

# Lead Extracted from Ceramics under Household Conditions

The Swedish National Food Administration (NFA) and  
the Norwegian Food Safety Authority (Mattilsynet)

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## Summary

Lead (Pb) exposure from ceramics is an old and well-known hazard. In order to investigate the lead leakage from ceramics at more realistic test conditions and with more realistic test simulants like beverages, the following tests were carried out.

A total of 310 straight white mugs were prepared, with a volume of 230 ml. The inside of the mugs were decorated with an enamel colour containing lead, mimicing a lead glaze.

Red wine, orange juice, coffee, tea with lemon and water were used as test simulants and compared with the standard test solution of 4 % acetic acid. Red wine was stored for 24 hours, which is equal to the standard method (using acetic acid). For orange juice a longer storage time of 72 hours at 8° C was chosen, while for coffee and lemon tea, 30 minutes at 80° C was chosen.

Orange juice and red wine both extracted approximately 20-30 % of lead, relative to 4 % acetic acid at standard conditions. The longer time period using orange juice at low temperature gave similar results. Unexpected results were found for tea with lemon, which extracted approximately the same amount of lead as 4 % acetic acid in a short time at high temperature. Coffee, which has a higher pH than lemon tea, was a weaker extractant at the same conditions.

The implications of these tests are that 4 % acetic acid in most cases are a realistic worst case extractant, although attention should be paid to other realistic test simulants, such as different beverages, tested under realistic conditions. In this report we also discuss possible amendments of the test method to better reflect real exposure.

The results from this report may be used by EFSA in a risk assessment of lead exposure from ceramics.

## Introduction

Ceramics have been associated with lead toxicity since ancient times. Gilfillan (1) and Ramazzini (2) vividly described occupational lead poisoning among potters producing lead-glazed earthenware. Acute effects like fatigue, restless legs, sleep disturbances, abdominal pain and nausea have been observed as well as chronic effects on the central nervous system and inhibition of the formation of erythrocytes in the blood. During the previous century, strict legislation for the use of lead in society has made obvious lead poisoning a rarity in economically developed parts of the world, which may have reduced the awareness of the risks associated with lead in the general public.

The manufacture of ceramics with a surface glazed with lead oxide is a rather difficult and demanding procedure. The glaze may be combined with other oxides or salts to give certain colours or other properties. After having been covered with the glaze the ceramic articles are fired at a specific temperature, which is dependent on the combination of glaze and other components. If the temperature during the firing is too low, the glaze will not be stable and may gradually be dissolved by the food or beverage with which it is in contact. If the firing temperature is too high the glaze may be unstable and/or evaporate to some degree during the process. The temperature during the firing is thus of the utmost importance. Furthermore, the temperature must be as gradient-free as possible within the kiln, otherwise parts of a batch may be fired at an erroneous temperature. It is today rather unusual to find unsatisfactory products from major ceramic producers. Small scale producers, however, may not always have the competence or the economy to safeguard the quality of their produce.

It is thus possible to purchase lead-glazed ceramics as handicraft which does not stand up to the present standards of testing (EU) and health-based limits. This has been observed lately in a number of cases as lead intoxication from drinking juice or wine from articles made of ceramics from the Mediterranean area. Also import from other parts of the world may be a problem of concern.

In the European Union legal limits and the basic test method for migration of lead and cadmium from food contact ceramics are given by Directive 84/500/EEC (3) (updated by the Directive 2005/31/EC; 4). Testing is performed with 4 % acetic acid for 24 hours at 22° C. Ceramic articles that can be filled, e.g., a mug or a jug have to comply with a limit for lead of 4.0 mg Pb/L of the volume of the article. However, some questions have been raised whether or not this limit is low enough to ensure public health. The limit is far higher than the limits for different foodstuffs, ranging between 0.02 – 1.5 mg/kg fresh weight as decided in Regulation 1881/2006/EC (5). Also, in comparison with the Plastics Directive 2002/72/EC (6; 2 mg Pb/kg plastic) the restriction also seems to be far too high, as well as if we compare with restrictions for drinking water; 0.01 mg/L (7)

In order to estimate exposure it is interesting to compare test results with real use of ceramic articles, i.e. to gain knowledge on the influence of time, temperature and food type. In many cases, 24 hours of testing may not be representative for real use. For example, when drinking coffee, a realistic time from filling a mug with coffee to consumption may be only a few minutes, whereas orange juice may be filled in a jug and be kept refrigerated for several days before consumption.

The following study was carried out to examine lead release at realistic conditions, in order to give the European Food Safety Authority (EFSA) some further basis for its risk assessment (toxicological evaluation) of lead intake from ceramics. For several years, Sweden, Germany and Norway have been involved in a discussion with the Commission if the lead limits are valid, i.e., if they really protect public health. Lately Belgium has also started to investigate the same topic (8).

## **Materials and Methods**

In order to find the most suitable test mugs, four batches of straight white mugs, each with a different lead containing enamel colour, were produced and tested for release of lead. The batch using enamel colour Flame orange 97H1003 was found to have a suitable concentration level in the extract solution (for production details see below). The stability of this enamel was tested on six mugs by six consecutive extractions and determination of lead using method EN1388-1 (9).

For the project another 310 mugs, each with a volume of 230 ml, were first glazed with a lead free glaze made up of quartz, nephelinsyenite, whiting, dolomite and alumina and then fired in 1400° C. The inside of the mugs were thereafter decorated with the enamel colour Flame orange. The enamel was applied as transfers, one piece on the vertical side of the mug and one piece in the bottom. The transfer pieces were screen printed from the same screen so as to get the same amount of pigment and size of the print in each mug (Pictures 1 and 2).



*Picture 1*



*Picture 2*

The 2<sup>nd</sup> firing of the enamel on the mugs was done in an intermittent kiln to 850° C in a firing schedule of 5 hours to top temperature and 0.5 hour holding time followed by 10 hours cooling to 50° C. The variation of temperature or heat work in the kiln is shown by the difference in bending of the Orton cones that were in different places in the kiln during the firing (Picture 3). From these a temperature difference of less than 20° C between the highest and lowest temperature could be measured.

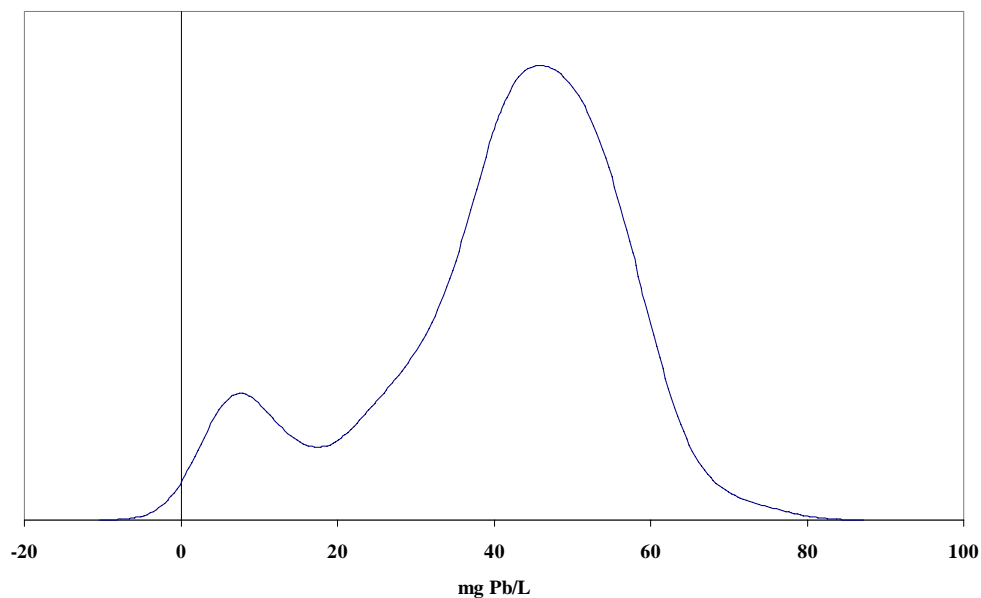


*Picture 3. Orton cones are designed to indicate a temperature interval. They are usually used in sets of three which reacts slightly different to the temperature, and can thus indicate which temperature was actually reached during the firing. The cones in the right row that accompanied the mugs during the firing shows that the temperature interval for which they were chosen was complied with. At too low a temperature the cones would not bend enough and at too high a temperature they would bend too much. The left row show unused cones for different temperatures.*

Before use, the mugs were washed using a standard detergent and tap water, and then rinsed with deionised water. Thereafter the mugs were extracted using the method EN 1388-1 (9), according to which ceramic articles are placed in contact with 4 % acetic acid for 24 hours at 22° C to extract lead, if present, from the surface of the mugs. Lead was then determined in the extracting solution using flame atomic absorption spectrometry (FAAS; 10) using a Varian SpectrAA-220FS. This first extraction was done to remove loosely bound lead-containing particles that may be distributed through the furnace during the firing process, prior to the commencement of the actual release tests.

The results of the first extraction of all 310 mugs showed a bimodal distribution of lead, which turned out to be advantageous. It was decided to use 12 mugs for tests 1-3 in order to make it possible to detect reasonably small differences between the groups. The individual mugs were chosen from the result of the first extraction. Twelve mugs in the range 20 – 80 mg/L were selected to give an

average of 40-45 mg/L/group (Figure 1). For test 4 six mugs per group was considered to be sufficient. These mugs were selected from the left mode (Figure 1) in order to give a mean around 5 mg/L in each group, which approximates the maximum allowed limit (3) for lead release from ceramics.



*Figure 1. The distribution of lead, using the Kernel density plot for the 310 mugs after the first extraction using method EN 1388-1 (9). Mugs for tests 1-3 were chosen within the range 20 to 80 mg of Pb/L. For test 4, mugs with concentrations of approximately 5 mg of Pb/L were selected.*

Four different tests were carried out, all based on method EN 1388-1 (9), but with a variation of extraction solutions, time and temperature as described in Table 1.

Table 1. Extractants, times and temperatures used for the release tests of lead containing mugs.

Test	Extractant	Time	Temperature	Treatment of extract
1	4 % Acetic acid	24 h	22° C	None
	Tap water	24 h	22° C	None
	Orange juice	24 h	22° C	Dry ashing
	Red dry wine	24 h	22° C	Dry ashing
2	4 % Acetic acid	30 min.	80° C	None
	Coffee	30 min.	80° C	Dry ashing
	Tea with lemon	30 min.	80° C	Dry ashing
3	4 % Acetic acid	72 h	8° C	None
	Orange juice	72 h	8° C	Dry ashing
4	4 % Acetic acid	24 h	22° C	None
	Orange juice	24 h	22° C	Dry ashing

The orange juice was reconstituted from concentrated juice according to the recommendation on the container, using tap water (one part juice concentrate plus four parts of water). The pH was determined to 4.0.

The coffee was brewed in an automatic coffee-machine using finely ground coffee and hot (80° C) tap water. The pH was determined to 5.2.

The tea was made from hot water (80° C) in which the tea was extracted (8 g of tea to 1 L of water). Lemon juice was added to the tea in the ratio 20 ml concentrated lemon juice/L of tea. This corresponds to 1/8 of a lemon/mug. The pH was determined to 3.7.

The red wine was an Australian Cabernet Sauvignon with an alcohol strength of 13.5 %. The pH was determined to 3.6.

The pH was measured using a pH-meter Orion SA-520 with an electrode 9104SC Komb Ag/AgCl, EH-glas. The pH buffert Merck 7.00 ± 0.01 and Merck 4.00 ± 0.01 were used.

Extraction solutions consisting of beverages were dry ashed according to NMKL-method no.139 (10) prior to the determination of lead with FAAS (Table 1).

The FAAS instrument was regularly checked by analysing standards for analytical control to safeguard against drift in the system. Analytical blanks were run together with the samples to monitor any contamination.

The mugs used in this project were manufactured at the site of the former

Rörstrand factory. The enamel transfers were made by Print och Design and the 2<sup>nd</sup> firing of the mugs by Porslinsfabriken. All situated in Lidköping, Sweden

## **Results and Discussion**

The objective of this study was to simulate realistic contact conditions for the ceramic articles with realistic foods/beverages, and to compare those results with results using EN 1388-1 (9). Both in the case of orange juice kept in a ceramic jug in the refrigerator for several days, or wine (in a decanter) at room temperature, are actual cases of which the former has resulted in severe lead intoxication. The method is standardized to the use of 4 % acetic acid at 22° C for 24 hours, which means that it does not take into account the actual beverage, storage temperature or time period that can occur in a household.

The consecutive extractions of the six pre-fabricated mugs showed that the lead level in the extracts gradually decreased, and was, after six extractions, close to the maximum limit of 4 mg Pb/L (Figure 2). This shows that the pigment was “stable” and only gradually dissolved into the extractant. The release of lead from a poorly applied (i.e. soluble) lead-glaze is usually rather unpredictable, since the surface of the glaze changes with each extraction. If the surface initially is rather smooth an extraction may result in folds and creases, which increases the surface area considerably. The next extraction may therefore result in a much higher lead level. Since this extraction may dissolve protruding ridges and bumps, the surface may again become smoother, whereby the following extraction may give a lower lead level.

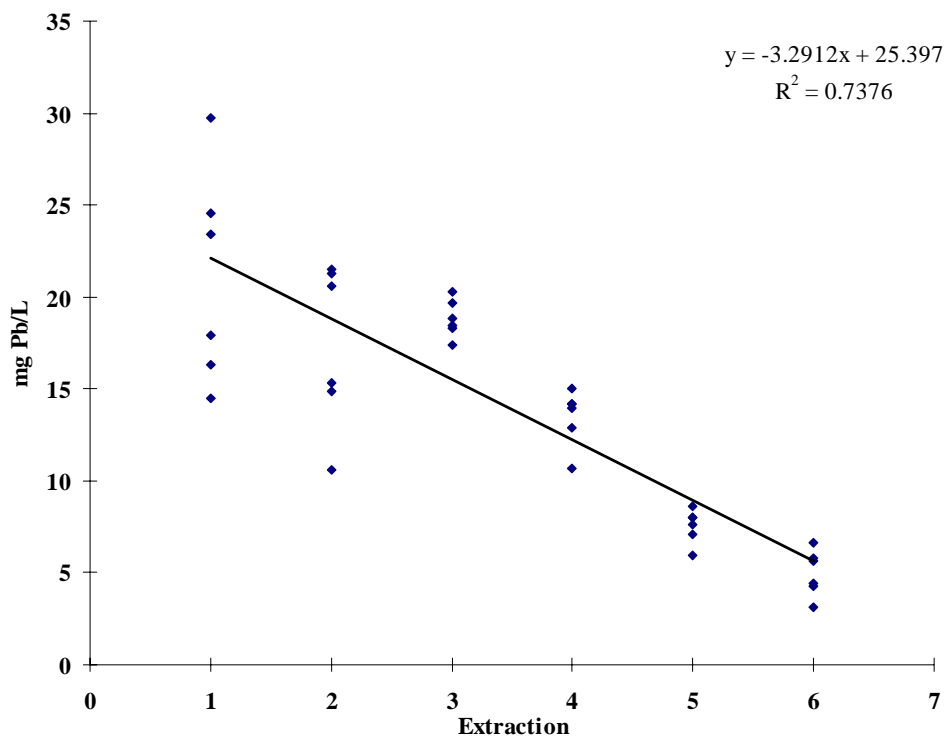


Figure 2. The decrease of extracted lead from six mugs over six consecutive extractions using method EN 1388-1 (9).

**Test 1:** Three different extractants, tap water, orange juice and red dry wine were compared with 4 % acetic acid using 12 mugs (for tap water 6; see Materials and Methods) for each extractant under standard conditions, 24 hours and 22° C. Tap water did not extract any detectable lead (<0.2 mg/l), whereas red wine and orange juice extracted roughly 20 % of the level extracted by 4 % acetic acid (Table 2).

**Test 2:** Mugs may be used for coffee or tea, which can be assumed to have similar extraction properties. In order to test at realistic conditions, 12 mugs each were extracted at 80° C with coffee or tea with lemon (for preparation see Materials and Methods) for 30 minutes. The result showed that coffee is a rather weak extractant whereas lemon tea is nearly as aggressive as 4 % acetic acid. This fact requires that attention should be paid to realistic conditions not yet explored (Table 2).

**Test 3:** The mugs were extracted with orange juice for 72 h (three days) at 8° C, which was considered to be a realistic situation. In the Nordic countries it is common to reconstitute juice from a concentrate. This juice can be kept in a jug in a refrigerator for several days. The result showed that approximately the same amount of lead is extracted during that temperature/time period (72 h/8° C) as compared to orange juice at standard test conditions (24 h/22° C) (Table 2).

**Test 4:** In order to check if lead at the maximum limit for lead (4 mg/L) behaved in the same way as for the higher levels, test 4 was carried out with orange juice using 6 mugs, that showed an average lead-content of 5 mg/L after the first extraction. The ratio of released lead between 4 % acetic acid and orange juice was approximately 3 which is the same as refrigerated 4 % acetic acid and orange juice and similar to what was found at room temperature.

Table 2. Result of tests 1-4

Test	Time/temp	Extractant	pH	Pb mg/L				
				n	Mean	SD	Min	Max
1	24 h/22° C	4 % Acetic acid	2.7	12	25	2.1	22	28
	24 h/22° C	Tap water	7.8	6	<0.2			
	24 h/22° C	Orange juice	4.0	12	4.6	1.2	2.3	5.8
	24 h/22° C	Red dry wine	3.6	12	4.5	0.8	2.5	5.5
2	30 min/80° C	4 % Acetic acid	2.7	12	13	2.6	7.6	16
	30 min/80° C	Coffee	5.2	12	1.7	0.8	0.6	2.8
	30 min/80° C	Tea with lemon	3.7	12	12	2.1	7.5	15
3	72 h/8° C	4 % Acetic acid	2.7	12	17	4.1	7.8	23
	72 h/8° C	Orange juice	4.0	12	4.5	0.8	2.8	5.2
4	24 h/22° C	4 % Acetic acid	2.7	6	1.0	0.2	0.8	1.3
	24 h/22° C	Orange juice	4.0	6	0.3	0	0.3	0.4

### Risk assessments on ceramics

In an ideal world no migration of metals should occur from ceramic articles. However, metals are still used in e.g. pigments or glaze. Lead glaze is known for giving an attractive surface of ceramics, although used less frequently today.

This survey shows that an enamel colour containing lead applied on ceramics, mimicking a lead glaze, produced in an uncontrolled firing process constitute a considerable risk for the consumer. Beverages such as wine, orange juice and lemon tea are frequently consumed from ceramic mugs and jugs. As can be seen

from Table 2 lead levels well above the maximum limit (4 mg/L) were obtained after extraction of mugs with such beverages.

Despite the fact that lead levels in this study cannot entirely be compared with realistic conditions for lead glazed ceramics on the market, the following can be assumed: Assuming a person (60 kg body weight; bw) consuming half a litre of orange juice or tea with lemon or red wine per day at a level of 4 mg Pb/L, would give rise to an intake of 0.033 mg/kg bw and day (0.5 L x 4 mg/60 kg bw) or approximately 0.230 mg/kg bw and week. This value could be compared with the PTWI (provisional tolerable weekly intake) set by JECFA (Joint FAO/WHO Expert Committee on Food Additives) in 1986; 0.025 mg/kg bw for children, and confirmed in 1993 and 2000 (to apply also for adults). The result show that even at this low level of lead, 4 mg/L, the exposure is 10 times higher than the PTWI.

In 2004 the Norwegian Scientific Committee for Food Safety (VKM) carried out a risk assessment for migration of lead, cadmium and barium from ceramic articles (11). The risk assessment concludes on TDI for lead, cadmium and barium. Due to exposure from other sources VKM only accepts a fraction of the TDI allocated to migration from ceramic articles for lead and cadmium.

#### **Use of simulants vs. testing in real food**

It would be a great advantage for future guidelines on ceramics if simulants used for testing of ceramic articles were harmonized with the EU Plastics Directives 2002/72/EC (6), because plastics legislation generally is recognized as a reasonable approach to the migration concept. For articles filled by food industry, the general principle is that migration to food prevails over migration to simulants.

Typically ceramic articles are sold directly to consumer, without having been brought into contact with food. We therefore, for simplicity reasons, suggest to continue using a simulant as a realistic worst case approach. For metals "worst case" generally can be assumed as acidic food, due to the general tendency of metals to be more soluble at a lower pH. Metals generally do not tend to migrate into fatty foods. According to the Plastics Directive 2002/72/EC (6), 3 % acetic acid is used, while traditionally 4 % acetic acid has been used for ceramic articles.

According to this study 4 % acetic acid seems to be an appropriate simulant for acidic food, we therefore suggest to continue using this simulant for ceramics. In this test, both red wine and orange juice extracted approximately 20-30 % of the amount of lead extracted with 4 % acetic acid. Also storing orange juice in a jug in the refrigerator (72 h/8° C), indicated that 4 % acetic acid is an appropriate simulant at room temperature or lower.

The study with lemon tea and coffee showed that tea with lemon has a high extractive power, almost equal to 4 % acetic acid. This fact needs attention and amended test conditions should be considered as shown below.

### **Realistic contact time and temperature**

To follow the concept of migration from the Plastics Directive 2002/72/EC (6), real contact time should be simulated. Realistic contact time for hot drinks such as coffee or tea, acidic soft drinks or wine, which we pour over in a mug or a glass before consumption, is considerable shorter than storage in mugs or bowls.

We propose that a realistic contact time for mugs and glass made of ceramics up to 500 ml, should be tested in contact with acetic acid in 30 minutes at 80° C and 4 hours in 22° C. A combination of these may cover both situations in one single test:

***- Temperature 80° C/30 minutes – letting the article cool down to room temperature for 3 ½ hours - for cups and glass made of ceramics***

For long time storage we may also have a glance at the Plastics Directive 2002/72/EC (6). Ten days at 40° C are indicated. However, 2-3 days in the refrigerator may be seen as a realistic worst case for a jug of juice or wine. Due to higher kinetic rates the 24 hour test at room temperature in contact with acetic acid seems appropriate for long time storage of food in ceramic articles. We therefore suggest:

***- Room temperature for 24 hour - for long term storage in ceramic articles***

Ceramic articles > 500 ml also made for short term contact with a hot liquid, should also be tested for higher temperatures. Here a combination of the two may be applicable:

***- Temperature 80° C/30 minutes – letting the article cool down to room temperature for 23 ½ hours - for ceramics with no restrictions in use***

### **Repeated use**

Lower migration levels after repeated contact with foodstuffs may be an essential factor. Therefore the migration after the article has been used several times is essential. A refined test method should take into account the leaking of lead over time, not only high levels that may be the case when the product is new.

However it is important to notice that in this test no detergent is applied in between the re-fillings. More studies therefore is necessary to evaluate the possible alignment of the migration curve after several times repeated washing and use. Also storing time between washing and repeated contact with acetic acid may be relevant.

### **Acknowledgement**

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