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# Batch leaching tests of motherboards to assess environmental contamination by bromine, platinum group elements and other selected heavy metals



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### HIGHLIGHTS

- Metals stripping from motherboards depend on the used leaching solution.
- Water, acetic acid, nitric acid and synthetic acid rain were used as leaching media.
- Heavy metals including PGEs were preferably leached with synthetic acid rain.
- Acetic acid solution dissolved the highest amounts of bromine.
- Motherboards should be considered as hazardous and treated prior to disposal.

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# GRAPHICAL ABSTRACT



# ABSTRACT

In this study, a batch leaching test was executed to evaluate the toxicity associated with chemicals contained in motherboards. The leaching solutions used were distilled water, nitric acid, acetic acid and synthetic acid rain solution. A total of 21 elements including Ag, As, Au, Br, Cd, Co, Cr, Cu, Hf, Ir, Mn, Ni, Os, Pb, Pd, Pt, Rd, Rh, Se, U and Zn were analyzed. In this study, the pH values of all the leachates fell within the range of 2.33–4.88. The highest concentrations of metals were obtained from the acid rain solution, whilst the maximum value of bromine was achieved with solution of acetic acid. Appreciable concentrations of platinum group elements were detected with concentrations around 3.45, 1.43, 1.21 and 22.19  $\mu$ g L<sup>-1</sup> for Ir, Pd, Pt and Rh, respectively. The different leaching of the motherboards revealed the predominant presence of the toxic substances in the leached from the e-waste.

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# 1. Introduction

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http://dx.doi.org/10.1016/j.chemosphere.2015.08.035 0045-6535/© 2015 Elsevier Ltd. All rights reserved. The global electronic waste (e-waste) production is assessed at 20–50 Mt per year (Robinson, 2009), equal to 1–3% of the estimated global urban waste production (1636 Mt) (Cobbing, 2008; OECD, 2008). Personal computers (PCs), cell phones and TVs con-

tributed 5.7 Mt in 2010 and the estimates for 2015 amount to 9.8 Mt (Widmer et al., 2005). Electronic computers with an average 3-year life cycle (Betts, 2008) contribute to the greatest extent to the total e-waste flow. Most e-waste is disposed in landfills, because effective reprocessing technology, which recovers the valuable materials with minimal environmental impact, is expensive (Robinson, 2009). According to Frazzoli et al. (2010), the most chemicals implicated in e-waste, are responsible of endocrine disruption and effects on the nervous and immune systems. Also, this hazardous components cause critical changes in pre-, postnatal stages of development of the most species. The PCs weigh from 8 to 14 kg; the two heaviest components are the housing and steel frame that constitutes 65–85% of the total weights of the PCs. The third largest components are the motherboards which account for 6–9%. However, this component should be treated as hazardous waste when being discarded because of the high concentration of heavy metals (Li et al., 2009a).

The implementation of the appropriate toxicity characteristic leaching procedure test (TCLP) (USEPA, 1999) has shown that ewaste discarded at urban waste dumping sites does not produce leachates with heavy metals concentrations exceeding the environmental limits established in Code of Federal Regulations, D-list (Spalvins et al., 2008). Nevertheless, this chemical cocktail generated from several electronic items was toxic for aquatic organisms (Dagan et al., 2007). Another factor to consider is that most components are metallic electronic waste and the leaching of pollutants begins with corrosion (Li et al., 2009b). TCLP is used to simulate the heavy metals leach from the PC components in the worstcase scenario in landfills. However, Jang and Townsend (2003) concluded that the amounts of heavy metals leached were much greater in the TCLP extraction fluid than in the landfill leachate, suggesting that the TCLP is over-conservative when used to estimate the leaching of heavy metals from e-waste in landfills. It could be because TCLP requires the samples to be crushed into particles of 9.5 mm; however, not all components could be reasonably sampled in such a small size. In this context, Li et al. (2009b) tested the leaching from heavy metals and argues that most ewaste components are metallic and the leaching of contaminants starts with corrosion and the 18 h<sup>-1</sup> extraction in the TCLP test is not sufficient for the corrosion and leaching to complete.

Platinum group elements, known as PGEs (Pt, Pd, Rh, Ru, Ir and Os) can be naturally found only at very low concentration in the earth crust. These metals are predominantly used in automobile catalytic converters to reduce toxic exhaust fumes (Osterauer et al., 2011; Wiseman and Zereini, 2011). The PGEs are also used in electronic components because of their properties such as high melting points, high resistance to corrosion and low electrical resistance (Balcerzak, 2002). However, they have been associated with asthma, nausea, increased hair loss, increased spontaneous abortion, dermatitis and other serious health problems in humans (Pawlak et al., 2014). According to some experts, PGEs are mainly emitted in metallic form, and thus exhibit low toxicity. However, some of them are converted into soluble forms, which become bioavailable and begin to constitute a serious threat to animals and plants (Ravindra et al., 2004; Moldovan, 2007). The concentration of these metals has increased significantly in the last decades in diverse environmental matrices; like airborne particulate matter, soil, roadside dust and vegetation, river, coastal and oceanic environment (Merget and Rosner, 2001; Ek et al., 2004; Ravindra et al., 2004; Pawlak et al., 2014). The content of Pt in the tested food products from the Australian market varied and amounted to, e.g. 8.11  $\mu$ g kg<sup>-1</sup> in chicken liver and 0.13  $\mu$ g L<sup>-1</sup> in milk. Daily consumption of Pt was estimated at 1.73  $\mu$ g for men and 1.15  $\mu$ g for women (Pawlak et al., 2014). Hence, the determination of PGEs traces in environmental matrices requires a highly sensitive analytical methodology.

Bromine is another contaminant typically encountered in ewaste, which is likely the major source of bromine in the environment (Choi et al., 2009). The harmful effects of bromine on the body come from its interaction with iodine, which can lead to thyroid disease. Moreover, the bromine dumped in the environment has very negative health effects on daphnia, fish, lobsters and algae. Organobromine compounds are also damaging to mammals, especially when they accumulate in the bodies. The most important effects on animals are nerve damage and next to that deoxyribonucleic acid damage, which can also enhance the chances of development of cancer. Organobromine compounds are not very biodegradable; when they are decomposed inorganic bromines will consist. These can damage the nerve system when high doses are absorbed (Fu et al., 2011). In this context, primitive recycling of e-waste has become a global environmental problem introducing bromine and organic brominated compounds into soils and water resource.

There have been a number studies used to classify the hazards of e-waste by TCLP test, however, to our knowledge this is the first attempt of studying PGEs from electronic devices as potential pollutants of landfill. The main objective of this study was to examine the leaching of toxic PGMs and bromine from motherboards using different extraction fluids. This study will provide insight into how the PGMs and bromine from e-waste could pollute the landfills. Bromine, PGEs and fourteen heavy metal elements including Ag, As, Au, Cd, Co, Cr, Cu, Hf, Mn, Ni, Pb, Se, U and Zn were examined.

#### 2. Sample treatment and methods

#### 2.1. Sample preparation

Motherboards were tested for bromine and twenty metals without being reduced to a small size according to Li (2009). Four leaching solutions were used: distilled water (DW) had an initial pH of 4.8; nitric acid 0.1 M (NA) pH 3.68; acetic acid 0.5 M (AA) pH 4.20 and a synthetic acid rain solution (AR). This solution was prepared adding a percent mixture 60/40 weight of sulfuric acid and nitric acid to ultrapure water (18.2 M $\Omega$  cm) until the pH was 4.20  $\pm$  0.05. The motherboards were placed into polytetrafluoroethylene (PTFE) vessels within the different solution at a liquidto-solid ratio of 20:1 on weight basis to evaluate the mobility of metals. The collected samples were filtered to determine the concentrations of the different elements. The total leaching period was 120 days.

## 2.2. Analysis of leachate

The total contents of fourteen heavy metals in the motherboards were determined using an ELAN DRC-e quadrupole ICP-MS instrument (Perkin Elmer SCIEX, Thornhill, Canada). An HFresistant and high performance perfluoracetate (PFA) nebulizer model PFA-ST, coupled to a quartz cyclonic spray chamber with internal baffle and drain lines, cooled with the PC<sup>3</sup> system from ESI (Omaha–NE, USA) was used.

For calibration purposes, six concentration levels were prepared and calibration curve were built for all elements. Each level of concentration was made in quintuplicate. The calibration curves were linear, with correlation coefficients with values above 0.995. The limit of detection (LOD) and the limit of quantification (LOQ), were calculated as the concentration of metals required to yield a concentration equal to 3.3 and 10 times the standard deviation of the blank records following IUPAC recommendations (Currie, 1999). The solutions used were acetic acid, distilled water, nitric acid and synthetic acid rain solution. The elements analyzed and their detection limits ( $\mu$ g L<sup>-1</sup> in parentheses) were: Ag (0.08), As (0.02), Au (0.01), Cd (0.003), Co (0.008), Cr (0.05), Cu (0.009), Hf (0.002), Ir (0.005), Mn (0.01), Ni (0.02), Pb (0.004), Pd (0.01), Pt (0.003), Rh (0.01), Os (0.04), Se (0.08), U (0.001) and Zn (0.04). Special attention was paid to the control of spectral interference, especially in the case of Pd and Rh. Mathematical corrections were used to take into account the isobaric spectral overlaps.

The determinations of Br were performed in DRC mode (dynamic reaction cell), measuring BrO<sup>+</sup> at m/z 95 using O<sub>2</sub> (6.5 mL min<sup>-1</sup>) as reaction gas. Thus, the polyatomic ion interference <sup>38</sup>Ar<sup>40</sup>Ar<sup>1</sup>H<sup>+</sup> at m/z 79 was avoided. The argon and oxygen gas with a minimum purity of 99.996% were supplied by Air Liquide (Córdoba, Argentina).

All measurements of pH were performed using a Thermo Orion Dual-Star pH meter equipped with Thermo Scientific Orion ROSS Ultra Refillable pH/ATC Triode Combination Electrode. The electrode was calibrated with Orion standard pH 4.01 and 7.00 buffers.

#### 2.3. Quality assurance/quality control

All leaching experiments were run in triplicate. The quality of analytical processes was assessed by checking the reproducibility of calibration, blanks, sample triplicates and recovery processes. For each batch of 10 samples a solvent blank and procedural blanks were processed to check for contamination. Spike-recovery tests were carried out to check the accuracy. For this purpose, aliquots of the leached solution were fortified with known concentrations of each analyte; ensuring a recovery in true sample matrix. Duplicate spiked samples were analyzed each 15 samples. The accuracy of the proposed methodology was then evaluated as recoveries of the spikes as 100\*[(found value - base value)/added amount]. The results were always better than 97.2%, showing that the procedure is accurate. Finally, at least two control standards (low and high level) were included in each run to confirm calibration validity each 20 samples and also at the end of each batch analysis.

# 3. Results and discussion

# 3.1. Chemical analysis

As discussed in Section 2.1, batch leaching tests were run in parallel and supernatant aliquots were sampled at different times (from 0 to 120 days). The collected samples were further analyzed to determine target elements and pH values throughout the entire process.

#### 3.1.1. Trace metal determination by ICP-MS

All metal determinations were accomplished by conventional ICP-MS, using a microconcentric nebulizer and a quartz cooled cyclonic spray chamber as discussed above. Before elemental analysis of leachates, an overall instrument optimization including RF Power (watts), nebulizer Ar flow rate (mL min<sup>-1</sup>) and isotope selection, was carried out aiming to reduce matrix effects and possible spectral overlaps. The optimized operational conditions of the ICP-MS analysis are shown in Table 1.

#### 3.1.2. Measurement of pH during leaching tests

Metal dissolution of the e-waste materials are expected to be very different in acidic medium compared to alkaline ones, as predicted by simple acid-base equilibrium principles. Consequently, accurate pH measurements are required to reach deeper insights about the chemical behavior of the motherboards in each leaching solution. In this study, pH profiles in terms of time were drawn in Fig. 1. The obtained profiles from DW, NA and AR leachates showed similar trends reaching a final pH around of 2.3, whilst the AA leachate descended down to pH 3.34. In the case of AR leachates, a different pattern behavior was advised at the starting sampling times, when the pH values increased up to 5.0, and about sixtieth day decreased again to reach the final value of 2.32.

## 3.2. Batch leaching tests from motherboards

The TCLP method is used like regulatory test to rapidly screen which solid wastes warrant more stringent management as hazardous wastes. However, the TCLP was not intended, per se, to be a method for predicting pollutant leachate concentrations from a typical landfill where a potential hazardous waste is disposed (Jang and Townsend, 2003). The leaching of pollutants from solid can be evaluated by constructing and operating simulated landfills. These tests, however, can be time consuming and expensive (Gould et al., 1988; Farquhar, 1989). According to Li et al. (2009b), the leaching levels of different metals were much higher in TCLP extraction fluid than in synthetic precipitation leaching procedure (SPLP). Nevertheless, TCLP may thus be more conservative for some elements (and some wastes) and less conservative for others (Hooper et al., 1998). In this context, it is necessary select the solution which extracts the highest amounts of the analytes of interest to avoid TCLP underestimate the amount of metals leached in landfill leachate.

The results of the composition analysis of leachate from the motherboards are summarized in Table 2. The mean values and the standard derivations were calculated based on triplicate measurements. Among the 21 elements analyzed, the elements detected were bromine, four PGEs (Ir, Pd, Pt and Rh), and fourteen heavy metals (Ag, As, Au, Cd, Co, Cr, Cu, Hf, Mn, Ni, Pb, Se, U and Zn). Most of the elements exhibited a variable concentration with time and the concentrations among the solutions were dissimilar.

#### 3.2.1. Heavy metals leaching

The highest concentrations were obtained from the AR and NA, while leaching with DW showed lowest concentration. Among the detected metals, Ag, As, Cd, Cr, Ni, Pb, Se, U and Zn are on the EPA list (D-list) of the toxicity characteristic substances for TCLP (USEPA, 1999). The amounts of Cd, Ni and Zn leached by AR, NA and AA solution were greater than others elements. Furthermore, the leached by AR was significantly higher than NA. Although AR and NA solutions had a final pH of 2.3, AR in the first sixty days had a pronounced increase of pH, which would seem to be decisive in the leaching of heavy metals.

Uranium was detected in NA, AR and DW leaching solutions; however it was not possible to determine its presence in AA, because in experimental pH values (3.33-4.24), the predominant species is UO<sub>2</sub> <sup>2+</sup>. Under these conditions the uranyl ion precipitates in the presence of acetate ion (Lide, 2006).

#### 3.2.2. Leaching of PGEs

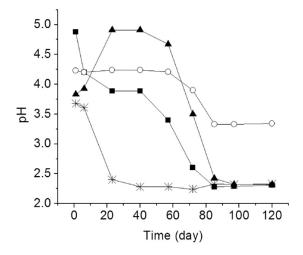
The cumulative amounts of PGEs detected over time are shown in Fig. 2. The values for each time were obtained by multiplying the concentrations by the volume of the extraction fluid in the leaching vessel, then adding to the values of the previous time. Thus, the slopes of the cumulative curves are the rates of leaching of the elements in  $\mu$ g d<sup>-1</sup>. Unexpectedly, Os and Ru were not detected in the leachate from any of solutions.

In the AR batch leaching tests, Ir concentrations ranged from 0.41 to 3.45  $\mu$ g L<sup>-1</sup>, Pd concentrations ranged from 0.14 to 1.43  $\mu$ g L<sup>-1</sup> and Pt concentrations ranged from 0.02 to 1.21  $\mu$ g L<sup>-1</sup>. Rhodium had the highest concentrations for PGEs leaching (22.19  $\mu$ g L<sup>-1</sup>) with a minimum value of 0.21  $\mu$ g L<sup>-1</sup> and

#### Table 1

Instrument settings and data acquisition parameters for DRC-ICP-MS.

Sample uptake rate $(\mu L \min^{-1})$	1000
Sample introduction	Nebulizer model PFA-ST, coupled to a quartz cyclonic spray chamber with internal baffle and drain line, cooled with the PC3 system from ESI
	(Omaha–NE, USA)
RF power (W)	Standard mode: 1100
	DRC mode: 1300
Gas flow rates (L min <sup>-1</sup> )	Standard mode: Plasma, 13.5; auxiliary, 1.2; nebulizer, 0.85
	DRC mode: Plasma, 13.5; auxiliary, 1.2; nebulizer, 0.85
Interface	Ni cones (sampler and skimmer)
Cell gas	BrO+; Reaction gas: O <sub>2</sub>
Standard mode	${}^{107}\text{Ag}^+, {}^{75}\text{As}^+, {}^{197}\text{Au}^+, {}^{111}\text{Cd}^+, {}^{59}\text{Co}^+, {}^{52}\text{Cr}^+, {}^{63}\text{Cu}^+, \\ \text{Hf}^+, {}^{193}\text{Ir}^+, {}^{55}\text{Mn}^+, {}^{60}\text{Ni}^+, {}^{108}\text{Pb}^+, {}^{106}\text{Pd}^+, {}^{195}\text{Pt}^+, {}^{103}\text{Rh}^+, {}^{192}\text{Os}^+, {}^{82}\text{Se}^+, {}^{238}\text{U}^+, \\ \text{and}, {}^{66}\text{Zn}^+, {}^{106}\text{Pd}^+, {}^{106}\text$
Scanning mode	Peak hopping
Dwell time (ms)	Standard mode: 30
	DRC mode: 50
Number of replicates	10



**Fig. 1.** Measurement of pH at different times throughout the entire leaching studies (Acetic acid (-o-), Acid rain (-▲-), Distilled water (-■-) and Nitric acid (-\*-).

Table 2 Results of metal leachability for the different solutions expressed in  $\mu g \ L^{-1} \pm$  standard deviation (n = 3).

	Nitric acid	Acid rain	Distilled water	Acetic acid
Ag	$2.59~\pm~1.35$	$3.52~\pm~1.61$	0.11 ± 0.08	0.24 ± 0.11
As	$5.12~\pm~1.35$	$6.75~\pm~3.21$	$0.14\pm0.08$	$0.24\pm0.14$
Au	$0.81~\pm~0.35$	$2.32~\pm~0.72$	$0.33\pm0.16$	$0.05\pm0.01$
Cd	$8.63^{a} \pm 4.35$	$18.4^{a}~\pm~13.6$	$19.8\pm3.7$	$1.44\pm0.45$
Со	$59.2~\pm~21.1$	$122.8~\pm~43.6$	$0.13\pm0.06$	$18.8\pm10.4$
Cr	$43.1~\pm~16.85$	$187.5~\pm~47.5$	$1.08\pm0.38$	$5.53 \pm 2.32$
Cu	$176.7~\pm~38.4$	$332.5 \pm 133.1$	$37.3 \pm 12.7$	$49.3 \pm 15.3$
Hf	$0.11~\pm~0.05$	$1.22~\pm~0.31$	ND	$0.014\pm0.004$
Mn	$0.41^a~\pm~0.16$	$0.85^{\texttt{a}}~\pm~0.35$	$4.1~\pm~1.8$	$0.22^{a} \pm 0.07$
Ni	$13.7^{a} \pm 9.3$	$33.5^{a} \pm 8.1$	91.5 ± 37.1	4.15 <sup>a</sup> ±1.45
Pb	$79.7~\pm~17.7$	$98.1~\pm~21.7$	$157.6 \pm 35.4$	$197.1 \pm 21.3$
Se	$4.34~\pm~1.25$	$47.7~\pm~14.9$	$5.11~\pm~1.92$	$9.91 \pm 1.55$
U	$0.05~\pm~0.02$	$0.08~\pm~0.03$	$0.024\pm0.005$	ND
Zn	$3.11^a~\pm~1.09$	$14.3^a~\pm~4.9$	$30.1\pm6.8$	1.32 <sup>a</sup> ±0.31

<sup>a</sup> Value expressed in mg L<sup>-1</sup>, ND: no detected element.

an average of  $3.92 \ \mu g \ L^{-1}$ . The huge difference between AR and the other solutions is due to these compounds are soluble in a solution consisting of nitric and sulfuric acid (Balcerzak, 2002; Park and Fray, 2009). Moreover, these results are supported by previous

research about PGEs extraction from waste electronics and electrical equipment. In this study, the presence of Ag, Sn and Zn could affect the dissolution of PGEs; so the utilization of HNO<sub>3</sub> allowed an appropriated dissolution and reduction of interferences in the ICP-MS (Sheng and Etsell, 2007; Park and Fray, 2009). Although good leaching yields were observed in the presence of nitric acid, much better results were obtained when sulfuric acid was added.

# 3.3. Comparison of bromine leachate results

Fig. 3 displays the bromine concentrations in the motherboard samples from the different solutions. Bromine was detected in all samples, with a concentration range of 0.27–3.13 mg L<sup>-1</sup>. The maximum values of bromine were achieved with solutions of AR and AA. Nevertheless, the AR solution reached the maximum value after 120 days, whilst AA did it after approximately 70 days. In addition, the geometric mean concentration in DW (1.21 mg L<sup>-1</sup>) was twice lower than AA (2.31 mg L<sup>-1</sup>); while the concentration of NA was one unit under the final concentration for AA. The accumulated values of bromine for the different leachates were 14.82 mg, 8.57 mg, 9.66 mg and 15.01 mg for AR, NA, DW and AA respectively.

# 4. Conclusions

This study showed that the best leaching of metal of the ewaste was carried out with synthetic acid rain solution followed of nitric acid; but acetic acid was more appropriate for the determination of bromine. Interestingly, pH played an important role in the leachability of the different metals. An increase of pH in the early days of extraction could favor the leachability of metals. As conclusion, the authors recommend the utilization of a synthetic solution of acid nitric and sulfuric acid for the study of e-waste leaching of heavy metals and acetic acid for bromine.

Motherboards are a hazardous source of heavy metals (including uranium and platinum group elements) and bromine. The different leaching of the motherboards revealed the predominant presence of the toxic substances in the leach from the e-waste. Moreover, most data demonstrated exponential distributions, suggesting that the amounts of these elements which are leaching into the environment through landfilling are rapidly and continuously increasing. Furthermore, this could be dangerous and even more magnified with acid rain fallout.

Finally, according to this study, landfill leachate should be considered a serious environmental, especially when electronic waste is present. Ideally, this type of waste should be collected and treated prior to disposal.

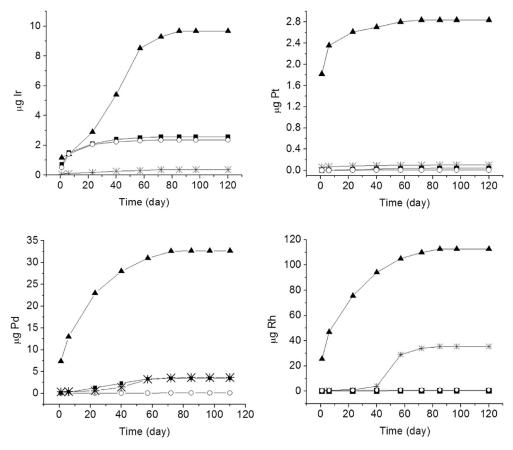
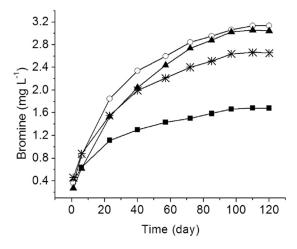


Fig. 2. Cumulative amounts leached (µg) out over time (day) from different motherboards (Acetic acid (-o-), Acid rain (-A-), Distilled water (-A-), and Nitric acid (-\*-).



**Fig. 3.** Comparisons of bromine leaching from acetic acid (- $\circ$ -), acid rain (- $\blacktriangle$ -), distilled water (- $\blacksquare$ ;-) and nitric acid (- $\ast$ -).

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