Metrology for the recycling of Technology Critical Elements to support Europe's circular economy

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INTRODUCTION

Technology critical elements (TCEs) are irreplaceable raw materials that are vastly used in consumer products throughout society; including phones, computers, and renewable energy products. Dwindling supplies of TCEs, as well as rapidly changing geopolitical climates, threaten to disrupt technology production worldwide. Therefore, the European Union (EU) strives for a circular economy approach.

PROBLEM

New targets for the recycling of TCE-containing waste are not being met due to:

- Lack of knowlege of TCE stocks and flows in urban mine waste.
- Lack of standardisation of analytical methods and sampling procedures.
- Lack of suitable reference materials to allow comparability of analytical results.

MetroCycleEU

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SOLUTION

The MetroCycleEU project aims to **develop new reference methods** and **materials** to:

- Enable reliability, traceability, and comparability of sampling strategies and analytical results.
- Improve knowledge of TCE stocks in the recycling industry and inform on the recycling process.

Target matrixes: printed circuit boards (PCBs), light emitting diodes (LEDs), and Li-ion batteries.

THE CHALLENGES

SAMPLING

The first major challenge for the analysis of electronic waste is to obtain a representative sample. E-wastes, such as PCBs, are extremely heterogeneous materials. They are also very difficult to break down to small particle sizes. As seen in **Fig. 1**, some large metal particulates (approx. 80 µm diameter) are present in the powdered material. The grinding process and sample mass analysed are important to consider.

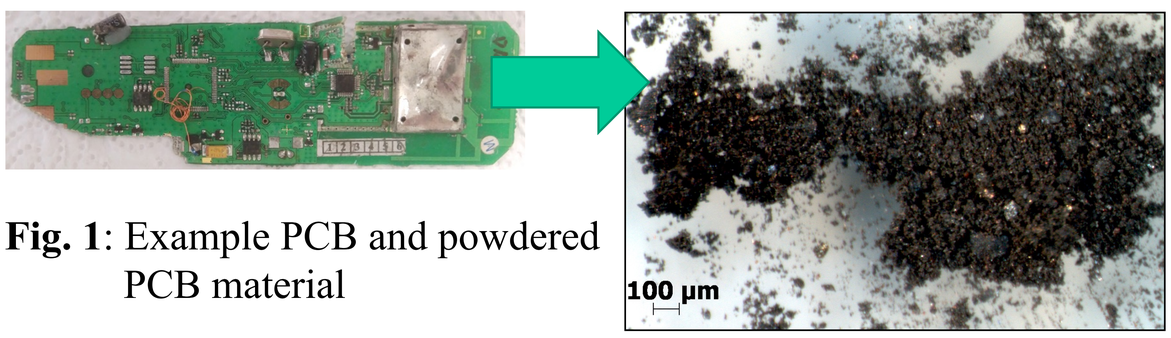

Fig. 1: Example PCB and powdered PCB material

SAMPLE PREPARATION

Samples must be prepared depending on the analysis technique. **Fig. 2** depicts two possible sample preparation methods: acid digestion and pelletizing material. For TCE analysis, acid digestion is typically preferred, however complete digestion of e-waste is very difficult and typically requires harsh, toxic reagents, such as hydrofluoric acid. As such, the development of improved digestion methods are a key focus.

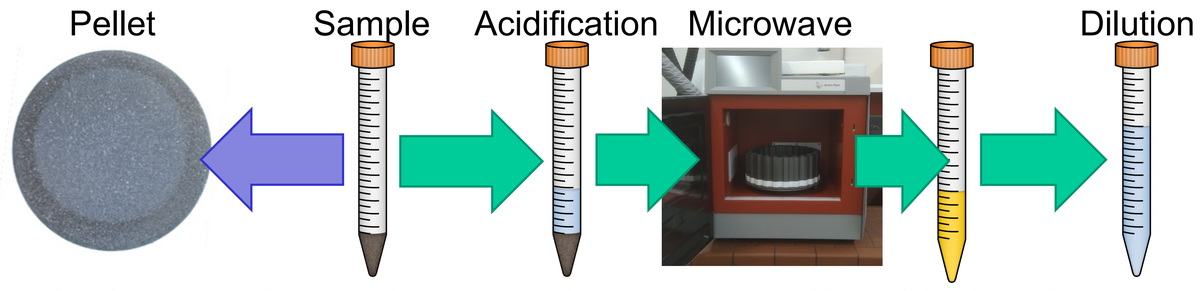

Fig. 2: Example sample preparation: pressed pellets (blue) and acid digestion (green)

ANALYSIS

1) Inductively coupled plasma tandem mass spectrometry (ICP-MS/MS) is a widely used tool for routine analysis that is able to resolve interferences from other elements and provide reliable results. However, some interferences remain challenging. Additionally, samples first require digestion, which means that samples currently take a long time to be processed.

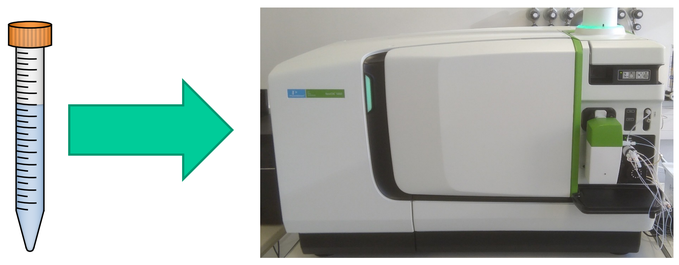

Fig. 3: PerkinElmer NexION 5000 ICP-MS/MS

2) X-ray fluorescence spectrometry (XRF) requires minimal sample preparation and can provide rapid analysis of materials. However, low sensitivity makes detection of low quantities of TCEs challenging. Additionally, there is currently a lack of sufficient XRF calibration standards for e-waste materials.

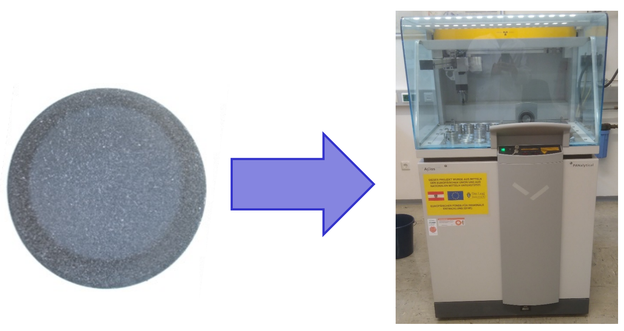

Fig. 4: PANalytical WD-XRF

3) Laser ablation (LA)-ICP-MS is an established technique for the direct analysis of solid materials. This approach has not yet been applied to the analysis of TCEs in e-waste and it is a simpler approach considered as part of this project. No e-waste calibration materials for LA-ICP-MS currently exist. Additionally, sample heterogeneity is a also great challenge.

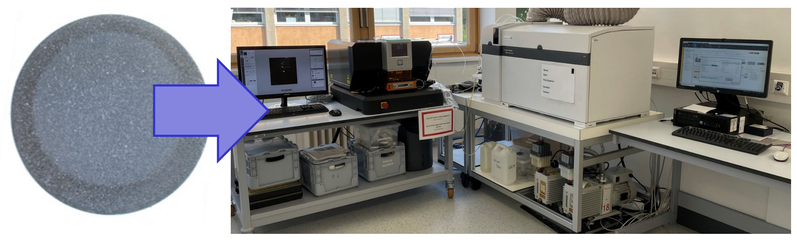

Fig. 5: NWR Laser with Agilent 8800 ICP-MS

OUTLOOK

Given the high demand for TCEs, and a greater focus on recycling, it is likely that the economic value of TCE-containing waste will rise.
The improved TCE analysis for electronic waste matrices through the MetroCycleEU project will be a great asset to industry.
Further outcomes of the project include:

- **Provision of "best practice" guides** that allow more laboratories to perform high-quality TCE measurements at lower costs.
- **Support through training materials** to help industrial partners implement these new analytical methods.

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