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JRC VALIDATED METHODS, REFERENCE METHODS AND MEASUREMENTS REPORT

Testing approaches for the release of metals from ceramic articles

In support of the revision of the Ceramic Directive 84/500/EEC

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Abstract

The overall aim of this study was to investigate the release of metals from ceramics into food and simulants, and to develop adequate methodologies for testing these articles including at lower limits. In the present work, the release of metals from 73 ceramic samples including 2 reference materials was investigated under different conditions. The sets of samples studied aimed to represent a vast variety of articles including hand crafted and highly decorated samples more prone to release a broader range and greater levels of metals. A comparison between two simulants and a benchmark food (acetic acid 4% for 24 h at 22°C, citric acid 0.5% for 2 h at 70°C and hot tomato sauce) showed that migration was higher in either simulants than into tomato sauce (2 h at 70°C). Results also showed that the migration profile in acetic acid was more representative for the tomato sauce. It was observed that is necessary to use of at least four replicates for each migration experiment due to the heterogeneity of the samples that Pb to a large variability of data. The relationship between the third and the first migration was shown to be not always constant and most of the time higher than 10% with respect to the first migration. This implies that prediction of values for the third migration based on the value of a first migration may not be derivable. Experiments on accelerated testing showed that increasing the acidity in an attempt to substitute the first two migrations in repeat testing were not successful from lack of clear relationships of the impact of acidity on release kinetics or mechanisms for different metals. No significant difference was observed between a test protocol with daily successive migrations and one with a lag phase (e.g. two days, representing a weekend). A longer storage (representing occasionally used articles) did not affect considerably the metals release for ceramics tableware. This implies that a repeat use testing is adequate regardless of the frequency of use of tableware articles, and that testing is not subjected to start on certain days of the weeks, which means productivity can be maintained if establishing a repeat use testing. For cookware, which had a smaller sample set of six different articles, three different profiles of Pb released were observed. For two samples the release of Pb over repeat testing in acetic acid 4% (24 h at 22°C) gave much higher values than those in boiling tomato sauce (6 h). For three samples the respective releases of Pb were comparable in both media. One sample presented higher releases of Pb and Cd in tomato sauce than in acetic acid 4%.

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Abstract

Materials and articles in contact with foods fall under a specific legislation at the EU level to ensure the safety of the consumer while facilitating trade. Specific limits are imposed on potential release of substances during contact with foods. For ceramics, limits are in place for lead (Pb) and cadmium (Cd) [1]. Scientific data has shown the need to lower the current limits [2-4]. Limits to the release of other metals such as arsenic (As), chromium (Cr), cobalt (Co), copper (Cu), manganese (Mn), nickel (Ni), etc., are also under consideration [5-11].

The overall aim of this study was to investigate the release of metals from ceramics into food and simulants, and to develop adequate methodologies for testing these articles including at lower limits. In the present work, the release of metals from 73 ceramic samples including 2 reference materials was investigated under different conditions. The sets of samples studied aimed to represent a vast variety of articles including hand crafted and highly decorated samples more prone to release a broader range and greater levels of metals.

Three different methodologies were compared and optimised. The first one currently in use foresees the use of acetic acid 4% as simulating liquid under test conditions of 22°C for 24 h. This procedure was repeated three times to better represent the repeated use regime. Two additional methodologies were investigated: 1) citric acid 0.5% 2 h at 70°C, three consecutives migrations and 2) an attempt of an accelerated test using a "conditioning" with acetic acid 10% (5 h at 22°C) followed by a single migration in acetic acid 4% (24 h at 22°C). The release of metals was also studied into tomato sauce as an example of benchmark food. The impact of a lag phase between consecutive tests was investigated. A study of the impact of storage on metals release was also performed to assess the effect of occasional use of articles. Testing conditions were compared to real use under cooking conditions for the articles intended for use as bakeware and cookware.

In the sets of samples investigated, a number of metals were found in leaching solutions e.g. aluminium (Al), iron (Fe), zinc (Zn), cobalt (Co), lithium (Li), barium (Ba), manganese (Mn), vanadium (V), lead (Pb), cadmium (Cd), antimony (Sb), titanium (Ti), chromium (Cr), nickel (Ni), copper (Cu) and arsenic (As). In general the release of metals seemed to be higher into citric acid 0.5% (2 h at 70°C) than into acetic acid 4% (24 h at 22°C). The migration was higher in either simulants than into tomato sauce (2 h at 70°C), with the results of the migration profile in acetic acid being more representative for the tomato sauce. Experiments on accelerated testing showed that the conditioning step led to releases much above the third migration in the repeat-use conventional test in acetic acid 4%.

No significant difference was observed between a test protocol with daily successive migrations and one with a lag phase (e.g. two days, representing a week end), when the ceramic article were left dry. The storage representing occasionally used articles did not affect considerably the metals release for ceramics tableware.

For cookware which had a smaller sample set of six different articles, three different profiles of Pb released were observed. For two samples the release of Pb over repeat testing in acetic acid 4% (24 h at 22°C) gave much higher values than those in boiling tomato sauce (6 h). For three samples the respective releases of Pb were comparable in both media. One sample presented higher releases of Pb and Cd in tomato sauce than in acetic acid 4%.

1. Introduction

Ceramic articles are manufactured from a mixture of inorganic materials with a generally high argillaceous or silicate content to which small quantities of organic materials may have been added. These articles are shaped and permanently fixed by firing [1]. Before firing a glaze can be applied by spraying or dipping [12]. The glaze melts and forms a glassy coating upon firing which improves the strength, cleanliness and appearance of the ceramic ware [13]. Glaze formulations can include different metals to achieve desired colourations, such as cobalt (Co) for blue, chromium (Cr) for green, cadmium (Cd), selenium (Se) and sulphur (S) for red/yellow/orange. Copper (Cu), magnesium (Mg), antimony (Sb) and vanadium can give stability or depth to a colour and calcium (Ca) and lead (Pb) are used to adjust the tone of the colour. The process and metal salts combination give the desired end colour [14]. While some studies showed the possibility of producing ceramicware without Pb in the glaze formulation [15], lead oxide (PbO) is still used in the glaze for its functional properties over a wide range of ceramicware compositions and firing ranges [4, 15].

During contact with food products, a release of trace elements such as Pb, Cd and other metals may occur from ceramics. The release/leaching process is a chemical attack on the glaze surface by acid-base reaction. It has been demonstrated that the leaching of metals from ceramic depends on several factors, including glaze composition, firing conditions, pH, temperature, physical properties of the food and time of contact [7].

Directive 84/500/EEC establishes specific migration limits (table 1) and basic rules for determining the migration of Pb and Cd in ceramics. It also reports test conditions to carry out the migration test using acetic acid 4% and an exposure contact time of 24 ± 0.5 h at 22 ± 2°C [1]. The European Commission may consider a potential revision the current Directive 84/500/EEC. Further restrictions have been proposed to take into account the revised EFSA opinions for these elements. So called "discussing starting values" (DSV) are around 400 times and 60 times lower respectively for Pb and Cd.

*Table 1 - Current limits for Pb and Cd (Directive 84/500/EEC) and DSVs**

Category	Current limits		DSV *	
	Pb	Cd	Pb	Cd
1ª	0.8 mg/dm^2	0.07 mg/dm^2	2.0 µg/ dm^2**	1.0 µg/dm^2**
2ª	4.0 mg/L	0.3 mg/L	**10 µg/kg**	**5 µg/kg**
3ª	1.5 mg/L	0.1 mg/L	3.8 µg/kg**	1.9 µg/kg**

* DSV: Discussing Starting Values; ** calculated related to the 2ª category

1ª Articles that cannot be filled and articles that can be filled; the internal depth does not exceed 25 mm; 2ª Articles that can be filled; 3ª Cooking ware; packaging and storage vessels having a capacity of > 3L.

The Council of Europe has listed specific release levels (SRLs) for components in Metals and Alloys used in food contact materials and articles (2013) [16]. The DSVs for several metals that could be present in ceramic samples are listed in table 2 in comparison to SRLs used by the CoE for metals and alloys. It is not necessarily the intention to add all these metals to a future legislation.

Table 2 – DSVs and SRLs (CoE Metals 2013) in µg/kg for elements other than Pb and Cd

Metal	DSV	SRL	Metal	DSV	SRL	Metal	DSV	SRL	Metal	DSV	SRL
Al	1000	5000	Cr	100000	250	Sn	50000	100000	Zn	1500	5000
As	18	2	Cu	1000	4000	Ni	72	140	Li	600	48
Ba	1000	1200	Fe	2500	40000	Ag	50	80	V	1200	10
Co	84	20	Mn	400	1800	Sb	40	40			

Metals other than Pb and Cd are not yet regulated at European level, but limits for some metals are under discussion as well (see section results). An expansion of scope could also be considered for similar articles such as crystalware.

2. Experimental design

Establishing lower limits implies that testing also needs to be updated to a more realistic repeated use test of three migrations. AS the duration of the test becomes 3 days, options to potentially accelerate a repeat-use testing were considered for comparisons. An increase of temperature to reduce the time of exposure, while feasible, would cause implementation issues for acetic acid (fumes). Alternative conditions were considered such as that used by Council of Europe (CoE) for Metals and Alloys with 0.5% citric acid 2 h at 70°C. The introduction of a accelerating pre-conditioning step of higher acidity [8] followed by one only single migration for with acetic acid 4% (22°C, 24 h) was also attempted.

In addition to food simulants also a real food was used to test the release of metals from ceramic. Tomato sauce was proposed as a worst case acidic foodstuff for ceramic articles. Food composition data indicate that tomato sauce products can have a pH as low as 3.5 [18]. Tomato sauce prepared ad-hoc with a 3.5 pH was used as benchmark food to represent the worst case acidic foodstuff for ceramic articles. Repeat tests into tomato sauce of commercial pH (4.2) and of acidified level to pH 3.5 were compared under conditions of hot fill (2 h at 70°C) for both articles of category 1[a] and 2[a].

Potential effects related to repeated use regime were investigated. Literature suggested that leaving overglaze decorated ceramic articles (i.e. overnight or week end) between repeat tests may cause higher release of Pb [19]. In the present study, the results of multiple migrations with a so called "weekend gap" of 48 hours in between migrations were investigated to evaluate its effect on the result of a compliance test.

The scope of the study encompassed both daily use ceramic articles as well as several luxury porcelain likely to be used on a more occasional basis. In this context, it included a study on the effect of storage on the release of metals release. Articles were tested for migration, stored for different periods from six months to one week, and re-tested to assess the possible evolution in the release of metals.

Testing conditions for cookware[1] were investigated. Kinetic studies into tomato sauce were performed in boiling tomato sauce of 3.5 pH up to 6 hours and analysing Pb release at several time points. Repeated use tests under different conditions in both simulants and food were compared.

The experimental plan is summarised below:

Optimisation of the release test:	Effects of different time gaps between three consecutive exposures:	Storage effect: Repeat testing of the same ceramic articles	Study on cookware:
3 migration tests with acetic acid 4% (22°C, 24 h) 3 migration tests with citric acid 0.5% (70°C, 2 h) 3 migration tests with tomato sauce (regular/acidified), (70°C, 2 h) Pre-conditioning test with acetic acid 10% (22°C, 5 h) followed by one migration test with acetic acid 4% (22°C, 24 h)	Testing with 48hrs between 1st and 2nd release tests Testing with week end gap between 2nd and 3rd release tests	after six months of storage after one month of storage after one week of storage 3 migration tests with acetic acid 4% (22°C, 24 h)	3 consecutive release tests with acetic acid 4% (22°C, 24 h) 3 consecutive kinetics with boiling tomato sauce (pH 3.5) for 6 h

[1] articles of category 3a intended to be heated in the course of preparation of food and drinks

3. Materials and methods

Migration tests were performed in food simulants: acetic acid 4% and citric acid 0.5%, and in tomato sauce (as conventional base of a worst-case common foodstuff).

3.1 Reagents

Acetic acid, 99-100% purity, Sigma-Aldrich; Citric acid, 99.5% purity, Fluka; Ultrapure Milli-Q water (≥18MΩ); Hydrogen Peroxide solution, TraceSELECT® ≥ 30,0% (RT), Fluka; Nitric acid, TraceSELECT®, for trace analysis ≥ 69,0% (T), Fluka; Metal standards 1000 µg/mL (1% HNO3), Analytical technology, Milano.

3.2 Benchmark food

Double concentrate Tomato "Carlo Manzella" was used and diluted 1:3 in order to reduce the viscosity of the product.

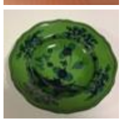
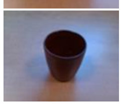
3.3 Ceramic samples

The samples used in this study were supplied by the stakeholders of this project. Both commercially available and ad-hoc produced samples with a significant release of metals were used in this project. Ad-hoc produced samples aimed to assess more precisely the difference between the testing modes under study. Three different categories were studied: flatware (1st category), hollowware (2nd category) and cookware (3rd category). The ceramic test articles used are reported in table 3 and the ad-hoc samples in table 4.

Table 3 - Ceramic samples

Sample	Description and test performed	Sample	Description and test performed
	Category 1ª (Flatware)		
	Multicolour plate (Ø = 22 cm) acetic acid 4% (22°C, 24 h) ; citric acid 0.5%; pre-conditioning		Blue coloured plate (Ø = 18 cm) acetic acid 4% (22°C, 24 h) ; citric acid 0.5%
	Multicolour plate (Ø = 16.5 cm) acetic acid 4% (22°C, 24 h) ; citric acid 0.5%		Multicolour plate (Ø = 19 cm) acetic acid 4% (22°C, 24 h) ; citric acid 0.5%
	Overglaze decorated plate (Ø = 19 cm) acetic acid 4% (22°C, 24 h) ; citric acid 0.5%; pre-conditioning		Overglaze decorated plate (Ø = 18.5 cm) acetic acid 4% (22°C, 24 h) ; citric acid 0.5%; pre-conditioning
	Yellow coloured plate (Ø = 12 cm) acetic acid 4% (22°C, 24 h) ; citric acid 0.5%; pre-conditioning		Green coloured plate (Ø = 12 cm) acetic acid 4% (22°C, 24 h) ; citric acid 0.5%; pre-conditioning
	Red coloured plate (Ø = 12 cm) acetic acid 4% (22°C, 24 h) ; citric acid 0.5%; pre-conditioning		Blue coloured plate (Ø = 20 cm) acetic acid 4% (22°C, 24 h) ; citric acid 0.5%; tomato sauce
	Pink coloured plate (Ø = 11 cm) acetic acid 4% (22°C, 24 h) ; citric acid 0.5%; pre-conditioning		Purple coloured plate (Ø = 11 cm) acetic acid 4% (22°C, 24 h) ; citric acid 0.5%; pre-conditioning

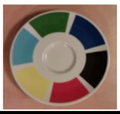
	Overglaze decorated plate (Ø = 30 cm) acetic acid 4% (22°C, 24 h) ; citric acid 0.5%; pre-conditioning		Overglaze decorated plate (Ø = 23.5 cm) acetic acid 4% (22°C, 24 h) ; citric acid 0.5%; pre-conditioning
	Multicolour plate (Ø = 21 cm) acetic acid 4% (22°C, 24 h)		Blue decorated plate (Ø = 22 cm) acetic acid 4% (22°C, 24 h)
	Multicolour plate (Ø = 26.5 cm) acetic acid 4% (22°C, 24 h)		Multicolour plate (Ø = 21 cm) acetic acid 4% (22°C, 24 h)
	Multicolour plate (Ø = 18.5 cm) acetic acid 4% (22°C, 24 h); citric acid 0.5%; Tomato sauce; pre-conditioning		Multicolour plate (Ø = 14.5 cm) acetic acid 4% (22°C, 24 h); citric acid 0.5%; pre-conditioning
	Black decorated plate (Ø = 10 cm) acetic acid 4% (22°C, 24 h); citric acid 0.5%;		Blue decorated plate (Ø = 10 cm) acetic acid 4% (22°C, 24 h); citric acid 0.5%;
	Brown decorated plate (Ø = 10 cm) acetic acid 4% (22°C, 24 h); citric acid 0.5%;		Multicolour plate (Ø = 28 cm) acetic acid 4% (22°C, 24 h); citric acid 0.5%; pre-conditioning
	Multicolour plate (Ø = 23.5cm) acetic acid 4% (22°C, 24 h); citric acid 0.5%; pre-conditioning		Blue decorated plate (Ø = 19.3 cm) acetic acid 4% (22°C, 24 h); citric acid 0.5%; pre-conditioning

Category 2ª (Hollowware)			
	Enamelled metal plate acetic acid 4% (22°C, 24 h); citric acid 0.5%		Enamelled measuring jug acetic acid 4% (22°C, 24 h); citric acid 0.5%
	Enamelled metal vase acetic acid 4% (22°C, 24 h); citric acid 0.5%		Blue coloured cup acetic acid 4% (22°C, 24 h); citric acid 0.5%
	Blue coloured bowl acetic acid 4% (22°C, 24 h); citric acid 0.5%		Red coloured cup acetic acid 4% (22°C, 24 h); citric acid 0.5%
	Multicolour decorated soup plate acetic acid 4% (22°C, 24 h)		Multicolour decorated soup plate acetic acid 4% (22°C, 24 h)
	Blue decorated soup plate acetic acid 4% (22°C, 24 h)		Multicolour decorated tray acetic acid 4% (22°C, 24 h)
	Multicolour decorated soup plate acetic acid 4% (22°C, 24 h)		Brown cup acetic acid 4% (22°C, 24 h); citric acid 0.5%; tomato sauce; pre-conditioning
	Yellow bowl acetic acid 4% (22°C, 24 h); citric acid 0.5%		Coloured bowl acetic acid 4% (22°C, 24 h); citric acid 0.5%
	Yellow mortar acetic acid 4% (22°C, 24 h)		Brown cup acetic acid 4% (22°C, 24 h); citric acid 0.5%

	Brown bowl acetic acid 4% (22°C, 24 h); citric acid 0.5%; pre-conditioning		Green cup acetic acid 4% (22°C, 24 h); citric acid 0.5%; pre-conditioning
	Brown cup acetic acid 4% (22°C, 24 h)		Light blue coloured plate acetic acid 4% (22°C, 24 h); citric acid 0.5%
	Jar acetic acid 4% (22°C, 24 h); citric acid 0.5%		Jar acetic acid 4% (22°C, 24 h); citric acid 0.5%
	Blue bowl acetic acid 4% (22°C, 24 h);		Green bowl acetic acid 4% (22°C, 24 h);

Category 3ª (Cooking ware)			
	Brown bowl acetic acid 4% (22°C, 24 h); citric acid 0.5%; tomato sauce		Brown bowl acetic acid 4% (22°C, 24 h); citric acid 0.5%;
	Brown bowl acetic acid 4% (22°C, 24 h); citric acid 0.5%; tomato sauce		Brown bowl acetic acid 4% (22°C, 24 h); citric acid 0.5%
	Brown bowl acetic acid 4% (22°C, 24 h); citric acid 0.5%		Brown bowl acetic acid 4% (22°C, 24 h); citric acid 0.5%
	Brown square tray acetic acid 4% (22°C, 24 h); tomato sauce		Crème broule bowl acetic acid 4% (22°C, 24 h); citric acid 0.5%; tomato sauce
	Brown bowl acetic acid 4% (22°C, 24 h); citric acid 0.5%; tomato sauce ; pre-conditioning		Black bowl acetic acid 4% (22°C, 24 h); citric acid 0.5%; tomato sauce ; pre-conditioning
	Brown bowl acetic acid 4% (22°C, 24 h); citric acid 0.5%; tomato sauce		Brown bowl acetic acid 4% (22°C, 24 h);
	Ramekin regency green, acetic acid 4% (22°C, 24 h)		Ramekin Imperial blue, acetic acid 4% (22°C, 24 h)
	Red bowl, acetic acid 4% (22°C, 24 h), tomato sauce		Blue tray, decorated inside in grey, acetic acid 4% (22°C, 24 h)
	Blue bowl, acetic acid 4% (22°C, 24 h), tomato sauce		Blue/white bowl, acetic acid 4% (22°C, 24 h)
	Cast iron cookware, acetic acid 4% (22°C, 24 h)		Steel cookware, acetic acid 4% (22°C, 24 h)
	Steel cookware, acetic acid 4% (22°C, 24 h)		

Table 4 - Ceramic samples manufactured ad-hoc

Sample	Description and type of test performed
	Overglaze decorated plate (Ø = 10 cm) acetic acid 4% (22°C, 24 h); citric acid 0.5%; tomato sauce
	Underglaze decorated plate (Ø = 10 cm) acetic acid 4% (22°C, 24 h); citric acid 0.5%; tomato sauce

3.4. Apparatus

3.4.1 ICP-MS

Two quadrupole inductively-coupled plasma mass spectrometers was used in this study: A PerkinElmer NexIon 300D (figure 1A) equipped with a concentric nebulizer Meinhard, a glass cyclonic spray chamber and a standard torch (2.5 mm i.d.) and an Agilent Serie 7700 (figure 1B) equipped with a concentric nebulizer, a Scott quartz spray chamber and a standard torch (2.5 mm i.d.).

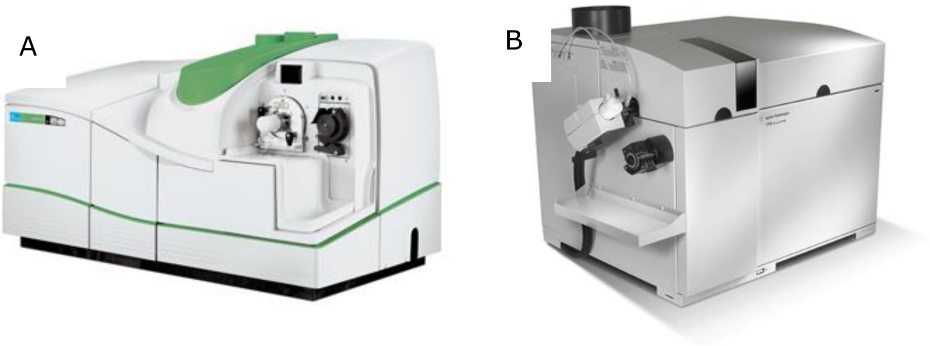


Figure 1 – A: ICP-MS Perkin Elmer NexIon 300D; B: ICP-MS Agilent 7700

The Working Operating Procedure (WOP) used for the determination of Pb, Cd and other metals such as Ag, Al, As, Ba, Co, Cr, Cu, Fe, Li, Mn, Mo, Ni, Sb, Sn, Sr, Ti, V, Zn and Zr in acetic acid 4% (v/v) or citric acid 0.5% (w/v) using ICP-MS is reported in annex 1. The periodic table of elements is reported in annex 2.

3.4.2 Digestor

An Anton Paar multiwave 3000 SOLV - XF 100-8 positions rotor was used (figure 2).

Figure 2 – Microwave digestion system

3.5 Migration methodologies in food simulants

3.5.1 Migration in acetic acid 4% (22°C, 24 h) (84/500/EEC)

Test liquid: 4% (v/v) acetic acid, in a freshly prepared aqueous solution.

Procedure: 4 identical units were taken and cleaned with non-acidic diluted detergent and tap water at approximately 40°C followed by Milli-Q water and dried. The articles were filled with the test liquid to a level no more than 1 mm from the overflow point (measured from the upper rim of the sample). Samples with a flat or slightly sloping rim were filled so that the distance between the surface of the liquid and the overflow point was no more than 6 mm measured along the rim. The test was carried out at a temperature of 22 ± 2°C for 24 ± 0.5 h using an incubator to maintain the temperature control. The samples were exposed to the simulant three times. After the first migration (I), the samples were washed with Milli-Q water and dried. Afterwards, they were refilled with fresh simulant and incubated for the second migration[2] (II). The same procedure was followed to conduct the third migration experiment (III).

3.5.2 Migration in citric acid 0.5%, 70°C, 2 h

Test liquid: 0.5% (w/v) citric acid, in a freshly prepared aqueous solution.

Procedure: The simulant was pre-heated using a hot plate or an oven to 70°C and the test pieces also equilibrated to 70°C. The temperature of the oven was set so that the temperature of the simulant was 70°C during the migration test[3]. 4 identical units were taken and cleaned with non-acidic diluted detergent and tap water at approximately 40°C followed by Milli-Q water and dried. Each sample was filled with a citric acid 0.5% inside the oven in order to minimize the drop in temperature. The test was carried out at a temperature of 70 ± 2°C for 2 h ± 5 min using an incubator. The samples were exposed to the simulant three times as described above.

3.5.3 Accelerated test in acetic acid (10%, 22°C, 5 h + 4%, 22°C, 24 h)

Pre-treatment liquid: 10% (v/v) acetic acid aqueous solution.

Test liquid: 4% (v/v) acetic acid, in a freshly prepared aqueous solution.

Procedure: 4 identical units were taken and cleaned with non-acidic diluted detergent and tap water at approximately 40 °C followed by Milli-Q water and dried. The articles were filled with a 10% (v/v) acetic acid solution to as close as possible to the edge (i.e. almost overflowing). This ensured that the area contacted by the subsequent 4% acetic acid solution had all been exposed[4]. The test items were left covered in the incubator at a temperature of 22 ± 2°C for 5 h ± 5 minutes[5]. The articles were then removed and the acetic acid solution discarded. They were rinsed with Milli-Q water and allowed to dry before further contact with acetic acid 4%. Following the pre-treatment step, the items

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[2] Note: The time between each successive migration should be as short as possible anyway no more than one hour. Long time between each migration could increase the migration of the following migration step.

[3] Note: It is recommended to track the temperature of the blank sample with a temperature probe to maintain the traceability of the temperature of the simulant itself.

[4] Note: drinkware that is to test by immersion may simply be immersed after any areas not to be tested have been covered with a suitable paraffin wax (or similar). There is no specific volume/ratio required, but ware should be covered.

[5] Note: It is recommended to track the temperature of the blank sample with a temperature probe to maintain the traceability of the temperature of the simulant itself.

were filled with a 4% (v/v) acetic acid solution and the test was carried out at a temperature of 22 ± 2°C for 24 ± 0.5 h using an incubator[6].

3.5.4 Sample treatment after migration

Samples obtained applying these methods (3.5.1- 3.5.3) were treated in the same manner before analysing them by ICP-MS: 1 mL of simulant was added with 100 µL of internal standard solution at 1000 µg/L (^{185}Re, ^{45}Sc, ^{103}Rh, ^{89}Y) and filled up to 10 mL with Milli-Q water (dilution 1/10). The dilution depended strictly from the concentration of each individual metal[7]. The internal standard concentration was kept constant independently from the dilution applied (10 µg/L).

3.5.5 Preparation of the calibration curve

Calibration curves were prepared by using standard solutions of 1000 µg/mL of each metal and diluting with a suitable percentage of acetic 4% or citric acid 0.5% in ultrapure water. These acids were added to the standard in order to have the same matrix effect as in the sample during the measurements. Calibration curves were prepared as follows: 1 mL of acetic acid 4% (v/v) or citric acid 0.5% (w/v), 100 µL of Internal standards solution (1000 µg/L) of ^{185}Re, ^{45}Sc, ^{103}Rh, ^{89}Y to have a final concentration of 10 µg/L, the proper aliquot of metals stock solution to reach the concentration required and fill up to 10 mL with Milli-Q water. The calibration curve was built up to cover the entire range of the concentration of metals in the sample. Samples that required a dilution 1/100 were quantified using the calibration curve prepared as follows: 100 µL of acetic acid 4% (v/v) or citric acid 0.5% (w/v), 100 µL of internal standard solution (1000 µg/L), the proper aliquot of metals stock solution to reach the concentration required and fill up to 10 mL with Milli-Q water.

3.6 Migration experiments in a benchmark food

Tomato sauce was chosen as a representative worst case food for ceramics. Food composition tables report a range of pH for tomato sauce from 3.5 to 4.7 [18]. A survey over ca. 30-40 brands conducted in house did not identify a tomato commercially available with a pH as low as 3.5. All samples had a pH around 4.2. A comparison was performed using a tomato sauce with pH 4.2 vs. a tomato sauce acidified to pH 3.5, in order to assess the effect of pH on the migration of metals.

3.6.1 Tableware: migration into tomato sauce at 70°C for 2 h

The conventional test conditions for tomato under "real use" for ceramic tableware were chosen as isothermal conditions 2 h at 70°C, which mimic food being kept hot and is used as a convention for hot fill for plastics.

Test liquid: Acidified tomato of pH 3.5 was prepared as follows: 670 g of double concentrated tomato sauce was added with 120 g of citric acid 20 % (w/v) solution and 1210 g of Milli-Q and mixed carefully in order to reach a 3.5 pH and to dilute the double concentrated tomato three times (1:3). The pH was checked with a pH-metre and eventually adjusted to pH 3.5.

Procedure: 4 identical units were taken and cleaned with diluted non-acidic detergent and tap water at approximately 40°C followed by Milli-Q water and dried. The test was carried out at a temperature of 70°C for 2 hours. The tomato sauce and the ceramic ware samples were both pre-heated to 70°C before filling. The test specimens were filled with tomato sauce to a level no more than 1 mm from the overflow point; the distance

[6] Note: The laps of time between pre-treatment with acetic acid 10% (v/v) and the migration with acetic acid 4% (v/v) migrations should be no more than one hour.

[7] It was sometimes necessary to dilute the samples 100 times before the analysis.

was measured from the upper rim of the sample. These specimens exhibiting a flat or slightly sloping rim were filled so that the distance between the surface of the liquid and the overflow point was no more than 6 mm measured along the rim. The weight of tomato sauce used to fill the sample was recorded. The temperature of the oven was set to ensure that the temperature of the tomato sauce itself was 70°C during the entire migration test. After 2 hours the entire tomato sample was collected in a Schott bottle, the bottle was closed and shaken carefully to ensure the homogeneity of sample.

The migration procedure was repeated three times. Between consecutive migrations, ceramic articles were washed with distilled water followed by Milli-Q water and dried. The ceramic articles were refilled with fresh tomato sauce at 70°C and incubated for the second migration. The same procedure was followed to conduct the third migration. The lag time between consecutive migrations was no more than 1 hour.

3.6.2 Cookware: Kinetics into boiling tomato sauce for 6 hours

Test liquid: Acidified tomato of pH 3.5 was prepared as in paragraph 3.6.1.

Procedure: 4 identical units were taken and cleaned with diluted non-acidic detergent and tap water at approximately 40°C followed by Milli-Q water and dried. The test used dedicated large size hot plates to ensure controlled temperature. The tomato sauce as well as test specimens were pre-heated. The test was carried out in boiling tomato sauce for 6 hours. The test samples were filled with tomato sauce to a level no more than 1 mm from the overflow point and the distance was measured from the upper rim of the sample. The weight of tomato sauce used to fill the sample was recorded. The temperature of the hot plate was controlled to keep a slow boil of the tomato sauce during the entire test. Evaporation was controlled by covering the samples with unleaded glass or Teflon lids and by adding boiling Milli-Q water, when necessary, to keep the volume constant. After 0.5, 1, 2, 3, 4, 5 and 6 h aliquots of 5 grams of tomato sauce were taken for analysis and the same amount of fresh tomato sauce was restored into ceramic articles immediately after sampling. Before each sampling the tomato sauce was stirred with Teflon rods for around 30 seconds to ensure the sample homogeneity.

The kinetic was repeated three times. Between consecutive kinetics, ceramic articles were washed with distilled water followed by Milli-Q water and dried.

Because of the loss of Pb in the extraction tomato sauce resulting from periodically sampling, the concentration of Pb was corrected using the following equation:

$$y_j = x_j + \frac{V_{taken}}{V_{total}} \cdot x_{j-1} \qquad (y_1 = x_1;\ j = 2,3,..,n)$$

Where y_j is the concentration corrected and x_j is the measured value [20].

3.6.3 Sample treatment

The tomato sample was digested using a microwave digestor (4.4.2) prior to analysis with ICP-MS, as follows: 4 g of tomato sauce (after migration) was weighed in a Teflon tube, to which 7 mL of HNO_3 and 3 mL of H_2O_2 were added and left under the fume hood for 30 minutes. The samples were then placed into the microwave digestion system using the programme reported in table 5.

Table 5 - Tomato sauce digestion program

Power	Ramp	Hold	Fan
800 W	10 min	10 min	1
0 W		15 min	3
p-rate: 0.5 bar/s	IR: 180°C	p:60 bar	Stirrer: Off

The digested tomato sauce was weighed and then diluted to 30 g with of Milli-Q to reach an acidity of approximately 30%. A volume of 1.5 mL of this 30% acidic solution received 100 µL of internal standard and was filled up to 10 mL with Milli-Q. This further dilution was necessary to have a solution of an acidity of approximately 5% adequate for the ICP-MS analysis.

3.6.4 Preparation of the calibration curve

Calibration curves were prepared in the same matrix as the sample. A blank sample of tomato sauce was digested and used to prepare the standards of the calibration curve. Acid concentration in calibration standards and sample were the same. 1.5 mL of digested blank tomato sauce was added with 100 µL of internal standard solution (1000 µg/L) and the proper aliquot of metals stock solution to reach the concentration required and filled up to 10 mL with Milli-Q water. The calibration curve was built up to cover the entire range of the concentration of metals in the sample.

4. Results and discussion

4.1 Characteristics of the analytical method

All results reported in the tables were average values and the corresponding standard deviation was derived from four replicates obtained after first (I), second (II) and third (III) migration. The results for flatware (Cat. 1[a]) were expressed in µg/dm^2 and for hollowware (Cat. 2[a]) and cooking ware (Cat. 3[a]) in µg/kg. Results for a number of samples were analysed in collaboration with NRL-BE, NRL-DE and NRL-UK and are reported in annex 3 and 4.

Method detection limits (LOD) were calculated from the standard deviation of the blank. For the calculation of LOD, 10 determinations of the blank samples were analysed according to the same analytical method and their standard deviation was calculated. The LOD of the entire method was calculated as: LOD $=x_{bl} + 3\ sd_{bl}$, where x_{bl} is the means concentration calculates from the counts of the noise peak for the 10 determinations; sd_{bl} is the standard deviation of the analysis [21]. The LODs are reported in table 6a.

Table 6a– LOD values in µg/kg for different methods

Acetic Acid 4% [µg/kg]									
Li	**Al**	**Mn**	**Cu**	**Ti**	**Co**	**Zn**	**Cd**	**Ba**	**Pb**
0.05	0.55	0.15	0.10	0.10	0.11	8.21	0.01	0.04	0.26
Zr	**Fe**	**V**	**Ni**	**Cr**	**Ag**	**Sn**	**Sb**	**As**	
1.87	2.92	0.11	0.19	0.06	0.04	0.54	0.01	0.09	
Citric Acid 0.5% [µg/kg]									
Li	**Al**	**Mn**	**Cu**	**Ti**	**Co**	**Zn**	**Cd**	**Ba**	**Pb**
0.01	2.07	0.27	0.29	0.24	0.05	6.83	0.03	0.16	0.20
Zr	**Fe**	**V**	**Ni**	**Cr**	**Ag**	**Sn**	**Sb**	**As**	
0.11	0.42	0.04	0.12	0.26	0.06	0.10	0.01	0.16	
Tomato sauce [µg/kg]									
Li	**Al**	**Mn**	**Cu**	**Ti**	**Co**	**Zn**	**Cd**	**Ba**	**Pb**
0.42	65.6	11.8	11.8	32.3	0.04	16.9	1.85	10.9	3.01
Zr	**Fe**	**V**	**Ni**	**Cr**	**Sb**	**As**			
0.05	9.19	1.49	5.38	1.78	0.09	0.04			

4.2 Background levels of the reference food

The metal background of tomato sauce was measured in order to establish the concentration of metals already present in the matrix. Table 6b shows the values of metals in tomato sauce diluted three times with Milli-Q water.

Table 6b – Values of metals in tomato sauce diluted 1:3

Metal	Concentration [µg/kg]	RSD [%]
Cd	19	9.7
Pb	5	15.5
Li	10	8.4
Al	1206	11.8
Ti	601	4.7
V	26	9.7
Cr	62	8.9
Ni	150	6.5
Cu	1569	8.1
Zn	2600	4.5
Sn	448	28.3
Ba	137	6.0
Mn	1394	4.6
Fe	5627	8.1
Co	7	5.4

The table indicates that for several metals it was possible to discriminate the amount of metals migrated from the ceramics articles from those already present in the matrix only if the level of migrated metals from the articles themselves was high enough. This was the case of Al, Cu, Zn, Mn or Fe present in high concentration in the food.

4.2 Comparison of release tests

A number of metals were released in different concentrations from the ceramic articles tested. The metals released into food simulants and tomato sauce were for example Li, Al, Mn, Cu, Ti, Cr, V, Fe, Zn, Ba, Ni, Mo, As, Co, Cu, Zr, Sr, Sn, Cd and Pb. For many items significant "article to article" variability was observed in the migration data thus the use of at least four items remains a strong recommendation for testing compliance. The amount and combination of metals depended on the sample tested and methodology used. The release values sometimes were very low and the standard deviations were high. This gave an indication of the migration of metals from real samples.

4.2.1 Acetic acid 4% (22°C, 24 h) versus citric acid 0.5 % (70°C, 2 h)

47 samples of the three categories were used to compare the two methodologies. For all samples three consecutive exposures were carried out. Both tests presented advantages and drawbacks. The release test with acetic acid 4% (22°C, 24 h) is easier to manage, it has the possibility to use a migration room as alternative to individual ovens thus a larger number of samples can be analysed in parallel. However acetic acid has a strong smell and the repeat testing amounts to three days and requires a more careful planning of experiments (to avoid week-ends). The release test with citric acid 0.5% (70°C, 2 h) is less time consuming and citric acid is odourless but due to the use of a higher temperature, a number of ovens is required to perform parallel experiment resulting in high energy consumption. There are also several additional issues owing to the use of a higher temperature such as evaporation and equilibration of the simulant and control of the temperature during the exposure. Safety concerns have to be taken into account managing hot simulant with larger articles. All these considerations are extremely important to identify an appropriate release test that can be used routinely by both industry and control laboratories.

Pb and Cd concentration values in leachates obtained in the first and third exposure with acetic acid 4% (22°C, 24 h) and with citric acid 0.5% (70°C, 2 h) are reported in table 7a (hollowware and cookware) and in table 7b (flatware).

Table 7a –Pb and Cd release in acetic acid 4% (22°C, 24 h) versus citric acid 0.5% (70°C, 2 h) from hollowware and cooking ware

I MIGRATION						**III MIGRATION**					
Sample		**Cd [µg/kg]**		**Pb [µg/kg]**		**Sample**		**Cd [µg/kg]**		**Pb [µg/kg]**	
		AA4%	**CA0.5%**	**AA4%**	**CA0.5%**			**AA4%**	**CA0.5%**	**AA4%**	**CA0.5%**
C6	Av	0.12	0.08	1583.32	1458.63	C6	Av	0.12	0.07	1203.32	1632.96
	RSD%	33.92	20.77	13.73	7.18		RSD%	33.92	20.77	23.14	15.70
C14	Av	0.06	0.07	27.02	88.73	C14	Av	0.05	0.07	14.95	68.81
	RSD%	27.21	17.60	24.54	77.60		RSD%	84.31	41.06	25.06	96.87
113C01	Av	0.07	0.527	0.99	0.942	113C01	Av	<LOD	0.132	<LOD	<LOD
	RSD%	29.46	99.56	33.69	35.73		RSD%		69.802		
113C02	Av	<LOD	0.21	1.31	10.45	113C02	Av	<LOD	1.08	0.69	0.61
	RSD%		44.87	96.12	26.48		RSD%		-		77.90
113C03	Av	1.45	1.92	<LOD	2.29	113C03	Av	0.17	0.17	<LOD	<LOD
	RSD%	107.23	46.65		14.25		RSD%	95.95	44.91		
213C07	Av	0.01	0.02	0.58	0.40	213C07	Av	0.01	0.01	0.31	0.19
	RSD%	43.26	7.03	28.09	34.02		RSD%	11.87	26.72	63.35	112.33
213C09	Av	<LOD	0.23	<LOD	<LOD	213C09	Av	<LOD	<LOD	<LOD	<LOD
	RSD%		-				RSD%				
413C06	Av	0.07	0.13	0.80	0.38	413C06	Av	41.93	0.03	4.10	0.10
	RSD%	25.53	61.58	76.31	88.67		RSD%	141.39	67.51	142.82	59.61
713C01	Av	0.53	0.71	347.50	679.77	713C01	Av	0.29	0.59	41.40	325.43
	RSD%	6.74	9.41	6.84	5.54		RSD%	20.92	10.04	9.15	10.25
713C02	Av	0.10	<LOD	764.39	1250.93	713C02	Av	0.05	<LOD	152.33	430.55
	RSD%	45.66		27.16	22.79		RSD%	76.66		35.46	34.97
713C03	Av	<LOD	<LOD	211.58	464.00	713C03	Av	<LOD	0.07	13.91	42.81
	RSD%			23.41	12.12		RSD%		10.88	18.70	19.47
713C04	Av	<LOD	0.21	243.72	808.59	713C04	Av	<LOD	<LOD	53.79	152.47
	RSD%		-	57.79	29.06		RSD%			10.22	30.72
713C05	Av	<LOD	<LOD	7.39	12.36	713C05	Av	<LOD	<LOD	1.67	2.90
	RSD%			51.51	43.54		RSD%			32.18	29.20
713C06	Av	0.39	0.43	1094.09	1522.06	713C06	Av	0.52	0.42	1423.31	2465.90
	RSD%	71.86	28.31	52.27	40.83		RSD%	47.90	15.81	25.21	13.44
713C07	Av	<LOD	<LOD	1.06	1.46	713C07	Av	<LOD	<LOD	<LOD	<LOD
	RSD%			47.49	34.36		RSD%				
713C08	Av	11.49	0.28	2973.80	3353.47	713C08	Av	0.08	0.13	586.32	1659.80
	RSD%	167.82	79.23	71.03	92.34		RSD%	32.16	41.56	12.29	52.54
713C11	Av	<LOD	<LOD	466.36	509.73	713C11	Av	<LOD	<LOD	335.19	452.23
	RSD%			42.22	21.07		RSD%			52.14	37.98
713C12	Av	<LOD	<LOD	489.84	467.38	713C12	Av	<LOD	<LOD	106.38	454.80
	RSD%			13.02	26.54		RSD%			14.19	52.58
713C15	Av	0.67	1.00	11034.90	20851.90	713C15	Av	0.14	0.11	2498.33	875.08
	RSD%	25.10	36.37	12.88	39.75		RSD%	18.44	-	10.50	30.56
713C16	Av	0.02	0.01	1244.85	1018.05	713C16	Av	<LOD	<LOD	197.02	261.62
	RSD%	73.17	67.54	34.80	62.17		RSD%			39.38	90.14
713C23	Av	<LOD	<LOD	75.65	131.10	713C23	Av	<LOD	<LOD	7.74	58.23
	RSD%			18.17	17.14		RSD%			27.57	44.85
713C28	Av	1.67	2.11	19061.17	20357.65	713C28	Av	0.23	0.27	2332.85	2974.84
	RSD%	31.20	9.55	5.75	12.71		RSD%	31.51	27.54	10.11	21.83
913C03	Av	<LOD	<LOD	0.40	<LOD	913C03	Av	<LOD	<LOD	<LOD	<LOD
	RSD%			18.63			RSD%				
913C08	Av	0.01	0.01	0.33	<LOD	913C08	Av	<LOD	<LOD	<LOD	<LOD
	RSD%	104.75	107.96	17.60			RSD%				
913C09	Av	0.01	0.01	0.52	<LOD	913C09	Av	<LOD	<LOD	<LOD	<LOD
	RSD%	74.69	97.67	65.82			RSD%				

Table 7b –Pb and Cd release in acetic acid 4% (22°C, 24 h) versus citric acid 0.5% (70°C, 2 h) from flatware

I MIGRATION						III MIGRATION					
Sample		Cd [μg/dm²]		Pb [μg/dm²]		Sample		Cd [μg/dm²]		Pb [μg/dm²]	
		AA4%	CA0.5%	AA4%	CA0.5%			AA4%	CA0.5%	AA4%	CA0.5%
213C02	Av	0.003	0.004	0.07	0.045	213C02	Av	<LOD	0.001	<LOD	0.007
	RSD%	71.80	51.98	64.91	65.40		RSD%		138.67		-
213C08	Av	0.01	0.002	0.42	0.19	213C08	Av	0.01	0.02	0.26	0.07
	RSD%	126.31	30.50	135.96	90.09		RSD%	151.48	-	121.77	89.34
213C11	Av	<LOD	0.02	<LOD	0.04	213C11	Av	<LOD	0.01	0.21	0.08
	RSD%		4.71		-		RSD%		-	-	75.99
413C02	Av	11.73	13.01	181.65	182.24	413C02	Av	3.70	2.14	72.30	26.47
	RSD%	34.54	15.68	37.40	6.01		RSD%	16.54	20.56	32.18	12.41
413C04	Av	0.02	0.03	12.53	35.03	413C04	Av	<LOD	0.0043	2.01	8.285
	RSD%	53.19	48.40	15.98	49.38		RSD%		66.67	13.30	77.44
413C11	Av	0.007	0.009	7.12	12.15	413C11	Av	<LOD	0.002	0.61	2.74
	RSD%	16.22	7.28	7.10	4.41		RSD%		45.84	25.90	10.88
413C13	Av	0.001	0.0004	0.08	0.01	413C13	Av	0.001	<LOD	0.09	<LOD
	RSD%	29.55	151.64	10.49	-		RSD%	12.28		19.34	
413C15	Av	15.44	34.00	111.29	251.56	413C15	Av	3.66	22.84	25.93	166.38
	RSD%	14.14	28.56	14.60	28.87		RSD%	18.63	47.75	20.38	50.88
413C17	Av	38.68	64.01	150.37	253.79	413C17	Av	7.30	26.60	26.19	73.26
	RSD%	6.04	8.22	4.71			RSD%	1.70	6.78	3.03	12.67
413C18	Av	0.003	0.22	11.60	13.00	413C18	Av	<LOD	<LOD	0.71	3.39
	RSD%	100.07	198.96	29.01	29.35		RSD%			29.06	36.24
413C21	Av	0.03	0.12	155.52	646.15	413C21	Av	<LOD	0.04	50.36	236.67
	RSD%	58.34	71.51	62.47	67.95		RSD%		67.27	38.67	63.32
413C22	Av	0.70	4.88	63.83	371.83	413C22	Av	0.10	3.39	9.31	240.68
	RSD%	20.34	46.58	15.13	44.55		RSD%	26.12	51.29	22.25	51.02
413C26	Av	0.02	0.13	3.46	7.15	413C26	Av	0.005	0.006	1.20	0.63
	RSD%	14.21	79.01	15.48	64.39		RSD%	29.08	54.01	31.52	22.54
413C27	Av	0.01	0.04	6.36	10.98	413C27	Av	0.003	0.007	1.74	2.26
	RSD%	13.94	20.39	16.39	28.41		RSD%	34.64	22.88	10.82	19.55
713C09	Av	0.02	0.007	40.95	90.00	713C09	Av	0.01	0.02	6.46	49.51
	RSD%	47.24	83.51	41.25	53.83		RSD%	55.38	87.94	36.74	76.41
713C17	Av	<LOD	<LOD	0.06	0.07	713C17	Av	<LOD	<LOD	<LOD	<LOD
	RSD%			142.59	14.88		RSD%				
713C18	Av	<LOD	<LOD	0.03	0.17	713C18	Av	<LOD	<LOD	<LOD	0.03
	RSD%			34.24	34.65		RSD%				117.21
713C19	Av	0.05	0.01	0.30	0.19	713C19	Av	<LOD	<LOD	0.03	0.32
	RSD%	101.02	-	79.62	118.16		RSD%			-	99.20
713C20	Av	0.0002	<LOD	20.96	42.29	713C20	Av	<LOD	<LOD	2.58	8.80
	RSD%	137.45		9.19	6.89		RSD%			11.13	26.41
713C22	Av	0.02	0.03	0.73	0.98	713C22	Av	0.01	0.02	0.19	0.47
	RSD%	45.40	39.45	31.30	12.06		RSD%	40.24	54.22	39.25	83.12
913C02	Av	0.09	0.27	1.79	3.74	913C02	Av	0.01	0.05	0.26	0.73
	RSD%	26.17	24.66	28.80	26.29		RSD%	27.33	14.57	27.65	13.38
913C05	Av	<LOD	<LOD	0.20	0.22	913C05	Av	<LOD	<LOD	0.02	0.002
	RSD%			30.96	71.65		RSD%			18.33	140.87

Usually the Pb and Cd release decreased across three consecutive exposures. However, this decrease was far from constant and was not necessarily the norm for other elements.

It was difficult to compare the two methods due to the difference in time and temperature conditions. In general the metal release was higher in citric acid 0.5% than in acetic acid 4%. However, the nature/type of the sample could influence the process. It was possible to identify few groups of articles behaving similarly.

The highest Pb release was often present in earthenware articles, for example samples C6, C14, 713C01, 713C02, 713C03, 713C04, 713C05, 713C06, 713C08, 713C09, 713C011, 713C12, 713C15, 713C16, 713C23 and 713C28. In those articles, the difference between using acetic acid 4% (22°C, 24 h) and citric acid 0.5% (70°C, 2 h) was also more evident. In the majority of the cases, the Pb release was higher in citric acid 0.5% than in acetic acid 4%. For this type of articles upon migration in acetic acid 4%, several samples showed cracks on the surface and presence of calcium carbonate, as is shown in figure 3. This phenomenon could have an effect on the concentration of metals released during migrations.

Figure 3 – Example of earthenware article a) not exposed, b) exposed to acetic acid 4% (22°C, 24 h) and c) exposed to citric acid 0.5%

Another group of overglaze decorated samples such as samples 413C04, 413C11, 413C26, 413C27, 713C20 and 913C02 presented a significant release of Pb. In the overglaze decoration process clay ware is fired at a high temperature, glazed, and fired a second time at a high temperature. A design is applied on top of the glaze and in order to fuse the decoration with the glaze, the item is re-fired at a lower temperature. Since overglaze colours are not subjected to high temperatures, they are more vivid than those used in high-temperatures underglaze decoration technique, but they are also more vulnerable to wear, damage and may leach during contact. In overglaze samples analysed in this study the Pb release was higher in citric acid 0.5% (70°C, 2 h) than in acetic acid 4% (22°C, 24 h). The same trend was confirmed with the test performed on ad-hoc prepared samples (613C04 and 613C05) realized with the same decoration applied on over and under glaze: the results for Li, Al, Fe, Co, Pb and Cd are shown in figure 4. For all metals, testing with citric acid 0.5% (70°C, 2 h) seemed to be more aggressive than the test with acetic acid 4% (22°C, 24 h). Taking into account the type of decoration, generally speaking, overglaze ad-hoc samples released higher concentrations of Li, Al, Fe, Pb and Cd. One exception was for Co, for which the highest amount of this element was present in leachates from samples with underglaze decoration.

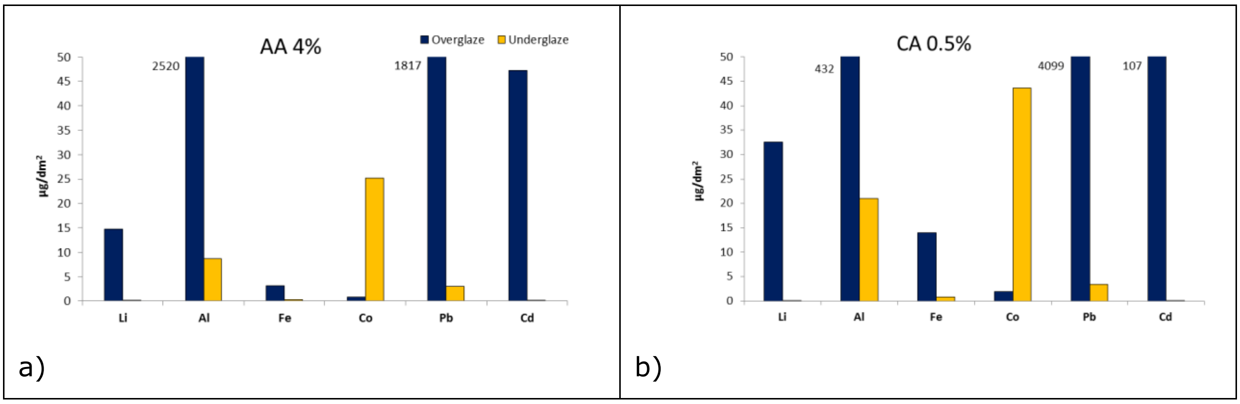


Figure 4 – Metals release from overglaze and underglaze decorated articles after first exposure to a) acetic acid 4% (22°C, 24 h) and b) citric acid 0.5% (70°C, 2 h)

Some samples with brilliant solid colours released Pb when in contact with simulants. The concentration values in leachates increased from pale yellow colour sample (413C13), green sample (413C15) to red sample (413C17). However, other samples with colourful decoration such as 213C02, 213C07, 213C08, 213C09, 213C11, 613C22 and 913C05 showed very low release of Pb and Cd (e.g. lower than the detection limit)

A few enamelled articles (113C01, 113C02 and 113C03) were also tested. The release of Pb and Cd was low with no difference between the two simulants. These enamelled samples released also other metals in particular Al, Mn, Fe, Co, Zn, Cu, Ba, Li and Ti.

Aluminium was released predominantly from Earthenware articles (e.g. samples C14, 713C01, 713C02, 713C04, 713C05, 713C06, 713C08, 713C09, 713C11, 713C12, 713C15, 713C16, 713C23 and 713C28). In a large proportion of these, Al migrated in higher amount in acetic acid (e.g. C14, 713C02, 713C04, 713C06, 713C08, 713C11, 713C12, 713C16 and 713C28) than in citric acid. The release of Al in acetic acid 4% did not exhibit a clear trend and did not always decrease through consecutive exposures (unlike the release of Pb). Indeed, the release of Al increased from the first to the third migration in some samples (C14, 713C06, 713C11 and 713C16), it decreased for other samples (713C02, 713C04, 713C05, 713C09, 713C12, 713C15 and 713C28), or remained almost constant through the three exposures for other samples (713C01, 713C08 and 713C23). The lack of trend (to increase of decrease in different migration tests) was noted not only for the test in acetic acid but also for the test using citric acid. This could potentially be due to the formation of small surface cracks during the test allowing Al to be released not only from the glazed surface. For all the other types of ceramic articles it was easier to identify a common behaviour, the Al concentrations were higher in citric acid 0.5% (70°C, 2 h) and decreased along three consecutive migrations.

Li and Ti were released in higher amount from enamelled samples (113C01, 113C02 and 113C03). As expected the amount of metals depended on the colour of the enamel. The white enamel released greater amounts of Ti than the black one probably due to the TiO_2 used in white colour. The amount of Mn, Co, Ba, Cu leached from the black enamel was significantly higher than for the white sample. The type of test had an effect with citric acid 0.5% (70°C, 2 h) being more aggressive than acetic acid 4% (22°C, 24 h).

Mn, Fe and Ba were released in most of the samples but not in very high concentrations. Usually the release was higher in citric acid than in acetic acid and decreased through consecutives leaching. However, this behaviour was not always constant and could be sample dependent.

Co was mainly present in samples with a strong blue coloration such as samples 615C12, 615C13, 615C14, 615C15, 615C16, 615C17, 615C18, 615C19 and 615C20.

For Zn it was difficult to establish whether citric acid 0.5% (70°C, 2 h) was more aggressive than acetic acid 4% (22°C, 24 h). In both of them the release decreased from the first exposure to the third one.

4.2.2 Relationship between third and first migration

Previous work [22] referred to a potential relation between results of third migration and the first migration. Following this relation the third migration should be approximately 10% of the first one. Therefore, it was considered whether a release of for example 100 µg/L of Pb in the first migration would give less than 10 µg/L in the third migration hence complying with the proposed limit. However, this factor should be demonstrated and potentially extended to other metals. Data were compared and Table 8 shows the percentage of the third migration respect to the first one for Pb leached from 18 samples in both food simulants. These values were obtained by dividing the concentration of the Pb in solution from migration III by the concentration of Pb from migration I solution and presented in percent. The present study could not confirm this relationship of 10%. The relation between the third and first migration was not constant and most of the time was greater than 10%.

Table 8 - 3rd migration as % of 1st migration for Pb in ceramics tableware

Ceramic samples	Method AA 0.4% [%]	Method CA 0.5% [%]
713C01	12	48
713C02	20	34
713C09	16	55
413C18	6	26
713C16	16	26
413C15	23	66
413C17	18	29
713C20	12	21
713C22	26	48
413C04	16	23
913C02	15	20
413C26	35	9
413C27	27	21
713C28	12	15

As a conclusion of this section (4.2.1), results were not amenable to draw convergences, as level of migration often depended on the sample and the metal considered. In general the test with citric acid 0.5% (70°C, 2 h) seemed more severe than the one with acetic acid 4% (22°C, 24 h) probably also due to the temperature that may play a significant role in the migration process. Literature indicates that when temperatures are between 20°C and 40°C, metals leach via an ion-exchange which is controlled by inter-diffusion of cations such as Pb^{+2}, Co^{+2} and H_3O^+ through the structure of the ceramic article. However, for temperatures higher than 60°C, migration of metals could be affected by dissolution of the network of the ceramicware [3, 20, 23]. In addition, citric acid is a tricarboxylic acid so it can be more severe as it could form tridentate complexes with heavy metals in glaze and may lead to higher migrations levels [8].

4.2.3 Acetic acid 4% (22°C, 24 h) versus preconditioning test

The test with acetic acid 4% (22°C, 24 h) under repeated use regime implies an experimental time of three days of exposure plus the analysis. Options to reduce this time were thus explored as part of the project. One way of minimizing this time of analysis, without resorting to accelerated leaching at elevated temperatures, could be to pre-condition the ware to an equivalent state using acetic acid of a higher concentration, after which a normal, acetic acid 4% (22°C, 24 h) extraction is carried out. The aim of the pre-conditioning is to accelerate the leaching process and achieve values of migration similar to those of the third migration of the conventional method with acetic acid 4% (22°C, 24 h). The release of Cd and Pb and other metals were compared for both testing regimes: conventional acetic acid 4% (22°C, 24 h) under repeat use by 3 successive tests, and preconditioning acetic acid 10% (22°C, 5 h) + one conventional migration in acetic acid 4% (22°C, 24 h).

Table 9 presents the differences in percentage between the Pb and Cd concentrations in leachates obtained applying single migration test (acetic acid 4% (22°C, 24 h) after pre-treatment with acetic acid 10% (22°C, 5h) compared to the third migration of 3 consecutive tests with acetic acid 4% (22°C, 24 h). Concentrations in leachates following the pre-treatment step are also reported.

The differences in percentage were calculated as follow:

$$\frac{(5\text{ h AA10\%}+24\text{ h AA4\%})-\text{III AA4\% 24 h}}{\text{III AA4\% 24 h}} * 100\ [\%]$$

Table 9 – Difference in percentage between method with acetic acid 4% (III migration) vs method pre-conditioning for Pb and Cd

Sample	Pb				Cd			
	5h AA10% [µg/kg]	**5h AA10%+ 24 h AA4%**	**III AA4% 24 h [µg/kg]**	**Precond vs AA III [%]**	**5h AA10% [µg/kg]**	**5h AA10%+ 24 h AA4%**	**III AA4% 24 h [µg/kg]**	**Precond vs AA III [%]**
713C23	183.82	83.87	7.74	**+ 983**	0.01	0.02	<LOD	**-**
713C01	281.86	255.72	41.40	**+ 518**	0.35	1.62	0.53	**+ 449**
413C22	1952.63	996.34	221.01	**+ 351**	21.97	13.23	16.71	**+ 461**
413C26	103.91	142.31	33.64	**+ 323**	1.35	0.62	0.68	**+ 360**
913C05	8.97	1.05	0.32	**+ 232**	0.01	<LOD	<LOD	**-**
413C04	395.41	202.11	66.09	**+ 206**	1.17	0.30	0.60	**+ 969**
713C02	477.73	450.47	152.33	**+ 196**	0.04	0.27	0.10	**+ 486**
413C11	123.01	46.37	16.39	**+ 183**	0.13	0.03	0.18	**+ 273**
713C09	407.69	324.80	115.44	**+ 181**	0.13	0.37	0.43	**+ 48**
713C28	17296.68	6325.76	2332.85	**+ 171**	1.66	0.55	1.67	**+ 138**
713C20	434.93	167.93	63.46	**+ 165**	<LOD	<LOD	<LOD	**-**
413C17	3048.15	1856.27	739.74	**+ 151**	971.64	805.42	1092.63	**+ 291**
713C16	645.63	481.09	197.02	**+ 144**	<LOD	<LOD	0.025	**-**
413C27	117.58	90.74	38.37	**+ 137**	0.26	0.13	0.29	**+ 121**
213C02	1.41	0.38	0.18	**+ 114**	0.08	0.04	0.11	**+ 382**
413C15	1935.00	1514.15	732.63	**+ 107**	261.54	220.22	436.05	**+ 113**
913C02	51.69	18.44	11.49	**+ 60**	2.28	1.16	3.82	**+ 100**
713C22	15.76	6.52	4.75	**+ 37**	0.37	0.16	0.13	**+ 23**
413C13	1.92	3.43	2.65	**+ 30**	0.04	0.07	0.02	**+ 165**
413C21	491.44	568.61	1196.04	**- 52**	0.13	0.16	0.78	**-**

The release values for Pb and Cd obtained with a preconditioning test regime were almost always higher than those obtained with acetic acid 4% (22°C, 24 h) during the third migration. In 16 out of 20 samples the Pb release values following preconditioning were higher than 100% with respect to those one obtained after the third migration with acetic acid 4%, in 3 out of 20 the Pb concentration values were between 30% and 60% higher and only 1 sample gave lower Pb release following the preconditioning. For Cd, the differences (%) between concentrations in leachates obtained after preconditioning and values obtained in the third migration of consecutive test with acetic acid 4% (22°C, 24 h) were in all cases higher (more than 100% higher in 13 out of 15 samples).

The same behaviour could be observed for other metals such as Al, Li, Co, Zn, Fe and Mn. For metals such as Ba and Ti this trend was not confirmed (table 10).

Pre-conditioning of the articles with acetic acid 10% (5 h at 22°C) followed by a single migration in acetic acid 4% (22°C, 24 h) usually resulted in higher concentration values than those obtained in the third migration with acetic acid 4% (22°C, 24 h) (current migration test 84/500/EEC). Thus the aim to quickly achieve (by conditioning) a release level that would be similar to the leachate of combined first and second successive exposures was not successful. It should also be noted that the articles were all different in terms of the glaze and pigments applied and so what was found appropriate for one article may not be appropriate for all.

Table 10 – Difference (in %) between acetic acid 4% (22°C, 24 h) (III migration) vs pre-conditioning for Li, Al. Ti, Mn, Fe, Co, Zn and Ba

Sample code	Li [µg/kg]				Al [µg/kg]				Ti [µg/kg]				Mn [µg/kg]			
	5h AA10%	5h AA10% +24 h A4%	III AA4% 24 h	Precond. vs AA III [%]	5h AA10%	5h AA10% +24 h A4%	III AA4% 24 h	Precond. vs AA III [%]	5h AA10%	5h AA10% +24 h A4%	III AA4% 24 h	Precond. vs AA III [%]	5h AA10%	5h AA10% +24 h A4%	III AA4% 24 h	Precond. vs AA III [%]
213C02	0.12	0.06	<LOD	**-**	34.21	20.00	13.01	**+54**	0.71	0.34	0.15	**+122**	0.77	0.35	<LOD	**-**
413C04	1.02	0.75	0.27	**+178**	166.98	57.32	20.67	**+177**	1.42	0.55	0.62	**-11**	0.88	0.21	0.45	**-52**
413C11	0.38	0.18	0.09	**+109**	89.06	-	-	**-**	4.75	0.41	<LOD	**-**	28.46	13.11	4.94	**+165**
413C13	0.29	1.50	3.17	**-53**	15.60	144.51	131.94	**+10**	0.66	0.96	6.02	**-84**	<LOD	0.48	0.46	**6**
413C15	20.11	20.47	9.12	**+125**	266.03	283.34	120.14	**+136**	3.78	8.20	1.99	**+313**	0.70	3.96	0.15	**+2498**
413C17	19.43	13.17	4.55	**+189**	542.19	367.42	121.62	**+202**	16.33	21.71	7.31	**+197**	0.73	1.83	<LOD	**-**
413C21	1.01	1.83	2.81	**-35**	38.82	67.98	113.27	**-40**	0.15	3.50	<LOD	**-**	<LOD	0.49	<LOD	**-**
413C22	8.08	6.51	1.43	**+356**	52.72	41.07	2.82	**+1356**	0.87	3.73	<LOD	**-**	<LOD	1.04	<LOD	**-**
413C26	10.31	10.19	5.01	**+103**	548.23	582.27	189.37	**+207**	15.49	9.44	5.25	**+80**	0.53	0.48	0.22	**+116**
413C27	20.16	28.26	11.30	**+150**	701.12	1597.73	682.25	**+134**	6.47	2.20	1.34	**+64**	0.89	0.88	0.40	**+119**
713C01	0.46	11.55	<LOD	**-**	141.11	-	246.44	**-**	0.46	<LOD	<LOD	**-**	2.94	76.05	15.63	**+387**
713C02	4.83	43.15	<LOD	**-**	5475.11	-	10963.54	**-**	9.30	81.26	<LOD	**-**	74.63	714.60	265.44	**+169**
713C09	0.13	5.42	<LOD	**-**	63.52	-	1079.48	**-**	0.34	<LOD	<LOD	**-**	0.52	38.44	23.65	**+63**
713C16	0.38	7.74	<LOD	**-**	37.03	-	463.70	**-**	<LOD	<LOD	<LOD	**-**	0.15	5.93	2.30	**+158**
713C20	0.06	<LOD	<LOD	**-**	17.16	13.35	5.30	**+152**	<LOD	0.22	0.41	**-47**	<LOD	0.39	<LOD	**-**
713C22	0.07	<LOD	0.18	**-**	13.24	15.68	11.79	**+33**	<LOD	0.14	0.38	**-64**	<LOD	<LOD	0.38	**-**
713C23	0.28	3.58	<LOD	**-**	9.53	-	84.16	**-**	0.73	<LOD	<LOD	**-**	0.53	26.16	<LOD	**-**
713C28	11.46	26.55	25.52	**+4**	1129.03	2010.05	1053.81	**+91**	109.33	110.27	79.57	**+39**	7.75	72.38	58.38	**+24**
913C02	2.59	1.65	0.78	**+111**	63.07	36.18	32.68	**+11**	0.47	0.48	0.60	**-19**	<LOD	0.24	<LOD	**-**
913C05	3.54	1.44	0.31	**+359**	71.74	24.45	8.68	**+182**	0.26	0.13	<LOD	**<LOD**	<LOD	<LOD	<LOD	**-**

Sample code	Fe [µg/kg]				Co [µg/kg]				Zn [µg/kg]				Ba [µg/kg]			
	5h AA10%	5h AA10% +24 h A4%	III AA4% 24 h	Precond. vs AA III [%]	5h AA10%	5h AA10% +24 h A4%	III AA4% 24 h	Precond. vs AA III [%]	5h AA10%	5h AA10% +24 h A4%	III AA4% 24 h	Precond. vs AA III [%]	5h AA10%	5h AA10% +24 h A4%	III AA4% 24 h	Precond. vs AA III [%]
213C02	15.65	<LOD	<LOD	**-**	0.56	0.27	0.09	**+203**	<LOD	<LOD	<LOD	**-**	0.63	0.34	0.14	**+133**
413C04	5.27	2.73	0.33	**+730**	1.71	0.72	0.16	**+359**	29.16	-	-	**-**	3.33	3.38	1.13	**+199**
413C11	22.24	12.91	1.16	**+1014**	81.20	51.35	28.18	**+82**	12.89	<LOD	<LOD	**-**	2.13	2.56	0.67	**+285**
413C13	9.47	10.33	9.27	**+11**	<LOD	<LOD	<LOD	**-**	<LOD	-	-	**-**	0.37	2.85	2.58	**+10**
413C15	21.39	20.15	13.35	**+51**	4.64	4.86	<LOD	**-**	113.58	96.36	-	**-**	8.90	5.32	<LOD	**-**
413C17	16.68	25.47	4.85	**+426**	0.19	0.18	<LOD	**-**	144.29	97.72	-	**-**	945.13	573.43	110.45	**+419**
413C21	28.01	6.20	5.82	**+7**	<LOD	<LOD	<LOD	**-**	<LOD	10.01	<LOD	**-**	1.37	2.48	<LOD	**-**
413C22	15.03	6.57	24.17	**-73**	<LOD	<LOD	<LOD	**-**	97.22	66.09	<LOD	**-**	2.52	1.50	<LOD	**-**
413C26	42.58	16.03	5.20	**+208**	5.33	5.67	8.29	**-32**	46.22	55.93	19.01	**+194**	92.81	100.19	60.22	**+66**
413C27	28.14	21.01	7.72	**+172**	0.89	0.32	0.13	**+143**	40.85	63.97	35.81	**+79**	69.38	151.35	92.80	**+63**
713C01	56.38	502.69	<LOD	**-**	0.51	8.68	<LOD	**-**	<LOD	57.69	<LOD	**-**	1.14	12.30	13.63	**-10**
713C02	1323.12	8708.74	<LOD	**-**	1.70	13.35	<LOD	**-**	25.99	125.05	40.10	**+212**	7.30	50.51	55.20	**-8**
713C09	23.13	475.58	96.34	**+394**	0.33	1.32	0.62	**+113**	<LOD	<LOD	<LOD	**-**	1.34	6.10	8.60	**-29**
713C16	14.76	138.08	31.64	**+336**	0.12	4.37	<LOD	**-**	146.19	194.97	5.89	**+3211**	9.57	13.51	13.14	**+3**
713C20	5.42	<LOD	<LOD	**-**	6.46	22.13	21.94	**+1**	<LOD	-	-	**-**	1.02	0.96	0.70	**+37**
713C22	<LOD	<LOD	1.63	**-**	<LOD	0.42	0.21	**+97**	<LOD	-	-	**-**	0.28	0.25	0.64	**-61**
713C23	12.33	81.27	2.79	**+2808**	<LOD	0.22	0.02	**+1194**	<LOD	<LOD	<LOD	**-**	0.43	2.44	9.50	**-74**
713C28	353.32	266.58	106.54	**+150**	1.76	4.87	2.32	**+110**	1263.80	-	204.29	**-**	189.84	97.79	36.89	**+165**
913C02	<LOD	<LOD	<LOD	**-**	10.28	0.71	0.17	**+316**	24.40	-	-	**-**	23.51	15.45	2.34	**+561**
913C05	26.71	<LOD	<LOD	**-**	1.18	0.37	0.10	**+283**	27.17	<LOD	<LOD	**-**	51.56	62.60	67.78	**-8**

4.2.4 Acidified tomato sauce versus non-acidified tomato

In addition to food simulants a food was used as benchmark for comparison, to describe the release of metals from ceramic. Tomato sauce was proposed as a worst case acidic foodstuff for ceramic articles. Food composition data tables indicate that tomato sauce products can be as low as pH 3.5. The determination of the pH for about 50 different tomato sauce products showed all values of pH around 4.2.

A comparison between acidified tomato sauce (pH 3.5) and non-acidified tomato sauce (pH 4.2) was conducted with a focus on the release of Cd and Pb, since those are the metals under priority consideration. Highly releasing articles were chosen as test materials to perform this comparison test (613G03, C6 and 713C02). The release of Pb and Cd in acidified and non-acidified tomato sauce is shown in figure 5. The amount of Pb migrated into acidified tomato sauce was higher than the amount migrated into the non-acidified matrix. The same was observed also for Cd released from sample 613G03.

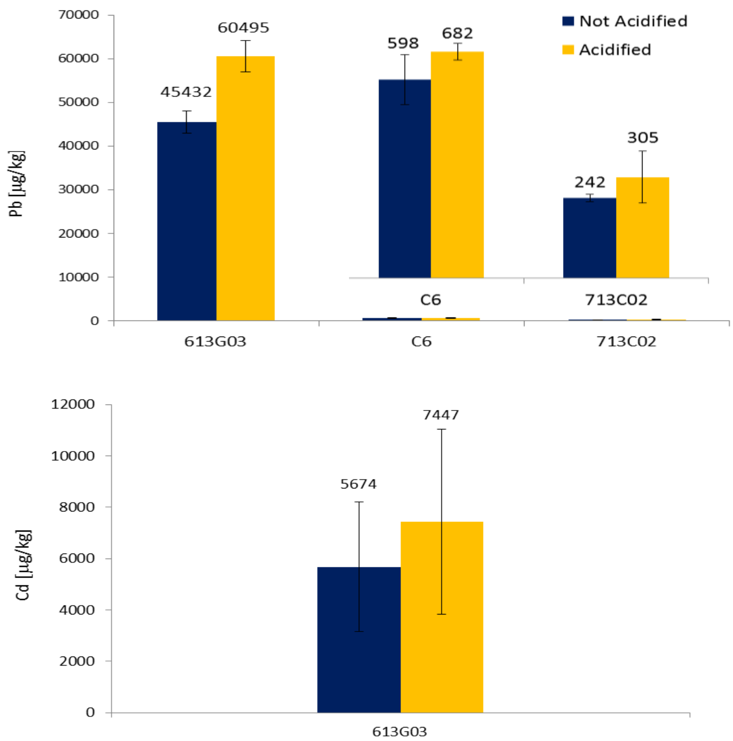


Figure 5 – Difference in Pb and Cd release between regular vs. acidified tomato sauce

The results from the first migration were sufficient to identify that the level of acidity induced a difference in migration. Therefore the decision was to reduce of the pH of a commercial source of tomato by addition of citric acid for all experiments using tomato as benchmark food. The reasoning was that it could not be demonstrated that a tomato sauce would never be as low as 3.5, and thus the lowest pH from the composition tables had to be taken as reference for worst case.

4.2.5 Food simulants versus acidified tomato sauce

Three consecutive migrations in acidified tomato sauce were performed (70°C, 2 h). The Pb release was compared with results obtained using acetic acid 4% (22°C, 24 h) and citric acid 0.5% (70°C, 2 h). Highly releasing samples were chosen as test materials to perform this comparison test (C14, 713C01, 713C16, 713C09, 413C18 and 613C04). Figure 6 shows the results of this comparison.

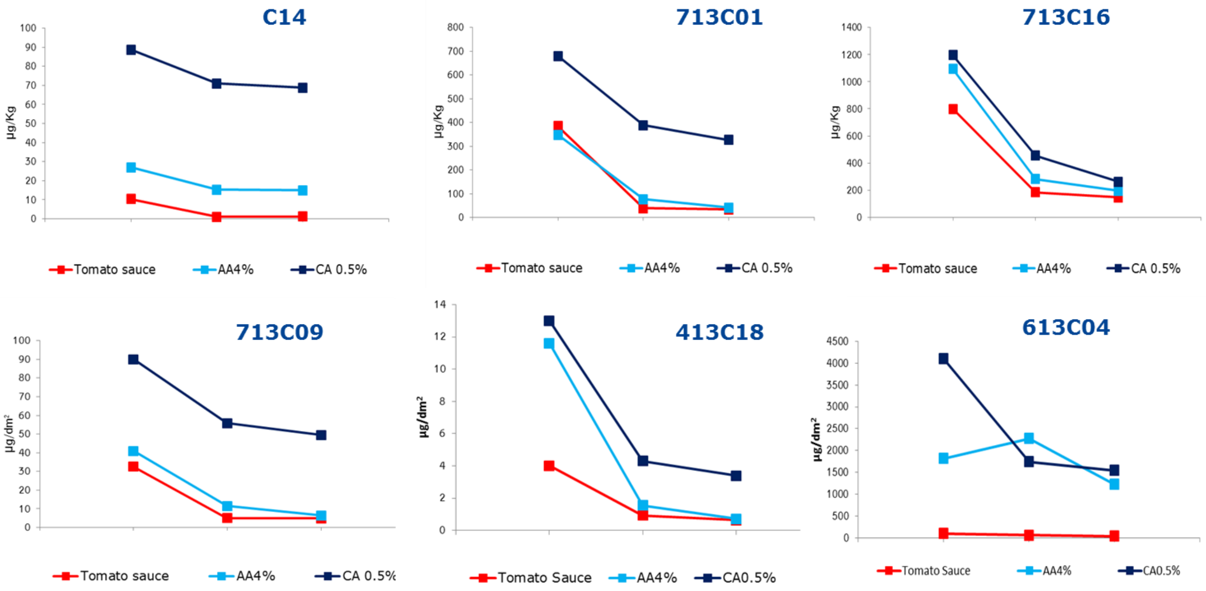


Figure 6 - Migration of Pb from hollowware into acetic acid 4% (22°C, 24 h), citric acid 0.5% (70°C, 2 h) and acidified tomato sauce

Results of testing with citric acid 0.5% (70°C, 2 h) always gave higher release than with acetic acid 4% (22°C, 24 h). It is important to point out that the migration profile in tomato sauce was more similar to that in acetic acid 4% especially in the third release. The amount of Pb released in the food simulants was, for the samples tested, always higher than the amount released into food (tomato sauce), ensuring that the migration into food simulants covered the worst case scenario and gave a safe margin.

4.3 Effects of different time gaps between three consecutive exposures

Some studies had suggested that leaving overglaze decorated ceramic articles overnight caused higher release of Pb [19]. The effects observed could have been attributable to the action of atmospheric carbon dioxide and water vapour on the overglaze decoration. Glass and glazes absorb water from the atmosphere to produce a film which can be removed only by heating. Such a film, which must actually be a solution of carbonic acid and other elements, can attack overglaze enamels, resulting in the breakdown and possible ultimate disappearance of portions of the decoration [19]. These observations suggest that several hours of contact between a 'dry dish' and the atmosphere may be long enough for chemical reactions to occur between the absorbed acidic film and basic compounds in the decoration, whereas the few minutes of air-glaze contact between leachings on a single day is not enough to facilitate release of metals from decorations.

One implication from this literature could therefore be that compliance tests using repeat use regime may be impacted if not done consecutively for all three. This in turn would imply a mandatory start only on Mondays or Tuesdays to avoid the occurrence of a week-end between migrations. As this would be a strong impediment to productivity for control or compliance laboratories, a dedicated study was conducted to assess the effect of a time gap between migrations, both between the first and second and more importantly between the second and third migration.

Multiple migrations with time gaps between migrations were investigated to evaluate the overall effect of lag phase on the final result of the compliance test. Releases under repeated use regime were carried out with a weekend gap of 72 h between first and second, or second and third migration tests. Alternative treatments were also tested using a fill of standard tap water (ISO 10531:2011-06) during the time gaps [24]. All results were compared using t-Student test.

The experimental plan (table 11) included three types of tests: (1) consecutive migration tests; (2) migration tests with a weekend gap between the first and second migrations and (3) migration tests with a weekend gap between the second and third migrations. One set of half the ceramic test samples were filled with standard tap water during the time gap and the other set of half the samples were left empty during the time gap (see Table 11). Four sets of different types of ceramic articles 713C21, 713C23, 713C30 and 413C11 were chosen as most test materials for this study. All migrations were performed in acetic acid 4% (22°C, 24 h). For the consecutive migration tests, the same migration procedure was immediately (less than 1h) repeated for two more times.

For migrations with a time gap between the first and second migrations, after the first migration tests, 4 items for each ceramic set were filled with the standard tap water and left in incubator at 22 ± 2°C and other 4 items of the same set were left empty (cleaned and dried) at room temperature (in contact with the atmosphere). Following the "week-end" gap the consecutive second and third migrations were performed.

For migrations with a weekend gap between the second and third migrations, two consecutive migration tests were performed and after the second migrations. Half of the samples (4 for each kind of ceramics) were filled with the standard tap water and left in an incubator at 22 ± 2°C and the rest of the samples were left empty (cleaned and dried) at room temperature. After the weekend the final migration tests were conducted.

Table 11 – Week end gap test experimental plan

TEST	MONDAY	TUESDAY	WEDNESDAY	THURSDAY	FRIDAY	W-END	MONDAY	TUESDAY	WEDNESDAY
Consecutive		4 Mortars 4 Bowls 4 Cups → 4 Plates **1st Migration**	4 Mortars 4 Bowls 4 Cups → 4 Plates **2nd Migration**	4 Mortars 4 Bowls 4 Cups → 4 Plates **3rd Migration**	End				
weekend 2nd- 3rd (H_2O)			4 Mortars 4 Bowls 4 Cups → 4 Plates **1st Migration**	4 Mortars 4 Bowls 4 Cups → 4 Plates **2nd Migration**	Rinse Dry +H_2O	→	4 Mortars 4 Bowls 4 Cups → 4 Plates **3rd Migration**	End	
weekend 2nd- 3rd (no H_2O)			4 Mortars 4 Bowls 4 Cups → 4 Plates **1st Migration**	4 Mortars 4 Bowls 4 Cups → 4 Plates **2nd Migration**	Rinse Dry	→	4 Mortars 4 Bowls 4 Cups → 4 Plates **3rd Migration**	End	
weekend 1st-2nd (H_2O)				4 Mortars 4 Bowls 4 Cups → 4 Plates **1st Migration**	Rinse Dry +H_2O	→	4 Mortars 4 Bowls 4 Cups → 4 Plates **2nd Migration**	4 Mortars 4 Bowls 4 Cups → 4 Plates **3rd Migration**	End
weekend 1st-2nd (no H_2O)				4 Mortars 4 Bowls 4 Cups → 4 Plates **1st Migration**	Rinse Dry	→	4 Mortars 4 Bowls 4 Cups → 4 Plates **2nd Migration**	4 Mortars 4 Bowls 4 Cups → 4 Plates **3rd Migration**	End

For all types of ceramic samples used for this comparison and exposed to acidic media, the majority of Pb was released during the first migration tests and decreased considerably with repeated contact [25-26]. This was not the case for all other elements as it strongly depended on the type of ceramic article and on the metals measured.

Results showed that for all four ceramic articles undergoing the study, the Pb concentrations obtained after consecutive migration tests and after tests left empty over a weekend gap were not significantly different (applying the Student t –test). If the samples were left filled with standard tap water over the weekend between the second and the third migrations, slightly higher releases of Pb for samples 713C23 and 713C30 were observed. When the weekend gap was between the first and the second migrations a slight increase of Pb release in the third exposure was observed for the samples 713C21 and 713C30.

There was no effect for time gap with or without filling with respect to the release of Cd. For other metals it was more difficult to identify common trends. Heavy metals concentrations obtained after third migration tests for the four ceramic articles are reported in table 12.

Considering all results obtained for the ceramic articles used in this comparison, no significant difference was observed between a protocol with successive migrations and one with a lag phase when the articles were left empty in contact with the ambient air.

Table 12 – Concentrations obtained after 3rd migration tests for four ceramic articles

	Test		Pb	Cd	Ba	Co	Mn	Al	Li	Ti	Zn	As	Ni
Mortar	Consecutive	Av [µg/L]	1.10	0.03	0.59	<LOD	<LOD	0.71	<LOD	<LOD	<LOD	0.20	<LOD
		RSD [%]	39.3	10.1	29.9			5.9				20.4	
	weekend 1st-2nd	Av [µg/L]	2.32	<LOD	3.96	<LOD	<LOD	80.43	0.26	0.15	13.08	0.22	<LOD
	(H_2O)	RSD [%]	20.4		151.5			179.4	101.4	-	-	12.7	
	weekend 1st-2nd	Av [µg/L]	2.85	<LOD	1.23	<LOD	0.20	14.37	<LOD	<LOD	0.39	0.23	<LOD
	(no H_2O)	RSD [%]	71.9		27.1		75.5	71.2				10.6	
	weekend 2nd-3rd	Av [µg/L]	3.27	<LOD	1.03	<LOD	<LOD	18.15	0.38	<LOD	<LOD	0.18	<LOD
	(H_2O)	RSD [%]	98.0		35.6			78.7	-			1.9	
	weekend 2nd-3rd	Av [µg/L]	3.35	<LOD	0.84	<LOD	<LOD	8.20	<LOD	<LOD	<LOD	0.20	<LOD
	(no H_2O)	RSD [%]	72.4		42.4			85.5				6.2	
Bowl	Consecutive	Av [µg/L]	42.79	0.07	4.19	0.85	59.92	1065.79	11.68	1.11	13.87	8.49	<LOD
		RSD [%]	56.4	27.0	86.6	111.3	89.7	119.8	92.4	127.0	43.6	106.2	
	weekend 1st-2nd	Av [µg/L]	47.22	0.02	3.60	0.60	41.42	894.35	12.52	1.06	15.74	3.87	<LOD
	(H_2O)	RSD [%]	68.2	65.3	89.2	145.9	63.4	164.7	99.7	-	66.1	57.9	
	weekend 1st-2nd	Av [µg/L]	46.27	0.02	4.29	0.75	48.70	1013.78	14.02	0.41	13.48	3.18	<LOD
	(no H_2O)	RSD [%]	41.9	69.2	46.2	93.0	52.3	90.6	73.5	135.9	58.2	56.1	
	weekend 2nd-3rd	Av [µg/L]	79.69	<LOD	1.91	0.25	22.12	168.46	7.03	0.13	11.88	1.39	<LOD
	(H_2O)	RSD [%]	17.3		18.5	20.8	37.2	31.6	9.3	114.5	12.3	31.5	
	weekend 2nd-3rd	Av [µg/L]	54.32	0.03	4.63	0.47	37.53	751.53	8.40	0.56	12.42	14.92	<LOD
	(no H_2O)	RSD [%]	62.1	48.2	31.9	58.8	22.7	76.3	39.6	-	19.5	135.4	
Cup	Consecutive	Av [µg/L]	76.38	0.25	6.94	1.02	62.42	906.18	8.94	0.90	<LOD	1.20	2.26
		RSD [%]	17.4	36.6	15.3	8.3	9.1	13.0	10.5	7.0		25.3	23.7
	weekend 1st-2nd	Av [µg/L]	129.70	0.15	11.13	1.04	40.48	1279.01	9.27	1.01	<LOD	1.00	1.92
	(H_2O)	RSD [%]	16.4	113.2	1.6	44.2	63.3	53.8	23.5	26.1		70.5	32.3
	weekend 1st-2nd	Av [µg/L]	65.40	0.29	9.97	1.54	67.52	2091.65	15.54	1.75	10.04	1.86	3.09
	(no H_2O)	RSD [%]	24.9	113.9	19.4	30.5	33.1	36.6	31.4	42.8	47.1	36.9	32.0
	weekend 2nd-3rd	Av [µg/L]	107.10	0.18	7.15	0.46	25.24	355.04	6.12	0.51	<LOD	0.60	1.05
	(H_2O)	RSD [%]	14.1	35.9	10.1	32.1	33.4	16.0	9.9	9.9		15.5	24.9
	weekend 2nd-3rd	Av [µg/L]	88.24	0.26	11.33	1.95	100.6	2605.69	14.51	1.52	12.13	5.23	3.31
	(no H_2O)	RSD [%]	21.7	99.8	16.4	24.9	34.0	26.6	11.3	36.1	23.7	36.9	15.3
Plate	Consecutive	Av [µg/dm2]	0.713	<LOD	0.037	0.694	0.233	0.867	<LOD	0.120	<LOD	0.002	0.061
		RSD [%]	47.8		72.8	29.5	57.8	69.2		25.1		76.9	116.2
	weekend 1st-2nd	Av [µg/dm2]	0.471	<LOD	0.043	0.675	0.169	0.545	<LOD	0.089	<LOD	0.001	0.127
	(H_2O)	RSD [%]	22.1		46.0	27.9	18.0	55.6		14.0		115.0	42.6
	weekend 1st-2nd	Av [µg/dm2]	0.412	<LOD	0.025	0.689	0.121	0.317	<LOD	0.060	<LOD	0.000	0.044
	(no H_2O)	RSD [%]	11.8		48.5	31.8	6.5	29.3		3.9		59.5	136.0
	weekend 2nd-3rd	Av [µg/dm2]	0.592	<LOD	0.036	1.063	0.221	1.107	<LOD	0.190	<LOD	0.038	0.011
	(H_2O)	RSD [%]	20.6		20.9	17.9	29.5	19.9		8.9		195.4	66.2
	weekend 2nd-3rd	A [µg/dm2]	0.443	<LOD	0.026	0.649	0.132	0.532	<LOD	0.068	<LOD	0.001	0.037
	(no H_2O)	RSD [%]	8.6		13.6	27.5	2.6	8.4		5.0		72.7	108.1

4.3 Storage effect

The scope of this experiment was to evaluate the effect of storage of tableware on release metals from ceramic ware. Studies had indicated that storage of crystal glass articles led to increased release closer to those seen in a first migration test [27]. Thus, experiments were also conducted to verify the level of release for a set of tests performed again after a long lag phase. These experiments could also give an indication whether a test regime with three consecutive migrations would still be adequate for articles that are used only a few times a year.

Nine different types of luxury or niche ceramic articles (615C12, 615C13, 615C14, 615C15, 615C16, 615C17, 615C18, 615C19 and 615C20) were used in this study. Leachates from ceramic articles were obtained applying the migration test with acetic acid 4% (22°C, 24 h), under repeated use regime (3 times). Once the third migration was completed the samples were rinsed, dried and stored for defined time periods. The samples were stored first for 6 months, tested for repeated use migration, stored for month and retested, and finally stored for one week and retested (figure 7).

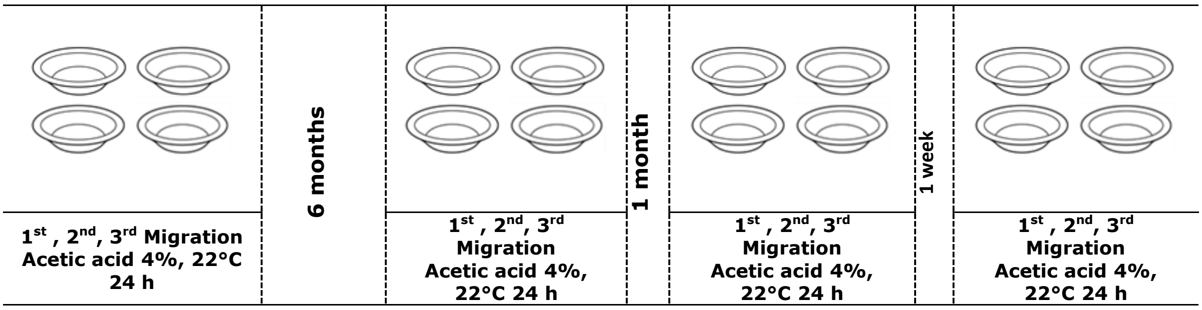


Figure 7 – Storage effect study scheme

The results obtained for the ceramics articles tested (table 13 and annex 5) suggested that the majority of Pb, Co, Al and Zn was released after the first migration and the concentrations of the successive leachates even after 6 months of storage were always lower than the first test (unlike crystal).

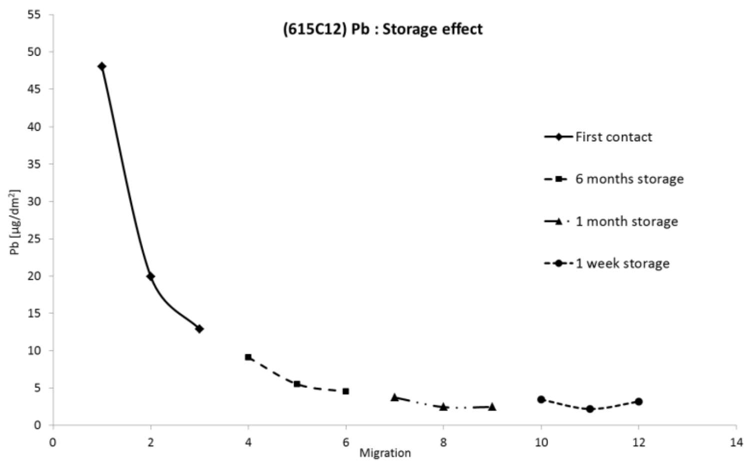
The nature and type of the sample had an important effect on the leaching process.

The storage time did not affect the Pb, Co, Al and Zn release for samples 615C12, 615C13, 615C14 and 614C15, and their concentrations in leachates decreased continuously when the test was repeated after 6 months of storage of the article. Figure 8a presents an example of the profile obtained for Pb, where it was observed that after four exposures with acetic acid the release achieved constant levels. In samples 615C16, 615C17, 615C18, 615C19 and 615C20 the Al, Co, Pb and Zn concentration values in the first migration after 6 months of storage were slightly higher than those of the third migration of the previous test, but they were still far lower from those of the initial exposure (figure 8b). Also for these samples a constant level was reached after the fourth migration. For Cd (present only in samples 615C12 and 615C13), the concentration values decreased continuously across all exposures.

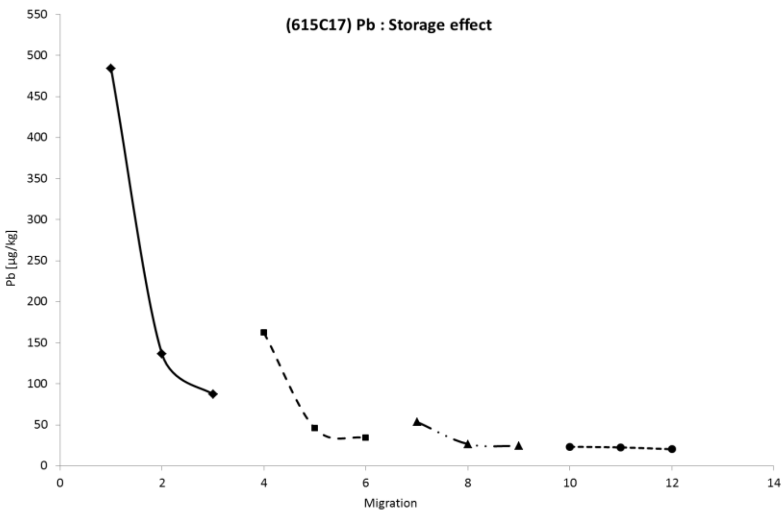
Table 13 – Results of storage effect for Pb

Samples			Pb [µg/kg]	Storage time	Pb [µg/kg]	Storage time	Pb [µg/kg]	Storage time	Pb [µg/kg]
615C13	I	Av	216.58		36.42		26.30		17.89
		RSD [%]	29.41		12.21		25.74		22.58
	II	Av	80.45		16.01		14.99		15.16
		RSD [%]	48.23		2.64		21.40		20.31
	III	Av	78.02		14.11		11.88		15.05
		RSD [%]	39.00		3.62		24.29		22.10
615C15	I	Av	230.90		28.67		18.41		12.67
		RSD [%]	18.61		28.06		24.69		33.24
	II	Av	41.39		8.27		7.21		12.93
		RSD [%]	26.56		4.90		28.61		36.93
	III	Av	39.28		6.85		7.07		11.14
		RSD [%]	53.82		29.80		32.14		37.33
615C17	I	Av	483.86		162.16		53.66		23.45
		RSD [%]	11.54		30.48		41.55		23.88
	II	Av	136.36	6 months	45.78	1 month	26.74	1 week	22.72
		RSD [%]	16.16		20.65		32.72		23.48
	III	Av	87.19		34.11		24.36		20.46
		RSD [%]	13.90		13.84		27.74		30.21
615C18	I	Av	317.86		81.03		38.48		16.38
		RSD [%]	7.80		18.23		13.61		12.84
	II	Av	87.41		33.10		21.47		14.80
		RSD [%]	11.03		17.07		9.42		8.32
	III	Av	55.04		23.83		16.58		14.25
		RSD [%]	7.99		12.04		15.51		15.63
615C19	I	Av	707.63		297.72		168.51		87.08
		RSD [%]	9.44		10.67		10.14		15.55
	II	Av	239.36		121.71		94.00		87.27
		RSD [%]	11.14		7.97		10.88		16.68
	III	Av	195.15		97.58		86.69		82.79
		RSD [%]	23.87		2.79		11.69		12.29

Samples			Pb [µg/dm²]	Storage time	Pb [µg/ dm²]	Storage time	Pb [µg/ dm²]	Storage time	Pb [µg/ dm²]
615C12	I	Av	48.08	6 months	9.09	1 month	3.73	1 week	3.44
		RSD [%]	23.52		11.22		23.07		29.46
	II	Av	19.94		5.51		2.44		2.19
		RSD [%]	32.21		34.30		41.33		11.11
	III	Av	12.88		4.51		2.47		3.19
		RSD [%]	35.34		23.21		36.27		24.52
615C14	I	Av	80.84		9.09		6.15		4.10
		RSD [%]	28.97		37.67		36.32		47.83
	II	Av	16.70		2.72		3.02		2.68
		RSD [%]	27.09		25.39		63.28		49.08
	III	Av	11.14		2.12		1.70		2.27
		RSD [%]	51.66		40.17		36.75		56.56
615C16	I	Av	188.79		60.18		17.73		12.79
		RSD [%]	19.96		75.34		68.29		22.90
	II	Av	61.53		24.39		11.72		10.80
		RSD [%]	20.27		20.79		21.98		16.53
	III	Av	39.22		16.84		9.54		9.16
		RSD [%]	20.01		15.31		21.27		25.25
615C20	I	Av	135.48		53.75		22.61		15.47
		RSD [%]	10.52		14.37		14.82		18.55
	II	Av	50.65		19.52		14.73		14.54
		RSD [%]	16.95		6.84		14.84		23.06
	III	Av	39.63		17.28		13.33		12.85
		RSD [%]	4.45		8.31		9.77		15.23



a)



b)

Figure 8 – Pb migrations after different storage time for sample a) 615C12 and b) 615C17

4.4 Cookware – preliminary studies

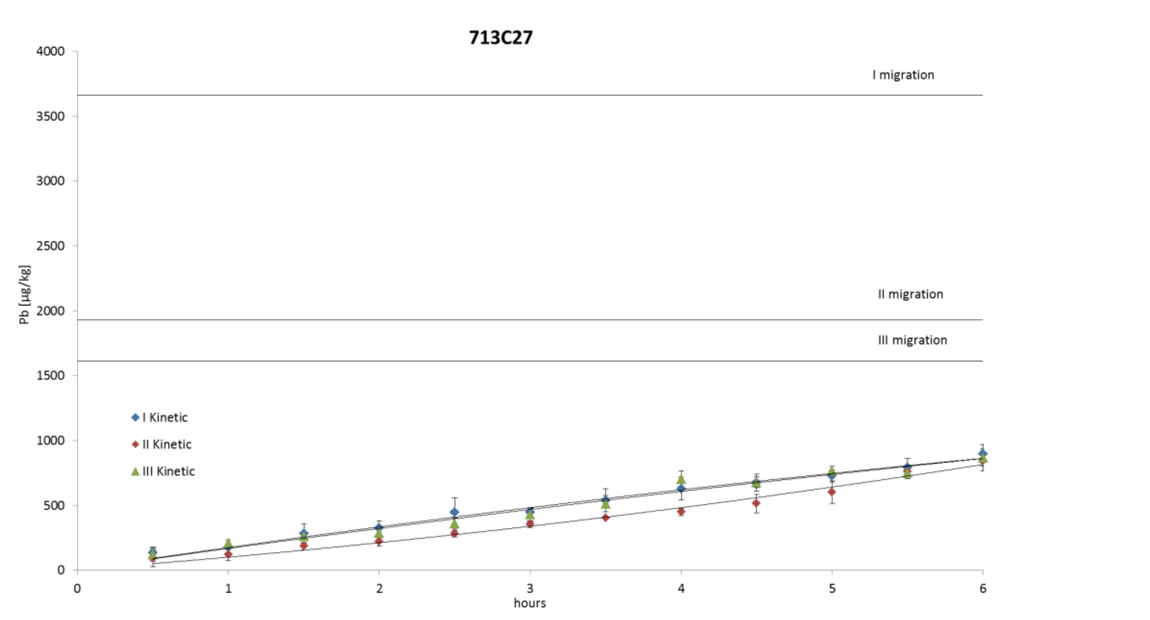
Cookware was also investigated under the scope of these studies. These articles are referred to as articles of category 3[a] as opposed to category 1[a] (flatware) and 2[a] (hollowware). There is a particular concern for cookware because the normal conditions of use (heating acid foods for prolonged periods) are conducive to higher release of the metals into food.

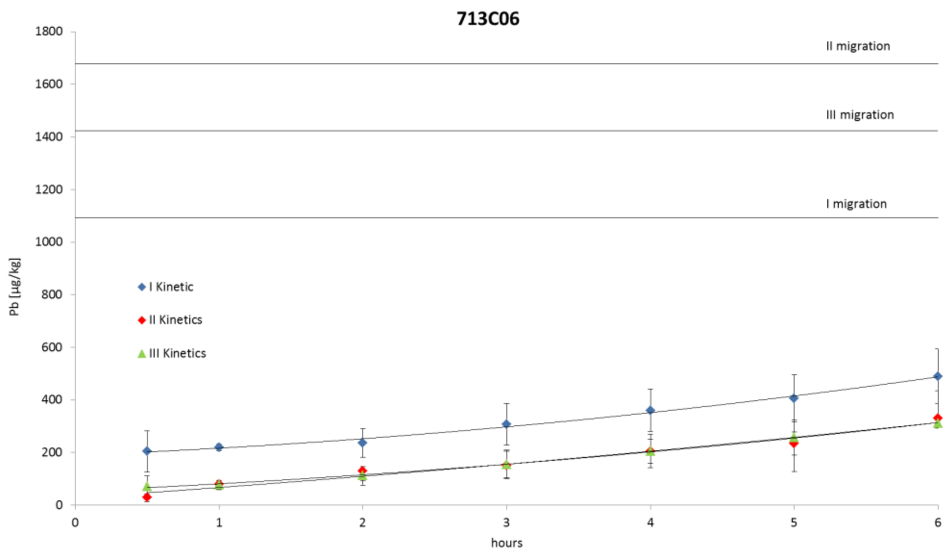
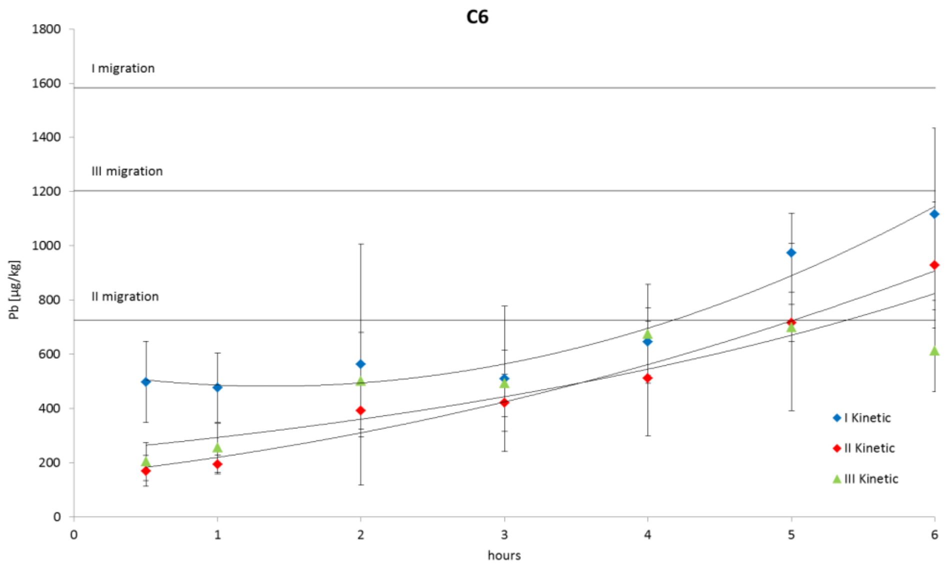
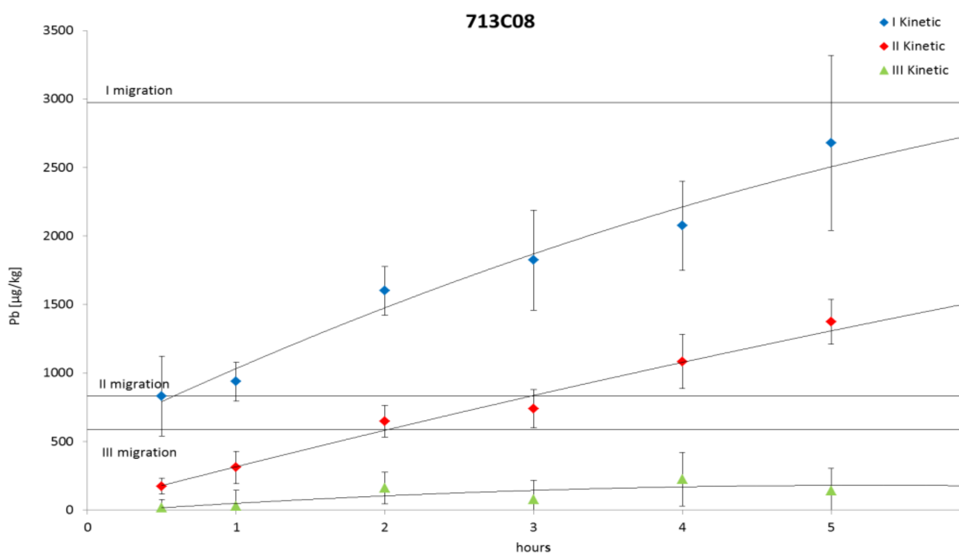
Cookware was tested with acetic acid 4% (22°C, 24 h) as laid down in Directive 84/500 EEC. However, some standards exist that imply the use of temperature. One of them, ISO Standard 8391-1 (1986) for release of Pb and Cd from ceramic cookware in contact with food, provides test conditions where hot acetic acid solution of 4% is used for 2 h [28]. Due to the presence of acetic acid fumes coming from the boiling simulant, these types of test are not easy to manage in the laboratory and pose safety concerns when applied routinely. Thus, the conventional test using room temperature was chosen to perform preliminary studies.

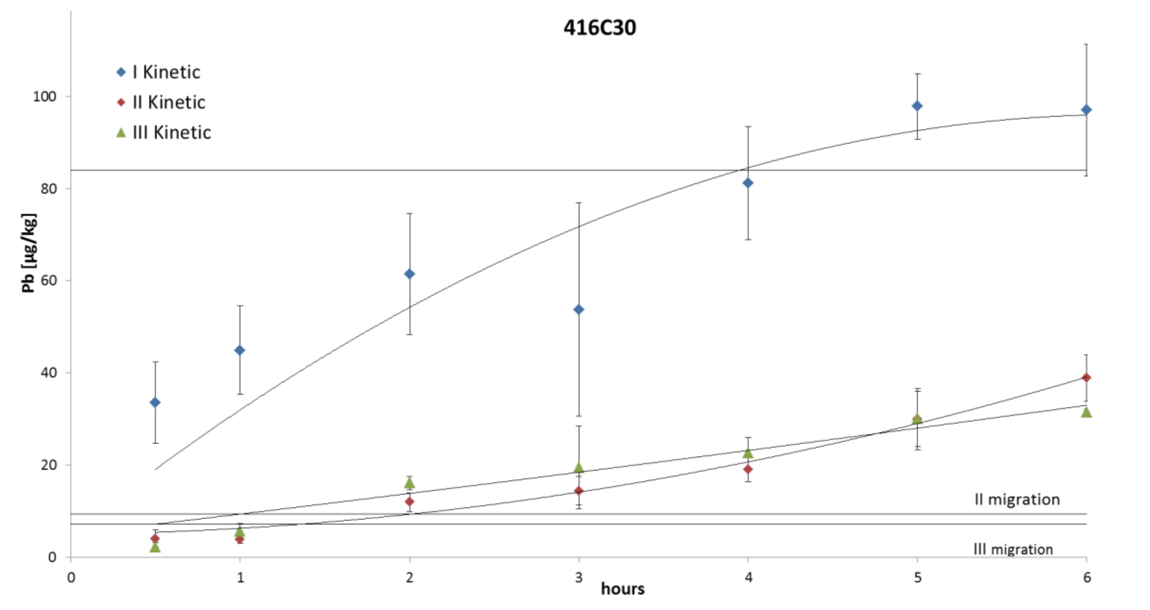
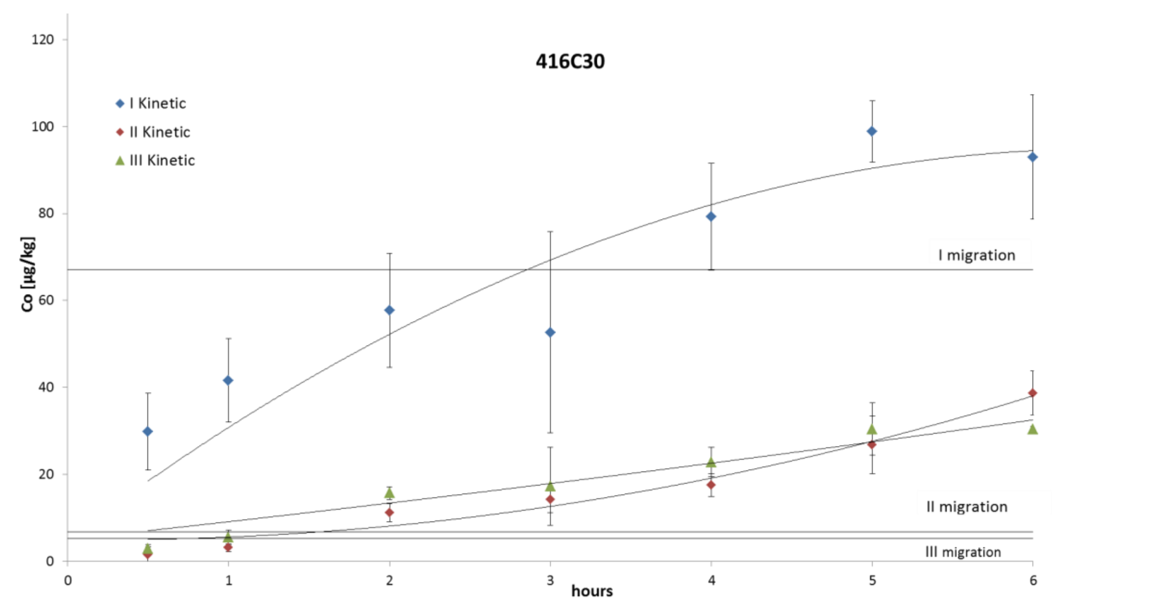
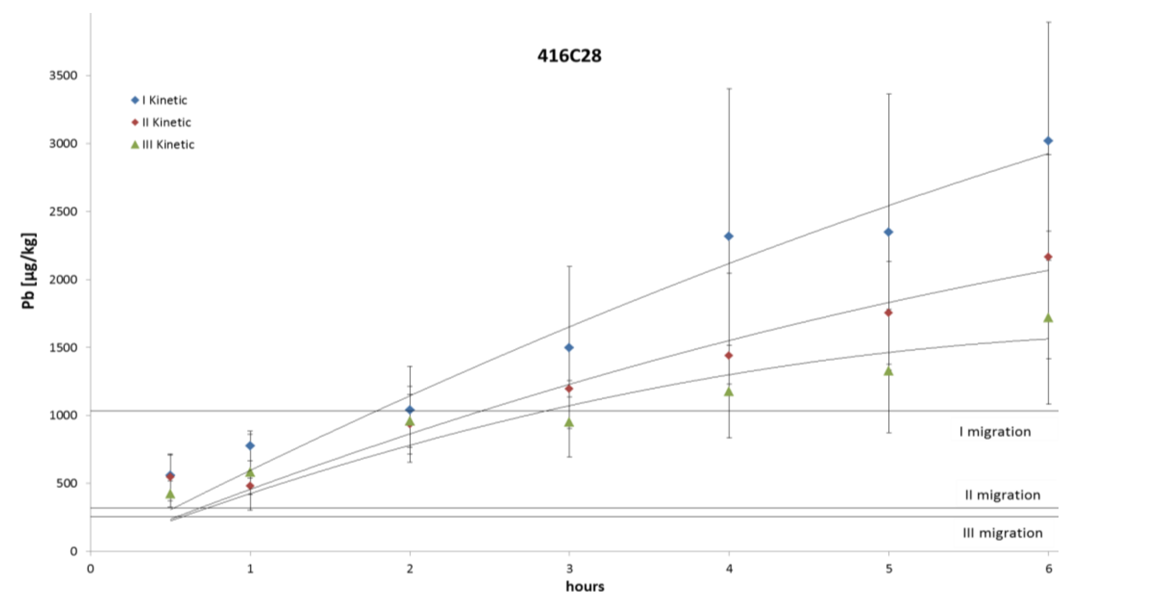
Twenty one samples of cookware were previously exposed three consecutive times to acetic acid 4% (22°C, 24 h) and release of the metals was determined. Six samples exhibiting the highest Pb release were than chosen for kinetic studies using hot tomato sauce. The kinetic studies into tomato sauce were performed boiling tomato sauce (pH 3.5) for ca. 6 hours, and analysing Pb, Cd and Co release at several time points. Three consecutive kinetics of 6 hours were performed over three days (repeat use scenario).

The Pb concentration values were plotted as graphs on figure 9a-h. For two samples the concentration of both Cd and Co was plotted. Results of conventional and repeated tests with acetic acid 4% (22°C, 24 h) were also shown.

a



b	 713C06 Pb [µg/kg] hours II migration III migration I migration I Kinetic II Kinetics III Kinetics 0 200 400 600 800 1000 1200 1400 1600 1800 0 1 2 3 4 5 6
c	 C6 Pb [µg/kg] hours I migration III migration II migration I Kinetic II Kinetic III Kinetic 0 200 400 600 800 1000 1200 1400 1600 1800 0 1 2 3 4 5 6
d	 713C08 Pb [µg/kg] hours I migration II migration III migration I Kinetic II Kinetic III Kinetic 0 500 1000 1500 2000 2500 3000 3500 0 1 2 3 4 5 6

e	
f	
g	

416C30
I Kinetic
II Kinetic
III Kinetic
Pb [µg/kg]
hours
II migration
III migration

416C30
I Kinetic
II Kinetic
III Kinetic
Co [µg/kg]
hours
I migration
II migration
III migration

416C28
I Kinetic
II Kinetic
III Kinetic
Pb [µg/kg]
hours
I migration
II migration
III migration

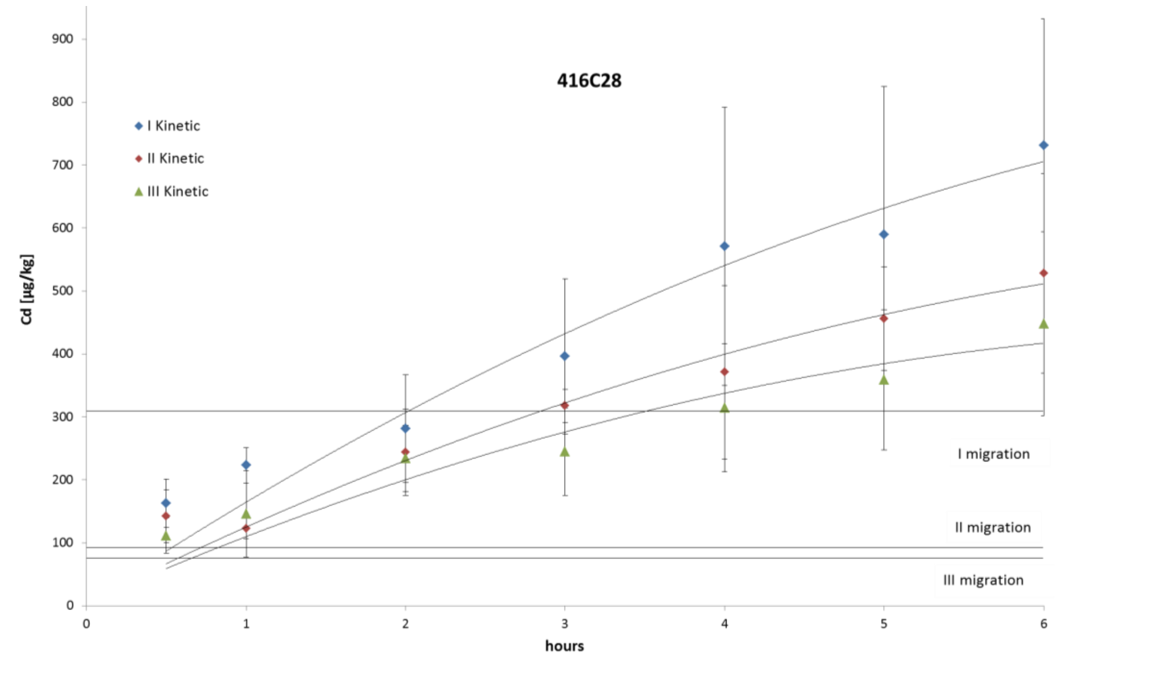
h	

Figure 9 – Pb, Cd and Co release during kinetics in boiling tomato vs migrations in acetic acid 4% 24 h at 22°C for samples a) 713C27, b) 713C06, c) C6, d) 713C08, e-f) 416C28 and g-h)416C30

Samples 713C27 and 713C06 had similar plots. Repeated exposures (Acetic acid 4%, 22°C, 24 h) gave much higher Pb release values compared to those obtained with the test conditions using boiling tomato sauce. The Pb concentrations increased over the 6 hours and the release profile remained constant in all three consecutive kinetics for sample 713C27. For sample 713C06, the first kinetic into tomato was slightly higher compared to the second and the third one.

For other three samples C6, 713C08 and 416C30,

- The Pb release in the first migration in acetic acid 4% (22°C, 24 h) was comparable to those obtained after 6 hours with boiling tomato sauce considering the standard deviations.

- The Pb release in the second and third migrations in acetic acid was often lower compared to values obtained in the first kinetic with boiling tomato sauce.

- The Pb release the third migration in acetic acid was higher than the third kinetic into boiling tomato sauce (apart for sample 416C30). Release of Co behaved similarly to that of Pb.

- The Pb and Cd release from sample 416C28 was lower in acetic acid than in boiling tomato sauce

For 5 out of 6 samples, the Pb release in the first migration with acetic acid was always higher or equal than those obtained after 6 hours with boiling tomato sauce. Furthermore, it always (for all 6 samples) covers 2 h in boiling tomato sauce.

As the set of samples received was quite limited, further work could be carried out on a larger set of samples to evaluate the adequateness of the release test in acetic acid 4% without use of temperature under repeated use regime. It would require the collection of appropriate ceramic articles containing other metals beyond Pb.

5. Conclusions

The overall aim of this study was to investigate the release of metals from ceramics into food and simulants, and to develop adequate methodologies for testing these articles including at lower limits. In the present work, the release of metals from 73 ceramic samples including 2 reference materials was investigated under different conditions. The sets of samples studied aimed to represent a vast variety of articles including hand crafted and highly decorated samples more prone to release a broader range and greater levels of metals.

In the sets of samples investigated, a number of metals were found in leaching solutions e.g. aluminium (Al), iron (Fe), zinc (Zn), cobalt (Co), lithium (Li), barium (Ba), manganese (Mn), vanadium (V), lead (Pb), cadmium (Cd), antimony (Sb), titanium (Ti), chromium (Cr), nickel (Ni), copper (Cu) and arsenic (As).

A comparison between two simulants and a benchmark food (acetic acid 4% for 24 h at 22°C, citric acid 0.5% for 2 h at 70°C and hot tomato sauce) showed that migration was higher in either simulants than into tomato sauce (2 h at 70°C). Results also showed that the migration profile in acetic acid was more representative for the tomato sauce. It was observed that is necessary to use of at least four replicates for each migration experiment due to the heterogeneity of the samples that Pb to a large variability of data.

The relationship between the third and the first migration was shown to be not always constant and most of the time higher than 10% with respect to the first migration. This implies that prediction of values for the third migration based on the value of a first migration may not be derivable.

Experiments on accelerated testing showed that increasing the acidity in an attempt to substitute the first two migrations in repeat testing were not successful from lack of clear relationships of the impact of acidity on release kinetics or mechanisms for different metals.

No significant difference was observed between a test protocol with daily successive migrations and one with a lag phase (e.g. two days, representing a weekend). A longer storage (representing occasionally used articles) did not affect considerably the metals release for ceramics tableware. This implies that a repeat use testing is adequate regardless of the frequency of use of tableware articles, and that testing is not subjected to start on certain days of the weeks, which means productivity can be maintained if establishing a repeat use testing.

For cookware, which had a smaller sample set of six different articles, three different profiles of Pb released were observed. For two samples the release of Pb over repeat testing in acetic acid 4% (24 h at 22°C) gave much higher values than those in boiling tomato sauce (6 h). For three samples the respective releases of Pb were comparable in both media. One sample presented higher releases of Pb and Cd in tomato sauce than in acetic acid 4%.

Table 14 – Results of metals release in the 1st migration in acetic acid 4% from flatware

		Li	Al	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	As	Cd	Sn	Sb	Ba	Pb
DSV [µg/dm²]		120	200		240	20	20	500	17	14	200	300	4	1	10000		200	2
Sample Id		I MIGRATION ACETIC ACID 4% [µg/dm²]																
213C02	Av	0.005	3.72	0.04	0.01	0.01	0.05	0.25	0.03	0.002	0.02	0.23		0.003	0.04	0.002	0.03	0.07
	RSD [%]	27.89	72.02	25.09	35.02	40.49	41.51	19.76	29.73	47.34	49.22	35.63		71.80	32.28	44.07	32.59	64.91
213C08	Av	0.03	10.40	-	0.18	0.18	0.49	3.01	5.52	<LOD	0.88	1.56	0.53	0.01	0.32	0.04	0.54	0.42
	RSD [%]	46.40	61.14		134.65	42.19	45.69	6.61	140.25		-	18.55	-	126.31	71.67	118.00	23.06	135.96
213C11	Av		2.72					0.37	0.08					<LOD	0.34	0.17	<LOD	<LOD
	RSD [%]		6.92					24.53								14.84		
413C02	Av	0.74	40.99			0.11	0.23	3.02	5.01	0.08	0.09	13.03	0.05	11.73	0.23	0.09	4.05	181.65
	RSD [%]	41.20	31.49			62.76	50.38	28.03	32.44	39.33	62.36	33.29	40.76	34.54	18.02	93.17	44.22	37.40
413C04	Av	0.04	6.52	0.04	0.01	0.13	0.03	0.60	0.07	0.02	0.07	0.99	0.05	0.02	0.04	0.02	0.13	12.53
	RSD [%]	12.37	10.64	7.96	22.40	19.61	7.54	1.31	18.32	9.18	78.54	21.71	62.78	53.19	16.15	36.26	16.40	15.98
413C11	Av	0.02		0.18	0.03	0.01	1.67	1.34	5.39	0.06	0.06	0.41		0.007	0.03		0.15	7.12
	RSD [%]	4.69		9.38	9.91	23.34	3.29	4.97	8.52	4.66	10.85	26.24		16.22	36.50		12.69	7.10
413C13	Av	0.03	3.80	0.04	0.003	0.01	0.01	0.24	0.001	0.01	0.01	0.08	0.003	0.001	0.06	0.03	0.05	0.08
	RSD [%]	22.79	17.04	12.70	73.69	73.17	12.61	22.59	40.15	28.97	8.21	108.72	48.95	29.55	60.12	7.39	27.12	10.49
413C15	Av	1.21	21.29	0.27	0.06	0.04	0.06	1.51	0.30	0.13	0.11	6.81	0.05	15.44	0.41	0.04	0.50	111.29
	RSD [%]	13.94	15.06	16.65	47.08	18.02	18.93	17.25	9.45	77.22	15.93	15.36	22.61	14.14	28.23	31.62	13.47	14.60
413C17	Av	1.09	38.50	1.03	0.02	0.03	0.08	1.77	0.01	0.10	0.31	9.49	0.02	38.68	0.19	0.03	51.66	150.37
	RSD [%]	1.85	6.76	2.28	6.74	19.82	36.32	11.29	36.36	19.22	56.62	15.88	52.36	6.04	68.37	7.16	5.01	4.71
413C18	Av		8.40				0.01	0.07	0.88	0.01	0.26			0.003			0.21	11.60
	RSD [%]		16.33				149.39	260.27	25.20	23.18	26.59			100.07			31.41	29.01
413C21	Av	0.32	20.68	0.07	0.01	<LOD	0.02	0.81	0.01	0.01	0.35	24.28	0.004	0.03	0.10	0.01	0.35	155.52
	RSD [%]	58.88	41.59	29.58	24.36		45.98	48.26	126.36	37.62	38.30	171.44	38.06	58.34	10.53	56.57	54.31	62.47
413C22	Av	0.33	3.75	0.10	0.004		0.03	0.33	0.002	0.01	0.09	5.14	0.002	0.70	0.13	0.003	0.08	63.83
	RSD [%]	19.28	13.73	18.46	38.95		48.90	43.50	22.12	52.21	38.69	30.78	149.41	20.34	7.60	17.26	16.70	15.13
413C26	Av	0.46	29.61	0.59	0.01	0.47	0.03	1.02	0.27	0.08	0.55	1.91		0.02	0.01		4.17	3.46
	RSD [%]	9.81	11.58	2.60	2.77	3.23	14.11	9.32	4.34	4.81	16.99	8.64		14.21	9.53		13.18	15.48
413C27	Av	1.68	77.87	0.36	0.02	0.25	0.07	1.31	0.05	0.02	0.36	3.65		0.01	0.12	0.03	6.54	6.36
	RSD [%]	13.22	12.47	5.49	8.47	15.66	14.51	14.11	13.98	21.39	15.92	7.91		13.94	17.68	17.60	10.53	16.39
615C12	Av		12.94		0.16	0.30	0.03	3.15	34.01		0.27	63.67		1.85			0.51	48.08
	RSD [%]		16.69		87.90	38.79	20.52	29.93	13.31		25.73	12.56		30.94			8.96	23.52
615C14	Av		14.21		0.06	0.13	0.20	4.96	795.21		1.82	1325.30		0.19			0.16	80.84
	RSD [%]		21.73		126.23	31.36	23.54	22.87	4.08		9.71	3.92		23.88			27.31	28.97
615C16	Av		102.37		0.10	0.17	0.09	5.52	173.22		0.64	123.38		0.02			1.09	188.79
	RSD [%]		12.13		25.39	27.24	20.02	24.98	17.08		22.18	18.62		41.21			15.95	19.96
615C20	Av		131.70		0.14	0.10	0.11	12.81	55.43		1.18	72.17		0.67			0.62	135.48
	RSD [%]		10.62		33.87	25.12	8.97	12.77	11.16		72.15	11.39		10.79			7.10	10.52
713C09	Av		132.77			0.03	1.16	16.05	0.08	0.02	<LOD			0.02			0.59	40.95
	RSD [%]		49.59			78.91	22.28	13.05	47.61	50.48				47.24			29.48	41.25
713C17	Av		1.20			0.02	0.06	0.11	1.97	0.62	0.16			<LOD			0.36	0.06
	RSD [%]		16.68			52.32	24.69	67.93	38.88	31.52	43.77						48.07	142.59
713C18	Av		5.25			0.02		0.06	0.16	0.01	<LOD			<LOD			0.40	0.03
	RSD [%]		44.99			69.87		165.00	39.65	41.63							4.70	34.24
713C19	Av	0.01	6.28		0.12		0.03	2.07	0.10		0.32	0.86	0.54	0.05	0.12	0.04	0.03	0.30
	RSD [%]	48.25	42.42		126.56		79.67	81.43	101.22		149.36	90.65	129.79	101.02		74.25	36.92	79.62
713C20	Av	0.005	1.43	0.03	0.001	0.01	0.01	0.05	0.74	0.003	0.02	0.12	0.003	0.0002	0.01	0.01	0.06	20.96
	RSD [%]	7.39	4.02	18.15	14.73	14.73	107.46	12.03	39.94	47.99	55.23	39.38	29.93	137.45	24.72	24.36	24.90	9.19
713C22	Av	0.01	1.15	0.02	0.001	0.003	0.003	0.01	0.004	0.0004	<LOD	0.56	0.001	0.02	0.03	0.001	0.06	0.73
	RSD [%]	12.57	14.78	12.22	54.77	47.63	116.18	80.81	28.47	144.90		38.27	20.34	45.40	53.48	42.59	146.04	31.30
913C02	Av	0.12	2.97	0.03	0.001	0.03	0.01	0.08	0.11	0.01	0.03	0.33	0.001	0.09	0.002	0.004	0.87	1.79
	RSD [%]	21.07	18.28	15.44	27.13	11.69	100.18	22.37	81.30	37.53	59.65	50.27	64.43	26.17	6.75	20.90	3.87	28.80
913C05	Av	0.13	2.87	0.01	0.003	0.002	0.01	0.01	0.04	0.001	0.03	0.73		<LOD	0.07	0.004	3.86	0.20
	RSD [%]	9.72	7.49	10.21	54.58	76.66	8.96	140.49	22.26	74.72	40.58	10.59			12.52	47.90	20.88	30.96

Table 15 – Results of metals release in the 3rd migration in acetic acid 4% from flatware

		Li	Al	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	As	Cd	Sn	Sb	Ba	Pb
DSV [µg/dm²]		120	200		240	20	20	500	17	14	200	300	4	1	10000		200	2
Sample Id		III MIGRATION ACETIC ACID 4% [µg/dm²]																
213C02	Av	<LOD	0.34	0.004	<LOD	<LOD	<LOD	<LOD	0.002	<LOD	0.01	<LOD		<LOD	0.031	<LOD	0.004	<LOD
	RSD [%]		33.45	39.95					28.63		50.20				37.49		33.05	
213C08	Av	0.01	9.32	-	0.01	0.18	0.29	3.48	0.09	0.030	0.18	1.07	0.02	0.01	0.22	0.02	0.30	0.26
	RSD [%]	30.21	3.07		-	94.12	-	-	54.48	-	-	48.21	135.91	151.48	121.13	37.54	58.55	121.77
213C11	Av	0.01	3.97		0.04	0.03	<LOD	0.48	<LOD	0.03	0.03	0.51	0.12	<LOD	0.39	0.56	<LOD	0.21
	RSD [%]	-	2.02		35.09			15.44		79.06	-	78.02	61.06		40.98	-		-
413C02	Av	0.26	12.56		0.06	0.04	0.09	0.97	2.07	0.09	<LOD	4.48	0.05	3.70	0.18	0.15	0.94	72.30
	RSD [%]	25.07	24.91		12.89	91.55	6.05	22.04	51.89	46.47		27.78	-	16.54	53.11	62.04	21.86	32.18
413C04	Av	0.01	0.63	0.02	<LOD	0.006	0.01	0.01	0.005	<LOD	0.005		0.01	<LOD	0.03	0.005	0.03	2.01
	RSD [%]	12.47	9.65	8.37		8.51	144.32	134.42	24.45		55.95		32.82		14.82	57.18	41.40	13.30
413C11	Av	0.003		<LOD	<LOD	0.01	0.18	0.04	1.05	0.008	<LOD	<LOD		<LOD	0.16		0.02	0.61
	RSD [%]	62.78				307.69	31.20	231.83	43.17	54.28					65.77		35.45	25.90
413C13	Av	0.11	4.67	0.21	<LOD	0.006	0.02	0.33	<LOD	0.014	0.026		0.002	0.001	0.15	<LOD	2.05	0.09
	RSD [%]	67.77	8.99	101.81		19.72	41.17	23.61		41.61	25.39		46.51	12.28	71.16		110.56	19.34
413C15	Av	0.32	4.25	0.07	0.03	0.02	0.01	0.47	<LOD	<LOD	<LOD		0.03	3.66	0.57	<LOD	<LOD	25.93
	RSD [%]	14.77	20.49	15.48	44.03	87.06	1465.14	137.68					56.38	18.63	74.82			20.38
413C17	Av	0.16	4.31	0.26	0.008	0.01	<LOD	0.17	<LOD	<LOD	<LOD		0.01	7.30	0.10	<LOD	3.91	26.19
	RSD [%]	3.94	4.76	11.90	30.25	38.95		11.51					98.07	1.70	24.38		11.15	3.03
413C18	Av		3.39			<LOD	<LOD	<LOD	0.06	<LOD	<LOD			<LOD			0.13	0.71
	RSD [%]		44.04						25.81								35.99	29.06
413C21	Av	0.12	4.77	-	0.01	<LOD	<LOD	0.24	<LOD	<LOD	<LOD	-	0.007	<LOD	0.16	<LOD	-	50.36
	RSD [%]	31.38	40.21		26.43			195.64					84.20		14.88			38.67
413C22	Av	0.06	0.12	<LOD	<LOD	<LOD	<LOD	1.02	<LOD	<LOD	<LOD	<LOD	0.004	0.10	0.08	<LOD	<LOD	9.31
	RSD [%]	17.80	460.56					121.81					162.08	26.12	1.99			22.25
413C26	Av	0.18	6.76	0.19	<LOD	0.06	0.008	0.19	0.30	0.11	0.18	0.68		0.005	<LOD		2.15	1.20
	RSD [%]	18.05	32.58	13.06		28.02	24.31	41.60	6.82	7.61	18.17	19.88		29.08			11.37	31.52
413C27	Av	0.51	30.90	0.06	<LOD	0.05	0.02	0.35	0.006	<LOD	0.11	1.62		0.003	0.039	0.0027	4.20	1.74
	RSD [%]	10.76	9.25	7.82		14.97	19.80	11.92	14.84		24.73	20.65		34.64	1.84	16.79	6.65	10.82
615C12	Av		2.97		0.01	0.05	<LOD	0.99	2.33		0.02	3.52		0.45			0.14	12.88
	RSD [%]		27.72		18.00	16.46		69.32	15.78		123.56	14.21		38.30			17.03	35.34
615C14	Av		2.32		0.01	0.03	<LOD	0.46	6.62		0.02	9.63		0.01			0.03	11.14
	RSD [%]		42.65		128.05	46.68		90.32	50.45		195.27	52.11		62.42			27.73	51.66
615C16	Av		21.36		0.01	0.04	0.005	1.14	40.30		0.07	24.55		0.004			0.22	39.22
	RSD [%]		13.32		100.57	31.88	82.83	38.64	24.85		59.15	21.13		51.21			14.67	20.01
615C20	Av		40.26		0.04	0.02	0.03	4.01	17.29		0.16	22.37		0.18			0.18	39.63
	RSD [%]		9.08		53.75	21.25	36.14	15.17	8.30		31.80	6.96		4.41			19.29	4.45
713C09	Av		60.42			0.03	1.32	5.39	0.03	0.02	<LOD			0.01			0.48	6.46
	RSD [%]		75.22			104.87	38.57	88.28	45.36	22.47				55.38			20.11	36.74
713C17	Av		<LOD			<LOD	0.02	0.19	0.45	0.18	0.34			<LOD			0.33	<LOD
	RSD [%]						24.95	46.97	32.17	27.68	199.28						15.82	
713C18	Av		3.37			<LOD	<LOD	<LOD	0.02	0.004	<LOD			<LOD	<LOD	<LOD	0.28	<LOD
	RSD [%]		156.16						34.57	96.52							12.62	
713C19	Av	<LOD	0.51		0.01	<LOD	<LOD	0.80	0.01	<LOD	0.03	<LOD	0.01	<LOD	<LOD	<LOD	<LOD	0.03
	RSD [%]		32.27		-			-	-		81.91		24.38					-
713C20	Av	0.002	0.22	0.02	<LOD	<LOD	<LOD	<LOD	0.89	<LOD	0.01		<LOD	<LOD	<LOD	0.001	0.03	2.58
	RSD [%]	9.43	12.60	9.08					44.19		170.18					60.90	86.12	11.13
713C22	Av	0.01	0.50	0.02	<LOD	<LOD	0.02	0.07	0.01	<LOD	0.01		<LOD	0.01	<LOD	<LOD	0.03	0.19
	RSD [%]	81.85	36.61	6.68			106.33	321.50	99.36		83.18			40.24			48.13	39.25
913C02	Av	0.02	0.75	0.01	<LOD	0.003	<LOD	<LOD	0.004	<LOD	0.01		<LOD	0.01	<LOD	<LOD	0.05	0.26
	RSD [%]	25.72	47.08	9.43		26.45			32.07		91.78			27.33			29.91	27.65
913C05	Av	0.02	0.41	<LOD	<LOD	<LOD	<LOD	<LOD	0.005	<LOD	0.006	<LOD		<LOD	0.02	0.0006	3.24	0.02
	RSD [%]	15.32	8.38						14.83		60.00				40.06	20.42	20.29	18.33

Table 16 – Results of metals release in the 1st migration in acetic acid 4% from hollowware

		Li	Al	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	As	Mo	Cd	Sn	Ba	Pb
DSV [µg/kg]		600	1000		1200	100	100	2500	84	72	1000	1500	18	10	5	50000	1000	10
Sample Id		I MIGRATION ACETIC ACID 4% [µg/kg]																
113C01	Av	135.15		257.38	1.98	0.39	0.84	183.06	7.02	0.51	23.97	<LOD	0.31	0.38	0.07	<LOD	6.69	0.99
	RSD [%]	11.40		9.20	0.10	22.40	19.70	5.50	10.62	19.44	18.14		25.12	4.07	29.46		13.45	33.69
113C02	Av	13.14	465.95		1.09	0.37		19.91	1.71	0.04	4.39	<LOD		0.46		1.92	44.62	1.31
	RSD [%]	0.32	14.10		-	-		14.56	17.14	-	14.79			-		-	23.90	96.12
113C03	Av	94.83		103.80	2.22	11.25	291.89	202.01	53.26	98.03	135.63	<LOD		1.95	1.45	0.55	340.26	<LOD
	RSD [%]	26.53		18.40	20.09	26.59	24.90	27.50	26.39	23.05	27.52			13.70	107.23	60.02	12.51	
213C07	Av	0.08	50.36	-	0.02	0.78	1.15	5.14	7.63	0.21	0.49	4.51	0.02	0.23	0.01	0.81	0.93	0.58
	RSD [%]	49.19	1.36		39.56	12.96	5.87	-	13.28	21.28	76.98	27.60	4.45	32.52	43.26	5.61	26.79	28.09
213C09	Av	<LOD	29.29	<LOD	0.16	<LOD	<LOD	<LOD	8.90	<LOD	2.88	5.42	<LOD	<LOD	<LOD	1.28	<LOD	<LOD
	RSD [%]		24.70		-				4.00		24.35	86.68				2.61		
413C06	Av	0.12	44.19	-	0.02	0.85	2.45	54.98	0.01	0.18	0.30	7.18	0.06	0.17	0.07	0.50	5.63	0.80
	RSD [%]	12.57	2.94		21.53	34.92	97.43	-	66.51	14.21	19.57	7.92	96.98	12.98	25.53	14.68	27.54	76.31
615C13	Av		83.16		0.20	1.97	<LOD	24.60	154.76		0.81	291.32			6.75		2.48	216.58
	RSD [%]		24.74		54.71	31.81		39.88	25.65		38.74	20.47			58.01		38.64	29.41
615C15	Av		65.79		0.78	0.63	0.59	27.61	2196.10		5.38	3578.33			0.57		0.92	230.90
	RSD [%]		23.76		37.13	33.68	12.87	3.86	5.38		13.57	5.67			6.54		64.49	18.61
615C17	Av		379.95		0.69	0.76	0.38	26.62	612.90		2.17	411.98			0.02		3.48	483.86
	RSD [%]		14.62		9.23	38.51	31.18	32.35	15.02		59.04	14.66			46.93		36.02	11.54
615C18	Av		176.72		1.38	0.40	<LOD	7.78	316.83		0.49	210.52			0.06		1.90	317.86
	RSD [%]		3.69		60.44	35.64		62.40	4.09		16.48	5.92			55.97		5.07	7.80
615C19	Av		1099.57		0.56	0.46	1.02	116.21	617.66		7.70	794.41			0.39		228.73	707.63
	RSD [%]		10.70		25.02	16.42	12.66	2.99	12.09		17.03	12.86			37.61		35.19	9.44
713C01	Av		288.64		1.49	0.31	8.51						0.56		0.53		8.11	347.50
	RSD [%]		39.12		29.19	21.88	40.98						28.24		6.74		18.01	6.84
713C03	Av	0.08	15.80				0.14	23.56	1.86	6.07	0.24	3.97					0.65	211.58
	RSD [%]	23.09	6.75				44.61		27.35		29.46						20.80	23.41
713C04	Av	1.15	5229.22		1.64		3.65	147.74	0.26	2.01			0.39				6.93	243.72
	RSD [%]	51.59	22.33		38.62		32.11	58.40	21.36	17.46			26.02				74.16	57.79
713C11	Av	1.82	822.22	6.35	0.95	<LOD	8.34	138.81	0.34	<LOD	2.77	6.70	0.76	0.15	<LOD	<LOD	10.58	466.36
	RSD [%]	54.63	145.11	64.28	93.87		103.79	93.22	111.83		21.04	48.57	89.26	82.28			16.39	42.22
713C21	Av	<LOD	65.84	0.27	0.11	40.87	0.24	74.47	0.11	<LOD	0.20	<LOD	0.33		0.01	1.28	2.20	11.22
	RSD [%]		28.55	86.19	74.72	1.64	31.76	2.84	171.77		22.02		23.74		127.74	77.39	37.49	29.35
713C23	Av	<LOD	116.68	<LOD	<LOD	<LOD	0.41	<LOD	0.01	<LOD	5.16		<LOD		<LOD		6.30	75.65
	RSD [%]		50.16				93.98		19.12		236.96						6.98	18.17
713C28	Av	39.15	2418.82	147.89	23.36	5.78	37.33	586.91	<LOD	0.42	5512.22	3167.56	11.23		1.67	2.22	227.45	19061.17
	RSD [%]	2.63	5.77	3.01	9.64	10.15	8.82	2.56		216.40	4.11	30.25	27.23		31.20	117.81	7.16	5.75
713C30	Av	13.16	3891.77	5.13	11.93	<LOD	77.80	<LOD	2.20	4.10	0.67	13.70	3.99		0.45	<LOD	16.92	321.89
	RSD [%]	11.59	9.65	15.52	7.60		14.38		8.03	4.77		27.23	17.63		68.45		43.57	33.62
913C03	Av	0.19	63.05	0.63	0.25	<LOD	0.60	<LOD	0.17	<LOD	3.39	108.71	0.03		<LOD	<LOD	6.01	0.40
	RSD [%]	4.66	4.64	5.19	21.98		12.05		13.84		20.35	14.95	60.24				8.87	18.63
913C08	Av	0.96	18.92	0.22	<LOD	0.12	<LOD	<LOD	<LOD	<LOD	0.73	10.61	0.02		0.01	0.62	0.16	0.33
	RSD [%]	14.44	14.76	7.97		32.48					47.80	24.87	62.46		104.75	9.75	14.59	17.60
913C09	Av	1.74	20.58	0.20	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	0.37	8.93	0.01		0.01	0.58	0.20	0.52
	RSD [%]	6.28	5.20	16.21							45.19	48.32	52.44		74.69	6.62	33.11	65.82

Table 17 – Results of metals release in the 3rd migration in acetic acid 4% from hollowware

		Li	Al	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	As	Mo	Cd	Sn	Ba	Pb
DSV [µg/kg]		600	1000		1200	100	100	2500	84	72	1000	1500	18	10	5	50000	1000	10
Sample Id		III MIGRATION ACETIC ACID 4% [µg/kg]																
113C01	Av	17.90		188.89	0.27	<LOD	<LOD	23.04	0.99	<LOD	2.56	<LOD	<LOD	0.05	<LOD	1.28	1.09	<LOD
	RSD [%]	12.74		9.19	14.49			16.05	11.19		19.13			6.34		57.43	35.41	
113C02	Av	4.04	144.55		0.55	<LOD	1.98	152.65	1.21	0.39	1.25	<LOD	<LOD	0.91	<LOD	<LOD	6.56	0.69
	RSD [%]	3.31	8.27		5.75			132.25	30.91	136.00	6.04			55.66			19.31	
113C03	Av	13.50		40.66	0.36	2.08	47.30	48.50	8.65	11.37	18.22	<LOD	<LOD	0.33	0.17	0.89	55.76	<LOD
	RSD [%]	6.99		14.01	15.42	9.60	8.03	29.67	8.49	6.40	7.66			13.49	95.95	79.62	10.29	
213C07	Av	0.03	36.82	-	0.01	0.93	1.50	10.62	0.62	0.24	<LOD	3.80	0.02	0.22	0.01	0.38	1.35	0.31
	RSD [%]	22.78	4.69		78.55	27.44	41.20	6.22	16.94	39.78		10.83	31.00	7.28	11.87	9.52	53.29	63.35
213C09	Av	<LOD	4.55	2.31	<LOD	<LOD	<LOD	<LOD	0.39	<LOD	1.97	2.47	<LOD	<LOD	<LOD	0.32	<LOD	<LOD
	RSD [%]		-	-					31.04		31.62	37.94				-		
413C06	Av	0.39	41.64	-	4.74	10.89	28.16	880.06	0.01	16.35	97.02	167.55	17.34	0.24	41.93	33.67	1.92	4.10
	RSD [%]	155.48	11.63		172.53	160.38	164.76	171.78	-	170.23	140.63	168.13	172.96	45.62	141.39	171.96	45.65	142.82
615C13	Av		19.42		<LOD	0.44	<LOD	4.70	17.09		<LOD	26.33			2.80		1.03	78.02
	RSD [%]		31.69			58.45		70.31	57.96			58.54			54.07		39.54	39.00
615C15	Av		8.54		0.24	1.49	<LOD	6.25	91.81		3.30	148.04			0.04		0.13	39.28
	RSD [%]		63.66		108.47	162.27		54.04	122.60		132.86	123.24			89.52		64.10	53.82
615C17	Av		64.61		<LOD	0.14	<LOD	7.48	108.65		<LOD	68.52			<LOD		0.48	87.19
	RSD [%]		18.90			36.63		86.53	21.74			19.12					40.52	13.90
615C18	Av		27.48		<LOD	0.13	<LOD	<LOD	80.86		<LOD	36.51			<LOD		1.07	55.04
	RSD [%]		3.74			64.46			4.87			9.54					153.95	7.99
615C19	Av		326.65		<LOD	0.13	0.25	34.75	183.96		0.99	238.21			0.11		53.17	195.15
	RSD [%]		21.60			78.34	41.58	19.20	24.42		61.63	25.39			38.06		31.12	23.87
713C01	Av		246.44		2.30	0.15	15.63				<LOD	<LOD	1.31		0.29		13.63	41.40
	RSD [%]		16.71		19.74	28.47	15.81						29.51		20.92		60.43	9.15
713C03	Av	<LOD	<LOD		<LOD	<LOD	<LOD	<LOD	0.18	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	13.91
	RSD [%]								24.02									18.70
713C04	Av	3.94	2643.62		1.44	<LOD	11.38	167.11	0.52	4.59	0.34	<LOD	0.63	<LOD	<LOD	<LOD	7.02	53.79
	RSD [%]	45.18	50.59		51.57		40.74	62.31	30.03	37.68	70.28		46.01				69.17	10.22
713C11	Av	3.06	655.03	3.15	1.29	<LOD	12.72	120.70	0.53	<LOD	0.84	8.60	1.11	0.27	<LOD	<LOD	9.70	335.19
	RSD [%]	45.68	103.71	-	55.59		58.52	67.56	67.35		62.63	26.48	51.60	78.41			11.29	52.14
713C21	Av	<LOD	0.71	<LOD	0.04	40.53	<LOD	69.70	<LOD	<LOD	0.68	<LOD	0.20		0.03	0.99	0.59	1.10
	RSD [%]		5.88		72.84	0.82		1.44					20.42		10.15	28.34	29.86	39.33
713C23	Av		84.16	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	1.06				<LOD		9.50	7.74
	RSD [%]		43.40								154.37						54.73	27.57
713C28	Av	25.52	1053.81	79.57	23.13	2.81	58.38	106.54	2.32	0.54	611.76	204.29	10.40		0.23	4.00	36.89	2332.85
	RSD [%]	8.56	11.96	6.24	14.17	14.78	5.97	5.01	16.99	20.22	9.92	8.71	16.37		31.51	8.59	7.88	10.11
713C30	Av	8.94	906.18	0.90	2.74	<LOD	62.42	<LOD	1.02	2.26	<LOD	<LOD	1.20		0.25	<LOD	6.94	76.38
	RSD [%]	10.52	12.96	7.00	16.43		9.10		8.26	23.71			25.33		36.56		15.26	17.40
913C03	Av	<LOD	6.90	0.37	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	-	<LOD		<LOD	<LOD	0.55	<LOD
	RSD [%]		34.42	14.29													21.62	
913C08	Av	0.11	1.83	0.36	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	-	<LOD		<LOD	<LOD	0.14	<LOD
	RSD [%]	16.94	17.38	16.12													181.06	
913C09	Av	0.22	3.38	0.38	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	-	<LOD		<LOD	<LOD	0.06	<LOD
	RSD [%]	6.66	57.78	10.34													38.50	

Table 18 – Results of metals release in the 1st migration in acetic acid 4% from cooking ware

		Li	Al	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	As	Mo	Cd	Sn	Ba	Pb
DSV [µg/kg]															1.9			3.8
Sample Id										I MIGRATION ACETIC ACID 4% [µg/kg]								
C6	Av			1.67			36.04	258.89	0.29		14.23	<LOD			0.12		100.98	1583.32
	RSD [%]			21.54			41.02	33.99	30.62		31.54				33.92		10.27	13.73
C14	Av	98.26	17928.72	13.04		16.46	368.07		5.96		18.02		99.25		0.06		271.70	27.02
	RSD [%]	17.18	37.01	53.16		28.00	40.11		56.13		15.16		16.20		27.21		41.71	24.54
416C28	Av	-	282.62	-	-	-	3.28	21.28	<LOD	-	4.75	84.88	-	-	308.98	-	5.76	1029.87
	RSD [%]		29.40				22.75	12.57			129.61	41.37			38.63		20.17	35.10
416C29	Av	-	12.74	-	-	-	<LOD	4.71	0.47	-	0.73	<LOD	-	-	<LOD	-	0.12	0.61
	RSD [%]		92.65					109.44	17.37		38.97						32.52	13.98
416C30	Av	-	58.57	-	-	-	<LOD	4.48	67.06	-	1.17	<LOD	-	-	0.21	-	5.99	83.90
	RSD [%]		17.87					24.81	19.54		95.06				37.62		25.74	28.32
416C31	Av	-	31.25	-	-	-	<LOD	17.69	<LOD	-	2.38	20.40	-	-	0.07	-	1.78	17.41
	RSD [%]		4.07					167.29			151.14	153.66			27.79		167.88	24.19
713C02	Av		40197.50		32.93	21.83	525.88			9.24	19.00	95.36	17.95		0.10		65.50	764.39
	RSD [%]		30.14		35.14	51.15	49.62			52.69	66.77	51.01	38.01		45.66		48.40	27.16
713C05	Av	6.03	2839.44		1.45		13.19	74.64	0.26	3.66	2.00	1378.73	7.18				457.84	7.39
	RSD [%]	40.66	33.49		75.71		48.62	39.52	45.62		35.93	21.58	34.60				17.70	51.51
713C06	Av	31.74	1440.67	2.10	21.14	<LOD	47.82	66.60	0.48	1.63	6.77	54.45	2.45	2.57	0.39	<LOD	26.39	1094.09
	RSD [%]	95.07	50.34	-	105.29		79.72	73.34	86.26	87.59	30.37	65.99	81.32	111.15	71.86		80.36	52.27
713C07	Av	<LOD	79.17		42.00	1.77	61.33		<LOD	1.40	2.70	56.37	12.04		<LOD		31.93	1.06
	RSD [%]		30.44		22.94	51.46	26.58			30.68	33.91	29.67	30.93				20.62	47.49
713C08	Av	28.29	833.73	-	17.23	8.35	44.24	252.24	1.23	1.18	24.80	95.18	5.21	2.34	11.49	17.31	75.18	2973.80
	RSD [%]	67.11	64.06		40.96	71.18	32.19	61.79	64.32	53.29	115.75	59.81	18.67	81.73	167.82	171.65	66.04	71.03
713C12	Av	9.42	11041.89		20.44		21.47	6.35	0.13		0.81		1.01				43.38	489.84
	RSD [%]	65.48	150.10		33.21		40.18		44.85		54.97		39.04				21.28	13.02
713C15	Av	8.83	533.90		0.32		2.37	235.56	0.06		1020.61	15.47	0.67		0.67		102.81	11034.90
	RSD [%]	11.26	16.72		35.59		6.40	6.14	8.00		18.56	7.59	8.06		25.10		21.30	12.88
713C16	Av	<LOD	285.13	<LOD		8.94	1.76	21.93		2.05	1.00	27.51	<LOD		0.02		19.60	1244.85
	RSD [%]		40.08			98.43	67.50	133.14		41.20	97.16	22.44			73.17		30.61	34.80
713C27	Av	<LOD	14672.17	28.83	48.94	20.02	92.44	2859.78	1.98	<LOD	15.21	780.10	15.77	<LOD	<LOD	2.49	192.23	3664.29
	RSD [%]		32.36	18.45	17.93	25.99	36.71	21.92	31.98		29.21	30.64	23.15			60.99	35.88	30.86
916C16	Av	-	355.40	399.58	-	0.60	90.97	126.67	76.37	0.23	1488.69	1711.36	-	-	1.52	-	32.53	0.77
	RSD [%]		40.25	34.17		47.45	39.42	42.26	39.73	56.90	30.38	34.80			135.94		36.48	67.10
916C17	Av	-	93.23	193.14	-	0.20	31.14	31.80	63.17	<LOD	6.09	482.62	-	-	0.14	-	1.31	<LOD
	RSD [%]		9.99	6.56		45.82	8.44	3.79	8.53		15.00	8.62			10.45		18.55	
1015C01		-	250.87	-	<LOD	0.75	0.60	7.22	<LOD	-	0.98	43.97	-	-	<LOD	-	39.82	1.67
	RSD [%]		37.68			27.21	117.17	83.75			88.05	26.36					33.57	15.56
1016C02		-	1717.32	192.92	-	4.65	326.65	55561.61	5.46	8.24	7.93	19.25	-	-	1.67	-	49.90	42.00
	RSD [%]		65.44	57.33		77.96	73.24	74.94	70.69	71.59	65.97	55.03			73.13		64.71	47.28
1016C03		-	810.88	120.89	-	0.54	10.79	676.34	2.42	0.70	31.54	7.48	-	-	0.02	-	20.35	0.51
	RSD [%]		11.16	30.85		114.57	196.68	191.55	87.15	190.09	171.56	107.41			41.06		174.81	93.06
1016C04		-	40.31	7.24	-	1.11	16.90	25.47	9.00	<LOD	12.39	<LOD	-	-	<LOD	-	11.22	<LOD
	RSD [%]		17.69	16.56		19.42	10.16	6.18	10.86		8.28						7.35	

Table 19 – Results of metals release in the 3rd migration in acetic acid 4% from cooking ware

		Li	Al	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	As	Mo	Cd	Sn	Ba	Pb
DSV [µg/kg]															1.9			3.8
Sample Id										III MIGRATION ACETIC ACID 4% [µg/kg]								
C6	Av			22.72			51.80	1400.62	0.34		9.53	<LOD			0.12		173.70	1203.32
	RSD [%]			34.19			40.69	33.39	9.27		31.76				33.92		23.91	23.14
C14	Av	82.60	10714.46	7.31		7.29	498.93		4.79		13.06		120.28		0.05		555.75	14.95
	RSD [%]	33.21	29.42	33.26		49.74	34.60		51.21		37.32		20.66		84.31		56.32	25.06
416C28	Av	-	83.78	-	-	-	0.52	<LOD	<LOD	-	0.50	23.30	-	-	75.16	-	1.07	254.65
	RSD [%]						34.11				61.31	14.18			17.80		22.41	18.92
416C29	Av	-	<LOD	-	-	-	<LOD	<LOD	<LOD	-	<LOD	<LOD	-	-	<LOD	-	<LOD	<LOD
	RSD [%]																	
416C30	Av	-	6.39	-	-	-	<LOD	<LOD	5.30	-	0.27	<LOD	-	-	<LOD	-	0.79	7.06
	RSD [%]		25.53						9.95		99.95						35.45	19.81
416C31	Av	-	6.22	-	-	-	<LOD	<LOD	<LOD	-	0.24	<LOD	-	-	<LOD	-	0.31	1.63
	RSD [%]		34.70								19.20						91.75	22.43
713C02	Av		10963.54		7.85	4.72	265.44			2.90	5.39	40.10	9.33		0.05		55.20	152.33
	RSD [%]		40.80		40.12	41.07	38.96			48.49	55.06	44.78	53.78		76.66		49.72	35.46
713C05	Av	2.77	1647.37		0.56	<LOD	12.71	44.22	0.16	2.10	0.28	64.95	1.12	<LOD	<LOD	<LOD	133.80	1.67
	RSD [%]	24.41	39.61		27.15		31.32	29.28	34.22	26.34	35.94	50.75	28.86				52.23	32.18
713C06	Av	51.38	2409.85	<LOD	11.15	<LOD	65.27	152.06	2.54	30.70	2.99	77.25	1.89	0.76	0.52	<LOD	35.16	1423.31
	RSD [%]	14.29	4.07		31.25		23.29	4.80	38.57	164.39	21.57	55.80	39.33	29.10	47.90		37.02	25.21
713C07	Av		52.21		25.97	1.56	96.42			0.88	1.11	20.94	13.07		<LOD		76.92	<LOD
	RSD [%]		9.70		8.89	16.59	3.90			9.85	12.71	6.53	9.37				3.53	
713C08	Av	28.44	616.33	-	13.74	8.21	128.93	95.77	1.91	2.71	38.41	135.97	4.68	5.38	0.08	81.66	216.07	586.32
	RSD [%]	42.74	77.74		17.23	60.15	64.97	85.64	71.14	99.71	165.33	144.88	23.47	106.05	32.16	172.91	62.59	12.29
713C12	Av	13.69	687.01		25.03	<LOD	52.98	<LOD	0.49	<LOD	1.64	<LOD	0.64	<LOD	<LOD	<LOD	56.06	106.38
	RSD [%]	143.08	59.47		117.88		87.91		70.20		150.68		134.53				30.13	14.19
713C15	Av	1.68	117.10		<LOD	<LOD	0.54	58.89	<LOD	<LOD	155.20	<LOD	0.16	<LOD	0.14	<LOD	19.80	2498.33
	RSD [%]	10.03	8.48				6.09	12.90			16.52		10.21		18.44		15.34	10.50
713C16	Av		463.70	<LOD		5.99	2.30	31.64		0.79	<LOD	<LOD			<LOD		13.14	197.02
	RSD [%]		14.56			135.86	50.26	89.42		59.47							21.00	39.38
713C27	Av	<LOD	8475.15	24.88	60.77	8.73	66.52	1359.21	2.87	<LOD	10.64	311.24	13.79	<LOD	<LOD	1.64	79.23	1615.44
	RSD [%]		22.38	25.06	24.14	18.95	34.96	13.69	24.68		22.43	20.86	36.08			60.14	23.49	18.96
916C16	Av	-	5.44	20.11	-	<LOD	1.02	3.90	0.76	<LOD	6.61	13.95	-	-	0.02	-	0.17	<LOD
	RSD [%]		3.56	50.41			23.88	114.06	20.63		17.84	17.16			68.85		50.39	
916C17	Av	-	6.90	9.18	-	<LOD	1.12	23.28	2.05	<LOD	<LOD	20.97	-	-	<LOD	-	0.43	<LOD
	RSD [%]		10.78	25.60			39.77	54.17	37.36			28.31					203.72	
1015C01		-	203.61	-	<LOD	<LOD	-	<LOD	<LOD	-	0.26	19.63	-	-	<LOD	-	22.75	<LOD
	RSD [%]		25.26								147.71	38.03					28.60	
1016C02		-	779.67	59.56	-	14.19	-	115901.44	3.37	6.66	3.19	9.62	-	-	0.51	-	23.10	<LOD
	RSD [%]		97.04	86.24		138.45		119.72	83.17	77.29	91.94	37.63			105.75		97.27	
1016C03		-	107.72	44.35	-	<LOD	-	34.57	0.28	<LOD	0.48	<LOD	-	-	<LOD	-	0.51	<LOD
	RSD [%]		20.17	6.48				82.28	2.45		98.32						13.61	
1016C04		-	3.09	1.59	-	0.18	1.84	3.09	1.00	<LOD	1.37	<LOD	-	-	<LOD	-	1.35	<LOD
	RSD [%]		13.25	16.79		21.95	4.48	54.90	2.87		28.19						7.00	

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Annex 1: Work Operating Procedure for metals determination by ICP-MS

Determination of lead, cadmium and other metals into acetic acid 4% (v/v) or citric acid 0.5% (w/v) using ICP-MS

1. SCOPE AND FIELD OF APPLICATION

This procedure describes an ICP-MS method for the determination of lead (Pb), cadmium (Cd) and other metals such as silver (Ag), aluminium (Al), arsenic (As), barium (Ba), cobalt (Co), chromium (Cr), copper (Cu), iron (Fe), lithium (Li), manganese (Mn), molybdenum (Mo), nickel (Ni), antimony (Sb), tin (Sn), strontium (Sr), titanium (Ti), vanadium (V), zinc (Zn) and zirconium (Zr) in acetic acid 4 % (v/v) or citric acid 0.5%(w/v) leachates obtained after the extraction test.

2. NORMATIVE REFERENCES

ISO 17294-1:2005, Water quality — Application of inductively coupled plasma mass spectrometry (ICP-MS) for the determination of elements — Part 1: General guidelines and basic principles

ISO 17294-2005, Water quality — Application of inductively coupled plasma mass spectrometry (ICP-MS) for the determination of elements — Part 2: Determination of 62 elements

3. TERMS AND DEFINITIONS

ICPMS — Inductively coupled plasma mass spectrometry

ANALYTICAL BLANK — Value determined by a blank sample covering the complete analytical procedure including extraction, clean-up.

BLANK CALIBRATION SOLUTION

Solution prepared in the same way as the calibration solution, but leaving out the analyte

CALIBRATION SOLUTION

Solution used to calibrate the instrument, prepared from stock solutions or from a certified standard

CHECK CALIBRATION SOLUTION

Solution of known composition within the range of the calibration solutions

INSTRUMENT DETECTION LIMIT

Smallest concentration that can be detected with a defined statistical probability using a contaminant-free instrument and blank calibration solution.

DETECTION LIMIT — The limit of detection is expressed as the mean analytical blank value plus three times the standard deviation of the analytical blank

QUANTIFICATION LIMIT

Limit above which a quantification of the measurands is possible, expressed as the mean analytical blank value plus, either, five to ten times the standard deviation of the analytical blank

WOP — Working Operating Procedure

4. PRINCIPLE

Determination of Pb, Cd and other elements by inductively coupled plasma mass spectrometry (ICP-MS) consists of the following steps:

1. Introduction of a measuring solution into radiofrequency plasma to cause dissolution, atomization and ionization of elements;
2. Extraction of the ions from plasma through a differentially pumped vacuum interface and separation on the basis of their mass-to-charge ratio by a mass spectrometer;
3. Transmission of the ions through the mass separation unit and detection, usually by a continuous dynode electron multiplier assembly, and ion information processing by a data handling system;
4. Quantitative determination after calibration with suitable calibration solutions.

The relationship between signal intensity and mass concentration is usually a linear one over at least five orders of magnitude. For more details refer to ISO 17294-1:2005.

5. INTERFERENCES

It is important underline that when using ICP-MS, the presence of concomitant elements in the sample can cause interferences, for instance systematic errors in the measurement of the signal. Interferences are classified into spectral and non-spectral interferences.

The components that can cause spectral interferences are the following:

1. An isotope of another element having the same nominal mass-to charge-ratio as the analyte isotope, for example 114Cd (analyte) and 114Sn (interferant). Isobaric interferences may be corrected using the abundance of a different isotope of the interfering element; an example for Pb and Cd is reported in table 1. However correction options are often included in the instrument software. The isotope for measurements can usually be chosen free from isobaric interferences.
2. Polyatomic or molecular and doubly charged ion interferences. In many cases this ions contain argon (plasma gas) and/or oxygen originating from the water of the solution aspirated, for example 114MoO+ (interferant) and 111Cd, 114Cd (analyte). Significant molecular and doubly charged interferences shall be corrected for.

Example:

Corrected Cd signal (using natural isotopes abundances for coefficient approximations):

114Cd = (m/z 114 signal) - (0.02684)(118Sn signal).

Non spectral physical interferences are associated with the sample nebulisation and transport processes as well as with ion-transmission efficiencies. Nebulisation and transport processes can be affected if a matrix component causes a change in surface tension or viscosity. Changes in matrix composition can cause relevant signal suppression or enchantement. Dissolved solids can deposit on the nebuliser tip of a pneumatic nebuliser and on the interface skimmers. Total solid levels below 0.2% (2.000 mg/l) are recommended to minimise solid deposition. An internal standard can be used to correct for physical interferences, if it is carefully matched to the measurement element so that the two elements are similarly affected by matrix changes. Dilution of the sample fivefold will usually eliminate the problem.

Detailed information on spectral and non-spectral interferences for other metals are given in clause 6.1 of ISO 17294-1.

Table 1. Pb and Cd spectral interferences

Element	Isotope	Abundance %	Theoretical interferences		Interference with practical relevance
			Inter-element	Polyatomic ions	
Cd	111	12.8	-	MoO, MoOH, ZrOH,K_2O_2H	$^{94}Zr^{16}O^{1}H$ $^{95}Mo^{16}O$
	113	12.2	In	MoO, ZrOH, Ca_2O_2H, Ar_2O_2H, RuO	In, $^{97}Mo^{16}O$
	114	28.7	Sn	MoO, MoOH, RuO	Sn, $^{98}Mo^{16}O$

Pb*	206	24.1	-	PtO	-
	207	22.1	-	IrO	-
	208	52.4	-	PtO	-

*All three isotopes must be used to quantify Pb to allow for the variability of Pb isotopes in nature.

The following correction equation must be applied: (1.000) (206Pb) + (1.000) (207Pb) + (1.000) (208Pb).

6. REAGENTS AND MATERIALS

6.1 Reagents

All reagents shall be of recognised analytical grade.

6.1.1 Distilled water or water of equivalent purity (grade 3 water complying with the requirements of ISO 3696) shall be used throughout.

6.1.2 Acetic acid, (CH_3COOH), glacial, ρ = 1.05 g/ml, CAS 64-19-7.

6.1.3 Citric acid, ($HOC(COOH)(CH_2COOH)_2$, assay ≥ 99.5%, CAS 77-92-9.

6.1.4 Acetic acid test solution, 4 % (v/v) solution

Add 40 ml of acetic acid (6.1.2) to distilled water (6.1.1) and fill to 1 l. This solution shall be freshly prepared for use. Proportionately greater quantities may be prepared.

6.1.5 Citric acid test solution, 0.5 % (w/v) solution

Add 5g of citric acid (6.1.3) to distilled water (6.1.1) and fill to 1 l. This solution shall be freshly prepared for use. Proportionately greater quantities may be prepared.

6.1.6 Nitric acid, $r(HNO_3)$ = 1.4 g/ml.

NOTE: Nitric acid is available both as:

$r(HNO_3)$ = 1.40 g/ml equivalent to $w(HNO_3)$ = 650 g/kg;

$r(HNO_3)$ = 1.42 g/ml equivalent to $w(HNO_3)$ = 690 g/kg.

Both are suitable for use in this method.

6.1.7 Elements stock solution

Single-element stock solutions and multi-element stock solutions with adequate specification stating the acid used and the preparation technique are commercially available. For example element stock solutions with concentrations of the analytes of 1000 mg/l are suitable. These solutions are considered to be stable for more than one year, but in reference to guaranteed stability, the recommendations of the manufacturer should be considered.

6.1.8 Standard solutions

ρ (Pb, Cd, Ag, Al, As, Ba, Co, Cr, Cu, Fe, Li, Mn, Mo, Ni, Sb, Sn, Sr, Ti, V, Zn and Zr) = 10 mg/l

Pipette 10 ml of elements stock solutions of 1000 mg/l (6.1.7) separately or together, if suitable, into a 1000 ml glass volumetric flask. Add 10 ml of nitric acid (6.1.6). Bring to volume with water (6.1.1) and transfer to a suitable storage bottle.

Elements standard solutions are considered to be stable for several months, if stored in the dark. This does not apply to elements and multi-element standard solutions that are prone to hydrolysis, in particular solutions of Mo, Sn, Sb and Zr. In reference to guaranteed stability of all standard solutions, see the recommendations of the manufacturer.

6.1.9 Intermediate standard solutions

ρ (Pb, Cd, Ag, Al, As, Ba, Co, Cr, Cu, Fe, Li, Mn, Mo, Ni, Sb, Sn, Sr, Ti, V, Zn and Zr) = 1 mg/l

Pipette 10 ml of elements standard solutions of 10 mg/l (6.1.8) separately or together, if suitable, into a 100 ml glass volumetric flask. Bring to volume with water (6.1.1) and transfer to a suitable bottle. Prepare the intermediate standard solutions freshly before each use.

6.1.10 Internal standard solution (reference element solution)

The choice of elements for the reference-element solution depends on the analytical problem. Solutions of these elements should cover the mass range of interest. Generally an internal standard should be no more than 50 amu removed from the analyte. The concentrations of these elements in the sample should be negligibly low.

The elements for example ^{45}Sc, ^{85}Y, ^{103}Rh, ^{165}Ho and ^{187}Re can be suitable for this purpose.

Internal standard solution, which contain one or more of proposed elements may be used: ρ (Sc, Y, Rh, Ho and Re) = 5 mg/l.

Pipette 5 ml of each element stock solution (1000 mg/l of each Sc, Y, Rh, Ho and/or Re) into a 1000 ml volumetric flask. Add 10 ml of nitric acid (6.1.6). Bring to volume with water and transfer to a suitable storage bottle.

A suitable concentration ranges of the internal standard in samples and calibration solutions is 10 - 100 µg/l.

6.1.11 Calibration solutions

Prepare the calibration solution(s) that cover the required working range by diluting the element standard solutions (6.1.8) or intermediate element standard solutions (6.1.9). Add an adequate volume of acetic acid 4% (v/v) (6.1.4) or citric acid 0.5% (w/v) (6.1.5) to make the composition of the calibration solutions equal to the composition of the test sample solutions to minimise the matrix effect. If necessary, add internal standard solution (6.1.10) to a concentration of for example 10 µg/l or 100 µg/l of the reference elements before bringing up to volume.

6.1.12 Calibration blank solution

The calibration blank solution is prepared in the same way as the calibration solutions, but leaving out the analytes. Prepare the calibration blank solution by adding an adequate volume of acetic acid 4% (v/v) (6.1.4) or citric acid 0.5% (w/v) (6.1.5) to make the composition of the calibration blank solutions equal to the composition of the test sample. If necessary, add internal standard solution (6.1.10) to a concentration of for example 10 µg/l or 100 µg/l of the reference elements before bringing up to volume.

6.1.13 Initial calibration verification solution

The calibration verification solution is prepared by combining Pb, Cd and other elements from a standard source different from that of the calibration standard, and at concentration near the midpoint of the calibration curve. This standard may also be purchased. The solution should be prepared in the same acid composition (matrix) of the calibrations and the test samples.

6.1.14 Continuing calibration verification solution

The continuing calibration verification standard solution should be prepared combining Pb, Cd and other metals from the same standards used for calibration, at a concentration near the mid-point of the calibration curve. The solution should be prepared in the same acid composition (matrix) of the calibrations and the test samples.

6.1.15 Interference check solution

The interference check solution (ICS) is prepared to contain known concentrations of interfering elements (for Pb and Cd see Table 1; other elements see clause 6.1 of ISO 17294-1) that will demonstrate the magnitude of interferences and provide an adequate test of any corrections, for example Mo serves (if not presented in the samples) to indicate oxide effects on Cd isotopes. The other components are present to evaluate the ability of the measurement system to correct for various molecular-ion isobaric interferences. The ICS is used to verify that the interference levels are corrected by the data system within quality control limits. These solutions must be prepared from ultrapure reagents or they can be obtained commercially.

6.1.16 Optimisation solution

The optimisation solution, commercially available, serves for mass calibration and for optimization of the ICP-MS apparatus conditions, for example adjustment of maximal sensitivity with respect to minimal oxide formation rate and minimal formation of doubly charged ions. It should contain elements covering the entire mass range, as well as elements prone to a high oxide formation rate or to the formation of doubly charged ions.

7. APPARATUS

7.1 Inductively coupled plasma mass spectrometer

ICP-MS system includes:

- Sample introduction system (pump, nebuliser, spray chamber);
- Inductively coupled plasma (radio-frequency generator, load coil, torch);
- Quadrupole or time-of-flight mass spectrometer, capable of scanning a mass range from 5 m/z (amu) to 240 m/z (AMU) with a resolution of at least 1 m/z peak width at 5 % of peak height, or sector field mass spectrometer;
- Collision/reaction cell that can be pressurized with helium and kinetic energy discrimination for polyatomic interference attenuation;
- Process control and data processing equipment;
- Argon gas supply - high purity grade, i.e. > 99.99 %;
- Hellium for collision cell – Ultra high purity grade, i.e > 99.999 %;
- Optional autosampler or additional (peristaltic) pump.

For more detailed information on the instrumentation, refer to ISO 17294-1:2005.

7.2 Accessories

The stability of test samples and calibration solutions depends to a high degree on the container material. The material shall be checked according to the specific purpose. For the determination of metals in acetic acid 4% (v/v) or citric acid 0.5% (w/v) leachates, high density polyethene (HDPE) or polytetrafluoroethene (PTFE) containers (e.g. falcon tubes and storage bottles) are allowed.

Immediately before use, all glassware used to prepare stock and standard solutions should be washed thoroughly with warm diluted nitric acid e.g. w(HNO_3) = 10 %, and then rinsed several times with water (6.1.1).

The use of piston pipettes is permitted and also enables the preparation of lower volumes of calibration solutions. The application of dilutors is also allowed. For more detailed information on the instrumentation, refer to ISO 17294-1:2005.

8. PROCEDURE

8.1 Instrument set up

Adjust the instrumental parameters of the ICP-MS system in accordance with the manufacturer's manual. Wait at least 30 min to stabilise the plasma and adjust the instrument to working condition.

For guidance consult ISO 17294-1. For the selection of suitable isotopes refer to table 1 and clause 6.1 of ISO 17294-1. Use the recommended optimisation solution (6.1.16) to optimise or check the sensitivity and the stability of the system. Check the resolution and the mass calibration as often as required by the manufacturer. Define the relative atomic masses and the corresponding corrections. Define take-up and rinsing times to avoid memory effects.

Both standard mode or spectrum helium mode (KED - kinetic energy discrimination) are acceptable. KED mode should be used for elements with more interferences.

The use of an internal standard is recommended. Add the internal standard solution (6.1.10) to the interference check solution (6.1.15), to calibration solutions (6.1.11), to the blank calibration solutions (6.1.12) and to all test portions of samples before the analysis or add the internal standard solution (6.1.10) on-line using two channel sample-introduction pump. The mass concentration of the reference elements shall be the same in all solutions.

8.2 Calibration

When the analytical system is first evaluated, establish a calibration curve for Pb, Cd and other elements using at least five measuring points (for example, the blank calibration solution (6.1.12) and four calibration solutions

(6.1.11) over a linear range. The calibration range should encompass the elements concentrations of the sample.

The working range in general may cover the range of 0.2 µg/l to 200 µg/l or a part of this.

For work on a daily basis, one blank solution (6.1.12) and one to two calibration solutions (6.1.11) are enough to set up a calibration graph, but check the validity of the calibration curve with a certified reference sample, a standard sample, or a suitable internal control sample.

For more details refer to ISO 17294-1 clause 9.2.

Linear regression correlation coefficient (r) must be ≥ 0.998. If correlation coefficient is < 0.998, repeat calibration.

8.3 Determination of Pb, Cd, Ag, Al, As, Ba, Co, Cr, Cu, Fe, Li, Mn, Mo, Ni, Sb, Sn, Sr, Ti, V, Zn and Zr

After establishing the calibration curves, measure the blanks and the interference check solution to establish interference correction or to check presence of interferences. Run the test samples and if the metals concentrations of the extraction solutions are found to be higher than the highest calibration point, dilute suitable aliquot portions to reduce concentrations within the working range with test solutions (6.1.4) or (6.1.5) or water (6.1.1) to have the same acidity composition of the calibration curve.

Within sufficient small intervals (for example, every 25 samples or less and at the beginning and end of the sample run) check the accuracy of at least one certified reference sample or one standard sample or a suitable internal control sample. If necessary, re-calibrate.

9. EXPRESSION OF RESULTS

State as many significant figures as are acceptable according to the precision of the measuring values, but not more than three significant figures.

10. QUALITY CONTROL

10.1 Blank

Result of the calibration blank check shall be within 3 times the instrumental detection limit.

10.2 Calibration verification and drift

Result of the initial and continuing calibration verification solutions shall not deviate more than 10%.

10.3 Internal standard abundance

Internal standard shell not deviate more than 20%.

10.4 Interference

The impact on the measured value of uncorrected isobaric, molecular and doubly charged interferences shall not be higher than 5% or three times the instrumental detection limit. Successive values of a correction factor shall not differ more than 20%.

11. PRECISION

An Interlaboratory trial, carried out among European reference and official control laboratories for food contact materials in 2014, yielded the results given in the Table 2. The sample used in the Interlaboratory trial was an acetic acid 4% (v/v) solution spiked with the elements reported in Table 3.

Table 2. Precision data for the determination of Pb, Cd, Al, Ba, Co, Mn and Ni in acetic acid 4% (v/v) in ICP-MS

Element	*l*	*n*	$\bar{\bar{X}}$ [µg/l]	σ_R [µg/l]	Reproducibility CV [%]	σ_r [µg/l]	Repeatability CV [%]
Pb	30	114	9.58	0.99	10.32	0.20	2.13
Cd	29	111	4.89	0.24	4.89	0.09	1.77
Al	28	106	728.78	66.06	9.06	17.29	2.37
Ba	26	99	785.23	38.17	4.86	8.71	1.11
Co	28	107	51.27	1.85	3.62	0.88	1.72
Mn	26	101	397.82	17.67	4.44	5.54	1.39
Ni	28	108	69.92	3.62	5.17	1.25	1.79

l is the number of laboratories;
n is the number of values;
$\bar{\bar{X}}$ is the robust mean;
σ_R is the reproducibility standard deviation;
CV is the coefficient of variation;
σ_r is the repeatability standard deviation;

Pb, Cd, Al, Ba, Co, Mn and Ni were measured in acetic acid 4% (v/v) solutions.
All precision criteria were derived applying robust statics without elimination of outliers (ISO 5725-5)

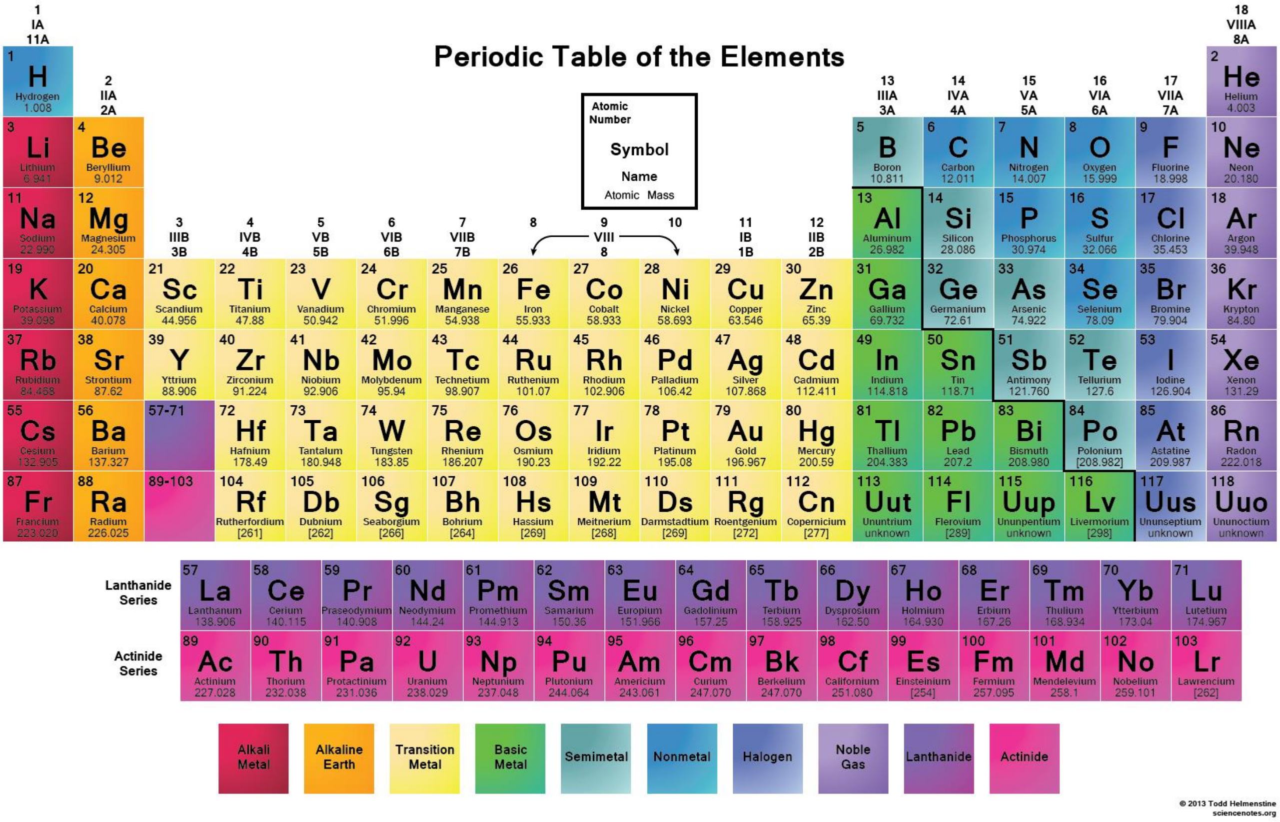
Table 3. Sample used for the Interlaboratory trial

Sample matrix	Spiked element	*Concentration [µg/l]
Acetic acid 4% v/v	Pb	9.44
	Cd	4.89
	Ba	787
	Co	51.4
	Mn	401
	Ni	70.8
	As	13.0
	Al	727
* robust mean from participants results		

12. SAFETY

General safety instructions should be followed at all times. All appropriate protective safety equipment should be worn and a fume cupboard must be used.

Annex 2: Tables of elements



Annex 3: Metals leached from flatware samples

Table 1 - Metals leached from sample 213C02 into acetic acid 4% and citric acid 0.5%

Sample	213C02								
Simulant			**Al (µg/dm²)**	**Ti (µg/dm²)**	**V (µg/dm²)**	**Cr (µg/dm²)**	**Mn (µg/dm²)**	**Fe (µg/dm²)**	**Co (µg/dm²)**
AA4%	I	**Av**	**3.72**	**0.04**	**0.005**	**0.012**	**0.05**	**0.25**	**0.03**
		std	2.68	0.01	0.002	0.005	0.02	0.05	0.01
	II	**Av**	**0.58**	**0.011**	**<LOD**	**0.003**	**0.009**	**0.03**	**0.005**
		std	0.32	0.004		0.001	0.004	0.02	0.002
	III	**Av**	**0.34**	**0.004**	**<LOD**	**<LOD**	**<LOD**	**<LOD**	**<LOD**
		std	0.11	0.002					
CA0.5%	I	**Av**	**3.54**	**0.05**	**0.006**	**0.01**	**0.06**	**0.09**	**0.05**
		std	1.33	0.02	0.002	0.01	0.02	0.02	0.01
	II	**Av**	**0.65**	**0.003**	**<LOD**	**<LOD**	**0.007**	**<LOD**	**<LOD**
		std	0.08	0.001			0.001		
	III	**Av**	**0.82**	**0.01**	**<LOD**	**<LOD**	**0.02**	**<LOD**	**0.01**
		std	0.69	0.01			0.02		0.01
Simulant			**Zn (µg/dm²)**	**Zr (µg/dm²)**	**Cd (µg/dm²)**	**Sn (µg/dm²)**	**Ba (µg/dm²)**	**Pb (µg/dm²)**	**Li (µg/dm²)**
AA4%	I	**Av**	**0.23**	**<LOD**	**0.003**	**0.04**	**0.03**	**0.07**	**0.005**
		std	0.08		0.002	0.01	0.01	0.04	0.001
	II	**Av**	**<LOD**	**<LOD**	**0.001**	**0.04**	**0.007**	**0.02**	**<LOD**
		std			0.001	0.02	0.003	0.01	
	III	**Av**	**<LOD**	**<LOD**	**<LOD**	**0.03**	**0.004**	**<LOD**	**0.018**
		std				0.01	0.001		0.002
CA0.5%	I	**Av**	**0.20**	**0.25**	**0.004**	**0.08**	**0.31**	**0.04**	**0.009**
		std	0.11	0.09	0.002	0.03	0.10	0.03	0.003
	II	**Av**	**<LOD**	**0.038**	**<LOD**	**0.013**	**0.031**	**<LOD**	0.001
		std		0.008		0.003	0.002		0.0004
	III	**Av**	**<LOD**	**0.04**	**<LOD**	**0.013**	**0.34**	**0.01**	**0.003**
		std		0.03		0.006	0.03	0.04	0.005

Table 2 - Metals leached from sample 213C08 into acetic acid 4% and citric acid 0.5%

Sample	213C08 (DE)							
Simulant			**Li (µg/dm²)**	**Al (µg/dm²)**	**V (µg/dm²)**	**Cr (µg/dm²)**	**Mn (µg/dm²)**	**Fe (µg/dm²)**
AA4%	I	**Av**	**0.03**	**10.40**	**0.18**	**0.18**	**0.49**	**3.01**
		std	0.01	6.36	0.25	0.08	0.22	0.20
	II	**Av**	**0.03**	**20.16**	**0.01**	**0.39**	**0.58**	**8.66**
		std	0.03	24.37	0.01	-	-	-
	III	**Av**	**0.009**	**9.32**	**0.01**	**0.19**	**0.29**	**3.48**
		std	0.003	3.07	-	0.17	-	-
CA0.5%	I	**Av**	**0.01**	**10.01**	**0.01**	**0.17**	**0.22**	**21.96**
		std	-	0.51	-	0.16	0.26	-
	II	**Av**	**0.02**	**11.94**	**-**	**0.10**	**0.06**	**28.46**
		std	0.02	11.58	-	0.08	0.01	-
	III	**Av**	**0.01**	**7.10**	**0.003**	**0.08**	**0.45**	**-**
		std	-	1.00	-	0.01	-	-
Simulant			**As (µg/dm²)**	**Sn (µg/dm²)**	**Ba (µg/dm²)**	**Pb (µg/dm²)**	**Co (µg/dm²)**	**Zn (µg/dm²)**
AA4%	I	**Av**	**0.53**	**0.32**	**0.54**	**0.42**	**5.52**	**1.56**
		std	-	0.23	0.12	0.58	7.75	0.29
	II	**Av**	**0.01**	**0.46**	**0.63**	**0.14**	**0.34**	**1.95**
		std	0.00	0.33	0.83	-	0.39	2.40
	III	**Av**	**0.02**	**0.22**	**0.30**	**0.26**	**0.09**	**1.07**
		std	0.03	0.27	0.17	0.31	0.05	0.52
CA0.5%	I	**Av**	**0.03**	**0.44**	**0.38**	**0.19**	**2.14**	**1.03**
		std	-	0.05	0.32	**0.17**	0.74	0.51
	II	**Av**	**0.01**	**0.18**	**1.23**	**0.05**	**0.37**	**1.62**
		std	-	0.19	1.71	0.07	0.45	1.65
	III	**Av**	**0.01**	**0.09**	**0.44**	**0.07**	**0.08**	**1.59**
		std	-	0.01	0.14	0.06	0.01	-

Table 3 - Metals leached from sample 213C11 into acetic acid 4% and citric acid 0.5%

Sample			**213C11 (BE)**						
Simulant			**Al ($\mu g/dm^2$)**	**Fe ($\mu g/dm^2$)**	**V ($\mu g/dm^2$)**	**Zn ($\mu g/dm^2$)**	**Sn ($\mu g/dm^2$)**	**Sb ($\mu g/dm^2$)**	**Pb ($\mu g/dm^2$)**
AA4%	I	**Av**	**2.72**	**0.37**	**<LOD**	**<LOD**	**0.34**	**0.17**	**<LOD**
		std	0.19	0.09	-	-	-	0.02	-
	II	**Av**	**0.43**	**0.41**	**0.25**	**0.65**	**0.29**	**0.63**	**0.04**
		std	0.08	0.01	0.02	0.28	0.02	0.17	0.04
	III	**Av**	**3.97**	**0.48**	**0.04**	**0.51**	**0.39**	**0.56**	**0.21**
		std	0.08	0.07	0.01	0.40	0.16	-	-
CA0.5%	I	**Av**	**4.22**	**1.46**	**0.19**	**0.36**	**0.09**	**0.23**	**0.04**
		std	0.16	0.07	0.02	0.10	0.07	0.07	-
	II	**Av**	**0.84**	**0.31**	**0.19**	**0.11**	**0.15**	**0.30**	**0.11**
		std	0.12	0.03	0.01	0.10	-	-	0.10
	III	**Av**	**0.48**	**0.25**	**0.07**	**0.09**	**0.11**	**0.20**	**0.08**
		std	0.14	0.16	0.02	0.07	0.04	0.07	0.06

Table 4 - Metals leached from sample 413C02 into acetic acid 4% and citric acid 0.5%

Sample				**413C02 (BE)**									
Simulant			**Li ($\mu g/dm^2$)**	**Al ($\mu g/dm^2$)**	**Cr ($\mu g/dm^2$)**	**Mn ($\mu g/dm^2$)**	**Fe ($\mu g/dm^2$)**	**Co ($\mu g/dm^2$)**	**Zn ($\mu g/dm^2$)**	**Cd ($\mu g/dm^2$)**	**Sn ($\mu g/dm^2$)**	**Ba ($\mu g/dm^2$)**	**Pb ($\mu g/dm^2$)**
AA4%	I	**Av**	**0.74**	**40.99**	**0.11**	**0.23**	**3.02**	**5.01**	**13.03**	**11.73**	**0.23**	**4.05**	**181.65**
		std	0.31	12.91	0.07	0.12	0.85	1.63	4.34	4.05	0.04	1.79	67.94
	II	**Av**	**0.35**	**13.94**	**0.12**	**0.17**	**2.13**	**1.52**	**5.17**	**5.23**	**0.53**	**1.12**	**77.33**
		std	0.09	4.67	0.15	0.12	2.17	0.74	1.38	1.26	0.35	0.31	21.28
	III	**Av**	**0.26**	**12.56**	**0.04**	**0.09**	**0.97**	**2.07**	**4.48**	**3.70**	**0.18**	**0.94**	**72.30**
		std	0.07	3.13	0.04	0.01	0.21	1.07	1.24	0.61	0.10	0.21	23.27
CA0.5%	I	**Av**	**0.86**	**45.90**	**0.49**	**0.98**	**5.94**	**6.63**	**15.59**	**13.01**	**0.27**	**7.30**	**182.24**
		std	0.05	2.41	0.08	0.08	1.45	0.56	1.51	2.04	0.06	2.21	10.95
	II	**Av**	**0.22**	**5.11**	**0.11**	**0.64**	**1.75**	**1.87**	**2.39**	**2.65**	**0.07**	**1.20**	**31.89**
		std	0.02	0.64	0.01	0.02	0.90	0.11	0.35	0.63	0.02	0.22	4.64
	III	**Av**	**0.14**	**4.24**	**0.17**	**0.66**	**1.29**	**1.76**	**2.07**	**2.14**	**0.03**	**0.81**	**26.47**
		std	0.01	0.59	0.03	0.09	0.50	0.26	0.30	0.44	0.02	0.18	3.29

Table 5 - Metals leached from sample 413C04 into acetic acid 4% and citric acid 0.5%

Sample			**413C04**								
Simulant			**Li ($\mu g/dm^2$)**	**Al ($\mu g/dm^2$)**	**Ti ($\mu g/dm^2$)**	**V ($\mu g/dm^2$)**	**Cr ($\mu g/dm^2$)**	**Mn ($\mu g/dm^2$)**	**Fe ($\mu g/dm^2$)**	**Co ($\mu g/dm^2$)**	**Ni ($\mu g/dm^2$)**
AA4%	I	**Av**	**0.043**	**6.52**	**0.042**	**0.010**	**0.13**	**0.033**	**0.60**	**0.07**	**0.022**
		std	0.005	0.69	0.003	0.002	0.02	0.002	0.01	0.01	0.002
	II	**Av**	**0.011**	**0.78**	**0.017**	**<LOD**	**0.012**	**0.008**	**<LOD**	**0.012**	**0.003**
		std	0.002	0.11	0.004		0.004	0.005		0.004	0.001
	III	**Av**	**0.008**	**0.63**	**0.019**	**<LOD**	**0.0056**	**0.01**	**<LOD**	**0.005**	**<LOD**
		std	0.001	0.06	0.002		0.0005	0.02		0.001	
CA0.5%	I	**Av**	**0.18**	**8.60**	**0.047**	**0.005**	**0.13**	**0.03**	**0.21**	**0.25**	**0.03**
		std	0.13	0.81	0.007	0.001	0.02	0.01	0.19	0.22	0.04
	II	**Av**	**0.07**	**1.39**	**<LOD**	**<LOD**	**<LOD**	**<LOD**	**<LOD**	**0.04**	**0.005**
		std	0.06	0.47						0.03	0.002
	III	**Av**	**0.05**	**0.75**	**<LOD**	**0.011**	**<LOD**	**<LOD**	**<LOD**	**0.03**	**<LOD**
		std	0.04	0.18		0.018				0.02	

Simulant			**Cu ($\mu g/dm^2$)**	**Zn ($\mu g/dm^2$)**	**Zr ($\mu g/dm^2$)**	**Cd ($\mu g/dm^2$)**	**Sr ($\mu g/dm^2$)**	**Sb ($\mu g/dm^2$)**	**Ba ($\mu g/dm^2$)**	**Pb ($\mu g/dm^2$)**
AA4%	I	**Av**	**0.07**	**0.99**	**<LOD**	**0.02**	**0.015**	**0.016**	**0.13**	**12.53**
		std	0.06	0.21		0.01	0.001	0.006	0.02	2.00
	II	**Av**	**0.01**	**0.17**	**<LOD**	**0.003**	**0.004**	**0.009**	**0.04**	**2.38**
		std	0.01	0.06		0.002	0.004	0.006	0.01	0.48
	III	**Av**	**0.005**	**-**	**<LOD**	**<LOD**	**0.004**	**0.005**	**0.03**	**2.01**
		std	0.003	-			0.004	0.003	0.01	0.27
CA0.5%	I	**Av**	**0.03**	**2.63**	**0.106**	**0.03**	**0.017**	**0.019**	**0.21**	**35.03**
		std	0.01	1.89	0.005	0.02	0.003	0.004	0.09	**17.30**
	II	**Av**	**<LOD**	**0.54**	**0.02**	**0.007**	**<LOD**	**0.006**	**0.06**	**12.30**

		std		0.45	0.01	0.004		0.004	0.02	8.55
	III	**Av**	**<LOD**	**0.37**	**0.02**	**0.004**	**<LOD**	**0.004**	**0.03**	**8.28**
		std		0.34	0.02	0.003		0.003	0.02	6.42

Table 6 - Metals leached from sample 413C11 into acetic acid 4% and citric acid 0.5%

Samples	**413C11**									
Simulant			**Li (µg/dm^2)**	**Ti (µg/dm^2)**	**V (µg/dm^2)**	**Cr (µg/dm^2)**	**Mn (µg/dm^2)**	**Fe (µg/dm^2)**	**Co (µg/dm^2)**	**Ni (µg/dm^2)**
AA4%	I	**Av**	**0.022**	**0.18**	**0.026**	**0.015**	**1.67**	**1.34**	**5.39**	**0.061**
		std	0.001	0.02	0.003	0.003	0.06	0.07	0.46	0.003
	II	**Av**	**0.005**	**0.03**	**0.006**	**<LOD**	**0.42**	**0.32**	**1.44**	**0.017**
		std	0.002	0.06	0.003		0.28	0.29	0.52	0.009
	III	**Av**	**0.003**	**<LOD**	**<LOD**	**0.01**	**0.18**	**<LOD**	**1.05**	**0.008**
		std	0.002			0.03	0.06		0.45	0.004
CA0.5%	I	**Av**	**0.047**	**1.11**	**0.045**	**0.05**	**3.88**	**30.35**	**13.85**	**0.13**
		std	0.002	0.10	0.005	0.01	0.22	23.38	2.31	0.01
	II	**Av**	**0.019**	**0.84**	**0.016**	**0.023**	**1.38**	**1.72**	**11.34**	**0.08**
		std	0.001	0.12	0.002	0.004	0.15	0.33	4.47	0.01
	III	**Av**	**0.014**	**0.64**	**0.0107**	**0.018**	**1.00**	**0.66**	**10.29**	**0.07**
		std	0.001	0.09	0.0001	0.005	0.09	0.16	5.20	0.01
Simulant			**Cu (µg/dm^2)**	**Zn (µg/dm^2)**	**Zr (µg/dm^2)**	**Cd (µg/dm^2)**	**Sn (µg/dm^2)**	**Ba (µg/dm^2)**	**Pb (µg/dm^2)**	
AA4%	I	**Av**	**0.063**	**0.41**	**<LOD**	**0.007**	**0.03**	**0.15**	**7.12**	
		std	0.007	0.11		0.001	0.01	0.02	0.50	
	II	**Av**	**0.12**	**<LOD**	**<LOD**	**0.0010**	**0.25**	**0.05**	**1.23**	
		std	0.21			0.0004	0.11	0.03	0.45	
	III	**Av**	**<LOD**	**<LOD**	**<LOD**	**<LOD**	**0.16**	**0.02**	**0.61**	
		std					0.10	0.01	0.16	
CA0.5%	I	**Av**	**0.12**	**1.68**	**0.52**	**0.009**	**0.67**	**0.35**	**12.15**	
		std	0.01	0.48	0.03	0.001	0.06	0.09	0.54	
	II	**Av**	**0.029**	**0.59**	**0.20**	**0.0024**	**0.31**	**0.20**	**4.01**	
		std	0.003	0.26	0.03	0.0004	0.05	0.03	0.49	
	III	**Av**	**0.022**	**0.34**	**0.17**	**0.002**	**0.24**	**0.14**	**2.74**	
		std	0.002	0.03	0.03	0.001	0.03	0.01	0.30	

Table 7- Metals leached from sample 413C13 into acetic acid 4% and citric acid 0.5%

Sample	**413C13**								
Simulant			**Li (µg/dm^2)**	**Al (µg/dm^2)**	**Ti (µg/dm^2)**	**Cu (µg/dm^2)**	**Pb (µg/dm^2)**	**Sb (µg/dm^2)**	**Ba (µg/dm^2)**
AA4%	I	**Av**	**0.03**	**3.80**	**0.04**	**0.014**	**0.08**	**0.029**	**0.05**
		std	0.01	0.65	0.01	0.001	0.01	0.002	0.01
	II	**Av**	**0.04**	**3.58**	**0.02**	**0.014**	**0.09**	**0.002**	**0.08**
		std	0.01	0.83	0.01	0.005	0.01	0.000	0.02
	III	**Av**	**0.11**	**4.67**	**0.21**	**0.026**	**0.09**	**0.002**	**2.05**
		std	0.08	0.42	0.22	0.007	0.02	0.002	2.26
CA0.5%	I	**Av**	**0.031**	**1.46**	**0.02**	**0.38**	**<LOD**	**0.04**	**0.68**
		std	0.003	0.25	0.01	0.74		0.01	1.33
	II	**Av**	**0.006**	**0.07**	**<LOD**	**<LOD**	**<LOD**	**<LOD**	<LOD
		std	0.001	0.11					
	III	**Av**	**0.005**	**<LOD**	**<LOD**	**<LOD**	**<LOD**	**<LOD**	**0.09**
		std	0.001						0.14

Table 8 - Metals leached from sample 413C15 into acetic acid 4% and citric acid 0.5%

Sample	413C15									
Simulant			**Li ($\mu g/dm^2$)**	**Al ($\mu g/dm^2$)**	**Ti ($\mu g/dm^2$)**	**V ($\mu g/dm^2$)**	**Cr ($\mu g/dm^2$)**	**Mn ($\mu g/dm^2$)**	**Fe ($\mu g/dm^2$)**	**Co ($\mu g/dm^2$)**
AA4%	I	**Av**	**1.21**	**21.29**	**0.27**	**0.06**	**0.04**	**0.06**	**1.51**	**0.30**
		std	0.17	3.21	0.04	0.03	0.01	0.01	0.26	0.03
	II	**Av**	**0.38**	**5.22**	**0.17**	**0.021**	**0.02**	**0.02**	**1.80**	**0.09**
		std	0.07	0.74	0.01	0.003	0.02	0.01	2.24	0.01
	III	**Av**	**0.32**	**4.25**	**0.07**	**0.03**	**0.02**	**0.01**	**0.47**	**<LOD**
		std	0.05	0.87	0.01	0.01	0.02	0.08	0.65	
CA0.5%	I	**Av**	**3.47**	**41.97**	**0.91**	**0.009**	**0.05**	**0.10**	**1.80**	**0.64**
		std	0.94	8.86	0.23	0.002	0.01	0.03	0.33	0.16
	II	**Av**	**1.81**	**23.51**	**0.63**	**0.004**	**0.03**	**0.05**	**0.47**	**0.35**
		std	0.80	10.39	0.30	0.002	0.01	0.02	0.43	0.14
	III	**Av**	**2.16**	**28.52**	**0.82**	**0.004**	**0.03**	**0.05**	**0.63**	**0.41**
		std	1.10	13.77	0.47	0.002	0.02	0.03	0.56	0.20
Simulant			**Ni ($\mu g/dm^2$)**	**Cu ($\mu g/dm^2$)**	**Sr ($\mu g/dm^2$)**	**Zn ($\mu g/dm^2$)**	**Cd ($\mu g/dm^2$)**	**Zr ($\mu g/dm^2$)**	**Ba ($\mu g/dm^2$)**	**Pb ($\mu g/dm^2$)**
AA4%	I	**Av**	**0.13**	**0.11**	**0.24**	**6.81**	**15.44**	**<LOD**	**0.50**	**111.29**
		std	0.10	0.02	0.08	1.05	2.18		0.07	16.25
	II	**Av**	**0.11**	**0.04**	**0.04**	**4.18**	**4.33**	**<LOD**	**0.10**	**30.50**
		std	0.11	0.01	0.01	0.95	0.61		0.02	5.57
	III	**Av**	**<LOD**	**<LOD**	**0.03**	-	**3.66**	**<LOD**	**<LOD**	**25.93**
		std			0.05	-	0.68			5.29
CA0.5%	I	**Av**	**0.11**	**0.17**	**0.42**	**16.81**	**34.00**	**1.90**	**1.14**	**251.56**
		std	0.05	0.07	0.09	3.74	9.71	0.19	0.50	72.63
	II	**Av**	**0.04**	**0.07**	**0.06**	**10.44**	**19.49**	**1.01**	**0.33**	**142.89**
		std	0.02	0.04	0.02	3.55	8.06	0.31	0.06	61.61
	III	**Av**	**0.03**	**0.10**	**0.06**	**11.22**	**22.84**	**1.53**	**0.24**	**166.38**
		std	0.01	0.02	0.04	5.26	10.90	0.59	0.09	84.65

Table 9- Metals leached from sample 413C17 into acetic acid 4% and citric acid 0.5%

Sample	413C17									
Simulant			**Li ($\mu g/dm^2$)**	**Al ($\mu g/dm^2$)**	**Ti ($\mu g/dm^2$)**	**Mn ($\mu g/dm^2$)**	**Fe ($\mu g/dm^2$)**	**V ($\mu g/dm^2$)**	**Co ($\mu g/dm^2$)**	**Ni ($\mu g/dm^2$)**
AA4%	I	**Av**	**1.09**	**38.50**	**1.03**	**0.08**	**1.77**	**0.017**	**0.011**	**0.10**
		std	0.02	2.60	0.02	0.03	0.20	0.001	0.004	0.02
	II	**Av**	**0.21**	**6.13**	**0.52**	**0.03**	**0.55**	**0.011**	**0.006**	**0.05**
		std	0.01	0.51	0.01	0.02	0.08	0.002	0.001	0.04
	III	**Av**	**0.16**	**4.31**	**0.26**	**<LOD**	**0.17**	**0.008**	**<LOD**	**<LOD**
		std	0.01	0.21	0.03		0.02	0.002		
CA0.5%	I	**Av**	**2.09**	**53.90**	**2.86**	**0.05**	**1.07**	**0.008**	**0.02**	**0.07**
		std	0.17	4.70	0.09	0.003	0.15	0.002	0.01	0.01
	II	**Av**	**0.46**	**11.41**	**0.86**	**<LOD**	**<LOD**	**0.003**	**0.004**	**0.02**
		std	0.09	1.81	0.18			0.001	0.002	0.01
	III	**Av**	**0.50**	**11.22**	**0.86**	**<LOD**	**<LOD**	**0.002**	**0.003**	**0.02**
		std	0.05	1.49	0.17			0.001	0.001	0.02
Simulant			**Cu ($\mu g/dm^2$)**	**Sr ($\mu g/dm^2$)**	**Zr ($\mu g/dm^2$)**	**Cd ($\mu g/dm^2$)**	**Sb ($\mu g/dm^2$)**	**Ba ($\mu g/dm^2$)**	**Pb ($\mu g/dm^2$)**	**Zn ($\mu g/dm^2$)**
AA4%	I	**Av**	**0.31**	**0.29**	**0.15**	**38.68**	**0.030**	**51.7**	**150.4**	**9.49**
		std	0.17	0.03	0.01	2.34	0.002	2.6	7.1	1.15
	II	**Av**	**0.08**	**0.08**	**0.04**	**8.62**	**0.012**	**11.0**	**33.2**	**2.31**
		std	0.06	0.03	0.01	0.55	0.001	2.0	1.8	0.27
	III	**Av**	**<LOD**	**<LOD**	**0.016**	**7.30**	**<LOD**	**3.9**	**26.2**	**-**
		std			0.003	0.12		0.4	0.8	-
CA0.5%	I	**Av**	**0.03**	**0.34**	**3.32**	**64.01**	**0.042**	**59.0**	**253.8**	**15.91**
		std	0.03	0.02	0.40	5.26	0.003	8.7	19.0	2.89
	II	**Av**	**0.00**	**0.06**	**0.95**	**23.74**	**0.007**	**12.4**	**64.3**	**4.88**
		std	0.02	0.01	0.22	2.93	0.002	2.0	13.4	0.75
	III	**Av**	**<LOD**	**0.05**	**0.99**	**26.60**	**0.010**	**10.8**	**73.3**	**5.12**
		std		0.01	0.26	1.80	0.002	1.0	9.3	0.88

Table 10 - Metals leached from sample 413C18 into acetic acid 4% and citric acid 0.5% and Tomato sauce

Sample	413C18								
Simulant			**Al (µg/dm²)**	**Mn (µg/dm²)**	**Fe (µg/dm²)**	**Co (µg/dm²)**	**Cu (µg/dm²)**	**Pb (µg/dm²)**	**Ba (µg/dm²)**
AA4%	I	**Av**	**8.40**	**0.01**	**0.1**	**0.88**	**0.26**	**11.60**	**0.21**
		std	1.37	0.01	0.2	0.22	0.07	3.36	0.06
	II	**Av**	**4.32**	**<LOD**	**<LOD**	**0.12**	**0.02**	**1.55**	**0.27**
		std	0.84			0.03	0.01	0.40	0.10
	III	**Av**	**3.39**	**<LOD**	**<LOD**	**0.06**	**<LOD**	**0.71**	**0.13**
		std	1.49			0.02		0.21	0.05
CA0.5%	I	**Av**	**11.30**	**0.009**	**0.48**	**1.05**	**0.39**	**13.00**	**0.16**
		std	1.65	0.010	0.34	0.27	0.16	3.82	0.03
	II	**Av**	**12.34**	**0.009**	**0.25**	**0.36**	**0.07**	**4.29**	**0.39**
		std	9.89	0.010	0.15	0.05	0.03	0.99	0.10
	III	**Av**	**4.99**	**<LOD**	**<LOD**	**0.28**	**0.032**	**3.39**	**0.25**
		std	0.92			0.09	0.008	1.23	0.06
Food			**Pb (µg/dm²)**						
Tomato 2h 70°C	I	**Av**	**4.01**						
		std	0.67						
	II	**Av**	**0.93**						
		std	0.16						
	III	**Av**	**0.64**						
		std	0.10						

Table 11 - Metals leached from sample 413C21 into acetic acid 4% and citric acid 0.5%

Sample	413C21										
Simulant			**Li (µg/dm²)**	**Al (µg/dm²)**	**Ti (µg/dm²)**	**V (µg/dm²)**	**Mn (µg/dm²)**	**Fe (µg/dm²)**	**Co (µg/dm²)**	**Ni (µg/dm²)**
AA4%	I	**Av**	**0.32**	**20.68**	**0.07**	**0.007**	**0.02**	**0.81**	**0.01**	**0.013**
		std	0.19	8.60	0.02	0.002	0.01	0.39	0.02	0.005
	II	**Av**	**0.15**	**7.37**	**0.07**	**0.008**	**0.03**	**0.42**	**0.005**	**0.012**
		std	0.06	3.02	0.02	0.003	0.02	0.25	0.002	0.007
	III	**Av**	**0.12**	**4.77**	**-**	**0.006**	**<LOD**	**0.24**	**<LOD**	**<LOD**
		std	0.04	1.92	-	0.001		0.48		
CA0.5%	I	**Av**	**0.86**	**70.87**	**0.30**	**0.010**	**0.03**	**2.29**	**0.009**	**0.015**
		std	0.84	45.72	0.17	0.007	0.03	1.33	0.006	0.008
	II	**Av**	**1.22**	**43.39**	**0.33**	**<LOD**	**0.03**	**0.89**	**0.006**	**0.012**
		std	1.06	27.79	0.25		0.01	1.03	0.003	0.007
	III	**Av**	**0.54**	**27.57**	**0.22**	**<LOD**	**<LOD**	**0.31**	**<LOD**	**<LOD**
		std	0.32	17.56	0.14			0.52		
Simulant			**Cu (µg/dm²)**	**Zn (µg/dm²)**	**Cd (µg/dm²)**	**Sr (µg/dm²)**	**Ba (µg/dm²)**	**Pb (µg/dm²)**		
AA4%	I	**Av**	**0.35**	**24.28**	**0.03**	**0.03**	**0.35**	**155.52**		
		std	0.13	41.62	0.02	0.03	0.19	97.16		
	II	**Av**	**0.13**	**3.29**	**0.015**	**0.03**	**0.19**	**64.70**		
		std	0.06	1.65	0.005	0.02	0.06	26.35		
	III	**Av**	**<LOD**	**-**	**<LOD**	**<LOD**	-	**50.36**		
		std		-			-	19.47		
CA0.5%	I	**Av**	**1.18**	**4.35**	**0.12**	**0.13**	**1.57**	**646.15**		
		std	0.75	2.92	0.08	0.11	0.93	439.08		
	II	**Av**	**0.63**	**3.52**	**0.07**	**0.07**	**0.93**	**389.07**		
		std	0.37	1.24	0.04	0.05	0.52	240.97		
	III	**Av**	**0.40**	**2.03**	**0.04**	**0.03**	**0.59**	**236.67**		
		std	0.23	1.50	0.03	0.04	0.30	149.85		

Table 12 - Metals leached from sample 413C22 into acetic acid 4% and citric acid 0.5%

Sample	413C22								
Simulant			Li (µg/dm²)	Al (µg/dm²)	Ti (µg/dm²)	Mn (µg/dm²)	Fe (µg/dm²)	Ni (µg/dm²)	Zr (µg/dm²)
AA4%	I	Av	0.33	3.75	0.10	0.03	0.33	0.01	<LOD
		std	0.06	0.51	0.02	0.01	0.14	0.01	
	II	Av	0.07	0.61	0.05	0.01	0.13	<LOD	<LOD
		std	0.01	0.06	0.02	0.01	0.06		
	III	Av	0.06	0.12	<LOD	<LOD	1.02	<LOD	<LOD
		std	0.01	0.55			1.24		
CA0.5%	I	Av	2.17	9.35	0.13	0.03	0.04	<LOD	0.57
		std	0.92	2.25	0.03	0.04	0.80		0.05
	II	Av	1.30	7.44	0.11	<LOD	<LOD	0.05	0.15
		std	0.54	3.30	0.07			0.09	0.05
	III	Av	1.30	11.65	0.23	<LOD	0.81	<LOD	0.27
		std	0.65	4.13	0.15		1.64		0.17
Simulant			Cu (µg/dm²)	Zn (µg/dm²)	Cd (µg/dm²)	Sr (µg/dm²)	Ba (µg/dm²)	Pb (µg/dm²)	
AA4%	I	Av	0.09	5.14	0.70	0.02	0.08	63.83	
		std	0.03	1.58	0.14	0.01	0.01	9.66	
	II	Av	0.04	3.55	0.15	0.01	0.05	11.90	
		std	0.06	1.38	0.04	0.01	0.02	2.85	
	III	Av	<LOD	-	0.10	<LOD	<LOD	9.31	
		std		-	0.03			2.07	
CA0.5%	I	Av	0.25	23.77	4.88	0.07	0.60	371.83	
		std	0.10	10.67	2.27	0.05	0.15	165.65	
	II	Av	0.13	16.37	3.29	0.03	0.37	238.37	
		std	0.05	8.10	1.42	0.04	0.14	102.48	
	III	Av	0.13	16.08	3.39	0.03	0.26	240.68	
		std	0.07	7.99	1.74	0.03	0.14	122.80	

Table 13 - Metals leached from sample 413C26 into acetic acid 4% and citric acid 0.5%

Sample	413C26									
Simulant			Li (µg/dm²)	Al (µg/dm²)	Ti (µg/dm²)	V (µg/dm²)	Cr (µg/dm²)	Mn (µg/dm²)	Fe (µg/dm²)	Co (µg/dm²)
AA4%	I	Av	0.46	29.61	0.59	0.0078	0.47	0.029	1.02	0.27
		std	0.05	3.43	0.02	0.0002	0.02	0.004	0.09	0.01
	II	Av	0.16	8.83	0.24	<LOD	0.08	0.007	0.22	0.16
		std	0.03	2.04	0.01		0.02	0.002	0.04	0.02
	III	Av	0.18	6.76	0.19	<LOD	0.06	0.008	0.19	0.30
		std	0.03	2.20	0.02		0.02	0.002	0.08	0.02
CA0.5%	I	Av	1.41	60.77	3.14	0.016	0.79	0.09	2.71	1.34
		std	0.50	26.69	0.79	0.004	0.15	0.05	1.38	0.49
	II	Av	0.62	23.89	0.92	0.003	0.17	0.019	0.82	0.36
		std	0.14	8.96	0.37	0.001	0.06	0.008	0.39	0.13
	III	Av	0.13	4.69	0.19	<LOD	0.023	<LOD	<LOD	0.08
		std	0.01	0.67	0.02		0.004			0.01
Simulant			Ni (µg/dm²)	Cu (µg/dm²)	Zn (µg/dm²)	Zr (µg/dm²)	Cd (µg/dm²)	Sn (µg/dm²)	Ba (µg/dm²)	Pb (µg/dm²)
AA4%	I	Av	0.079	0.55	1.91	<LOD	0.024	<LOD	4.17	3.46
		std	0.004	0.09	0.16		0.003		0.55	0.54
	II	Av	0.06	0.17	0.53	<LOD	0.13	<LOD	1.94	1.90
		std	0.01	0.04	0.08		0.18		0.36	0.51
	III	Av	0.11	0.18	0.68	<LOD	0.005	<LOD	2.15	1.20
		std	0.01	0.03	0.13		0.001		0.25	0.38
CA0.5%	I	Av	0.50	0.99	6.98	9.37	0.13	0.53	14.75	7.15
		std	0.14	0.25	3.24	3.59	0.10	0.45	5.53	4.60
	II	Av	0.17	0.48	2.19	3.42	0.03	0.15	6.67	2.15
		std	0.07	0.18	0.41	0.99	0.03	0.04	1.49	0.25
	III	Av	0.03	0.06	0.45	0.82	0.006	0.032	1.22	0.63
		std	0.01	0.01	0.07	0.11	0.003	0.003	0.11	0.14

Table 14 - Metals leached from sample 413C27 into acetic acid 4% and citric acid 0.5%

Sample	413C27									
Simulant			Li (µg/dm2)	Al (µg/dm2)	Ti (µg/dm2)	V (µg/dm2)	Cr (µg/dm2)	Mn (µg/dm2)	Fe (µg/dm2)	Co (µg/dm2)
AA4%	I	Av	1.68	77.87	0.36	0.020	0.25	0.07	1.31	0.046
		std	0.22	9.71	0.02	0.002	0.04	0.01	0.19	0.006
	II	Av	0.76	47.89	0.10	<LOD	0.07	0.022	0.65	0.009
		std	0.07	4.14	0.01		0.01	0.003	0.07	0.001
	III	Av	0.51	30.90	0.061	<LOD	0.05	0.018	0.35	0.006
		std	0.06	2.86	0.005		0.01	0.004	0.04	0.001
CA0.5%	I	Av	2.87	151.33	0.62	0.026	0.35	0.13	2.70	0.055
		std	0.56	25.84	0.12	0.003	0.09	0.04	0.65	0.006
	II	Av	0.94	59.23	0.13	0.003	0.08	0.04	1.61	0.011
		std	0.23	11.01	0.03	0.001	0.03	0.01	1.81	0.002
	III	Av	0.70	44.64	0.09	<LOD	0.05	0.022	0.54	0.008
		std	0.10	5.73	0.02		0.02	0.005	0.03	0.001
Simulant			Cu (µg/dm2)	Zn (µg/dm2)	Zr (µg/dm2)	Cd (µg/dm2)	Sn (µg/dm2)	Sb (µg/dm2)	Ba (µg/dm2)	Pb (µg/dm2)
AA4%	I	Av	0.36	3.65	0.14	0.013	0.12	0.028	6.54	6.36
		std	0.06	0.29	0.02	0.002	0.02	0.005	0.69	1.04
	II	Av	0.13	2.13	0.05	0.003	0.060	0.005	5.48	2.73
		std	0.04	0.31	0.01	0.001	0.005	0.001	0.21	0.27
	III	Av	0.11	1.62	0.03	0.003	0.039	0.0027	4.20	1.74
		std	0.03	0.33	0.01	0.001	0.001	0.0004	0.28	0.19
CA0.5%	I	Av	0.46	8.09	11.29	0.037	2.76	0.04	18.75	10.98
		std	0.18	1.87	4.71	0.008	1.01	0.01	2.82	3.12
	II	Av	0.10	2.74	4.08	0.009	1.01	0.004	10.30	3.07
		std	0.05	0.67	1.46	0.002	0.26	0.002	1.71	0.77
	III	Av	0.07	1.84	3.32	0.007	0.83	0.001	8.15	2.26
		std	0.02	0.32	0.72	0.002	0.16	0.001	1.19	0.44

Table 15 - Metals leached from sample 615C12 into acetic acid 4%

Sample	615C12						
Simulant			Al (µg/dm^2)	V (µg/dm^2)	Cr (µg/dm^2)	Fe (µg/dm^2)	Co (µg/dm^2)
AA4%	I	Av	12.94	0.16	0.30	3.15	34.01
		std	2.16	0.14	0.12	0.94	4.53
	II	Av	4.51	0.03	0.06	0.72	5.86
		std	1.44	0.01	0.01	0.18	1.36
	III	Av	2.97	<LOD	0.05	0.99	2.33
		std	0.82		0.01	0.68	0.37
Simulant			Cu (µg/dm^2)	Zn (µg/dm^2)	Cd (µg/dm^2)	Ba (µg/dm^2)	Pb (µg/dm^2)
AA4%	I	Av	0.27	63.67	1.85	0.51	48.08
		std	0.07	8.00	0.57	0.05	11.31
	II	Av	0.06	9.36	0.84	0.21	19.94
		std	0.01	2.37	0.31	0.04	6.42
	III	Av	0.02	3.52	0.45	0.14	12.88
		std	0.03	0.50	0.17	0.02	4.55

Table 16 - Metals leached from sample 615C14 into acetic acid 4%

Sample	615C14						
Simulant			Al (µg/dm²)	V (µg/dm²)	Cr (µg/dm²)	Fe (µg/dm²)	Co (µg/dm²)
AA4%	I	Av	14.21	0.06	0.13	4.96	795.21
		std	3.09	0.07	0.04	1.13	32.46
	II	Av	3.74	0.02	0.06	1.40	63.99
		std	0.86	0.02	0.01	0.75	35.92
	III	Av	2.32	<LOD	0.03	0.46	6.62
		std	0.99		0.02	0.41	3.34
Simulant			Cu (µg/dm²)	Zn (µg/dm²)	Cd (µg/dm²)	Ba (µg/dm²)	Pb (µg/dm²)
AA4%	I	Av	1.82	1325.30	0.19	0.16	80.84
		std	0.18	51.98	0.04	0.04	23.42
	II	Av	0.16	102.96	0.02	0.05	16.70
		std	0.07	59.53	0.002	0.02	4.52
	III	Av	0.02	9.63	0.01	0.03	11.14
		std	0.03	5.02	0.01	0.01	5.76

Table 17 - Metals leached from sample 615C16 into acetic acid 4%

Sample	615C16						
Simulant			Al (µg/dm²)	V (µg/dm²)	Cr (µg/dm²)	Fe (µg/dm²)	Co (µg/dm²)
AA4%	I	Av	102.37	0.10	0.17	5.52	173.22
		std	12.42	0.03	0.05	1.38	29.58
	II	Av	32.94	0.03	0.06	1.93	59.08
		std	4.95	0.01	0.01	0.65	12.96
	III	Av	21.36	<LOD	0.04	1.14	40.30
		std	2.84		0.01	0.44	10.01
Simulant			Cu (µg/dm²)	Zn (µg/dm²)	Cd (µg/dm²)	Ba (µg/dm²)	Pb (µg/dm²)
AA4%	I	Av	0.64	123.38	0.02	1.09	188.79
		std	0.14	22.98	0.01	0.17	37.69
	II	Av	0.12	37.85	0.01	0.35	61.53
		std	0.02	8.08	0.003	0.05	12.47
	III	Av	0.07	24.55	0.004	0.22	39.22
		std	0.04	5.19	0.002	0.03	7.85

Table 18 - Metals leached from sample 615C20 into acetic acid 4%

Sample	615C20						
Simulant			Al (µg/dm²)	V (µg/dm²)	Cr (µg/dm²)	Fe (µg/dm²)	Co (µg/dm²)
AA4%	I	Av	131.70	0.14	0.10	12.81	55.43
		std	13.99	0.05	0.02	1.64	6.18
	II	Av	49.64	0.05	0.02	4.45	21.52
		std	9.17	0.02	0.01	1.05	4.02
	III	Av	40.26	0.04	0.02	4.01	17.29
		std	3.66	0.02	0.004	0.61	1.43
Simulant			Cu (µg/dm²)	Zn (µg/dm²)	Cd (µg/dm²)	Ba (µg/dm²)	Pb (µg/dm²)
AA4%	I	Av	1.18	72.17	0.67	0.62	135.48
		std	0.85	8.22	0.07	0.04	14.26
	II	Av	0.19	27.79	0.24	0.20	50.65
		std	0.07	5.08	0.04	0.03	8.59
	III	Av	0.16	22.37	0.18	0.18	39.63
		std	0.05	1.56	0.01	0.04	1.76

Table 19 - Metals leached from sample 713C09 into acetic acid 4% and citric acid 0.5% and Tomato sauce

Sample	713C09								
Simulant			**Al (µg/dm²)**	**Mn (µg/dm²)**	**Fe (µg/dm²)**	**Co (µg/dm²)**	**Pb (µg/dm²)**	**Cd (µg/dm²)**	**Ba (µg/dm²)**
AA4%	I	**Av**	**132.77**	**1.16**	**16.05**	**0.08**	**40.95**	**0.02**	**0.59**
		std	65.84	0.26	2.10	0.04	16.89	0.01	0.17
	II	**Av**	**109.12**	**1.40**	**14.85**	**0.05**	**11.51**	**0.02**	**0.46**
		std	75.79	0.61	4.91	0.03	4.79	0.01	0.15
	III	**Av**	**60.42**	**1.32**	**5.39**	**0.03**	**6.46**	**0.01**	**0.48**
		std	45.45	0.51	4.76	0.02	2.37	0.01	0.10
CA0.5%	I	**Av**	**12.97**	**0.40**	**3.76**	**<LOD**	**90.00**	**0.01**	**3.09**
		std	6.43	0.04	1.48		48.44	0.01	0.83
	II	**Av**	**146.73**	**1.30**	**23.33**	**<LOD**	**55.85**	**0.01**	**2.61**
		std	8.33	0.61	4.45		39.54	0.02	0.91
	III	**Av**	**181.13**	**1.57**	**31.00**	**<LOD**	**49.51**	**0.02**	**3.19**
		std	62.62	0.61	12.66		37.83	0.02	0.91
Food			**Pb (µg/dm²)**						
Tomato 2h 70°C	I	**Av**	**32.57**						
		std	4.85						
	II	**Av**	**5.04**						
		std	1.68						
	III	**Av**	**4.90**						
		std	1.54						

Table 20 - Metals leached from sample 713C17 into acetic acid 4% and citric acid 0.5%

Sample	713C17										
Simulant			**Al (µg/dm²)**	**Cr (µg/dm²)**	**Mn (µg/dm²)**	**Fe (µg/dm²)**	**Co (µg/dm²)**	**Ni (µg/dm²)**	**Cu (µg/dm²)**	**Ba (µg/dm²)**	**Pb (µg/dm²)**
AA4%	I	**Av**	**1.20**	**0.02**	**0.06**	**0.11**	**1.97**	**0.62**	**0.16**	**0.36**	**0.06**
		std	0.20	0.01	0.02	0.08	0.77	0.20	0.07	0.17	0.08
	II	**Av**	**<LOD**	**<LOD**	**0.02**	**0.14**	**0.63**	**0.22**	**0.19**	**0.35**	**0.02**
		std			0.01	0.08	0.27	0.07	0.25	0.16	0.03
	III	**Av**	**<LOD**	**<LOD**	**0.02**	**0.19**	**0.45**	**0.18**	**0.34**	**0.33**	**<LOD**
		std			0.01	0.09	0.14	0.05	0.68	0.05	
CA0.5%	I	**Av**	**6.57**	**0.021**	**<LOD**	**0.17**	**0.62**	**0.23**	**<LOD**	**0.39**	**0.07**
		std	2.25	0.002		0.03	0.24	0.13		0.09	0.01
	II	**Av**	**6.51**	**<LOD**	**<LOD**	**<LOD**	**0.10**	**0.05**	**<LOD**	**0.33**	**0.067**
		std	5.77				0.05	0.02		0.04	0.004
	III	**Av**	**<LOD**	**<LOD**	**<LOD**	**<LOD**	**0.07**	**0.03**	**<LOD**	**0.40**	**<LOD**
		std					0.04	0.02		0.19	

Table 21 - Metals leached from sample 713C18 into acetic acid 4% and citric acid 0.5%

Sample	713C18							
Simulant			**Al (µg/dm²)**	**Cr (µg/dm²)**	**Co (µg/dm²)**	**Cu (µg/dm²)**	**Ba (µg/dm²)**	**Pb (µg/dm²)**
AA4%	I	**Av**	**5.25**	**<LOD**	**0.16**	**<LOD**	**0.40**	**0.03**
		std	2.36		0.06		0.02	0.01
	II	**Av**	**2.86**	**<LOD**	**0.03**	**<LOD**	**0.37**	**0.06**
		std	3.17		0.01		0.07	0.01
	III	**Av**	**3.37**	**<LOD**	**0.02**	**<LOD**	**0.28**	**<LOD**
		std	5.26		0.01		0.04	
CA0.5%	I	**Av**	**8.52**	**0.02**	**0.42**	**0.11**	**0.36**	**0.17**
		std	3.86	0.02	0.06	0.04	0.20	0.06
	II	**Av**	**4.40**	**0.02**	**0.09**	**0.50**	**0.37**	**0.11**
		std	6.58	0.03	0.02	0.63	0.10	0.19
	III	**Av**	**<LOD**	**<LOD**	**0.08**	**<LOD**	**0.36**	**0.03**
		std			0.03		0.10	0.04

Table 22 - Metals leached from sample 713C19 into acetic acid 4% and citric acid 0.5%

Sample				**713C19 (BE)**					
Simulant			**Li (µg/dm²)**	**Al (µg/dm²)**	**Fe (µg/dm²)**	**Zn (µg/dm²)**	**Cu (µg/dm²)**	**As (µg/dm²)**	**Pb (µg/dm²)**
AA4%	I	**Av**	**0.01**	**6.28**	**2.07**	**0.86**	**0.32**	**0.54**	**0.30**
		std	0.01	2.67	1.68	0.78	0.48	0.70	0.24
	II	**Av**	**<LOD**	**0.96**	**0.84**	**<LOD**	**0.04**	**0.02**	**1.23**
		std	-	0.54	0.21	-	0.04	0.02	-
	III	**Av**	**<LOD**	**0.51**	**0.80**	**<LOD**	**0.03**	**0.01**	**0.03**
		std	-	0.17	-	-	0.03	0.00	-
CA0.5%	I	**Av**	**0.02**	**7.98**	**1.42**	**0.18**	**0.04**	**0.01**	**0.19**
		std	0.01	2.58	0.11	0.06	0.01	0.00	0.23
	II	**Av**	**0.03**	**1.74**	**1.94**	**0.10**	**0.07**	<LOD	**0.14**
		std	0.01	0.95	0.95	0.03	0.01	-	0.15
	III	**Av**	**0.02**	**1.03**	**0.79**	**0.18**	**0.06**	**0.01**	**0.32**
		std	0.01	0.68	0.29	0.04	0.02	0.01	0.32

Table 23 - Metals leached from sample 713C20 into acetic acid 4% and citric acid 0.5%

Sample	**713C20**									
Simulant			**Al (µg/dm²)**	**Ti (µg/dm²)**	**Co (µg/dm²)**	**Sb (µg/dm²)**	**Ni (µg/dm²)**	**Zr (µg/dm²)**	**Ba (µg/dm²)**	**Pb (µg/dm²)**
AA4%	I	**Av**	**1.43**	**0.025**	**0.74**	**0.011**	**<LOD**	**0.008**	**0.06**	**20.96**
		std	0.06	0.005	0.30	0.003		0.001	0.02	1.93
	II	**Av**	**0.26**	**0.006**	**0.62**	**0.002**	**<LOD**	6.33	**0.03**	**3.36**
		std	0.03	0.001	0.27	0.001		0.007	0.01	0.14
	III	**Av**	**0.22**	**0.016**	**0.89**	**0.001**	**<LOD**	0.006	**0.03**	**2.58**
		std	0.03	0.001	0.39	0.001		0.007	0.02	0.29
CA0.5%	I	**Av**	**2.66**	**<LOD**	**4.82**	**0.011**	**0.011**	**0.13**	**0.49**	**42.29**
		std	0.07		1.58	0.002	0.012	0.01	0.14	2.91
	II	**Av**	**0.52**	**<LOD**	**7.97**	**0.003**	**0.008**	5.90	**0.15**	**11.69**
		std	0.23		2.57	0.001	0.003	0.01	0.03	3.76
	III	**Av**	**0.23**	**<LOD**	**8.63**	**0.002**	**0.007**	0.01	**0.05**	**8.80**
		std	0.18		1.89	0.001	0.002	0.03	0.01	2.32

Table 24 - Metals leached from sample 713C22 into acetic acid 4% and citric acid 0.5%

	713C22									
Simulant			**Li (µg/dm²)**	**Al (µg/dm²)**	**Co (µg/dm²)**	**Sr (µg/dm²)**	**Zn (µg/dm²)**	**Cd (µg/dm²)**	**Ba (µg/dm²)**	**Pb (µg/dm²)**
AA4%	I	**Av**	**0.006**	**1.15**	**<LOD**	**0.01**	**0.56**	**0.018**	**0.06**	**0.73**
		std	0.001	0.17		0.01	0.22	0.008	0.08	0.23
	II	**Av**	**0.004**	**0.31**	**0.006**	**0.02**	**0.60**	**0.007**	**0.02**	**0.23**
		std	0.002	0.08	0.009	0.01	0.20	0.004	0.02	0.13
	III	**Av**	**0.007**	**0.50**	**0.009**	**0.03**	**-**	**0.005**	**0.03**	**0.19**
		std	0.006	0.18	0.009	0.02	-	0.002	0.01	0.08
CA0.5%	I	**Av**	**0.011**	**2.93**	**0.01**	**0.010**	**1.61**	**0.03**	**0.03**	**0.98**
		std	0.002	0.84	0.01	0.002	0.71	0.01	0.01	0.12
	II	**Av**	**0.003**	**2.83**	**0.01**	**<LOD**	**0.44**	**0.02**	**0.06**	**0.50**
		std	0.001	3.62	0.01		0.21	0.01	0.09	0.23
	III	**Av**	**0.01**	**5.90**	**0.02**	**0.015**	**0.62**	**0.02**	**0.02**	**0.47**
		std	0.01	10.06	0.02	0.029	0.31	0.01	0.03	0.39

Table 25 - Metals leached from sample 913C02 into acetic acid 4% and citric acid 0.5%

Sample	913C02									
Simulant			Li (µg/dm²)	Al (µg/dm²)	Ti (µg/dm²)	Cr (µg/dm²)	Co (µg/dm²)	Ni (µg/dm²)	Cu (µg/dm²)	Zn (µg/dm²)
AA4%	I	Av	0.12	2.97	0.032	0.033	0.11	0.007	0.027	0.33
		std	0.02	0.54	0.005	0.004	0.09	0.003	0.016	0.16
	II	Av	0.031	1.07	0.017	0.005	0.014	<LOD	0.049	-
		std	0.005	0.35	0.001	0.001	0.008		0.012	-
	III	Av	0.018	0.75	0.014	0.003	0.004	<LOD	0.013	-
		std	0.005	0.35	0.001	0.001	0.001		0.012	-
CA0.5%	I	Av	0.25	15.53	0.057	0.060	0.44	0.006	0.012	1.40
		std	0.05	10.35	0.013	0.008	0.24	0.003	0.007	1.52
	II	Av	0.09	7.31	0.010	0.009	0.04	0.004	<LOD	0.24
		std	0.04	6.31	0.011	0.007	0.02	0.002		0.18
	III	Av	0.05	2.74	<LOD	<LOD	0.05	0.004	<LOD	<LOD
		std	0.02	1.19			0.03	0.001		

Simulant			Cd (µg/dm²)	Sr (µg/dm²)	Ba (µg/dm²)	Pb (µg/dm²)
AA4%	I	Av	0.09	0.03	0.87	1.79
		std	0.02	0.01	0.03	0.51
	II	Av	0.023	0.01	0.11	0.44
		std	0.002	0.01	0.03	0.06
	III	Av	0.013	0.007	0.05	0.26
		std	0.004	0.003	0.02	0.07
CA0.5%	I	Av	0.27	0.14	1.42	3.74
		std	0.07	0.11	0.18	0.98
	II	Av	0.09	0.05	0.38	1.34
		std	0.05	0.06	0.14	0.76
	III	Av	0.05	0.02	0.13	0.73
		std	0.01	0.01	0.01	0.10

Table 26 - Metals leached from sample 913C05 into acetic acid 4% and citric acid 0.5%

Sample	913C05									
Simulant			Li (µg/dm²)	Al (µg/dm²)	Co (µg/dm²)	Cu (µg/dm²)	Zn (µg/dm²)	Sn (µg/dm²)	Ba (µg/dm²)	Pb (µg/dm²)
AA4%	I	Av	0.13	2.87	0.04	0.03	0.73	0.073	3.86	0.205
		std	0.01	0.21	0.01	0.01	0.08	0.009	0.81	0.063
	II	Av	0.027	0.59	0.008	0.007	<LOD	0.05	4.41	0.024
		std	0.004	0.01	0.001	0.005		0.02	1.21	0.007
	III	Av	0.015	0.41	<LOD	0.006	<LOD	0.016	3.24	0.015
		std	0.002	0.03		0.004		0.006	0.66	0.003
CA0.5%	I	Av	0.13	3.07	0.04	0.03	0.69	<LOD	1.76	0.22
		std	0.10	2.24	0.03	0.01	0.27		0.60	0.16
	II	Av	0.06	1.75	0.02	0.04	0.17	<LOD	0.94	0.009
		std	0.04	1.27	0.01	0.05	0.02		0.52	0.007
	III	Av	0.02	0.53	0.01	<LOD	<LOD	<LOD	0.74	<LOD
		std	0.01	0.35	0.01				0.25	

Table 27 - Metals leached from sample 613C04 into acetic acid 4% and citric acid 0.5%

Sample	**613C04**							
Simulant			**Li (µg/dm²)**	**Al (µg/dm²)**	**Cr (µg/dm²)**	**Mn (µg/dm²)**	**Fe (µg/dm²)**	**Co (µg/dm²)**
AA4%	I	**Av**	**14.81**	**2519.75**	**0.30**	**0.11**	**3.15**	**0.77**
		std	8.43	472.91	0.16	0.26	0.92	0.09
	II	**Av**	**17.60**	**2215.59**	**0.36**	**<LOD**	**2.60**	**0.55**
		std	3.51	523.92	0.08		0.94	0.20
	III	**Av**	**9.34**	**1353.24**	**0.21**	**<LOD**	**1.15**	**0.37**
		std	1.66	285.60	0.04		0.65	0.17
CA0.5%	I	**Av**	**32.56**	**432.27**	**0.93**	**0.27**	**14.04**	**1.97**
		std	3.50	51.06	0.12	0.06	0.79	0.61
	II	**Av**	**14.14**	**201.61**	**0.39**	**0.23**	**6.24**	**1.19**
		std	2.44	28.21	0.08	0.12	0.96	0.40
	III	**Av**	**12.98**	**178.75**	**0.35**	**0.11**	**5.39**	**1.28**
		std	3.28	43.97	0.12	0.04	1.21	0.44
Simulant			**Ni (µg/dm²)**	**Cu (µg/dm²)**	**Zn (µg/dm²)**	**Cd (µg/dm²)**	**Ba (µg/dm²)**	**Pb (µg/dm²)**
AA4%	I	**Av**	**0.03**	**0.96**	**87.63**	**47.30**	**4.18**	**1817.04**
		std	0.02	0.19	56.85	24.99	0.98	1127.75
	II	**Av**	**0.02**	**0.81**	**111.18**	**55.46**	**3.01**	**2277.40**
		std	0.03	0.34	24.57	13.26	0.56	506.22
	III	**Av**	**<LOD**	**0.21**	**57.58**	**30.84**	**2.10**	**1221.55**
		std		0.08	10.33	5.60	0.39	212.00
CA0.5%	I	**Av**	**0.16**	**2.45**	**208.41**	**107.44**	**5.95**	**4098.87**
		std	0.02	0.05	34.23	13.38	0.64	586.84
	II	**Av**	**0.06**	**0.99**	**88.78**	**46.66**	**2.82**	**1737.37**
		std	0.02	0.19	19.27	9.74	0.25	412.66
	III	**Av**	**0.05**	**0.75**	**82.08**	**43.76**	**2.24**	**1545.05**
		std	0.01	0.24	29.61	12.40	0.67	445.41

Table 28 - Metals leached from sample 613C05 into acetic acid 4% and citric acid 0.5%

Sample	**613C05**						
Simulant			**Li (µg/dm²)**	**Al (µg/dm²)**	**Cr (µg/dm²)**	**Mn (µg/dm²)**	**Fe (µg/dm²)**
AA4%	I	**Av**	**0.004**	**8.76**	**0.02**	**0.02**	**0.33**
		std	0.005	2.82	0.02	0.02	0.35
	II	**Av**	**0.002**	**8.21**	**0.01**	**0.01**	**1.17**
		std	0.004	7.50	0.01	0.01	0.91
	III	**Av**	**<LOD**	**3.74**	**<LOD**	**<LOD**	**<LOD**
		std		0.47			
CA0.5%	I	**Av**	**0.010**	**20.96**	**0.011**	**0.014**	**0.82**
		std	0.002	1.43	0.006	0.003	0.10
	II	**Av**	**0.007**	**15.21**	**<LOD**	**0.012**	**0.65**
		std	0.002	2.98		0.006	0.46
	III	**Av**	**0.003**	**10.79**	**0.007**	**0.012**	**0.38**
		std	0.001	3.58	0.002	0.005	0.03
Simulant			**Co (µg/dm²)**	**Ni (µg/dm²)**	**Cd (µg/dm²)**	**Ba (µg/dm²)**	**Pb (µg/dm²)**
AA4%	I	**Av**	**101.16**	**0.02**	**0.02**	**579.93**	**3.05**
		std	153.08	0.03	0.01	191.59	2.03
	II	**Av**	**46.06**	**0.01**	**0.005**	**283.86**	**1.28**
		std	88.48	0.02	0.005	145.71	1.65
	III	**Av**	**1.83**	**<LOD**	**0.001**	**249.17**	**0.31**
		std	3.05		0.001	17.97	0.32
CA0.5%	I	**Av**	**43.59**	**0.022**	**0.018**	**558.27**	**3.41**
		std	33.39	0.004	0.004	54.80	0.57
	II	**Av**	**6.57**	**0.015**	**0.008**	**358.80**	**1.34**
		std	5.49	0.002	0.002	286.20	0.41
	III	**Av**	**3.10**	**0.012**	**0.005**	**184.70**	**0.88**
		std	1.15	0.001	0.002	42.38	0.16

Annex 4: Metals leached from hollowware and cooking ware samples

Table 1 - Metals leached from sample C6 into acetic acid 4%, citric acid 0.5% and Tomato sauce

Sample	**C6**								
Simulant			**Pb (µg/kg)**	**Cd (µg/kg)**	**Mn (µg/kg)**	**Ti (µg/kg)**	**Cu (µg/kg)**	**Zn (µg/kg)**	**Ba (µg/kg)**
AA4%	I	**Av**	**1583.32**	**0.12**	**36.04**	**1.67**	**14.23**	**2.18**	**180.98**
		std	217.39	0.04	14.78	0.36	4.49	1.29	18.59
	II	**Av**	**726.74**	**0.10**	**52.90**	**1.31**	**15.53**	**1.83**	**117.20**
		std	121.39	0.02	19.23	0.13	6.03	2.76	35.82
	III	**Av**	**1203.32**	**0.12**	**51.80**	**22.72**	**9.53**	**<LOD**	**173.70**
		std	278.50	0.04	21.08	7.77	3.03		41.52
CA0.5%	I	**Av**	**1458.63**	**0.08**	**6.97**	**3.40**	**0.42**	**<LOD**	**61.88**
		std	104.72	0.02	3.05	1.03	0.19		6.87
	II	**Av**	**906.20**	**<LOD**	**4.72**	**1.28**	**<LOD**	**<LOD**	**46.55**
		std	181.38		2.41	0.45			7.49
	III	**Av**	**1632.96**	**0.08**	**5.98**	**3.04**	**0.49**	**<LOD**	**71.01**
		std	256.30	0.02	2.73	0.87	0.40		5.28
Food			**Pb (µg/kg)**						
Tomato (Not A) 2h 70°C	I	**Av**	**598.28**						
		std	74.62						
	II	**Av**	**99.42**						
		std	11.54						
	III	**Av**	**93.29**						
		std	6.28						
Tomato (A) 2h 70°C	I	**Av**	**682.43**						
		std	25.24						
	II	**Av**	**112.75**						
		std	26.86						
	III	**Av**	**125.05**						
		std	44.27						
Tomato (A) 6h boiling	I	**Av**	**1116.05**						
		std	318.01						
	II	**Av**	**928.99**						
		std	232.38						
	III	**Av**	**612.50**						
		std	151.42						

(not acidified and acidified)

Table 2 - Metals leached from sample C14 into acetic acid 4%, citric acid 0.5% and Tomato sauce

Sample	**C14**										
Simulant			**Pb (µg/kg)**	**Cd (µg/kg)**	**Mn (µg/kg)**	**Ti (µg/kg)**	**Cu (µg/kg)**	**Li (µg/kg)**	**Al (µg/kg)**	**Cr (µg/kg)**	**Ba (µg/kg)**
AA4%	I	**Av**	**27.02**	**0.06**	**368.07**	**13.04**	**18.02**	**98.26**	**17928.72**	**16.46**	**271.70**
		std	6.63	0.02	147.63	6.93	2.73	16.89	6635.67	4.61	113.34
	II	**Av**	**15.35**	**0.05**	**415.53**	**6.87**	**11.69**	**76.70**	-	**8.52**	**285.53**
		std	2.87	0.01	73.16	2.29	1.43	15.15	-	3.21	118.57
	III	**Av**	**14.95**	**0.05**	**498.93**	**7.31**	**13.06**	**82.60**	**10714.46**	**7.29**	**555.75**
		std	3.75	0.04	172.65	2.43	4.87	27.43	3152.58	3.62	313.01
CA0.5%	I	**Av**	**88.73**	**0.07**	**109.42**	**58.69**	**18.98**	**89.47**	**8117.20**	**36.08**	**621.34**
		std	68.85	0.01	22.56	12.00	8.37	16.31	1896.45	39.09	211.31
	II	**Av**	**70.99**	**0.04**	**120.87**	**54.91**	**14.14**	**65.39**	-	**34.13**	**481.43**
		std	75.82	0.03	40.75	23.08	8.34	23.30	-	43.14	294.29
	III	**Av**	**68.81**	**0.07**	**185.78**	**73.42**	**17.31**	**103.93**	**11769.68**	**34.10**	**518.80**
		std	66.65	0.03	72.41	19.17	6.64	25.07	3898.74	39.51	255.85
Food			**Pb (µg/kg)**								
Tomato 2h 70°C	I	**Av**	**10.31**								
		std	10.00								
	II	**Av**	**<LOD**								
		std									
	III	**Av**	**<LOD**								
		std									

Table 3 - Metals leached from enamelled sample 113C02 into acetic acid 4% and citric acid 0.5%

Sample	113C02 (BE)										
Simulant			**Li (µg/kg)**	**Al (µg/kg)**	**V (µg/kg)**	**Cr (µg/kg)**	**Fe (µg/kg)**	**Zn (µg/kg)**	**Sb (µg/kg)**	**Ba (µg/kg)**	**Pb (µg/kg)**
AA4%	I	**Av**	**13.14**	**465.95**	**1.09**	**0.39**	**19.91**	**1.67**	**2.57**	**44.62**	**1.31**
		std	0.04	65.69	-	-	2.90	1.64	0.09	10.66	1.26
	II	**Av**	**5.47**	**164.70**	**2.25**	**0.35**	**159.07**	**8.73**	**5.54**	**12.86**	**5.31**
		std	1.03	30.83	0.23	-	189.26	3.97	0.17	4.87	7.01
	III	**Av**	**4.04**	**144.55**	**0.55**	**0.02**	**152.65**	**3.33**	**3.97**	**6.56**	**0.69**
		std	0.13	11.95	0.03	-	201.88	2.22	1.89	1.27	-
CA0.5%	I	**Av**	**31.65**	**1103.80**	**1.85**	**0.17**	**55.72**	**4.03**	**5.33**	**40.44**	**10.45**
		std	6.39	513.64	-	-	28.16	1.44	2.85	37.79	2.77
	II	**Av**	**5.49**	**152.55**	**1.25**	**0.71**	**10.69**	**0.92**	**3.73**	**7.03**	**2.05**
		std	0.16	4.03	0.11	-	3.98	0.21	0.82	8.34	0.01
	III	**Av**	**4.19**	**204.05**	**0.53**	**0.20**	**11.46**	**2.65**	**2.74**	**3.65**	**0.61**
		std	1.77	92.70	0.21	-	3.59	3.32	-	3.11	0.48

Table 4 - Metals leached from enamelled sample 213C07 into acetic acid 4% and citric acid 0.5%

Sample	213C07 (DE)									
Simulant			**Li (µg/kg)**	**Al (µg/kg)**	**Co (µg/kg)**	**Mo (µg/kg)**	**Cr (µg/kg)**	**Mn (µg/kg)**	**Fe (µg/kg)**	**Ag (µg/kg)**
AA4%	I	**Av**	**0.08**	**50.36**	**7.63**	**0.23**	**0.78**	**1.15**	**5.14**	**2.87**
		std	0.04	0.69	1.01	0.07	0.10	0.07	-	1.34
	II	**Av**	**0.02**	**43.45**	**1.14**	**0.16**	**0.76**	**1.32**	**20.30**	**2.22**
		std	0.01	7.63	0.26	0.01	0.27	0.44	1.11	0.52
	III	**Av**	**0.03**	**36.82**	**0.62**	**0.22**	**0.93**	**1.50**	**10.62**	**4.14**
		std	0.01	1.73	0.11	0.02	0.26	0.62	0.66	0.87
CA0.5%	I	**Av**	**0.06**	**30.91**	**12.02**	**0.24**	**0.28**	**0.61**	**4.04**	**3.51**
		std	0.01	5.68	2.29	0.02	0.11	0.02	-	0.81
	II	**Av**	**0.02**	**-**	**1.19**	**0.37**	**0.37**	**0.97**	**9.21**	**2.83**
		std	0.01	-	0.08	0.01	0.21	0.20	6.52	0.49
	III	**Av**	**0.02**	**-**	**0.86**	**0.24**	**0.35**	**0.76**	**3.20**	**3.85**
		std	0.01	-	0.22	0.01	0.12	0.09	2.76	1.72
Simulant			**Sn (µg/kg)**	**Zn (µg/kg)**	**Sb (µg/kg)**	**Ba (µg/kg)**	**Pb (µg/kg)**			
AA4%	I	**Av**	**0.81**	**4.51**	**0.10**	**0.93**	**0.58**			
		std	0.05	1.25	0.02	0.25	0.16			
	II	**Av**	**0.51**	**4.14**	**0.056**	**1.86**	**1.04**			
		std	0.09	2.14	0.003	1.99	1.63			
	III	**Av**	**0.38**	**3.80**	**0.07**	**1.35**	**0.31**			
		std	0.04	0.41	0.01	0.72	0.20			
CA0.5%	I	**Av**	**2.85**	**4.42**	**0.16**	**2.89**	**0.40**			
		std	0.38	0.70	0.02	0.40	0.14			
	II	**Av**	**0.68**	**4.48**	**0.107**	**2.96**	**0.32**			
		std	0.02	1.11	0.001	0.40	0.21			
	III	**Av**	**0.59**	**4.50**	**0.108**	**2.75**	**0.19**			
		std	0.05	0.72	0.001	0.42	0.22			

Table 5 - Metals leached from sample 213C09 into acetic acid 4% and citric acid 0.5%

Sample		**213C09 (UK)**										
Simulant			**Mg (µg/kg)**	**Al (µg/kg)**	**Co (µg/kg)**	**Cu (µg/kg)**	**V (µg/kg)**	**Zn (µg/kg)**	**As (µg/kg)**	**Sr (µg/kg)**	**Zr (µg/kg)**	**Sn (µg/kg)**
AA4%	I	**Av**	**7.26**	**29.29**	**8.90**	**2.88**	**0.16**	**5.42**	**< LOD**	**0.18**	**0.11**	**1.28**
		std	1.10	7.23	0.36	0.70	-	4.70	-	0.02	0.02	0.03
	II	**Av**	**4.59**	**< LOD**	**0.76**	**2.42**	**< LOD**	**5.42**	**< LOD**	**0.13**	**0.02**	**0.38**
		std	-	-	0.09	0.59	-	4.66	-	0.03	0.00	0.02
	III	**Av**	**5.09**	**4.55**	**0.39**	**1.97**	**< LOD**	**2.47**	**< LOD**	**0.11**	**0.02**	**0.32**
		std	-	0.00	0.12	0.62	-	0.94	-	0.03	0.01	-
CA0.5%	I	**Av**	**34.42**	**49.21**	**46.92**	**7.92**	**7.07**	**16.50**	**7.97**	**0.47**	**0.42**	**9.95**
		std	47.60	57.24	74.60	9.86	-	19.51	-	0.60	0.59	15.84
	II	**Av**	**8.46**	**11.33**	**4.52**	**0.88**	**0.89**	**11.20**	**0.97**	**0.08**	**0.06**	**1.16**
		std	5.60	9.17	7.25	0.73	-	6.99	-	0.05	0.06	1.23
	III	**Av**	**< LOD**	**6.98**	**1.79**	**0.40**	**0.31**	**5.67**	**0.37**	**0.07**	**0.05**	**0.80**
		std	NA	-	2.19	0.22	-	4.47	-	0.03	0.01	0.67

Table 6 - Metals leached from sample 413C06 into acetic acid 4%

Sample	**413C06 (DE)**								
Simulant			**Li (µg/kg)**	**Al (µg/kg)**	**Mo (µg/kg)**	**Cr (µg/kg)**	**Mn (µg/kg)**	**Zn (µg/kg)**	**Ag (µg/kg)**
AA4%	I	**Av**	**0.12**	**44.17**	**0.17**	**0.85**	**2.45**	**7.18**	**2.70**
		std	0.01	1.30	0.02	0.30	2.39	0.57	0.88
	II	**Av**	**0.21**	**44.71**	**18.91**	**4.18**	**4.75**	**96.32**	**272.80**
		std	0.16	5.63	32.46	4.17	3.69	92.24	357.22
	III	**Av**	**0.369**	**41.64**	**0.24**	**10.89**	**28.16**	**167.55**	**49.32**
		std	0.61	4.84	0.11	17.47	46.40	281.70	70.80
CA0.5%	I	**Av**	**0.25**	**12.12**	**0.23**	**0.22**	**0.59**	**7.88**	**3.47**
		std	0.08	0.77	0.01	0.11	0.03	0.21	1.30
	II	**Av**	**0.04**	**11.79**	**0.32**	**0.40**	**0.86**	**6.57**	**4.52**
		std	0.01	-	0.08	0.10	0.31	1.24	3.64
	III	**Av**	**0.04**	**-**	**0.28**	**.0.29**	**0.69**	**5.13**	**6.49**
		std	0.01	-	0.07	0.02	0.02	0.30	4.42
Simulant			**As (µg/kg)**	**Sn (µg/kg)**	**Cd (µg/kg)**	**Sb (µg/kg)**	**Ba (µg/kg)**	**Pb (µg/kg)**	
AA4%	I	**Av**	**0.06**	**0.50**	**0.07**	**0.07**	**5.63**	**0.80**	
		std	0.06	0.07	0.02	0.01	1.55	0.61	
	II	**Av**	**4.83**	**39.94**	**42.01**	**0.06**	**4.03**	**9.08**	
		std	8.32	35.90	19.76	0.01	3.61	7.72	
	III	**Av**	**17.34**	**33.67**	**41.93**	**6.84**	**1.92**	**4.10**	
		std	30.00	57.89	59.29	11.71	0.88	5.85	
CA0.5%	I	**Av**	**0.03**	**1.42**	**0.13**	**0.12**	**9.42**	**0.38**	
		std	0.01	0.11	0.08	0.001	1.22	0.34	
	II	**Av**	**0.02**	**0.91**	**0.05**	**0.11**	**5.37**	**0.57**	
		std	0.01	0.12	0.03	0.01	1.49	0.28	
	III	**Av**	**0.031**	**0.94**	**0.03**	**0.12**	**3.73**	**0.10**	
		std	0.002	0.06	0.02	0.01	0.44	0.06	

Table 7 - Metals leached from sample 416C28 into acetic acid 4% and tomato sauce

Sample	416C28									
Simulant			**Al (µg/kg)**	**Fe (µg/kg)**	**Mn (µg/kg)**	**Cu (µg/kg)**	**Zn (µg/kg)**	**Cd (µg/kg)**	**Ba (µg/kg)**	**Pb (µg/kg)**
AA4%	I	**Av**	**282.62**	**21.28**	**3.28**	**4.75**	**84.88**	**308.98**	**5.76**	**1029.87**
		std	83.10	2.67	0.75	6.16	35.11	119.35	1.16	361.51
	II	**Av**	**101.76**	**8.72**	**0.86**	**0.82**	**25.78**	**92.81**	**1.80**	**319.29**
		std	44.42	1.54	0.25	0.34	8.83	35.42	0.67	134.00
	III	**Av**	**83.78**	**5.73**	**0.52**	**0.50**	**23.30**	**75.16**	**1.07**	**254.65**
		std	19.74	1.62	0.18	0.31	3.30	13.38	0.24	48.19
Food			**Cd (µg/kg)**	**Pb (µg/kg)**						
Tomato 6 h boiling	I	**Av**	**731.48**	**3017.41**						
		std	201.55	874.28						
	II	**Av**	**528.17**	**2166.39**						
		std	158.80	750.95						
	III	**Av**	**447.89**	**1717.84**						
		std	146.03	636.14						

Table 8 - Metals leached from sample 416C29 into acetic acid 4%

Sample	416C29								
Simulant			**Al (µg/kg)**	**Fe (µg/kg)**	**Co (µg/kg)**	**Cu (µg/kg)**	**Cd (µg/kg)**	**Ba (µg/kg)**	**Pb (µg/kg)**
AA4%	I	**Av**	**12.74**	**4.71**	**0.47**	**0.73**	**<LOD**	**0.12**	**0.61**
		std	11.80	5.15	0.08	0.29		0.04	0.09
	II	**Av**	**1.64**	**<LOD**	**0.09**	**0.37**	**<LOD**	**0.15**	**<LOD**
		std	0.62		0.01	0.29		0.18	
	III	**Av**	-	-	-	-	-	-	-
		std							

Table 9 - Metals leached from sample 416C30 into acetic acid 4%

Sample	416C30								
Simulant			**Al (µg/kg)**	**Fe (µg/kg)**	**Co (µg/kg)**	**Cu (µg/kg)**	**Cd (µg/kg)**	**Ba (µg/kg)**	**Pb (µg/kg)**
AA4%	I	**Av**	**58.57**	**4.48**	**67.06**	**1.17**	**0.21**	**5.99**	**83.90**
		std	10.47	1.11	13.11	1.11	0.08	1.54	23.76
	II	**Av**	**8.67**	**<LOD**	**6.82**	**2.75**	**<LOD**	**0.85**	**9.39**
		std	2.64		1.12	4.80		0.18	1.81
	III	**Av**	**6.39**	**<LOD**	**5.30**	**0.27**	**<LOD**	**0.79**	**7.06**
		std	1.63		0.53	0.27		0.28	1.40
Food			**Co (µg/kg)**	**Pb (µg/kg)**					
Tomato 6h boiling	I	**Av**	**92.98**	**97.04**					
		std	10.12	14.28					
	II	**Av**	**38.72**	**38.90**					
		std	2.83	5.03					
	III	**Av**	**30.38**	**31.51**					
		std	1.62	0.70					

Table 10 - Metals leached from sample 416C31 into acetic acid 4%

Sample	416C31								
Simulant			Al (µg/kg)	Fe (µg/kg)	Cu (µg/kg)	Zn (µg/kg)	Cd (µg/kg)	Ba (µg/kg)	Pb (µg/kg)
AA4%	I	Av	31.25	17.69	2.38	20.40	0.07	1.78	17.41
		std	1.27	29.60	3.60	31.34	0.02	2.98	4.21
	II	Av	9.44	<LOD	5.89	9.80	<LOD	0.16	2.94
		std	2.34		9.14	10.38		0.04	1.50
	III	Av	6.22	<LOD	0.24	5.05	<LOD	0.31	1.63
		std	2.16		0.05	1.39		0.28	0.37

Table 11 - Metals leached from sample 615C13 into acetic acid 4%

Sample	615C13											
Simulant			Al (µg/kg)	V (µg/kg)	Cr (µg/kg)	Fe (µg/kg)	Co (µg/kg)	Cu (µg/kg)	Zn (µg/kg)	Cd (µg/kg)	Ba (µg/kg)	Pb (µg/kg)
AA4%	I	Av	83.16	0.20	1.97	24.60	154.76	0.81	291.32	6.75	2.48	216.58
		std	20.57	0.11	0.63	9.81	39.69	0.31	59.64	3.92	0.96	63.69
	II	Av	20.48	<LOD	0.45	5.09	16.49	<LOD	24.88	2.74	0.96	80.45
		std	12.93		0.12	3.54	16.55		26.51	2.34	0.57	38.80
	III	Av	19.42	<LOD	0.44	4.70	17.09	<LOD	26.33	2.80	1.03	78.02
		std	6.15		0.26	3.30	9.90		15.42	1.52	0.41	30.43

Table 12 - Metals leached from sample 615C15 into acetic acid 4%

Sample	615C15											
Simulant			Al (µg/kg)	V (µg/kg)	Cr (µg/kg)	Fe (µg/kg)	Co (µg/kg)	Cu (µg/kg)	Zn (µg/kg)	Cd (µg/kg)	Ba (µg/kg)	Pb (µg/kg)
AA4%	I	Av	65.79	0.78	0.63	27.61	2196.10	5.38	3578.33	0.57	0.92	230.90
		std	15.63	0.29	0.21	1.07	118.06	0.73	202.81	0.04	0.59	42.97
	II	Av	8.40	0.21	0.33	3.74	98.63	<LOD	157.63	0.05	<LOD	41.39
		std	4.84	0.13	0.08	2.69	29.56		47.68	0.02		10.99
	III	Av	8.54	0.24	1.49	22.59	91.81	<LOD	148.04	0.04	0.13	39.28
		std	5.43	0.27	2.42	32.79	112.57		182.44	0.04	0.08	21.14

Table 13 - Metals leached from sample 615C17 into acetic acid 4%

Sample	615C17											
Simulant			Al (µg/kg)	V (µg/kg)	Cr (µg/kg)	Fe (µg/kg)	Co (µg/kg)	Cu (µg/kg)	Zn (µg/kg)	Cd (µg/kg)	Ba (µg/kg)	Pb (µg/kg)
AA4%	I	Av	379.95	0.69	0.76	26.62	612.90	2.17	411.98	0.02	3.48	483.86
		std	55.55	0.06	0.29	8.61	92.07	1.28	60.39	0.01	1.25	55.82
	II	Av	108.57	0.18	0.33	9.33	174.26	<LOD	115.26	<LOD	0.61	136.36
		std	22.49	0.08	0.19	2.49	40.03		24.52		0.15	22.04
	III	Av	64.61	<LOD	0.14	7.48	108.65	<LOD	68.52	<LOD	0.48	87.19
		std	12.21		0.05	6.47	23.62		13.10		0.19	12.12

Table 14 - Metals leached from enamelled 113C01 and 113C03 samples into acetic acid 4% and citric acid 0.5%

Sample			Li (µg/kg)		Ti (µg/kg)		V (µg/kg)		Cr (µg/kg)		Mn (µg/kg)		Fe (µg/kg)		Co (µg/kg)		Ni (µg/kg)	
			AA4%	CA0.5%	AA4%	CA0.5%	AA4%	CA0.5%	AA4%	CA0.5%	AA4%	CA0.5%	AA4%	CA0.5%	AA4%	CA0.5%	AA4%	CA0.5%
113C01	I	Av	**135.15**	**171.52**	**257.38**	**1563.87**	**1.98**	**2.46**	**0.39**	**0.82**	**0.84**	**1.23**	**183.06**	**204.31**	**7.02**	**11.04**	**0.51**	**0.52**
		std	15.41	16.42	23.68	257.09	0.11	0.45	0.09	0.43	0.17	0.40	10.15	49.88	0.74	1.38	0.10	0.06
	II	Av	**21.48**	**76.55**	**250.73**	**819.43**	**0.34**	**0.99**	**0.06**	**<LOD**	**0.26**	**0.43**	**35.81**	**73.97**	**2.66**	**5.16**	**<LOD**	**0.22**
		std	3.01	17.97	8.99	64.21	0.08	0.17	0.04		0.33	0.03	7.46	9.31	2.90	0.44		0.02
	III	Av	**17.90**	**38.57**	**188.89**	**425.46**	**0.27**	**0.39**	**<LOD**	**<LOD**	**<LOD**	**0.36**	**23.04**	**33.20**	**0.99**	**2.67**	**<LOD**	**0.20**
		std	2.28	8.61	17.36	7.03	0.04	0.08				0.29	3.70	6.31	0.11	0.15		0.14
113C03	I	Av	**94.83**	**558.25**	**103.80**	**937.62**	**2.22**	**8.60**	**11.25**	**56.32**	**291.89**	**1771.63**	**202.01**	**741.50**	**53.26**	**365.89**	**69.32**	**333.26**
		std	25.15	296.09	19.08	435.40	0.44	3.14	2.99	30.18	72.70	924.48	55.55	253.18	14.06	181.55	15.98	128.09
	II	Av	**14.00**	**223.43**	**54.25**	**618.32**	**0.41**	**4.30**	**2.44**	**29.67**	**47.71**	**802.53**	**50.74**	**359.30**	**8.61**	**156.51**	**11.22**	**160.86**
		std	1.14	27.30	14.39	99.32	0.07	0.91	0.39	1.46	4.87	109.31	18.98	51.59	0.68	25.88	0.98	21.48
	III	Av	**13.50**	**84.30**	**40.66**	**299.78**	**0.36**	**1.67**	**2.08**	**12.64**	**47.30**	**324.55**	**48.50**	**145.08**	**8.65**	**65.78**	**11.37**	**69.72**
		std	0.94	20.75	5.70	62.56	0.06	0.37	0.20	2.09	3.80	77.35	14.39	26.56	0.73	17.55	0.73	13.09

Sample			Zn (µg/kg)		Cu (µg/kg)		Zr (µg/kg)		Mo (µg/kg)		Cd (µg/kg)		Sn (µg/kg)		Ba (µg/kg)		Pb (µg/kg)	
			AA4%	CA0.5%	AA4%	CA0.5%	AA4%	CA0.5%	AA4%	CA0.5%	AA4%	CA0.5%	AA4%	CA0.5%	AA4%	CA0.5%	AA4%	CA0.5%
113C01	I	Av	**<LOD**	**9.12**	**23.97**	**28.34**	**4.17**	**358.39**	**0.38**	**0.03**	**0.07**	**0.53**	**<LOD**	**<LOD**	**6.69**	**8.61**	**0.99**	**0.94**
		std		3.18	4.35	12.70	0.25	95.39	0.02	0.13	0.02	0.52			0.90	2.18	0.33	0.34
	II	Av	**<LOD**	**<LOD**	**3.36**	**10.65**	**<LOD**	**231.98**	**0.08**	**<LOD**	**0.010**	**0.21**	**1.14**	**<LOD**	**1.28**	**3.55**	**<LOD**	**<LOD**
		std			0.52	5.50		22.53	0.01		0.003	0.18	0.67		0.22	0.78		
	III	Av	**<LOD**	**<LOD**	**2.56**	**4.83**	**<LOD**	**201.21**	**0.048**	**<LOD**	**0.006**	**0.13**	**1.28**	**<LOD**	**1.09**	**2.11**	**<LOD**	**<LOD**
		std			0.49	2.50		14.00	0.003		0.002	0.09	0.73		0.39	0.43		
113C03	I	Av	**<LOD**	**39.55**	**135.63**	**869.94**	**<LOD**	**7.88**	**1.95**	**4.83**	**1.45**	**1.92**	**0.55**	**<LOD**	**340.26**	**1578.37**	**<LOD**	**2.29**
		std		59.01	37.32	547.88		3.69	0.27	0.86	1.55	0.89	0.33		42.58	89.54		0.33
	II	Av	**<LOD**	**8.82**	**19.12**	**315.04**	**<LOD**	**5.55**	**0.42**	**2.75**	**0.20**	**0.53**	**<LOD**	**<LOD**	**60.02**	**884.46**	**<LOD**	**0.91**
		std		2.45	2.31	11.17		1.22	0.12	0.72	0.20	0.21			5.96	77.47		0.48
	III	Av	**<LOD**	**<LOD**	**18.22**	**124.37**	**<LOD**	**5.15**	**0.33**	**0.84**	**0.17**	**0.17**	**0.89**	**<LOD**	**55.76**	**348.70**	**<LOD**	**<LOD**
		std			1.40	19.86		0.65	0.04	0.35	0.16	0.08	0.71		5.74	67.27		

Table 15 - Metals leached from sample 615C18 into acetic acid 4%

Sample	615C18											
Simulant			**Al (µg/kg)**	**V (µg/kg)**	**Cr (µg/kg)**	**Fe (µg/kg)**	**Co (µg/kg)**	**Cu (µg/kg)**	**Zn (µg/kg)**	**Cd (µg/kg)**	**Ba (µg/kg)**	**Pb (µg/kg)**
AA4%	I	**Av**	**176.72**	**1.38**	**0.40**	**7.78**	**316.83**	**0.49**	**210.52**	**0.06**	**1.90**	**317.86**
		std	6.52	0.83	0.14	4.86	12.95	0.08	12.45	0.03	0.10	24.80
	II	**Av**	**45.97**	**0.17**	**0.13**	**<LOD**	**110.30**	**<LOD**	**55.37**	**<LOD**	**0.39**	**87.41**
		std	4.87	0.19	0.04		10.73		5.56		0.05	9.64
	III	**Av**	**27.48**	**<LOD**	**0.13**	**<LOD**	**80.86**	**<LOD**	**36.51**	**<LOD**	**1.07**	**55.04**
		std	1.03		0.08		3.94		3.48		1.65	4.40

Table 16 - Metals leached from sample 615C19 into acetic acid 4%

Sample	615C19											
Simulant			**Al (µg/kg)**	**V (µg/kg)**	**Cr (µg/kg)**	**Fe (µg/kg)**	**Co (µg/kg)**	**Cu (µg/kg)**	**Zn (µg/kg)**	**Cd (µg/kg)**	**Ba (µg/kg)**	**Pb (µg/kg)**
AA4%	I	**Av**	**1099.57**	**0.56**	**0.46**	**116.21**	**617.66**	**7.70**	**794.41**	**0.39**	**228.73**	**707.63**
		std	117.67	0.14	0.07	3.47	74.67	1.31	102.14	0.15	80.50	66.79
	II	**Av**	**396.82**	**0.28**	**0.07**	**38.59**	**224.75**	**1.23**	**284.74**	**0.14**	**69.49**	**239.36**
		std	51.46	0.13	0.08	7.49	32.16	0.48	39.88	0.05	26.96	26.65
	III	**Av**	**326.65**	**<LOD**	**0.13**	**34.75**	**183.96**	**0.99**	**238.21**	**0.11**	**53.17**	**195.15**
		std	70.57		0.10	6.67	44.92	0.61	60.47	0.04	16.55	46.59

Table 17 - Metals leached from sample 713C01 into acetic acid 4%, citric acid 0.5% and Tomato sauce

Sanple	713C01									
Simulant			**Al (µg/kg)**	**V (µg/kg)**	**Cr (µgkg)**	**Mn (µg/kg)**	**As (µg/kg)**	**Pb (µg/kg)**	**Cd (µg/kg)**	**Ba (µg/kg)**
AA4%	I	**Av**	**288.64**	**1.49**	**0.31**	**8.51**	**0.56**	**347.50**	**0.53**	**8.11**
		std	112.93	0.44	0.07	3.49	0.16	23.76	0.04	1.46
	II	**Av**	**422.35**	**3.50**	**0.39**	**13.53**	**1.87**	**75.96**	**0.31**	**14.00**
		std	63.91	0.72	0.07	1.93	0.42	7.49	0.09	3.65
	III	**Av**	**246.44**	**2.30**	**0.15**	**15.63**	**1.31**	**41.40**	**0.29**	**13.63**
		std	41.18	0.45	0.04	2.47	0.39	3.79	0.06	8.24
CA0.5%	I	**Av**	**114.90**	**0.15**	**0.24**	**1.33**	**0.13**	**679.77**	**0.71**	**11.67**
		std	59.86	0.05	0.05	0.21	0.04	37.69	0.07	1.35
	II	**Av**	**200.45**	**3.99**	**0.17**	**6.06**	**1.78**	**388.64**	**0.50**	**9.94**
		std	30.42	1.33	0.05	1.51	0.64	14.27	0.06	2.92
	III	**Av**	**613.93**	**11.85**	**0.55**	**12.59**	**4.00**	**325.43**	**0.59**	**15.54**
		std	412.87	3.79	0.29	3.43	0.91	33.36	0.06	3.14
Food			**Pb (µg/kg)**							
Tomato 2h 70°C	I	**Av**	**384.23**							
		std	45.98							
	II	**Av**	**75.96**							
		std	7.49							
	III	**Av**	**41.40**							
		std	3.79							

Table 18 - Metals leached from sample 713C02 into acetic acid 4%, citric acid 0.5% and Tomato sauce (not acidified and acidified)

Sample	713C02										
Simulant			**Al (µg/kg)**	**V (µg/kg)**	**Cr (µg/kg)**	**Mn (µg/kg)**	**Ni (µg/kg)**	**Cu (µg/kg)**	**Zn (µg/kg)**	**As (µg/kg)**
AA4%	I	**Av**	**40197.50**	**32.93**	**21.83**	**525.88**	**9.24**	**19.00**	**95.36**	**17.95**
		std	12113.69	11.57	11.16	260.96	4.87	12.69	48.65	6.82
	II	**Av**	**18304.82**	**10.69**	**11.10**	**328.69**	**4.19**	**7.47**	**66.39**	**8.14**
		std	4207.92	3.60	4.41	135.47	1.89	4.37	30.48	3.18
	III	**Av**	**10963.54**	**7.85**	**4.72**	**265.44**	**2.90**	**5.39**	**40.10**	**9.33**
		std	4472.74	3.15	1.94	103.40	1.41	2.97	17.96	5.02
CA0.5%	I	**Av**	**1243.49**	**23.29**	**1.55**	**31.16**	**0.53**	**2.45**	**<LOD**	**6.11**
		std	343.86	4.61	0.48	11.18	0.20	1.75		1.04
	II	**Av**	**1857.59**	**47.49**	**3.43**	**51.87**	**1.25**	**3.88**		**9.61**
		std	420.84	7.62	0.83	17.35	0.49	2.61		0.75
	III	**Av**	**2449.27**	**56.13**	**5.00**	**62.51**	**0.67**	**1.11**		**10.33**
		std	510.83	6.10	1.03	19.91	0.14	0.33		0.56

Simulant			**Cd (µg/kg)**	**Ba (µg/kg)**	**Pb (µg/kg)**
AA4%	I	**Av**	**0.10**	**65.50**	**764.39**
		std	0.05	31.70	207.64
	II	**Av**	**0.04**	**51.82**	**246.08**
		std	0.03	21.88	74.16
	III	**Av**	**0.03**	**55.20**	**152.33**
		std	0.04	27.44	54.01
CA0.5%	I	**Av**	**<LOD**	**19.00**	**1250.93**
		std		5.30	285.12
	II	**Av**		**20.22**	**566.43**
		std		6.27	210.54
	III	**Av**		**16.47**	**430.55**
		std		3.49	150.54
Food			**Pb (µg/kg)**		**Pb (µg/kg)**
Tomato (Not A) 2h 70°C	I	**Av**	**242.04**	**Tomato (A) 2h 70°C**	**304.72**
		std	11.35		78.22
	II	**Av**	**112.70**		**106.01**
		std	21.08		12.52
	III	**Av**	**101.12**		**114.11**
		std	29.87		19.79

Table 19 - Metals leached from sample 713C03 into acetic acid 4% and citric acid 0.5%

Sample	713C03 (BE)					
Simulant			**Al (µg/kg)**	**Co (µg/kg)**	**Ba (µg/kg)**	**Pb (µg/kg)**
AA4%	I	**Av**	**15.80**	**1.86**	**0.65**	**211.58**
		std	1.07	0.51	0.14	49.53
	II	**Av**	**6.14**	**0.28**	**0.17**	**29.46**
		std	0.30	0.09	0.03	6.13
	III	**Av**	**-**	**0.18**	**<LOD**	**13.91**
		std	-	0.04	-	2.60
CA0.5%	I	**Av**	**44.58**	**3.57**	**<LOD**	**464.00**
		std	2.19	0.51	-	56.25
	II	**Av**	**44.36**	**15.14**	**<LOD**	**128.81**
		std	37.00	21.68	-	18.59
	III	**Av**	**39.88**	**10.91**	**0.91**	**42.81**
		std	19.92	11.22	-	8.33

Table 20 - Metals leached from sample 713C04 into acetic acid 4% and citric acid 0.5%

Sample	713C04 (BE)										
Simulant			Li (µg/kg)	Al (µg/kg)	V (µg/kg)	Mn (µg/kg)	Fe (µg/kg)	As (µg/kg)	Ni (µg/kg)	Ba (µg/kg)	Pb (µg/kg)
AA4%	I	**Av**	**1.15**	**5229.22**	**1.64**	**3.65**	**147.74**	**0.39**	**2.01**	**6.93**	**243.72**
		std	0.59	1167.64	0.63	1.17	86.28	0.10	0.35	5.14	140.85
	II	**Av**	**4.49**	**2086.92**	**1.43**	**8.51**	**72.02**	**0.41**	**4.23**	**7.39**	**78.48**
		std	2.91	1132.75	0.80	3.24	39.63	0.18	1.59	4.89	20.77
	III	**Av**	**3.94**	**2643.62**	**1.44**	**11.38**	**167.11**	**0.63**	**4.59**	**7.02**	**53.79**
		std	1.78	1337.52	0.74	4.64	104.13	0.29	1.73	4.86	5.50
CA0.5%	I	**Av**	**1.47**	**103.36**	**10.46**	**2.45**	**60.68**	**3.04**	**1.25**	**3.76**	**808.59**
		std	0.21	37.76	9.91	1.52	25.91	2.71	0.31	1.42	234.97
	II	**Av**	**11.95**	**218.31**	**32.06**	**6.89**	**107.49**	**3.56**	**2.48**	**4.98**	**307.77**
		std	2.74	60.79	10.65	3.56	45.64	0.36	0.97	-	74.19
	III	**Av**	**16.66**	**239.74**	**20.07**	**3.09**	**60.27**	**2.81**	**1.02**	**1.88**	**152.47**
		std	4.09	35.41	6.55	0.41	20.51	0.46	0.22	-	46.83

Table 21 - Metals leached from sample 713C05 into acetic acid 4% and citric acid 0.5%

Sample	713C05 (BE)											
Simulant			Li (µg/kg)	Al (µg/kg)	V (µg/kg)	Mn (µg/kg)	Fe (µg/kg)	Ni (µg/kg)	Zn (µg/kg)	As (µg/kg)	Ba (µg/kg)	Pb (µg/kg)
AA4%	I	**Av**	**6.03**	**2839.44**	**1.45**	**13.19**	**74.64**	**3.66**	**1378.73**	**7.18**	**457.84**	**7.39**
		std	2.45	950.85	1.10	6.41	29.49	-	297.47	2.49	81.06	3.81
	II	**Av**	**5.32**	**2437.71**	**1.01**	**18.87**	**71.02**	**3.57**	**300.91**	**2.28**	**234.03**	**2.86**
		std	1.32	414.62	0.25	2.85	13.15	0.82	223.92	0.64	41.56	1.17
	III	**Av**	**2.77**	**1647.37**	**0.56**	**12.71**	**44.22**	**2.10**	**64.95**	**1.12**	**133.80**	**1.67**
		std	0.68	652.54	0.15	3.98	12.94	0.55	32.96	0.32	69.88	0.54
CA0.5%	I	**Av**	**5.96**	**5621.47**	**23.05**	**2.99**	**118.45**	**1.72**	**883.22**	**11.88**	**644.45**	**12.36**
		std	1.72	672.08	12.81	1.17	29.82	0.57	271.67	3.05	97.83	5.38
	II	**Av**	**3.25**	**2647.15**	**4.43**	**3.15**	**67.75**	**2.92**	**348.60**	**3.77**	**346.93**	**5.92**
		std	0.94	756.08	1.05	2.31	18.16	1.77	111.56	1.25	107.57	0.55
	III	**Av**	**2.57**	**908.74**	**4.59**	**4.97**	**37.18**	**0.94**	**142.08**	**2.82**	**132.22**	**2.90**
		std	0.90	91.46	2.52	3.54	4.98	0.28	19.81	0.66	13.68	0.85

Table 22 - Metals leached from sample 713C06 into acetic acid 4%, citric acid 0.5% and tomato sauce

Sample	713C06 (UK)											
Simulant			**Li (µg/kg)**	**Mg (µg/kg)**	**Al (µg/kg)**	**Ti (µg/kg)**	**V (µg/kg)**	**Mn (µg/kg)**	**Fe (µg/kg)**	**Co (µg/kg)**	**Ni (µg/kg)**	**Cu (µg/kg)**
AA4%	I	**Av**	**31.74**	**199.57**	**1440.67**	**2.10**	**21.14**	**47.82**	**66.60**	**0.48**	**1.63**	**6.77**
		std	30.17	150.11	725.30	-	22.26	38.12	48.85	0.41	1.43	2.06
	II	**Av**	**49.00**	**428.87**	**2464.99**	**<LOD**	**13.60**	**60.71**	**137.95**	**1.82**	**31.91**	**5.01**
		std	18.22	144.60	364.87	-	4.22	15.87	31.38	0.69	33.38	0.72
	III	**Av**	**51.38**	**424.56**	**2409.85**	**<LOD**	**11.15**	**65.27**	**152.06**	**2.54**	**30.70**	**2.99**
		std	7.35	83.57	98.11	-	3.48	15.20	7.30	0.98	50.47	0.64
CA0.5%	I	**Av**	**47.03**	**251.17**	**994.45**	**5.92**	**19.11**	**53.94**	**67.07**	**0.92**	**1.57**	**11.28**
		std	23.65	51.83	237.85	4.65	6.72	4.48	15.68	0.53	1.02	4.22
	II	**Av**	**36.86**	**289.45**	**1402.54**	**4.39**	**15.47**	**58.90**	**103.64**	**2.36**	**2.17**	**2.35**
		std	10.47	42.36	196.48	3.04	6.87	6.39	18.11	0.88	1.06	0.58
	III	**Av**	**34.29**	**347.36**	**1870.82**	**7.46**	**11.08**	**69.77**	**156.35**	**3.58**	**3.22**	**1.65**
		std	14.37	40.17	325.23	2.01	1.05	3.87	29.88	0.97	2.10	0.22
Simulant			**As (µg/kg)**	**Se (µg/kg)**	**Sr (µg/kg)**	**Zr (µg/kg)**	**Cd (µg/kg)**	**Mo (µg/kg)**	**Ba (µg/kg)**	**Pb (µg/kg)**	**Zn (µg/kg)**	
AA4%	I	**Av**	**2.45**	**0.42**	**4.92**	**0.82**	**0.39**	**2.57**	**26.39**	**1094.09**	**54.45**	
		std	1.99	0.25	2.92	0.70	0.28	2.85	21.20	571.85	35.93	
	II	**Av**	**2.21**	**0.45**	**10.63**	**0.55**	**0.55**	**1.25**	**44.98**	**1677.89**	**101.64**	
		std	0.81	0.18	2.45	0.47	0.19	0.82	19.91	546.52	20.32	
	III	**Av**	**1.89**	**0.46**	**11.31**	**0.39**	**0.52**	**0.76**	**35.16**	**1423.31**	**77.25**	
		std	0.74	0.11	0.46	0.25	0.25	0.22	13.02	358.80	43.11	
CA0.5%	I	**Av**	**1.84**	**0.32**	**6.10**	**1.37**	**0.43**	**4.38**	**33.26**	**1522.06**	**36.94**	
		std	0.50	0.16	1.29	0.19	0.12	0.70	12.06	621.51	14.66	
	II	**Av**	**1.57**	**0.28**	**9.37**	**1.29**	**0.41**	**3.43**	**45.82**	**2076.69**	**39.97**	
		std	0.40	0.05	0.81	0.48	0.08	1.21	11.01	389.43	19.15	
	III	**Av**	**1.21**	**0.33**	**11.38**	**1.39**	**0.42**	**2.60**	**45.13**	**2465.90**	**34.94**	
		std	0.16	0.10	0.72	0.24	0.07	0.73	7.48	331.31	8.32	
Food			**Pb (µg/kg)**	**Food**			**Pb (µg/kg)**					
Tomato 2h 70°C	I	**Av**	**59.05**	**Tomato (A) 6h boiling (EURL)**	I	**Av**	**489.57**					
		std	26.04			std	103.63					
	II	**Av**	**58.02**		II	**Av**	**329.74**					
		std	38.25			std	106.19					
	III	**Av**	**88.15**		III	**Av**	**311.14**					
		std	57.05			std	19.00					

Table 23 - Metals leached from sample 713C07 into acetic acid 4% and citric acid 0.5%

Sample	713C07											
Simulant			**Al (µg/kg)**	**V (µg/kg)**	**Cr (µg/kg)**	**Mn (µg/kg)**	**Ni (µg/kg)**	**Cu (µg/kg)**	**Zn (µg/kg)**	**As (µg/kg)**	**Ba (µg/kg)**	**Pb (µg/kg)**
AA4%	I	**Av**	**79.17**	**42.00**	**1.77**	**61.33**	**1.40**	**2.70**	**56.37**	**12.04**	**31.93**	**1.06**
		std	24.10	9.64	0.91	16.30	0.43	0.92	16.72	3.73	6.58	0.50
	II	**Av**	**66.74**	**28.43**	**1.63**	**96.26**	**1.31**	**2.14**	**38.89**	**13.38**	**54.91**	**<LOD**
		std	16.92	5.14	0.39	19.07	0.39	0.44	9.29	2.27	5.78	
	III	**Av**	**52.21**	**25.97**	**1.56**	**96.42**	**0.88**	**1.11**	**20.94**	**13.07**	**76.92**	**<LOD**
		std	5.06	2.31	0.26	3.76	0.09	0.14	1.37	1.23	2.72	
CA0.5%	I	**Av**	**61.17**	**6.49**	**0.31**	**7.83**	**0.21**	**1.25**	**<LOD**	**1.24**	**12.51**	**1.46**
		std	43.78	3.32	0.17	5.30	0.32	1.38		0.99	2.94	0.50
	II	**Av**	**91.22**	**5.67**	**0.49**	**15.72**	**0.22**	**1.28**	**<LOD**	**4.14**	**16.30**	**<LOD**
		std	46.78	3.65	0.11	6.11	0.19	0.18		1.14	3.66	
	III	**Av**	**107.91**	**7.54**	**0.28**	**16.93**	**0.16**	**<LOD**	**<LOD**	**4.67**	**21.20**	**<LOD**
		std	64.47	4.90	0.23	6.90	0.17			1.93	6.01	

Table 24 - Metals leached from sample 713C08 into acetic acid 4%, citric acid 0.5% and tomato sauce

Sample	**713C08 (DE)**											
Simulant			**Li (µg/kg)**	**Al (µg/kg)**	**V (µg/kg)**	**Ag (µg/kg)**	**Cu (µg/kg)**	**Mo (µg/kg)**	**Cr (µg/kg)**	**Mn (µg/kg)**	**Fe (µg/kg)**	**Co (µg/kg)**
AA4%	I	**Av**	**28.29**	**833.73**	**17.23**	**97.86**	**24.80**	**2.34**	**8.35**	**44.24**	**252.24**	**1.23**
		std	18.99	534.08	7.06	165.86	28.70	1.91	5.95	14.24	155.86	0.79
	II	**Av**	**34.26**	**741.48**	**13.30**	**3.15**	**2.22**	**2.49**	**8.55**	**73.36**	**88.51**	**1.13**
		std	10.55	376.34	0.68	0.86	0.42	0.78	4.37	19.16	50.58	0.55
	III	**Av**	**28.44**	**616.33**	**13.74**	**59.66**	**38.41**	**5.38**	**8.21**	**128.93**	**95.77**	**1.91**
		std	12.16	479.16	2.37	99.53	63.51	5.70	4.94	83.77	82.02	1.36
CA0.5%	I	**Av**	**2.29**	**157.72**	**0.96**	**3.29**	**4.59**	**0.30**	**1.07**	**4.20**	**125.88**	**0.24**
		std	1.66	138.62	0.49	1.43	6.12	0.05	0.87	2.18	110.20	0.27
	II	**Av**	**8.15**	**249.10**	**9.78**	**3.62**	**1.27**	**0.77**	**1.56**	**16.63**	**98.27**	**0.25**
		std	3.20	90.33	1.67	1.03	0.83	0.08	1.77	4.91	51.92	0.14
	III	**Av**	**28.61**	**370.60**	**25.47**	**3.84**	**2.24**	**2.87**	**2.49**	**22.29**	**128.62**	**0.27**
		std	10.21	123.49	1.79	1.91	-	0.59	1.65	3.920	57.39	0.15

Simulant			**Sn (µg/kg)**	**Zn (µg/kg)**	**Sb (µg/kg)**	**Cd (µg/kg)**	**As (µg/kg)**	**Ba (µg/kg)**	**Pb (µg/kg)**
AA4%	I	**Av**	**17.31**	**95.18**	**1.60**	**11.49**	**5.21**	**75.18**	**2973.80**
		std	29.72	56.93	2.43	19.28	0.97	49.64	2112.21
	II	**Av**	**0.16**	**20.54**	**0.10**	**0.10**	**3.89**	**203.88**	**830.45**
		std	0.02	6.62	0.03	0.01	0.58	132.13	243.82
	III	**Av**	**81.66**	**135.97**	**3.09**	**0.08**	**4.68**	**216.07**	**586.32**
		std	141.19	196.99	5.21	0.03	1.10	135.24	72.05
CA0.5%	I	**Av**	**0.42**	**71.39**	**0.32**	**0.28**	**0.54**	**136.27**	**3353.47**
		std	0.02	69.42	0.24	0.22	0.37	147.58	3096.53
	II	**Av**	**0.41**	**35.53**	**0.24**	**0.13**	**4.85**	**465.53**	**1164.11**
		std	0.02	20.82	0.10	0.06	0.16	693.35	764.51
	III	**Av**	**0.39**	**33.56**	**0.24**	**0.13**	**6.03**	**368.44**	**1659.80**
		std	0.01	17.81	0.09	0.06	0.40	543.59	872.05

Food			**Pb (µg/kg)**	**Food**			**Pb (µg/kg)**
Tomato 2h 70°C	I	**Av**	**434.72**	**Tomato 6h boiling (EURL)**	I	**Av**	**2686.73**
		std	165.38			std	553.95
	II	**Av**	**231.89**		II	**Av**	**1492.55**
		std	11.82			std	478.31
	III	**Av**	**172.46**		III	**Av**	**992.68**
		std	21.34			std	190.52

Table 25- Metals leached from sample 713C11 into acetic acid 4% and citric acid 0.5%

Sample	**713C11 (UK)**											
Simulant			**Li (µg/kg)**	**Mg (µg/kg)**	**Al (µg/kg)**	**Ti (µg/kg)**	**V (µg/kg)**	**Mn (µg/kg)**	**Fe (µg/kg)**	**Co (µg/kg)**	**Ni (µg/kg)**	**Cu (µg/kg)**
AA4%	I	**Av**	**1.82**	**3703.85**	**822.22**	**6.35**	**0.95**	**8.34**	**138.81**	**0.34**	**< LOD**	**2.77**
		std	1.00	4330.42	1193.13	4.08	0.89	8.65	129.40	0.38	-	0.58
	II	**Av**	**3.35**	**6761.87**	**1074.43**	**2.31**	**1.72**	**13.16**	**158.04**	**0.52**	**< LOD**	**1.57**
		std	1.53	6722.88	1508.63	-	1.72	11.91	165.22	0.53	-	0.19
	III	**Av**	**3.06**	**6657.94**	**655.03**	**3.15**	**1.29**	**12.72**	**120.70**	**0.53**	**< LOD**	**0.84**
		std	1.40	5245.92	679.35	-	0.71	7.44	81.55	0.36	-	0.53
CA0.5%	I	**Av**	**1.25**	**517.53**	**144.91**	**3.95**	**1.39**	**2.68**	**83.35**	**0.07**	**3.09**	**5.75**
		std	0.81	501.40	102.13	-	1.89	2.54	35.00	-	0.24	2.60
	II	**Av**	**3.24**	**1556.77**	**366.10**	**3.75**	**5.25**	**8.42**	**154.11**	**0.16**	**4.30**	**1.52**
		std	3.17	1239.93	228.82	2.14	5.94	6.64	121.08	0.10	1.88	0.97
	III	**Av**	**4.84**	**2284.50**	**904.76**	**3.37**	**5.19**	**8.71**	**209.71**	**0.18**	**< LOD**	**1.95**
		std	3.39	816.79	189.61	1.16	5.62	6.29	50.24	0.04	-	0.97

Simulant			**As (µg/kg)**	**Sr (µg/kg)**	**Zr (µg/kg)**	**Mo (µg/kg)**	**Ba (µg/kg)**	**Pb (µg/kg)**	**Zn (µg/kg)**
AA4%	I	**Av**	**0.76**	**7.31**	**0.50**	**0.15**	**10.58**	**466.36**	**6.70**
		std	0.68	7.23	0.14	0.12	1.73	196.89	3.25
	II	**Av**	**1.18**	**9.68**	**0.17**	**0.39**	**10.07**	**335.38**	**10.59**
		std	1.01	8.74	0.10	0.34	2.73	171.53	7.85
	III	**Av**	**1.11**	**8.80**	**0.15**	**0.27**	**9.70**	**335.19**	**8.60**
		std	0.57	5.85	0.13	0.21	1.10	174.77	2.28
CA0.5%	I	**Av**	**0.84**	**2.41**	**0.56**	**0.22**	**11.85**	**509.73**	**5.70**
		std	0.36	0.67	0.12	0.12	4.74	107.42	1.04
	II	**Av**	**1.65**	**3.55**	**0.26**	**0.77**	**19.11**	**430.30**	**5.31**
		std	1.06	1.84	0.08	0.85	14.71	161.58	1.67
	III	**Av**	**1.89**	**4.77**	**0.36**	**0.89**	**33.60**	**452.23**	**5.67**
		std	1.25	0.70	0.08	1.03	17.00	171.77	2.91

Table 26 - Metals leached from sample 713C12 into acetic acid 4% and citric acid 0.5%

Sample	**713C12 (BE)**									
Simulant			**Li (µg/kg)**	**Al (µg/kg)**	**V (µg/kg)**	**Mn (µg/kg)**	**Co (µg/kg)**	**As (µg/kg)**	**Ba (µg/kg)**	**Pb (µg/kg)**
AA4%	I	**Av**	**9.42**	**11041.89**	**20.44**	**21.47**	**0.13**	**1.01**	**43.38**	**489.84**
		std	6.17	16573.83	6.79	8.63	0.06	0.39	9.23	63.78
	II	**Av**	**12.53**	**1163.43**	**22.88**	**40.73**	**0.34**	**0.62**	**46.14**	**104.33**
		std	5.25	636.98	7.71	9.20	0.10	0.31	6.97	18.28
	III	**Av**	**13.69**	**687.01**	**25.03**	**52.98**	**0.49**	**0.64**	**56.06**	**106.38**
		std	19.58	408.56	29.50	46.57	0.34	0.86	16.89	15.10
CA0.5%	I	**Av**	**9.54**	**307.96**	**8.17**	**68.73**	**0.94**	**0.86**	**22.75**	**467.38**
		std	6.88	185.80	5.89	38.82	0.53	0.28	9.35	124.02
	II	**Av**	**13.43**	**428.51**	**2.87**	**83.41**	**1.31**	**0.47**	**41.45**	**461.11**
		std	8.70	144.55	1.08	25.50	0.41	0.22	14.90	106.77
	III	**Av**	**18.50**	**541.36**	**7.69**	**137.19**	**1.79**	**1.31**	**48.30**	**454.80**
		std	14.37	262.01	1.43	30.91	0.44	1.64	29.45	239.11

Table 27- Metals leached from sample 713C15 into acetic acid 4% and citric acid 0.5%

Sample	**713C15 (BE)**											
Simulant			**Li (µg/kg)**	**Al (µg/kg)**	**V (µg/kg)**	**Mn (µg/kg)**	**Fe (µg/kg)**	**Cu (µg/kg)**	**Zn (µg/kg)**	**Cd (µg/kg)**	**Ba (µg/kg)**	**Pb (µg/kg)**
AA4%	I	**Av**	**8.83**	**533.90**	**0.32**	**2.37**	**235.56**	**1020.61**	**15.47**	**0.67**	**102.81**	**11034.90**
		std	0.99	89.26	0.11	0.15	14.47	189.42	1.17	0.17	21.90	1421.39
	II	**Av**	**5.31**	**286.60**	**7.80**	**9.76**	**81.12**	**241.37**	**5.60**	**0.19**	**32.92**	**3222.33**
		std	5.47	220.14	13.23	18.01	12.48	61.38	0.68	0.04	7.39	788.95
	III	**Av**	**1.68**	**117.10**	<LOD	**0.54**	**58.89**	**155.20**	**3.91**	**0.14**	**19.80**	**2498.33**
		std	0.17	9.93	-	0.03	7.59	25.63	0.24	0.03	3.04	262.37
CA0.5%	I	**Av**	**11.45**	**1032.09**	**0.42**	**3.79**	**542.09**	**1483.61**	**29.93**	**1.00**	**139.47**	**20851.90**
		std	5.81	559.83	0.12	1.98	217.20	799.57	13.02	0.36	68.43	8289.47
	II	**Av**	**1.74**	**93.49**	**0.40**	**0.76**	**88.07**	**85.30**	**4.75**	**0.18**	**12.44**	**1954.21**
		std	1.07	35.88	-	0.29	30.71	93.60	2.55	0.08	6.31	762.51
	III	**Av**	**0.97**	**43.94**	<LOD	**0.38**	**39.61**	**26.87**	**1.25**	**0.11**	**6.36**	**875.08**
		std	-	16.87	-	0.09	7.10	36.58	-	-	-	267.43

Table 28 - Metals leached from sample 713C16 into acetic acid 4%, citric acid 0.5% and tomato sauce

Sample	**713C16**										
Simulant			**Al (µg/kg)**	**Cr (µg/kg)**	**Mn (µg/kg)**	**Fe (µg/kg)**	**Cu (µg/kg)**	**Ni (µg/kg)**	**Zn (µg/kg)**	**Pb (µg/kg)**	**Ba (µg/kg)**
AA4%	I	**Av**	**285.13**	**8.94**	**1.76**	**21.93**	**1.00**	**2.05**	**27.51**	**1244.85**	**19.60**
		std	114.28	8.80	1.18	29.20	0.97	0.84	6.17	433.26	6.00
	II	**Av**	**439.14**	**6.38**	**1.95**	**32.99**	**5.06**	**0.78**	**11.59**	**283.34**	**13.38**
		std	94.72	8.80	1.56	33.91	2.71	0.52	3.74	107.29	7.56
	III	**Av**	**463.70**	**5.99**	**2.30**	**31.64**	**<LOD**	**0.79**	**<LOD**	**197.02**	**13.14**
		std	67.50	8.13	1.15	28.30		0.47		77.58	2.76
CA0.5%	I	**Av**	**129.62**	**2.02**	**1.09**	**14.01**	**2.61**	**2.17**	**139.64**	**1018.05**	**30.05**
		std	41.30	1.15	0.38	7.94	4.53	1.21	180.39	632.91	6.75
	II	**Av**	**201.61**	**2.25**	**1.42**	**34.34**	**2.18**	**2.02**	**63.99**	**454.93**	**17.09**
		std	150.10	2.38	0.79	23.80	0.59	1.04	82.91	384.69	5.73
	III	**Av**	**181.31**	**2.15**	**0.75**	**17.68**	**1.23**	**0.76**	**11.48**	**261.62**	**14.95**
		std	122.35	2.67	0.36	13.27	1.27	0.52	3.49	235.82	2.27
Food			**Pb (µg/kg)**								
Tomato 2h 70°C	I	**Av**	**798.83**								
		std	737.08								
	II	**Av**	**185.61**								
		std	206.19								
	III	**Av**	**147.72**								
		std	169.13								

Table 29 - Metals leached from sample 713C21 into acetic acid 4%

Sample	**715C21**										
Simulant			**Al (µg/kg)**	**V (µg/kg)**	**Cr (µg/kg)**	**Fe (µg/kg)**	**As (µg/kg)**	**Cu (µg/kg)**	**Zn (µg/kg)**	**Sn (µg/kg)**	**Ba (µg/kg)**	**Pb (µg/kg)**
AA4%	I	**Av**	**65.84**	**0.11**	**40.87**	**74.47**	**0.33**	**0.20**	**2.95**	**1.28**	**2.20**	**11.22**
		std	18.80	0.08	0.67	2.11	0.08	0.04	0.63	0.99	0.83	3.29
	II	**Av**	**8.56**	**0.07**	**38.65**	**105.08**	**0.21**	**0.64**	**0.77**	**0.37**	**1.57**	**3.89**
		std	2.66	0.04	2.97	67.26	0.01	-	0.57	0.14	1.63	3.55
	III	**Av**	**0.71**	**0.04**	**40.53**	**69.70**	**0.20**	**0.68**	<LOD	**0.99**	**0.59**	**1.10**
		std	0.04	0.03	0.33	1.00	0.04	-	-	0.28	0.18	0.43

Table 30 - Metals leached from sample 713C23 into acetic acid 4% and citric acid 0.5%

Sample	713C23								
Simulant			Al (µg/kg)	Mn (µg/kg)	Fe (µg/kg)	Ni (µg/kg)	Cu (µg/kg)	Ba (µg/kg)	Pb (µg/kg)
AA4%	I	**Av**	**116.68**	**0.41**	**<LOD**	**<LOD**	**5.16**	**6.30**	**75.65**
		std	58.52	0.39			12.23	0.44	13.75
	II	**Av**	**113.52**	**0.17**	**<LOD**	**<LOD**	**<LOD**	**7.04**	**11.84**
		std	5.23	0.34				1.10	2.84
	III	**Av**	**84.16**	**<LOD**	**<LOD**	**<LOD**	**1.06**	**9.50**	**7.74**
		std	36.52				1.64	5.20	2.13
CA0.5%	I	**Av**	**33.34**	**<LOD**	**12.52**	**0.14**	**1.38**	**10.82**	**131.10**
		std	37.47		5.60	0.11	1.14	2.83	22.47
	II	**Av**	**350.15**	**3.99**	**52.86**	**1.04**	**4.30**	**9.22**	**56.10**
		std	257.77	4.26	43.46	0.07	3.38	3.83	25.14
	III	**Av**	**424.97**	**9.62**	**72.38**	**0.42**	**1.08**	**9.41**	**58.23**
		std	255.43	7.09	53.45	0.20	1.18	3.82	26.11

Table 31 - Metals leached from sample 713C24 into acetic acid 4% and tomato sauce

Sample	713C24							
Simulant			Co (µg/kg)	Sn (µg/kg)	Mn (µg/kg)	Ti (µg/kg)	Cu (µg/kg)	Al (µg/kg)
AA4%	I	**Av**	**354.92**	**20.06**	**81.88**	**10.67**	**4.94**	**12929.11**
		std	54.69	13.59	45.05	2.41	1.00	3969.12
	II	**Av**	**278.40**	**3.81**	**78.62**	**7.25**	**5.50**	**10318.89**
		std	72.44	1.15	25.98	1.23	0.96	2138.05
	III	**Av**	**198.49**	**0.73**	**106.65**	**9.39**	**4.98**	**10967.43**
		std	60.12	0.37	50.50	5.73	0.88	1808.95
			Zn (µg/kg)	Pb (µg/kg)	Ba (µg/kg)	As (µg/kg)	V (µg/kg)	Fe (µg/kg)
AA4%	I	**Av**	**224.02**	**1583.75**	**247.34**	**26.32**	**71.17**	**1581.63**
		std	31.09	222.28	24.83	9.67	23.48	620.52
	II	**Av**	**201.46**	**1277.48**	**210.26**	**24.90**	**87.80**	**982.16**
		std	41.99	324.80	46.64	4.09	6.98	207.17
	III	**Av**	**165.02**	**881.89**	**155.56**	**31.77**	**119.77**	**1192.00**
		std	43.95	268.39	40.47	5.93	18.96	143.63
Food			Pb (µg/kg)					
Tomato 6h boiling	I	**Av**	**1693.38**					
		std	88.81					
	II	**Av**	**948.12**					
		std	184.34					
	III	**Av**	**792.77**					
		std	51.88					

Table 32 - Metals leached from sample 713C25 into acetic acid 4%

Sample	**713C25**							
Simulant			**Co (µg/kg)**	**Cr (µg/kg)**	**Mn (µg/kg)**	**Ti (µg/kg)**	**Cu (µg/kg)**	**Al (µg/kg)**
AA4%	I	**Av**	**4.81**	**8.10**	**207.12**	**13.31**	**7.41**	**21306.78**
		std	0.76	2.72	96.56	2.23	0.99	2615.76
	II	**Av**	**3.72**	**4.97**	**220.97**	**9.01**	**6.40**	**15529.31**
		std	0.71	1.50	117.04	1.41	1.33	2833.50
	III	**Av**	**2.74**	**3.49**	**225.21**	**7.79**	**5.61**	**12406.45**
		std	0.37	0.43	54.25	1.41	0.52	1301.53
			Zn (µg/kg)	**Pb (µg/kg)**	**Ba (µg/kg)**	**As (µg/kg)**	**V (µg/kg)**	**Fe (µg/kg)**
AA4%	I	**Av**	**923.54**	**495.13**	**138.85**	**33.31**	**121.41**	**2389.10**
		std	141.69	71.08	18.14	8.94	7.11	274.03
	II	**Av**	**568.60**	**295.37**	**98.82**	**32.58**	**145.26**	**1531.26**
		std	81.90	44.35	9.73	8.97	18.63	319.62
	III	**Av**	**378.71**	**191.09**	**74.14**	**29.06**	**113.11**	**1223.82**
		std	31.46	19.51	2.08	3.53	15.00	227.48

Table 33 - Metals leached from sample 713C27 into acetic acid 4% and tomato sauce

Sample	**713C27**							
Simulant			**Co (µg/kg)**	**Cr (µg/kg)**	**Mn (µg/kg)**	**Ti (µg/kg)**	**Cu (µg/kg)**	**Al (µg/kg)**
AA4%	I	**Av**	**4.98**	**20.02**	**92.44**	**28.83**	**15.21**	**14672.17**
		std	1.59	5.20	33.93	5.32	4.44	4747.60
	II	**Av**	**3.34**	**10.07**	**75.73**	**25.59**	**11.76**	**10177.39**
		std	0.58	1.54	21.77	3.60	2.00	1732.79
	III	**Av**	**2.87**	**8.73**	**66.52**	**24.88**	**10.64**	**8475.15**
		std	0.71	1.65	23.26	6.23	2.39	1896.43
			Zn (µg/kg)	**Pb (µg/kg)**	**Ba (µg/kg)**	**As (µg/kg)**	**V (µg/kg)**	**Fe (µg/kg)**
AA4%	I	**Av**	**780.10**	**3664.29**	**192.23**	**15.77**	**48.94**	**2859.78**
		std	239.00	1130.71	68.98	3.65	8.77	626.94
	II	**Av**	**379.39**	**1931.12**	**90.31**	**14.40**	**57.38**	**1572.37**
		std	53.22	276.56	20.13	5.33	10.63	145.70
	III	**Av**	**311.24**	**1615.44**	**79.23**	**13.79**	**60.77**	**1359.21**
		std	64.92	306.29	18.61	4.98	14.67	186.03
Food			**Pb (µg/kg)**					
Tomato 6h boiling	I	**Av**	**894.90**					
		std	73.89					
	II	**Av**	**844.80**					
		std	78.07					
	III	**Av**	**866.64**					
		std	66.86					

Table 34 - Metals leached from sample 713C28 into acetic acid 4% and citric acid 0.5%

Sample	713C28									
Simulant			**Li (μg/kg)**	**Al (μg/kg)**	**Ti (μg/kg)**	**V (μg/kg)**	**Cr (μg/kg)**	**Mn (μg/kg)**	**Fe (μg/kg)**	**Ni (μg/kg)**
AA4%	I	**Av**	**39.15**	**2418.82**	**147.89**	**23.36**	**5.78**	**37.33**	**586.91**	**0.42**
		std	1.03	139.61	4.45	2.25	0.59	3.29	15.03	0.91
	II	**Av**	**34.96**	**1384.77**	**109.53**	**27.33**	**4.47**	**55.07**	**158.70**	**0.99**
		std	2.37	118.15	3.08	3.00	0.78	5.25	12.25	0.15
	III	**Av**	**25.52**	**1053.81**	**79.57**	**23.13**	**2.81**	**58.38**	**106.54**	**0.54**
		std	2.18	126.07	4.97	3.28	0.42	3.49	5.34	0.11
CA0.5%	I	**Av**	**23.90**	**1673.72**	**443.53**	**5.43**	**1.92**	**12.49**	**568.90**	**1.71**
		std	2.41	160.00	37.56	1.62	0.28	1.90	61.89	0.32
	II	**Av**	**20.58**	**540.22**	**103.85**	**19.47**	**1.45**	**14.60**	**146.08**	**0.91**
		std	2.04	88.20	13.73	2.80	0.32	1.01	32.38	0.10
	III	**Av**	**23.14**	**399.12**	**73.70**	**27.23**	**1.51**	**14.09**	**99.50**	**0.91**
		std	1.13	58.79	17.27	1.10	0.18	0.89	23.01	0.15
Simulant			**Cu (μg/kg)**	**Zn (μg/kg)**	**Sr (μg/kg)**	**Zr (μg/kg)**	**Cd (μg/kg)**	**Ba (μg/kg)**	**Pb (μg/kg)**	
AA4%	I	**Av**	**5512.22**	**3167.56**	**40.13**	**2.49**	**1.67**	**227.45**	**19061.17**	
		std	226.68	958.28	1.66	0.63	0.52	16.29	1095.95	
	II	**Av**	**1077.94**	**363.53**	**37.78**	**<LOD**	**0.56**	**62.28**	**4121.23**	
		std	106.78	33.99	2.14		0.03	2.51	207.76	
	III	**Av**	**611.76**	**204.29**	**30.21**	**<LOD**	**0.23**	**36.89**	**2332.85**	
		std	60.71	17.80	4.31		0.07	2.91	235.77	
CA0.5%	I	**Av**	**4880.88**	**1782.00**	**15.98**	**95.72**	**2.11**	**248.23**	**20357.65**	
		std	394.74	223.64	2.03	11.39	0.20	47.87	2588.06	
	II	**Av**	**1192.56**	**404.96**	**7.33**	**32.95**	**0.47**	**63.68**	**4978.40**	
		std	242.71	93.19	1.17	3.73	0.18	17.58	1327.19	
	III	**Av**	**756.67**	**266.24**	**5.94**	**21.11**	**0.27**	**40.39**	**2974.84**	
		std	127.36	36.68	0.54	3.95	0.07	8.19	649.26	

Table 35 - Metals leached from sample 713C30 into acetic acid 4%

Sample	715C30													
Simulant			**Li (μg/kg)**	**Al (μg/kg)**	**Ti (μg/kg)**	**V (μg/kg)**	**Mn (μg/kg)**	**Co (μg/kg)**	**Ni (μg/kg)**	**Zn (μg/kg)**	**As (μg/kg)**	**Cd (μg/kg)**	**Ba (μg/kg)**	**Pb (μg/kg)**
AA4%	I	**Av**	**13.16**	**3891.77**	**5.13**	**11.93**	**77.80**	**2.20**	**4.10**	**13.70**	**3.99**	**0.45**	**16.92**	**321.89**
		std	1.53	375.60	0.80	0.91	11.19	0.18	0.20	3.73	0.70	0.31	7.37	108.23
	II	**Av**	**14.58**	**2006.93**	**2.55**	**5.54**	**79.95**	**1.62**	**3.31**	**8.32**	**1.81**	**0.47**	**10.72**	**158.46**
		std	3.12	737.02	1.10	1.49	23.52	0.42	0.56	2.19	0.68	0.19	4.73	106.19
	III	**Av**	**8.94**	**906.18**	**0.90**	**2.74**	**62.42**	**1.02**	**2.26**	**3.46**	**1.20**	**0.25**	**6.94**	**76.38**
		std	0.94	117.43	0.06	0.45	5.68	0.08	0.54	0.53	0.30	0.09	1.06	13.29

Table 36 - Metals leached from samples 913C03, 913C08 and 913C09 into acetic acid 4% and citric acid 0.5%

Sample			Li (µg/kg)		Al (µg/kg)		Ti (µg/kg)		Cu (µg/kg)		Zn (µg/kg)		Ba (µg/kg)	
			AA 4%	**CA 0.5%**	**AA 4%**	**CA 0.5%**	**AA 4%**	**CA 0.5%**	**AA 4%**	**CA 0.5%**	**AA 4%**	**CA 0.5%**	**AA 4%**	**CA 0.5%**
913C03	**I**	**Av**	**0.19**	**0.33**	**63.05**	**187.85**	**0.63**	**0.50**	**3.39**	**4.18**	**108.71**	**162.97**	**6.01**	**10.64**
		std	0.01	0.02	2.93	100.63	0.03	0.06	0.69	0.25	16.26	11.98	0.53	0.84
	II	**Av**	**<LOD**	**0.03**	**7.90**	**39.02**	**0.20**	**<LOD**	**0.22**	**0.22**	**11.47**	**13.29**	**0.75**	**0.92**
		std		0.01	0.59	36.88	0.06		0.07	0.14	2.39	2.84	0.07	0.45
	III	**Av**	**<LOD**	**0.05**	**6.90**	**49.23**	**0.37**	**<LOD**	**<LOD**	**0.33**	-	**7.98**	**0.55**	**0.56**
		std		0.10	2.38	76.50	0.05			0.53		2.17	0.12	0.16
913C08	**I**	**Av**	**0.96**	**2.26**	**18.92**	**27.35**	**0.22**	**<LOD**	**0.73**	**1.36**	**10.61**	**11.80**	**0.16**	**<LOD**
		std	0.14	0.48	2.79	4.82	0.02		0.35	1.12	2.64	3.01	0.02	
	II	**Av**	**0.18**	**0.44**	**2.97**	**9.80**	**<LOD**	**<LOD**	**0.24**	**1.27**	**13.81**	**<LOD**	**0.06**	**0.43**
		std	0.03	0.06	0.49	5.24			0.41	2.47	22.59		0.06	0.64
	III	**Av**	**0.11**	**0.28**	**1.83**	**<LOD**	**0.36**	**<LOD**	**<LOD**	**<LOD**	-	**<LOD**	**0.14**	**<LOD**
		std	0.02	0.06	0.32		0.06						0.25	
913C09	**I**	**Av**	**1.74**	**3.54**	**20.58**	**24.53**	**0.20**	**<LOD**	**0.37**	**0.42**	**8.93**	**<LOD**	**0.20**	**<LOD**
		std	0.11	0.09	1.07	1.24	0.03		0.17	0.11	4.31		0.07	
	II	**Av**	**0.33**	**0.71**	**4.32**	**6.13**	**0.16**	**<LOD**	**<LOD**	**<LOD**	**<LOD**	**<LOD**	**0.18**	**<LOD**
		std	0.01	0.03	2.37	0.36	0.08						0.24	
	III	**Av**	**0.22**	**0.42**	**3.38**	**<LOD**	**0.38**	**<LOD**	**<LOD**	**<LOD**	-	**<LOD**	**0.06**	**<LOD**
		std	0.01	0.02	1.95		0.04						0.02	

Table 37 - Metals leached from samples 916C16 into acetic acid 4%

Sample	**916C16**											
Simulant			**Al (µg/kg)**	**Mn (µg/kg)**	**Ti (µg/kg)**	**Fe (µg/kg)**	**Co (µg/kg)**	**Cu (µg/kg)**	**Zn (µg/kg)**	**Cd (µg/kg)**	**Ba (µg/kg)**	**Pb (µg/kg)**
AA4%	I	**Av**	**355.40**	**90.97**	**399.58**	**126.67**	**76.37**	**1488.69**	**1711.36**	**1.52**	**32.53**	**0.77**
		std	143.05	35.85	136.53	53.54	30.34	452.26	595.57	2.06	11.87	0.51
	II	**Av**	**13.42**	**1.96**	**85.58**	**<LOD**	**1.52**	**19.75**	**38.93**	**0.04**	**0.72**	**<LOD**
		std	8.23	1.36	52.01		0.94	11.83	27.44	0.03	0.36	
	III	**Av**	**5.44**	**1.02**	**20.11**	**3.90**	**0.76**	**6.61**	**13.95**	**0.02**	**0.17**	**<LOD**
		std	0.19	0.24	10.14	4.45	0.16	1.18	2.39	0.01	0.09	

Table 38 - Metals leached from samples 916C17 into acetic acid 4%

Sample	**916C17**								
Simulant			**Al (µg/kg)**	**Mn (µg/kg)**	**Ti (µg/kg)**	**Fe (µg/kg)**	**Co (µg/kg)**	**Zn (µg/kg)**	**Pb (µg/kg)**
AA4%	I	**Av**	**93.23**	**31.14**	**193.14**	**31.80**	**63.17**	**482.62**	**<LOD**
		std	9.31	2.63	12.67	1.20	5.39	41.60	
	II	**Av**	**7.62**	**0.97**	**19.06**	**<LOD**	**1.89**	**19.30**	**<LOD**
		std	0.82	0.38	1.26		0.65	5.39	
	III	**Av**	**6.90**	**1.12**	**9.18**	**23.28**	**2.05**	**20.97**	**<LOD**
		std	0.74	0.44	2.35	12.61	0.77	5.94	

Table 39 - Metals leached from samples 1015C01 into acetic acid 4%

Sample	1015C01									
Simulant			Al (µg/kg)	Cr (µg/kg)	Fe (µg/kg)	Mn (µg/kg)	Cu (µg/kg)	Zn (µg/kg)	Ba (µg/kg)	Pb (µg/kg)
AA4%	I	**Av**	**250.87**	**0.75**	**7.22**	**0.60**	**0.98**	**43.97**	**39.82**	**1.67**
		std	94.52	0.20	6.05	0.71	0.86	11.59	13.37	0.26
	II	**Av**	**269.18**	**0.42**	**7.54**	**1.41**	**0.21**	**32.28**	**33.56**	**0.98**
		std	99.16	0.56	11.41	1.55	0.14	19.31	12.59	1.21
	III	**Av**	**203.61**	**<LOD**	**<LOD**	**0.90**	**0.26**	**19.63**	**22.75**	**<LOD**
		std	51.43			1.24	0.39	7.47	6.51	

Table 40 - Metals leached from samples 1016C02 into acetic acid 4%

Sample	1016C02											
Simulant			Al (µg/kg)	Mn (µg/kg)	Ti (µg/kg)	Fe (µg/kg)	Co (µg/kg)	Cu (µg/kg)	Zn (µg/kg)	Ni (µg/kg)	Ba (µg/kg)	Pb (µg/kg)
AA4%	I	**Av**	**1717.32**	**326.65**	**192.92**	**55561.61**	**5.46**	**7.93**	**19.25**	**8.24**	**49.90**	**0.42**
		std	1123.73	239.24	110.60	41637.31	3.86	5.23	10.59	5.90	32.29	0.20
	II	**Av**	**464.56**	**206.40**	**48.77**	**46576.38**	**1.54**	**1.57**	**6.74**	**2.99**	**13.30**	**<LOD**
		std	846.09	402.56	57.87	89840.70	2.90	1.47	7.64	5.80	22.60	
	III	**Av**	**779.67**	**598.26**	**59.56**	**115901.44**	**3.37**	**3.19**	**9.62**	**6.66**	**23.10**	**<LOD**
		std	756.55	740.88	51.37	138759.69	2.80	2.94	3.62	5.15	22.47	

Table 41 - Metals leached from samples 1016C03 into acetic acid 4%

Sample	1016C03									
Simulant			Al (µg/kg)	Mn (µg/kg)	Ti (µg/kg)	Fe (µg/kg)	Co (µg/kg)	Cu (µg/kg)	Ba (µg/kg)	Pb (µg/kg)
AA4%	I	**Av**	**810.88**	**10.79**	**120.89**	**676.34**	**2.42**	**31.54**	**20.35**	**0.51**
		std	90.52	21.22	37.30	1295.52	2.11	54.11	35.58	0.47
	II	**Av**	**128.10**	**0.55**	**59.28**	**138.04**	**0.33**	**9.51**	**0.91**	**0.60**
		std	6.38	0.87	6.56	199.38	0.02	15.56	0.45	0.98
	III	**Av**	**107.72**	**<LOD**	**44.35**	**34.57**	**0.28**	**0.48**	**0.51**	**<LOD**
		std	21.73		2.88	28.44	0.01	0.47	0.07	

Table 42 - Metals leached from samples 1016C04 into acetic acid 4%

Sample	1016C04										
Simulant			Al (µg/kg)	Mn (µg/kg)	Ti (µg/kg)	Fe (µg/kg)	Co (µg/kg)	Cu (µg/kg)	Cr (µg/kg)	Ba (µg/kg)	Pb (µg/kg)
AA4%	I	**Av**	**40.31**	**16.90**	**7.24**	**25.47**	**9.00**	**12.39**	**1.11**	**11.22**	**<LOD**
		std	7.13	1.72	1.20	1.58	0.98	1.03	0.22	0.82	
	II	**Av**	**6.29**	**2.61**	**2.27**	**4.84**	**1.43**	**1.92**	**0.25**	**2.11**	**<LOD**
		std	0.66	0.05	0.49	1.80	0.02	0.17	0.06	0.38	
	III	**Av**	**3.09**	**1.84**	**1.59**	**3.09**	**1.00**	**1.37**	**0.18**	**1.35**	**<LOD**
		std	0.41	0.08	0.27	1.70	0.03	0.39	0.04	0.09	

Annex 5: Storage effect for Cd, Co, Al and Zn

Table 1 – Results of storage effect for Cd

Samples			Cd [µg/kg]	Storage time	Cd [µg/kg]	Storage time	Cd [µg/kg]	Storage time	Cd [µg/kg]
615C13	I	Av	6.75	6 months	0.45	1 month	0.32	1 week	0.25
		RSD [%]	58.01		29.05		37.97		33.23
	II	Av	2.74		0.24		0.20		0.22
		RSD [%]	85.44		40.92		40.50		36.61
	III	Av	2.80		0.23		0.16		0.23
		RSD [%]	54.07		58.86		36.98		48.15
615C15	I	Av	0.57		0.03		0.03		0.01
		RSD [%]	6.54		32.58		25.83		62.85
	II	Av	0.05		0.01		0.01		0.01
		RSD [%]	32.36		42.51		21.17		100.04
	III	Av	0.04		0.01		<LOD		0.01
		RSD [%]	89.52		40.31		-		44.04
615C17	I	Av	0.02		0.01		0.01		<LOD
		RSD [%]	46.93		53.53		213.46		-
	II	Av	<LOD		0.01		<LOD		<LOD
		RSD [%]	-		56.94		-		-
	III	Av	<LOD		0.02		<LOD		<LOD
		RSD [%]	-		106.33		-		-
615C18	I	Av	0.06		0.02		<LOD		<LOD
		RSD [%]	55.97		38.71		-		-
	II	Av	<LOD		0.01		<LOD		<LOD
		RSD [%]	-		47.17		-		-
	III	Av	<LOD		0.01		<LOD		<LOD
		RSD [%]	-		34.51		-		-
615C19	I	Av	0.39		0.16		0.08		0.04
		RSD [%]	37.61		36.82		37.92		45.88
	II	Av	0.14		0.07		0.04		0.05
		RSD [%]	36.29		38.07		43.27		51.03
	III	Av	0.11		0.06		0.04		0.04
		RSD [%]	38.06		18.85		34.83		51.55
Samples			**Cd [µg/dm^2]**	**Storage time**	**Cd [µg/dm^2]**	**Storage time**	**Cd [µg/dm^2]**	**Storage time**	**Cd [µg/dm^2]**
615C12	I	Av	1.85	6 months	0.20	1 month	0.09	1 week	0.10
		RSD [%]	30.94		23.33		39.01		39.56
	II	Av	0.84		0.16		0.06		0.04
		RSD [%]	36.34		46.65		55.24		18.23
	III	Av	0.45		0.12		0.06		0.10
		RSD [%]	38.30		31.27		52.71		34.42
615C14	I	Av	0.19		0.006		0.005		0.004
		RSD [%]	23.88		57.33		40.32		56.27
	II	Av	0.02		0.004		0.003		0.002
		RSD [%]	7.73		49.69		87.22		71.25
	III	Av	0.01		0.002		0.02		0.002
		RSD [%]	62.42		40.76		52.44		69.36
615C16	I	Av	0.018		0.006		0.001		0.001
		RSD [%]	41.21		79.92		47.33		70.91
	II	Av	0.006		0.003		0.001		0.001
		RSD [%]	47.73		38.52		76.54		88.19
	III	Av	0.004		0.002		0.001		0.001
		RSD [%]	51.21		38.99		29.32		67.70
615C20	I	Av	0.67		0.18		0.08		0.06
		RSD [%]	10.79		8.29		14.59		19.24
	II	Av	0.24		0.07		0.05		0.06
		RSD [%]	18.11		7.89		10.74		18.75
	III	Av	0.18		0.06		0.04		0.05
		RSD [%]	4.41		4.54		7.72		16.35

Table 2 – Results of storage effect for Co

Samples			Co [µg/kg]	Storage time	Co [µg/kg]	Storage time	Co [µg/kg]	Storage time	Co [µg/kg]
615C13	I	Av	154.76		2.00		1.17		0.87
		RSD [%]	25.65		25.54		33.93		16.61
	II	Av	16.49		0.88		0.74		0.70
		RSD [%]	100.35		31.84		42.63		28.77
	III	Av	17.09		0.86		0.61		0.76
		RSD [%]	57.96		41.35		29.45		27.83
615C15	I	Av	2196.10		31.82		5.22		4.94
		RSD [%]	5.38		76.09		28.98		33.31
	II	Av	98.63		14.17		4.80		4.16
		RSD [%]	29.98		99.06		20.62		32.00
	III	Av	91.81		14.47		4.01		3.85
		RSD [%]	122.60		92.54		37.09		17.49
615C17	I	Av	612.90		178.40		56.86		30.70
		RSD [%]	15.02		36.57		56.06		47.92
	II	Av	174.26	6 months	52.05	1 month	31.56	1 week	30.70
		RSD [%]	22.97		35.39		55.79		46.83
	III	Av	108.65		40.43		28.90		27.60
		RSD [%]	21.74		31.36		51.43		52.25
615C18	I	Av	316.83		90.01		57.11		47.52
		RSD [%]	4.09		12.53		11.89		15.91
	II	Av	110.30		55.42		48.25		47.92
		RSD [%]	9.73		15.62		14.19		13.14
	III	Av	80.86		48.74		42.29		46.09
		RSD [%]	4.87		10.71		20.97		19.45
615C19	I	Av	617.66		240.73		126.52		70.05
		RSD [%]	12.09		13.49		14.48		19.78
	II	Av	224.75		100.75		71.77		70.38
		RSD [%]	14.31		13.13		11.84		15.61
	III	Av	183.96		79.19		64.67		66.24
		RSD [%]	24.42		5.21		15.77		15.39
Samples			**Co [µg/dm^2]**	**Storage time**	**Co [µg/dm^2]**	**Storage time**	**Co [µg/dm^2]**	**Storage time**	**Co [µg/dm^2]**
615C12	I	Av	34.01		0.67		0.33		0.26
		RSD [%]	13.31		37.32		34.05		24.58
	II	Av	5.86		0.48		0.17		0.12
		RSD [%]	23.15		37.91		51.16		9.79
	III	Av	2.33		0.33		0.17		0.36
		RSD [%]	15.78		21.00		42.29		82.81
615C14	I	Av	795.21		2.14		1.77		1.82
		RSD [%]	4.08		18.04		13.17		34.09
	II	Av	63.99		1.34		4.74		16.61
		RSD [%]	56.14		39.48		139.33		39.70
	III	Av	6.62		0.85		1.00		0.76
		RSD [%]	50.45	6 months	29.88	1 month	23.49	1 week	31.11
615C16	I	Av	173.22		49.90		17.88		19.82
		RSD [%]	17.08		77.47		76.42		53.24
	II	Av	59.08		26.52		14.78		18.32
		RSD [%]	21.94		37.96		39.63		51.71
	III	Av	40.30		21.80		14.17		15.97
		RSD [%]	24.85		35.34		43.41		52.99
615C20	I	Av	55.43		18.71		8.60		6.17
		RSD [%]	11.16		13.63		16.68		22.06
	II	Av	21.52		7.90		5.47		5.80
		RSD [%]	18.69		9.68		14.44		21.17
	III	Av	17.29		7.02		4.95		5.14
		RSD [%]	8.30		9.12		12.09		17.94

Table 3 – Results of storage effect for Al

Samples			Al [µg/kg]	Storage time	Al [µg/kg]	Storage time	Al [µg/kg]	Storage time	Al [µg/kg]
615C13	I	Av	83.16	6 months	9.28	1 month	15.34	1 week	14.43
		RSD [%]	24.74		18.19		25.49		12.21
	II	Av	20.48		6.48		9.80		12.69
		RSD [%]	63.11		21.11		14.64		14.43
	III	Av	19.42		5.14		7.34		10.97
		RSD [%]	31.69		40.39		7.71		22.39
615C15	I	Av	65.79		4.30		7.39		11.46
		RSD [%]	23.76		19.57		14.16		55.05
	II	Av	8.40		1.35		4.59		7.01
		RSD [%]	57.64		24.86		12.50		19.17
	III	Av	8.54		1.33		3.04		7.92
		RSD [%]	63.66		38.75		13.97		16.57
615C17	I	Av	379.95		103.26		45.06		23.81
		RSD [%]	14.62		35.01		63.12		20.48
	II	Av	108.57		30.48		18.46		23.19
		RSD [%]	20.71		24.53		38.48		27.20
	III	Av	64.61		26.02		16.40		20.35
		RSD [%]	18.90		20.73		30.17		27.13
615C18	I	Av	176.72		35.11		18.79		19.45
		RSD [%]	3.69		16.57		14.16		42.62
	II	Av	45.97		15.36		16.88		13.20
		RSD [%]	10.59		16.07		18.23		15.51
	III	Av	27.48		11.40		16.02		14.43
		RSD [%]	3.74		8.72		19.18		31.42
615C19	I	Av	1099.57		376.69		201.97		149.49
		RSD [%]	10.70		9.69		12.17		15.42
	II	Av	396.82		165.16		139.23		155.51
		RSD [%]	12.97		10.13		21.96		15.77
	III	Av	326.65		129.07		113.93		143.55
		RSD [%]	21.60		3.47		17.77		10.81
Samples			Al [µg/dm^2]	Storage time	Al [µg/dm^2]	Storage time	Al [µg/dm^2]	Storage time	Al [µg/dm^2]
615C12	I	Av	12.94	6 months	1.97	1 month	1.19	1 week	1.63
		RSD [%]	16.69		12.12		20.34		27.07
	II	Av	4.51		1.49		0.85		1.24
		RSD [%]	31.81		32.17		32.45		17.51
	III	Av	2.97		1.20		0.88		1.62
		RSD [%]	27.72		21.33		40.12		45.53
615C14	I	Av	14.21		0.80		1.00		1.17
		RSD [%]	21.73		27.53		13.78		49.37
	II	Av	3.74		0.49		0.71		0.92
		RSD [%]	23.05		34.67		44.02		35.54
	III	Av	2.32		0.34		0.49		0.82
		RSD [%]	42.65		38.26		31.48		69.26
615C16	I	Av	102.37		24.44		7.47		8.26
		RSD [%]	12.13		72.36		65.04		27.50
	II	Av	32.94		11.38		5.50		6.78
		RSD [%]	15.04		14.26		21.59		10.60
	III	Av	21.36		8.16		4.45		5.66
		RSD [%]	13.32		9.61		14.97		19.07
615C20	I	Av	131.70		37.91		17.88		17.41
		RSD [%]	10.62		11.04		16.28		18.14
	II	Av	49.64		17.33		12.60		16.37
		RSD [%]	18.47		6.49		11.28		15.82
	III	Av	40.26		15.40		11.49		14.91
		RSD [%]	9.08		4.53		14.86		19.34

Table 4 – Results of storage effect for Zn

Samples			Zn [µg/kg]	Storage time	Zn [µg/kg]	Storage time	Zn [µg/kg]	Storage time	Zn [µg/kg]
615C13	I	Av	291.32	6 months	<LOD	1 month	19.23	1 week	<LOD
		RSD [%]	20.47		-		14.44		-
	II	Av	24.88		<LOD		20.92		<LOD
		RSD [%]	106.55		-		16.71		-
	III	Av	26.33		<LOD		17.51		<LOD
		RSD [%]	58.54		-		11.16		-
615C15	I	Av	3578.33		52.49		27.46		10.71
		RSD [%]	5.67		81.00		21.55		40.70
	II	Av	157.63		21.38		26.90		<LOD
		RSD [%]	30.25		109.83		6.80		-
	III	Av	148.04		20.37		8.30		<LOD
		RSD [%]	123.24		109.39		19.94		-
615C17	I	Av	411.98		116.06		40.94		20.00
		RSD [%]	14.66		34.52		40.90		34.08
	II	Av	115.26		31.99		19.90		15.46
		RSD [%]	21.28		25.58		45.64		39.70
	III	Av	68.52		24.34		23.03		14.93
		RSD [%]	19.12		43.08		26.33		44.23
615C18	I	Av	210.52		47.92		30.01		19.19
		RSD [%]	5.92		12.01		22.36		32.49
	II	Av	55.37		21.91		26.64		13.73
		RSD [%]	10.05		17.82		25.89		14.29
	III	Av	36.51		15.59		18.27		13.49
		RSD [%]	9.54		13.73		25.37		25.00
615C19	I	Av	794.41		309.53		162.46		97.69
		RSD [%]	12.86		12.87		14.90		11.09
	II	Av	284.74		131.17		89.99		94.20
		RSD [%]	14.00		11.03		9.74		16.81
	III	Av	238.21		97.02		83.61		89.59
		RSD [%]	25.39		5.38		15.53		17.91
Samples			Zn [µg/dm^2]	Storage time	Zn [µg/dm^2]	Storage time	Zn [µg/dm^2]	Storage time	Zn [µg/dm^2]
615C12	I	Av	63.67	6 months	0.97	1 month	2.29	1 week	0.67
		RSD [%]	12.56		39.16		39.47		48.54
	II	Av	9.36		0.66		1.50		0.39
		RSD [%]	25.30		42.12		3.04		84.87
	III	Av	3.52		0.32		1.58		0.48
		RSD [%]	14.21		58.47		6.29		97.54
615C14	I	Av	1325.30		2.76		3.75		2.56
		RSD [%]	3.92		17.49		9.80		41.60
	II	Av	102.96		1.86		8.78		4.53
		RSD [%]	57.82		49.90		123.90		79.46
	III	Av	9.63		0.94		2.80		0.96
		RSD [%]	52.11		37.13		15.93		31.85
615C16	I	Av	123.38		33.05		10.28		9.54
		RSD [%]	18.62		76.46		62.89		37.42
	II	Av	37.85		14.39		7.71		8.00
		RSD [%]	21.35		27.53		27.70		37.92
	III	Av	24.55		10.69		6.48		6.93
		RSD [%]	21.13		25.04		31.05		41.01
615C20	I	Av	72.17		24.53		11.24		8.27
		RSD [%]	11.39		13.42		13.78		23.17
	II	Av	27.79		9.94		6.96		7.74
		RSD [%]	18.28		9.39		13.37		20.82
	III	Av	22.37		8.71		6.99		17.14
		RSD [%]	6.96		8.28		15.11		122.03

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