowest Overall Release Samples - Baseline Results – SLS Tableware/Ovenware.

6.2.4 Lead Crystal Ware (Baseline Tests)

The results of migration testing for lead crystal ware are shown graphically in Figure 13. Only elements detected above their relevant reporting limit during testing are listed as migrants. For the purpose of graphical treatment results below reporting limit for particular samples are treated as a zero result.

Figure 13 shows that migration of only eight elements was detected under baseline conditions above reporting limits. The results are the average of two independent determinations, carried out by testing each of a matched pair of articles respectively.

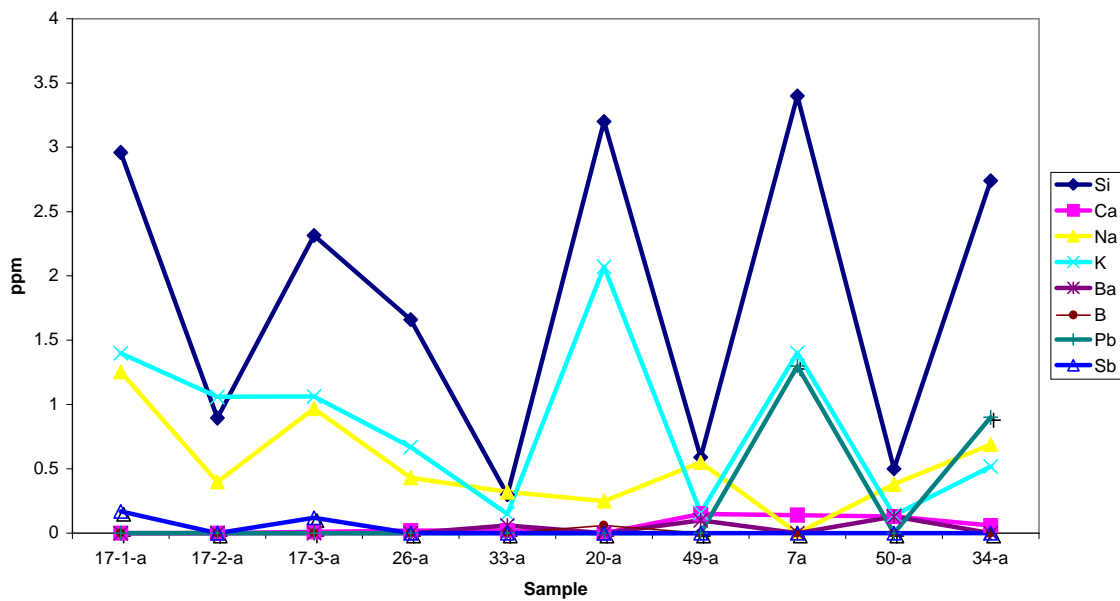


Figure 13: Baseline Migration Results for Lead Crystal Ware

The results of this testing are shown in table 21.

Element	No Of Samples Tested	Number of samples with result equal to or greater than reporting limit	Reporting Limit (ppm)	Min (ppm)	Max (ppm)	Mean (ppm)
Si	10	10	0.1	0.3	3.4	1.9
Ca	10	7	0.01	<0.01	0.15	0.05
Na	10	9	0.02	<0.02	1.26	0.52
K	10	10	0.01	0.14	2.07	0.86
B	10	1	0.03	<0.03	0.06	<0.03
Ba	10	3	0.01	<0.01	0.13	0.03
Pb	10	2	0.1	<0.10	1.3	0.2
Sb	10	2	0.01	<0.01	0.17	0.03

Table 21: Results of baseline Migration Tests On Lead Crystal Ware

The sample with overall highest release in this category was sample 7, which was a 32% lead crystal champagne flute manufactured in the Republic of Ireland.

The sample with overall lowest release in this category was sample 33, which was a spirits tumbler glass produced in the UK. The release figures for both of these samples are given in Figure 14.

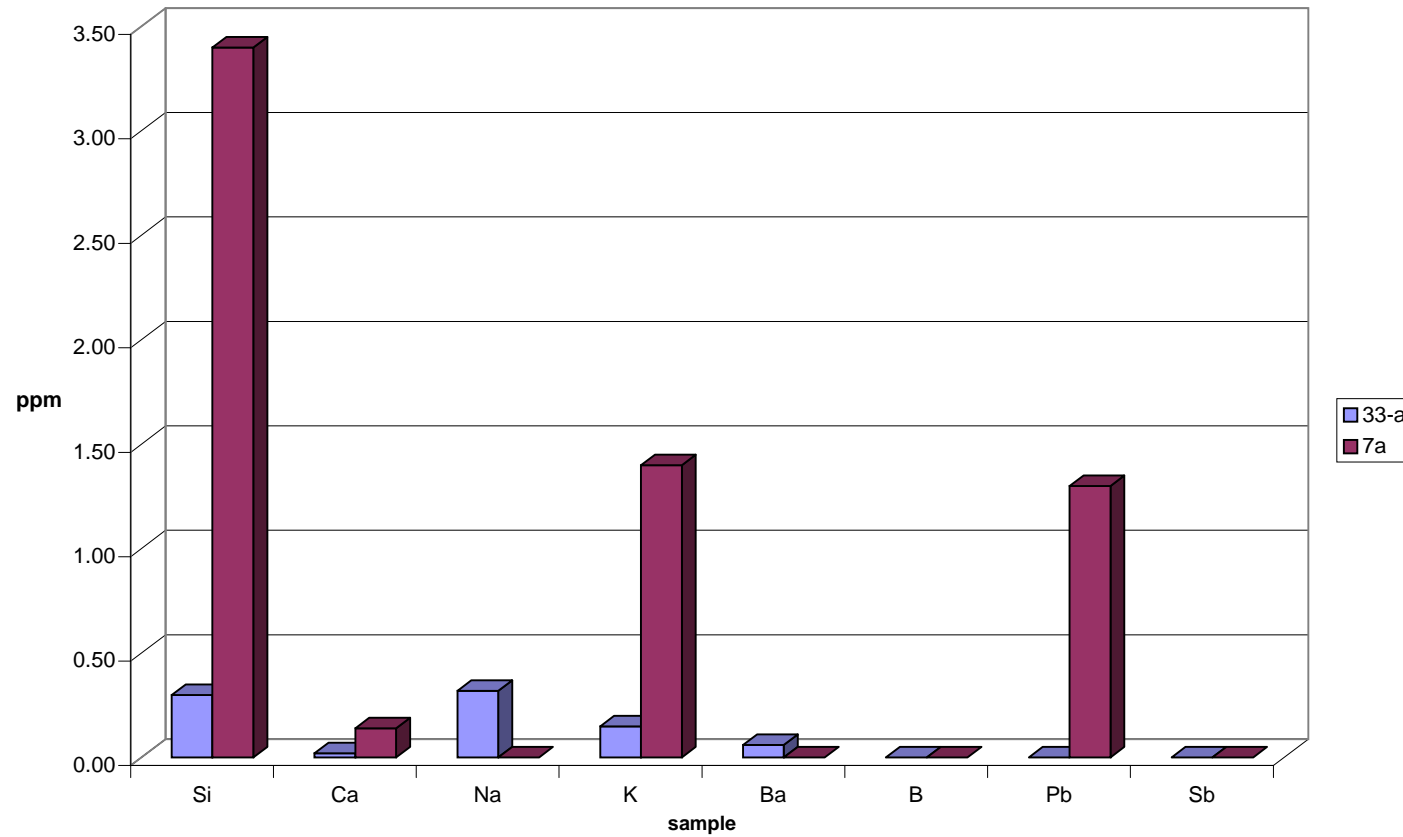


Figure 14: Highest Vs Lowest Overall Release Samples - Baseline Results – Lead Crystal Ware.

6.2.5 Borosilicate & Glass Ceramic Ware (Baseline Tests)

The results of migration testing for Borosilicate and Glass Ceramic Ware are shown graphically in Figure 15. Only elements detected above their relevant reporting limit during testing are listed as migrants. For the purpose of graphical treatment results below reporting limit for particular samples are treated as a zero result. Figure 15 shows that migration of only four elements were detected under baseline conditions above reporting limits. The results are the average of two independent determinations, carried out by testing each of a matched pair of articles respectively.

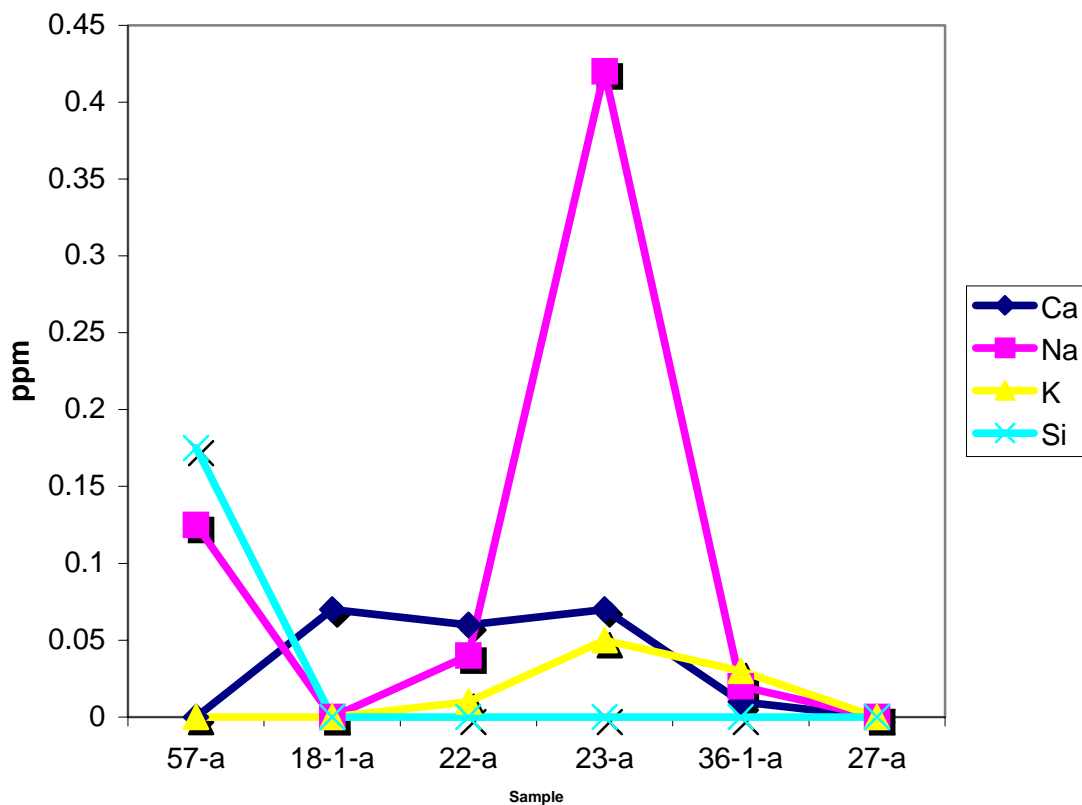


Figure 15: Baseline Migration Results for Borosilicate and Glass Ceramic Ware.

The results of the testing are shown in table 22

Element	No Of Samples Tested	Number of samples with result equal to or greater than reporting limit	Reporting Limit (ppm)	Min (ppm)	Max (ppm)	Mean (ppm)
Si	6	1	0.1	<0.1	0.2	<0.1
Ca	6	4	0.01	<0.01	0.07	0.04
Na	6	4	0.02	<0.02	0.42	0.10
K	6	4	0.01	<0.01	0.05	0.02

Table 22: Results of Baseline Migration Tests On Borosilicate / Glass Ceramic Ware

The sample with overall highest release in this category was sample 23-a, which was a borosilicate of unknown origin. The sample with overall lowest release in this category is sample 27, which was a glass ceramic article, produced in France. The release figures for both of these sample are given in Figure 16.

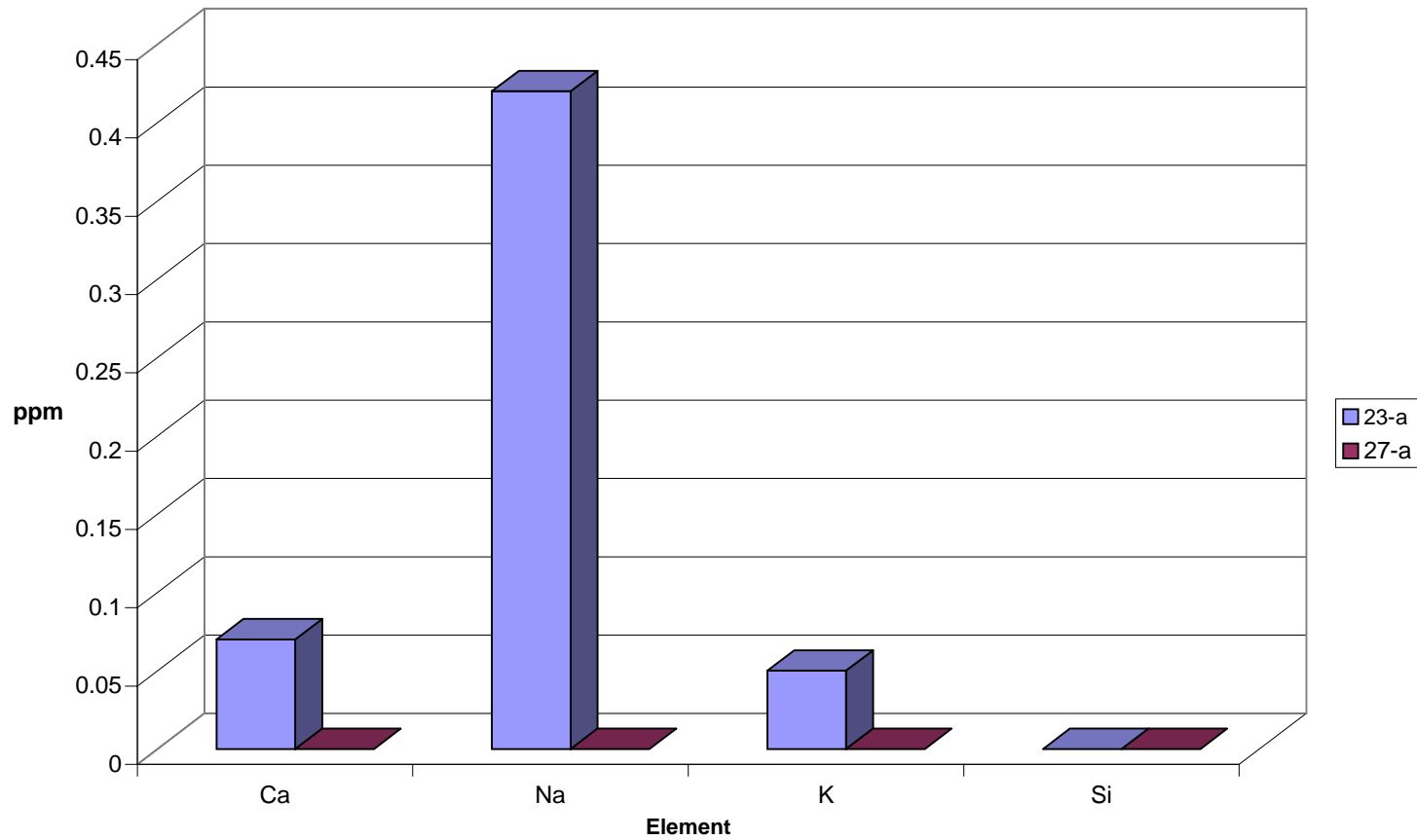


Figure 16: Highest Vs Lowest Overall Release Samples - Baseline Results –Borosilicate / Glass Ceramic Ware.

6.2.6 Decorated Ware (Baseline Tests)

The results of migration testing for decorated ware are shown graphically in Figure 17. Only elements detected above their relevant reporting limit during testing are listed as migrants. For the purpose of graphical treatment results below reporting limit for particular samples are treated as a zero result.

Figure 17 shows that migration of only six elements was detected under baseline conditions above reporting limits. The results are the average of two independent determinations, carried out by testing each of a matched pair of articles respectively.

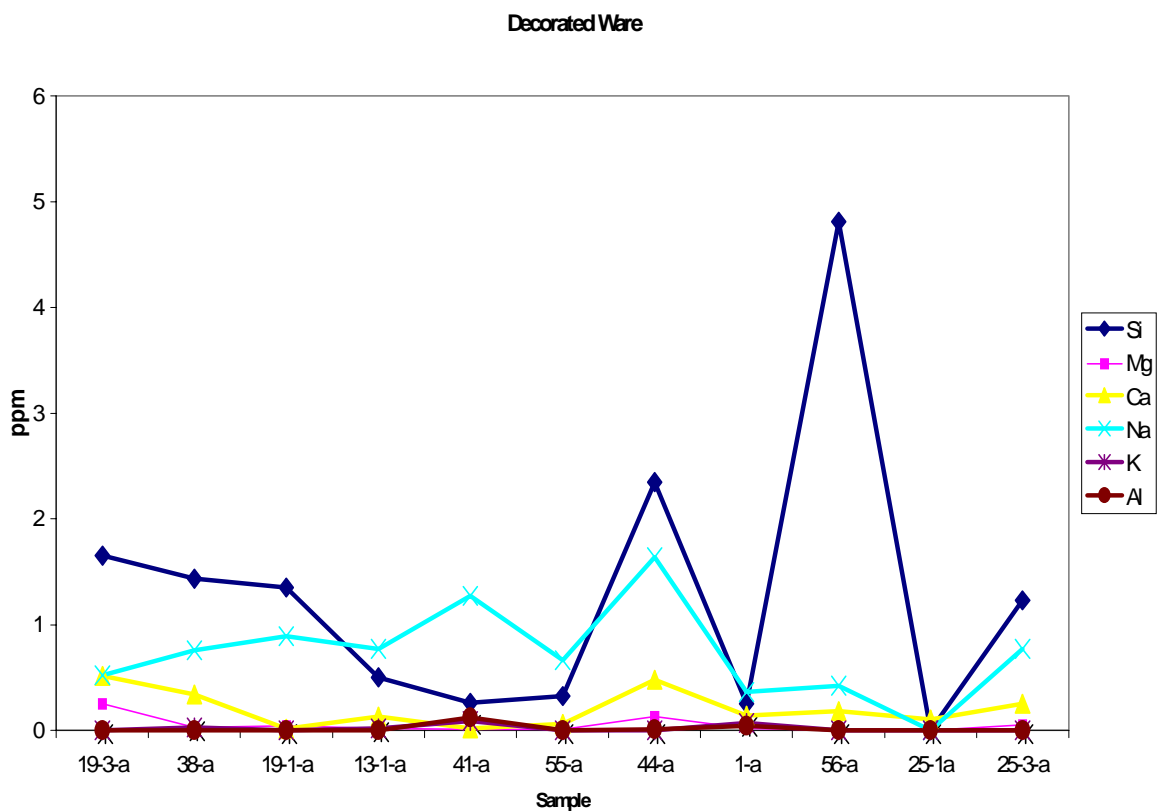


Figure 17: Baseline Migration Results for Decorated Ware

The results of the testing are shown in table 23

Element	No Of Samples Tested	Number of samples with result equal to or greater than reporting limit	Reporting Limit (ppm)	Min (ppm)	Max (ppm)	Mean (ppm)
Si	11	9	0.1	<0.1	4.8	1.3
Mg	11	5	0.01	<0.01	0.25	0.05
Ca	11	11	0.01	0.02	0.52	0.20
Na	11	10	0.02	<0.02	1.64	0.73
K	11	4	0.01	<0.01	0.09	0.02
Al	11	1	0.01	<0.01	0.12	0.01

Table 23: Results of Baseline Migration Tests On Decorated Ware

The sample with overall highest release in this category was sample 56-a, which was a decorated Pb glass article. The sample with overall lowest release in this category appears to be sample 25-1-a, which was a decorated SLS tumbler. The release figures for both of these sample are given in Figure 16. It should be remembered that in these particular tests the filled article does not necessarily have decoration in contact with the test solution, as that decoration may be on the outer surface of the article in question. This was the case for article 56-a.

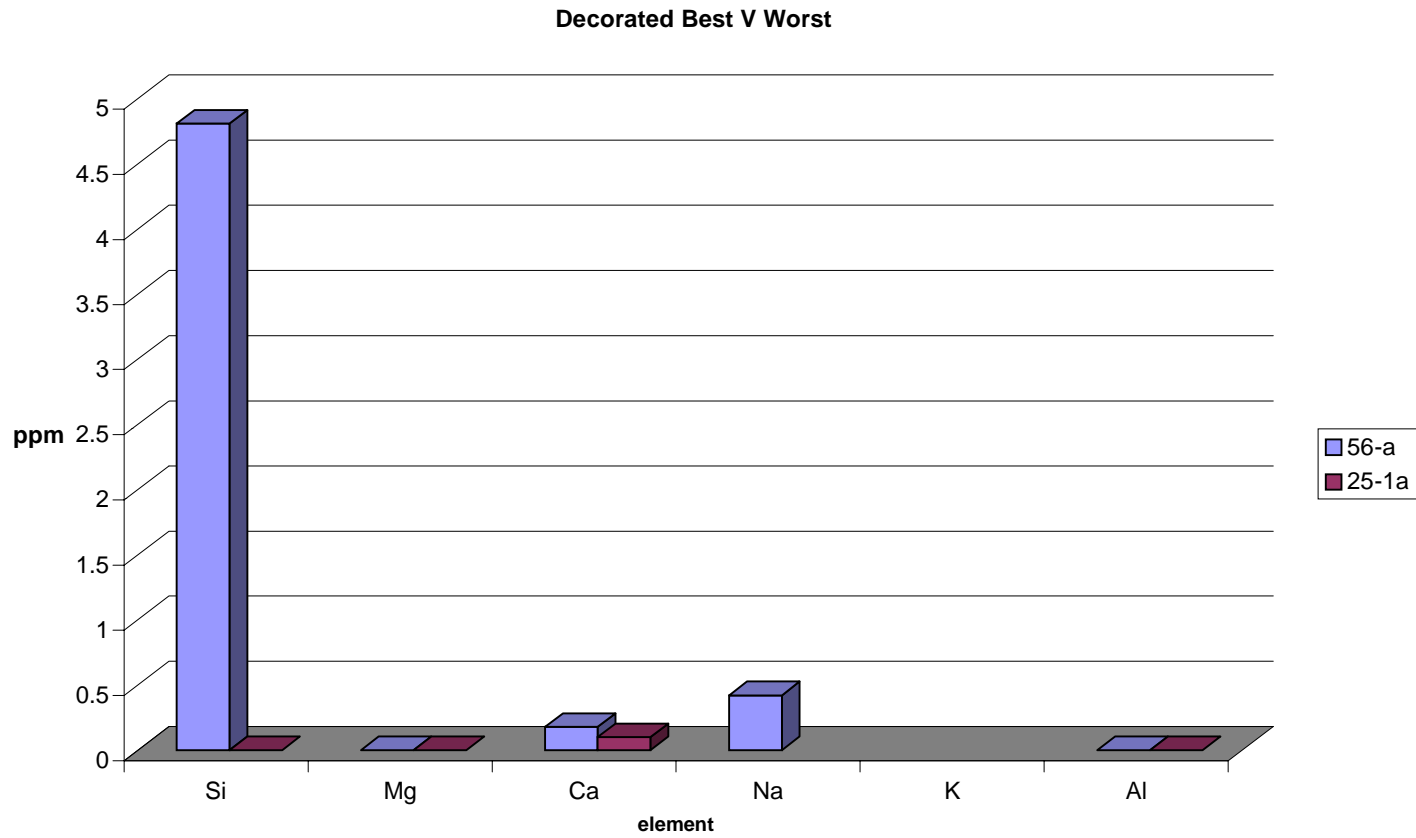


Figure 18: Highest Vs Lowest Overall Release Samples - Baseline Results –Decorated Ware.

6.2.7 Summary Of Baseline Results (Maximum Recorded Values ppm)

The highest recorded results in the baseline testing are recorded in table 22. These results show that the highest overall result was for a piece of soda lime silica tableware (article 42, a shop bought green bowl), which released relatively high levels of sodium and potassium. This article was unusual within its category.

Glass Category	Si	Mg	Ca	Na	K	Fe	V	Al	Ba	B	Pb	Sb
White Flint Soda Lime Silica Container	5.9	0.10	1.27	2.37	0.07	0.03	0.02	0.19	nd	nd	nd	nd
Coloured Soda Lime Silica Container	1.2	0.06	0.25	0.90	0.10	Nd	nd	nd	nd	nd	nd	nd
Soda Lime Silica Table/Ovenware	4.4	0.18	0.71	22.78	18.85	Nd	0.01	0.80	nd	nd	nd	nd
Lead Crystal Ware	3.4	Nd	0.15	1.26	2.07	Nd	nd	nd	0.13	0.06	1.3	0.17
Borosilicate & Glass Ceramic Ware	0.2	Nd	0.07	0.42	0.05	Nd	nd	nd	nd	nd	nd	nd
Decorated Articles	4.8	0.25	0.52	1.64	0.09	Nd	nd	0.12	nd	nd	nd	nd

Table 24: Summary Of Baseline Testing

Nd = not detected at or above reporting limit.

6.3 Surface Damage / Pasteurisation / Caustic Washing

6.3.1 Surface Damage Trials

Surface damage tests were carried out as per the method set out in section 5.2. The results are the average of two independent determinations, carried out by testing each of a matched pair of articles respectively.

6.3.1.1 Soda Lime Silica Containers (White Flint and Coloured)

Table 25 shows the effects of surface damage on soda lime silica containers (both white flint and coloured) with the figures representing the total release and additional release over the recorded baseline figure for each article. (i.e the increase in migration due to surface damage). This increase in elemental release is thought to be related to the increased contact surface area for migration and is shown graphically in Figure 19. Surface damage of soda lime silica containers is possible when in use by the general public. One example would be the introduction of a metal knife in to a jar of mustard.

The analysis shows that for soda lime silica containers surface damage does impart an overall increase in migration. Generally this migration is confined to the elements Si, Mg, Ca, Na and K with less consistent release of Al, V and in one instance Pb also being observed. Release of all elements apart from the three main glass constituent elements Si, Na and Ca is at levels less than 1ppm even for all surface damaged soda lime silica ware. Lead was released from one amber article at 0.3 ppm level after surface damage. The release of this element was not observed in the standard baseline test for this article type. Amber glass does have relatively high recycling rates (discussed in section 4.1) and as a result does have the potential to contain low levels of lead (typically 0 -100ppm).

Analyte	4-1 Total Release	4-1 Additional Migration Over Baseline Test	5-1 Total Release	5-1 Additional Migration Over Baseline Test	6-5 Total Release	6-5 Additional Migration Over Baseline Test	2-2 Total Release	2-2 Additional Migration Over Baseline Test	6-4 Total Release	6-4 Additional Migration Over Baseline Test	12-2 Total Release	12-2 Additional Migration Over Baseline Test	12-3 Total Release	12-3 Additional Migration Over Baseline Test
Si	5.0	3.1	4.9	1.9	6.6	3.9	0.9	0.5	3.2	1.0	2.0	1.6	3.6	2.7
Mg	0.14	0.09	0.14	0.05	0.16	0.09	0.04	0.02	0.09	0.03	0.03	0.03	0.06	0.05
Ca	1.07	0.63	1.12	0.46	1.14	0.62	0.20	0.13	0.68	0.16	0.30	0.23	0.79	0.61
Na	1.78	0.83	1.89	0.30	2.02	0.88	0.68	0.13	1.49	0.50	1.16	0.56	1.74	0.84
K	0.03	0.03	0.05	0.02	0.08	0.08	0.02	0.02	0.04	0.04	0.05	0.05	0.03	0.01
V	0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Al	0.06	0.06	0.09	0.02	0.17	0.17	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
Pb	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	0.3	0.3	<0.1	<0.1

Table 25: Effect of Surface Damage On Soda Lime Silica Containers Tested Under Baseline Conditions (Units ppm)

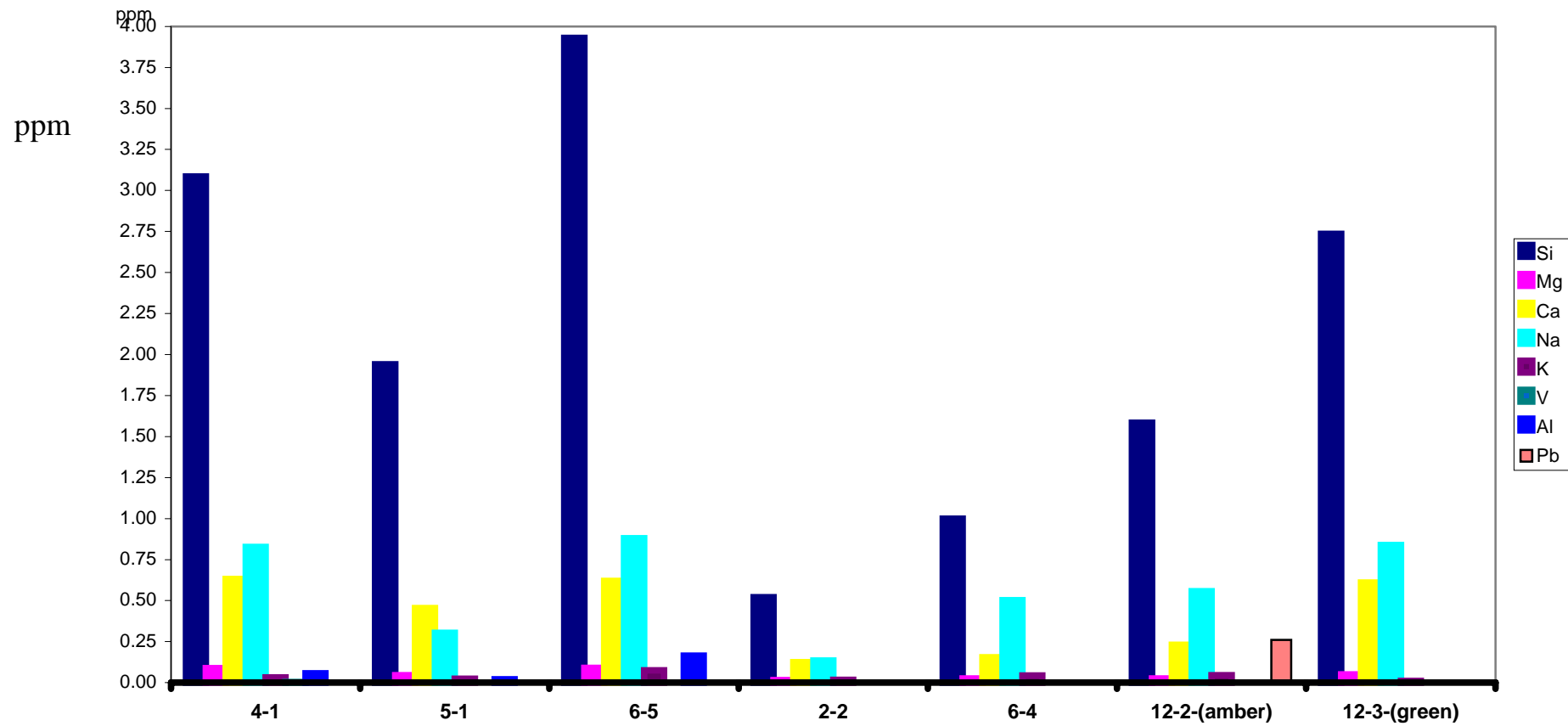


Figure 19: Effect of Surface Damage On Soda Lime Silica Containers -- Additional Release Over Baseline Test

6.3.1.2 Soda Lime Silica Tableware

Tableware / oven to tableware has the potential to be scratched and damaged in everyday use by contact with items such as cutlery or scouring during cleaning. Surface damage testing of soda lime silica tableware is detailed in Table 26 and Figure 20.

Analyte	13-2 Total Release	13-2 Additional Migration Over Baseline Test	37-1 Total Release	37-1 Additional Migration Over Baseline Test	35 Total Release	35 Additional Migration Over Baseline Test
Si	1.9	0.8	0.7	0.7	1.7	0.6
Mg	0.06	0.02	0.03	0.02	0.04	0.01
Ca	0.40	0.12	0.23	0.12	0.36	0.13
Na	1.17	0.36	1.28	0.05	0.81	0.14
K	0.07	0.07	0.06	0.01	0.01	0.01
Al	<0.05	<0.05	<0.05	-0.17	<0.05	<0.05
V	0.01	0.01	<0.01	<0.01	<0.01	<0.01

Table 26: Effect of Surface Damage On Soda Lime Silica Tableware Tested Under Baseline Conditions. (Elemental Release ppm)

Again surface damage does appear to have an influence (i.e to cause a slight increase) on the release of elements from the glass and again this is largely confined to the main glass constituent elements of Si, Na and Ca. In one case there was actually a reduction in the amount of aluminium released in to solution when compared to the baseline tests. However all other elements released from the sample in question were released at higher levels than in the baseline test and this reduction in Al was below 0.2ppm, which suggests that the significance of this result may be low. Vanadium was released in a single sample at the reporting limit of 0.01ppm. No vanadium was released from this sample type when tested in the undamaged state during baseline testing. Vanadium can enter glass at very low levels as a contaminant in fuel oils used to fire furnaces.

Again care should be taken in drawing firm conclusions from results at such low levels.

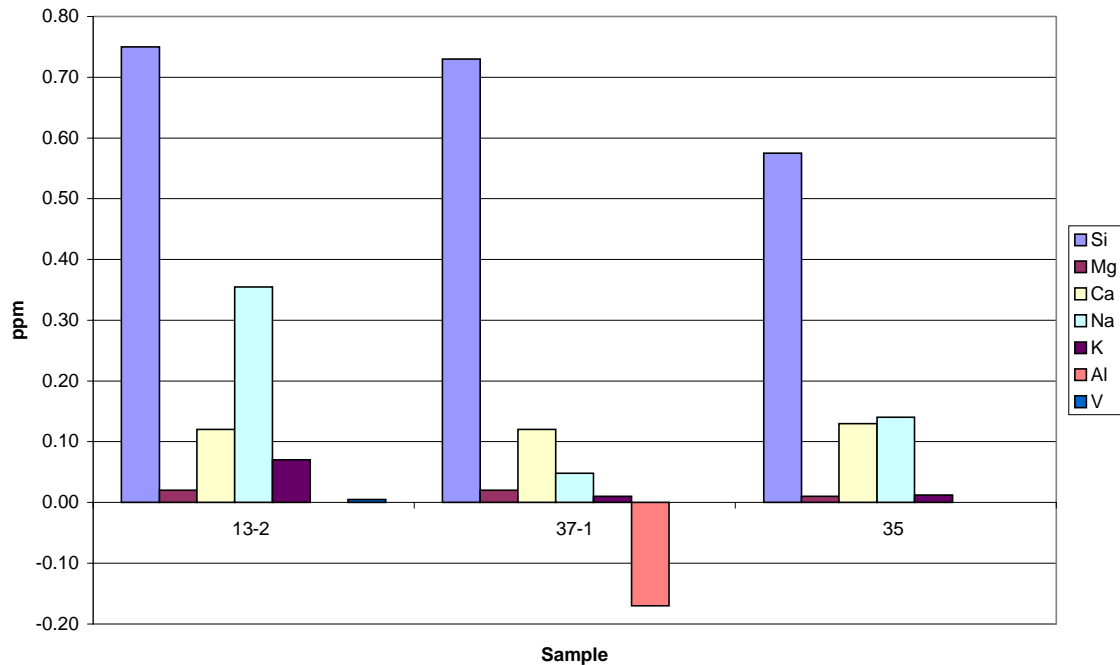


Figure 20: Effect of Surface Damage On Soda Lime Silica Additional Migration Over Baseline Test.

6.3.1.3 Decorated Ware

The effect of surface damage on decorated ware was again to increase the overall elemental release relative to a standard baseline test. The elements released were Si, Mg, Na, K and in one instance 0.05ppm of cobalt was released after surface damage. This sample type did not release detectable levels of cobalt in normal baseline conditions. Again the changes observed were generally small with only Si being released at levels approaching 1ppm greater than undamaged samples tested under baseline test conditions. The results of this testing are shown in Table 27 and Figure 21.

Analyte	1 Total Release	1 Additional Migration Over Baseline Test	13-1 Total Release	13-1 Additional Migration Over Baseline Test	25-3 Total Release	25-3 Additional Migration Over Baseline Test
Si	1.1	0.9	1.5	1.0	1.4	0.2
Mg	0.04	0.02	0.05	0.03	0.06	0.01
Ca	0.15	0.01	0.33	0.20	0.26	0.01
Na	0.53	0.17	0.94	0.17	0.81	0.04
K	0.15	0.08	0.03	0.01	0.01	0.01
Co	0.05	0.05	<0.02	<0.02	<0.02	<0.02

Table 27: Effect of Surface Damage On Decorated Ware Tested Under Baseline Conditions. (Elemental Release ppm)

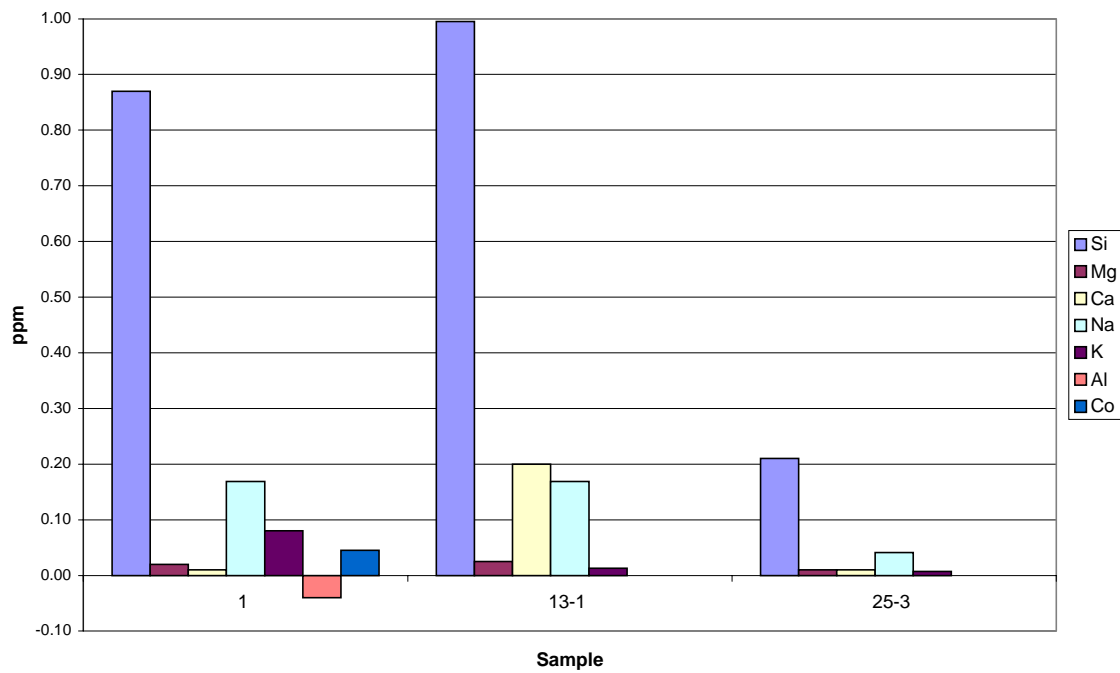


Figure 21: Effect of Surface Damage On Decorated Ware Additional Migration Over Baseline Testing

6.3.1.4 Borosilicate / Glass Ceramic Ware

Borosilicate and glass ceramic cookware have the potential to become surface damaged as a result of repeated contact with cooking utensils and scouring action. The effect of surface damage on borosilicate and glass ceramic ware was less marked than other glass types for the samples tested, leading to a very slight increase in sodium release from sample 27 (glass ceramic) and a slight increase in release of sodium and potassium in the case of sample 22 (borosilicate). There was actually a slight reduction in release of Ca from sample 22 (-0.05ppm) when compared to baseline testing. These changes in overall release between baseline tests and surface damage tests were less than 0.1ppm for all elements detected and are probably of the same order as article to article variability. The overall release from these articles both in their undamaged and damaged state was low suggesting that they are inert materials even relative to other glass compositions. This data is shown in Table 28 and Figure 22.

Analyte	22 Total Release	22 Additional Migration Over Baseline Test	27 Total Release	27 Additional Migration Over Baseline Test
Ca	0.02	-0.05	0.00	0.00
Na	0.07	0.03	0.05	0.05
K	0.03	0.02	0.00	0.00
Si	<0.1	<0.1	<0.1	<0.1

Table 28: Effect of Surface Damage On Borosilicate / Glass Ceramic Ware Under Baseline Test Conditions (Elemental Release ppm)

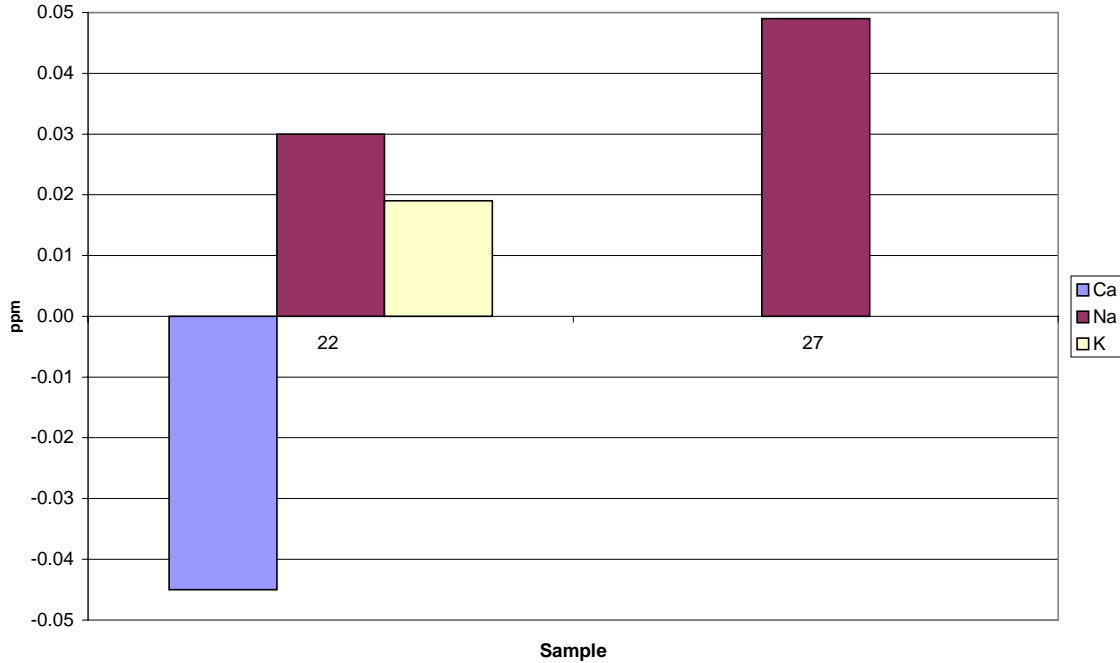


Figure 22: Effect of Surface Damage Borosilicate / Glass Ceramic Tableware Tested Under Baseline Conditions

6.3.1.5 Lead Crystal Ware

The effect of surface damage on lead crystal ware was to generally increase the amount of elemental release for the elements Si, Na (2 out of 3 samples) and K (all samples). In one case there was a small amount of barium detected after surface damage. Barium was not detected in undamaged baseline testing of similar undamaged articles. In two of the three samples tested there was actually a reduction in the amount of lead and calcium released and in one sample a very slight reduction in the amount of boron released. Lead crystal is made in smaller volumes relative to other glass types, and is often “hand worked” which gives greater opportunity for article to article inhomogeneity both in terms of chemical composition and thermal history, than the high volume mass market glasses such as borosilicate and soda lime silica glass. This may explain some of the variation seen.

This data is shown in Table 29 and Figure 23.

Analyte	34 Total Release	34 Additional Migration Over Baseline Test	20 Total Release	20 Additional Migration Over Baseline Test	7 Total Release	7 Additional Migration Over Baseline Test
Si	3.9	1.1	5.9	2.7	6.3	2.9
Ca	0.03	-0.03	0.02	0.02	0.03	-0.11
Na	0.69	0.00	0.43	0.18	0.38	0.38
K	0.82	0.30	3.60	1.53	4.05	2.65
Ba	0.02	0.02	<0.01	<0.01	<0.01	<0.01
B	<0.03	<0.03	0.05	-0.01	<0.03	<0.03
Pb	0.5	-0.4	0.1	0.1	<0.1	-1.3
Sb	<0.10	<0.10	0.14	0.14	<0.10	<0.10

Table 29: Effect of Surface Damage On Lead Crystal Ware Tested Under Baseline Conditions. (Elemental Release ppm)

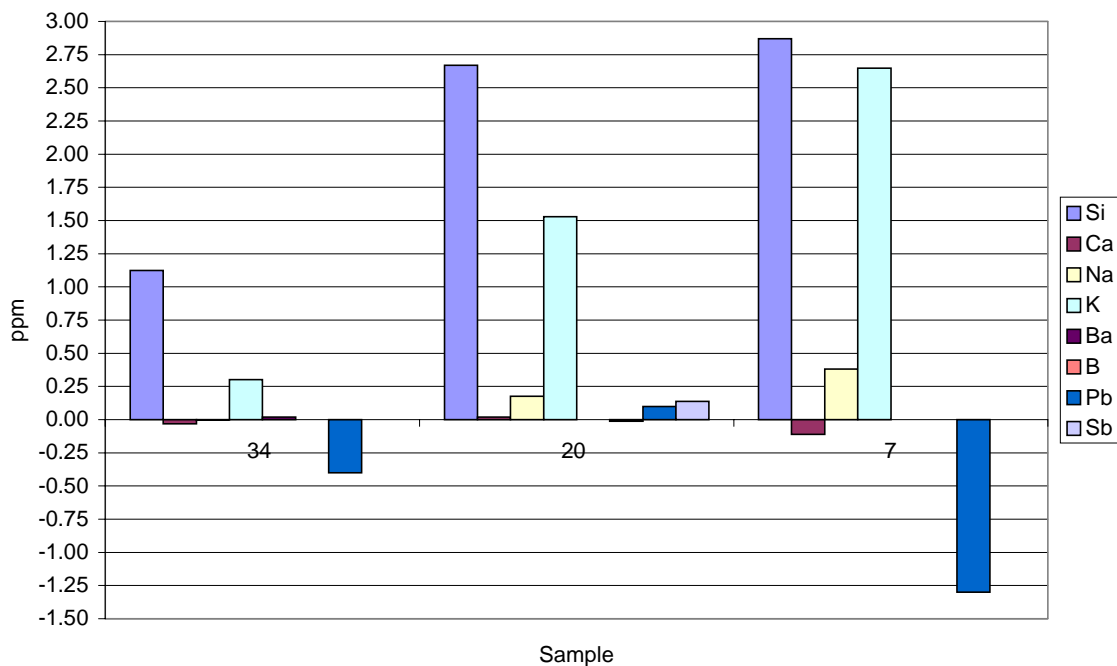


Figure 23: Effect of Surface Damage On Lead Crystal Ware On Migration Additional To Baseline Result.

6.3.2 Caustic Washing Trials

Caustic washing trials were restricted to soda lime silica containers as this treatment is used only in the washing of returnable bottles. There does not seem to be a consistent effect with caustic washing. However changes in behaviour appear to be generally small. The biggest difference recorded over original baseline results was a reduction of Si release of 1.5ppm for a white flint soda lime silica container. Sodium release is lower in all instances after caustic washing but again differences are below 1ppm between samples that have and have not been caustic washed. In two instances (sample 2-1 and sample 11-1) V was released at the reporting limit of 0.01ppm for the original (not caustic washed) baseline test, however no vanadium was detected in any of the caustic washing samples. Release appears to be limited to key glass composition components Si, Na, Mg, Ca and K (with no Al or colourants such as Cr, Ni, Fe being detected)

Table 30 and Figure 24 show the differences between the recorded results after caustic washing and original baseline values. A negative figure denotes a lower migration for that particular element relative to the baseline migration for the article type in question.

Analyte	2-1	11-1	12-1	43-3	15-1	43-2	2-3	12-2	12-3	14-1	11-2	15-2
Si	-1.46	0.07	0.52	-0.58	0.13	-0.49	0.23	0.62	0.40	0.10	-0.24	0.01
Mg	0.28	0.01	0.02	0.30	0.06	0.03	0.13	0.02	0.04	0.25	0.02	0.06
Ca	-0.29	-0.03	0.14	-0.04	0.11	-0.11	0.03	0.14	0.10	0.06	-0.08	0.11
Na	-0.74	-0.25	-0.09	-0.80	-0.40	-0.44	-0.25	-0.14	-0.28	-0.49	-0.39	-0.24
K	0.09	0.19	0.05	0.01	0.14	0.03	-0.02	0.23	0.03	0.06	0.13	0.01
V	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01

Table 30: Difference in Migration Result After Caustic Washing Relative to Original Baseline Migration. (Units ppm)

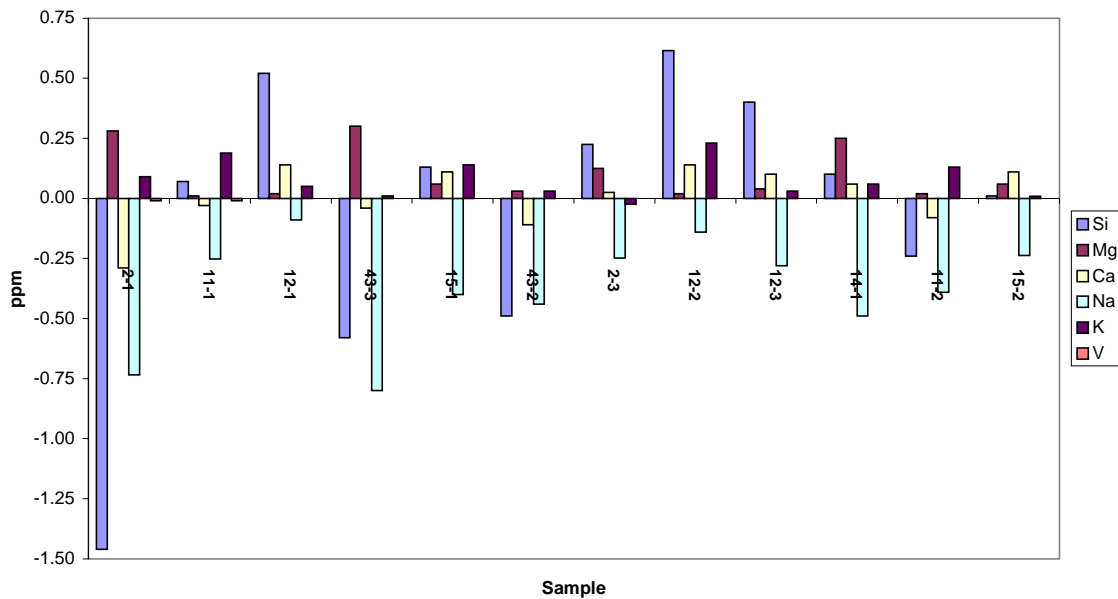


Figure 24. Effect Of Caustic Washing Relative To Baseline Results For Soda Lime Silica Containers.

6.3.3 Pasteurisation Tests

Pasteurisation tests were limited to soda lime silica containers. This article type generally undergoes pasteurisation treatment in real use situations. As can be seen from Tables 31 and 32 and Figures 25 & 26, the pasteurisation process appears to lead to a lower elemental release than that observed under baseline conditions. This is not surprising when considering the reduced test temperature involved. (See section 5 for detailed methods)

Analyte	2-3a	2-3e	15-1a	15-1e	8-1a	8-1e	11-2a	11-2e	12-2a	12-2e	12-3a	12-3e	14-1a	14-1e	43-3a	43-3e	15-2a	15-2e
Si	0.7	<0.1	0.4	<0.1	0.1	<0.1	1.1	0.2	0.4	<0.1	0.9	0.2	0.6	<0.1	1.2	0.2	0.2	<0.1
Mg	0.04	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.01	0.01	0.02	<0.01	0.06	0.04	<0.01	<0.01
Ca	0.17	0.03	0.02	0.04	0.04	0.03	0.25	0.09	0.07	0.05	0.18	0.10	0.12	0.05	0.23	0.07	0.06	0.05
Na	0.52	0.14	0.45	0.08	0.15	0.16	0.78	0.36	0.60	0.20	0.90	0.22	0.83	0.16	0.89	0.21	0.25	0.17
K	0.02	<0.01	<0.01	<0.01	0.10	<0.01	<0.01	0.04	<0.01	<0.01	0.02	0.04	<0.01	<0.01	0.02	<0.01	0.08	<0.01

Table 31: Baseline (a) and Pasteurisation (e) Results For Coloured SLS Containers (Elemental Release ppm)

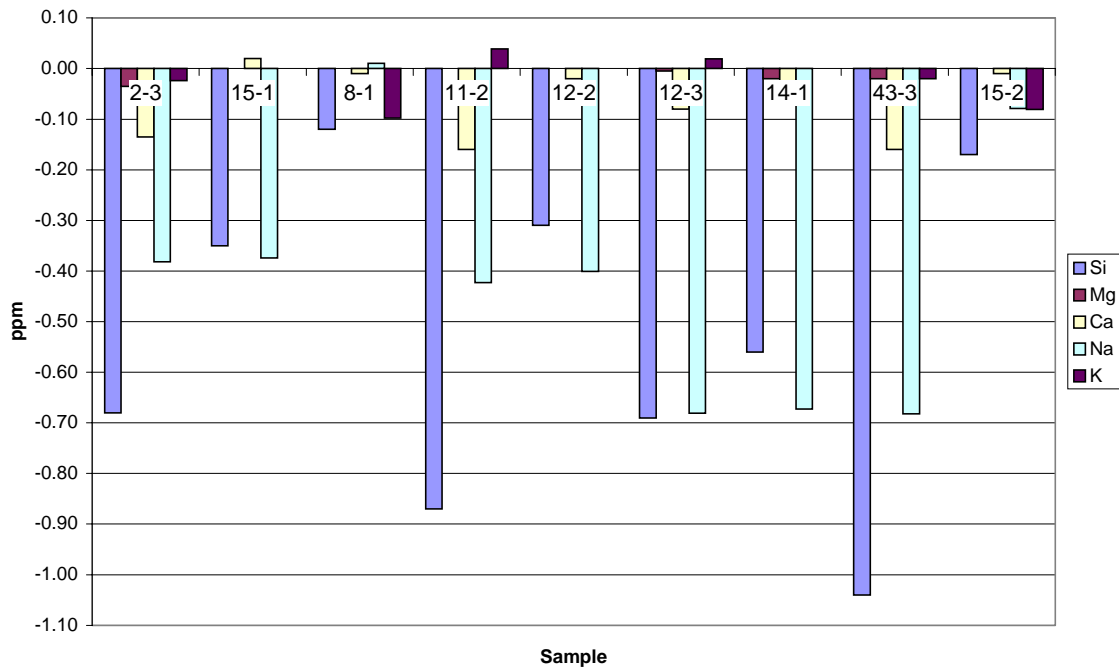


Figure 25. Effect Of Pasteurisation Relative To Baseline Results For Coloured Soda Lime Silica Containers.

Analyte	2-1a	2-1e	2-2a	2-2e	4-1a	4-1e	4-3a	4-3e	5-1a	5-1e	11-1a	11-1e	12-1a	12-1e	6-2a	6-2e	6-3a	6-3e
Si	2.0	<0.1	0.4	<0.1	1.9	0.2	1.0	0.3	3.0	0.5	1.8	0.9	1.1	0.3	1.7	0.3	3.5	0.8
Mg	0.08	0.05	0.02	<0.01	0.06	0.01	0.03	0.05	0.09	0.02	<0.01	<0.01	0.01	<0.01	0.05	0.01	0.10	0.03
Ca	0.47	0.05	0.07	0.03	0.44	0.08	0.22	0.07	0.66	0.16	0.45	0.25	0.23	0.09	0.38	0.09	0.68	0.23
Na	0.99	0.17	0.55	0.08	0.96	0.19	0.67	0.29	1.59	0.47	1.00	0.68	0.79	0.35	0.86	0.32	1.41	0.56
K	<0.01	<0.01	<0.01	0.43	0.02	0.08	<0.01	0.03	0.03	0.09	0.02	0.07	<0.01	0.03	0.04	0.14	0.06	<0.01
V	0.01	<0.01	<0.01	<0.01	0.01	<0.01	<0.01	<0.01	0.02	<0.01	0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Al	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	0.07	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05

Table 32: Baseline (a) and Pasteurisation (e) Results For White Flint SLS Containers (Elemental Release ppm)

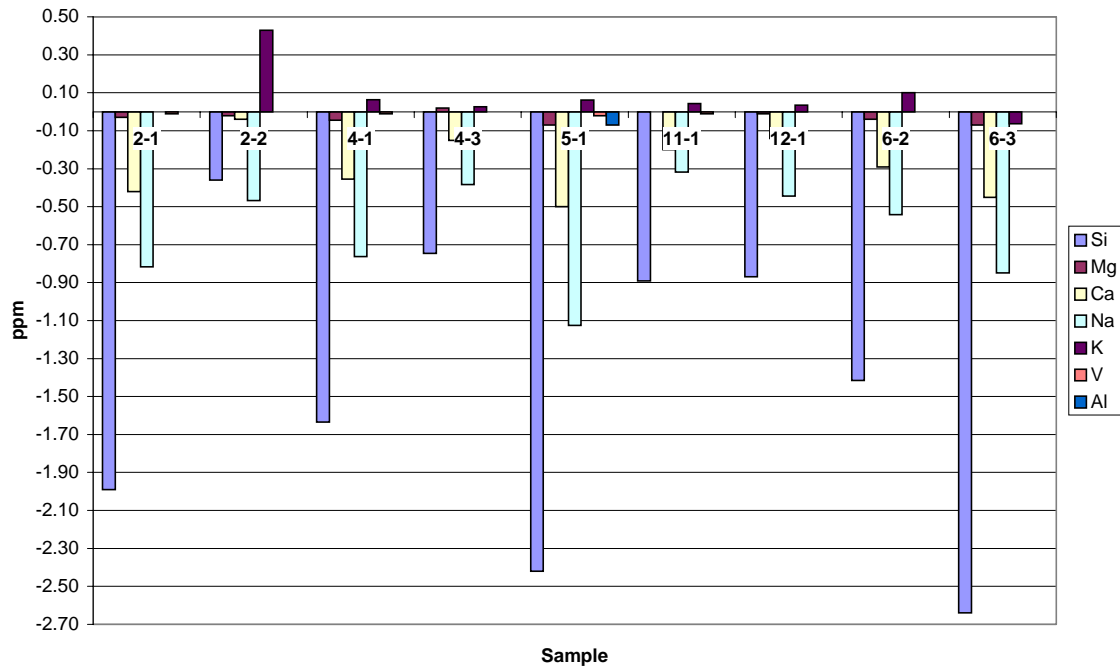


Figure 26. Effect Of Pasteurisation Relative To Baseline Results For White Flint Soda Lime Silica Containers

6.3 Food Simulants And FoodStuffs

The results are the average of two independent determinations, carried out by testing each of a matched pair of articles respectively.

6.3.1 Acetic Acid Testing

Testing was carried out as detailed in section 5.3

6.4.1.1 Acetic Acid Testing of Soda Lime Silica Containers

Acetic acid migration testing using the conditions described in section 5.3 has led to an overall reduced migration for soda lime silica containers. The data from these tests is shown in Table 33. This is shown clearly in Figure 27.

Analyte	21-1a	21-1b	43-1a	43-1b	43-2a	43-2b	43-3a	43-3b	43-4a	43-4b	14-1a	14-1b	21-2a	21-2b
Si	1.0	<0.1	0.9	<0.1	0.7	<0.1	1.2	<0.1	0.6	<0.1	0.6	0.1	1.0	<0.1
Mg	0.02	<0.01	0.03	0.01	0.03	0.01	0.06	0.04	0.04	0.02	0.02	0.03	<0.01	<0.01
Ca	0.17	0.04	0.18	0.05	0.17	0.06	0.23	0.17	0.18	0.10	0.12	0.21	0.25	0.06
Na	0.64	0.28	0.47	0.25	0.44	0.23	0.89	0.32	0.66	0.30	0.83	0.38	0.62	0.23
K	<0.01	<0.01	<0.01	<0.01	<0.01	0.02	0.02	0.01	0.01	<0.01	<0.01	0.02	<0.01	<0.01
Fe	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	0.03	<0.02

Table 33: Baseline (a) and Acetic Acid (b) Results For SLS Containers (Elemental Release ppm)

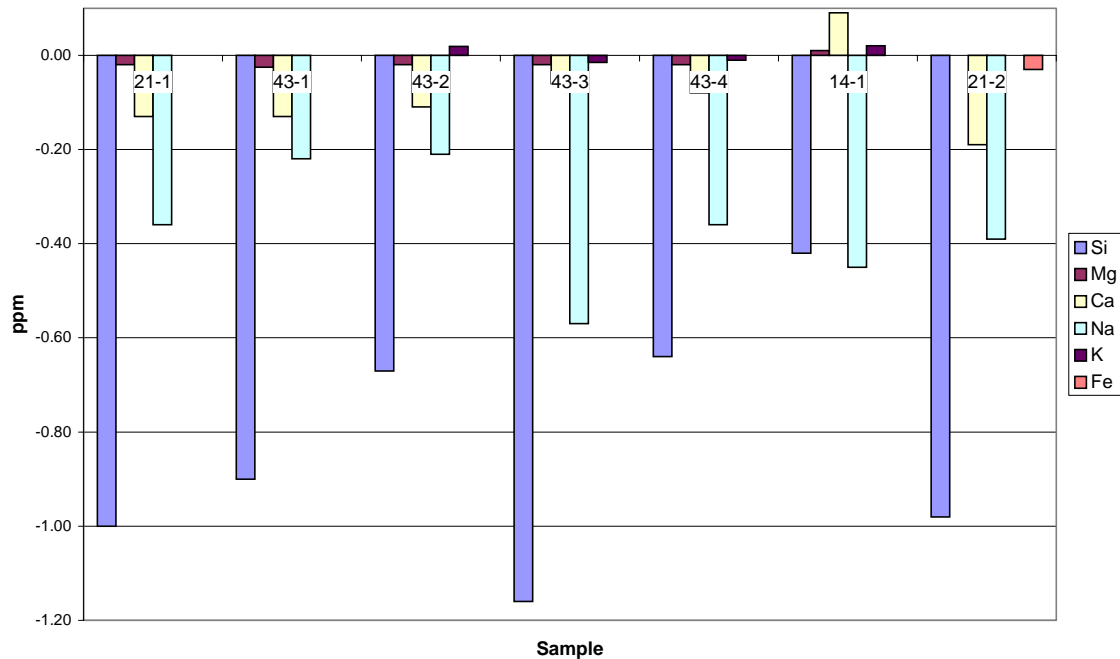


Figure 27: Acetic Acid results Relative To Baseline Results For SLS Containers (Units ppm)

6.4.1.2 Acetic Acid Testing Of Soda Lime Silica Tableware

A similar effect was generally observed with soda lime silica tableware tested with acetic acid as with soda lime silica containers (i.e a general reduction in migration – see Table 34 and Figure 28). The acetic acid tests released Pb from three of the four samples tested in the range of 0.4 to 1.2 ppm. Pb was not released in the corresponding baseline migration test.

Analyte	37-1-a	37-1-b	13-3-a	13-3-b	19-2-a	19-2-b	19-4-a	19-4-b
Si	<0.1	<0.1	1.8	<0.1	1.1	0.1	0.8	<0.10
Mg	0.01	0.01	0.04	0.02	0.04	0.02	0.03	0.02
Ca	0.11	0.19	0.17	0.10	0.28	0.16	0.20	0.10
Na	1.23	0.31	1.89	0.79	0.78	0.50	0.71	0.44
K	0.05	0.02	<0.01	<0.01	<0.01	0.04	<0.01	0.03
Al	0.17	<0.05	0.12	0.08	<0.05	0.09	<0.05	<0.05
Pb	<0.1	<0.1	<0.1	1.2	<0.1	0.4	<0.1	0.7
Fe	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	0.02
V	<0.01	<0.01	<0.01	<0.01	0.01	<0.01	<0.01	<0.01

Table 34: Baseline (a) and Acetic Acid (b) Results For SLS Tableware (Units ppm)

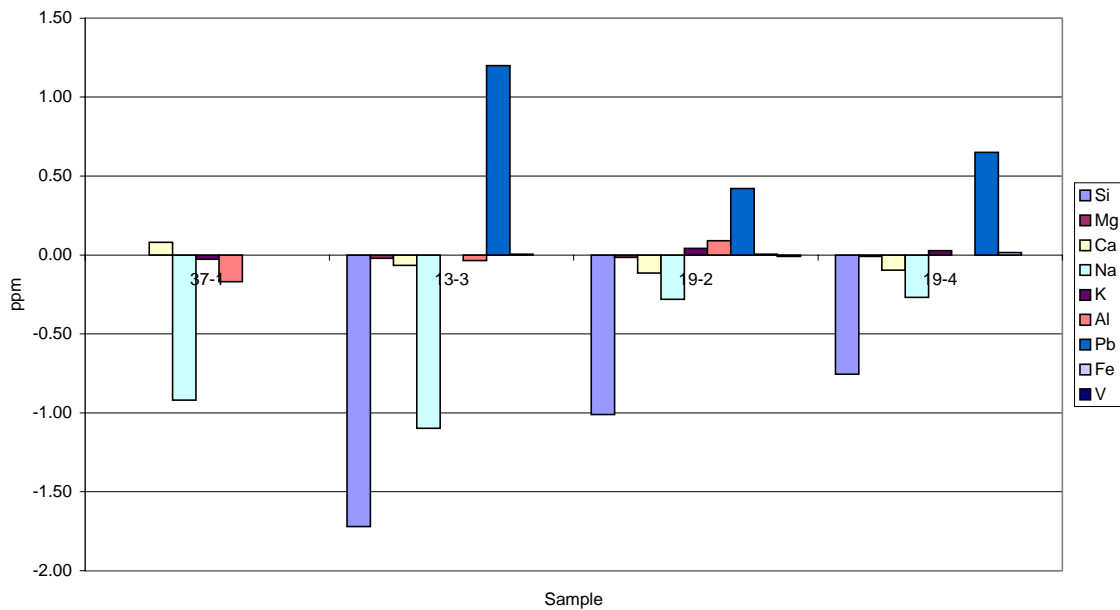


Figure 28: Acetic Acid results Relative To Baseline Results For SLS Tableware (Units ppm)

6.4.1.3 Acetic Acid Testing Of Lead Crystal Ware

Significant recent work already exists on the effects of acetic acid and other simulants on lead crystal ware and as a result testing was restricted to a single article. Acetic acid appears to have led to the release of Pb, Fe and V at sub 1ppm levels, which was not detected in baseline tests. At the same time there has been a drop in the release of Si, Na and K from this article type on acetic acid testing relative to baseline testing. This would support the literature discussed in section 3.2 suggesting that acetic acid is effective for assessing the release of lead from glassware.

The results of this testing are shown in Table 35.

Analyte	17-1-a	17-1b
Si	3.0	<0.1
Ca	<0.01	<0.01
Na	1.26	0.94
K	1.40	1.09
Ba	<0.01	<0.01
B	<0.03	<0.03
Pb	<0.1	0.9
Fe	<0.02	0.68
V	<0.01	0.04
Sb	0.17	<0.01

Table 35: Baseline (a) and Acetic Acid (b) Results For Lead Crystal Ware (Units ppm)

6.4.1.4 Acetic Acid Testing Of Borosilicate And Glass Ceramic Ware

One article of each type was selected for testing with acetic acid, sample 27 a glass ceramic cookware dish and sample 36, a borosilicate cookware dish. Both articles showed high migration resistance with acetic acid as the test medium. This observed performance was similar to that obtained with water as the attacking medium in the original baseline testing. These results again suggest that this category of ware is inert even relative to other glass types.

The results of this testing are shown in Table 36

Analyte	27-a	27-b	36-a	36-b
Ca	<0.01	<0.01	0.01	0.12
Na	<0.02	<0.02	0.02	<0.02
K	<0.01	<0.01	0.03	0.01

Table 36: Baseline (a) and Acetic Acid (b) Results For Glass Ceramic and Borosilicate Tableware (Units ppm)

6.4.1.5 Acetic Acid Testing Of Decorated Ware

The results for acetic acid testing (method described in section 5.3) for elemental release from the decorated ware are shown relative to the original baseline figures for that particular type of article tested. (see Table 37 and Figure 29). This acetic acid attack does appear to lead to appreciable elemental release for one article type in particular. Article 1, a ceramic slip/glaze decorated glass bowl manufactured in Greece is of particular interest as acetic acid testing has led to the release of 228ppm of Al along with 5ppm of Pb, 2.5ppm of Fe, 0.7ppm of Co, 0.4ppm of Zn and 0.9ppm of Mn. This was an article included after discussion with the Food Standards Agency in which it was requested that decorated articles such as bowls, that might be intended as ornamental articles but which could perhaps also be used to hold foodstuffs, be included in the testing. It would appear from this testing that the decoration covering this article (which in this case was in full contact with the test solution) can release high levels of elemental migrants into acetic acid.

None of these elements were released above the reporting limit when this particular design of glass bowl was tested under baseline conditions with water as the attacking medium. Elemental release was lower for the other decorated articles tested using acetic acid. Lead was also released from articles 19-3, a decorated SLS tumbler (1.2ppm) and 19-1 (1.0ppm), a decorated SLS mug. Cd was also released from this article (0.03ppm).

Analyte	19-3-a	19-3b	19-1-a	19-1b	1-a	1b
Si	1.7	0.2	1.4	0.1	0.3	1.7
Mg	0.25	0.25	0.04	0.03	0.02	0.32
Ca	0.52	0.07	0.02	0.19	0.14	0.84
Na	0.52	0.34	0.89	0.49	0.36	4.49
K	<0.01	0.03	<0.01	0.02	0.07	1.91
Mn	<0.01	<0.01	<0.01	<0.01	<0.01	0.91
Fe	<0.02	0.02	<0.02	<0.02	<0.02	2.48
Cu	<0.03	<0.03	<0.03	<0.03	<0.03	0.03
Zn	<0.03	<0.03	<0.03	<0.03	<0.03	0.36
Pb	<0.10	1.2	<0.1	1.0	<0.1	5.0
Co	<0.02	<0.02	<0.02	<0.02	<0.02	0.74
Cd	<0.01	<0.01	<0.01	0.03	<0.01	<0.01
Al	<0.05	<0.05	<0.05	<0.05	<0.05	228.19

Table 37: Baseline (a) and Acetic Acid (b) Results For Decorated Ware (Units ppm)

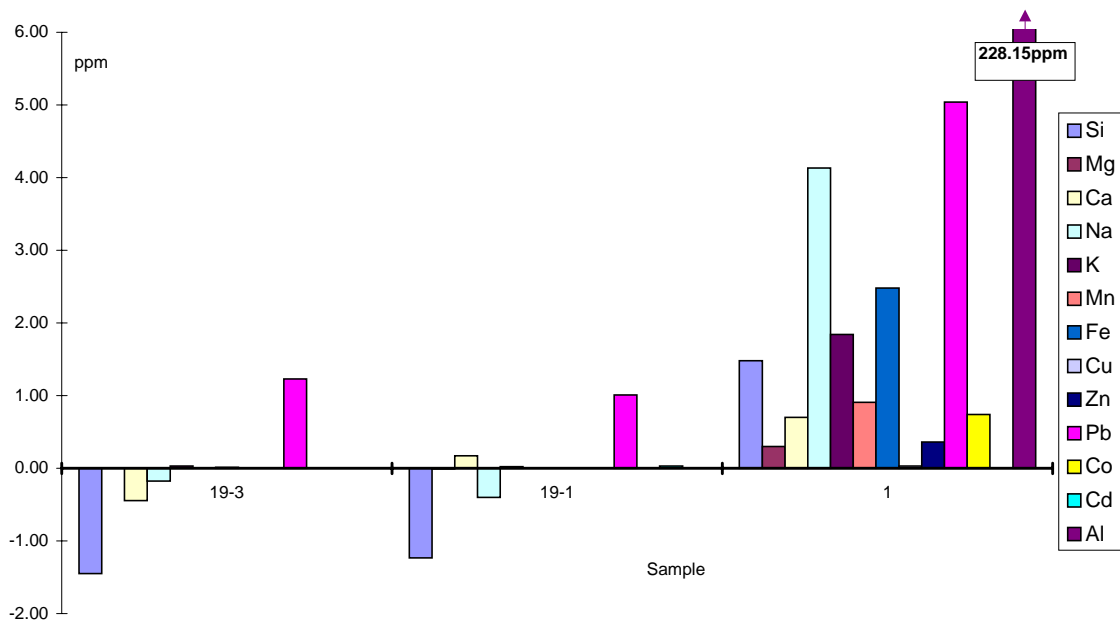


Figure 29: Acetic Acid Results Relative To Baseline Results For Decorated Ware (Units ppm)

6.4.2 15% Ethanol Testing

6.4.2.1 Ethanol Testing Of White Flint Soda Lime Silica Containers

White flint SLS containers are often used to contain alcoholic beverages such as spirits, alcopops and some wines and beers. The results of migration testing of white flint SLS containers with ethanol solutions, compared to their original baseline migration figures are shown in Table 38 and graphically in Figure 30. It would appear that in the case of soda lime silica containers ethanol testing generally results in lower migration than baseline migration testing with water.

Analyte	12-4-a	12-4b	21-2-a	21-2b
Si	2.1	<0.10	1.0	<0.1
Mg	0.06	0.01	<0.01	<0.01
Ca	0.36	0.06	0.25	0.05
Na	1.22	0.63	0.62	0.38
K	0.05	0.01	<0.01	<0.01
Fe	<0.02	<0.02	0.03	<0.02

Table 38: Baseline (a) and Ethanol(b) Results For White Flint Soda Lime Silica Containers(Units ppm)

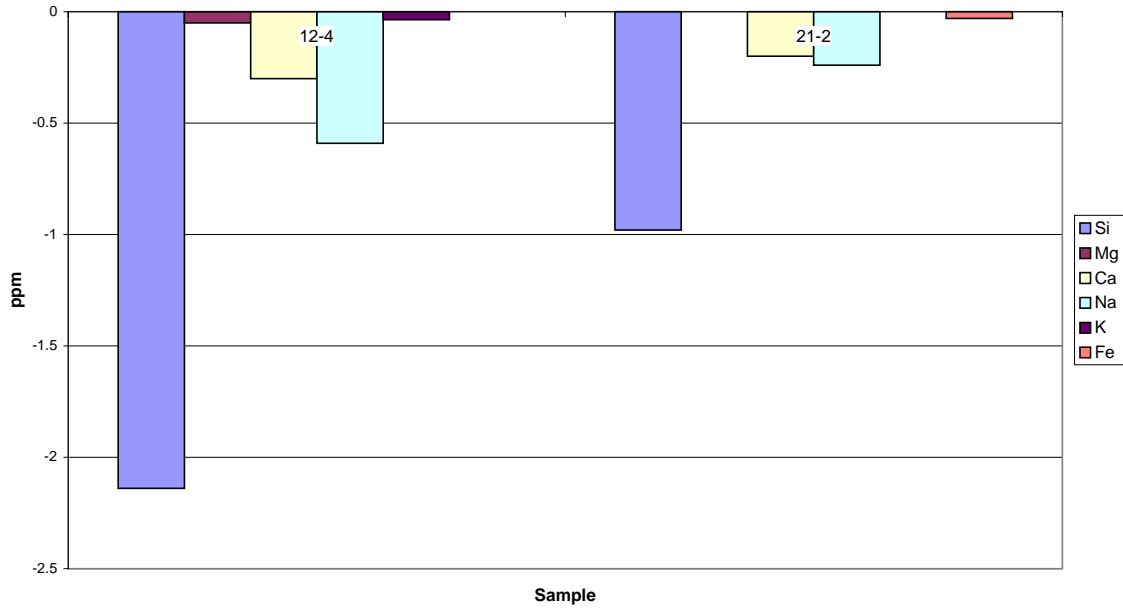


Figure 30: Ethanol Results Relative To Baseline Results For White Flint Soda Lime Silica Containers (Units ppm)

6.4.2.2 Ethanol Testing Of Coloured Soda Lime Silica Containers

Coloured SLS containers are often used to contain alcoholic beverages such as some spirits, wines, beers and fortified wines such as Sherries and Ports. The results of migration testing of coloured SLS containers with ethanol solutions, compared to their original baseline migration figures are shown in Table 39 and graphically in Figure 31. It would appear that in the case of coloured soda lime silica containers, ethanol testing generally results in lower migration than baseline migration testing with water.

Analyte	43-1-a(green)	43-1b	43-2-a(amber)	43-2b	43-3-a(green)	43-3b	43-4-a(green)	43-4b	14-1-a(blue)	14-1b	21-1-a(green)	21-1b
Si	0.9	<0.1	0.7	<0.1	1.2	0.6	0.6	<0.1	0.6	<0.1	1.0	<0.1
Mg	0.03	<0.01	0.03	<0.01	0.06	0.04	0.04	0.01	0.02	0.01	0.02	<0.01
Ca	0.18	0.04	0.17	0.04	0.23	0.12	0.18	0.06	0.12	0.05	0.17	0.04
Na	0.47	0.28	0.44	0.27	0.89	0.43	0.66	0.26	0.83	0.44	0.64	0.39
K	<0.01	0.01	<0.01	0.02	0.02	0.03	0.01	0.01	<0.01	0.01	<0.01	<0.01

Table 39: Baseline (a) and Ethanol (b) Results For Coloured Soda Lime Silica Containers(Units ppm)

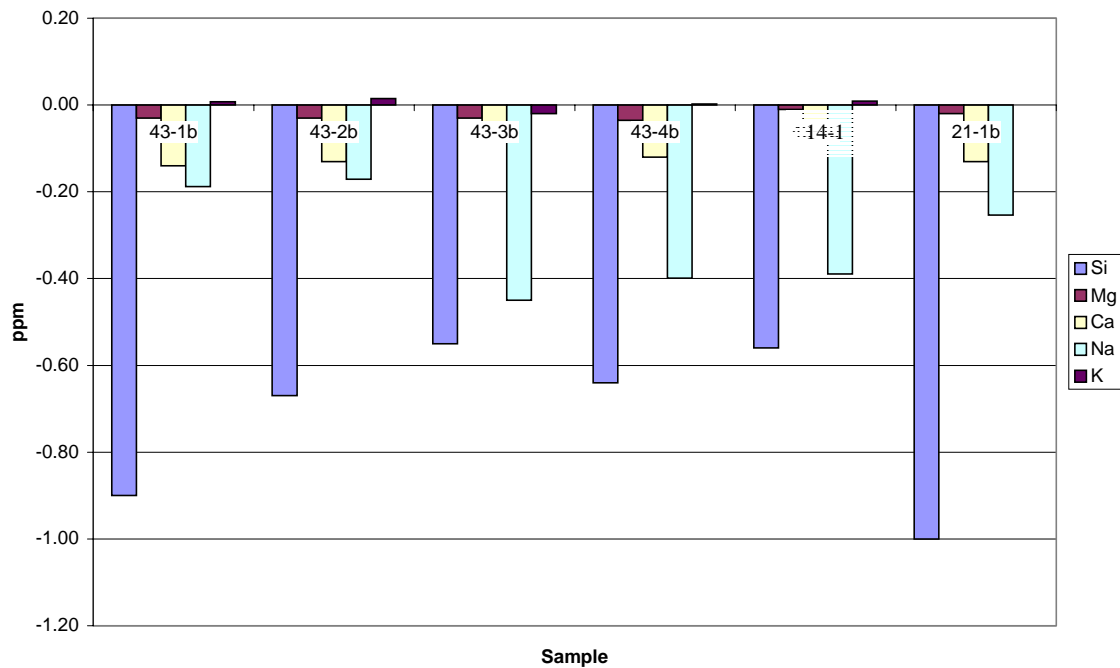


Figure 31: Ethanol Results Relative To Baseline Results For Coloured Soda Lime Silica Containers (Units ppm)

6.4.2.3 Ethanol Testing Of Soda Lime Silica Tableware

SLS tableware includes drinking glasses used to contain alcoholic beverages. The results of migration testing of SLS tableware with ethanol solutions, compared to their original baseline migration figures are shown in Table 40 and graphically in Figure 32. It would appear that in the case of soda lime silica tableware, ethanol testing generally results in lower migration than baseline migration testing with water.

Analyte	13-3-a	13-3b	19-2-a	19-2b	19-4-a	19-4b	31-a	31b
Si	1.8	0.4	1.1	0.3	0.8	0.2	<0.1	<0.1
Mg	0.04	0.03	0.04	0.02	0.03	0.01	<0.010	<0.01
Ca	0.17	0.09	0.28	0.12	0.20	0.09	<0.01	<0.01
Na	1.89	1.47	0.78	0.60	0.71	0.53	0.13	0.08
K	<0.01	0.07	<0.01	0.03	<0.01	0.01	0.01	<0.01
Al	0.12	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
V	<0.01	<0.01	0.01	<0.01	<0.01	<0.01	<0.01	<0.01

Table 40: Baseline (a) and Ethanol (b) Results For Soda Lime Silica Tableware (Units ppm)

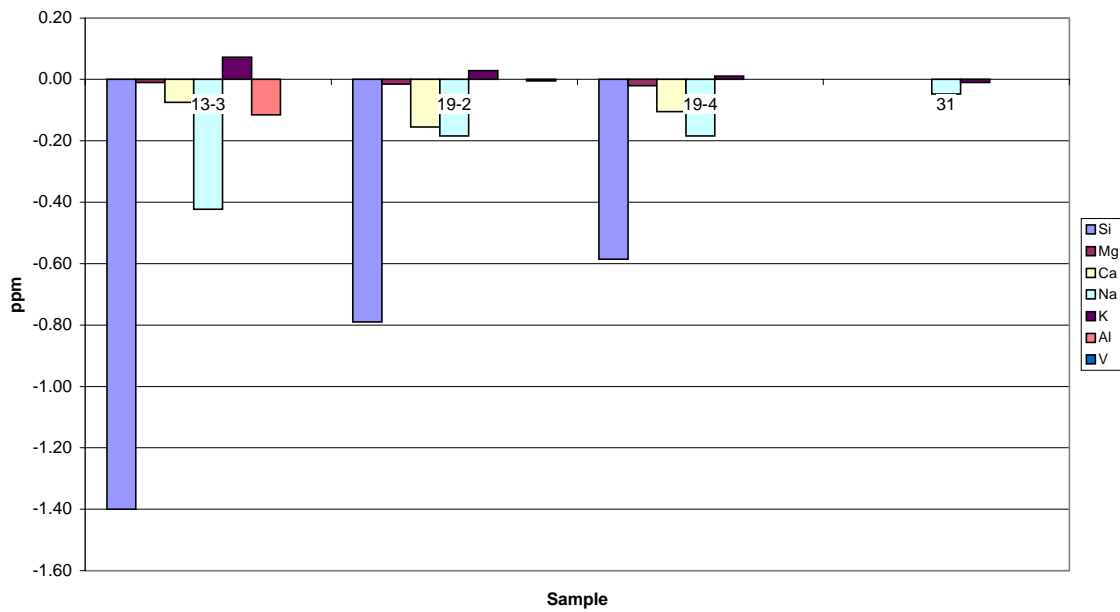


Figure 32: Ethanol Results Relative To Baseline Results For Soda Lime Silica Tableware (Units ppm)

6.4.2.4 Ethanol Testing Of Decorated Ware

Ethanol testing of three samples of decorated ware compared to baseline testing does not display a general trend. This is shown in Table 41 and graphically in Figure 33. It appears that articles 19-1 and 19-3 released less elements in terms of total migration into the test solutions than they did on baseline testing, whilst article 25-1 appeared to release more into the ethanol test solution than in baseline testing.

Analyte	19-1-a	19-1b	19-3-a	19-3b	25-1a	25-1b
Si	1.4	<0.1	1.7	1.0	<0.1	0.2
Mg	0.04	0.02	0.25	0.21	<0.01	0.02
Ca	0.02	0.11	0.52	0.44	0.10	0.08
Na	0.89	0.53	0.52	0.44	<0.02	0.48
K	<0.01	0.02	<0.01	0.02	<0.01	0.02
Al	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05

Table 41: Baseline (a) and Ethanol (b) Results For Decorated Ware (Units ppm)

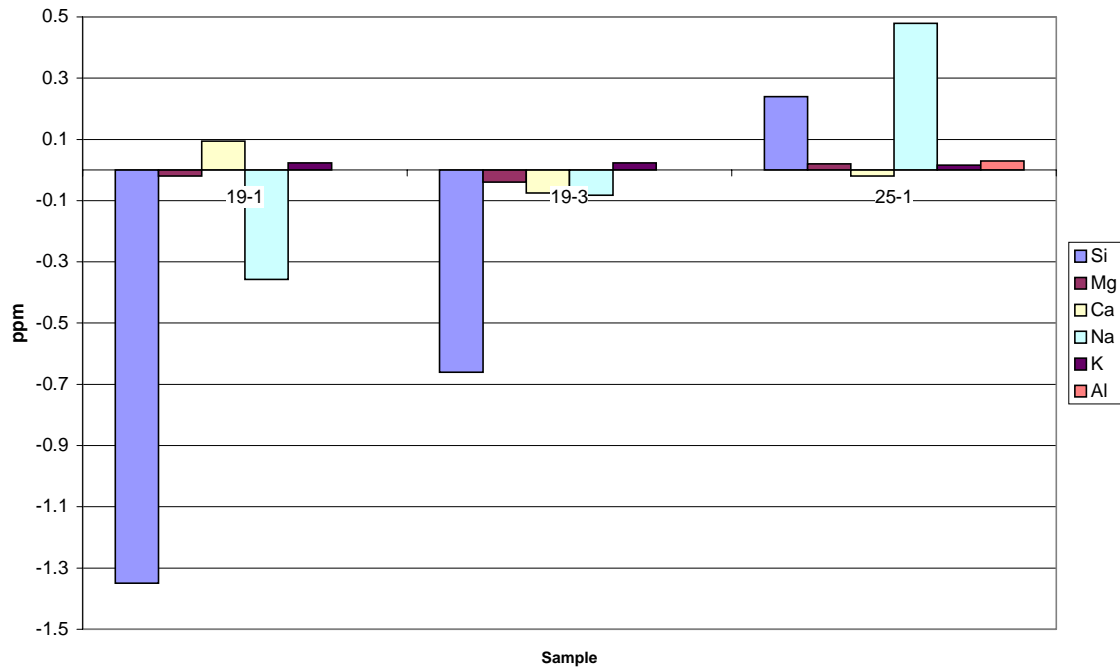


Figure 33: Ethanol Results Relative To Baseline Results For Decorated Ware (Units ppm)

6.4.2.5 Ethanol Testing Of Lead Crystal Ware

Ethanol testing of lead crystal ware compared to baseline testing of similar articles, appeared to yield a slight reduction in migration. The article selected for testing was a 24% lead crystal tumbler, which was intended for spirits use.

Analyte	17-1-a	17-1b
Si	3.0	1.4
Ca	<0.01	<0.01
Na	1.26	0.95
K	1.40	1.04
Ba	<0.01	<0.01
B	<0.03	<0.03
Pb	<0.1	<0.1
Sb	0.17	<0.10

Table 42: Baseline (a) and Ethanol (b) Results For Lead Crystal Ware (Units ppm)

6.4.2.6 Ethanol Testing Of Borosilicate / Glass Ceramic Ware

No testing of borosilicate or glass ceramic ware was carried out with ethanol solutions.

6.4.2.7 Summary Of Ethanol Testing

Ethanol testing displayed a trend to lower elemental migration than baseline testing for all articles tested. The single exception to this finding was sample 25-1 (a decorated soda lime silica tumbler), which appeared to have a slightly higher overall elemental migration than a similar sample, which had undergone baseline testing. The differences involved were below 0.5ppm for all elements released and this discrepancy was limited to the elements Si, Mg, Na and K. This is believed to be due to a low level of contamination of the sample during storage (dust), which may have persisted post rinsing, rather than sample to sample variation as this variability is very low in mass produced soda lime silica glass production. In all tests, the elements were released in levels below the permissible limit in drinking water (See Appendix 2).

6.4.3 Rectified Olive Oil Testing

As olive oil testing was a relatively time consuming analytical procedure testing was limited.

The additional dissolution/dilution step in MIBK as outlined in section 5.3.1.3 necessary to analyse by ICP-OES elements migrated into olive oil had a subsequent effect on reporting limits, which were higher than those applied when testing in water and acetic acid. This extra step meant it was only possible to analyse for a limited range of elements. The elements tested for and their reporting limits compared to other testing, including baseline testing, for similar article types are shown in Table 43.

Analyte	Olive Oil reporting limit	Normal (<i>baseline / acetic acid / ethanol</i>) reporting limit
Si	<0.5	<0.1
Pb	<2	<0.1
Na	<0.5	<0.02
Mg	<0.1	<0.01
K	<1	<0.01
Ca	<0.1	<0.01
Al	<0.5	<0.05

Table 43: Comparison Of Reporting Limits for Olive Oil and Baseline Testing

6.4.3.1 Olive Oil Testing Of Soda Lime Silica Containers

Olive oil is predominantly stored in flint soda lime silica bottles. For this reason a typical flint soda lime silica bottle design that may be used to store olive oil was tested. Table 44 shows the results of the olive oil testing and the corresponding baseline testing result. Despite the differences in reporting limits it is possible to say that for Si and Na there is more release using water as a simulant than olive oil for this example.

Analyte	5-1-a	5-1b
Si	2.96	<0.5
Mg	0.09	<0.1
Ca	0.66	<0.1
Na	1.59	<0.5
K	0.03	<1
**V	0.02	Not determined
Al	0.07	<0.5

Table 44: Baseline (a) and Olive Oil (b) Results For SLS Bottle (Units ppm)

*Note ** It was not possible to determine V in the olive oil simulant experiments due to the lack of suitable standards for instrument calibration.*

6.4.3.2 Olive Oil Testing Of Soda Lime Silica Tableware

No detectable migration above the reporting limits for the technique in question occurred in the two samples tested. It is possible to say that for sample 51 there was less migration of Na and Ca when tested using an olive oil simulant than in baseline test conditions however the large differences in reporting limits quoted between baseline conditions and olive oil test conditions makes further interpretation not possible. Only elements with a detectable migration in either the baseline or olive oil test are listed in Table 45. The results shown are actual results of the tests undertaken and are not “relative results” (i.e. test-baseline)

Analyte	51-a	51-b	32-a	32-b
Si	0.49	<0.5	<0.10	<0.5
Mg	0.02	<0.1	<0.01	<0.1
Ca	0.17	<0.1	0.08	<0.1
Na	0.64	<0.5	0.22	<0.5
K	0.02	<1	<0.01	<1

Table 45: Baseline (a) and Olive Oil (b) Results For SLS Tableware (Units ppm)

6.4.3.3 Olive Oil Testing Of Lead Crystal Ware

Lead crystal ware was omitted from olive oil testing because due to its high cost and poor thermal characteristics, it is not generally used for cooking in or storing/eating foodstuffs from and is mostly used for the storage of alcohol (decanters) and drinking from (glasses and tumblers).

6.4.3.4 Olive Oil Testing Of Borosilicate/Glass Ceramic Ware

One article of borosilicate oven to tableware (18-1) and one article of glass ceramic ware were tested with olive oil. The results of this testing are shown in Table 46 compared to the original baseline migration for this type of article. It would appear that in the case of glass ceramic ware there has been a slight increase in sodium migration relative to baseline testing for the two articles tested.

Analyte	18-1-a	18-1b	27-a	27b
Ca	0.07	<0.1	<0.01	<0.1
Na	<0.02	1.40	<0.02	0.50
K	<0.01	<1	<0.01	<1
Si	<0.1	<0.5	<0.10	<0.5

Table 46: Baseline (a) and Olive Oil (b) Results For Borosilicate/Glass Ceramic Oven/Tableware (Units ppm)

6.4.3 Real Foodstuffs

Drawing firm conclusions from such testing was at the outset known to be difficult. Despite efforts to thoroughly mix real foodstuffs they are by their nature relatively inhomogeneous and often contain high levels of the elements being investigated. Increases in elements on testing may be a factor of sample inhomogeneity rather than due to migration. Unfortunately despite repeated attempts the results of migration in to foodstuffs showed too much variability from one duplicate to another to be considered as robust. It is for this reason that data from migration into real foodstuffs is not presented. Contributing factors thought to be involved in the variability were

- Incomplete digestion of some components of the foodstuff
- Re-precipitation of foodstuffs from the digestion solution prior to analysis
- High background levels of the elements of interest leading to the need for excessive dilution steps prior to analysis which in turn increases error
- Swamping of the detector and interferences
- Plasma instability in ICP-OES when aspirating foodstuffs

6.5 Lip & Rim Testing / Microwaving

The results are the average of two independent determinations, carried out by testing each of a matched pair of articles respectively.

6.5.1 Lip & Rim Testing

Lip and rim testing was carried out on a series of articles with decoration within 2cm of the rim that may come in to contact with food. Testing was carried out as described in section 5.4 to the ASTM method C927.80 (1999)¹², and the results of the testing are shown in Table 47 and graphically in Figure 34. This data is intended as a set of stand alone data rather than for comparison to baseline testing which was carried out on filled articles. In lip and rim testing, elemental release is assessed from only the decorated portion in the top 2cm of the inverted article. Baseline testing assesses elemental release from the filled article, which may be decorated on the external or internal surfaces or only in the lip and rim area. Of particular note are sample 19-1 (decorated SLS tumbler) which released 30.5ppm of Pb and 2.70ppm of Cd, sample 38 (decorated SLS tumbler) which released 6.32ppm of Pb; and sample 44 (decorated SLS tumbler) which released 6.47ppm of Cu.

The relevant drinking water limits (see Appendix 2 at the rear of this report) are given as a point of reference to these figures to allow them to be put in to perspective. It should be stated that exposure to elemental species in drinking water is likely to greatly exceed exposure to the species from lip and rim decoration and that the figures shown are based on a significant “safety margin” approach. This is discussed on the web site. Results which are in excess of drinking water limits are highlighted in ***bold italic*** typeface.

Tests such as lip and rim tests and the baseline / acetic acid tests of some internally decorated articles appear to be more a test of the inertness of the applied decoration than inertness, in terms of elemental release, of the glass itself, as similar undecorated glasses (i.e. similar glass composition and similar manufacturing history) have not given the same levels of migration. As the range of compositions used to decorate glass articles is varied, establishing trends is difficult. It would appear from this testing that the decoration used for some articles of tableware when tested using acetic acid, leads to relatively high levels of migration of some elements relative to the levels permitted in drinking water. (For drinking water limits see Appendix 2)

Analyte	19-3	19-1	44	17-5	25-4	25-2	17-4	56	38	13-1	Drinking Water Limits
Si	0.2	1.5	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	0.2	<0.1	Not Given
Mg	0.91	0.39	0.19	0.07	0.34	0.22	0.03	0.01	0.04	0.05	Not Given
Ca	3.15	0.77	0.07	0.65	1.09	0.88	0.64	0.15	0.56	0.53	250
Na	1.18	2.43	0.31	0.37	0.70	0.44	0.57	0.36	1.26	0.90	150
K	<0.01	0.16	0.08	0.05	<0.01	0.09	<0.01	0.74	<0.01	<0.01	Not Given
Al	0.07	0.18	0.04	0.15	0.49	3.46	0.11	<0.05	0.09	0.07	0.2
Fe	<0.02	0.03	0.04	0.03	0.03	0.05	0.31	<0.02	0.03	0.03	0.2
Zn	<0.03	0.05	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	0.07	<0.03	5
Cu	<0.03	0.32	6.47	<0.03	<0.03	0.03	<0.03	0.06	<0.03	5.00	3
Pb	0.6	30.5	<0.1	0.1	<0.1	<0.1	<0.1	0.7	6.3	<0.1	0.05
Cd	0.02	2.70	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.76	<0.01	0.005
Co	0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	Not Given

Table 47: Lip And Rim Testing (Results ppm)

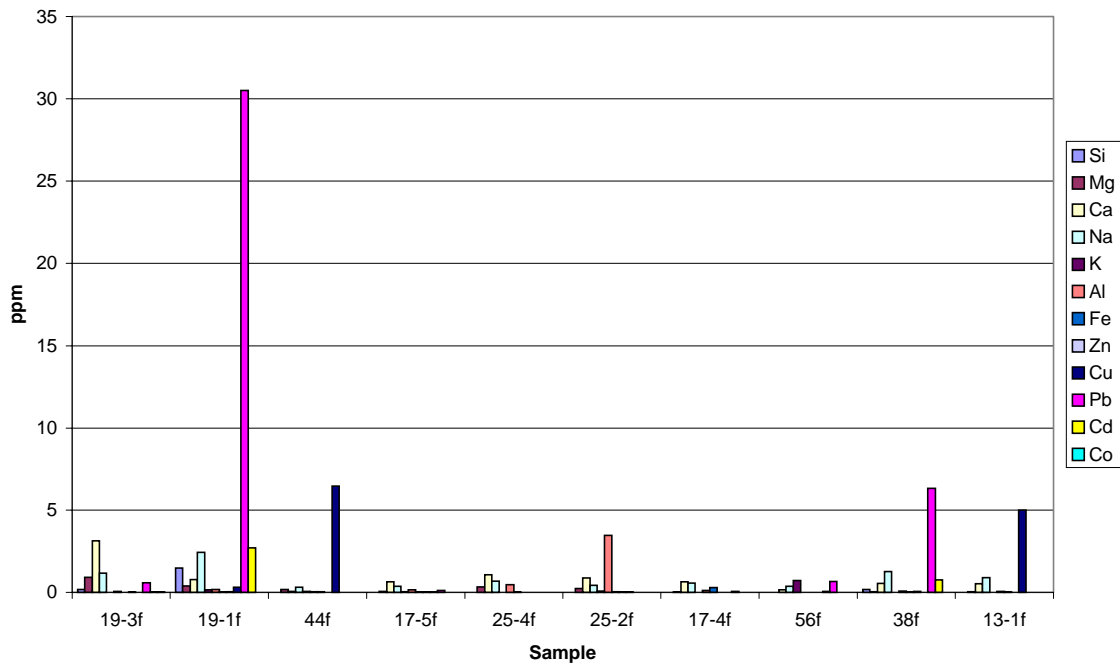


Figure 34: Lip And Rim Testing

6.5.2 Microwaving

Microwave testing was carried out as described in section 5.4.

6.5.2.1 Microwave Testing Of SLS Containers

Whilst it is uncommon for soda lime silica containers to be microwaved, it is possible to imagine items such as baby food jars being heated in this way. As a result two small soda lime silica jars were selected for microwave testing and the results of this testing are shown in Table 48 compared to the results achieved on similar articles from the same sample batch under baseline conditions. Migration from these articles was low and was restricted to four elements (Si, Mg, Ca and Na).

Analyte	6-7-a	6-7-b	6-5-a	6-5-b
Si	0.80	0.25	2.67	0.39
Mg	0.03	0.02	0.07	0.02
Ca	0.23	0.07	0.52	0.07
Na	0.63	0.42	1.14	0.42

Table 48: Baseline Testing (a) Compared To Microwave Testing (b) of SLS Jars (units ppm).

6.5.2.2 Microwave Testing of SLS Tableware

Items of SLS oven to table ware were selected that may be used to microwave cook in. Such items are generally toughened/tempered to give them improved thermal shock characteristics. The articles chosen were sample 32 (an SLS desert bowl) and sample 51 (a toughened SLS coffee mug). The results of this testing are shown in Table 49 compared to the results achieved on similar articles from the same sample batch under baseline conditions. Elemental release was low (<1ppm) for Si, Mg, Ca, Na, K. No other elements were detected in microwave testing at or above reporting limit.

Analyte	32-a	32b	51-a	51b
Si	<0.1	<0.1	0.5	0.4
Mg	<0.01	<0.01	0.02	0.02
Ca	0.08	<0.01	0.17	0.15
Na	0.22	0.14	0.64	0.51
K	<0.01	<0.01	0.02	0.01

Table 49: Baseline Testing (a) Compared To Microwave Testing (b) of SLS Tableware (units ppm).

6.5.2.3 Microwave Testing of Borosilicate Ware Glass / Ceramic Ware

Items of borosilicate/glass ceramic ovenware were selected that may be used to microwave cook in. This type of ware is commonly used for cooking in and a range of articles were selected for testing. The results of this testing are shown in Table 50 compared to the results achieved on similar articles from the same sample batch under baseline conditions. Elemental release was low (<1ppm) for Si, Mg, Ca, Na, K. No other elements were detected in microwave testing at or above reporting limit.

Analyte	57-a	57b	36a	36b	18a	18b	27a	27b
Mg	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.01	0.01
Ca	<0.01	<0.01	0.01	<0.01	0.07	0.08	<0.01	0.06
Na	0.13	0.20	0.02	<0.02	<0.02	0.07	<0.02	<0.02
K	0.06	0.03	0.03	<0.01	<0.01	<0.01	<0.01	<0.01
Si	0.2	0.2	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1

Table 50: Baseline Testing (a) Compared To Microwave Testing (b) of Borosilicate/Glass Ceramic Ware (units ppm).

6.5.2.4 Summary of Microwave Testing

Microwave testing generally led to a lower elemental release than baseline testing. This is probably a result of the reduced time period spent at elevated temperature during microwave testing when compared to baseline testing.

6.6 Elevated Pressure / Elevated Temperature Tests

The results are the average of two independent determinations, carried out by testing each of a matched pair of articles respectively.

6.6.1 Elevated Pressure/Elevated Temperature Testing of White Flint SLS Containers

Testing was carried out as detailed in section 5.5. The results of testing a series of articles under European Pharmacopoeia Time – Temperature conditions are shown in Figure 35 and in Table 51.

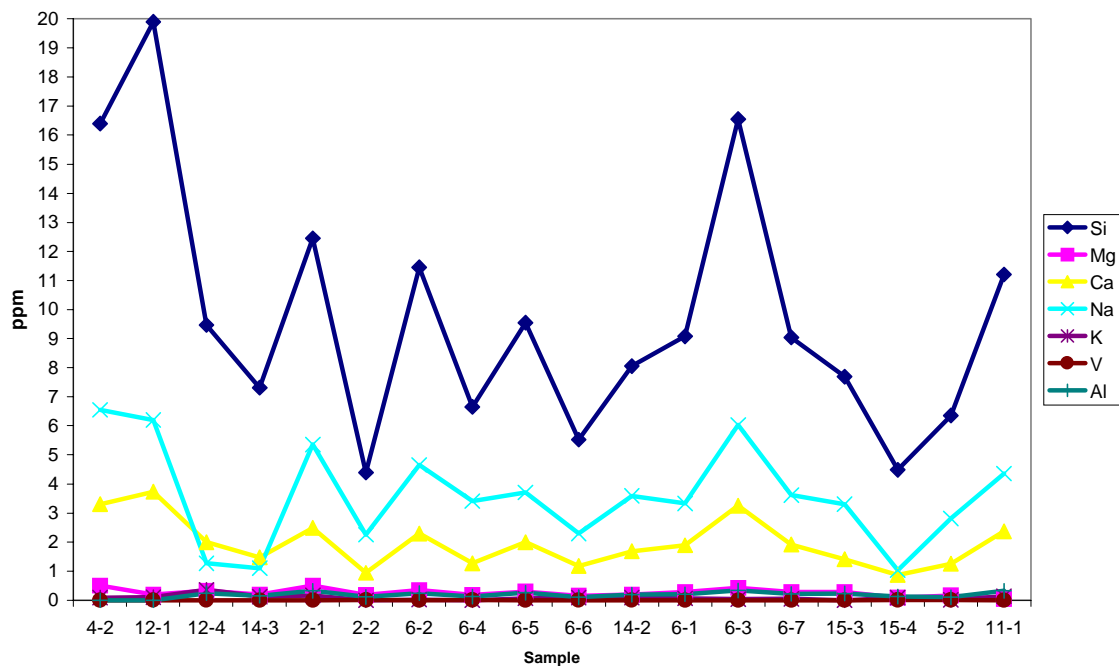


Figure 35: Migration Results of Flint Soda Lime Silca Containers Tested Under European Pharmacopoeia Conditions. (Units ppm)

Element	No Of Samples Tested	Number of samples with result equal to or greater than reporting limit	Reporting Limit (ppm)	Min (ppm)	Max (ppm)	Mean (ppm)
Si	18	18	0.1	4.4	19.9	9.75
Mg	18	18	0.01	0.05	0.51	0.26
Ca	18	18	0.01	0.88	3.74	1.97
Na	18	18	0.02	1.04	6.55	3.64
K	18	15	0.01	<0.01	0.36	0.09
V	18	4	0.01	<0.01	0.01	<0.01
Al	18	16	0.05	<0.05	0.34	0.19

Table 51: Elevated Temperature/Pressure Data For White Flint SLS Containers

These results generally show a marked increase in elemental release relative to testing under baseline conditions. This supports the relationship described by Lyle as discussed in section 3.1 about the effect of elevated temperature on migration.

6.6.2 Elevated Pressure/Elevated Temperature Testing of Coloured SLS Containers

Testing was carried out as detailed in section 5.5. The results of testing a series of articles under European Pharmacopoeia conditions are shown in Figure 36 and Table 52.

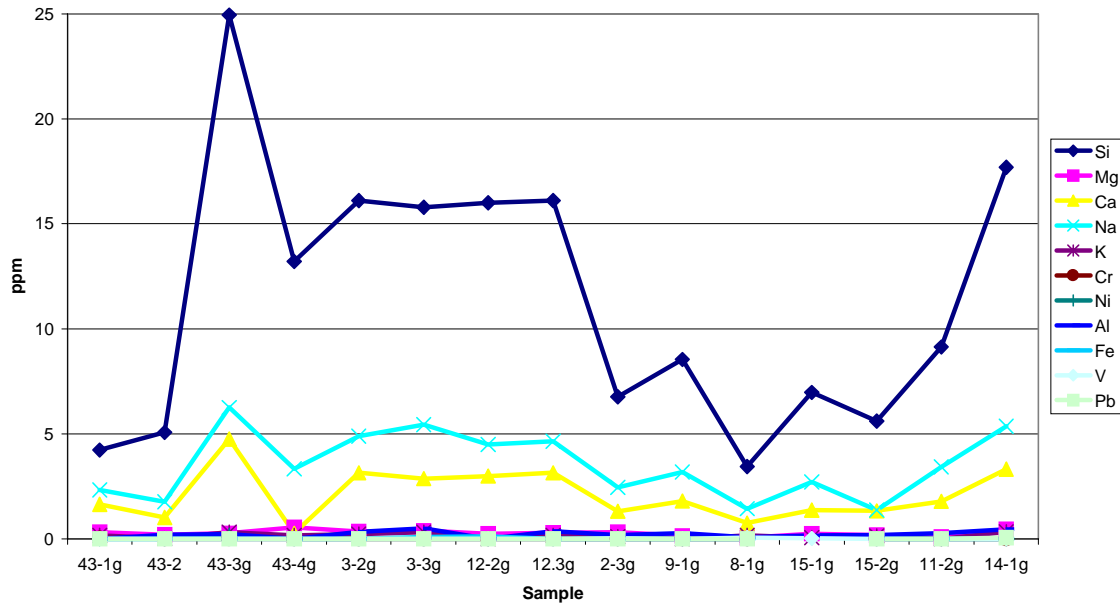


Figure 36: Migration Results of Coloured Soda Lime Silica Containers Tested Under European Pharmacopoeia Conditions.

Element	No Of Samples Tested	Number of samples with result equal to or greater than reporting limit	Reporting Limit (ppm)	Min (ppm)	Max (ppm)	Mean (ppm)
Si	15	15	0.1	3.45	24.95	11.31
Mg	15	15	0.01	0.06	0.54	0.27
Ca	15	15	0.01	0.27	4.75	2.10
Na	15	15	0.02	1.37	6.25	3.54
K	15	12	0.01	<0.01	0.33	0.14
V	15	3	0.01	<0.01	0.06	<0.01
Al	15	12	0.05	<0.05	0.50	0.21
Fe	15	2	0.02	<0.02	0.12	<0.02
Cr	15	1	0.03	<0.03	0.03	<0.03
Ni	15	1	0.02	<0.02	0.15	<0.02

Table 52: Elevated Temperature/Pressure Data For Coloured SLS Containers

The elevated temperature/ pressure has, in a few instances led to the detectable migration of glass colorants (Fe, Cr and Ni) at sub 1ppm levels. No evidence of recycling contaminants (i.e Pb) were detected above reporting limits. These results represent a marked increase in overall elemental release when compared to baseline testing conditions.

6.6.3 Elevated Pressure/Elevated Temperature Testing of SLS Tableware

Article 51 (a toughened SLS cup) was tested under European Pharmacopoeia autoclave conditions. The results of this testing are shown in Table 53 compared to the results achieved on similar articles from the same sample batch under baseline conditions. As can be seen the elevated temperature testing has lead to a marked increase in release for most elements.

Analyte	51-a	51b
Si	0.5	4.5
Mg	0.02	0.09
Ca	0.17	1.17
Na	0.64	2.71
K	0.02	<0.01
Al	<0.05	0.08
V	<0.01	0.01

Table 53: Baseline Testing (a) Compared To 121⁰C Testing (b), of SLS Tableware (units ppm).

6.6.4 Elevated Pressure/Elevated Temperature Testing of Borosilicate / Glass Ceramic ware

Articles 27 (a glass ceramic dish) and 18-1 (a borosilicate dish) were tested under European Pharmacopoeia autoclave conditions. The results of this testing are shown in Table 54 compared to the results achieved on similar articles from the same sample batch under baseline conditions. As can be seen the elevated temperature testing has led to a slight increase in elemental release. These types of articles again appear to give very low migration.

Analyte	27-a	27-1b	18-1-a	18-1b
Ca	<0.01	0.05	0.07	0.11
Na	<0.02	0.70	<0.02	0.19
K	<0.01	0.13	<0.01	<0.01
Si	<0.1	<0.1	<0.1	<0.1

Table 54: Baseline Testing (a) Compared To 121⁰C Testing (b), of Borosilicate / Glass Ceramic Ware (Units ppm)

6.6.5 Elevated Pressure/Elevated Temperature Testing of Lead Crystal Ware

Articles 17-2 (a 24% lead crystal glass) was tested under European Pharmacopoeia autoclave conditions. The results of this testing are shown in Table 55 compared to the results achieved on similar articles from the same sample batch under baseline conditions. As can be seen the elevated temperature testing has led to a marked increase in elemental release.

This elevated temperature has also lead to the release of Pb and Sb at detectable levels, which were not present under baseline testing conditions.

Analyte	17-2-a	17-2-b
Si	0.9	3.6
Ca	<0.01	<0.01
Na	0.40	1.39
K	1.06	2.86
Ba	<0.01	<0.01
B	<0.03	<0.03
Pb	<0.1	0.5
Sb	<0.01	0.12

Table 55: Baseline Testing (a) Compared To 121°C Testing (b), of Lead Crystal Ware(units ppm).

6.7 Spiked Melts

Melts with a series of intentional dopants / spike elements were prepared as detailed in section 5.6. It should be emphasized that grain testing represents a much more severe test than a whole article test due to the extremely high contact surface area of the test sample from which migration can take place. The results are the average of two independent determinations, carried out by testing each of a matched pair batch of prepared grains respectively.

6.7.1 Soda Lime Silica Glass Grains

6.7.1.1 Water Tests (Grains)

As shown in Table 56 no detectable nickel was released into water even when the element was doped into the soda lime silica composition at 10x typical levels. Cr was released in to water at progressively higher levels up to a value of 0.36ppm at 10x dopant levels. Pb release was more intermittent with Pb being released in to water from the typical level melt at 0.2ppm and the 5x typical melt at 0.4ppm. No detectable lead was released from the 2x and 10x typical melts. This may suggest inhomogeneity for some of the Pb spiked melts arising from difficulties in preparation as described in section 4.1.2. Cd was not released at detectable levels from any of the melts prepared.

Analyte	SLS			
	Typical	SLS x2	SLS x5	SLS x10
Ni	<0.01	<0.01	<0.01	<0.01
Cr	0.07	0.12	0.22	0.36
Pb	0.2	<0.1	0.4	<0.1
Cd	<0.01	<0.01	<0.01	<0.01

Table 56: SLS Spiked Melt Grain Testing With Water Test Solution (units ppm).

6.7.1.2 Acetic Acid Tests (Grains)

As shown in Table 57 nickel was released into acetic acid at the 10x typical dopant level (0.03ppm). Cr was released into acetic acid at low levels from all dopant level glasses getting progressively higher up to a value of 0.30ppm at 10x dopant levels. Pb release was more intermittent with Pb being released into acetic acid from the 2x typical level melt at 0.33ppm . No detectable lead was released from the typical, 5x and 10x typical melts. This may suggest inhomogeneity for some of the Pb spiked melts. Cd was released at detectable levels from the 5x and 10x typical levels.

Analyte	SLS			
	Typical	SLS x2	SLS x5	SLS x10
Ni	<0.01	<0.01	<0.01	0.03
Cr	0.05	0.10	0.20	0.30
Pb	<0.1	0.3	<0.1	<0.1
Cd	<0.01	<0.01	0.02	0.03

Table 57: SLS Spiked Melt Grain Testing With Acetic Acid Test Solution (units ppm).

6.7.2 Borosilicate Glass (Grains)

6.7.2.1 Water Tests (Grains)

Borosilicate glass did not release any Pb, Cd, Cr or Ni at detectable levels for melts prepared at typical and 5x typical addition levels when tested with water.

Analyte	Borosil Typical	Borosil x5
Cr	<0.01	<0.01
Pb	<0.1	<0.1
Cd	<0.01	<0.01
Ni	<0.01	<0.01

Table 58: Borosilicate Spiked Grain Melts With Water Test Solution (units ppm).

6.7.2.2 Acetic Acid Tests (Grains)

Borosilicate glass did not release any Pb, Cd, Cr or Ni at detectable levels for typical dopant level melts. At 5x typical addition levels low levels of Cr (0.09ppm) and Ni (0.03ppm) were detected.

Analyte	Borosil Typical	Borosil x5
Cr	<0.01	0.09
Pb	<0.1	<0.1
Cd	<0.01	<0.01
Ni	<0.01	0.03

Table 59: Borosilicate Spiked Grain Melts With Acetic Acid Test Solution (units ppm).

6.7.3 Lead Crystal Glass (Glass Grains)

6.7.3.1 Water Tests

The arsenic and antimony release in to water from spiked grains is shown in Table 60. Both elements were released at <1ppm levels during this testing. However compared to levels permissible in drinking water (see appendices at rear of report) the amounts detected are relatively high.

Analyte	Pb Glass Typical	Pb Glass x5
Sb	0.19	0.83
As	0.12	0.87

Table 60: Lead Crystal Spiked Grains Melts With Water Test Solution (units ppm).

6.7.3.2 Acetic Acid Tests (Grains)

The arsenic and antimony release into acetic acid from spiked grains was high. This is shown in Table 61. Both elements were released at >150ppm levels during this testing. The high figures may be a feature of a number of factors.

- Sb and As may be released more readily into acetic acid solutions than water solutions, although no evidence for such a relationship was found on the subsequent pressed cup testing.
- The effect of using glass grains is to significantly increase migration relative to manufactured articles due to the high contact surface area.
- The fact that the “typical” results are higher than the “5x typical” results would also suggest there was a degree of inhomogeneity in this batch of melts which may have affected compositional stability and therefore inertness of the final glass.

No results of this order have been seen from manufactured articles during water and acetic acid testing of lead crystal.

Analyte	PbGlass Typical	Pb Glass x5
Sb	235	177
As	287	185

Table 61: Lead Crystal Spiked Grain Melts With Acetic Acid Test Solution (units ppm).

6.7.4 Pressed Cups

Melts with a series of intentional dopants / spike elements were prepared and pressed into cups as detailed in section 5.6. It should be noted that instrumental changes at the subcontract laboratory between the initial spiked grain work and the follow up pressed cup work has resulted in a slight change in reporting limits. All results are the average of duplicate determinations. The results from pressed cups are probably more indicative of real world articles than those from grain tests.

6.7.4.1 Soda Lime Silica Water Tests (Pressed Cups)

As shown in Table 62 no detectable nickel, chromium, lead or cadmium was released in to water when doped individually or simultaneously at typical and 5x typical levels. The difference in performance between grain tests and the pressed articles is probably a feature of contact surface area.

CUP	Ni (ppm)	Cr(ppm)	Cd(ppm)	Pb(ppm)
SLS Blank	<0.02	<0.03	<0.01	<0.1
SLS Ni Typical	<0.01	<i>Intentionally blank</i>	<i>Intentionally blank</i>	<i>Intentionally blank</i>
SLS Ni x5	<0.01	<i>Intentionally blank</i>	<i>Intentionally blank</i>	<i>Intentionally blank</i>
SLS Cr Typical	<i>Intentionally blank</i>	<0.03	<i>Intentionally blank</i>	<i>Intentionally blank</i>
SLS Cr x5	<i>Intentionally blank</i>	<0.03	<i>Intentionally blank</i>	<i>Intentionally blank</i>
SLS Cd Typical	<i>Intentionally blank</i>	<i>Intentionally blank</i>	<0.01	<i>Intentionally blank</i>
SLS Cd x5	<i>Intentionally blank</i>	<i>Intentionally blank</i>	<0.01	<i>Intentionally blank</i>
SLS Pb Typical	<i>Intentionally blank</i>	<i>Intentionally blank</i>	<i>Intentionally blank</i>	<0.1
SLS Pb x5	<i>Intentionally blank</i>	<i>Intentionally blank</i>	<i>Intentionally blank</i>	<0.1
SLS Ni, Cr, Cd, Pb Typical	<0.02	<0.03	<0.01	<0.1
SLS Ni, Cr, Cd, Pb x5	<0.02	<0.03	<0.01	<0.1

Table 62: SLS Cup Spiked Melt Testing With Water Test solution (units ppm).

6.7.4.2 Acetic Acid Tests (Pressed Cups)

As can be seen in Table 63 no detectable nickel, chromium, lead or cadmium was released in to acetic acid when doped individually or simultaneously at typical and 5x typical levels. The difference in performance between grain tests and the pressed articles is probably a feature of contact surface area.

CUP	Ni (ppm)	Cr(ppm)	Cd(ppm)	Pb(ppm)
SLS Blank	<0.02	<0.03	<0.01	<0.1
SLS Ni Typical	<0.01	<i>Intentionally blank</i>	<i>Intentionally blank</i>	<i>Intentionally blank</i>
SLS Ni x5	<0.01	<i>Intentionally blank</i>	<i>Intentionally blank</i>	<i>Intentionally blank</i>
SLS Cr Typical	<i>Intentionally blank</i>	<0.03	<i>Intentionally blank</i>	<i>Intentionally blank</i>
SLS Cr x5	<i>Intentionally blank</i>	<0.03	<i>Intentionally blank</i>	<i>Intentionally blank</i>
SLS Cd Typical	<i>Intentionally blank</i>	<i>Intentionally blank</i>	<0.01	<i>Intentionally blank</i>
SLS Cd x5	<i>Intentionally blank</i>	<i>Intentionally blank</i>	<0.01	<i>Intentionally blank</i>
SLS Pb Typical	<i>Intentionally blank</i>	<i>Intentionally blank</i>	<i>Intentionally blank</i>	<0.1
SLS Pb x5	<i>Intentionally blank</i>	<i>Intentionally blank</i>	<i>Intentionally blank</i>	<0.1
SLS Ni, Cr, Cd, Pb Typical	<0.02	<0.03	<0.01	<0.1
SLS Ni, Cr, Cd, Pb x5	<0.02	<0.03	<0.01	<0.10

Table 63: SLS Cup Spiked Melt Testing With Acetic Acid Test Solution (units ppm).

6.7.5 Borosilicate Glass (Pressed Cups)

6.7.5.1 Water Tests (Pressed Cups)

As can be seen in Table 64 no detectable nickel, chromium, lead or cadmium was released in to water when doped individually or simultaneously at typical and 5x typical levels. The difference in performance between grain tests and the pressed articles is probably a feature of contact surface area.

CUP	Ni (ppm)	Cr(ppm)	Cd(ppm)	Pb(ppm)
BS Blank	<0.02	<0.03	<0.01	<0.1
BS Ni Typical	<0.01	<i>Intentionally blank</i>	<i>Intentionally blank</i>	<i>Intentionally blank</i>
BS Ni x5	<0.01	<i>Intentionally blank</i>	<i>Intentionally blank</i>	<i>Intentionally blank</i>
BS Cr Typical	<i>Intentionally blank</i>	<0.03	<i>Intentionally blank</i>	<i>Intentionally blank</i>
BS Cr x5	<i>Intentionally blank</i>	<0.03	<i>Intentionally blank</i>	<i>Intentionally blank</i>
BS Cd Typical	<i>Intentionally blank</i>	<i>Intentionally blank</i>	<0.01	<i>Intentionally blank</i>
BS Cd x5	<i>Intentionally blank</i>	<i>Intentionally blank</i>	<0.01	<i>Intentionally blank</i>
BS Pb Typical	<i>Intentionally blank</i>	<i>Intentionally blank</i>	<i>Intentionally blank</i>	<0.1
BS Pb x5	<i>Intentionally blank</i>	<i>Intentionally blank</i>	<i>Intentionally blank</i>	<0.1
BS Ni, Cr, Cd, Pb Typical	<0.02	<0.03	<0.01	<0.1
BS Ni, Cr, Cd, Pb x5	<0.02	<0.03	<0.01	<0.1

Table 64: Borosilicate Cup Spiked Melts With Water Test Solution (units ppm).

6.7.5.2 Acetic Acid Tests (Pressed Cups)

As can be seen in Table 65 no detectable nickel, chromium, or cadmium was released in to acetic acid when doped individually or simultaneously at typical and 5x typical levels. Low levels of Pb were detected in the Pb typical spike (0.1ppm) and the Pb,Cd,Cr,Ni combined typical spike (0.1ppm). These results are sub-ppm and are at the reporting limit for the procedure.

CUP	Ni (ppm)	Cr(ppm)	Cd(ppm)	Pb(ppm)
BS Blank	<0.02	<0.03	<0.01	<0.1
BS Ni Typical	<0.01	<i>Intentionally blank</i>	<i>Intentionally blank</i>	<i>Intentionally blank</i>
BS Ni x5	<0.01	<i>Intentionally blank</i>	<i>Intentionally blank</i>	<i>Intentionally blank</i>
BS Cr Typical	<i>Intentionally blank</i>	<0.03	<i>Intentionally blank</i>	<i>Intentionally blank</i>
BS Cr x5	<i>Intentionally blank</i>	<0.03	<i>Intentionally blank</i>	<i>Intentionally blank</i>
BS Cd Typical	<i>Intentionally blank</i>	<i>Intentionally blank</i>	<0.01	<i>Intentionally blank</i>
BS Cd x5	<i>Intentionally blank</i>	<i>Intentionally blank</i>	<0.01	<i>Intentionally blank</i>
BS Pb Typical	<i>Intentionally blank</i>	<i>Intentionally blank</i>	<i>Intentionally blank</i>	0.1
BS Pb x5	<i>Intentionally blank</i>	<i>Intentionally blank</i>	<i>Intentionally blank</i>	<0.1
BS Ni, Cr, Cd, Pb Typical	<0.02	<0.03	<0.01	0.1
BS Ni, Cr, Cd, Pb x5	<0.02	<0.03	<0.01	<0.1

Table 65: Borosilicate Spiked Grain Melts With Acetic Acid Test Solution (units ppm).

6.7.6 Lead Crystal Glass (Pressed Cups)

6.7.6.1 Water Tests (Pressed Cups)

The arsenic and antimony release into water from pressed cups is shown in Table 66. Sb at <1ppm levels was released from the Sb 5x typical sample and the combined Sb, As 5x typical sample. No Arsenic was detected at or above the reporting limits. No Sb release was recorded at typical levels.

CUP	Sb(ppm)	As(ppm)
LC Blank	<0.1	<0.1
LC Sb Typical	<0.1	<i>Intentionally blank</i>
LC Sb x5	0.3	<i>Intentionally blank</i>
LC As Typical	<i>Intentionally blank</i>	<0.1
LC As x5	<i>Intentionally blank</i>	<0.1
LC As,Sb Typical	<0.1	<0.1
LC As,Sb x5	0.3	<0.1

Table 66: Lead Crystal Cup Spiked Melts With Water Test Solution (units ppm).

6.7.6.2 Acetic Acid Tests (Pressed Cups)

The arsenic and antimony release into acetic acid from pressed cups was below the reporting limits. This was in stark contrast to the results obtained from the equivalent glass grains tests, which showed high levels of release for this test. This is probably a feature of the dramatically different surface area to volume ratios employed and may also suggest a problem with melt homogeneity with the equivalent grain samples, arising from difficulties in preparation as described in section 4.1.2.

CUP	Sb(ppm)	As(ppm)
LC Blank	<0.1	<0.1
LC Sb Typical	<0.1	<i>Intentionally blank</i>
LC Sb x5	<0.1	<i>Intentionally blank</i>
LC As Typical	<i>Intentionally blank</i>	<0.1
LC As x5	<i>Intentionally blank</i>	<0.1
LC As,Sb Typical	<0.1	<0.1
LC As,Sb x5	<0.1	<0.1

Table 67: Lead Crystal Spiked Grain Melts With Acetic Acid Test Solution (units ppm).

6.8 Surface Area To Volume Ratio

The effect on elemental migration of contact surface area to volume ratio was investigated by comparing the results of a range of articles produced in the same nominal glass composition (White Flint soda lime silica). This should remove any effects on migration resistance due to gross effects of compositional difference. Comparison between articles with the same prefix number is comparison of articles of different size originating from the same manufacturer and would also remove the effect of more subtle variations in composition from manufacturer to manufacturer. Migration of the key glass making elements Si, Na and Ca, (which have proved common migrants for this class of glass) have been tracked in Figure 37. As can be seen there appears to be a relationship in that the higher the surface area to fill volume ratio the higher the elemental release. This is illustrated in Table 68 and Figure 37

The articles chosen were previously untested flint soda lime silica containers and the effect on baseline migration was investigated.

Sample	Surface Area (cm ²)	Fill Volume (cm ³)	SA/Vol (cm ⁻¹)	Si ppm	Na ppm	Ca ppm
4-1	301.8	290	1.04	1.9	1.96	0.47
6-2	248.1	320	0.77	1.7	0.86	0.38
6-4	263.5	225	1.17	2.2	0.99	0.52
6-5	147.5	105	1.40	2.7	1.14	0.52
11-1	248.6	270	0.92	1.8	1.00	0.45

Table 64: Results of Comparison of Surface Area/Volume versus ppm release

Surface Area To Volume Relationship For Migration from White Flint Soda Lime Silica Containers
(Baseline Conditions)

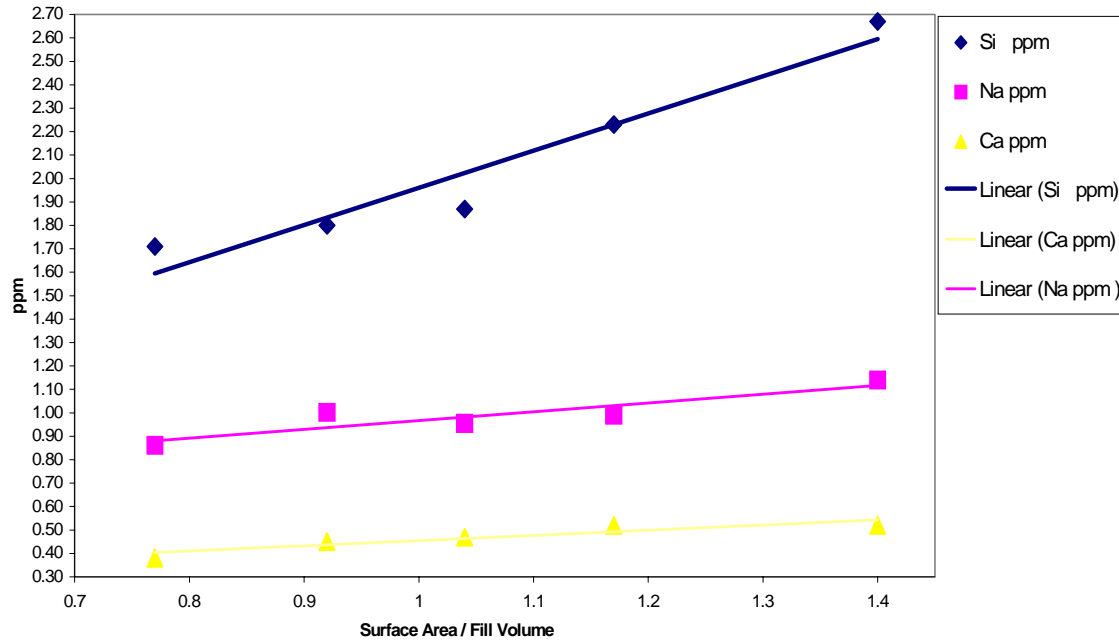


Figure 37: Effect of Surface Area to Volume ratio on Migration Result

7 Summary Of Data

This section presents a brief overview of the findings of the work carried out.

- In baseline testing, migration from soda lime silica (SLS) containers was generally only found for the major glass compositional elements with only intermittent low level release of V (typically <0.02ppm), Al (<0.2ppm) and Fe (one instance 0.03ppm). No evidence of the glass colourants Cr or Ni or common recycling contaminants such as Pb were detected from coloured SLS containers during baseline testing.

- In baseline tests, SLS tableware generally gave similar migration results to SLS containers. In one instance there was high release of Na (21.8ppm) and K (18.5ppm) although release of other elements from this article was relatively low.
- In baseline tests, lead crystal ware generally released Si (mean of 1.9ppm), Na (mean of 0.52ppm) and K (mean of 0.86ppm) with occasional release of Ca at sub 0.2ppm levels. The release of B, Ba and Sb was limited to only a few of the ten items tested and was generally at the sub 0.15ppm level. Pb was released from two articles under these conditions (1.3ppm and 0.9ppm) respectively.
- Baseline testing of glass ceramic and borosilicate articles showed release to be very low. Only Si, Na, Ca and K were detected and these were not detected for all articles tested. When detected, values recorded were close to reporting limit values.
- Baseline testing of decorated articles generally led to the release of similar elements to those detected in soda lime silica glass testing at similar levels.
- The effect of surface damage was generally to increase elemental release, which is probably linked to the increase in contact surface area. Generally, release was limited to the same elements released during baseline testing. In one instance, an amber soda lime silica was found to release 0.5ppm of Pb that was not released in baseline testing, and a decorated article was found to release 0.05ppm of Co after surface damage when none was detected on baseline testing. The effect of surface damage was relatively insignificant for borosilicate / glass ceramic ware which is a feature of the very high durability of these types of articles.

- Caustic washing did not have a consistent deleterious effect on migration from the SLS containers tested.
- Acetic acid testing resulted in lower elemental release from soda lime silica containers than water. The same was true for soda lime silica tableware, although acetic acid did lead to the release of Pb from 3 articles of SLS tableware. The migration of Pb was not detected from these articles with a water based test. Acetic acid testing also gave an increase in Pb migration from a lead crystal article over baseline values. Borosilicate and glass ceramic displayed high chemical resistance to acetic acid giving low migration figures.
- The effect of acetic acid testing on decorated ware was significant. The results show that on whole article tests, where the decoration contacts the test solution, some decoration has relatively low resistance to acidic solution testing and in one instance 228.2ppm of Al was released. This result is significantly higher than the allowable level of Al in drinking water (0.2ppm) found on the Yorkshire Water web site. Other elements released with acetic acid testing included Pb, Cd, Mn, Zn and Co. Lip and rim testing using the conditions described in ASTM C927.80 also showed that the resistance of certain decorations to acetic acid may be of concern with release of the elements Pb, Cd, Cu, Al and Fe exceeding prescribed drinking water limits for several articles, although not all of these articles released all the elements listed.
- Ethanol was found to be a less effective medium than acetic acid and water at promoting elemental release from all types of ware tested.

- Olive oil testing resulted in low migration figures from glass ware and was not as effective as acetic acid or water at promoting elemental release from the glassware tested. The differences in reporting limits for olive oil testing also make direct comparison with other simulants difficult.
- It is difficult to draw firm conclusions from the testing of real foodstuffs, due to the inhomogeneous nature of these materials and the high levels of certain elements of interest already present in these materials.
- Microwaving generally resulted in lower elemental release from the glassware tested than did baseline testing conditions.
- Testing carried out at 121⁰C led to an increase in elemental release relative to baseline testing conditions. This supports the findings of Lyle and Bacon and Birch (see section 3.1)
- Spiked melt testing showed that for extreme conditions of high contact surface area (glass grains) and elevated temperature, it was possible for the glasses studied to release elements of possible “toxicological interest”, when doped at “typical” or elevated concentration levels. However it should be remembered that these “typical” levels for Pb,Cd, Ni and Cr have been drawn from soda lime silica glass data and an arbitrary level has been taken for cadmium that is actually higher than might truly be expected. These dopant levels have then been applied to borosilicate glasses also. Despite this and the extremely high surface contact area of glass grain tests both soda lime silica and borosilicate glasses recorded generally sub 1ppm migration of these four elements even when doped up to 10x “typical levels”.

- Significant migration (100-300ppm) was recorded from grains of lead crystal glass spiked melts containing As and Sb when tested with acetic acid (even at “typical levels”). This may be a feature of the high contact surface area and glass in-homogeneity suggesting a melting problem with the laboratory scale batch of glass prepared for this test. When tested with water release was only recorded at sub 1ppm level for these elements.
- When spiked melts were actually pressed into articles and tested, migration of the elements Pb,Ni,Cd, Cr was not detectable for both water and acetic acid test solutions even up to 5x and 10x typical concentrations for soda lime silica glasses. A similar story was found with borosilicate glasses, although Pb was released into acetic acid at its reporting limit for 5x typical concentrations.
- The testing of pressed articles is more indicative of real world use than grain tests.
- No detectable As or Sb was released from typical lead crystal glass spiked melts pressed into cups when tested with acetic acid, however, in two instances Sb was released at sub 0.5 ppm level when doped to 5x typical levels. This suggests that the high acetic acid grain test result for release of these elements was probably a feature of increased contact surface area (perhaps coupled with melt inhomogeneity) and would probably not be indicative of real world exposure.

Overall Summary

- Glass has under accelerated migration testing conditions been found to be a material of high chemical inertness. However certain types of decorated glassware under acidic conditions were shown to release elements that may be of interest, if such decoration was used in contact with food.
- Whilst it could be argued that the acetic acid testing conditions employed (3% acetic acid, 50⁰C, 72hours,) were relatively severe compared to ISO7086/BS6748, significant migration from some decorated ware was also observed during lip and rim testing under the same contact conditions as this standard (4% acetic acid, 24hours, 20⁰C). There are numerous standards that could be used or adapted to cover decorated glass ware intended for food contact usage including ISO 7086, BS6748, ASTM AOAC 972.2 and the ASTM c927.80 lip and rim test. However, these standards are limited to the determination of Pb and Cd release at present and do not cover the other elements that may be of interest that were seen to be released from some articles. (e.g. Al, Cu, Mn, Co and Cu).
- Spiked melt testing has shown that, when pressed into an article, glass (lead crystal, soda lime silica and borosilicate) did not in general release detectable levels of the elements of possible physiological/toxicological interest (Pb, Cd, Ni, Cr, As, Sb) when subjected to accelerated migration testing. Where release was detected this was found to be at the sub 1ppm level and was generally not detected from articles with “typical” dopant content levels. Release from pressed articles was only detected when incorporated into the melt at 5x and 10x typical levels and was limited to Sb from lead crystal glass tested under baseline conditions.

- Pb was detected at the reporting limit from borosilicate glass spiked at “typical” levels, however these typical levels actually applied to soda lime silica glass and were arbitrarily applied to borosilicate glass to allow a direct comparison between the two glass types. The actual lead content level of borosilicate glass is generally an order of magnitude lower than this as the manufacture of borosilicate glass does not involve the use of external recycled glass, which prevents such contamination.
- It is recommended that a more detailed study of decoration on glassware which has the potential to contact foodstuffs be carried out, as this testing was only a limited aspect of the overall study carried out and data on a broader sample set of decorated ware would be desirable.

Appendix I – Samples Collected And Description Of Coding Used

First number indicates contributor/source, second number indicates a particular sample type.

Sample ref.	glass type
1	decorated tableware
2-1	flint SLS bottle
2-2	flint SLS jar
2-3	green SLS bottle
3-1	green SLS bottle
3-2	green SLS bottle
3-3	amber SLS bottle
4-1	flint SLS bottle
4-2	flint SLS jar
4-3	flint SLS jar
5-1	flint SLS bottle
5-2	flint SLS bottle
5-3	flint SLS bottle
6-1	flint SLS bottle
6-2	flint SLS bottle
6-3	flint SLS bottle
6-4	flint SLS jar
6-5	flint SLS jar
6-6	flint SLS bottle
6-7	flint SLS jar
7	32% Pb wine glass
8	yellow/green SLS bottle
9-1	deep amber bottle
10	flint SLS perfume bottle
11-1	flint SLS bottle
11-2	amber SLS bottle
12-1	flint SLS bottle
12-2	amber SLS bottle
12-3	green SLS bottle
12-4	flint SLS jar
13-1	decorated SLS wine glass

TESTING CODES USED

a= baseline testing (water)
 b = simulant test (acetic acid, ethanol, olive oil)
 c = Surface damage tests
 d = Alkali washing tests
 e = pastuerisation tests
 f = lip & rim testing
 g = autoclave testing
 h = *not used as can be confused with J when viewed turned through 90 degrees on test articles*
 J = microwave testing
 k= real foodstuff tests

In this report legends such as 5-1-a appear in many figures. The result for sample 5-1-a would be the average result of two samples from supplier/contributor number 5 of design type 1, (in this case a white flint sls bottle) tested under baseline conditions.

Sample ref.	glass type
13-2	flint SLS wine glass
13-3	flint SLS tumbler
14-1	blue SLS bottle
14-2	flint SLS jar
14-3	flint SLS bottle
15-1	deadleaf green SLS bottle
15-2	pale amber sls bottle
15-3	flint SLS bottle
15-4	flint SLS bottle
17-1	24% Pb glass tumbler
17-2	24% Pb glass decanter
17-3	24% Pb glass champagne glass
17-4	decorated SLS tumbler
17-5	decorated SLS tumbler
17-6	24% Pb glass bowl
18	borosilicate dish
19-1	decoratd flint SLS mug
19-2	flint SLS tumbler
19-3	decorated SLS tumbler
19-4	flint SLS tumbler
20	Pb glass tumbler
21-1	green SLS bottle
21-2	flint SLS bottle
22	borosilicate bowl
23	borosilicate dish
24	flint SLS wine glass
25-1	decorated SLS tumbler
25-2	decorated SLS tumbler
25-3	decorated SLS tumbler
25-4	decorated SLS tumbler
26	24% Pb glass
27-1	glass ceramic dish
27-2	glass ceramic dish with lid
27-3	glass ceramic dish with lid
28	SLS casserole dish
29	flint SLS champagne flute
30	SLS sundae dish
31-1	smoked SLS flan dish
31-2	smoked SLS casserole dish
31-3	smoked SLS casserole dish
32	flint SLS dessert bowl
33	24% Pb tumblers
34	24% Pb wine glass

Sample ref.	glass type
35	flint SLS tumbler
36-1	borosilicate dish
36-2	borosilicate dish
36-3	borosilicate dish
37-1	blue SLS plate
37-2	blue SLS mug
37-3	blue SLS bowl
37-4	blue SLS small plate
38	decorated tumbler
39	flint SLS dessert bowl
40	flint+blue SLS glass
41	decorated tableware
42	'jade' green tableware
43-1	green SLS bottle
43-2	ext. dark amber SLS bottle
43-3	light green SLS bottle
43-4	pale green SLS bottle
44	decorated tumbler
45	flint SLS tumbler
46	flint+blue SLS bowl
47	pale green wine glass
48	decorated SLS pint glass
49	24% Pb tumbler
50	32% Pb wine glass
51	toughened SLS smoked glass mug
52	flint SLS sundae dish
53	flint SLS bowl
54	decorated SLS glass
55	decorated SLS glass
56	decorated Pb glass
57	glass ceramic saucepans with lids
58	SLS mixing bowl

APPENDIX 2

Elemental concentration limits for drinking water

		Current limit	New limits	Further changes
Al	Aluminium	0.2	0.2	
As	Arsenic	0.05	0.01	
B	Boron	2	1	
Ba	Barium	1		
Ca	Calcium	250		
Cd	Cadmium	0.005	0.005	
Cr	Chromium	0.05	0.05	
Cu	Copper	3	2	
Fe	Iron	0.2	0.2	
Mn	Manganese	0.05	0.05	
Na	Sodium	150	200	
Ni	Nickel	0.05	0.02	
Pb	Lead	0.05	0.025	0.01*
Sb	Antimony	0.01	0.005	
Se	Selenium	0.01	0.01	
Zn	Zinc	5		

Values for current limits are from The Water Supply (Water Quality) Regulations 1989 (SI#1147).

Values for new limits are from The Water Supply (Water Quality) Regulations 2000 (SI#3184) which sets limits that apply from 25th December 2003.

* will apply from 25th December 2015.

Glossary Of Terms

- WFC = white flint container (e.g clear glass bottles, jars etc.)
 - GC = green container (e.g green bottles, jars etc.)
 - AC = Amber container (e.g brown/amber bottles, jars etc.)
 - DLGC = Dead Leaf Green container (olive green bottles, jars etc.)
 - BC = Blue container (e.g blue bottles, jars etc.)
 - HWC = Half white container (e.g. bacardi type green/clear bottles, jars etc.)
 - TW SLS = Soda lime Silica Tableware (e.g. drinking glasses, dishes etc.)
 - TW DEC = Decorated (lip/rim) Tableware (e.g decorated with potential for food contact)
 - TW LC = Lead crystal Tableware (drinking glasses, decanters etc.)
 - CW SLS = Cookware Soda Lime Silica (oven dishes etc. usually thermally toughened)
 - CW/TW BS = Cookware/tableware borosilicate (oven to table ware, cafetieres etc. articles with good thermal shock resistance e.g. pyrex)
 - GC = Glass ceramic ware (example "visionware" – used for cookware)
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- AAS FES = Atomic Absorbtion Spectroscopy Flame Emission Spectroscopy. An analytical technique which uses excitement of elements by a flame to determine their concentration. Capable of parts per million sensitivity.
 - ICP-OES = Inductively Coupled Plasma Optical Emission Spectroscopy. An analytical technique which uses excitement of elements by a plasma to determine their concentration. This technique is generally capable of parts per billion sensitivity although this sensitivity does vary from element to element.

Chemical Element Abbreviations Used In This Report And Reporting Limits Used In General Analytical Data (Unless otherwise stated)

		Reporting Limit Used (ppm)	Method
Al	aluminium	0.05	ICP-OES
As	arsenic	0.1	ICP-OES
B	boron	0.03	ICP-OES
Ba	barium	0.01	ICP-OES
Bi	bismuth	0.1	ICP-OES
Ca	calcium	0.01	ICP-OES
Cd	cadmium	0.01	ICP-OES
Co	cobalt	0.02	ICP-OES
Cr	chromium	0.03	ICP-OES
Cu	copper	0.03	ICP-OES
Fe	iron	0.02	ICP-OES
K	potassium	0.01	AAS FES
Mg	magnesium	0.01	ICP-OES
Mn	manganese	0.01	ICP-OES
Na	sodium	0.02	AAS FES
Ni	nickel	0.02	ICP-OES
Pb	lead	0.1	ICP-OES
Sb	antimony	0.01	ICP-OES
Se	selenium	0.1	ICP-OES
Si	silicon	0.1	ICP-OES
Ti	titanium	0.1	ICP-OES
Zn	zinc	0.03	ICP-OES
Zr	zirconium	0.01	ICP-OES

- AAS FES operated by GTS Ltd – reporting limit quoted as 20x standard deviation by GTS (used as better sensitivity for Na and K analysis than ICP-OES)
- ICP-OES operated by London & Scandinavian Ltd (LSM) – reporting limit quoted as 10x standard deviation by LSM

Approximate Estimation Of Worst Case Experimental Uncertainty For The Data Presented

A simplified estimation of the uncertainty/error of data presented is given. This approximation attempts to take a worst case approach.

Algebraic sum of:-

errors encountered in multiple sample migration tests + errors encountered due to analysis technique + evaporation losses in testing

This data is available because

- GTS routinely carries out multiple sample migration testing and as a result has standard deviation and mean results for tests carried out on a series of (i.e. multiple) standard samples.
- Both GTS and LSM have statistics available on the analytical techniques used. (As multiple elements are analysed each with slightly differing standard deviations a worse case is assumed using sodium statistics)
- The evaporation loss error was calculated from weight loss of a range of test articles.

% Error has been calculated as (Std. Dev / mean) * 100

$$\sqrt{\frac{(\% \text{error migration testing})^2 + (\% \text{error analysis})^2 + (\% \text{evaporation loss})^2}{(N - 1)}}$$

$$= \sqrt{\frac{(5)^2 + (13.9)^2 + (45)^2}{2}}$$

$$= 33.5\%$$

$$= \text{Approximately } 35\% \text{ on final result}$$

i.e 1ppm = 1ppm ± 0.35ppm
 0.1ppm = 0.1ppm ± 0.035ppm
 0.01ppm = 0.01ppm ± 0.0035ppm

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