TREASURE

D5.3: Simulation and lab-scaled testing of the materials recovery processes

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EXECUTIVE SUMMARY

The main objective of the TREASURE project is to test innovative technologies to make the automotive sector more circular. To this end, TREASURE wants to implement an AI-based scenario assessment tool to support the development of circular supply chains in the automotive industry. Through a series of success stories deriving from applying the principles of the circular economy in three key value chains linked to the automotive sector, TREASURE wants to demonstrate in practice the real benefits deriving from the adoption of circular behavior. Key enabling technologies (KETs) will be integrated into selected processes to support the efficient design of automotive electronics and their subsequent disassembly and material recovery. Deliverable 5.3 “Simulation and lab-scale testing of materials recovery processes” is part of the WP5 “Pilot plants reconfiguration/optimization” activities. The purpose of this document is to provide all the information relating to the laboratory-scale treatment of numerous materials in the automotive sector using hydrometallurgical processes. The document presents a detailed analysis of the characterization of each material in terms of precious and base metal contents. In addition, the results collected, shared and managed through a common Microsoft OneDrive folder have been summarized and incorporated into this document.

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1. Introduction

1.1. Background

The study carried out on the different materials supplied was conducted by analyzing and possibly modifying two patented processes:

- GOLD-REC 1: Process For The Hydrometallurgical Treatment Of Electronic Boards [1].
- GOLD-REC 2: Hydrometallurgical Method For The Recovery Of Base Metals And Precious Metals From A Waste Material [2].

GOLD-REC 1

The current hydrometallurgical procedure consists in the following operations:

1. The waste printed circuit boards are firstly subjected to a physical mechanical procedure where the Al- and Fe-based components are removed manually (by using a heat gun) from PCBs surface. Then, the depopulated PCBs are shredded and milled to suitable particles sizes.
2. The milled PCBs are then leached with water, sulfuric acid and hydrogen peroxide for the extraction of base metals.
3. The separation of the solids from the leach liquid is carried out by filtration process followed by washing with water. The resulted solution (filtrate) is subjected to a precipitation process for Sn precipitation. Then, also this solid precipitate is separated from solution by filtration and further washed with water. The solution achieved after Sn recovery s sent to an electrowinning cell for Cu recovery.
4. The resulted solution is recycled in the first leaching for the treatment of another PCB material (counter-current leaching).
5. The solid residue of base metals leaching process is involved into another leaching process with thiourea as reagent, ferric sulfate as oxidant in diluted sulfuric acid for Au and Ag dissolution.
6. After the separation of the solid from the solution by filtration, the electrowinning process is also applied to this solution for Au and Ag recovery. Once the process is finished, the remaining solution discharged from Au and Ag can be also recycled for the leaching of precious metals for the treatment of other PCBs.

GOLD-REC 2

This hydrometallurgical process could be synthetically described as indicated below:

1. The chemical process can be applied on the e-waste without grinding (with whole waste PCBs as an example) avoiding important loss of precious metals also described in the literature.
2. The process uses a unique step of metals dissolution with a chemical leaching using HCl, H_2O_2, acetic acid in water solution at room temperature (21 °C ± 3 °C) with a solid/liquid ratio of 10-20%. The chloroacetic acid is produced by in-situ chemical process within two steps: firstly, hydrochloric acid reacts with hydrogen peroxide and acetic acid to produce peracetic acid, water and chlorine; in the second step chloroacetic acid and hydrochloric acid are produced by the chlorination of the unreacted acetic acid.
3. Precious (Au and Ag) and base metals (Cu, Sn, Zn, Ni, Pb) are dissolved leaving the waste PCBs with mainly epoxy resins and fiberglass structure intact (with some residues of metals);

4. The liquid solution is easily separated from the S/L system and selective reduction-precipitations steps are considered in the process to recover the dissolved metals. These steps are synthetically described in the follow:
    - Reduction and precipitation of Au chloride to its metallic form by ascorbic acid;
    - Cooling the solution to less than 15 °C for precipitation of AgCl;
    - Selective reduction and precipitation of Cu by metallic Sn or co-reduction of both copper and tin ions with iron metal;
    - Reduction and precipitation of $SnCl_2$ by metallic Zn;
    - Exploitation of the residual solution for its recycling within the process or by adding iron in order to produce a $FeCl_2$-$FeCl_3$ solution useful for coagulation processes in the treatment of wastewaters;
5. The main products are: Au (after melting process in an inductive electrical oven adding some slug forming compound), AgCl, Cu and Sn in powder forms (mainly in the range of 10-90 µm) and a residual chloride solution that can be regenerated by make-up with proper reagents concentration or treated with iron metal to achieve a high concentrated iron solution (extensively and usually utilized in the coagulation processes in wastewater treatments).

1.2. Objectives

The activity was aimed at studying the technical and economic feasibility of using different hydrometallurgical processes on numerous materials. The materials provided by the various partners were analyzed both to assess their intrinsic value and to have an idea of the recovery efficiencies of the hydrometallurgical processes for the extraction of the various precious and base metals contained therein. Below is a table summarizing all the materials analyzed during this activity on a laboratory scale.

Table 1. Summary of all studied materials

PARTNER SUPPLIER	SOURCE	MATERIAL
TNO	-	Flexible electronics
SEAT	Seat350 Leon II	Infotainment, Dashboard, Additional Brake Light, Speed and ABS Sensor, Rain Sensor, Mirror
	Seat370 Leon III	Infotainment, Dashboard, Additional Brake Light, Speed and ABS Sensor, Rain Sensor, Mirror, Rear View Mirror
	Seat250 Ibiza IV	Dashboard, Speed and ABS Sensor, Mirror, Rear View Mirror
EuroLCDs	-	Liquid crystal displays
	-	Printed circuit boards – AF81

2. Characterization

The chemical characterization of the different materials provided by the various partners was performed. The main analysis techniques used are:

- Inductively Coupled Plasma – Optical Emission Spectroscopy (ICP-OES)
  This definition identifies an analytical instrument capable of measuring the light (optical emission) produced by a liquid sample when introduced into an inductively coupled argon gas plasma. Through this mechanism it is possible to quantify the metals contained in the sample by measuring, for each one, the intensity of the light emitted with a specific optical bench (system of mirrors, lenses and gratings). For the determination in ICP-OES it is necessary to have samples in liquid form. In this regard, aqua regia leaching is often used to determine the metal content of a solid matrix. Aqua regia is a mixture of nitric acid and hydrochloric acid, optimally in a molar ratio of 1:3.

- X-ray fluorescence spectroscopy (XRF)
  XRF is a non-destructive analysis technique which allows to know the elemental composition of a sample through the study of X-ray fluorescence. This radiation is emitted by the atoms of the sample following excitation (which can also give a photoelectric effect), which typically achieved by irradiating the sample with high-energy X-rays and gamma rays. Being a semi-quantitative analysis, it has a lower precision than ICP-OES analyses.

Both techniques allow to know the quantity of metals present in the sample in terms of mass percentage of the element. However, with these procedures it is not possible to know the molecular form in which the metals are present. In order to know the mineralogical form, an X-ray diffraction (XRD) analysis is required.

2.1. Flexible electronics 1 (TNO)

TNO provided samples of Flexible electronics with optically transparent, viscoelastic, chemically and thermally stable composite matrix. In most cases, the substrates (on both sides) are PET. The spacer (in between PET) is either TPU or a commercial material. Figure 1 shows an example of the samples. In this sample, the silver was embedded.

Figure 1. Flex circuit sample

The sample was chemically etched with aqua regia for the determination of the silver content. A mean **Ag concentration of 0.31 wt%** was determined, taking into account the full weight of the sample.

2.2. In-mold electronics 2 (TNO)

In-mold electronics (IME) samples were provided by TNO. The samples mainly consist of plastic sheets with polycarbonate backing and different types of printed layers (black and white graphic inks, dielectrics) and silver paste printed with a polymer binder. D5.6 – "Simulation of the in-mold-structural electronics prototyping process" contains more information describing the device build-up and material composition of each layer. Some samples were not over molded with polycarbonate resin. For these samples, the silver was directly accessible: it is on the surface not embedded by a plastic layer, so can be directly exposed to the action of the acid. Below are presented two examples of the sample (A, B).

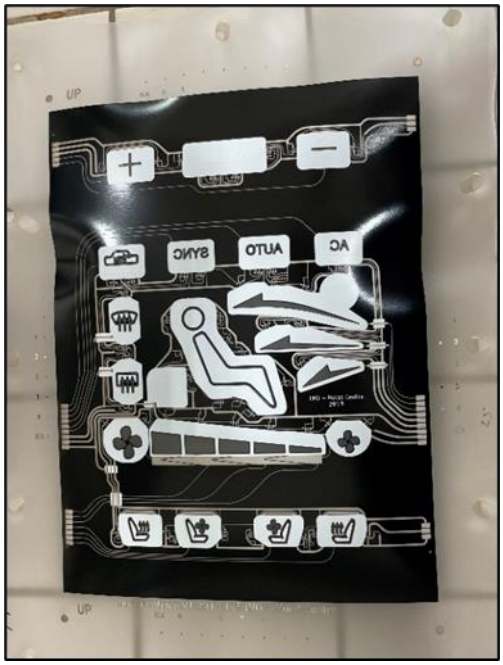


Figure 2. In-mold electronics: sample A

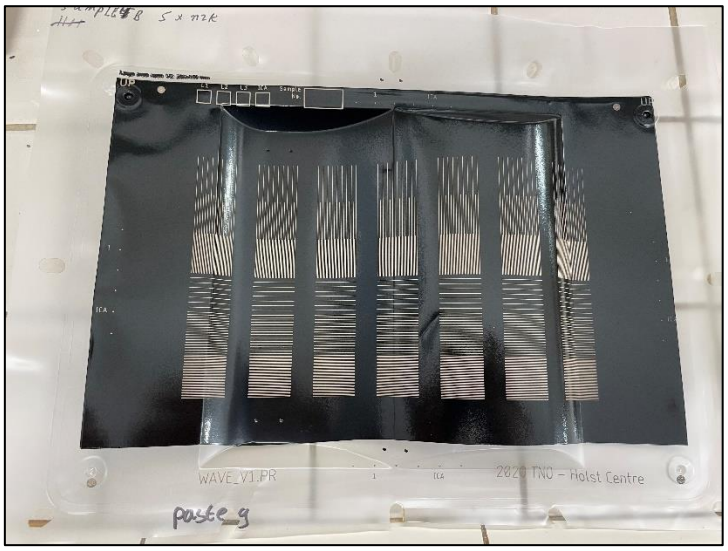


Figure 3. In-mold electronics: thermoformed sample B.

The initial concentration of silver in samples A and B was calculated through the analyses carried out on the solutions by ICP-OES.

Table 1. Silver concentration of E-waste – thermoformed samples.

	Ag Concentration (%)
Thermoformed sample A	0.36
Thermoformed sample B	0.63

In addition, also XRF analyses were performed to have qualitative information in order to detect also the other metals. An example of these analyses is provided in Table 2. In the following figures (Figures 4-5) it is possible to see a map showing areas where the analyses were performed; areas were selected by the different colors.

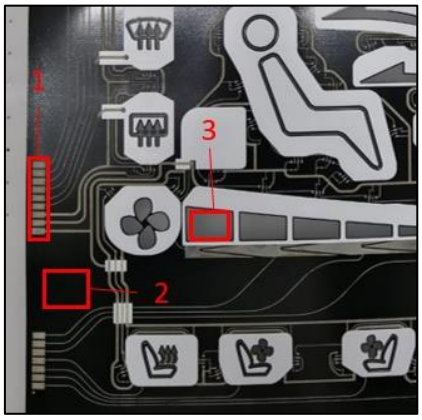


Figure 4. Map of performed XRF analyses for the characterization of sample A.

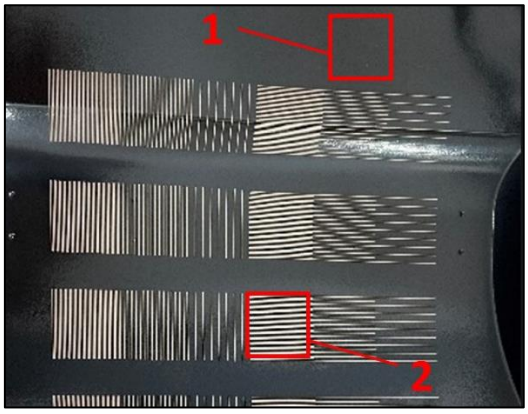


Figure 5. Map of performed XRF analyses for the characterization of sample B.

Table 3 shows the results obtained from XRF analyses for area 1 of sample A The other results are reported in the Appendix B.

Table 2. XRF of thermoformed sample A, area 2

Z	Symbol	Element	Norm. Int.	Concentration	Abs. Error
12	Mg	Magnesium	0.0000	< 0.0020 %	(0.0) %
13	Al	Aluminum	73.6823	< 0.0020 %	(0.0) %
14	Si	Silicon	913.0952	0.4782 %	0.0008 %
15	P	Phosphorus	31.0425	0.00623 %	0.00015 %
16	S	Sulfur	3264.5297	0.3101 %	0.0003 %
17	Cl	Chlorine	298.8645	0.01104 %	0.00003 %
19	K	Potassium	44.2475	0.01903 %	0.00021 %
20	Ca	Calcium	49.7790	0.00063 %	0.00001 %
22	Ti	Titanium	125.4881	0.01324 %	0.00009 %
23	V	Vanadium	21.0388	0.00141 %	0.00005 %
24	Cr	Chromium	31.4012	0.00111 %	0.00003 %
25	Mn	Manganese	14116.5111	0.4539 %	0.0003 %
26	Fe	Iron	12076.9126	0.6383 %	0.0005 %
27	Co	Cobalt	2.4330	< 0.00017 %	(0.0) %
28	Ni	Nickel	45.7436	0.00123 %	0.00002 %
29	Cu	Copper	310.8789	0.00672 %	0.00003 %
30	Zn	Zinc	22.7999	0.00034 %	0.00001 %
31	Ga	Gallium	0.3439	< 0.00005 %	(0.0) %
32	Ge	Germanium	0.0000	< 0.00005 %	(0.0) %
33	As	Arsenic	1.9967	0.00002 %	0.00001 %
34	Se	Selenium	2.5407	0.00002 %	0.00001 %
35	Br	Bromine	4.4707	< 0.00005 %	(0.00003) %
37	Rb	Rubidium	5.8103	< 0.00005 %	(0.00002) %
38	Sr	Strontium	39.0812	< 0.00005 %	(0.0) %
39	Y	Yttrium	0.0000	< 0.00005 %	(0.0) %
40	Zr	Zirconium	0.2934	< 0.00010 %	(0.0) %
41	Nb	Niobium	0.3178	0.00016 %	0.00007 %
42	Mo	Molybdenum	0.9827	0.00002 %	0.00001 %
47	Ag	Silver	1.0365	0.00007 %	0.00003 %
48	Cd	Cadmium	2.0584	0.00008 %	0.00001 %
50	Sn	Tin	3.4910	0.00428 %	0.00050 %
51	Sb	Antimony	1.1930	< 0.00030 %	(0.0) %
52	Te	Tellurium	3.0949	< 0.00030 %	(0.0) %
53	I	Iodine	0.0000	< 0.00030 %	(0.0) %
55	Cs	Cesium	0.0000	< 0.00040 %	(0.0) %
56	Ba	Barium	2.3420	< 0.00020 %	(0.0) %
57	La	Lanthanum	2.2980	< 0.00020 %	(0.0) %
58	Ce	Cerium	2.1024	< 0.00020 %	(0.0) %
59	Pr	Praseodymium	8.9735	0.00146 %	0.00012 %
60	Nd	Neodymium	17.4317	0.00241 %	0.00010 %
72	Hf	Hafnium	4.2243	< 0.00010 %	(0.0) %
73	Ta	Tantalum	27.5735	< 0.00010 %	(0.0) %
74	W	Tungsten	2.0377	< 0.00002 %	(0.00001) %
80	Hg	Mercury	3.0540	0.00004 %	0.00001 %
81	Tl	Thallium	4.5374	0.00004 %	0.00001 %
82	Pb	Lead	6.8112	0.00011 %	0.00001 %
83	Bi	Bismuth	0.0000	< 0.00010 %	(0.0) %
90	Th	Thorium	7.1449	0.00007 %	0.00001 %
92	U	Uranium	5.0199	< 0.00010 %	(0.0) %

2.3. Car components (SEAT)

SEAT has supplied automotive components for 3 different types of cars: SEAT 350 LEON II, SEAT 370 LEON III and SEAT IBIZA IV. The choice of the models and of the car components comes from a thermodynamic rarity assessment (D3.1). Table 3 summarizes the various components received with the relative codes and quantities.

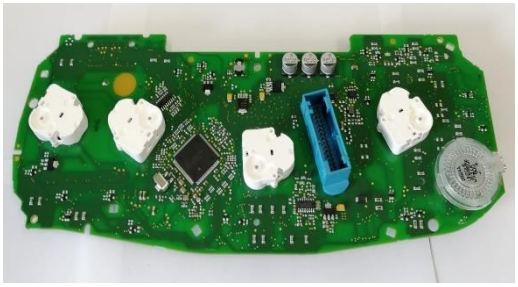
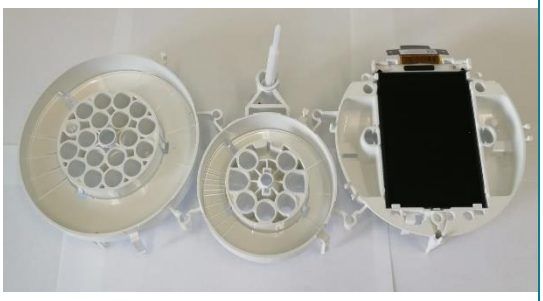
Table 3. Car components received from SEAT

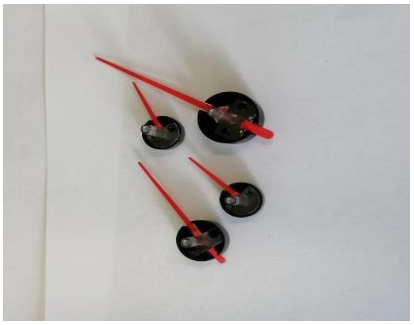
SEAT 350 LEON II	Qty.	SEAT 370 LEON III	Qty.	SEAT IBIZA IV	Qty.
INFOTAINMENT	x3	INFOTAINMENT	x3	-	-
DASHBOARD	x2	DASHBOARD	x2	DASHBOARD	x3
ADDITIONAL BRAKE LIGHT	x3	ADDITIONAL BRAKE LIGHT	x2	-	-
SPEED AND ABS SENSOR	x3	SPEED AND ABS SENSOR	x3	SPEED AND ABS SENSOR	x3
RAIN SENSOR	x3	RAIN SENSOR	x2	-	-
-	-	MIRROR	x3	MIRROR	x3
-	-	REAR VIEW MIRROR	x3	REAR VIEW MIRROR	x3

Each of the above components has been disassembled into its sub-components using the instructions provided by SEAT. Disassembly times were similar to those expected by SEAT. Subsequently, each sub-component was weighed by means of a technical balance and photographed.

Table 4. Example of the disassembly of the seat Leon II dashboard

N.	Photo	Weight [g]	wt %	Materials
0		844.2	100%	-
1		239.1	28.32%	Plastic

2		198.9	23.56%	See Table 6
3		197.9	23.44%	Plastic Glass
4		192.7	22.83%	Plastic
5		11.2	1.33%	Plastic

6		3.0	0.36%	Plastic
7		1.2	0.14%	Aluminum
Total	-	844.0	99.98%	-

A detailed analysis of the metal content was carried out for the electronic boards (component n. 2 of Table 5) of the dashboard of each SEAT model. In this respect, the connection devices, the transducers, the electromagnets, the display devices and the display board have been removed from the electronic board. Subsequently, the grinding with a knife mill of the board was carried out. Leaching with aqua regia or nitric acid was performed for each removed component and ground fraction. ICP-OES analyzed the resulting solution to determine the content of the different precious and base metals of interest. The following tables show the results obtained for each car model.

Table 5. Characterization dashboard electronic board SEAT Leon II

		g/ton			wt %	
Components	**wt %**	**Au**	**Ag**	**Pd**	**Ti**	**Cu**	**Sn**
Powders	66.52	59.08	169.0	19.20	715.7	11.54	1.05
Transducer	4.41	12.38	2.40	-	-	3.96	0.56
Electromagnets	23.01	11.48	0.01	-	-	0.10	1.73
Connection device	3.68	3.73	130.7	8.60	4.84	18.13	1.42
Display device	0.30	748.5	868.0	42.30	11.94	21.54	3.11
Display board	0.27	684.5	648.5	-	-	-	2.39
Losses	1.81						
Tot.	**100.0**						

Table 6. Characterization dashboard electronic board SEAT Leon III

		g/ton				wt %	
Components	**wt %**	**Au**	**Ag**	**Pd**	**Ti**	**Cu**	**Sn**
Powders	72.89	27.77	244.3	9.48	683.7	14.47	0.98
Transducer	6.20	1.59	1.05	-	-	6.43	2.30
Inductor	3.33	-	81.10	-	50.35	20.62	0.64
Electromagnets	12.07	0.08	0.00	-	-	43.84	0.10
Connection device	4.26	-	86.60	3.47	72.96	1.06	0.31
Display device	0.34	0.12	1389	0.00	-	21.54	3.11
Display board	0.00	0.91	513.1	75.12	-	22.01	2.10
Losses	0.91						
Tot.	**100.0**						

Table 7. Characterization dashboard electronic board SEAT Ibiza IV

		g/ton				wt %	
Components	**wt %**	**Au**	**Ag**	**Pd**	**Ti**	**Cu**	**Sn**
Powders	73.33	61.87	265.3	29.1	1637	20.46	1.23
Transducer	5.94	14.36	66.2	-	-	4.90	2.10
Electromagnets	14.75	0.00	2.50	-	-	0.52	0.09
Connection device	5.02	3.02	84.41	6.59	0.78	18.32	1.52
Losses	0.96						
Tot.	**100.0**						

The non-metallic fractions of the electronics boards are composed to fiber glass, epoxy resin and plastics [5].

2.4. Liquid crystal displays – ITO glass (EuroLCDs)

The samples were provided by EUROLCDs in the scope of TREASURE project (task 5.3 – lab scale activities). Figure 6 shows the photographic aspect of the sample.

Figure 6. LCD – ITO GLASS 2x3 cm pieces

The sample was attacked by aqua regia, and the resulting solution was subsequently analyzed by ICP-OES to determine the content of the metals of interest: In and Sn. The study of these two metals was dictated by literature analyses and by economic/critical interest [3].

Table 8 summarizes the composition of ITO glasses in terms of metals. Indium and tin were present as oxides.

Table 8. Chemical composition of ITO glasses (ICP-OES analysis).

Chemical attack	Mass [g]	In [g/t]	Sn [g/t]
1. double step aqua regia attack	4.032	71.12	25.17
2. single step aqua regia attack	3.834	60.90	8.87

The chemical composition was also defined by performing XRF analysis. The main components are shown in the following table. In addition to indium and tin were found only elements due to the glass.

Table 9. Chemical composition of ITO glass (XRF analysis)

Elements	Concentration
Si	37.9 %
Mg	4.4 %
Ca	4.8 %
S	1.6 %
In	**60 -70 g/t**
Sn	**15 - 20 g/t**

The other detected elements (K and P) have a concentration below 1 %.

2.5. Liquid crystal displays – printed circuit boards (EuroLCDs)

EuroLCDs has supplied Printed Circuit Boards (PCBs). Figure 7 shows an example of the sample.

Figure 7. PCBs Sample (AF81)

All the components on the board were detached, by heating and manual removal, in order to chemically characterize each type. Table 10 and Table 11 shows the results of this analysis. Then, the components have been processed using different methods to solubilize metals. The quantitative analyses were performed by inductively coupled plasma optical emission spectrometry (ICP-OES) on the solutions obtained by the chemical attacks. In this way it was possible to characterize the metallic fraction of the sample.

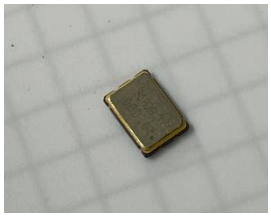
Table 10. Summary of component and relative percentage by weight

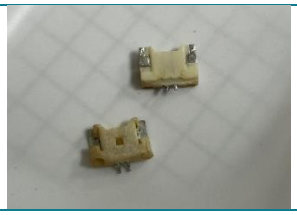
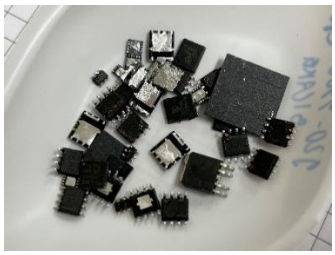
List of components			
components		weight, %	
inductors	inductors C102	22.58	27.96
	inductors DR73	5.38	
back panels	back panels	10.33	12.26
	back panels' hooks	1.93	
integrated circuit chips (ICC)		2.08	

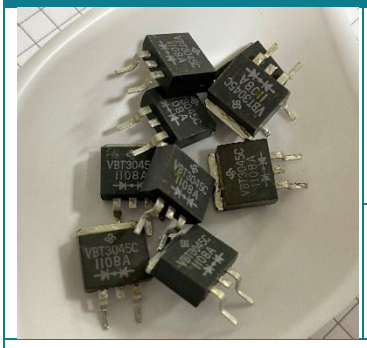
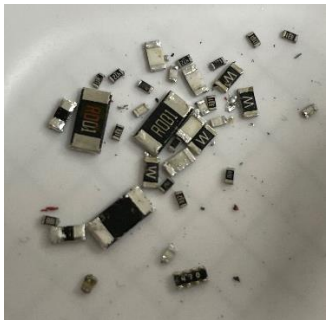
resistors	resistors	0.50	0.82
	chip resistors	0.32	
transistors		8.55	
capacitors	multilayer ceramic capacitors (MLCC)	3.39	4.10
	tantalum capacitors	0.49	
	solid state capacitors	0.22	
sensors		0.36	
electronic oscillator - amplifier		0.11	
board without components		43.20	
		total: 99.44 %	
		loss of material: 0.56 %	

Table 11. Results of components metal fraction characterization (ICP-OES analysis), expressed as element mass concentration

INDUCTORS (C 102) weight: 22.58 %		
	Cu: 33.6 % Fe: 1.27 % Zn: 0.17 %	Cr: 471.6 g/t Ag: 62.1 g/t
	Cu: 89.1 % Fe: 3.37 % Zn: 0.45 %	Cr: 1252 g/t Ag: 164.9 g/t
	description: black ceramic box with a copper wire internally other materials (62.3 %): plastics, ceramic materials, ferrite qualitative analysis: Sn	
method: they were broken and then the inner coil was submitted to aqua regia digestion		
INDUCTORS (DR73) weight: 5.38 %		
	Cu: 18.7 % Fe: 1.44 % Ni: 1.40 % Sn: 1.00 % Pb: 0.76 % Zn: 0.39 %	Ag: 143.4 g/t
	description: plastic/ceramic box with an inner copper coil and Sn-Ag solders other materials: plastics, ceramic qualitative analysis: Ca, Si	

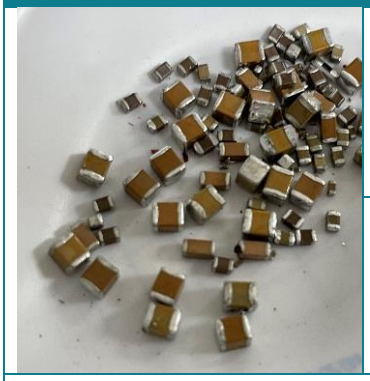
<table>
<tr><td colspan="4">method: they were broken and then the inner coil was submitted to aqua regia digestion</td></tr>
<tr><td colspan="4">ELECTRONIC OSCILLATOR – AMPLIFIER (TXC borde)
weight: 0.11 %</td></tr>
<tr><td rowspan="2">  </td><td>Fe: 13.7 %
Ni: 9.71 %
Sn: 1.47 %
Cu: 0.32 %</td><td colspan="2">Ag: 3675.3 g/t
Au: 181.2 g/t</td></tr>
<tr><td colspan="3">other materials: ceramic, quartz
qualitative analysis: Co</td></tr>
<tr><td colspan="4">method: aqua regia digestion</td></tr>
<tr><td colspan="4">BACK PANELS
weight: 10.05 %</td></tr>
<tr><td rowspan="2">  </td><td rowspan="2">  </td><td>Cu: 87.9 %
Sn: 0.91 %
Ni: 0.48 %
Zn: 0.48 %</td><td>Ag: 1260.7 g/t
Au: 1136.2 g/t
Pd: 132.4 g/t
Pb: 49.8 g/t
Al: 13.3 g/t</td></tr>
<tr><td colspan="2">qualitative analysis: Zr</td></tr>
<tr><td colspan="4">method: they were manually broken and then submitted to aqua regia digestion</td></tr>
<tr><td rowspan="2"> 
blue back panel</td><td colspan="2">Cu: 48.4 %
Zn: 38.9 %
B: 24.5 %
Al: 3.19 %
Sn: 3.02 %
Ni: 0.44 %
Ti: 0.14 %</td><td>Pd: 3068.6 g/t
Au: 963.8 g/t
Ag: 213.5 g/t
Pb: 47.2 g/t</td></tr>
<tr><td colspan="3">qualitative analysis: Ca, K, Na, Sb, Si, Zr</td></tr>
<tr><td rowspan="2"> 
black back panel</td><td colspan="2">Cu: 24.7 %
B: 2.92 %
Sn: 1.14 %
Al: 1.07 %
Ni: 0.62 %</td><td>Zn: 561.4 g/t
Au: 196.8 g/t
Ag: 89.8 g/t
Pb: 16.2 g/t
Pd: 14.1 g/t</td></tr>
<tr><td colspan="3">qualitative analysis: Ca, Na, K, Sb, Si, Ti, Zr</td></tr>
<tr><td rowspan="2"> 
box back panel</td><td colspan="2">Cu: 48.4 %
Zn: 19.5 %
B: 18.6 %
Al: 3.13 %
Sn: 1.24 %
Ni: 0.10 %</td><td>Au: 243.6 g/t
Ag: 196.4 g/t
Pb: 28.2 g/t</td></tr>
<tr><td colspan="3">qualitative analysis: Na, K, Pd, Si</td></tr>
<tr><td colspan="4">method: aqua regia digestion</td></tr>
<tr><td colspan="4">WHITE BACK PANELS
weight: 0.18 %</td></tr>
<tr><td></td><td colspan="2">Cu: 2.97 %
Sn: 0.30 %</td><td>Ni: 423.0 g/t
Ag: 260.7 g/t
Au: 39.9 g/t</td></tr>
</table>

	other materials: plastics qualitative analysis: Pd	
method: aqua regia digestion		
BACK PANELS' HOOKS **weight: 1.93 %**		
	Cu: 39.9 % Zn: 26.5 %	Ag: 620.2 g/t Ni: 416.0 g/t
	qualitative analysis: Sn	
method: aqua regia digestion		
SENSORS (bilf) **weight: 0.36 %**		
	Cu: 15.8 % Zn: 3.27 % Sn: 0.92 % Ni: 0.26 %	Ag: 1714 g/t Pd: 1516 g/t Au: 76.4 g/t
	description: plastic/ceramic box with a gold screw inside and Sn-Ag solders other materials: plastics, ceramic qualitative analysis: Al, Pb	
method: they were broken and then submitted to aqua regia digestion		
INTEGRATED CIRCUITS -CHIPS (ICC) **weight: 2.08 %**		
	Cu: 40.2 % Sn: 1.99 % Zn: 0.31 % Ni: 0.23 %	Au: 3688.5 g/t Ag: 319.6 g/t Pd: 103.1 g/t
	other materials: plastics qualitative analysis: Al, Ba, Ca, Fe, Pb	
method: they were cut and then submitted to aqua regia digestion		
RESISTORS **weight: 0.50 %**		
	Cu: 26.6 % Sn: 3.30 % B: 2.36 % Ti: 0.26 % Ni: 0.11 %	Ag: 689.4 g/t
	qualitative analysis: Al, Ba, Ca, Sb	
method: aqua regia digestion		

TRANSISTORS - (VBT) weight: 8.55 %		
	Cu: 52.0 % Sn: 1.06 % Al: 0.32 % Zn: 0.27 %	Ag: 414.7 g/t Au: 78.9 g/t Pd: 46.6 g/t Ni: 27.6 g/t
	other materials: silicon, ceramic (28 %) qualitative analysis: Pb, Si	
method 1: they were broken and then submitted to aqua regia digestion (detection of Cu, Sn, Al, Ni) method 2: they were submitted to thermal treatment with Na_2CO_3 (same amount as components) at 1060 °C for 1 h, then the melted components were quickly cooled, and then metallic alloy was submitted to aqua regia digestion (detection of Au, Ag, Pd)		
CHIP RESISTORS - (R001 - W) weight: 0.32 %		
	Cu: 47.5 % Mn: 5.62 % Ni: 5.05 % Sn: 4.19 %	Al: 584.0 g/t Pd: 490.8 g/t Ag: 483.6 g/t Pb: 300.2 g/t Ta: 65.6 g/t
	other materials: plastics qualitative analysis: In description: plastic and solders on the surface with inner electrode in precious metal	
method: they were broken and then submitted to aqua regia digestion		
SOLID STATE CAPACITORS weight: 0.22 %		
	Cu: 31.4 % Sn: 2.75 % Ti: 1.60 % Fe: 0.25 %	Ag: 605.6 g/t Au: 302.1 g/t Pd: 71.8 g/t
	other materials: ceramic qualitative analysis: Al, Ba, Ca, Er, Ni description: ceramic components with solders	
method: aqua regia digestion		
TANTALUM CAPACITORS weight: 0.49 %		
	Cu: 35.9 %	Ag: 1647 g/t Ta: 126.3 g/t Pd: 72.2 g/t Au: 28.4 g/t Mn: 19.3 g/t
	other materials: ceramic qualitative analysis: Al, K, Pb, Sn, Zn description: ceramic components with solders on two sides, inside there is a tantalum electrode	

method: they were broken, then were submitted to 1 hour thermal treatment at 350°C with KOH (two times the amount of solid), then the sample was dissolved in water at 90 °C for 1 h for the detection of Ta and Mn, the solid residue was submitted to nitric acid digestion for Ag and Cu detection and then to aqua regia digestion for Pd and Ru detection.

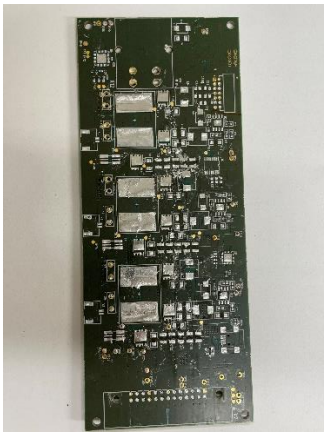
MULTI LAYER CERAMIC CAPACITORS (MLCC)
weight: 3.39 %



Ti: 10.23 % Cu: 3.29 % Nb: 0.49 %	Ag: 309.7 g/t Au: 15.5 g/t Pd: 6.3 g/t
other materials: ceramic qualitative analysis: Al, Ba, Ca, Fe, K, Dy, Mg, Ni, Si, Sn, Sr, Ti, Y, Zn, Zr	

method: they were broken, then were submitted to 1 hour thermal treatment at 350 °C with KOH (two times the amount of solid), then the sample was dissolved in water at 90 °C for 1 h for the detection of Nb, the solid residue was submitted to nitric acid for Ag and Cu digestion and then to aqua regia digestion for Au, Pd and Ti detection.

BOARD WITHOUT COMPONENTS
weight: 43.20 %

front side	back side
	

<table>
<tr><td> 
powders below to 2 mm</td><td> 
powders above to 2 mm have an average size of the particles of 5 mm, and 8-9 mm as maximum size</td></tr>
<tr><td colspan="2">Overall chemical composition of both fractions (below to 2 mm and above to 2 mm)</td></tr>
<tr><td>Cu: 35.1 %
Sn: 2.61 %
B: 1.35 %</td><td>Ag: 616.7 g/t
Pd: 68.9 g/t
Au: 46.6 g/t</td></tr>
<tr><td colspan="2">other materials: fiber glass, plastics
qualitative analysis: Al, Ca, Fe, Ni, Zn</td></tr>
<tr><td colspan="2">method: the board was cut and then grinded by a cut mill with a 2 mm grid; they were obtained two fractions, one below to 2 mm (75.7 %) and the other one above to 2 mm (24.3 %). The obtained powders were submitted to sampling and further digestion by aqua regia, three replications were performed.</td></tr>
</table>

The following figures are respectively the composition of PCBs in terms of components present (Figure 8) and in terms of metal content (Figure 9).

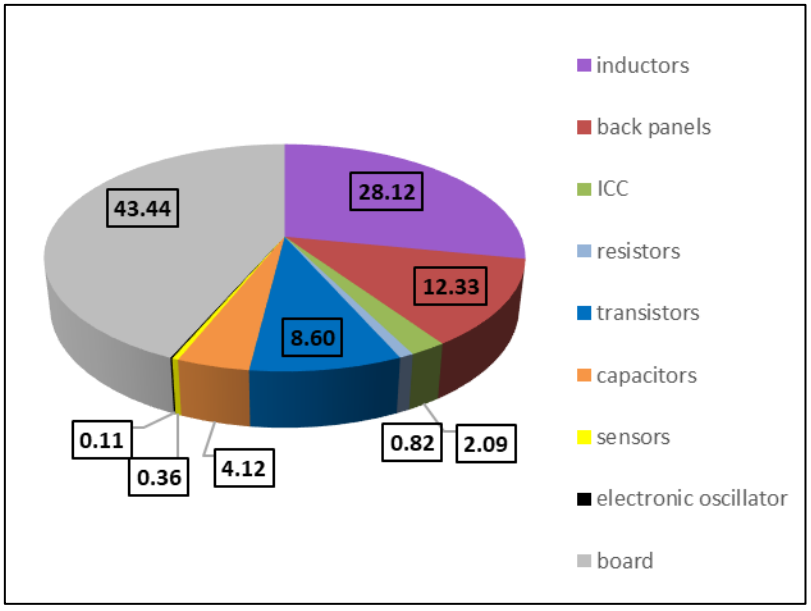


Figure 8. Weight distribution of PCBs components (normalized to 100 %)

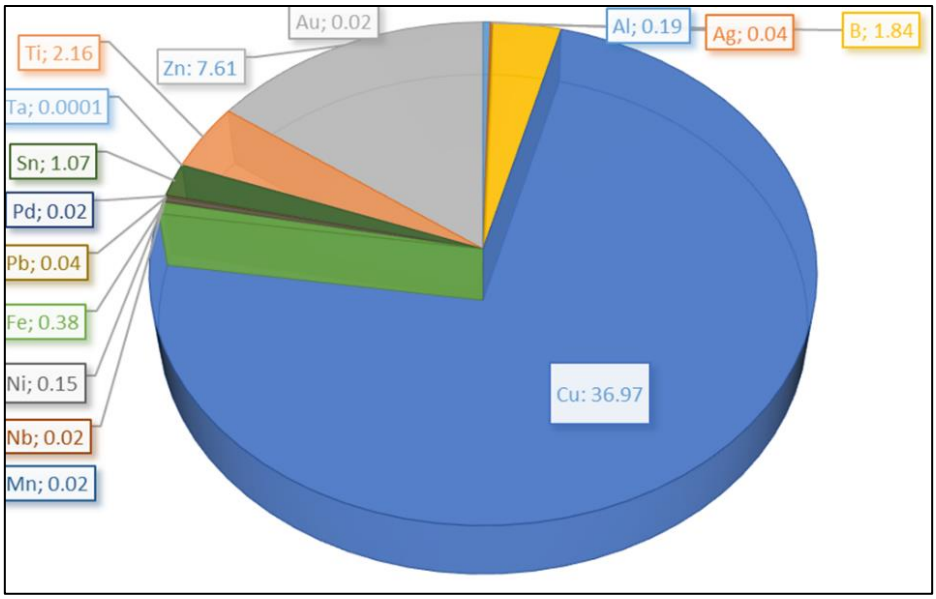


Figure 9. PCBs metals composition (normalized to 1)

3. Process recycling (lab-scale tests)

3.1 Flexible electronics 1 (TNO)

The flexible sensors (Figure 1) were subjected to two successive leaching steps (GOLD-REC1):

1. Dimethylformamide (DMF) Leaching aims to dissolve the plastic and facilitate access to the metal parts
2. The subsequent extraction of silver by Thiourea Leaching (Thiourea 20 g/L, $Fe_2(SO_4)_3$ 22 g/L, H_2SO_4 0.2 mol/L)

Iron from oxidation state +3 is reduced to oxidation state +2 leading to the oxidation of the silver which forms the complex $(Ag(SC(NH_2)_2)_3)^+$ with thiourea $(SC(NH_2)_2)$. $SC(NH_2)_2$ also partially reacts with Fe^{3+} leading to the formation of formamide disulfide $(C_2H_6N_4S_2)$ which decomposes into $SC(NH_2)_2$, cyanamide (CN_2H_2) to sulfur.

Leaching of silver occurs according to the following chemical reactions:

$$Ag + Fe^{3+} + SC(NH_2)_2 \rightarrow (Ag(SC(NH_2)_2)_3)^+ + Fe^{2+} \quad (1)$$

$$2\ SC(NH_2)_2 + 2\ Fe^{3+} \rightarrow C_2H_6N_4S_2 + 2\ Fe^{2+} + 2\ H^+ \quad (2)$$

$$2\ C_2H_6N_4S_2 \rightarrow SC(NH_2)_2 + CN_2H_2 + S \quad (3)$$

The residual silver content in solid material after thiourea leaching, was measured by attacking it with by use of aqua regia. Figure 10 shows the scheme of the process and Table 12 shows the results obtained.

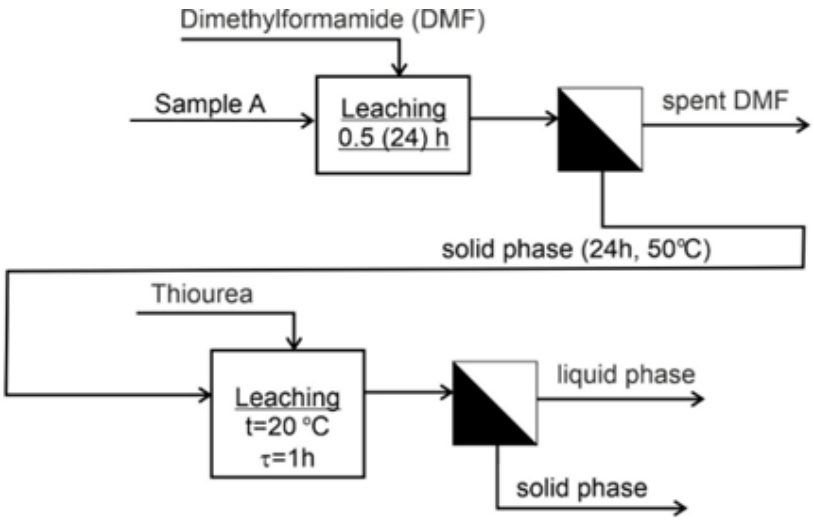


Figure 10. Simplified process scheme for flexible electronics 1

Table 12. Metal average content after Thiourea Leaching and Acid Attack

DMF treatment		Thiourea treatment	Acid Attack
Ag content in initial solid sample, **%**	**Ag** content in solid sample after DMF treatment**, %**	thiourea leaching **Ag, mg/L**	**Ag** content after Thiourea leaching, **%**
0.31	9.9	31.5	0.025

The thiourea leaching led to an Ag recovery yield of about **96.7%.**

Evaporation treatment has been studied to evaluate the effectiveness of a possible thermal treatment process for spent DMF. For testing, a laboratory rotary evaporator was used.

DMF was inserted into an evaporation flask that was immersed in a thermostatic bath to keep the solution at the appropriate temperature (Heidolph Rotavapor G3). The application of a depression helps to lower the boiling temperature of the solvents. Under vacuum, the solvents boil, and then evaporate, at lower temperatures than they would need working at atmospheric pressure. Furthermore, the rotation of the flask, creating a veil of solution, continuously renewed on the surface of the flask, allows to further increase the evaporation speed. The solvent vapors are removed and condense on contact with the cold surface of a coil in which cold mains water is made to flow. The condensate is collected in droplets in the collecting flask.

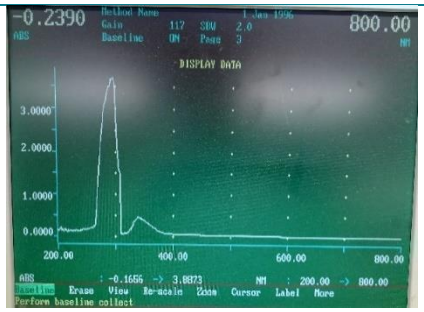
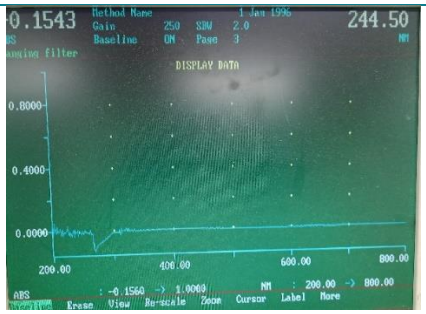
The test was carried out at a temperature of 68 °C working under vacuum (0.140 bar), the duration of the test was 30 min.

Table 13 shows the Fourier-transform infrared spectroscopy (FTIR[2]) spectra of the solid part of spent DMF after evaporation. Absorption spectrum of liquid part of spent DMF samples (distillate) (initial, after leaching and distillate after evaporation) were measured using UV–visible spectrophotometer (Table 14).

Table 13. UV-VIS spectrum of Spent DMF after leaching and after evaporation

Sample	Spent DMF after leaching	After evaporation (recycled DMF)
DMF		

[2] FTIR is a technique used to obtain an infrared spectrum of absorption or emission of a solid, liquid, or gas.

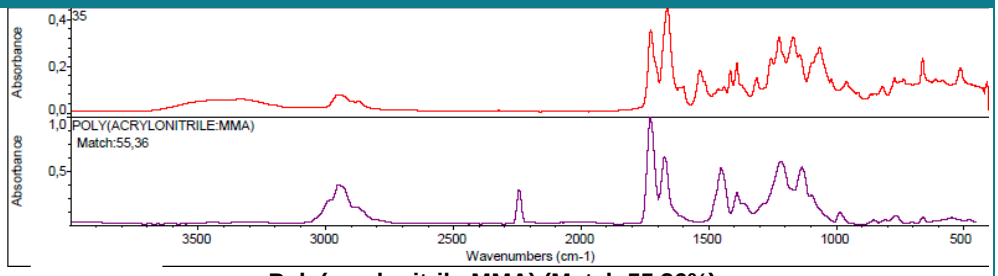
Flex circuits		

Comparison of the absorption spectra of the sample of DMF before leaching (baseline) with the spectra of samples of spent DMF (Table 14) shows that in all cases of spent DMF, after the leaching process, absorption maxima are observed at 280 and 348 nm. A similar pattern was observed for all spent DMF solutions after the leaching of studied spent flex circuits samples, although the ratio of intensities is somewhat different. It demonstrates the presence of dissolved impurities in the spent solution.

Instead, recycling the spent DMF solution by evaporation makes it possible to almost completely separate the dissolved impurities from the DMF distillate and thus allow its reuse in the leaching process.

The FTIR method carried out an analysis of the concentrate resulting from evaporation. Based on the FTIR spectra (Table 15), depending on the kind of flex circuits samples, after the leaching process, different chemicals were dissolved by DMF: Poly(acrylonitrile: MMA), 7,2-CHLOROETHYLTHEOPHYLLINE IN KB, Poly(ether-ester urethane) MBI, Polyurethane resin, Poly(ether-ester urethane) MBI, POLYETHERURETHANE, Poly(ester urethane).

Table 14. FTIR spectra of the solid part of spent DMF after evaporation

Sample	FTIR
Flex circuits	 **Poly(acrylonitrile:MMA) (Match 55.36%)**

3.2 In-mold electronics 2 (TNO)

A series of experimental tests were carried out to develop a process for the recovery of silver from the polycarbonate-based IME samples (A, B). The samples were submitted to lab-scale activities without reducing the size. The plastic frame was removed.

3.2.1 Thiourea leaching

For sample A, tests with three stages of thiourea leaching were performed: for each stage fresh leaching solutions were used.

Operative conditions:

- thiourea 20 g/L
- ferric sulphate 22.46 g/L
- H_2SO_4 0.2 mol/L
- pulp density 10 % w/v
- no stirring
- time 1 h.

Leaching of silver occurs according to reactions (1-3).

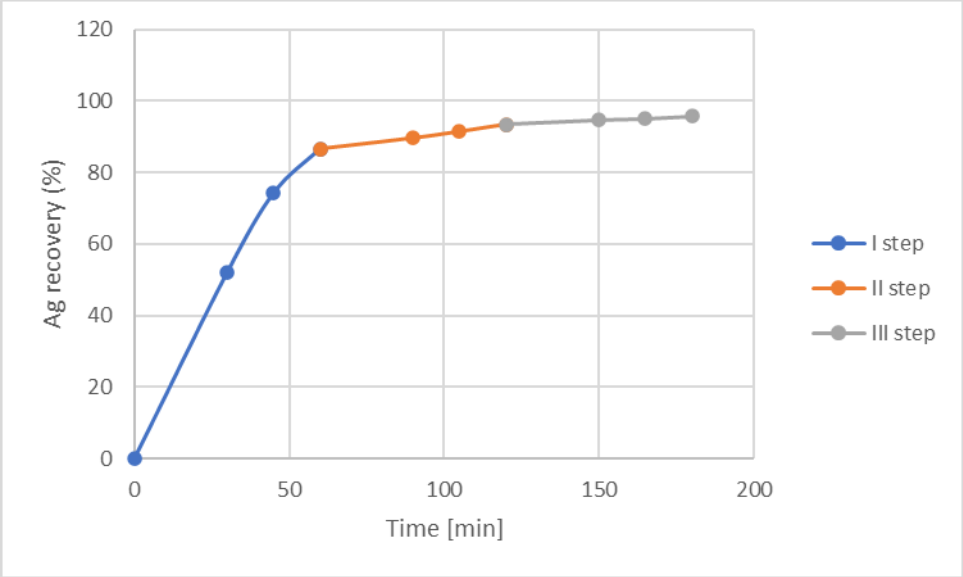


Figure 11. Results for sample A

Table 15. Results of silver dissolution by three stages of thiourea leaching for sample A

solid residue (98.9 % with respect to the initial solid)	Ag 156 ± 10 g/t
reconstituted feed	Ag 0.3623 ± 0.001%

For sample B, tests with two stages of thiourea leaching were performed: for each stage fresh leaching solutions were used.

Operative conditions:

- thiourea 20 g/L
- ferric sulphate 22.46 g/L
- H_2SO_4 0.2 mol/L
- pulp density 10 % w/v
- no stirring
- time 1 h

Table 16. Results for sample B

LEACHING OPERATIONS		
TREATMENT	Ag [mg/L]	Ag dissolution (%)
thiourea first step – 1 h	502.0	**65.0**
thiourea second step – 1 h	197.8	**90.7**

Table 17. Results of silver dissolution by three stages of thiourea leaching for sample B

solid residue (99.2 % with respect to the initial solid)	Ag 587 g/t
reconstituted feed	Ag 0.6284 %

In the next figure, a simplified scheme of the leaching process for the treatment of sample A was shown.

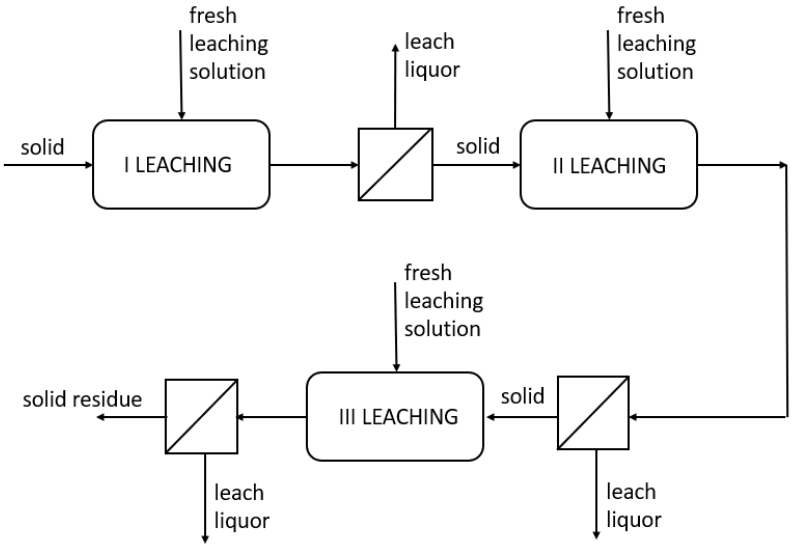


Figure 12. Simplified process scheme for leaching operation (sample A)

3.2.2 Thiourea leaching without oxidant agent

For sample B, tests with three stages of thiourea leaching were performed without using the oxidant agent: for each stage fresh leaching solutions were used.

Operative conditions:

- thiourea 20 g/L
- H_2SO_4 0.2 mol/L
- pulp density 10 % w/v
- no stirring
- time 1 h

Table 18. Results of silver dissolution by three stages of leaching without oxidant agent* for sample B

LEACHING OPERATIONS		
TREATMENT	**Ag [mg/L]**	**Ag cumulative dissolution (%)**
thiourea* first step – 1 h	19.5	2.4
thiourea* second step – 1 h	14.6	4.2
thiourea* third step – 1 h	9.9	5.4

The results showed that no significant Ag dissolution occurred performing thiourea leaching without the oxidant agent (Fe^{3+}).

3.2.3 Electrowinning – recovery of silver from the solution

Tests will be performed with electrodes of different materials and shapes. The figure below shows the photographic aspect of one of the tests performed.

Figure 13. Photographic aspect of electrolysis test 4

The reactions that occur during electrolysis are as follows:

$$Ag\,(SC(NH_2)_2)^{3+} + 3\,e^- \rightarrow Ag + 3\,(SC(NH_2)_2) \quad (1)$$

$$3\,C_2H_6N_4S_2 + 3\,H^+ + 3\,e^- \rightarrow 3\,SC(NH_2)_2 \quad (2)$$

The really important aspect that is noticed is the regeneration of the TU. In fact, thanks to this process it is possible to greatly reduce the amount of TU necessary for the process with consequent reduction of OPEX.

Table 19. Summary of electrowinning tests

TEST	solution	electrodes	operative conditions	results	purity of the obtained powder
n. 1	thiourea first step sample A (320 mg/L, volume 80 mL)	cathode and anode: copper, cylindrical shape	voltage: 1.5 V current density: 13 A/m^2	recovery: 65 % time: 2 h energetic consumption: 1.5 kWh/kg	Ag 68.1 % (as metal)
n. 2	thiourea first step sample A (328 mg/L, volume 80 mL)	cathode and anode: copper, cylindrical shape	voltage: 2.0 V current density: 37 A/m^2	recovery: 61 % time: 2 h energetic consumption: 4.8 kWh/kg	n.a.
n. 3	thiourea first step sample A (317 mg/L, volume 80 mL)	cathode: copper anode: graphite cylindrical shape	voltage: 1.5 V current density: 62 A/m^2	recovery: 79 % time: 2 h energetic consumption: 5.0 kWh/kg	n.a.
n. 4	thiourea first step sample B (500 mg/L, volume 90 mL)	cathode: copper anode: graphite cylindrical shape	voltage: 1.2 V current density: 43 A/m^2	**recovery: 95 %** time: 2 h energetic consumption: 2.9 kWh/kg	**Ag 75.4 %** (as metal)

The experimental tests, summarized in Table 19, allowed to identify the best operative conditions for the recovery of silver from the leaching solution by electrodeposition.

Cylindrical electrodes must be immersed in the solution to ensure good mixing of the solution in the equipment. Copper was used as cathode because in this case the silver powder can be directly obtained in the metallic form without a subsequent thermal refining phase [7]; graphite, instead, was used as anode. A silver recovery of 95 % occurred at the following conditions: voltage 1.2, current density 43 A/m^2. In Figure 14 is shown the silver recovery and current efficiency calculated at different times of test 4.

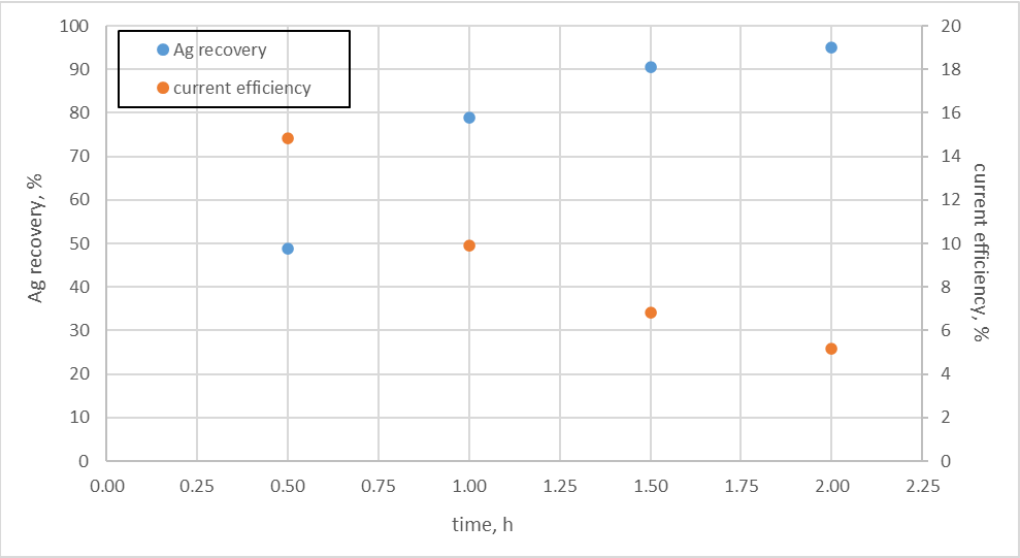


Figure 14. Silver recovery and current efficiency at different times of test 4

In the obtained powder was determined a grade of silver of around 75 %, graphite was detected as main impurity, probably due to the degradation of the anode.

In Figure 15 the X-Ray diffraction analysis of the obtained silver powder was showed. From the analysis the intensity of the peaks of silver as a metal is greater than that of silver as an oxide.

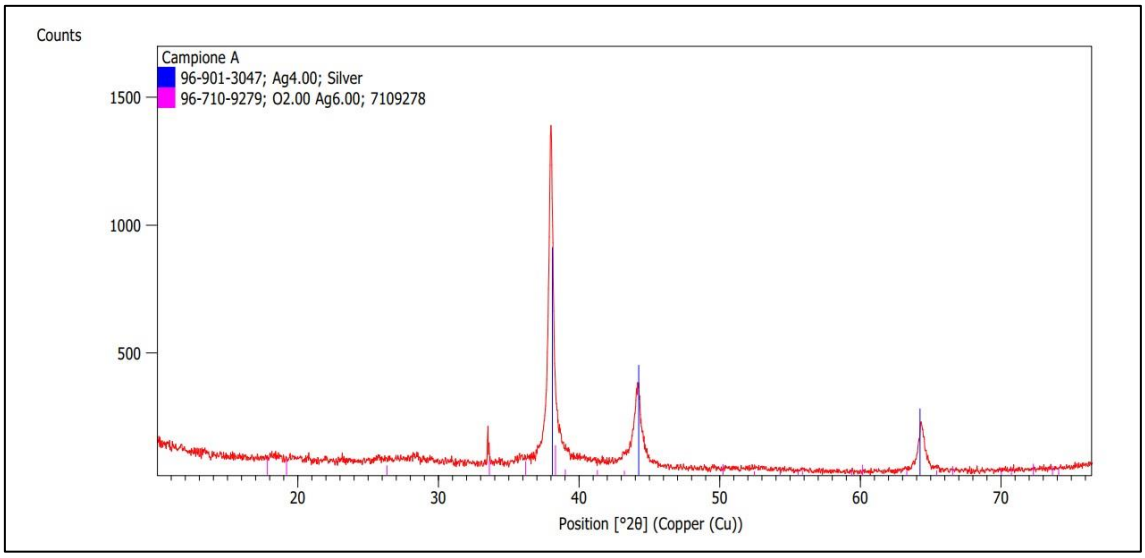


Figure 15. X-Ray diffraction analysis performed on silver powder (test 4)

3.2.4 Cross-leaching process

In order to study the cross-leaching process an iodometric titration method to detect the content of thiourea, Fe^{3+} and formamidine disulfide (FDS) was used. In addition, also a titration acid-base was performed to evaluate the sulfuric acid concentration. The results showed the opportunity to reuse the solution after the first step of leaching for the treatment of the same solid (second step of leaching) with a chemical make-up. After the electrodeposition operation, the solution was again titrated. So, the solution, discharged by the silver, can be reused for a new batch in the same way. The solutions can be recycled in 3 batches before disposing of as wastewater.

3.3 Car components (SEAT)

Several leaching tests were performed to evaluate the recovery efficiency of Au, Ag, Pd, Cu and Sn through Gold-Rec1 and Gold-Rec2 processes on the dashboard components of the various SEAT cars. The Printed Circuit Boards (PCBs) powders obtained by grinding with a knife mill as described in chapter 4.2 were subjected to the Gold-Rec1 process. The other components were treated with the Gold-Rec2 process.

Based on the Au content, the technical feasibility of the grinding and the background of UNIVAQ [4–6] it was decided to treat the components with high Au concentrations (i.e. Au > 300 ppm) with Gold-Rec2 without any reduction dimensional while the residual parts were ground and treated with powders using Gold-Rec1.

In addition, some components must be removed as they inhibit the recovery of gold, others cannot be subjected to grinding and others with high Cu concentrations lead to a significant increase in operating costs. Some of these components are batteries, Al electrolytic capacitors, Quartz oscillators or Inductors. Components with high copper concentrations are not convenient to treat with a hydrometallurgical process and a pyrometallurgical treatment is preferred.

3.3.1 PCBs powders Leaching

The conditions of the tests carried out on the powders are the following:

- Solid concentration 15% wt./vol.
- Mixing 200 rpm
- H_2O_2 20% vol.
- H_2SO_4 10% vol.
- Thiourea 20 g/L
- Ferric sulphate 22.46 g/L

The tests carried out were structured on 3 steps each. The first two 90 min steps for the leaching of the base metals and the third 60 min step for the leaching of gold and silver by thiourea. Table 20 shows the results obtained in terms of recovery yields.

Table 20. Extraction yields for different car model powders

Model	Extraction yields	Step I	Step II	Step III	Total
Leon II	Au	0.5%	0.0%	48.7%	**48.7%**
	Ag	4.5%	0.8%	43.8%	**43.8%**
	Pd	71.6%	0.0%	8.5%	**8.5%**
	Cu	87.1%	7.5%	0.3%	94.6%
Leon III	Au	1.8%	0.0%	58.8%	**58.8%**
	Ag	14.5%	0.3%	54.0%	**54.0%**
	Pd	63.7%	10.8%	8.5%	**8.5%**
	Cu	80.7%	9.8%	0.2%	90.5%
	Sn	8.8%	3.5%	0.5%	12.2%
Ibiza IV	Au	0.0%	0.0%	61.7%	**61.7%**
	Ag	37.5%	28.2%	21.1%	**21.1%**
	Pd	53.6%	13.6%	8.9%	**8.9%**
	Cu	83.5%	14.0%	0.1%	97.5%
	Sn	86.7%	3.3%	0.4%	90.0%

The obtained leaching yields are relatively low if compared with the yields obtained with the same process on materials very similar to those used. Some differences on yield of extraction from the models depend on the different components on the boards.

In this regard, the studied process is to be studied in more detail for its optimization. Unfortunately, the time available from the time of arrival of the materials did not allow for this in-depth analysis. The process optimization will be completed in the D5.4.

3.4 Liquid crystal displays (EuroLCDs)

A series of experimental tests was carried out starting from a literature review [3] in order to develop a process for the recovery of indium and tin. Preliminary tests aimed at evaluating the necessity of a preliminary treatment to free metals from coating's layers. Hence, before a test with acetone treatment before leaching operation was compared with that carried out without preliminary treatment.

3.4.1 Preliminary tests

TEST I. Acetone preliminary treatment – sulfuric acid leaching

This preliminary test was performed to understand the behavior of this material in different situations. Washing using acetone and water is necessary for the removal of the protective layer covering the metals in order to make them accessible during the leaching phase with sulfuric acid.

Table 21. Operative conditions for TEST I

STAGE		OPERATIVE CONDITIONS
1	washing by acetone solid 10.124 g volume 101 mL	solid concentration 10 % wt./vol. time 90 min, stirring 250 rpm
2	washing with water volume 100 mL	washing on the filter

3	sulfuric acid leaching solid 10.110 g volume 100 mL	sulfuric acid 2 mol/L solid concentration 10 % wt./vol. stirring 250 rpm, temperature 60 °C, time 60 – 90 min.
4	washing water in 2 steps/(15 mL and 15 mL)	washing on the filter
5	chemical attack of solid residue by aqua regia	time 120 min temperature 90 °C

Table 22. Results for TEST I

STAGE		RESULTS	YIELD OF DISSOLUTION (%)	
			In	Sn
1	washing by acetone	Loss of weight: 0.14%	-	-
2	washing by water	-	-	-
3	sulfuric acid leaching	1h: In 6.84 mg/L – Sn 0.88 mg/L 1h30: In 3.28 mg/L – Sn 1.12 mg/L	1h: **98.5%** 1h30: 52.4%	1h: 52.5% 1h30: **74.5%**
4	washing water 1 washing water 2	1: In 1.47mg/L – Sn 0.93 mg/L 2: In 0.41 mg/L – Sn 0.23 mg/L	-	-
5	chemical attack of solid residue	-	-	-

Reconstituted feed: **In: 68.60 g/t – Sn: 16.55 g/t.**

TEST II. Direct sulfuric acid leaching

Table 23. Operative Conditions for TEST II

	STAGE	OPERATIVE CONDITIONS
1	sulfuric acid leaching solid 9.961 g volume 100 mL	sulfuric acid 2 mol/L solid concentration 10 % w/v, stirring 250 rpm, temperature 60 °C, time 1h – 1h30.
2	Washing with water 30 mL	washing on the filter
3	chemical attack of solid residue by aqua regia	time 2 h temperature 90 °C

Table 24. Results for TEST II

	STAGE	RESULTS	YIELD OF DISSOLUTION (%)	
			In	Sn
1	sulfuric acid leaching	30': In 3.36 mg/L – Sn 1.16 mg/L 1h In 5.10 mg/L – Sn 1.88 mg/L	30': 54.9% 1h: **80.6%**	30': 37.8% 1h: **65.0%**
2	washing water	In 0.92 mg/L – Sn 0.75 mg/L	-	-
3	chemical attack of solid residue	-	-	-

Reconstituted feed: **In: 61.50 g/t – Sn: 30.80 g/t.**

3.4.2 Leaching tests: factorial design plan

Three factors are selected to carry out the factorial design plan with central composite: temperature, sulfuric acid concentration and pulp density. Every leach operation is carried out for 1.5 h. In the following table are shown their coded names and their high, mid and low levels.

Table 25. Coded names and levels

Factor	Coded Name	Low level (-)	Mid level (0)	High Level (+)
Temperature	A	25 °C	42,5°C	60 °C
H_2SO_4	B	1 mol/L	1,5 mol/L	3 mol/L
Solid %	C	5 % wt/vol	10 % wt/vol	15 % wt/vol

The following tables shows the results.

Table 26. Dissolution yields for indium obtained from factorial design plan

test	A	B	C	AB	AC	BC	ABC	A^2	B^2	C^2	In dissolution, %
1	-1	-1	-1	1	1	1	-1	1	1	1	0,91
2	1	-1	-1	-1	-1	1	1	1	1	1	98,91
3	-1	1	-1	-1	1	-1	1	1	1	1	0,58
4	1	1	-1	1	-1	-1	-1	1	1	1	99,85
5	-1	-1	1	1	-1	-1	1	1	1	1	2,37
6	1	-1	1	-1	1	-1	-1	1	1	1	99,29
7	-1	1	1	-1	-1	1	-1	1	1	1	5,05
8	1	1	1	1	1	1	1	1	1	1	99,69
9	-1,682	0	0	0	0	0	0	2,82912	0	0	1,25
10	1,682	0	0	0	0	0	0	2,82912	0	0	99,92
11	0	-1,682	0	0	0	0	0	0	2,82912	0	4,37
12	0	1,682	0	0	0	0	0	0	2,82912	0	23,18
13	0	0	-1,682	0	0	0	0	0	0	2,82912	4,1
14	0	0	1,682	0	0	0	0	0	0	2,82912	10,28
15	0	0	0	0	0	0	0	0	0	0	9,25
16	0	0	0	0	0	0	0	0	0	0	9,31
17	0	0	0	0	0	0	0	0	0	0	7,53

Table 27. Significative factors for indium dissolution

	Coefficients	*Standard error*	*Stat t*	*Significance value*	*Less then 95%*	*More then 95%*	*1-p*	*Significance*
Intercept	7,02	13,90	0,51	0,63	-26,98	41,03	0,37	36,87
A	40,62	6,53	6,22	0,00	24,65	56,59	1,00	99,92
B	2,59	6,53	0,40	0,71	-13,38	18,56	0,29	29,44
C	1,21	6,53	0,19	0,86	-14,76	17,18	0,14	14,11
AB	-0,13	8,53	-0,01	0,99	-20,99	20,74	0,01	1,13
AC	-0,71	8,53	-0,08	0,94	-21,58	20,15	0,06	6,40
BC	0,31	8,53	0,04	0,97	-20,56	21,17	0,03	2,77
ABC	-0,44	8,53	-0,05	0,96	-21,31	20,42	0,04	3,98
A^2	20,59	7,18	2,87	0,03	3,01	38,16	0,97	97,14
B^2	7,58	7,18	1,06	0,33	-10,00	25,15	0,67	66,80
C^2	5,25	7,18	0,73	0,49	-12,32	22,83	0,51	50,77

As it's shown in the previous table, the temperature results to be the only significative factor with a positive effect on the dissolution yield of indium. Also, a response curve can be developed from those results.

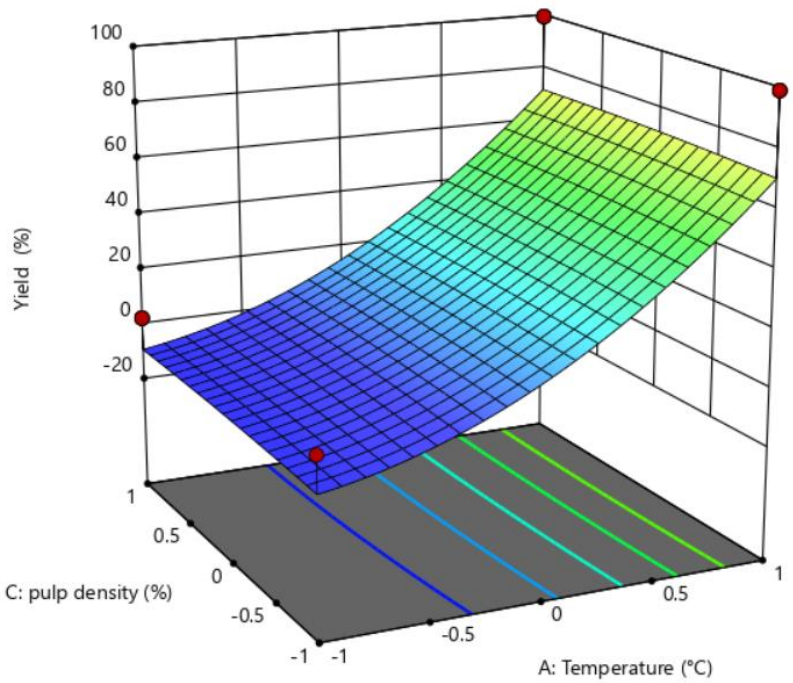


Figure 16. Response curve for indium dissolution yield

Table 28. Dissolution yields for tin obtained from factorial design plan

test	A	B	C	AB	AC	BC	ABC	A^2	B^2	C^2	Sn dissolution, %
1	-1	-1	-1	1	1	1	-1	1	1	1	76,62
2	1	-1	-1	-1	-1	1	1	1	1	1	22,16
3	-1	1	-1	-1	1	-1	1	1	1	1	73,22
4	1	1	-1	1	-1	-1	-1	1	1	1	68,02
5	-1	-1	1	1	-1	-1	1	1	1	1	9,2
6	1	-1	1	-1	1	-1	-1	1	1	1	47,02
7	-1	1	1	-1	-1	1	-1	1	1	1	14,36
8	1	1	1	1	1	1	1	1	1	1	90,71
9	-1,682	0	0	0	0	0	0	2,82912	0	0	82,46
10	1,682	0	0	0	0	0	0	2,82912	0	0	87,59
11	0	-1,682	0	0	0	0	0	0	2,82912	0	14,12
12	0	1,682	0	0	0	0	0	0	2,82912	0	67,15
13	0	0	-1,682	0	0	0	0	0	0	2,82912	72,22
14	0	0	1,682	0	0	0	0	0	0	2,82912	19,28
15	0	0	0	0	0	0	0	0	0	0	11,49
16	0	0	0	0	0	0	0	0	0	0	20,48
17	0	0	0	0	0	0	0	0	0	0	12,09

Table 29. Significative factors for tin dissolution

	Coefficients	*Standard error*	*Stat t*	*Significance value*	*Less then 95%*	*More then 95%*	*1-p*	*Significance*	*1-p*	*Significance*
Intercept	15,30	5,82	2,63	0,04	1,07	29,53	1,07	29,53	0,96	**96,10**
A	4,62	2,73	1,69	0,14	-2,06	11,30	-2,06	11,30	0,86	85,86
B	13,22	2,73	4,84	0,00	6,53	19,90	6,53	19,90	1,00	**99,71**
C	-12,28	2,73	-4,50	0,00	-18,97	-5,60	-18,97	-5,60	1,00	**99,59**
AB	10,97	3,57	3,08	0,02	2,24	19,70	2,24	19,70	0,98	**97,82**
AC	21,73	3,57	6,09	0,00	13,00	30,46	13,00	30,46	1,00	**99,91**
BC	0,80	3,57	0,22	0,83	-7,93	9,53	-7,93	9,53	0,17	16,97
ABC	-1,34	3,57	-0,38	0,72	-10,07	7,39	-10,07	7,39	0,28	28,01
A^2	22,75	3,01	7,57	0,00	15,39	30,10	15,39	30,10	1,00	**99,97**
B^2	7,06	3,01	2,35	0,06	-0,30	14,41	-0,30	14,41	0,94	94,28
C^2	8,86	3,01	2,95	0,03	1,51	16,22	1,51	16,22	0,97	**97,44**

As it's shown in the previous table, the sulfuric acid concentration and pulp density result to be significative factors respectively with a positive and a negative effect on the dissolution yield of indium; interactions between temperature and sulfuric acid concentration and between temperature and pulp density also result to be significative with a positive effect. Also, a response curve can be developed from those results.

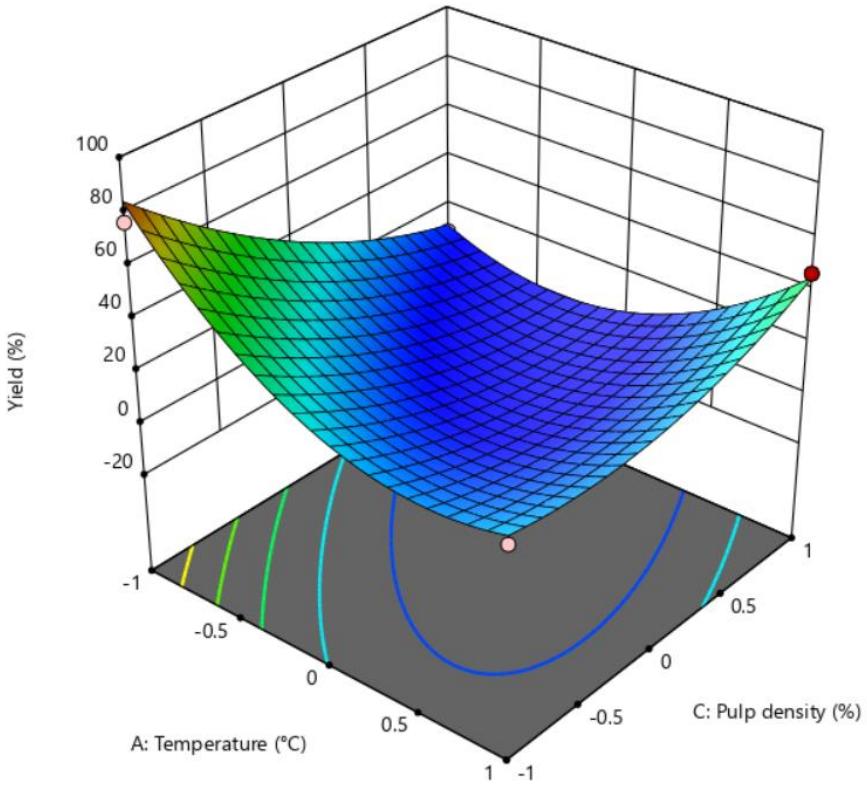


Figure 17. Response curve for tin dissolution yield

In order to maximize indium dissolution yield, because it's the precious metal, the best run results to be AC (T=60 °C; H_2SO_4 concentration= 1 mol/L; pulp density= 15 % wt/v) with a dissolution yield of 99.29 % for indium and 47.02 % for tin. Also, this run was chosen as the best one because it allows to reduce sulfuric acid consumption and to increase process potential.

To further reduce reagents consumption other tests with a sulfuric acid concentration of 0.5 mol/L were carried out. The dissolution yields were found to be very similar to the best run ones, so the perfect choice is to work with a sulfuric acid concentration.

To recover metals from leach liquors a tin and indium precipitations are necessary. Tin precipitation is performed by using polyamine and indium precipitation is about getting an insoluble indium salt that is insoluble in water (like indium carbonate).

The precipitation tests have not been carried out as it is possible to have high precipitation yields only by recirculating the leaching solution for numerous steps until reaching high concentrations of the two elements under consideration. The latter will be studied in detail on the pilot plant (D5.4).

3.5 Liquid crystal displays – printed circuit boards (EuroLCDs)

Several leaching tests were performed to evaluate the recovery efficiency of Au, Ag, Pd, Cu and Sn through Gold-Rec1 and Gold-Rec2 processes on the AF81 components. The Gold-Rec1 process was performed for electronic board powders (after grinding) while Gold-Rec2 was used for the different devices (for example Blue Back Panel and Large Black Connection Devices).

3.5.1 PCBs powders leaching

The conditions of the Gold-Rec1 test carried out on the powders are the following:

- Solid concentration 15% wt./vol.
- Mixing 200 rpm
- H_2O_2 20% vol.
- H_2SO_4 10% vol.
- Thiourea 20 g/L
- Ferric sulphate 22.46 g/L

The test carried out was structured on 4 steps. The first three 90 min steps for the leaching of the base metals and the third 60 min step for the leaching of gold and silver by thiourea. Table 31 shows the results obtained in terms of recovery yields.

Table 30. Characterization of PCBs powders

Element	Concentration
Au [ppm]	73.37
Ag [ppm]	548.43
Pd [ppm]	38.66
Cu (%)	10.01%
Sn (%)	2.50%

Table 31. Extraction yields for different AF81 powders

Extraction yields	Step I	Step II	Step III	Step IV	Total
Au	0.6%	1.0%	0.5%	24.3%	**24.3%**
Ag	4.3%	3.3%	2.7%	52.3%	**52.3%**
Pd	57.3%	14.0%	5.5%	8.1%	**8.1%**
Cu	10.0%	53.5%	20.2%	0.8%	83.7%
Sn	27.9%	6.9%	8.1%	0.3%	42.9%

The fact of having obtained such low extraction yields of precious metals certainly depends on the presence of some components that hinder leaching (for example, components with a high copper content). In order to have higher yields it is necessary to remove these components before proceeding with the grinding. In this sense, the percentage of copper or other substances (mainly organic) which have a negative effect on the yield is reduced. For example, it has been seen that inductances must be removed. In this way it is possible to minimize the amount of metals that remain in the matrix.

3.5.2 Blue Back Panel leaching

The conditions of the Gold-Rec2 test carried out on the Blue Back Panel are the following:

- Solid concentration 15% wt./vol.
- Mixing 200 rpm
- H_2O_2 20% vol.
- $C_2H_4O_2$ 10% vol.
- HCl 3.5 M

The test carried out was structured on 2 steps of 3 hours each. Table 33 Table 31shows the results obtained in terms of recovery yields.

Table 32. Characterization of Blue Back Panel

Element	Concentration
Au [ppm]	174.55
Ag [ppm]	204.62
Pd [ppm]	742.00
Cu (%)	28.99%
Sn (%)	1.45%

Table 33. Extraction yields for Blue Back Panel

Extraction yields	Step I	Step II	Total
Au	15.4%	67.9%	**83.4%**
Ag	60.8%	11.4%	**72.2%**
Pd	52.2%	45.1%	**97.3%**
Cu	85.5%	13.9%	99.5%
Sn	99.2%	0.4%	99.6%

3.5.3 Large Back Panel leaching

The conditions of the tests carried out on the Large Black Connection Devices are the following:

- Solid concentration 15% wt./vol.
- Mixing 200 rpm
- H_2O_2 20% vol.
- $C_2H_4O_2$ 10% vol.
- HCl 3.5 M

The test carried out was structured on 2 steps of 3 hours each. Table 35 Table 31shows the results obtained in terms of recovery yields.

Table 34. Characterization of Large Black Connection Devices

Element	Concentration
Au [ppm]	731.92
Ag [ppm]	544.12
Pd [ppm]	54.89
Cu (%)	74.00%
Sn (%)	3.92%

Table 35. Extraction yields for Large Black Connection Devices

Extraction yields	Step I	Step II	Total
Au	0.4%	8.1%	**8.6%**
Ag	42.0%	31.2%	**73.2%**
Pd	68.7%	31.3%	**100.0%**
Cu	64.2%	35.7%	99.9%
Sn	0.0%	98.0%	98.0%

The gold recovery yield is very low. This may be because the gold is less exposed to acid and therefore an additional stage of treatment may be required. Unfortunately, the material is no longer available to be able to proceed with further experimental tests.
All processes were carried out using new reagents for each step. To reduce consumption, a cross-leaching configuration will be carried out on a pilot scale. The obtained leaching yields are relatively low if compared with the yields obtained with the same process on materials very similar to those used. In this regard, the studied process is to be studied in more detail for its optimization.

4. Process analysis: simulation and cost analysis

4.1 Flexible electronics (TNO)

Batch leach tests were conducted by mixing the samples material with Dimethylformamide (DMF leaching) in a glass vessel, with agitation for 0.5 and 24 h respectively, and separating the leachate from solid material by centrifugation. Spent leachate (spent DMF) was subjected to evaporation treatment in order to evaluate the effectiveness of DMF recycling. Instead, solid material was subjected to subsequent Thiourea Leaching.

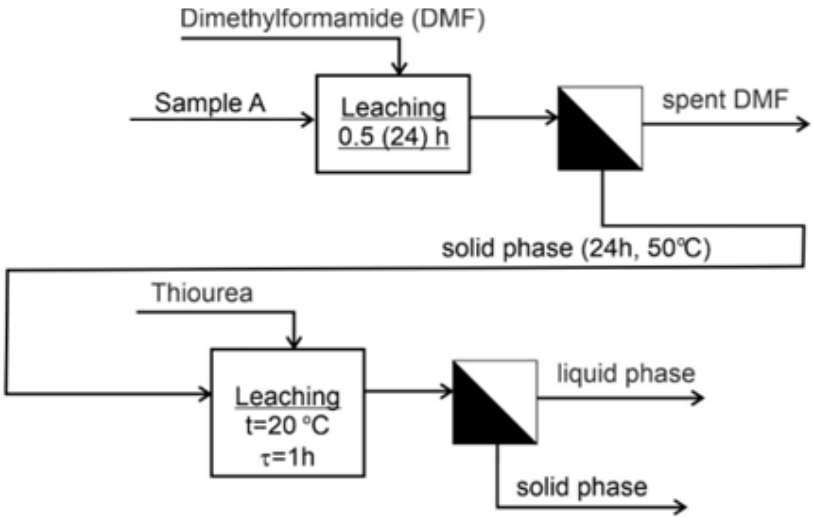


Figure 18. Simplified process scheme for flexible electronics 1

On the basis of the experimental tests carried out on a laboratory scale (chapter 3.1), the material balances were made. The recovery yield of Ag is about 97%. Thanks to the material balance it was possible to define the OPEX of the process both for a scenario without DMF recirculation, and for a scenario with the recirculation of about 70% of this reagent. The tables below report the results obtained.

Table 36. Mass balance for the treatment of a ton of Flex Circuits

INPUT		OUTPUT	
Description	kg	**Description**	kg
Solid (Flex Circuits)	1 000.0	Solid (spent plastic)	415.2
DMF	7 802.6	Silver powder	3.12
Water	3 693.3	Plastic after evaporation	613.5
Thiourea	70.9	Solid waste after Ag leaching	81.2
Ferric sulfate	78.1	Wastewater	3 836.0
Sulfuric acid, 50 %	106.7	Spent DMF non-recyclable	2 340.8

Table 37. Chemical consumption and costs for the recycling of silver from a ton of Flex Circuits

Chemicals	Mass, kg	Unit cost, €/kg	Cost, €
DMF	7 802.6	1.2	9 363.1
Thiourea	70.9	1.00	70.90
Ferric sulphate	78.1	0.30	23.43
Sulfuric acid, 50 %	106.7	0.13	13.8
Water	3 693.3	0.0015	5.54
			Total 9 476.7
OPEX			
items			cost, €
chemicals			**9 476.7**
wastewater to disposal	100 €/m^3		383.6
solid disposal			
silver powder	0.72 €/g		**2 246.4**
			Total - 7 613.9

Table 38. Chemical consumption and costs for the recycling of silver from a ton of Flex Circuits

Chemicals	Mass, kg	Unit cost, €/kg	Cost, €
Considering 70% DMF recycling	2340.8	1.20	2808.9
Thiourea	70.9	1.00	70.90
Ferric sulphate	78.1	0.30	23.43
Sulfuric acid, 50 %	106.7	0.13	13.8
Water	3693.3	0.0015	5.54
			Total 2 922.6
OPEX			
items			cost, €
chemicals			2 922.6
wastewater to disposal	100 €/m^3		383.6
solid disposal			recycling evaluation
silver powder	0.72 €/g		**2 246.4**
			Total - 1 059.8

The residual solid was not analyzed in detail but consists of spent plastic. The wastewater was characterized with semi-quantitative analysis by ICP-OES. The wastewater contains thiourea and related degradation compounds, with different metals. The main results obtained are summarized in the table below.

Table 39. Semi-quantitative analysis of wastewater (ICP-OES analysis)

Metal	Concentration [mg/L]
Fe	1000

Cu	60
Mn	10

For the moment, the price of the metal with a purity of 100% has been considered in the balance sheet. No other metal impurities were detected from the analyses on the silver dust obtained. Impurities are organic materials from chemicals and anode material. Probably, after refining, the purity of silver approaches 100%. Due to the small quantities obtained on the lab scale, this aspect has to be evaluated on a pilot scale.

4.2 In-mold electronics (TNO)

The proposed process for the treatment of this material consists of 2 leaching steps in cross-leaching configuration with make-up of chemicals in the second step. At the end of each of the two steps, the solid phase is separated from the liquid phase by means of a filtering system. From the second filtration, the resulting solution is sent to an electrolysis cell. From here comes out a liquid stream with about 25 mg/L of Ag which is recirculated at the head of the process, and a solid stream made up of silver powder.

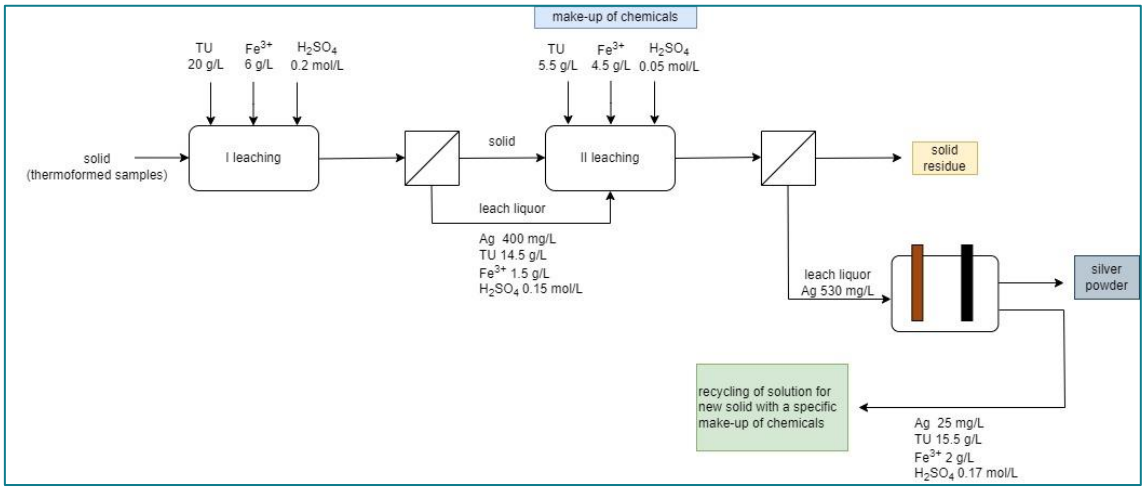


Figure 19. Flowsheet for the recycling of silver from thermoformed samples

On the basis of the experimental tests carried out on a laboratory scale (chapter 3.2), the material balances were made. The recovery yield of Ag is about 90%. Thanks to the material balance it was possible to define the OPEX of the process. Table 40 reports the material balance of the process while in Table 41 is shown the chemicals costs.

Table 42 summarizes the OPEX of the process.

Table 40. Mass balance for the treatment of a ton of thermoformed samples

INPUT		OUTPUT	
description	kg	description	kg
solid (thermoformed samples)	1000.0	solid (thermoformed samples)	991.6
water	3143.5	silver powder	4.4
thiourea	151.7	wastewater	3908.0
ferric sulphate	343.1		
sulfuric acid, 50 %	265.7		
Total	4904.0		4904.0

Table 41. Chemical consumption and costs for the recycling of a ton of thermoformed samples

CHEMICALS	MASS [kg]	UNIT COST [€/kg]	COST [€]
thiourea	151.7	1.00	151.70
ferric sulphate	343.1	0.30	102.93
sulfuric acid, 50 %	265.7	0.13	34.54
water	3143.5	0.0015	4.72
			Total 293.89 €

Table 42. Operating costs for the recycling of a ton of thermoformed samples

ITEMS		COST [€]
Chemicals		- 293.89
wastewater to disposal	100 €/m^3	- 379.50
solid disposal		…
energy consumption	electrowinning (3 kWh/kg) - 0.20 €/kWh	- 2.62
	agitators	…
silver powder	0.72 €/g	+ 3146.40
Total		**+ 2470,39**

The wastewater was characterized with semi-quantitative analysis by ICP-OES. The wastewater contains thiourea and related degradation compounds, with different metals summarized in the table below.

Table 43. Semi-quantitative analysis of wastewater

Metal	Concentration [mg/L]
Fe	1000
Cu	50
Mn	20

Currently, the price of the metal with a purity of 100% has been considered in the balance sheet. No other metal impurities were detected from the analyzes on the silver dust obtained. Impurities are organic materials from chemicals and anode material. Probably, after refining, the purity of silver approaches 100%. Due to the small quantities obtained on the lab scale, this aspect has to be evaluated on a pilot scale.

4.3 Electronic car components (SEAT

4.3.1 PCBs powders leaching

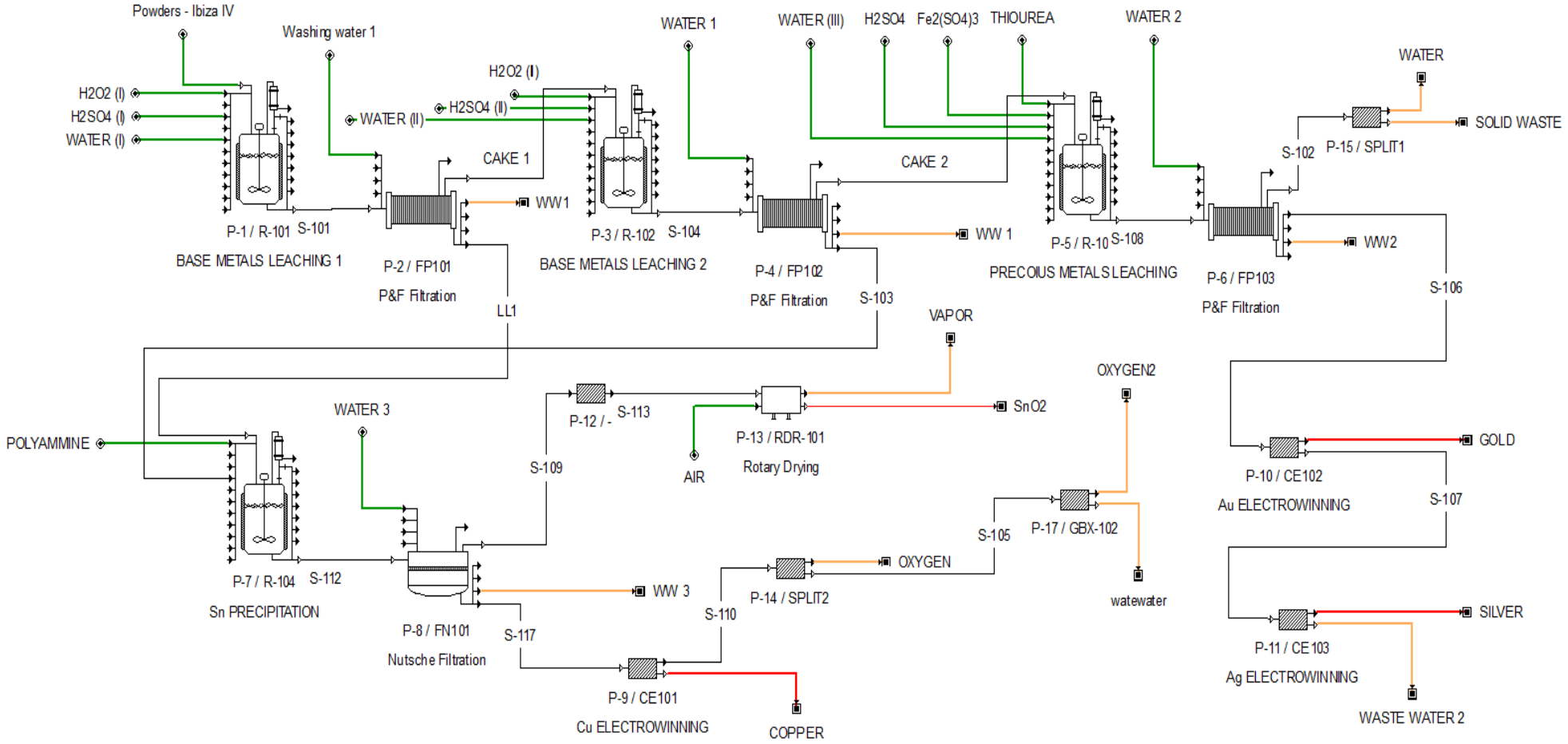


Figure 20. Simulation of Gold-Rec1 process for Ibiza IV powders treatment

On the basis of the experimental tests carried out on a laboratory scale (chapter 3.3.1), the material balances were made. The recovery yield of all elements is reported below:

- Gold: 62%
- Silver: 21%
- Copper: 98%
- Tin: 90%

Thanks to the material balance it was possible to define the OPEX of the process.

Table 44. Material balance for of Gold-Rec1 process for Ibiza IV powders treatment

INPUT		OUTPUT	
Solid [kg]	1000	Solid [kg]	766
STEP 1			
Hydrogen peroxide, 30% [L]	1333	Gold [kg]	0.032
Sulfuric Acid, 96% [L]	667	Silver[kg]	0.136
Water [L]	4667	Palladium [kg]	-
Washing Water [L]	2000	Copper [kg]	160.75
STEP 2		Tin Oxide [kg]	15.43
Hydrogen peroxide, 30% [L]	1333	Wastewater [L]	21802
Sulfuric Acid, 96% [L]	667		
Water [L]	4667		
Washing Water [L]	2000		
STEP 3			
Sulfuric Acid, 96% [L]	87		
Thiourea [kg]	154		
Ferric sulphate [kg]	173		
Water [L]	7636		
Washing Water [L]	2000		

Table 45. Operating costs for the recycling of a ton of dashboard electronic boards powders from Ibiza IV without recycling of water

Chemicals	Mass [kg]	Unit Cost [€/kg]	Cost [€]
Hydrogen peroxide, 35%	2537	0.40 €	1,014.86 €
Sulfuric acid, 50%	5016	0.13 €	652.10 €
Thiourea	154	1.00 €	154.46 €
Ferric sulphate	173	0.30 €	51.93 €
Water	21228	0.0015 €	31.84 €
Total			890.34 €
OPEX (1000 kg/h sample)	**(No recycling of water)**		
Chemicals		-	890.34 €
Wastewater (to disposal)	150 €/m^3	-	3,270.30 €
Solid (to disposal)	100 €/ton	-	76.60 €
Energy consumption	Agitators (6x20kW)	-	52.55 €
Precipitation section	Electrowinning (3x3kWh/kg)	-	169.42 €

	Chemicals	-	32.17 €
Total		-	4,491.38 €
Products	**Unit Cost [€/kg]**		
Gold	53,696.49 €		1,718.29 €
Silver	621.00 €		84.58 €
Palladium	63,272.67 €		- €
Copper	3.50 €		562.63 €
Tin Oxide	50.00 €		771.50 €
REVENUES		**-**	**1,354.39 €**

Table 46. Operating costs for the recycling of a ton of dashboard electronic boards powders from Ibiza IV with recycling of 60% of water

OPEX (1000 kg/h sample)	**(Recycling of 60% of water)**		
Chemicals		-	890.34 €
Wastewater (to disposal)	150 €/m^3	-	1,308.12 €
Solid (to disposal)	100 €/ton	-	76.60 €
Energy consumption	Agitators (6x20kW)	-	52.55 €
Precipitation section	Electrowinning (3x3kWh/kg)	-	169.42 €
	Chemicals	-	32.17 €
Total		-	2,529.20 €
Products	**Unit Cost [€/kg]**		
Gold	53,696.49 €		1,718.29 €
Silver	621.00 €		84.58 €
Palladium	63,272.67 €		- €
Copper	3.50 €		562.63 €
Tin Oxide	50.00 €		771.50 €
REVENUES			**607.79 €**

The residual solid was characterized respectively with elemental CHNS analysis and with semi-quantitative analysis by ICP-OES. The residual solid mainly contains plastics, glass fibers, carbon residues and unrecovered metal traces. The wastewaters were characterized with semi-quantitative analysis by ICP-OES. The main results obtained are summarized in the tables below.

Table 47. Elemental CHNS analysis results of the residual solid

Element	**Results (wt%)**
Carbon	17.03
Hydrogen	1.02
Nitrogen	0.56
Sulfur	0.36

Table 48. Semi-quantitative results of residual solid

Metal	**Concentration [g/t]**

Fe	50
Ti	50
Sn	30
Ni	20
Zr	<10
Zn	<5

Table 49. Wastewater characterization

Wastewater 1	acid, sulfuric solution, mainly containing zinc, nickel, aluminum, iron
Wastewater 2	weakly acidic solution with thiourea, urea, cyanamide, sulfur, iron and zinc

Table 50. Semi-quantitative results of wastewaters

	Metal	Concentration [mg/L]
Wastewater 1	Fe	500
	Ni	100
	Sn, Ti, Zn	<20
Wastewater 2	Fe	1000
	Cu	20
	Mn, Ni	<20

4.4 Liquid crystal displays (EuroLCDs)

The proposed process for the treatment of ITO glasses consists of two steps: pre-treatment and leaching. In the first step, the feeding stream is washed with water to remove the surface coating layer. After washing, filtration takes place to separate the wastewater from the solid which is sent to the leaching step. Leaching takes place at 60 °C using very diluted sulfuric acid. A new filter system is inserted downstream of the leaching system to separate the residual solid phase from the leach liquor.

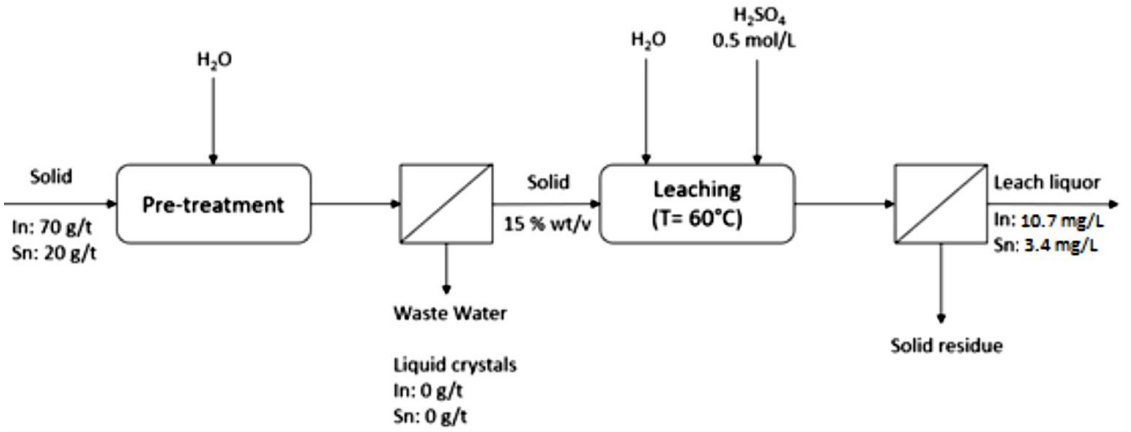


Figure 21. Pre-treatment and leaching process scheme

The precipitation of the tin is carried out by means of polyamine. The precipitation tests have not been carried out as it is possible to have high precipitation yields only by recirculating the leaching solution for numerous steps until reaching high concentrations of the two elements under consideration. The latter will be studied in detail on the pilot plan

4.5 Liquid crystal displays - printed circuit boards (EuroLCDs)

4.5.1 Blue Back Panel

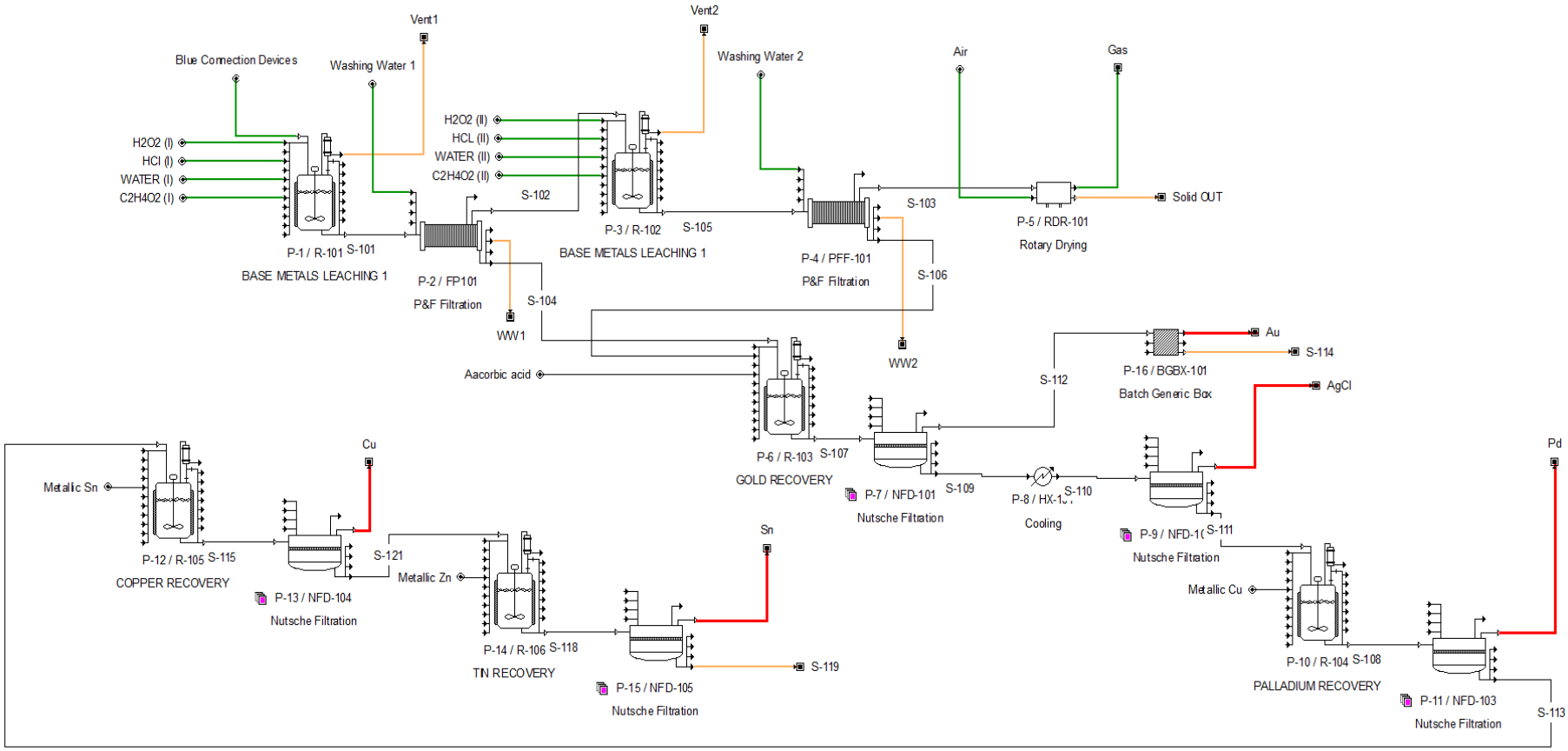


Figure 22. Simulation of Gold-Rec2 process for Blue Back Panel treatment

On the basis of the experimental tests carried out on a laboratory scale (chapter 3.5.1), the material balances were made. The recovery yield of all elements is reported below:

- Gold: 84%
- Silver: 73%
- Palladium: 98%
- Copper: 99.5%
- Tin: 99.5%

Thanks to the material balance it was possible to define the OPEX of the process.

Table 51. Material balance for of Gold-Rec2 process for Blue Back Panel treatment

INPUT		OUTPUT	
Solid [kg]	1000	Solid [kg] (plastic)	696
STEP 1			
Hydrogen peroxide, 30% [L]	1333	Gold [kg]	0.137
Hydrochloric Acid, 37% [L]	1945	Silver Chloride [kg]	0.185
Acetic Acid, 99.9% [L]	667	Palladium [kg]	0.678
Water [L]	2722	Copper [kg]	271
Washing Water [L]	2000	Tin [kg]	506
STEP 2		Wastewater [L]	17572
Hydrogen peroxide, 30% [L]	1333		
Hydrochloric Acid, 37% [L]	1945		
Acetic Acid, 99.9% [L]	667		
Water [L]	2722		
Washing Water [L]	2000		

Table 52. Operating costs for the recycling of a ton of Blue Back Panel without recycling of water

Chemicals	Mass [kg]	Unit Cost [€/kg]	Cost [€]
Hydrogen peroxide, 35%	2537	0.40 €	1,014.86 €
Hydrochloric Acid, 32.5%	5313	0.18 €	956.38 €
Acetic Acid, 80%	1748	0.60 €	1,048.95 €
Water	8951	0.0015 €	13.43 €
Total			2,018.76 €
OPEX (1000 kg/h sample)		**(No Recycling of water)**	
Chemicals			- 2,018.76 €
Wastewater (to disposal)		150 €/m^3	- 2,635.80 €

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Solid (to disposal)	100 €/ton	-	69.60 €
Energy consumption	Cooling Water (Ag precipitation)	-	52.55 €
	Agitators (6x20kW)	-	56.00 €
Precipitation section	Chemicals	-	5,766.55 €
Total		-	10,599.26 €
Products	**Unit Cost [€/kg]**		
Gold	53,696.49 €		7,356.42 €
Silver Chloride	50.45 €		9.33 €
Palladium	63,272.67 €		42,898.87 €
Copper	3.50 €		948.50 €
Tin	28.26 €		14,286.00 €
REVENUES			**54,899.86 €**

The residual solid and the wastewater were characterized. The residual solid mainly contains plastics. The wastewater contains hydrochloric acid with different metals summarizes in the table below.

Table 53. Composition of wastewater (to wastewater treatment) in terms of metals

Component		Concentration
$CuCl_2$	mg/L	842.2
$PdCl_2$	mg/L	1.6
$SnCl_2$	mg/L	1196
$ZnCl_2$	g/L	42.1

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5. Conclusions

Numerous laboratory-scale analyses were performed to characterize the different materials provided by the project partners. Using the results of these analyses, a sustainable recovery process that could guarantee high extraction yields was studied for each material. A technical and economic feasibility analysis was performed for the different processes studied and developed.

The main results obtained for the various components of interest within the Project are reported below: flexible electronics (2 types), car dashboard, printed circuit boards of LCD and ITO glass of LCD.

Flexible electronics (type 1)

- From the characterization of the material, an average silver concentration of 0.31 wt. % was determined, considering the full weight of the sample. The silver present in this type of material was embedded so it is required a pre-treatment to dissolve the plastic and facilitate access to the metal parts.
- The flexible electronics were subjected to two successive leaching steps (Gold-Rec1 modified): DMF treatment to remove the plastic layer and thiourea leaching to extract silver.
- A silver extraction yield of approximately 97% was achieved.
- Il processo proposto è stato studiato considerando un ricircolo di circa il 70% di DMF.

Flexible electronics/IME (type 2)

- From the characterization of the material, an average silver concentration between 0.36-0.63 wt. % was determined, considering the full weight of the sample. The silver present in this type of material was directly accessible (on the surface).
- The samples were subjected to different experimental tests to study the recovering of silver from the polycarbonate-based IME: thiourea leaching and thiourea leaching without oxidant agent. The first (2 steps) allowed a recovery over 90% while the second showed that no significant Ag dissolution occurred (recovery yield < 6%) performing thiourea leaching without the oxidant agent (Fe^{3+}).
- A recovery process using electrowinning has been studied and it has been seen that it is possible to recover about 95% of Ag if one works for 2 hours with a voltage 1.2 V and current density 43 A/m2.
  It has been seen that it is possible to optimize the process through a cross-leaching scheme. Indeed, it is possible to recycle the solution in 3 batches before disposing of as wastewater.

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Car dashboard of SEAT

- From the characterization of the material (for all three car models), the following composition of the device was determined: 76.3% plastic (with traces of glass), 23.56% electronic board and 0.14% aluminum (screws) (the results refer to the Leon II model but are similar for all three models). The electronic board has been characterized in detail in terms of metals present and in particular of Au, Ag, Pd, Cu and Sn; in addition to the metallic fraction is also composed to plastics, fiber glass and epoxy resin.
- The Printed Circuit Boards (PCBs) powders obtained by grinding were subjected to the Gold-Rec1 process. PCBs powders leaching has recorded relatively low recovery yields and is still being optimized today.
- It was seen that using the Gold-Rec1 process on the board powders without electronic components becomes sustainable only if there is recirculation of the water used in the process (at least 60% of the water used).

Printed circuit boards of LCD

- All the components on the board were detached, by heating and manual removal, in order to chemically characterize each type. The composition of each of the components and of the board itself was determined.
- The board has been milled and subjected to the Gold-Rec1 process while the components richer in precious metals (back panels) have been subjected to the Gold-Rec2 process. Also, for this material a low extraction yield of precious metals has been obtained which must be optimized by eliminating other components with a high copper content.
  For back panels the situation is different depending on the material being treated. In this case it could be the plastic material that influences the yields.
- For the powders and back panels from the PCBs of liquid crystal displays, it has been seen that the process is highly cost-effective. Suppose only the connection peripherals are considered. In that case, the revenue obtained is high and would allow the whole board to be treated while continuing to have an economically viable process.

ITO glass of LCD

- From the characterization of the material, the following composition of the device was determined: 60-70 g/t In and 15-25 g/t Sn.
- A series of experimental tests was carried out to understand the behavior of this material in different situations. Washing using acetone and water has used us pre-treatment for the removal of the protective layer covering the metals. When the pre-treatment was used, the leaching step (sulfuric acid leaching) allows recovery about 99% of In and 53% of Sn after 1h. Without the pre-treatment the leaching step allows recovery about 80.6% of In and 65% of Sn after 1h.
- To optimize the process, a factorial plan was created which made it possible to identify the best recovery conditions for In (of greatest interest), with almost total recovery (T=60 °C; H2SO4 concentration= 1 mol/L; pulp density = 15 % wt. /v); under these conditions the recovery of Sn was about 50%.

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- The indium and tin recovery processes are not economically feasible when treating ITO glasses when taken individually. To be feasible, the process of recovering these elements must be integrated into a process of recovering the other components of this material.

However, it should be remembered that all the tests carried out were conducted on materials supplied mainly by manufacturers and not disposers. On the pilot plant, the different processes studied will be evaluated, with all the background knowledge gained on these materials, also for materials from disposers (i.e. POLLINI, ILLSA etc.). Prior to pilot-scale experimentation, appropriate laboratory experimental verifications will be carried out on the materials from the disposers.

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Abbreviations

ICP-OES	Inductively Coupled Plasma – Optical Emission Spectroscopy
XRF	X-ray fluorescence spectroscopy
XRD	X-ray diffraction
PET	Polyethylene terephthalate
TPU	Thermoplastic polyurethane
IME	In-Mold Electronics
PCBs	Printed Circuit Boards
LCD	Liquid Crystal Displays
ITO	Indium-tin oxide
DMF	Dimethylformamide

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Appendix A: Arduino

A characterization study was carried out on a very widespread and standard board at a global level, namely the Arduino board. This study made it possible to define the main elements present in general in an electronic board and to define the source of each in terms of electronic components.

In order to carry out this analysis, we started with a removal of all the components present on the board and their identification based on the function performed. Subsequently, each of the removed elements was subjected to chemical attack (CA) by means of aqua regia or different acids.

The resulting solutions were analyzed by ICP-OES to determine the metal content.

Table 1.A. Results of components characterization

Photo	Name	Weight [g]	(Wt.%)	Elements reported by the qualitative analysis	Composition		Method
	TX-LX-L-Power Led	0.0031	0.006%	Ag, Al, Au, B, Cu, Fe, Ga, Ni, Pd, Sn, Zn	CA [g]	0.0031	Aqua regia digestion
					Cu (%)	24.47	
					Sn (%)	8.08	
					Ni (%)	4.97	
					Al (%)	2.11	
					Zn (%)	0.34	
					Fe (%)	0.19	
					Ag [ppm]	2096.77	
					Au [ppm]	1774.19	
					Pd [ppm]	161.29	
	PolyFuse	0.0281	0.054%	Ag, Al, Au, B, Cu, Ni, Pd, Sn, Zn	CA [g]	0.0282	Aqua regia digestion
					Cu (%)	38.41	
					Sn (%)	9.38	
					Ni (%)	0.67	
					Al (%)	0.49	
					Zn (%)	0.24	
					Ag [ppm]	2801.42	
					Au [ppm]	726.95	
					Pd [ppm]	53.19	
	M7 Diode	0.0646	0.125%	Ag, Au, Cu, Pb, Pd, Sn, Zn	CA [g]	0.0644	Aqua regia digestion
					Cu (%)	33.95	

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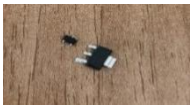
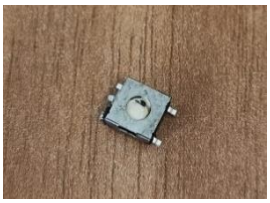
					Sn (%)	4.26	
					Zn (%)	0.21	
					Ag [ppm]	1374.22	
					Pd [ppm]	46.58	
	Voltage Regulator	0.1292	0.250%	Ag, Au, B, Co, Cu, Fe, Pd, Pt, Sn, Zn	CA [g]	0.129	They were cut and then submitted to aqua regia digestion
					Cu (%)	42.80	
					Sn (%)	2.96	
					Zn (%)	0.29	
					Fe (%)	0.02	
					Ag [ppm]	3573.64	
					Pd [ppm]	58.14	
	Reset	0.1965	0.381%	Ag, Al, Au, B, Cu, Fe, Mn, Pd, Pt, Rb, Sn, Zn	CA [g]	0.1972	Aqua regia digestion
					Cu (%)	13.85	
					Zn (%)	7.12	
					Sn (%)	0.87	
					Fe (%)	0.60	
					Al [ppm]	519.78	
					Ag [ppm]	403.14	
					Pd [ppm]	17.75	
	16 MHz Crystal Oscillator	0.5033	0.975%	Ag, Au, Co, Cu, Fe, Mn, Ni, Pd, Rb, Sn, Zn	CA [g]	0.5028	Aqua regia digestion
					Fe (%)	14.21	
					Cu (%)	9.62	
					Zn (%)	5.36	
					Sn (%)	0.05	
					Ni (%)	0.01	
					Ag [ppm]	904.93	
					Pd [ppm]	1.99	
	Capacitors	0.5409	1.048%	Ag, Al, Au, B, Cu, Fe, Pd, Pt, Rb, Sn, Zn	CA [g]	0.5338	They were cut and then submitted to aqua regia digestion
					Fe (%)	59.50	
					Al (%)	37.76	
					Cu (%)	0.41	
					Sn (%)	0.27	
					Zn (%)	0.01	
					Ag [ppm]	59.01	
					Pd [ppm]	0.94	
	ICC	0.6288	1.219%		CA [g]	0.626	

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				Ag, Au, Co, Cu, Fe, Ni, P, Pd, Sn, Zn	Cu (%)	28.75	They were cut and then submitted to aqua regia digestion
					Sn (%)	3.19	
					Fe (%)	1.67	
					Zn (%)	0.17	
					Ag [ppm]	638.57	
					Au [ppm]	190.77	
					Pd [ppm]	33.52	
	ICSP	0.7997	1.550%	Ag, Au, B, Cu, Ni, P, Pd, Sb, Sn, Zn	Plastic [g]	0.190	Aqua regia digestion
					CA [g]	0.607	
					Cu (%)	58.40	
					Zn (%)	28.99	
					Sn (%)	12.41	
					Ni (%)	1.02	
					Ag [ppm]	975.29	
					Au [ppm]	62.60	
					Pd [ppm]	59.31	
	DC Power Jack	1.3288	2.575%	Ag, Au, Cu, Fe, Mn, Ni, P, Pd, Pt, Rb, Sn, Zn	Plastic [g]	0.685	Aqua regia digestion
					CA [g]	0.643	
					Cu (%)	48.57	
					Zn (%)	25.53	
					Fe (%)	19.07	
					Sn (%)	7.03	
					Ni (%)	0.24	
					Ag [ppm]	810.89	
					Pd [ppm]	45.14	
	USB-B Port	3.406	6.601%	Ag, Au, Cu, Ni, Pd, Rb, Sn, Zn	Plastic [g]	1.531	They were broken and then submitted to aqua regia digestion
					CA [g]	1.875	
					Cu (%)	63.11	
					Zn (%)	25.81	
					Sn (%)	4.82	
					Ni (%)	0.07	
					Ag [ppm]	402.93	
					Pd [ppm]	49.87	
					Au [ppm]	4.53	
	External Pin	7.9289	15.366%	Ag, Au, Cu, Ni, Pd, Rb, Sn, Zn	Plastic [g]	5.505	Aqua regia digestion
					CA [g]	2.423	

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					Cu (%)	56.95	
					Zn (%)	25.84	
					Sn (%)	9.56	
					Ni (%)	1.00	
					Ag [ppm]	642.10	
					Au [ppm]	106.88	
					Pd [ppm]	45.19	
	Cover	15.23	29.515%	-	Plastic [g]	15.230	-
					CA [g]	-	
	Board	20.251	39.246%	Ag, B, Ba, Bi, Ca, Cu, Fe, Mg, Mn, Na, Ni, Pd, Si, Sn, Ti, Zn	CA [g]	18.377	It was cut and then submitted to aqua regia digestion
					Cu (%)	15.09	
					Sn (%)	3.36	
					Zn (%)	0.36	
					Ag [ppm]	350.33	
					Ti [ppm]	86.03	
					Pd [ppm]	14.80	
					Au [ppm]	2.39	
	Ceramic Multilayer Capacitors	0.164	0.318%	Ag, Ba, Bi, Ca, Cu, Dy, Fe, Mn, Ni, Pd, Si, Sn, Ti, Y, Zn, Zr	Ti (%)	15.72	They were broken, then were submitted to 1 hour thermal treatment at 350 °C with KOH (two times the amount of solid), then the sample was dissolved in water at 90 °C for 1 h for the detection of Nb, the solid residue was submitted to nitric acid for Ag and Cu digestion and then to aqua regia digestion for Au, Pd and Ti detection.
					Cu (%)	6.93	
					Sn (%)	6.07	
					Ag (%)	1.33	
					Zn (%)	0.40	
					Pd [ppm]	161.59	
					Au [ppm]	103.66	
-	Transistor	0.093	0.180%	Ag, Ca, Cu, Fe, Mn, Ni, Pb, Sn, Zn	Sn (%)	5.46	Aqua regia digestion
					Cu (%)	3.46	
					Zn (%)	0.04	
					Ag [ppm]	2032.26	

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					Au [ppm]	580.65	
-	Others	0.115	0.223%	Ag, Ba, Bi, Ca, Cu, Fe, Mn, Ni, Pb, Pd, Sn, Ti, Zn, Zr	Sn (%)	8.31	Aqua regia digestion
					Cu (%)	3.42	
					Ti (%)	0.48	
					Zn (%)	0.05	
					Ag [ppm]	1813.04	
					Pd [ppm]	300.00	
					Au [ppm]	52.17	

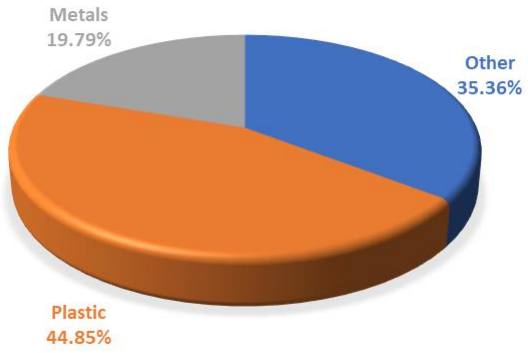

Figure 1.A. Arduino composition

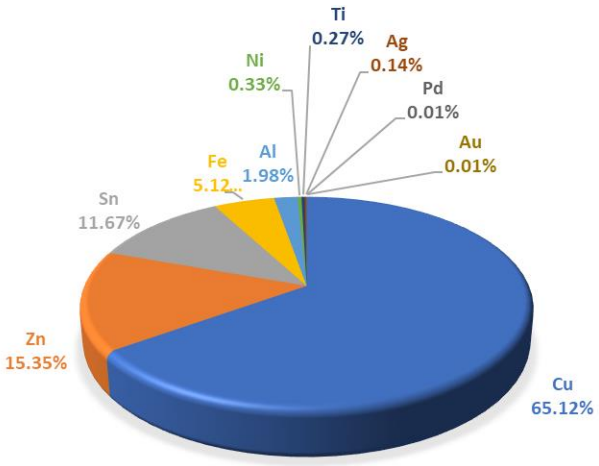

Figure 2.A. Metal fraction composition

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Appendix B: flexible electronics - XRF analysis

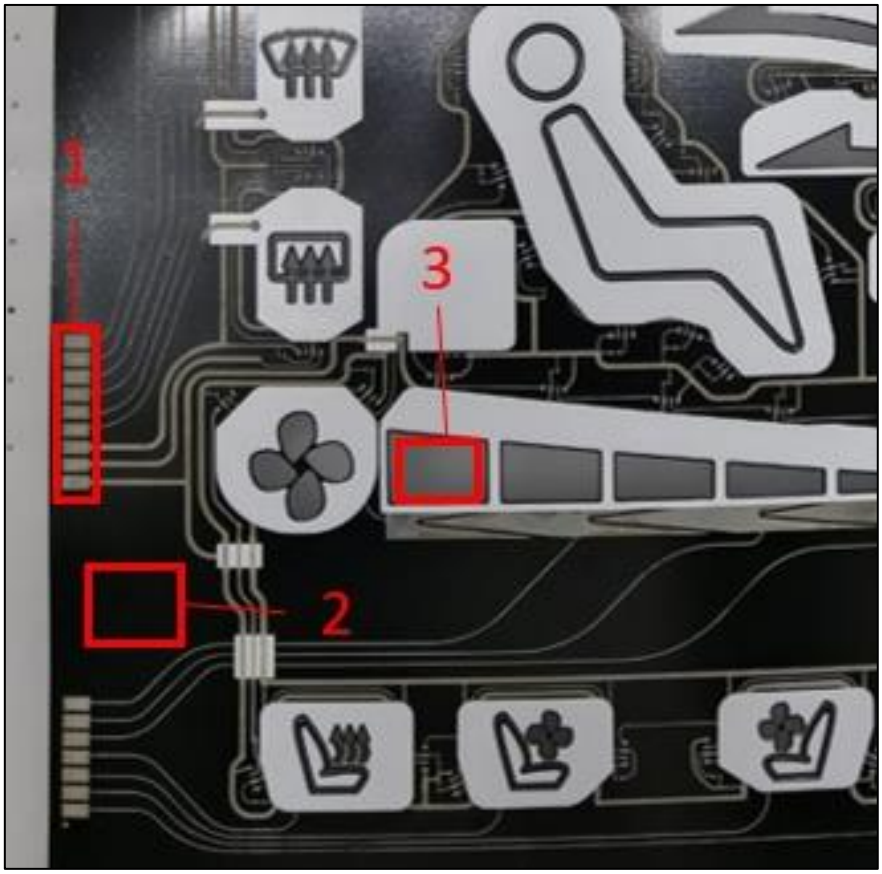

Figure 1.B. Map of performed XRF analyses for the characterization of sample A

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Table 1.B. XRF-analysis on flexible electronic (Sample A, area 1)

Z	Symbol	Element	Norm. Int.	Concentration	Abs. Error
12	Mg	Magnesium	0.0000	< 0.0020 %	(0.0) %
13	Al	Aluminum	15.0925	< 0.0020 %	(0.0) %
14	Si	Silicon	306.3534	0.1584 %	0.0006 %
15	P	Phosphorus	0.0000	< 0.00030 %	(0.0) %
16	S	Sulfur	1024.4259	0.09242 %	0.00016 %
17	Cl	Chlorine	7039.4736	0.3001 %	0.0002 %
19	K	Potassium	1309.8280	0.5606 %	0.0007 %
20	Ca	Calcium	0.0000	< 0.0010 %	(0.0) %
22	Ti	Titanium	30.9095	0.00357 %	0.00005 %
23	V	Vanadium	5.7787	0.00034 %	0.00002 %
24	Cr	Chromium	9.5832	0.00033 %	0.00001 %
25	Mn	Manganese	3871.0944	0.1323 %	0.0002 %
26	Fe	Iron	3820.3295	0.2194 %	0.0003 %
27	Co	Cobalt	0.9651	< 0.00030 %	(0.0) %
28	Ni	Nickel	27.1627	0.00066 %	0.00001 %
29	Cu	Copper	131.2786	0.00261 %	0.00002 %
30	Zn	Zinc	11.9189	0.00016 %	0.00001 %
31	Ga	Gallium	4.4748	0.00005 %	0.00001 %
32	Ge	Germanium	0.0000	< 0.00005 %	(0.0) %
33	As	Arsenic	0.0000	< 0.00005 %	(0.0) %
34	Se	Selenium	2.5214	< 0.00005 %	(0.00001) %
35	Br	Bromine	4.0084	< 0.00005 %	(0.00002) %
37	Rb	Rubidium	2.9093	< 0.00005 %	(0.0) %
38	Sr	Strontium	18.6010	< 0.00005 %	(0.0) %
39	Y	Yttrium	1.8379	< 0.00005 %	(0.0) %
40	Zr	Zirconium	0.0000	< 0.00010 %	(0.0) %
41	Nb	Niobium	0.0000	< 0.00010 %	(0.0) %
42	Mo	Molybdenum	1.0329	0.00002 %	0.00001 %
47	Ag	Silver	4304.2630	0.2938 %	0.0003 %
48	Cd	Cadmium	12.6772	0.00050 %	0.00001 %
50	Sn	Tin	0.0000	< 0.00030 %	(0.0) %
51	Sb	Antimony	0.0000	< 0.00030 %	(0.0) %
52	Te	Tellurium	2.5344	< 0.00030 %	(0.0) %
53	I	Iodine	0.0000	< 0.00030 %	(0.0) %
55	Cs	Cesium	0.0000	< 0.00040 %	(0.0) %
56	Ba	Barium	0.0000	< 0.00020 %	(0.0) %
57	La	Lanthanum	0.0000	< 0.00020 %	(0.0) %
58	Ce	Cerium	0.0000	< 0.00020 %	(0.0) %
59	Pr	Praseodymium	2.1031	0.00034 %	0.00006 %
60	Nd	Neodymium	3.9447	0.00053 %	0.00006 %
72	Hf	Hafnium	4.3778	0.00008 %	0.00001 %
73	Ta	Tantalum	16.0473	< 0.00010 %	(0.0) %
74	W	Tungsten	1.5193	< 0.00010 %	(0.0) %
80	Hg	Mercury	2.6738	< 0.00010 %	(0.00003) %
81	Tl	Thallium	3.3434	< 0.00010 %	(0.00003) %
82	Pb	Lead	3.8560	0.00006 %	0.00001 %
83	Bi	Bismuth	0.0000	< 0.00010 %	(0.0) %
90	Th	Thorium	3.4819	< 0.00010 %	(0.00003) %
92	U	Uranium	2.5352	< 0.00010 %	(0.0) %

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Table 2.B. XRF-analysis on flexible electronic (Sample A, area 2)

Z	Symbol	Element	Norm. Int.	Concentration	Abs. Error
12	Mg	Magnesium	0.0000	< 0.0020 %	(0.0) %
13	Al	Aluminum	73.6823	< 0.0020 %	(0.0) %
14	Si	Silicon	913.0952	0.4782 %	0.0008 %
15	P	Phosphorus	31.0425	0.00623 %	0.00015 %
16	S	Sulfur	3264.5297	0.3101 %	0.0003 %
17	Cl	Chlorine	298.8645	0.01104 %	0.00003 %
19	K	Potassium	44.2475	0.01903 %	0.00021 %
20	Ca	Calcium	49.7790	0.00063 %	0.00001 %
22	Ti	Titanium	125.4881	0.01324 %	0.00009 %
23	V	Vanadium	21.0388	0.00141 %	0.00005 %
24	Cr	Chromium	31.4012	0.00111 %	0.00003 %
25	Mn	Manganese	14116.5111	0.4539 %	0.0003 %
26	Fe	Iron	12076.9126	0.6383 %	0.0005 %
27	Co	Cobalt	2.4330	< 0.00017 %	(0.0) %
28	Ni	Nickel	45.7436	0.00123 %	0.00002 %
29	Cu	Copper	310.8789	0.00672 %	0.00003 %
30	Zn	Zinc	22.7999	0.00034 %	0.00001 %
31	Ga	Gallium	0.3439	< 0.00005 %	(0.0) %
32	Ge	Germanium	0.0000	< 0.00005 %	(0.0) %
33	As	Arsenic	1.9967	0.00002 %	0.00001 %
34	Se	Selenium	2.5407	0.00002 %	0.00001 %
35	Br	Bromine	4.4707	< 0.00005 %	(0.00003) %
37	Rb	Rubidium	5.8103	< 0.00005 %	(0.00002) %
38	Sr	Strontium	39.0812	< 0.00005 %	(0.0) %
39	Y	Yttrium	0.0000	< 0.00005 %	(0.0) %
40	Zr	Zirconium	0.2934	< 0.00010 %	(0.0) %
41	Nb	Niobium	0.3178	0.00016 %	0.00007 %
42	Mo	Molybdenum	0.9827	0.00002 %	0.00001 %
47	Ag	Silver	1.0365	0.00007 %	0.00003 %
48	Cd	Cadmium	2.0584	0.00008 %	0.00001 %
50	Sn	Tin	3.4910	0.00428 %	0.00050 %
51	Sb	Antimony	1.1930	< 0.00030 %	(0.0) %
52	Te	Tellurium	3.0949	< 0.00030 %	(0.0) %
53	I	Iodine	0.0000	< 0.00030 %	(0.0) %
55	Cs	Cesium	0.0000	< 0.00040 %	(0.0) %
56	Ba	Barium	2.3420	< 0.00020 %	(0.0) %
57	La	Lanthanum	2.2980	< 0.00020 %	(0.0) %
58	Ce	Cerium	2.1024	< 0.00020 %	(0.0) %
59	Pr	Praseodymium	8.9735	0.00146 %	0.00012 %
60	Nd	Neodymium	17.4317	0.00241 %	0.00010 %
72	Hf	Hafnium	4.2243	< 0.00010 %	(0.0) %
73	Ta	Tantalum	27.5735	< 0.00010 %	(0.0) %
74	W	Tungsten	2.0377	< 0.00002 %	(0.00001) %
80	Hg	Mercury	3.0540	0.00004 %	0.00001 %
81	Tl	Thallium	4.5374	0.00004 %	0.00001 %
82	Pb	Lead	6.8112	0.00011 %	0.00001 %
83	Bi	Bismuth	0.0000	< 0.00010 %	(0.0) %
90	Th	Thorium	7.1449	0.00007 %	0.00001 %
92	U	Uranium	5.0199	< 0.00010 %	(0.0) %

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Table 3.B. XRF-analysis on flexible electronic (Sample A, area 3)

Z	Symbol	Element	Norm. Int.	Concentration	Abs. Error
12	Mg	Magnesium	0.0000	< 0.0020 %	(0.0) %
13	Al	Aluminum	0.0000	< 0.0020 %	(0.0) %
14	Si	Silicon	0.0000	< 0.00051 %	(0.0) %
15	P	Phosphorus	0.0000	< 0.00030 %	(0.0) %
16	S	Sulfur	0.0000	< 0.00020 %	(0.0) %
17	Cl	Chlorine	270720.9910	19.07 %	0.01 %
19	K	Potassium	5.5596	0.00630 %	0.00024 %
20	Ca	Calcium	12.8424	< 0.0010 %	(0.0) %
22	Ti	Titanium	7.6467	0.00230 %	0.00007 %
23	V	Vanadium	1.3407	< 0.00010 %	(0.0) %
24	Cr	Chromium	3.6507	0.00025 %	0.00002 %
25	Mn	Manganese	1.7863	0.00016 %	0.00007 %
26	Fe	Iron	146.3358	0.01640 %	0.00010 %
27	Co	Cobalt	0.6166	< 0.00030 %	(0.0) %
28	Ni	Nickel	33.4337	0.00210 %	0.00003 %
29	Cu	Copper	13.9648	0.00072 %	0.00003 %
30	Zn	Zinc	25.7379	0.00093 %	0.00002 %
31	Ga	Gallium	3.7489	0.00011 %	0.00001 %
32	Ge	Germanium	0.0000	< 0.00005 %	(0.0) %
33	As	Arsenic	1.2916	0.00002 %	0.00001 %
34	Se	Selenium	1.6112	0.00002 %	0.00001 %
35	Br	Bromine	2.9298	0.00004 %	0.00001 %
37	Rb	Rubidium	1.2511	0.00001 %	0.00001 %
38	Sr	Strontium	1.9037	< 0.00005 %	(0.0) %
39	Y	Yttrium	5.9496	0.00004 %	0.00001 %
40	Zr	Zirconium	0.1604	< 0.00010 %	(0.0) %
41	Nb	Niobium	0.0000	< 0.00010 %	(0.0) %
42	Mo	Molybdenum	0.8312	0.00003 %	0.00001 %
47	Ag	Silver	0.0000	< 0.00020 %	(0.0) %
48	Cd	Cadmium	2.1921	0.00017 %	0.00002 %
50	Sn	Tin	3.3198	0.00828 %	0.00094 %
51	Sb	Antimony	0.0000	< 0.00030 %	(0.0) %
52	Te	Tellurium	3.4218	< 0.00030 %	(0.0) %
53	I	Iodine	0.7388	< 0.00001 %	(0.00001) %
55	Cs	Cesium	0.0000	< 0.00040 %	(0.0) %
56	Ba	Barium	0.0000	< 0.00020 %	(0.0) %
57	La	Lanthanum	0.0000	< 0.00020 %	(0.0) %
58	Ce	Cerium	0.0000	< 0.00020 %	(0.0) %
59	Pr	Praseodymium	0.2303	< 0.00012 %	(0.00012) %
60	Nd	Neodymium	0.0000	< 0.00020 %	(0.0) %
72	Hf	Hafnium	0.5130	< 0.00010 %	(0.0) %
73	Ta	Tantalum	20.1484	< 0.00010 %	(0.0) %
74	W	Tungsten	0.6391	< 0.00010 %	(0.0) %
80	Hg	Mercury	2.1332	0.00006 %	0.00001 %
81	Tl	Thallium	2.2727	0.00005 %	0.00001 %
82	Pb	Lead	2.0252	0.00006 %	0.00001 %
83	Bi	Bismuth	0.0000	< 0.00010 %	(0.0) %
90	Th	Thorium	2.6598	0.00006 %	0.00001 %
92	U	Uranium	1.5751	< 0.00010 %	(0.0) %

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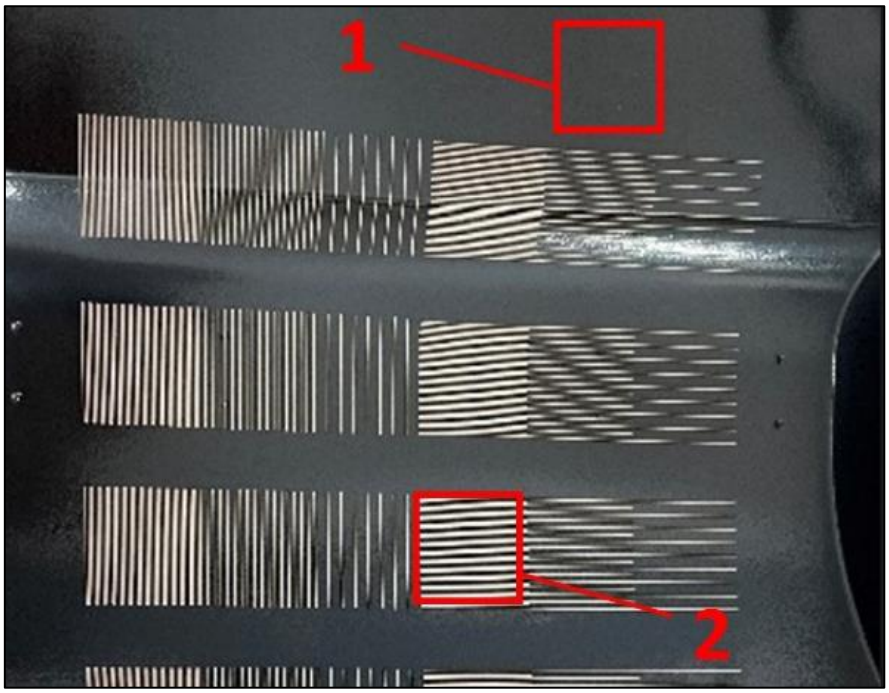

Figure 2.B. Map of performed XRF analyses for the characterization of sample B

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No 101003587

Table 4.B. XRF-analysis on flexible electronic (Sample B, area 1)

Z	Symbol	Element	Norm. Int.	Concentration	Abs. Error
12	Mg	Magnesium	0.0000	< 0.0020 %	(0.0) %
13	Al	Aluminum	91.3854	< 0.0020 %	(0.0) %
14	Si	Silicon	955.3650	0.5219 %	0.0009 %
15	P	Phosphorus	25.3750	0.00533 %	0.00017 %
16	S	Sulfur	3865.2044	0.3858 %	0.0003 %
17	Cl	Chlorine	321.3761	0.01268 %	0.00004 %
19	K	Potassium	64.0969	0.02909 %	0.00029 %
20	Ca	Calcium	291.7725	0.07120 %	0.00032 %
22	Ti	Titanium	270.7637	0.03110 %	0.00014 %
23	V	Vanadium	39.4186	0.00301 %	0.00009 %
24	Cr	Chromium	58.6370	0.00240 %	0.00005 %
25	Mn	Manganese	30443.7852	1.144 %	0.001 %
26	Fe	Iron	28800.9340	1.710 %	0.001 %
27	Co	Cobalt	3.8002	0.00016 %	0.00002 %
28	Ni	Nickel	53.2246	0.00212 %	0.00003 %
29	Cu	Copper	722.0398	0.02312 %	0.00007 %
30	Zn	Zinc	59.0909	0.00132 %	0.00002 %
31	Ga	Gallium	1.3793	0.00003 %	0.00001 %
32	Ge	Germanium	0.0000	< 0.00005 %	(0.0) %
33	As	Arsenic	1.7734	0.00002 %	0.00001 %
34	Se	Selenium	1.1429	0.00001 %	0.00001 %
35	Br	Bromine	5.1738	0.00005 %	0.00001 %
37	Rb	Rubidium	10.4546	0.00006 %	0.00001 %
38	Sr	Strontium	99.0798	0.00030 %	0.00001 %
39	Y	Yttrium	5.8382	0.00003 %	0.00001 %
40	Zr	Zirconium	0.2563	< 0.00010 %	(0.0) %
41	Nb	Niobium	0.0000	< 0.00010 %	(0.0) %
42	Mo	Molybdenum	1.5034	0.00007 %	0.00001 %
47	Ag	Silver	0.0000	< 0.00020 %	(0.0) %
48	Cd	Cadmium	2.0407	0.00011 %	0.00002 %
50	Sn	Tin	2.7209	0.00406 %	0.00057 %
51	Sb	Antimony	0.8035	< 0.00030 %	(0.0) %
52	Te	Tellurium	3.3223	< 0.00030 %	(0.0) %
53	I	Iodine	0.0000	< 0.00030 %	(0.0) %
55	Cs	Cesium	0.0000	< 0.00040 %	(0.0) %
56	Ba	Barium	5.4468	< 0.00020 %	(0.0) %
57	La	Lanthanum	0.0000	< 0.00020 %	(0.0) %
58	Ce	Cerium	2.2132	< 0.00020 %	(0.0) %
59	Pr	Praseodymium	19.1607	0.00343 %	0.00017 %
60	Nd	Neodymium	33.5189	0.00496 %	0.00014 %
72	Hf	Hafnium	2.7361	< 0.00010 %	(0.0) %
73	Ta	Tantalum	23.9550	< 0.00010 %	(0.0) %
74	W	Tungsten	3.6538	0.00007 %	0.00001 %
80	Hg	Mercury	1.8804	0.00003 %	0.00001 %
81	Tl	Thallium	5.2921	0.00007 %	0.00001 %
82	Pb	Lead	10.0493	0.00026 %	0.00001 %
83	Bi	Bismuth	0.0000	< 0.00010 %	(0.0) %
90	Th	Thorium	11.2372	0.00016 %	0.00001 %
92	U	Uranium	3.5524	< 0.00010 %	(0.0) %

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Table 5.B. XRF-analysis on flexible electronic (Sample B, area 2)

Z	Symbol	Element	Norm. Int.	Concentration	Abs. Error
12	Mg	Magnesium	0.0000	< 0.0020 %	(0.0) %
13	Al	Aluminum	56.0221	< 0.0020 %	(0.0) %
14	Si	Silicon	879.9840	0.4786 %	0.0009 %
15	P	Phosphorus	3.5905	0.00075 %	0.00020 %
16	S	Sulfur	6135.8149	0.6122 %	0.0005 %
17	Cl	Chlorine	599.3317	0.02586 %	0.00006 %
19	K	Potassium	336.4141	0.1545 %	0.0005 %
20	Ca	Calcium	81.9894	0.01036 %	0.00010 %
22	Ti	Titanium	225.4893	0.02652 %	0.00013 %
23	V	Vanadium	34.9249	0.00271 %	0.00008 %
24	Cr	Chromium	48.3154	0.00199 %	0.00004 %
25	Mn	Manganese	25404.2805	0.9592 %	0.0006 %
26	Fe	Iron	24484.8954	1.483 %	0.001 %
27	Co	Cobalt	4.1156	0.00032 %	0.00004 %
28	Ni	Nickel	47.2170	0.00178 %	0.00003 %
29	Cu	Copper	611.1902	0.01864 %	0.00006 %
30	Zn	Zinc	53.6945	0.00114 %	0.00001 %
31	Ga	Gallium	4.5156	0.00008 %	0.00001 %
32	Ge	Germanium	1.5509	0.00002 %	0.00001 %
33	As	Arsenic	1.2933	0.00001 %	0.00001 %
34	Se	Selenium	2.9045	0.00003 %	0.00001 %
35	Br	Bromine	8.1709	0.00007 %	0.00001 %
37	Rb	Rubidium	8.6477	0.00004 %	0.00001 %
38	Sr	Strontium	92.4721	0.00024 %	0.00001 %
39	Y	Yttrium	7.1790	0.00003 %	0.00001 %
40	Zr	Zirconium	3.4229	< 0.00010 %	(0.0) %
41	Nb	Niobium	0.2721	< 0.00016 %	(0.00015) %
42	Mo	Molybdenum	1.6669	0.00007 %	0.00001 %
47	Ag	Silver	734.7557	0.06899 %	0.00015 %
48	Cd	Cadmium	2.2654	0.00012 %	0.00002 %
50	Sn	Tin	0.0000	< 0.00030 %	(0.0) %
51	Sb	Antimony	0.5490	< 0.00030 %	(0.0) %
52	Te	Tellurium	4.2044	< 0.00030 %	(0.0) %
53	I	Iodine	1.3899	0.00005 %	0.00002 %
55	Cs	Cesium	0.0000	< 0.00040 %	(0.0) %
56	Ba	Barium	4.2044	< 0.00020 %	(0.0) %
57	La	Lanthanum	0.0000	< 0.00020 %	(0.0) %
58	Ce	Cerium	2.2308	< 0.00020 %	(0.0) %
59	Pr	Praseodymium	14.8152	0.00264 %	0.00016 %
60	Nd	Neodymium	28.7226	0.00434 %	0.00013 %
72	Hf	Hafnium	3.9950	< 0.00010 %	(0.0) %
73	Ta	Tantalum	20.9725	< 0.00010 %	(0.0) %
74	W	Tungsten	3.1675	0.00005 %	0.00001 %
80	Hg	Mercury	1.0741	< 0.00002 %	(0.00001) %
81	Tl	Thallium	3.9676	0.00005 %	0.00001 %
82	Pb	Lead	9.1957	0.00023 %	0.00001 %
83	Bi	Bismuth	0.0000	< 0.00010 %	(0.0) %
90	Th	Thorium	9.9739	0.00013 %	0.00001 %
92	U	Uranium	3.6662	< 0.00010 %	(0.0) %

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