


Metal release from coffee machines and electric kettles

Frederic D. Müller, Christin Hackethal, Roman Schmidt, Oliver Kappenstein, Karla Pfaff & Andreas Luch

To cite this article: Frederic D. Müller, Christin Hackethal, Roman Schmidt, Oliver Kappenstein, Karla Pfaff & Andreas Luch (2015) Metal release from coffee machines and electric kettles, Food Additives & Contaminants: Part A, 32:11, 1959-1964, DOI: [10.1080/19440049.2015.1086929](https://doi.org/10.1080/19440049.2015.1086929)


To link to this article: <http://dx.doi.org/10.1080/19440049.2015.1086929>

 View supplementary material [↗](#)

 Accepted author version posted online: 05 Oct 2015.
Published online: 15 Oct 2015.

 Submit your article to this journal [↗](#)

 Article views: 1822

 View related articles [↗](#)

 View Crossmark data [↗](#)

Metal release from coffee machines and electric kettles

Frederic D. Müller*, Christin Hackethal, Roman Schmidt, Oliver Kappenstein, Karla Pfaff and Andreas Luch

Department of Chemical and Product Safety, German Federal Institute for Risk Assessment (BfR), Berlin, Germany

(Received 8 June 2015; accepted 18 August 2015)

The release of elemental ions from 8 coffee machines and 11 electric kettles into food simulants was investigated. Three different types of coffee machines were tested: portafilter espresso machines, pod machines and capsule machines. All machines were tested subsequently on 3 days before and on 3 days after decalcification. Decalcification of the machines was performed with agents according to procedures as specified in the respective manufacturer's manuals. The electric kettles showed only a low release of the elements analysed. For the coffee machines decreasing concentrations of elements were found from the first to the last sample taken in the course of 1 day. Metal release on consecutive days showed a decreasing trend as well. After decalcification a large increase in the amounts of elements released was encountered. In addition, the different machine types investigated clearly differed in their extent of element release. By far the highest leaching, both quantitatively and qualitatively, was found for the portafilter machines. With these products releases of Pb, Ni, Mn, Cr and Zn were in the range and beyond the release limits as proposed by the Council of Europe. Therefore, a careful rinsing routine, especially after decalcification, is recommended for these machines. The comparably lower extent of release of one particular portafilter machine demonstrates that metal release at levels above the threshold that triggers health concerns are technically avoidable.

Keywords: metal release; food contact materials; coffee machine; electric kettle; ICP-MS; method validation

Introduction

The use of food contact materials (FCM) is often associated with the release of constituents and thus consumer exposure to certain chemicals. The release of metal ions in high concentrations from consumer products and the use of toxic alloying components represent an ongoing problem for surveillance authorities and are also reflected by a large number of notifications in the rapid alert system for food and feed of the European Commission (RASFF; http://ec.europa.eu/food/safety/rasff/index_en.htm). The release of metals from metallic FCM (Fekete et al. 2012; Kamerud et al. 2013) and from electric kettles and coffee machines (Berg et al. 2000) has been discussed in the literature. Hot beverage appliances were in the focus of a study published by Berg and colleagues (2000). Therein, electric kettles were found to release high amounts of nickel (Ni). In 2013 the French Agency for Food, Environmental and Occupational Health and Safety (ANSES) reported a slight increase of the concentrations of chromium (Cr), Ni, copper (Cu) and zinc (Zn) in espresso-type coffee prepared with pod or capsule machines when compared to filter coffee (ANSES 2013). Fully automatic coffee machines, which grind coffee beans followed by the preparation of a single cup of e.g. espresso, were tested by the Chemical and Veterinary Surveillance Office in Stuttgart (CVUA Stuttgart 2007). Release of high concentrations of lead (Pb) and Ni into

food simulants were detected, especially after decalcification of the machines.

Currently there are no release limits defined by European law concerning the leaching of elements from metallic FCM. Article 3(1) of the Commission Regulation (EC) No. 1935/2004 establishes general requirements for the release of constituents of FCM under normal or foreseeable conditions. The constituents must not be released in quantities which could endanger human health or convey an unacceptable change in the composition or organoleptic characteristics of the food. In 2013 the Council of Europe (CoE) passed the resolution CM/Res(2013)9 (CoE 2013) on metals and alloys used in food contact materials and articles. In the technical guide supplementing this resolution, methods for release testing are recommended and specific release limits (SRL) for 21 metals and metalloids (see Table 1) have been proposed (EDQM 2013a). These SRL are based on toxicological evaluations, taking the overall exposure via food into account. For the release of beryllium (Be), vanadium (V), Cr, cobalt (Co), Ni, arsenic (As), molybdenum (Mo), cadmium (Cd), antimony (Sb), mercury (Hg), thallium (Tl) and Pb, transitional limits have been accepted by the CoE (EDQM 2013b). The technical guide supplementing the CoE resolution states that the metal release of articles intended for repeated use should be tested three times (EDQM 2013a). The release of the elements in the third release

*Corresponding author. Email: Frederic.Mueller@bfr.bund.de

Table 1. Detected isotopes, used internal standard and specific release limit (SRL) as proposed by the CoE (EDQM 2013a, 2013b).

Analyte	SRL (SRL acceptable for a transitional period only) [mg/kg food]	Detected isotopes	Internal standard
Lithium (Li)	0.048	⁷ Li	Scandium (Sc)
Beryllium (Be)	0.01 (0.05)	⁹ Be	Scandium (Sc)
Aluminum (Al)	5	²⁷ Al	Scandium (Sc)
Vanadium (V)	0.01 (0.05)	⁵¹ V	Scandium (Sc)
Chromium (Cr)	0.25 (1)	⁵² Cr	Scandium (Sc)
Manganese (Mn)	1.8	⁵⁵ Mn	Scandium (Sc)
Iron (Fe)	40	⁵⁷ Fe	Scandium (Sc)
Cobalt (Co)	0.02 (0.1)	⁵⁹ Co	Scandium (Sc)
Nickel (Ni)	0.14 (0.7)	⁶⁰ Ni	Scandium (Sc)
Copper (Cu)	4	⁶⁵ Cu	Scandium (Sc)
Zinc (Zn)	5	⁶⁶ Zn	Scandium (Sc)
Arsenic (As)	0.002 (0.01)	⁷⁵ As	Rhodium (Rh)
Molybdenum (Mo)	0.12 (0.6)	⁹⁵ Mo	Rhodium (Rh)
Silver (Ag)	0.08	¹⁰⁷ Ag	Rhodium (Rh)
Cadmium (Cd)	0.005 (0.02)	¹¹¹ Cd	Rhodium (Rh)
Tin (Sn)	100	¹¹⁷ Sn	Rhodium (Rh)
Antimony (Sb)	0.04 (0.2)	¹²¹ Sb	Rhodium (Rh)
Barium (Ba)	1.2	¹³⁵ Ba	Rhodium (Rh)
Mercury (Hg)	0.003 (0.015)	²⁰² Hg	Bismuth (Bi)
Thallium (Tl)	0.0001 (0.0005)	²⁰⁵ Tl	Bismuth (Bi)
Lead (Pb)	0.01 (0.04)	²⁰⁶ Pb, ²⁰⁷ Pb, ²⁰⁸ Pb	Bismuth (Bi)

testing should not be higher than the SRL, while the sum of the first and second release testing should not exceed a level sevenfold the SRL.

Eleven electric kettle devices and eight coffee machines, among these two coffee pod machines, three coffee capsule machines and three portafilter espresso machines, were investigated regarding their release of elemental ions into food simulants under conditions of real use.

Materials and methods

Reagents and materials

All chemicals and standard compounds used in this study were of analytical grade unless otherwise stated. A list comprising all chemicals and standard compounds including CAS numbers and potential suppliers is given in the supplementary part (see Table S1). The nitric acid was purified in a duoPUR quartz sub-boiling distillation system (MLS GmbH, Leutkirch, Germany) before use and ultrapure water was obtained using a Milli-Q Advantage A10 water purification system (Millipore GmbH, Schwalbach am Taunus, Germany) equipped with a Millipore Q-POD Element Unit (Millipore GmbH).

The coffee machines and electric kettles were purchased online or in local stores in Berlin.

Release experiments

The synthetic tap water used for the release experiments was prepared according to German industrial norm (DIN 10531 (DIN 2011)) as stated in the aforementioned

guidelines supplementing Resolution CM/Res(2013)9 on metals and alloys used in food contact materials and articles (EDQM 2013a). In short, 300 mg NaHCO₃, 175 mg MgSO₄ and 300 mg CaCl₂ were dissolved in 5 l ultrapure water. The pH value of this solution was than adjusted to 7.5 by adding a solution of nitric acid (0.1 mol/l).

Eleven electric kettles and eight coffee machines, among these three portafilter espresso machines (PF1, PF2, PF3), three coffee pod machines (PM1, PM2, PM3) and two coffee capsule machines (CM1, CM2), were investigated in this study. The brand new coffee machines and electric kettles were set up according the manufacturer's specifications before the first release testing. All the decalcifications were performed using decalcification agents and procedures specified in the respective manuals.

From all coffee machines five consecutive samples of the volume of an espresso cup (25–35 ml) were taken on each single day on 3 days before and 3 days after decalcification of the machines. The samples taken from the coffee machines on these testing days were prepared without ground coffee, pods or capsules.

For the release experiments of the electric kettles three boiling processes were performed before and after decalcification. For each boiling process 0.5 l or the minimum volume specified by the manufacturer of synthetic tap water at room temperature was filled in the kettle. The water was brought to the boil and kept for a further 5 min in the kettle. After cooling to room temperature in PTFE beakers, aliquots were taken for quantification.

Sample preparation of the release solutions

After release testing into synthetic tap water, 0.3 ml of the internal standard solution (Sc, Rh, Bi; 500 µg/l) were added to an aliquot of 1.5 ml of the solution and the volume was made up to 15 ml by adding 3.4% nitric acid solution (200 µg/l Au). The solution contains gold ions (Au³⁺) to prevent potentially released Hg²⁺ from being reduced to Hg and thus from adsorbing at container walls (Thermo Electro Corporation 2003).

If target element levels exceeded the working range (see Tables S3 in the supplementary section), the samples were diluted appropriately.

Determination of elements by ICP-MS

For sample analysis an XSERIES 2 ICP-MS system (Thermo Scientific, Dreieich, Germany) equipped with an SC-4 DX Fast autosampler (ESI Elemental Service & Instruments GmbH, Mainz, Germany) was used. Each measurement was performed in triplicate. The detected isotopes and internal standards used for the respective analytes are given in Table 1. The ICP-MS operating parameters are provided in the supplementary section (Table S2). Data were processed with PlasmaLab 2.5.11.321 (Thermo Scientific).

The calibration standard with the lowest concentration was set to 3% of the respective SRL of the analytes, with exception of tin (Sn) and Cu due to their high SRL (see working ranges in Table S3 in the supplementary section).

Validation scheme

The ICP-MS method was validated for synthetic tap water.

The proposed ICP-MS method was validated in terms of linearity, precision, trueness, limit of detection (LOD) and limit of quantification (LOQ). The validation experiments were performed following the guidelines for performance criteria and validation procedures of analytical methods used in the control of FCM by the joint research centre of the European Commission (Bratinova *et al.* 2009).

For that purpose, a stock solution was prepared using the certified element standard solutions and ultrapure water. The concentrations of the analytes in this stock solution were set to be 20 times higher than the respective SRL as proposed by the CoE (EDQM 2013a). However, due to the high SRL of Sn and Cu, the respective concentrations of these two analytes in the stock solution were lower (Cu: 40 mg/l, SRL: 4 mg/l; Sn: 6.25 mg/l, SRL: 100 mg/l). The stock solution was then added to synthetic tap water, yielding samples that were spiked with three different analyte concentrations. These concentrations corresponded to 8%, 80% and 800% of the SRL of each element except Cu and Sn (see above). Six aliquots of each of these solutions plus non-spiked blank solutions were prepared, processed and measured similar to the real

testing solutions. The experiments were conducted three times on 3 days by different operators.

Prior to the determination of the method performance criteria, the data sets of the different batches were checked for outliers (Grubbs and Dixon test; Kromidas 1999) and normal distribution (Kolmogorov–Smirnov test; Zar 1999).

The LODs and LOQs were determined according to the German industrial norm (DIN) 32645 (DIN 2008) using the blank method. Repeatability (intra-day precision) and reproducibility (inter-day precision) were determined according to the guidelines for performance criteria and validation procedures of analytical methods used in the control of FCM by the joint research centre of the European Commission (Bratinova *et al.* 2009).

Results and discussion

Validation

The quantification method including the sample preparation steps was validated regarding its selectivity, sensitivity, precision and trueness for the matrix synthetic tap water. All elements for which an SRL has been proposed by the CoE guidelines (EDQM 2013a) were included in this validation.

For silver (Ag) inconsistent results and low recoveries were obtained, particularly for the higher concentrations. These findings might be explained by the presence of chloride ions in the synthetic tap water and the resulting formation of insoluble silver chloride. Due to these results, Ag was not considered in this study.

A sufficient selectivity was demonstrated via analysis of accompanying blanks. No interferences were detected in these measurements. Each day the ICP-MS system was optimised towards low formation rate of oxides using a routine protocol as described by the manufacturer of the ICP-MS.

The CoE guidelines on metals and alloys used in FCM (EDQM 2013a) request that the LODs and LOQs of the used quantification method should be lower than 10% and 20% of the SRL, respectively. These requirements were met for all elements. The LODs and LOQs obtained are given in the supplementary section (see Table S3).

To assess the precision of the method, the within-laboratory standard deviation (s_{WR}) under repeatability (intra-day precision) and reproducibility conditions (inter-day precision) were determined following Bratinova *et al.* (see Tables S3 in the supplementary section). The s_{WR} under reproducibility conditions should not exceed the level as calculated by the Horwitz equation (EDQM 2013a). This could be achieved for all analytes and all concentration levels excluding the highest concentration of Cu. In this case, the Horwitz level was exceeded by 1.4%.

By means of the spiked samples, the recovery rates for the analytes were assessed (see Tables S3 in the supplementary section). The CoE guidelines request recovery rates from 80% to 120% (EDQM 2013a). For the synthetic

tap water only the recovery rate for the lowest concentration of Hg (68.2%) lay outside of this range.

Coffee machines: release into synthetic tap water

The release patterns of all machines differed from each other. Most elements were released by the portafilter machines – qualitatively and quantitatively. In general, the release of a specific element continuously decreased from the first to the fifth sample taken in the course of 1 day. The release measured on a subsequent testing day was lower, but again increased significantly after decalcification of the machine.

Of all the elements for which the method has been validated, only lithium (Li), Be, Hg and barium (Ba) were not detected in the release testing solutions of the portafilter machines. The only elements released by these machines in concentrations above 3% of their respective SRL were Pb, Tl, Cd, Ni, Zn, As, manganese (Mn), Cr and Mo (see Tables S4–S9 in the supplementary section). Among these, Pb, Ni, Mn, Cr and Zn were detected in concentrations exceeding their individual SRL. The release patterns of the two portafilter machines PF1 and PF2 with regard to Pb are shown in Figure 1. Specifically, machine PF2 revealed with a high increase of the Pb release after decalcification. Before decalcification, the highest release of this machine was quantified as 0.54 µg/kg Pb in the first sample taken on the first testing day. No Pb release above the lower limit of the working range (3% SRL) could be detected on the second and third day prior to the decalcification. Directly after treatment of the machine with the decalcification agent, a release level of 1600 µg/kg Pb could be detected, a value >150 times the SRL. Starting with the fifth sample taken on the first

testing day after decalcification, the Pb concentrations again returned to a level lower than the SRL. In addition, Pb concentrations in the first and second sample taken on the two following days still exceeded the SRL. Machine PF1 constantly released Pb in concentrations above the SRL before decalcification was performed (10–21 µg/kg). Directly after decalcification, the Pb release was increased to 44 µg/kg. Only the samples starting with the second cup taken on the second testing day after decalcification complied with the SRL. As mentioned above, the SRL for Pb was set at 10 µg/kg food by CoE. For a transitional period it considers lead releases up to 40 µg/kg food as acceptable (EDQM 2013b). Taking this higher limit and the measurement uncertainty into account, only machine PF2 did not fulfil the requirements of the CoE.

Besides Pb, the concentrations of some other elements found in the first sample taken from machine PF2 after decalcification exceeded their respective SRL (see Table S7 in the supplementary section). In this sample Ni, Cr, Mn and Zn were found in concentrations two- to fourfold higher than their respective SRL. The course of the release of these elements from machine PF2 is shown in Figure 2. Similar to Pb, however, a higher SRL is accepted by the CoE in the case of Ni (700 µg/kg) and Cr (1000 µg/kg), but only for a transitional period (EDQM 2013b). Given this regulation, overall the detected releases of these two elements again concur with the limits as set by the CoE.

In comparison, the tested coffee pod and capsule machines released significantly less elements and in lower concentrations than the portafilter machines. Releases above 3% of the SRL from the pod machines were only detected for Pb, Ni, Mn, As, V, Cr, Co and Zn (see Tables S10–S19 in the supplementary section). All of

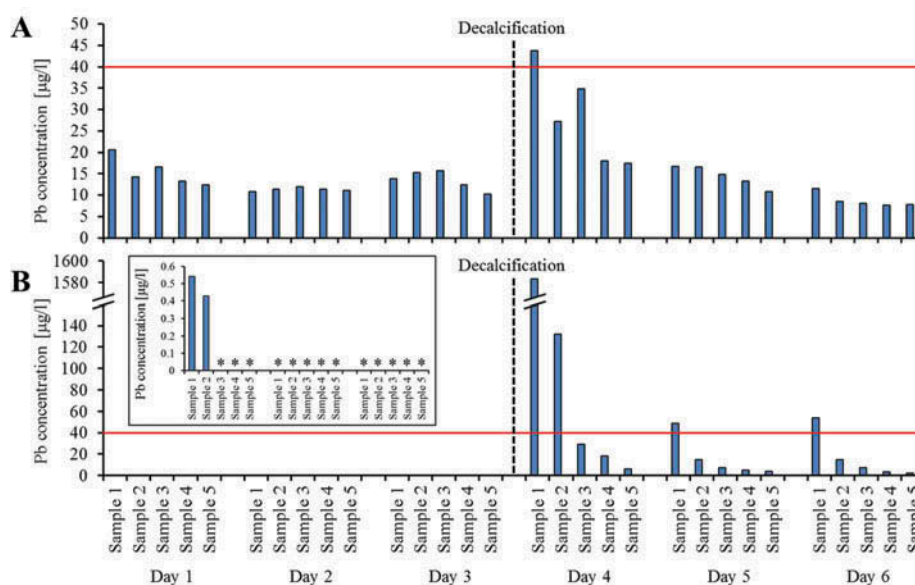


Figure 1. (colour online) Pb concentrations in the samples taken from the portafilter machines (A) PF1 and (B) PF2 in µg/l. The red line represents the specific release limit acceptable for a transitional period only (SRL; 40 µg/l; see Text for further explanation). The inset shows the Pb concentrations in the PF2 samples prior to decalcification. *Value below working range.

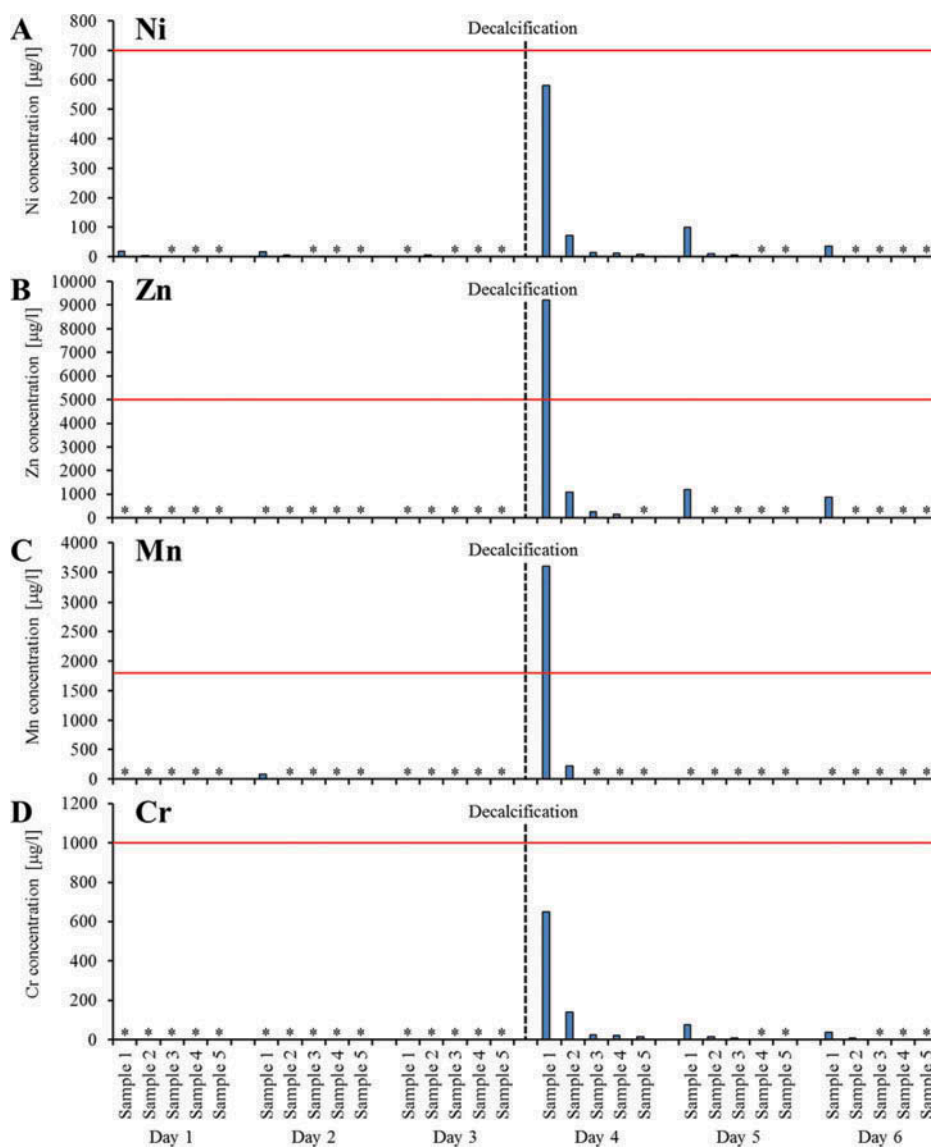


Figure 2. (colour online) Course of the release of (A) Ni, (B) Zn, (C) Mn and (D) Cr in $\mu\text{g/l}$ of portafilter machine PF2. Next to Pb, these were the only elements which exceeded their individual SRL in portafilter release solutions. The red line represents the SRL, in the case of Ni and Cr the depicted SRL is acceptable for a transitional period only (see Text for further explanation). *Value below working range.

the concentrations measured were considerably lower than the SRL with the exception of Ni as found in pod machine PM3. Although this machine showed only low releases before the decalcification step, the first sample taken after decalcification came up with $780 \mu\text{g/kg}$ Ni in the release testing solution. Subsequently, Ni releases were detected at all days following decalcification, but in general remained within the transitional limit taking the measurement uncertainty into account.

Release from electric kettles

For all operations, including the setting up of the kettles, the aforementioned synthetic tap water was used. Three

boiling steps were performed before and after decalcification of the kettles. Per boiling process either 0.5 l or the minimum volume as specified by the manufacturer of synthetic tap water were used. In none of these experiments could high releases be detected. Only the elements Pb, Ni and Cu could be quantified within the working range. The maximum releases of these metals (Pb: $1.7 \mu\text{g/l}$; Ni: $4.9 \mu\text{g/l}$; Cu: $96 \mu\text{g/l}$) were well below their respective SRLs.

Conclusions

Here it is shown that the protocol used on ICP-MS-based quantification of metal ion release into the matrix synthetic

tap water is fit-for-purpose. This method was validated for all elements for which an SRL has been proposed via the CoE guidelines (EDQM 2013a), with the exception of Ag.

Several elements were found to be released from the tested coffee machines, i.e. Mn, Cr, Zn, Ni and most prominently Pb, in concentrations higher than the SRL as proposed by the guidelines on metals and alloys used in FCM. In general, the extent of release decreased from the first to the last sample taken within a single day, whereas the highest releases were detected directly after decalcification of the machines. Based on this, it is to be recommended to strictly perform extensive rinsing steps subsequent to the decalcification procedure before the first use.

In general, the highest releases were found for the espresso portafilter machines, both qualitatively (number of different elements) as well as quantitatively (level of individual elements). With two among three tested machines, Pb was found leaching at levels capable of prompting health concern.

EU legislation prohibits the release of constituents of FCM in quantities which could endanger human health and requires FCM to be manufactured in compliance with good manufacturing practice (Article 3(1) of Commission Regulation (EC) No. 1935/2004). The CoE guidelines state that the release of Pb from FCM should be reduced as much as possible (EDQM 2013a). This is based on the observation that the level of Pb intake already exceeds doses that might cause adverse health effects in certain human subpopulations. Because one particular portafilter machine did not release any element at concentrations that causes concern, the aim stated in the CoE guideline reveals technically achievable.

In contrast to coffee machines and to findings reported in previous studies, no considerable element release was found in this study for electric kettles. As expected, Ni releases were detected, but to an extent far below the SRL as proposed by the CoE.

Disclosure statement

No potential conflict of interest was reported by the authors.

Supplemental data

Supplemental data for this article can be accessed here: <http://dx.doi.org/10.1080/19440049.2015.1086929>.

References

Berg T, Petersen A, Pedersen GA, Petersen J, Madsen C. 2000. The release of nickel and other trace elements from electric kettles and coffee machines. *Food Addit Contam.* 17:189–196.

- Bratinova S, Raffael B, Simoneau C. 2009. Guidelines for performance criteria and validation procedures of analytical methods used in controls of food contact materials (EUR 24105 EN). 1st ed. Ispra: European Commission, Joint Research Centre.
- [CVUA Stuttgart] Chemical and Veterinary Investigation Office Stuttgart, editor. 2007. Nickel und Blei aus dem Kaffeefullautomaten [Nickel and lead leached by automatic coffee machines]. In: *Lebensmittelüberwachung und Tierseuchendiagnostik: Jahresbericht 2007* [Food inspection and animal disease diagnostics: Annual Report 2007]. p. 44–45.
- [CoE] Council of Europe. 2013. Resolution CM/Res(2013)9 on metals and alloys used in food contact materials and articles. Adopted by the Committee of Ministers on 11 June 2013 at the 1173rd meeting of the Ministers' Deputies.
- [EDQM] European Directorate for the Quality of Medicines & HealthCare of the Council of Europe. 2013a. Metals and alloys used in food contact materials and articles: a practical guide for manufacturers and regulators. 1st ed. Strasbourg: Council of Europe (CoE).
- [EDQM] European Directorate for the Quality of Medicines & HealthCare of the Council of Europe. 2013b. Letter from the EDQM addressed to national authorities concerned with the surveillance of food contact materials of 18 November 2013. [cited 2015 Jul 29]. Available from: https://www.edqm.eu/medias/fichiers/recommendation_by_the_p_sc_emb_for_acceptable_deviation_from_the_srl_during_a_transitional_period_in.pdf
- Fekete V, Deconinck E, Bolle F, Van Loco J. 2012. Modelling aluminium leaching into food from different foodware materials with multi-level factorial design of experiments. *Food Addit Contam Part A.* 29:1322–1333.
- [ANSES] French Agency for Food, Environmental and Occupational Health and Safety. 2013. "Espresso" machine coffee pods and capsules do not increase exposure to chemical contaminants. [updated 2013 Sep 25; cited 2015 Jan 27]. Available from: <https://www.anses.fr/en/content/espresso-machine-coffee-pods-and-capsules-do-not-increase-exposure-chemical-contaminants>
- [DIN] German Industrial Norm. 2008. Chemical analysis - decision limit, detection limit and determination limit under repeatability conditions - terms, methods, evaluation. DIN 32645:2008-11. Berlin: Beuth.
- [DIN] German Industrial Norm. 2011. Food hygiene - production and dispense of hot beverages from hot beverage appliances - hygiene requirements, migration test. DIN 10531:2011-06. Berlin: Beuth.
- Kamerud KL, Hobbie A, Anderson KA. 2013. Stainless steel leaches nickel and chromium into foods during cooking. *J Agric Food Chem.* 61:9495–9501.
- Kromidas S. 1999. Validierung in der Analytik: Die Praxis der instrumentellen Analytik [Validation in analytics: the practice of instrumental analysis]. Weinheim: WILEY-VCH.
- Thermo Electro Corporation. 2003. Application Note AN_EO612: X Series ICP-MS Clinical Applications Note 3: Determination of Hg in urine. 3 p.
- Zar JH. 1999. *Biostatistical Analysis*. 4th ed. Upper Saddle River (NJ): Pearson Education.