Publishable Summary for 20IND01 MetroCycleEU
Metrology for the recycling of Technology Critical Elements to support Europe's circular economy agenda

Overview

Technology critical elements (TCE) are key materials for high level technology but their supply to the EU is challenging. A sustainable solution to this problem is through recovery and recycling, but the analysis of waste streams is difficult and a complicating factor. The overall objective of this project is to enable the representative SI traceable determination of TCE at the µg/g level in urban mine wastes. Transferable calibration methods and appropriate reference material(s) will be developed to improve TCE analysis for recycling in the context of the circular economy. Targeted industries in the recycling chain include multinational companies, SMEs and R&D institutes.

Need

TCE are a group of naturally occurring chemical elements increasingly used in high technology, including energy-efficient lighting, electric car batteries, sensors, electronic devices, mobile phones and computers, due to their unique characteristics (conductivity, magnetism, material properties). These elements are considered 'critical' because of their economic importance in essential technologies and their supply risk (stock depletion and/or few producers in, for example, China and Africa). In addition, relying on raw materials as the prime source for TCE has an environmental cost due to the industrial mining required to supply the scarce raw materials. As a result of the shift towards a greener economy through the deployment of renewable energy and e-mobility solutions (European Green Deal), the use of TCE is expected to increase exponentially in Europe. The value of raw materials in electronic waste generated in Europe in 2019 alone was estimated to be approximately 12.9 billon USD. The need to secure TCE supply has become even more pressing under the current health crisis and it is a major objective of the EU's Covid-19 Recovery Plan aimed at reinforcing Europe's resilience and autonomy.

In order to secure the TCE supply, the EU has promoted more efficient recycling and waste collection through the amended Waste Framework Directive (2018/851/EU). At the same time, the Circular Economy Action Plan has set a target of 50 % reduction in municipal waste disposal by 2030 and there are plans for various European legislation (on batteries, packaging, end-of-life vehicle and electronic equipment....) to be reviewed to stimulate better management of TCE-containing wastes, to increase the recycled content, and ensure high-quality recycling. In 2017, the EU established a list of the critical elements for Europe, which was updated in September 2020. The TCE included in the MetroCycleEU project (Cobalt (Co), Gallium (Ga), Germanium (Ge), Indium (In), Tantalum (Ta), Neodymium (Nd), Praseodymium (Pr), Dysprosium (Dy), Gadolinium (Gd), Lanthanum (La), Gold (Au), Platinum (Pt), Palladium (Pd), Rhodium (Rh) and Lithium (Li)) were selected from this list.

Wastes from the urban mine are extremely heterogeneous, which makes the estimation of their TCE content difficult. Currently there is a lack of knowledge at the European level about the TCE stocks and flows in the urban mine. Given the high volume of waste generated and received, fast reliable preparation and analytical methods as well as sampling and sample preparation strategies are needed to determine the economic value of the waste and of the final product, to decide on recycling route and to engage in new R&D for recycling. Prior to this project a survey was circulated by NMIs to identify the metrological needs of industries involved in urban mine waste recycling. This survey highlighted the lack of reference materials certified for TCE in these wastes and the need for specific validated methods and documentary standards for TCE to comply with ISO/IEC 17025 requirements.

Objectives

The overall objective of the project is to provide reliable and SI traceable determination of TCE in urban waste material at µg/g levels in order to increase the efficiency and accuracy of TCE recycling.

The specific objectives of the project are:

1. To develop validated SI-traceable reference methods (e.g. ICP-MS, INAA) to determine mass fractions at µg/g levels in urban waste materials with a target combined uncertainty below 10 % for the TCEs Co, Ga, Ge, In, Ta, Nd, Pr, Dy, Gd, La, Au, Pt, Pd, Rh and Li. In addition, sampling and digestion strategies for the reference methods will be optimised specifically for TCE in urban waste matrices.
2. To develop traceable and validated reference materials for the TCE in Objective 1 and to use them to validate TCE mass fraction measurements in urban mine waste with a target combined uncertainty below 20 %.
3. To validate the use of the methods and reference materials from objective 1 and 2 using inter-laboratory comparisons involving at least 10 industrial laboratories using both destructive methods (e.g. ICP-MS, ICP-OES) and non-destructive or quasi non-destructive analysis (e.g. WD-XRF, GD-OES, LIBS).
4. To facilitate the take up of the technology and measurement infrastructure developed in the project by the measurement supply chain, standards developing organisations (i.e. those associated with the Waste Framework Directive (2018/851/EU) and ISO/IEC 17025 and end users (recycling industry, analytical laboratories).

Progress beyond the state of the art and results

Reference TCE analysis methods, sampling and sample preparation for the selected TCEs in urban mine waste

Urban mine waste includes highly complex and heterogeneous materials which require suitable processing, digestion and sampling prior to analysis. However, there is a lack of information about the most suitable combination of reference methods for analysis of TCE in waste material and the most appropriate processing and digestion techniques, and due to the large heterogeneity of the matrix compositions of the urban mine waste, no harmonised sampling or sample preparation protocols exist. The project will improve the representativeness of sampling from large amounts of waste by applying the statistical Incremental Sampling methods specifically for TCEs analysis. In addition, sample preparation and reference TCE analysis methods will be optimised for three urban mine wastes (PCB, LED and Li-Ion batteries), with the aim of achieving 10 % expanded uncertainty for analysis at µg/g levels. Several sample preparation strategies (alkali fusion, high pressure acid digestion, plasma ashing) and interference correction instrumentation (HR-ICP-MS, ICP-MS/MS, ICP-CC-MS, INAA) will be optimised and compared.

Urban mine waste reference material for the determination of mass fractions of TCEs

Currently no suitable reference material exists for TCEs in urban mine wastes thus preventing method validation in compliance with quality standards (ISO/IEC 17025) and adequate calibration/optimisation of instruments. Three candidate materials will be evaluated and at least one will be certified for TCEs with an expanded uncertainty below 20 %. Homogeneity and stability of the material will be quantified according to ISO 17034. The value assignment of the produced reference material will be set through an interlaboratory comparison with measurements performed using the reference methods (ICP-MS and INAA) developed in the project.

Improvement of industrial routine analysis

The reference methods developed in the project will be relevant for the certification of reference materials but require specific capabilities that are too expensive and time-consuming for routine use. This project will improve the calibration and use of XRF instruments specifically for TCE analysis in urban waste materials. The performance of several different XRF instruments (portable XRF, WD-XRF) will be tested and compared. Furthermore, methods will be developed to enable the use of the materials characterised within the project as calibrants or quality controls to improve accuracy. An interlaboratory comparison involving laboratories beyond the consortium will be organised to validate the use of these methods and the reference material developed within the project.

Impact

Project outputs will include new analytical capabilities for TCE measurements and waste sample preparation, methods for more representative sampling, one certified matrix reference material (RM) and improved RM production processes, a technical guide for best practice use of XRF instrumentation for TCE analysis in urban waste samples, as well as provision of TCE data on three different waste types and their (in)homogeneity.

Impact on industrial and other user communities

The industrial end-users who will benefit from the project are: 1) industries that recycle and analyse TCE, 2) industries and organisations that collect and trade wastes (which are usually different from recycling

companies), 3) accredited analytical laboratories (recycling industry sub-contractors) and laboratories from R&D public institutes involved in waste recycling, 4) TCE analysis instruments manufacturers (e.g. XRF) and material providers. These end-users will be able to improve and validate their measurement methods for TCE analysis in their premises, improve the quality and accuracy of their data, calculate the uncertainties and improve their sampling strategies through the use of the reference material, the interlaboratory comparison (ILC), technical guides, videos and calculation tools available. As a result these end users will gain robust knowledge of the commercial values of their wastes to decide on a recycling route In the longer term, regulatory bodies and policy makers will benefit from the increased quality and quantity of data to take actions to promote TCE waste recycling. EMPIR

The methods and output generated in this project will be disseminated to the stakeholders through European databases, through the future European Raw Materials Alliance and through European and national industry networks e.g. EIT RawMaterial and Ecodom. A guide for representative sampling in the urban mine associated to a calculation tool will be submitted to EURAMET and will be available to end users. A workshop on sample preparation will be organised and training will be available through e-learning and online seminars for the end-users. The project's outputs will be presented at conferences and in open access scientific journals. The participation in the project of an industrial partner and the Stakeholder Committee will help align the project with industrial needs.

Impact on the metrology and scientific communities

At the primary level, the project will allow the NMIs/DIs to develop new measurement capabilities for the determination of TCE. These capabilities will be important in the future certification process of other matrix reference materials containing these TCE, produced by EC-JRC or AIEA for example. This will also allow NMIs/DIs to provide TCE analysis or to engage in further development for TCE tailored to the specific needs of the end-users. The metrological infrastructure will help the organisers of proficiency testing and large-scale reference material producers to use matrix matching reference materials in order to guarantee the quality of the TCE measurements under ISO/IEC 17025 accreditation scheme.

Impact on relevant standards

The project will support the implementation of the Circular Economy Action Plan from the European Green Deal, through the development of methods for TCE analysis in recyclable wastes from the urban mine. In addition, the consortium will promote the results of the project within the standardisation community and will provide input to the standardisation process (ISO, CEN), and in particular CEN TC444/WG3 "Environmental characterisation of solid matrices-inorganic chemistry". The reference measurement procedures developed will also be disseminated to NMIs and relevant DIs through participation in meetings and workshops organised on behalf of CCQM.

Longer-term economic, social and environmental impacts

With the expected increase in TCE recycling and demand, the economic value of the wastes should rise and the need for more high-quality data will increase. In 2019, in Europe, the amount of urban mine waste generated from electrical and electronic equipment (WEEE) was estimated to be around 12 million tonnes and the value of raw materials in this electronic waste approached 12.9 billon USD. The loss of TCE to waste is therefore also an economic loss and makes recycling a more pressing matter, including to resist a raw material crisis and international market fluctuations. This project will support the implementation of various Directives such as the WEEE Directive (2012/19/EU), Waste Framework Directive (2018/851/EU, Batteries and accumulators Directive (2006/66/EC), End-of-Life Vehicle Directive (2000/53/EC) and the Eco-design Directive (2009/125/EC). By making available best practice guides for routine analysis, more laboratories will be able to implement TCE measurements satisfying quality standards at lower cost.

Securing the TCE supply also affects the life of European citizens, as TCE are essential to provide the necessary digital products and services (e.g. smartphones, sensors…) in addition to critical industries such as the medical sector, the transportation sector, industry, power plants… and even to sectors that were traditionally not "connected" such as the agricultural sector.

More and more in Europe, reusing and improving the durability of equipment is demanded by citizens. Improving knowledge of TCE in waste can help better waste management, and even avoid waste production by promoting reuse of valuable pieces. Urban waste materials not only contain TCE, they also include hazardous substances (e.g. heavy metals such as mercury, lead, plastics containing Brominated Flame Retardants…), and similarly industrial mining of primary raw materials produces waste. Both sources include environmentally damaging materials which often contaminate and degrade the landscape. Limiting and decreasing waste and reducing the use of primary source raw material is one step towards a better

environment. Improving and increasing the spread of TCE analysis by fast reliable traceable methods will enable environmental agencies to set up national-scale monitoring programmes to determine the state of contamination by TCE, their sources (e.g. industrial discharge, WEEE disposal) and their fate in the environment.

List of publications

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<table>
<tr><td colspan="2">Project start date and duration:</td><td>01 June 2021, 36 months</td></tr>
<tr><td colspan="3">Coordinator: Johanna Noireaux, LNE Tel: 00 33 1 4043 3901 E-mail:johanna.noireaux@lne.fr
Project website address:</td></tr>
<tr><td>Internal Funded Partners:
1. LNE, France
2. BAM, Germany
3. IMBiH, Bosnia and Herzegovina
4. INRIM, Italy
5. LGC, United Kingdom
6. PTB, Germany
7. RISE, Sweden
8. SYKE, Finland
9. TUBITAK, Turkey</td><td>External Funded Partners:
10. BRGM, France
11. EID, France
12. Hereon, Germany
13. MUL, Austria</td><td>Unfunded Partners:
14. JSI, Slovenia
15. METAS, Switzerland</td></tr>
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