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Original

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Improving valuable metals recycling : best digestion method for retrieving Technology Critical Elements

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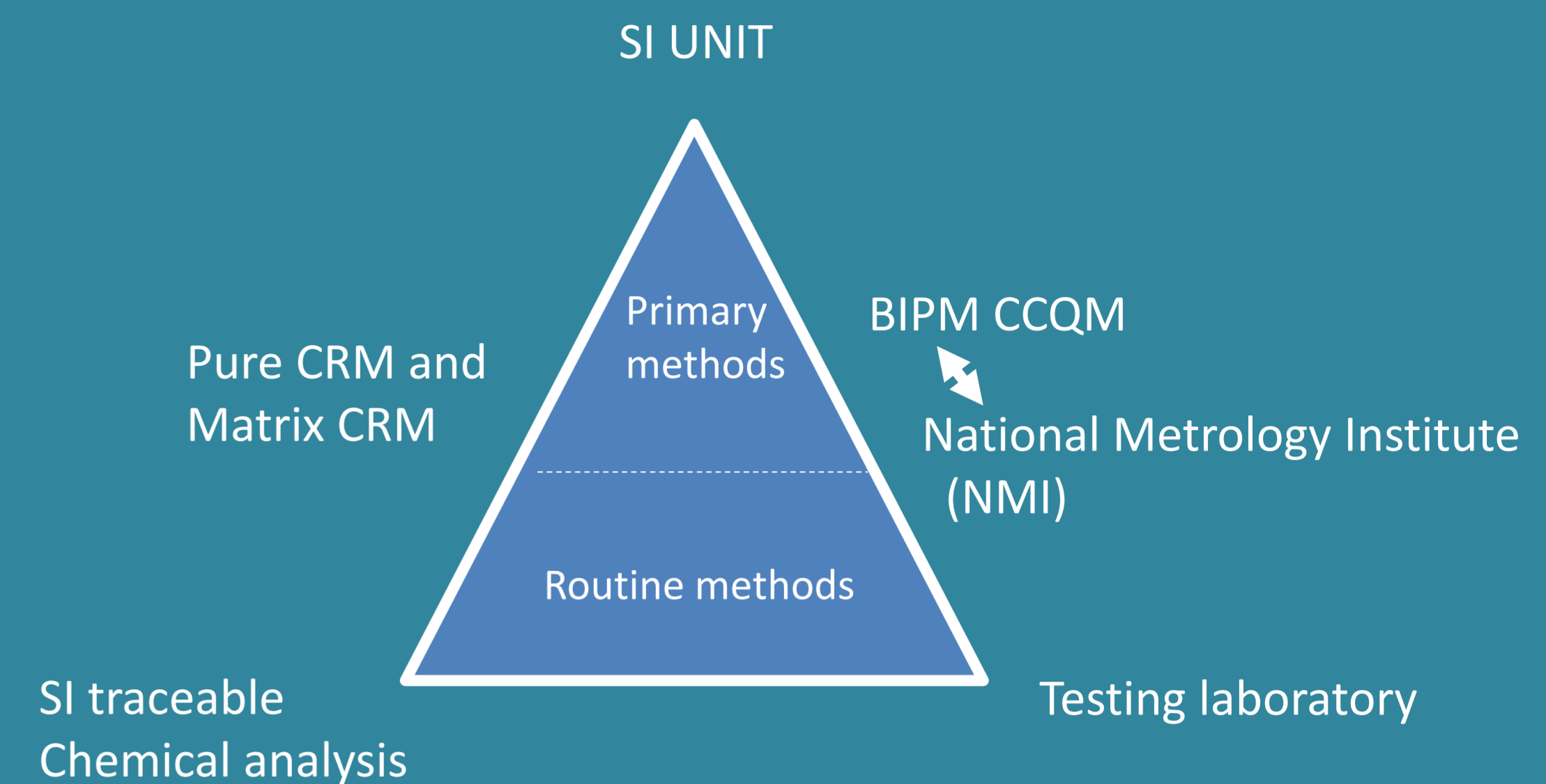


INTRODUCTION

- **Technology Critical Elements (TCE)** are at supply risk in the EU and are of high economic importance for the EU shift towards renewable energy and Green Deal strategy
- Measuring the TCE potential of **electrical wastes** is a first step in the development of TCE recycling and knowledge of its fluxes and loss to the environment
- One main objective of **MetroCycleEU** project is to develop a certified reference material (CRM) for TCE in electrical wastes
- As a first step, the sample digestion is being optimised for ICP-MS analysis with the help of INAA on a crushed Printed Circuit Board (PCB) sample

METROLOGICAL TRACEABILITY FOR CHEMICAL ANALYSIS

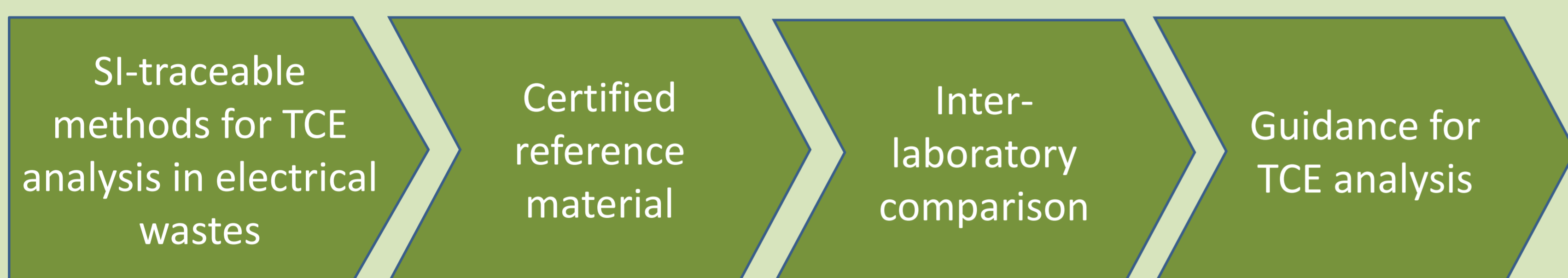
CRM is a reference material (RM) characterized by a metrologically valid procedure [...], accompanied by a RM certificate that provides the value of the specified property, its associated uncertainty, and a statement of metrological traceability



METROCYCLE EU PROJECT



metrocycle.eu



Co, Ga, Ge, In, Ta, Nd, Pr, Dy, Gd, La, Au, Pt, Pd, Rh, Li)

3 candidate CRM PCB, LED, LiB

ICPMS, XRF

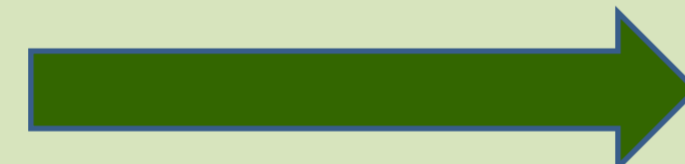
Guidance for TCE analysis

PRINTED CIRCUIT BOARD (PCB)



400 kg of PCB

4 Successive steps of shredding and milling

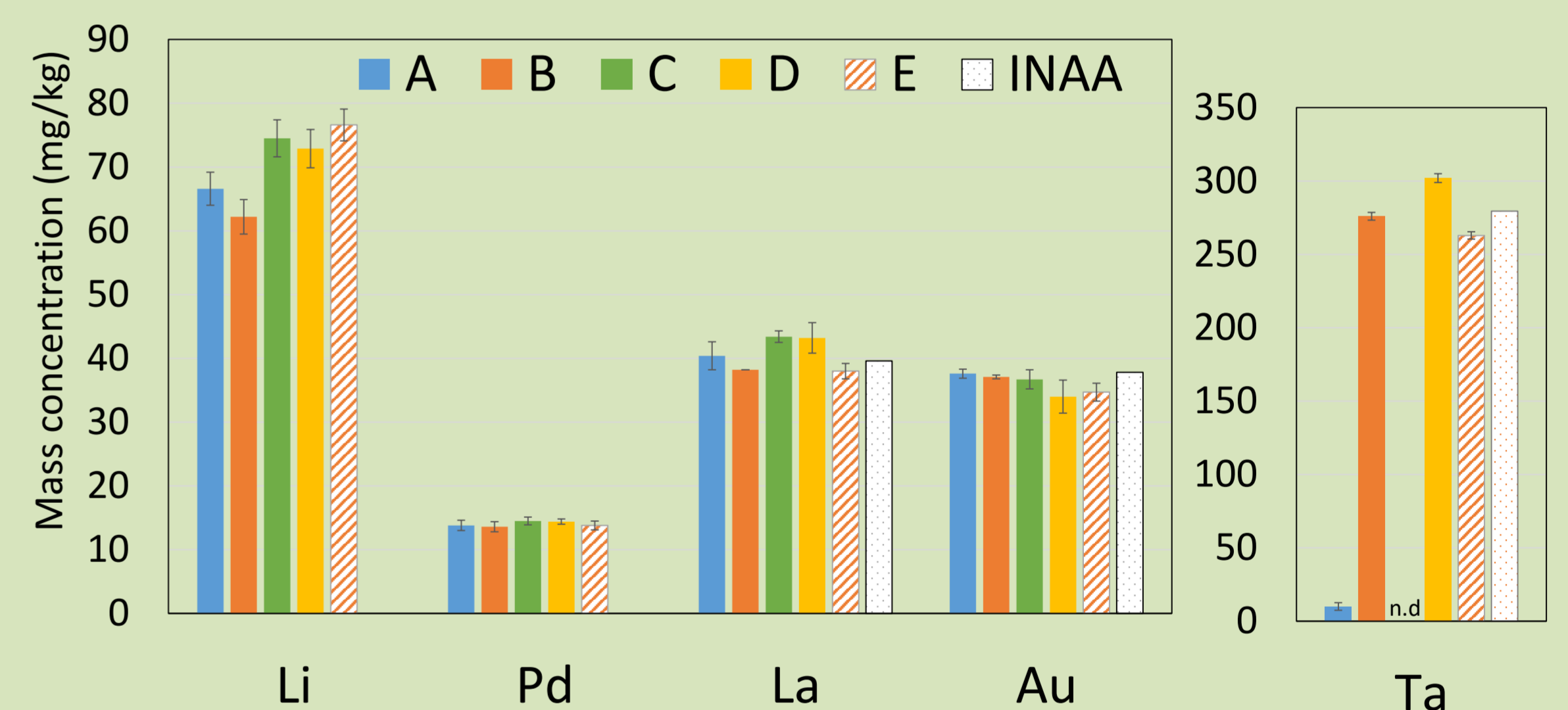


Sample ready for chemical analysis

<200µm

DIGESTION METHODS FOR ICP-MS

	Calcination 600°C	Acids/flux	Method
A	No	HNO ₃ +HCl+H ₂ O ₂	microwave 240°C
B	No	HF+H ₂ O ₂ +HNO ₃ +HCl and H ₃ BO ₃	microwave 240°C
C	Yes	HNO ₃ +HCl+H ₂ O ₂	microwave 240°C
D	Yes	HF+H ₂ O ₂ +HNO ₃ +HCl and H ₃ BO ₃	microwave 240°C
E	Yes	Na ₂ O ₂	Alkali fusion



INAA INSTRUMENTAL NEUTRON ACTIVATION ANALYSIS

Element	Au	La	Co	Ta
γ-lines / keV	411.8	1596.2	1173.2, 1332.5	1121.3, 1189.1, 1221.4, 1231.0
spectrum (counting distance)				
#1 (200 mm)	✓	✓		
#2 (100 mm)	✓	✓		
#3 (60 mm)			✓	✓
#4 (20 mm)			✓	✓



Pressed pellets



Stacked sample/Standards



Spectra acquired at different source-detector distances (200, 100, 60, 20 mm from end-cap)

ICP-MS ANALYSIS



Element XR (ThermoFisher)

Element	Isotope measured	Resolution
Li	6, 7	MR
Pd	104, 105	HR
La	139	HR
Au	197	HR
Ta	181	HR

WHAT'S NEXT?

- Compare the digestion for all 14 TCE elements
- Same study with LED and LiB
- Assignment of reference value to the RM, including homogeneity determination and uncertainty calculation
- Use of the RM for LA-ICPMS calibration
- Use of the RM for XRF calibration