

Article

Evaluation of US EPA Method 3052 Microwave Acid Digestion for Quantification of Majority Metals in Waste Printed Circuit Boards

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Abstract: Metal content determination is one of the critical aspects of preparing electronic waste for metal recycling. In spite of the fact that end-of-life printed circuit boards are considered to be a secondary resource reservoir, no standard procedure exists for determining the total metal content in this heterogeneous multicomponent material containing plastics, metals, alloys and ceramics. We investigated the utilization of United States Environmental Protection Agency (US EPA) microwave acid digestion (Method 3052) and various modifications of this procedure for effective releasing of Cu, Fe, Ni, Pb and Zn from waste printed circuit boards (WPCBs) from mobile phones. The maximum contents of Cu (22.6 wt.%), Fe (5.0 wt.%), Ni (2.0 wt.%) and Zn (2.6 wt.%) were obtained using the standard (unmodified) US EPA 3052 digestion procedure, but the total digestion of PCB material was not achieved. The solid residue material after digestion by means of the US EPA 3052 method consisted predominantly of oxides (Ca, Mg and Al) and fluorides (Ca and Mg), and some particles contained minor amounts of Fe and Cu.

Keywords: waste electrical and electronic equipment; printed circuit boards; acid microwave digestion; HR CS FAAS; heavy metals

1. Introduction

Older mobile phones types typically consist of electronic circuitry, a printed circuit board (PCB), a liquid crystal display, a battery, a keypad and a plastic case [1]. The waste PCBs (WPCBs) are diverse and complex in terms of their type, size, shape, components and composition [2]. The elemental composition of PCBs varies depending on their type and applications [3]. In general, these PCBs contain, in terms of weight, approximately 40% metals, 30% plastics or polymers, and 30% ceramics or refractory metal oxides [1,2,4–6]. The majority metals in PCBs are Cu, Pb, Zn, Fe, Ni and Sn [7]. Metallic material contains various elements, including precious metals, but also different hazardous elements such as heavy metals [3]. Three main types of recyclable materials can be retrieved from PCBs: metals (mainly Cu, Al, Sn and Pb) and precious metals (Au, Ag and Pt), polymeric materials and ceramic materials [8]. Each metal occurring in PCBs has its own specific properties depending on the particular point of view, such as weight share, economic value, environmental impacts, or natural resources depletion [9].

The results of quantitative analysis of highly heterogeneous materials such as e-waste may be influenced by uncertainties stemming from several sources, for example, sampling, digestion, dilution, calibration or other interferences [10].

The success of e-scrap treatment depends largely on the quality of sampling and assaying. Professional sampling and analysis is the basis for proper material assessment. Most sampling and assaying processes and technologies are still in-house developed [11–15]. The general procedure in preparing samples from WPCBs involves crushing them into smaller particles (less than 1 mm in size), followed by various techniques, including magnetic, electrostatic, electrowinning and selective dissolution in order to separate the components [16,17].

The effectivity of acid digestion reaction with solid waste material depends on several factors: the character and concentration of the acid used, the reaction time, external driving forces (heat, ultrasound, agitation and microwaves), and reaction conditions (especially temperature and solid-liquid ratio) [4]. The choice of acid or acid mixture for digestion largely depends on the metal forms present in the waste [4,18]. For example, hydrochloric acid is usually applied to release metals associated with carbonates, phosphates, borates and some oxides and sulphides [18], and metals bound to silicate fractions are dissolved by hydrofluoric acid [4,18]. The mixture of HNO_3 + HF provides the strongest chemical interaction and dissolving power, and the combination of HNO_3 with microwave (MW) energy also represents a high-energy system, with the result that both of these systems may lead to complete dissolving of any matrix [10]. The use of an aggressive combination of HNO_3 + HF + MW energy has been found to be more effective in releasing the majority metals Cu, Fe, Ni and Zn from WPCBs, compared with the utilization of hot aqua regia digestion.

The United States Environmental Protection Agency (US EPA) Solid Waste 846 Method 3052 is applicable to the microwave wet acid digestion of solid waste materials with siliceous, organics and other complex matrices [4]. This US EPA method employs conc. HNO_3 and conc. HF, and this mixture combined with MW energy creates a high energy system with strongest chemical interaction, oxidization and dissolving power [10].

Although this method was not originally designed for PCBs, it has already been used for this type of material (IT, telecommunication, large household, consumer equipment and lighting equipment) [7,19,20].

In our study, we decided to use a new approach based on the application of the original US EPA Method 3052 and its modifications to material from older mobile phone type PCBs in order to select the most effective digestion method. The effectivity of MW digestion procedures for releasing the majority elements (Cu, Pb, Zn, Fe and Ni) from PCBs was monitored through determination of their content in the solution after digestion by means of high-resolution continuum-source flame atomic absorption spectrometry (HR CS FAAS).

2. Materials and Methods

2.1. Equipment

Digestion tests on PCB material were carried out using an Ethos One pressurised closed-vessel laboratory microwave (MW) oven (Milestone Systems Italia Srl., Cinisello Balsamo, Italy), which ensures uniform distribution of MW radiation. The system was equipped with a high-pressure ten-position segmented rotor (SK-10), consisting of nine standard segments and one reference segment for temperature control. The digestion vessels, made from chemically-modified PTFE, were cleaned using 10 cm^3 conc. HNO_3 , heated for 15 min at $180 \text{ }^\circ\text{C}$ and rinsed with ultrapure water before each series of experiments.

The metals content in the solutions after individual digestion procedures was determined using the HR-CS FAAS method on a contrAA 700 spectrometer (Analytik Jena AG, Jena, Germany), which is operated by ASpect CS software. The most suitable working conditions for effective AAS measurements (fuel flow and burner height) were experimentally determined prior to the analyses using a calibration solution with the highest content of a particular element. The operating conditions of the HR-CS FAAS method, including wavelength, fuel (acetylene) flow, burner height and concentration of metals in the calibration solutions, are shown in Table 1.

Table 1. Operating conditions of high-resolution continuum-source flame atomic absorption spectrometry (HR CS FAAS) for element analysis.

Element	Wavelength [nm]	Fuel Flow [dm ³ h ⁻¹]	Burner Height [mm]	c _{Me} in Calibration Solution [mg dm ⁻³]
Cu	324.7840	50	6	0.1, 0.5, 1.0, 2.0, 4.0
Fe	248.3270	60	8	0.5, 1.0, 2.0, 4.0, 8.0
Zn	213.8570	50	6	0.1, 0.2, 0.4, 1.0
Ni	232.0030	45	7	0.2, 0.4, 1.0, 4.0, 8.0
Pb	217.0005	65	6	0.5, 1.0, 5.0, 10.0

The morphology (surface topography) of the solid residue after the most effective MW digestion procedure was determined by means of scanning electron microscopy (SEM) on an FE SEM MIRA 3 microscope (TESCAN, Brno, Czech Republic). The local surface elemental composition was examined using energy-dispersive X-ray spectroscopy (EDX) on an EDX microanalysis system (Oxford Instruments, Oxford, UK). The SEM EDX analysis was carried out at the Institute of Materials Research of Slovak Academy of Sciences, Košice. The ultrapure water for the solutions and cleaning operations was prepared using an Elga Labwater deionizer.

2.2. Reagents, Standards and Material

All the chemicals used were of analytical grade. The reagents for MW digestion were purchased from Fischer Chemical, UK and Centralchem, Czech Republic and included concentrated nitric acid (conc. HNO₃, 65% w/w), concentrated hydrochloric acid (conc. HCl, 37% w/w) and concentrated hydrofluoric acid (conc. HF, 40% w/w). Calibration solutions for the HR-CS FAAS method were prepared by diluting standard solutions of Cu, Fe, Ni, Pb and Zn (SIGMA-ALDRICH Co. LLC., St. Louis, MO, USA) with a solution of HCl (1% v/v) in ultrapure water. All laboratory flasks were washed with dilute HNO₃ (4 mol dm⁻³), followed by rinsing with ultrapure water.

The PCB material (analytical samples) for all MW digestion experiments was obtained by processing approx. 80 kg of end-of-life mobile phones with various years of production and from various manufacturers. Batteries and plastic covers from the mobile phones were removed by hand. The remaining PCBs from the mobile phones (Figure 1a) were used for the experiments. Their total weight was 2 kg. The crushing operation was carried out with a crusher hammer down to fraction $d \leq 8.0$ mm, and the crushed PCBs were homogenized. A sample of 1 kg was obtained by hand quartering, which was then divided three times with a mechanical divider. Magnetic separation was carried out to remove the magnetic fraction from the sample. Then, eight representative samples were milled separately in a hammer mill, combined to fraction $d \leq 1.0$ mm and sieved in order to obtain the final PCB analytical sample for experiments ($d \leq 0.5$ mm) (Figure 1b).

2.3. Microwave-Assisted Digestion Procedures

For quantitative metal liberation assessment, the sample of PCBs was subjected to four MW digestion procedures at 180 °C:

Method A1—the original US EPA Method 3052. In this digestion procedure, a 0.25 g PCB sample was digested in 4.5 cm³ conc. HNO₃ and 1.5 cm³ conc. HF for 10 min. Method A2—the US EPA Method 3052 with extension of digestion time to 20 min. Method A3—the US EPA Method 3052 with extension of digestion time to 30 min.

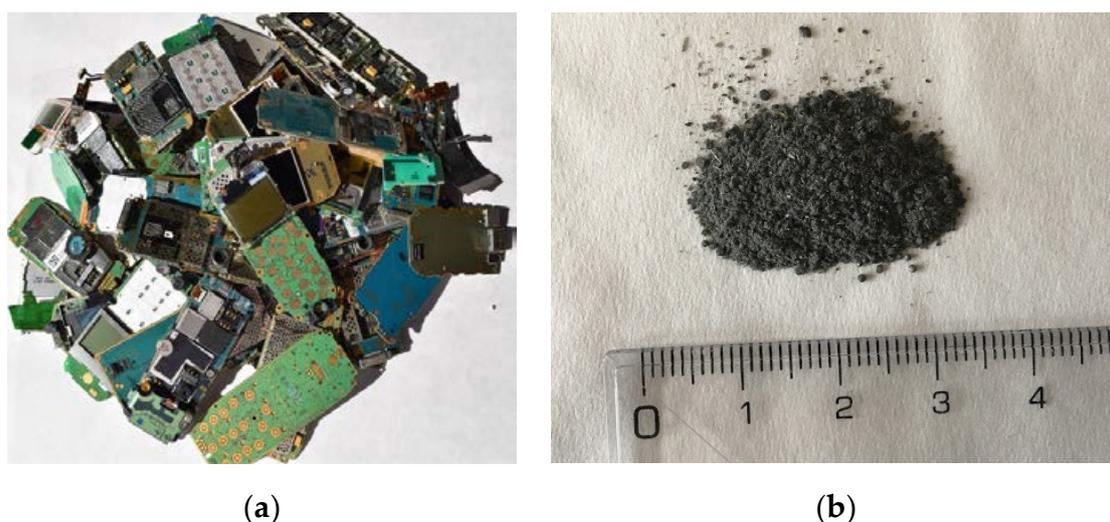


Figure 1. (a) Printed circuit boards (PCBs) of mobile phones; (b) analytical sample of PCBs (photos taken by author).

Method B—an adapted US EPA Method 3052. This digestion procedure consisted of two steps. In the first step, a 0.25 g PCB sample was digested in 4.5 cm³ conc. HNO₃ and 1.5 cm³ conc. HF for 10 min. In the second step, the MW digestion was stopped and the vessels were opened to release the reaction gases, and then after closing of the vessels the digestion continued for a period of 10 min.

Method C—an adapted US EPA Method 3052. This digestion procedure included two steps as well. In the first step, a 0.2 g PCB sample was digested in 9.0 cm³ conc. HNO₃ for 10 min with the aim of decomposing the organic (plastic) material from the sample. In the second step, the digestion vessels were opened in order to release the reaction gases, and then after addition of 3.0 cm³ conc. HF and 3.0 cm³ conc. HCl (for digestion of inorganic materials) the vessels were closed and the digestion continued for a period of 10 min.

Method D—an adapted US EPA Method 3052 [10]. In this digestion procedure, a 0.2 g PCB sample was digested in 9.0 cm³ conc. HNO₃ and 3.0 cm³ conc. HF (60% abundance of reagents compared to the original US EPA method) for 10 min.

After each digestion procedure, the content of the digestion vessels was filtered through quantitative filter paper into 100 cm³ volumetric flasks and supplemented with ultrapure water. These individual digestions performed with the aim of finding the most effective digestion procedure for complete release of selected elements from the PCB material were repeated four times, and analyses were carried out in triplicate. A summary of the complete procedural steps followed by sample digestion is schematically shown in Figure 2.

For statistical evaluation of the most effective digestion procedure, we performed ten repeated digestions. For elimination of the PCB material heterogeneity influence, we compared the contents of elements in 4/10 replicates of the most effective digestion procedure in 4/10 post-digestion solutions with their contents in the solution obtained by combining 4/10 post-digestion residues.

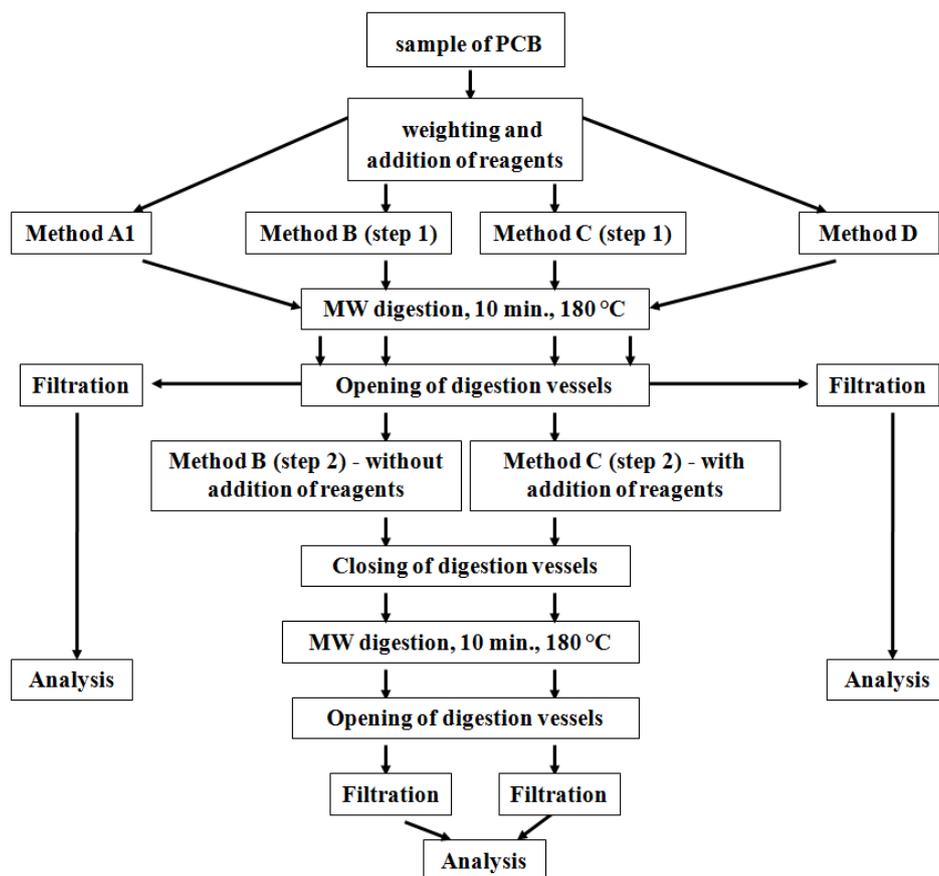


Figure 2. Block diagram of microwave (MW) digestion procedural steps.

3. Results and Discussion

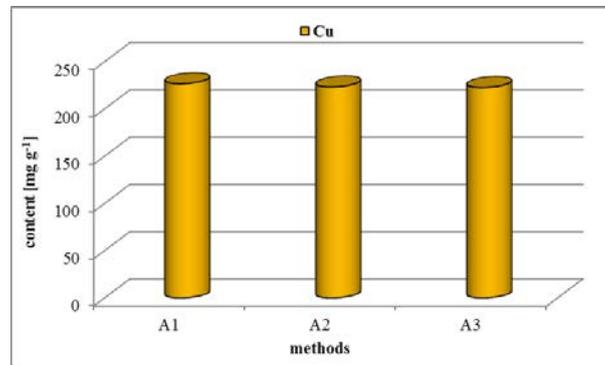
In the first series of experiments, we monitored the effect of digestion time (10 min for method A1, 20 min for method A2 and 30 min for method A3) with application of the original US EPA Method 3052 on the release of elements into the post-digestion solutions. The results of these experiments are shown in Figure 3.

The results indicate that the extension of digestion time, with the exception of Cu, does not influence the increase in content of elements in the solution after digestion, nor does it increase the effectivity of digestion. Decomposition for 30 min resulted in a 20% decrease in Cu recovery and up to a 60% decrease in Fe recovery. Although it is not possible expressly determine the reason for the decrease in decomposition efficiency by prolonging its decomposition time, it is most likely that the equilibrium of decomposition reactions were shifted due to excessive formation of reaction products. This result led us to apply Method B as a modification of the original US EPA Method 3052 in another series of experiments.

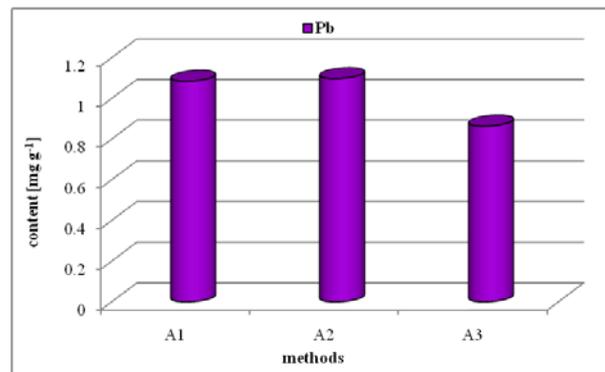
Our second series of experiments was focused on monitoring the effects of the three modifications of US EPA Method 3052 (Methods B, C and D) on the release of elements. The aim of the experiments was to shift the chemical balance to products of digestion reactions, which would lead to increased release of elements. The modifications of US EPA Method 3052 were as follows:

1. Opening digestion vessels to release gaseous products and repeating the digestion process in the second step (Method B);
2. Pre-digesting organic (plastic) materials with HNO_3 in the first step (the most widely-used primary oxidant for the decomposition of organic matter [21]), and adding HCl and HF for digestion of inorganic materials in the second step (Method C),

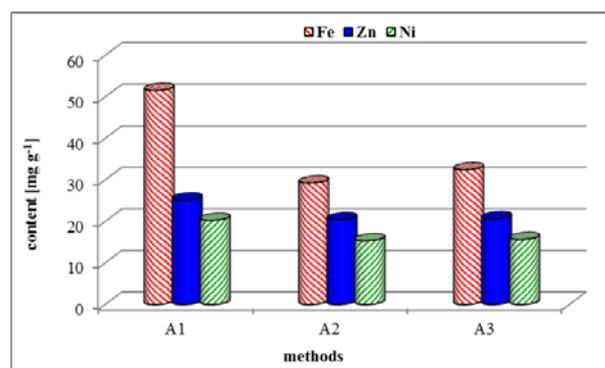
3. Increasing the HNO_3 and HF amounts in the digestion mixture by about 60% (Method D) over the original US EPA method. The results of these experiments in comparison with results of the original US EPA Method 3052 (Method A1) are shown graphically in Figure 4.



(a)



(b)



(c)

Figure 3. Content of Cu (a), Pb (b), Fe (c), Zn and Ni in post-digestion solutions obtained by application of the US EPA 3052 method (A1) and its modifications in time (A2, A3) on mobile phone PCBs.

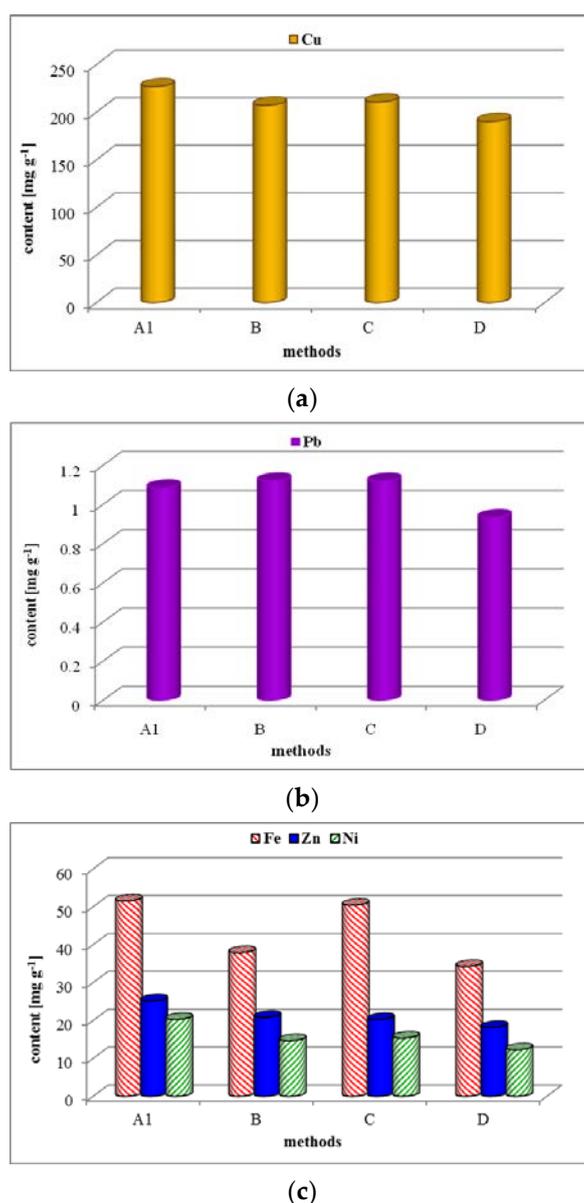


Figure 4. Content of Cu (a), Pb (b), Fe, Zn and Ni (c) in post-digestion solutions obtained by application of the US EPA 3052 Method (A1) and its modifications in the number of digestion steps (B, C) and amount of reagents (D).

The results (Figure 4) show that application of the chosen digestion procedures did not achieve any increase in the release efficiency of metals from the PCB material in comparison with the original US EPA method. The efficiency of two-step decomposition with the addition of HCl in the second step was comparable to that of the US EPA method. Increasing the ratio of sample mass to digestion reagent volume appeared to be the least effective. Increasing the amount of reagents to the same amount of sample (0.25 g), in comparison to the original US EPA Method 3052, could result in an increase of the fluorides and oxides formation as new forms in the solid residue after digestion (see discussion in the last stage of studies).

The relative standard deviation (RSD) values, representing the uncertainty of elements content determination caused by the heterogeneity of the material, calculated from the four repeated digestions for each digestion method (A1, A2, A3, B, C and D) are shown in Table 2.

Table 2. Relative standard deviation (RSD) values from four repeated digestions for each studied digestion method.

Element	RSD [%]					
	A1	A2	A3	B	C	D
Cu	5.879	5.952	5.501	5.481	12.35	6.410
Fe	15.33	17.63	20.56	18.97	14.32	18.09
Zn	7.672	4.981	8.530	6.689	18.95	20.48
Ni	16.04	11.67	18.93	17.44	28.62	28.16
Pb	26.31	39.23	20.26	27.39	22.16	11.47

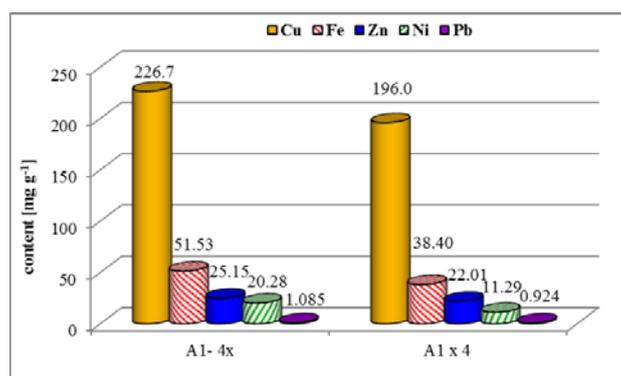
The RSD values (Table 2) ranged from 5 to 40 independently of the degree of dilution of the solution prior to analysis. The highest RSD values were found for determination of Pb content directly in the post-digestion solutions, and the lowest values were for Cu determination in the solutions with the highest dilution, suggesting that there was significant effect of material heterogeneity on the uncertainty of determining the content of the different elements. The effect of PCB material heterogeneity on the uncertainty of the analysis results may possibly be moderated by increasing the PCB amount in the digestion process. Considering the recommendations of the manufacturer of the MW digestion system, the maximum weight of a sample for digestion can be 0.5 g, and element content determined would be taken as the average of contents from two parallel digestions. The use of 0.5 g PCB material for digestion was not possible in our experiments, because unfavourable phenomena (explosions as a result of increased pressure within the closed vessel due to the inhomogeneity of sample) caused deformation and damage to the vessels.

In the next series of experiments, we compared the average content of the elements calculated from the four contents determined in the four post-digestion solutions obtained using method A1 (A1 – 4×) with the content determined in the single solution containing the elements released from four replicates of digestion method A1 (A1 × 4). This experiment was also repeated by applying ten repeats of the A1 method. Figure 5 shows the content of elements determined in this series of experiments.

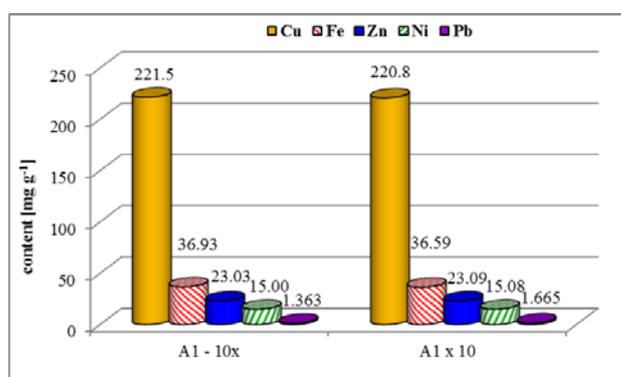
While the contents of the elements determined from four repeated digestions, i.e., 1.0 g of the sample, were significantly different for the two methods of evaluation (Figure 5a), in the case of the application of 10 repeated digestions, i.e., 2.5 g of the sample, these differences were minimal (Figure 5b).

In the solid residue after digestion using US EPA Method 3052 (method A1, Figure 6b), new formations were observed that were not present in the original sample (Figure 6a). These new formations may have originated in the presence of nitric acid as a result of the passivation of some metals by a layer of stable oxides (e.g., Al, Ca, Cr and Fe) or the hydrolysis of some metals to form hydroxides (Sn). The use of hydrofluoric acid could result in the formation of stable fluorides of alkaline earth metals, but also of Cu, Pb and Zn.

SEM analysis of the solid residue after digestion of PCB material using Method A1 revealed marked diversity in shape and size of particles (Figure 7a). Local chemical analysis of the solid surface using the EDX method confirmed the presence of fibrous material (Figure 7b, spectrum 5) that, considering the higher content of non-metallic elements, probably corresponds to non-decomposed organic matter. Element concentration on the different particle surfaces (Figure 7a) is given in Table 3. It is evident, from Table 3, that the presence of Fe was confirmed in the all analysed sites. Cu was found only in some sites (Figure 7, spectrum 6, 7 and 9). F, O, Ca and Mg were found on the all investigated sites. Presence of Al and Br was confirmed almost on the all sites.



(a)



(b)

Figure 5. Content of Cu, Fe, Zn, Ni and Pb obtained using method A1 expressed as (a) average of four contents from four replicates of digestion (A1—4×) vs. content in the single solution from four replicates of digestion (A1 × 4), (b) average of 10 contents from 10 replicates of digestion (A1—10×) vs. content in the single solution from 10 replicates of digestion (A1 × 10).

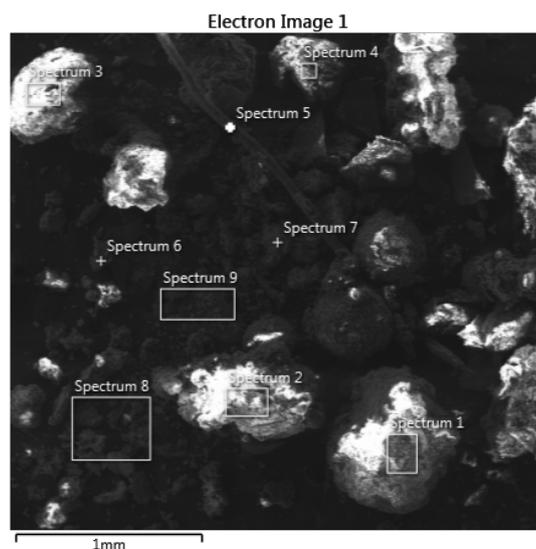


(a)

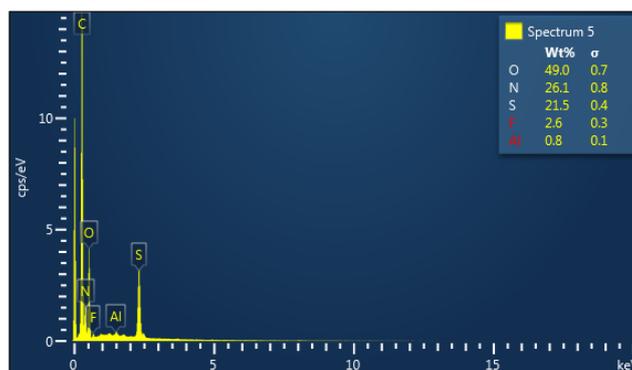


(b)

Figure 6. Images of (a) PCB analytical sample and (b) solid residue after digestion with United States Environmental Protection Agency (US EPA 3052) (method A1) (1.0:2.5, photos taken by author).



(a)



(b)

Figure 7. (a) SEM images of solid residue after digestion with US EPA method 3052; (b) chemical composition of specific particle surface material.

Table 3. Results of surface analysis of selected sites and elements.

Spectrum	Element [wt%]							
	F	Br	O	Al	Ca	Mg	Fe	Cu
1	38.3	7.4	35.0	9.3	3.1	4.0	1.8	–
2	32.8	7.8	40.1	8.2	2.9	4.1	1.6	–
3	16.9	38.6	29.8	–	5.1	3.1	4.6	–
4	38.2	–	40.4	8.9	3.7	5.0	1.6	–
6	9.1	21.9	25.7	–	20.2	2.8	11.3	3.9
7	29.6	7.1	15.1	13.5	6.0	10.4	2.3	1.0
8	19.8	24.7	28.0	5.9	5.2	4.2	2.6	–
9	33.5	–	19.9	7.5	16.1	5.3	8.9	7.6

Solid residue after decomposition in HF is probably created of fluorides, mainly Ca and Mg, respectively, formed from unreacted HF. The oxygen in the residue is probably in the form of Al, Fe and Ca oxides. The HNO₃ used in the decomposition mixture caused the formation of a layer of stable oxides of the given metals (passivation). Bromides were re-formed from Br (flame retardant), which was released into solution upon decomposition. The presence of Na (1–2.5 wt%), S (1–3.5 wt%), Cl (0.6–1.5 wt%) was found in some analyzed sites. It can be assumed that the presence of Br and Cl in

the solid residue may be due to incomplete decomposition of the organic material (plastics) contained in the WPCBs. In addition to the mentioned elements, the presence of Ag was detected in the analyzed sites (7 and 8). At site 7, it was detected even up to 7.4 wt% of Ag.

4. Conclusions

The choice of the most suitable digestion procedure was problematic because of the heterogeneity of the PCB material, incompleteness of digestions and unfavorable accompanying phenomena. The presence of Fe, Ca, Al and O in the solid residues after decomposition confirms incomplete digestion due to the formation of a stable oxide layer by the action of conc. HNO₃ [21]. The presence of Ca, Mg, Cu and F may indicate the formation of stable insoluble fluorides as a result of the reaction with conc. HF [21]. The problem of the formation of insoluble fluorides could be eliminated by the addition of H₂SO₄, HClO₃ or HClO₄. Mentioned unfavorable phenomena can lead to the incomplete release of the metals of interest into the solution or to their re-sorption to the surface of the emerging new forms (fluorides, oxides).

In addition to the incomplete digestion of the metal components, the organic material was not completely digested too. It could be supported by the addition of a strong oxidizing agent, e.g., H₂O₂.

For elimination of the effect of WPCBs material heterogeneity on the analysis results' uncertainty, it would be best to enhance the sample amount, which is possible through integration of the residues after ten digestions into one solution. This solution corresponds to the recommended 2.5 g sample for digestion.

Despite the effort to improve the original US EPA method (extending the digestion time, adding the 2nd digestion step, adding HCl, or excess of decomposing agents), it has been shown that the original US EPA Method 3052 seems to be the most effective procedure for releasing Cu, Fe, Ni and Zn.

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