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

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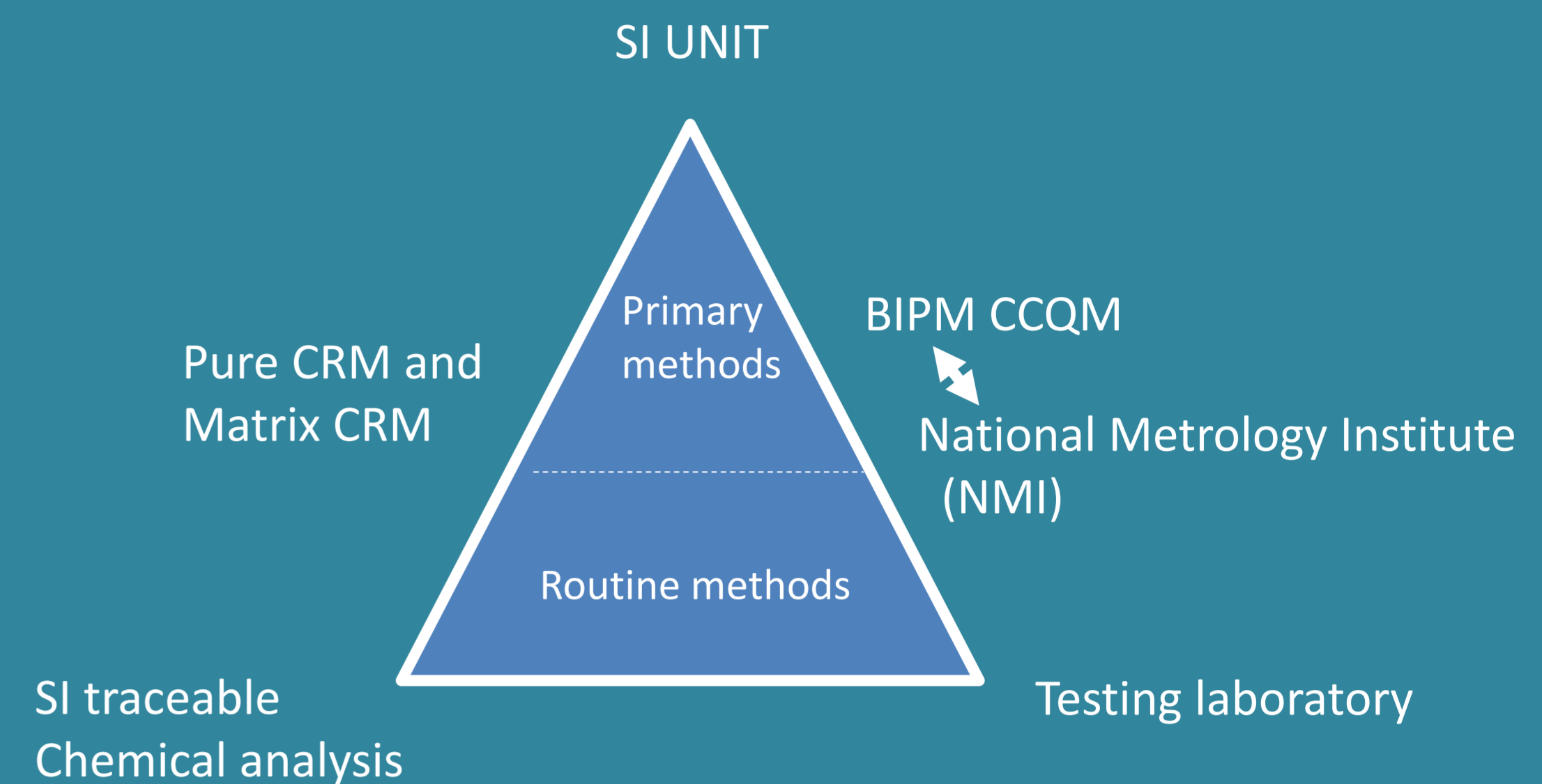
INTRODUCTION

- **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
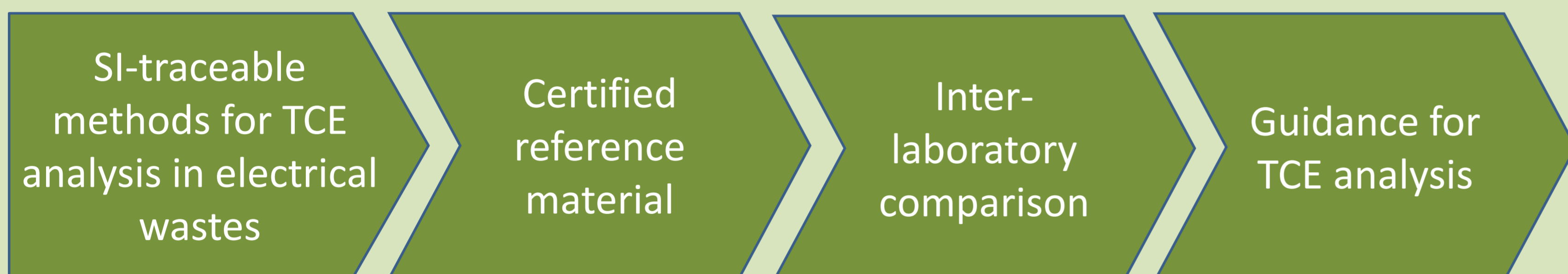
METROLOGICAL TRACEABILITY FOR CHEMICAL ANALYSIS

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

SI-traceable methods for TCE analysis in electrical wastes → Certified reference material → Inter-laboratory comparison → Guidance for TCE analysis

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

3 candidate CRM PCB, LED, LiB
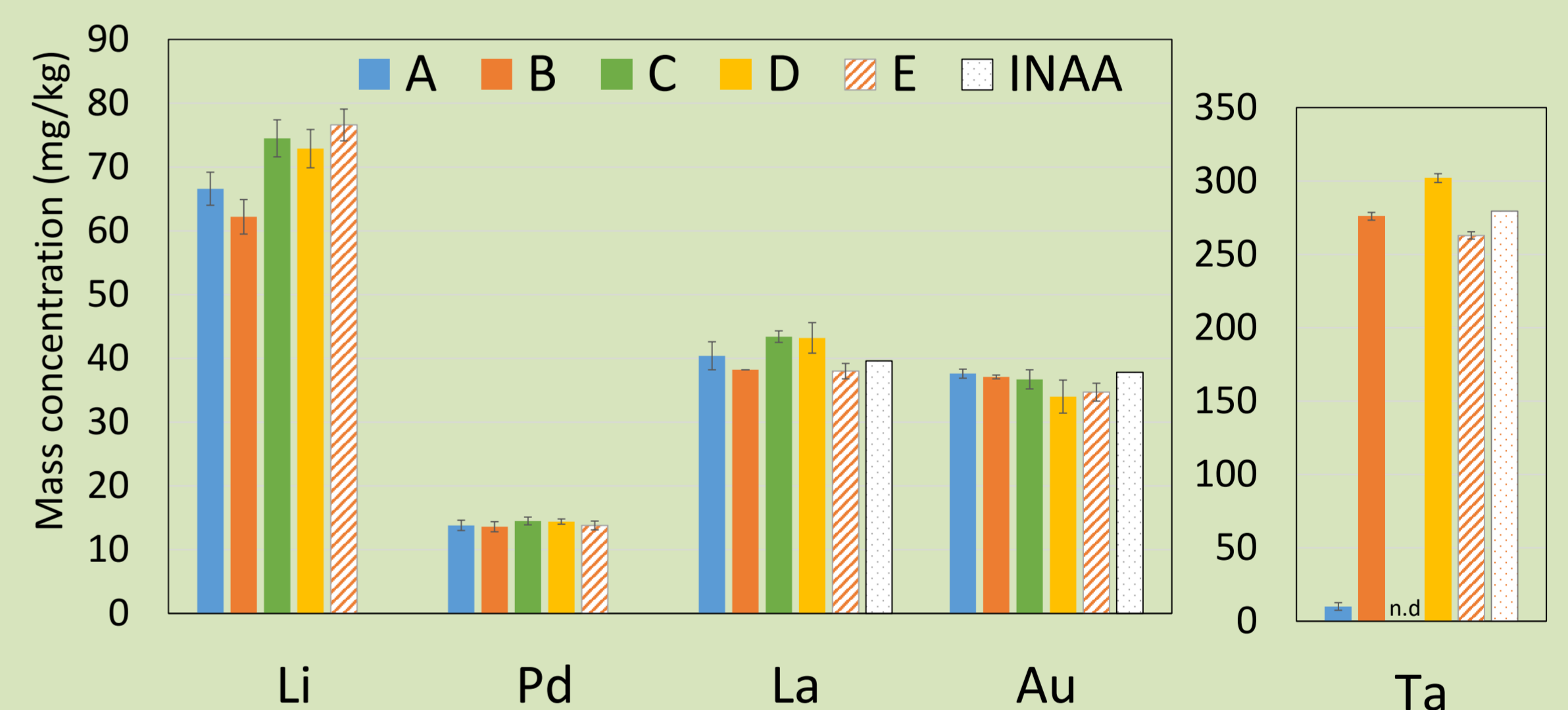
ICPMS, XRF

PRINTED CIRCUIT BOARD (PCB)

4 Successive steps of shredding and milling

Sample ready for chemical analysis

400 kg of PCB

<200µm

DIGESTION METHODS FOR ICP-MS

	Calcination 600°C	Acids/flux	Method
A	No	HNO3+HCl+H2O2	microwave 240°C
B	No	HF+H2O2+HNO3+HCl and H3BO3	microwave 240°C
C	Yes	HNO3+HCl+H2O2	microwave 240°C
D	Yes	HF+H2O2+HNO3+HCl and H3BO3	microwave 240°C
E	Yes	Na2O2	Alkali fusion

- For Pd, La and Au, all digestions are equivalent within uncertainty
- For Li, the calcination step is required
- For Ta, the digestion requires HF in the acid mixture
- La, Au and Ta concentrations obtained with INAA are in agreement with the data from ICP-MS

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
#1 (200 mm)	✓	✓		
#2 (100 mm)	✓	✓		
#3 (60 mm)			✓	✓
#4 (20 mm)			✓	✓

spectrum (counting distance)

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
- Assigment of reference value to the RM, including homogeneity determination and uncertainty calucation
- Use of the RM for LA-ICPMS calibration
- Use of the RM for XRF calibration