



Challenge

Ensure food safety compliance by detecting trace heavy metals (HMs) and rare earth elements (REEs) in recycled PET (rPET) used in food contact material.

Solution

A validated ICP-MS method enables accurate, sensitive, and repeatable quantification of 26 HMs and 4 REEs in PET and rPET matrices.

Intended audience

Food packaging manufacturers, recycling companies, regulatory bodies, and analytical laboratories.

ICP-MS analysis of trace metals and rare earth elements in PET and rPET materials

Introduction

The increasing demand for sustainable packaging solutions has driven the widespread adoption of recycled polyethylene terephthalate (rPET) in food contact materials (FCMs). While rPET supports circular economy goals and reduces environmental impact, its safety remains a critical concern due to the potential presence of non-intentionally added substances (NIASs), including trace heavy metals (HMs) and rare earth elements (REEs). These substances may originate from previous uses, degradation of additives, or contamination during the recycling process, posing risks to human health if they migrate into food or beverages. To safeguard consumer health, the European Union has established a robust regulatory framework for materials intended for food contact. Regulation (EC) No 1935/2004 sets out general safety principles, requiring that materials must not transfer constituents to food in quantities that could endanger human health or alter food quality. More specifically, Regulation (EU) No 10/2011 provides

detailed rules for plastic materials, including a Union list of authorized substances and specific migration limits. This regulation was further amended by Commission Regulation (EU) 2020/1245, which introduced stricter provisions for substances such as lanthanides used in plastic additives. According to this amendment, the migration of the sum of lanthanum (La), europium (Eu), gadolinium (Gd), and terbium (Tb) in ionic form must not exceed 0.05 mg/kg of food ^[1].

Despite these regulatory advances, analytical challenges persist. The detection and quantification of trace elements in PET and rPET require highly sensitive and validated methodologies capable of addressing complex matrices and ultra-trace concentrations. Traditional methods often fall short in terms of sensitivity, reproducibility, or scope. This application note presents a validated analytical method using inductively coupled plasma mass spectrometry

(ICP-MS) with the PlasmaQuant MS (Analytik Jena) for the simultaneous determination of 26 HMs and 4 REEs in PET and rPET matrices. The method employs optimized acid digestion and advanced mass spectrometric detection to ensure high sensitivity, accuracy, and repeatability. It is designed to support quality control in recycling workflows,

identify contamination hotspots, and ensure compliance with evolving food safety regulations. By enabling precise monitoring of elemental impurities, this approach contributes to safer rPET applications and reinforces the integrity of the circular economy.

Materials and methods

Instrument settings

Sample digestion was performed using a closed-vessel microwave-assisted digestion system (speedwave XPERT) with DAK-100X digestion vessels, ensuring complete decomposition of PET and rPET matrices under controlled temperature and pressure conditions. This method minimizes contamination and volatilization losses, particularly for volatile or trace elements.

Elemental analysis was conducted using the PlasmaQuant MS, equipped with integrated collision/reaction cell (iCRC) technology to effectively eliminate polyatomic interferences – especially those affecting first-row transition metals – by using helium and hydrogen as collision/reaction gases, respectively. This setup ensures high accuracy and low detection limits, even in complex PET and rPET matrices.

The ICP-MS system was coupled with a CETAC ASX-560 autosampler and a CETAC ASXpress plus injection valve, enabling automated high-throughput sample introduction

with efficient washout behavior. A standard sample introduction kit was employed, featuring a Scott-type double-pass spray chamber with Peltier cooling and a SeaSpray nebulizer, providing excellent matrix tolerance, chemical resistance, and signal stability.

All analyses were performed in a routine laboratory environment, rather than under cleanroom conditions, demonstrating the method's robustness and suitability for standard operational settings.

In total, 30 elements were determined in the PET and rPET samples, including 24 elements and 4 REEs. This extended scope provides a more comprehensive elemental profile, supporting advanced research, regulatory compliance, and quality control.

Instrument operating parameters, including plasma conditions, gas flows, and iCRC settings, are summarized in Table 1.

Table 1: Instrument settings – PlasmaQuant MS.

| Parameter | Specification | |
|---------------------|---|--|
| Plasma gas flow | 9.00 L/min | |
| Auxiliary gas flow | 1.50 L/min | |
| Sheath gas flow | 0.00 L/min | |
| Nebulizer gas flow | 1.00 L/min | |
| Sampling depth | 5.00 mm | |
| Plasma RF power | 1.25 kW | |
| Pump rate | 20 rpm – black/black PVC pump tubing (<1 mL/min) | |
| Stabilization delay | 10 s | |
| iCRC gas setting | He 120 mL/min | ⁴⁴ Ca, ⁷³ Ge, ¹¹⁴ Cd, ¹³⁹ La, ¹⁵³ Eu, ¹⁵⁷ Gd, and ¹⁵⁹ Tb |
| | He 150 mL/min | ²⁷ Al, ⁵¹ V, ⁵⁵ Mn, ⁵⁹ Co, ⁶⁰ Ni, ⁶⁵ Cu, and ⁶⁶ Zn |
| | No gas | ⁷ Li, ¹¹ B, ²³ Na, ²⁵ Mg, ³⁹ K, ⁸⁸ Sr, ⁹⁵ Mo, ¹¹⁸ Sn, ¹²¹ Sb, ¹³⁷ Ba, ²⁰² Hg, and ²⁰⁶⁺²⁰⁷⁺²⁰⁸ Pb |
| | H ₂ 150 mL/min | ⁵² Cr |
| | H ₂ 200 mL/min + boost (4 V) | ⁵⁶ Fe, ⁷⁵ As, and ⁷⁸ Se |
| Dwell time | 10 ms (no gas), 30 ms (He), and 50 ms (H ₂) | |

| Parameter | Specification |
|------------------------|---|
| Scan per replicate | 20 (peak hopping, 1pt/peak) |
| No. of replicates | 3 |
| Sample uptake time | 0 s – ASXpress plus sample introduction system used |
| Internal standards | ^{45}Sc , ^{89}Y , ^{115}In , ^{193}Ir , and ^{209}Bi at 50 $\mu\text{g/L}$, interpolate correction |
| Total acquisition time | 300 s (30 elements + 5 IS elements) |

Samples and reagents

The following high-purity reagents were used throughout method development to ensure trace-level accuracy and minimize contamination:

- Deionized water: >18.2 M Ω -cm, Millipore Milli-Q
- Nitric acid (HNO₃): 69%, sub-boiled, Analytik Jena GmbH+Co. KG
- Sulfuric acid (H₂SO₄): \geq 93%, NORMATOM[®]
- Hydrochloric acid (HCl): 37%, Supelco Suprapur[®]

These reagents were chosen for their ultra-trace purity, making them suitable for multi-elemental analysis by ICP-MS, particularly when analyzing both major and trace elements in complex PET samples.

Sample preparation

For method validation, two certified reference materials (CRMs) of low-density polyethylene were prepared in quadruplicate:

- ERM[®] – EC680m (Joint Research Centre, Geel, Belgium)
- ERM[®] – EC681m (Joint Research Centre, Geel, Belgium)

Each CRM, along with PET and rPET samples, was weighed to 0.1 g. Due to their larger surface area, the PET and rPET samples were ground using a Retsch ZM 200 mill at 8,000 rpm for three minutes (see Figure 1). The ground samples were then transferred into pre-cleaned DAK-100X digestion vessels of the speedwave XPERT microwave-assisted digestion system. A one-step digestion protocol was applied (see Table 2), to ensure complete matrix decomposition and compatibility with ICP-MS analysis.



Figure 1: PET and rPET samples before and after grinding using the Retsch ZM 200 mill at 8,000 rpm for three minutes.

Following digestion, samples were diluted to a final volume of 50 mL polypropylene (PP) tubes (Sarstedt, Germany) with deionized water. Prior to ICP-MS analysis, the digests were further diluted 5- and 10-fold with deionized water.

To assess recovery and matrix effects, each PET and rPET digest was also spiked post-digestion with a multi-element solution. Final spike concentrations ranged from 0.02 to 125 ppb, depending on the expected concentration range of each target element.

Calibration

Standard solutions for external calibration were prepared using 1% (v/v) HNO₃ by appropriate dilution in 50 mL PP tubes. Single element stock solutions (Certipur® 1,000 mg/L, in 2-3% HNO₃) of Al, As, Ba, B, Ca, Cd, Co, Cr, Cu, Fe, Ge, Li, Hg, Mg, Mn, Mo, Na, Ni, Pb, K, Se, Sb, Sn, Sr, V, Zn, La, Eu, Gd, and Tb were used to prepare an eight-point concentration curve as follows:

- Up to 4 µg/L for Hg, Mo, Sr, La, Eu, Gd and Tb
- 0.025 to 5 µg/L for Ba, Co, Ge and V
- 0.05 to 20 µg/L for As, B, Cr, Li, Mg and Mn
- 0.1 to 40 µg/L for Fe
- 0.125 to 50 µg/L for Ni, Pb, Se and Sn
- 0.25 to 100 µg/L for Cd, Cu, K and Sb
- 0.5 to 200 µg/L for Al and Ca
- 1.25 to 500 µg/L for Zn

Table 2: Digestion method parameters used by speedwave XPRT microwave digestion system.

| Parameter | Specification |
|--------------------------------|---|
| Sample amount | 0.1 g |
| HNO ₃ | 3 mL |
| H ₂ SO ₄ | 3 mL |
| HCl | 5 drops |
| Vessel | DAK-100X |
| Step 1: temp. / ramp / hold | 160 °C / 2 min / 5 min at 1,000 W (50%) |
| Step 2: temp. / ramp / hold | 225 °C / 2 min / 40 min at 1,000 W (50%) |
| Step 3: Cooling / time | Room / 30 min |
| Final volume | Fill up to 50 mL with DI H ₂ O |
| ICP-MS prior dilution | 1:10 and 1:5 with DI H ₂ O |

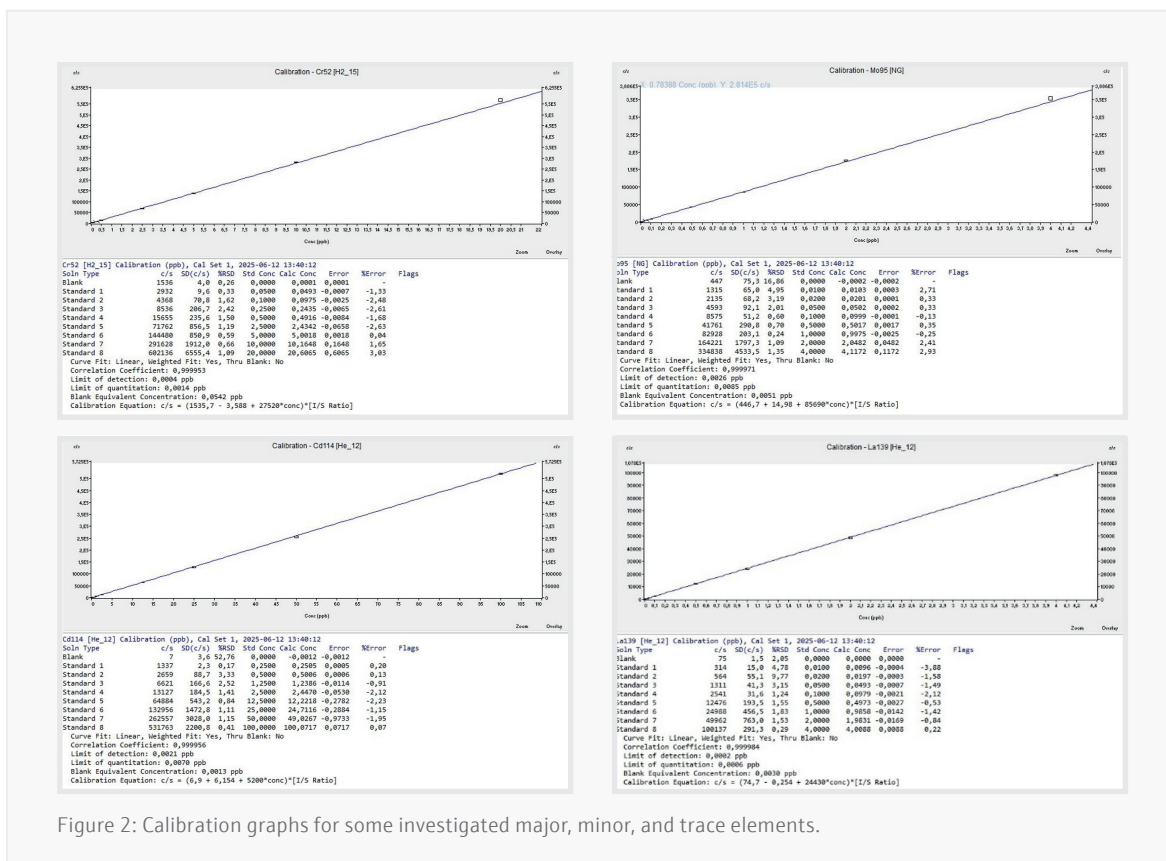


Figure 2: Calibration graphs for some investigated major, minor, and trace elements.

Measurement procedure and data acquisition

To ensure optimal performance, five measurement mode conditions were applied during each measurement cycle:

- Helium at 120 mL/min
- Helium at 150 mL/min
- No gas (standard mode)
- Hydrogen at 150 mL/min
- Hydrogen at 200 mL/min + boost (4 V)

Each condition was paired with specific isotopes, and all five modes were executed sequentially within a single measurement, with ten-second switching intervals between modes.

For data acquisition:

- Three replicate averages were calculated from 20 scans each.
- A final average and standard deviation were calculated from these three replicates.
- Total analysis time per sample (including rinsing and uptake): ~five minutes
- 30 elements and five internal standard elements were measured per sample.

Results and discussion

Digested blank solutions and method detection limits

To account for potential contamination from reagents and vessels, digested blank solutions were prepared using the same procedure as the sample digestions. This included identical volumes of the acid mixture and application of the same microwave-assisted digestion program used for the CRM samples.

The concentrations of Na, Al, K, Ca, Fe, and Ni detected in the procedural blanks were subtracted from the corresponding values measured in the CRM solutions. These elements were specifically corrected due to their presence in the blank at levels that could significantly impact the accuracy of the results – primarily due to trace contamination from the high-purity reagents.

Method detection limits (MDLs) were determined following a full calibration sequence. The MDLs, presented in Table 3, were calculated as ten times the standard deviation of the calibration blank measurement, multiplied by the dilution factor (2,500) to reflect the final sample concentrations.

Table 3: MDL of measured elements in PET and rPET samples obtained by PQMS after microwave-assisted sample digestion and dilution prior to analysis.

| Element | MDL (mg/kg) | Element | MDL (mg/kg) | Element | MDL (mg/kg) |
|---------|-------------|---------|-------------|---------|-------------|
| Li | 0.14 | Fe | 0.78 | Cd | 0.01 |
| B | 0.25 | Co | 0.03 | Sn | 0.07 |
| Na | 1.3 | Ni | 1.1 | Sb | 0.005 |
| Mg | 0.25 | Cu | 0.08 | Ba | 0.03 |
| Al | 1.6 | Zn | 0.44 | La | 0.008 |
| K | 3.1 | Ge | 0.23 | Eu | 0.003 |
| Ca | 27 | As | 0.36 | Gd | 0.02 |
| V | 0.04 | Se | 0.33 | Tb | 0.0008 |
| Cr | 0.01 | Sr | 0.004 | Hg | 0.005 |
| Mn | 0.14 | Mo | 0.01 | Pb | 0.02 |

Spike recoveries, CRM recoveries, and precision

Tables 4a and 4b present the results for the certified elements in the two analyzed CRMs. These tables include the mean concentrations measured across the four replicates, precision data, recoveries compared to the certified values, and Z-scores to assess accuracy. Additional elements that were measured are not included in these tables, as they were not certified, and since most were below the MDLs.

Figure 3 illustrates the overall recoveries of all certified elements from the two CRMs and the precision of the four replicates, while Figure 4 displays the Z-scores. The recoveries ranged from 86% to 105%, indicating excellent accuracy, as the Z-scores fell within the range of [-2.0, 2.0]. This suggests that the developed method was properly validated.

Table 4a: Metals and semi-metals concentration, recoveries, precision (RSD), and Z-score achieved in the ERM[®]-EC680m after microwave-assisted digestion (n=4).

| Isotope | Certified value ± U (mg/kg) | Measured conc. (mg/kg) | RSD (%) | Recovery (%) | Z-score |
|---------|-----------------------------|------------------------|---------|--------------|---------|
| Cr | 9.6 ± 0.5 | 9.8 | 9.1 | 102 | 0.4 |
| Zn | 194 ± 12 | 193 | 5.3 | 99 | -0.1 |
| As | 4.7 ± 0.4 | 4.6 | 9.0 | 99 | -0.2 |
| Cd | 20.8 ± 0.9 | 20.8 | 3.4 | 100 | 0.0 |
| Sn | 20.7 ± 1.6 | 20.6 | 6.6 | 99 | -0.1 |
| Sb | 9.6 ± 0.7 | 9.7 | 7.2 | 101 | -0.1 |
| Hg | 2.56 ± 0.16 | 2.6 | 3.8 | 101 | -0.2 |
| Pb | 11.3 ± 0.4 | 11.9 | 6.9 | 105 | 1.5 |

Table 4b: Metals and semi-metals concentration, recoveries, precision (RSD), and Z-score achieved in the ERM[®]-EC681m after microwave-assisted digestion (n=4).

| Isotope | Certified value ± U (mg/kg) | Measured conc. (mg/kg) | RSD (%) | Recovery (%) | Z-score |
|---------|-----------------------------|------------------------|---------|--------------|---------|
| Cr | 45.1 ± 1.9 | 44.6 | 2.4 | 99 | -0.3 |
| Zn | 1,170 ± 40 | 1,168 | 13 | 100 | 0.0 |
| As | 17 ± 1.2 | 18 | 11 | 104 | 0.6 |
| Cd | 146 ± 5 | 146 | 4.0 | 100 | -0.1 |
| Sn | 99 ± 6 | 89 | 5.1 | 90 | -1.7 |
| Sb | 86 ± 7 | 84 | 5.3 | 98 | -0.3 |
| Hg | 9.9 ± 0.8 | 8.5 | 4.8 | 86 | -1.8 |
| Pb | 69.7 ± 2.5 | 65 | 3.5 | 94 | -1.7 |

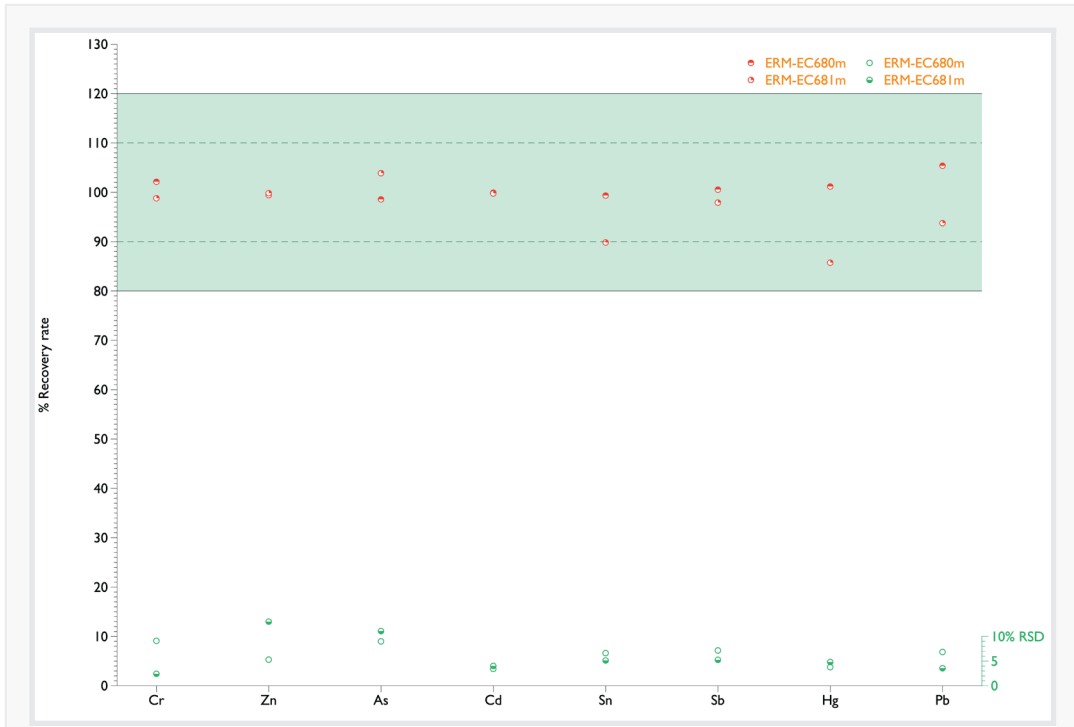


Figure 3: Recovery rate and precision of two CRMs. All measurements are within the range specified.

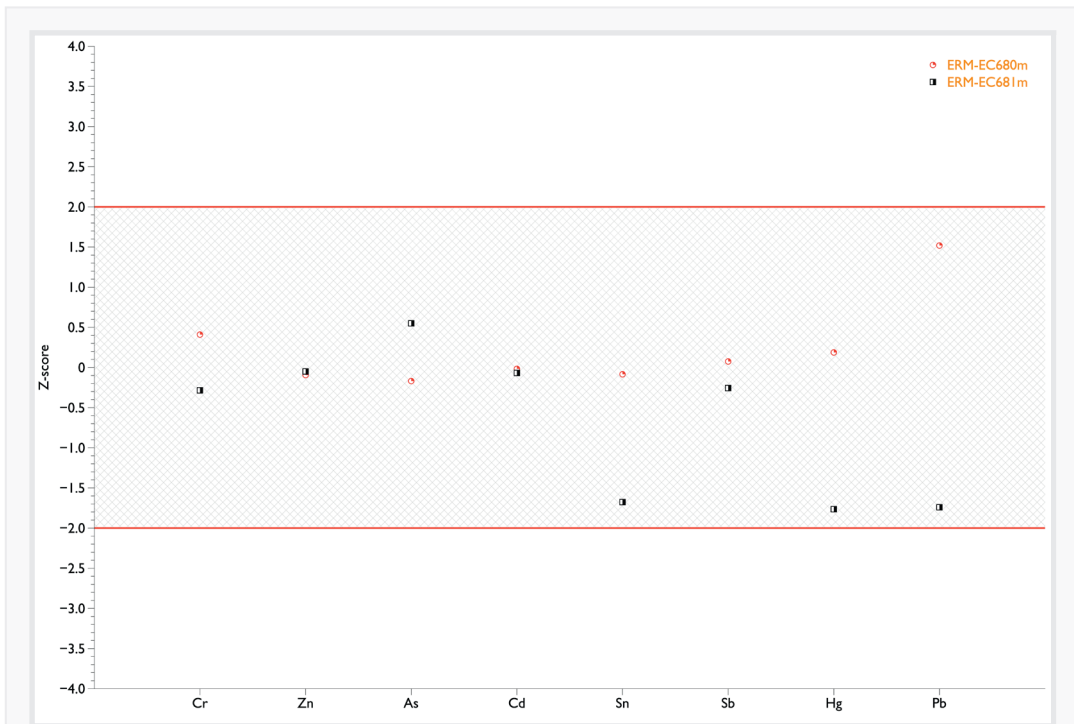
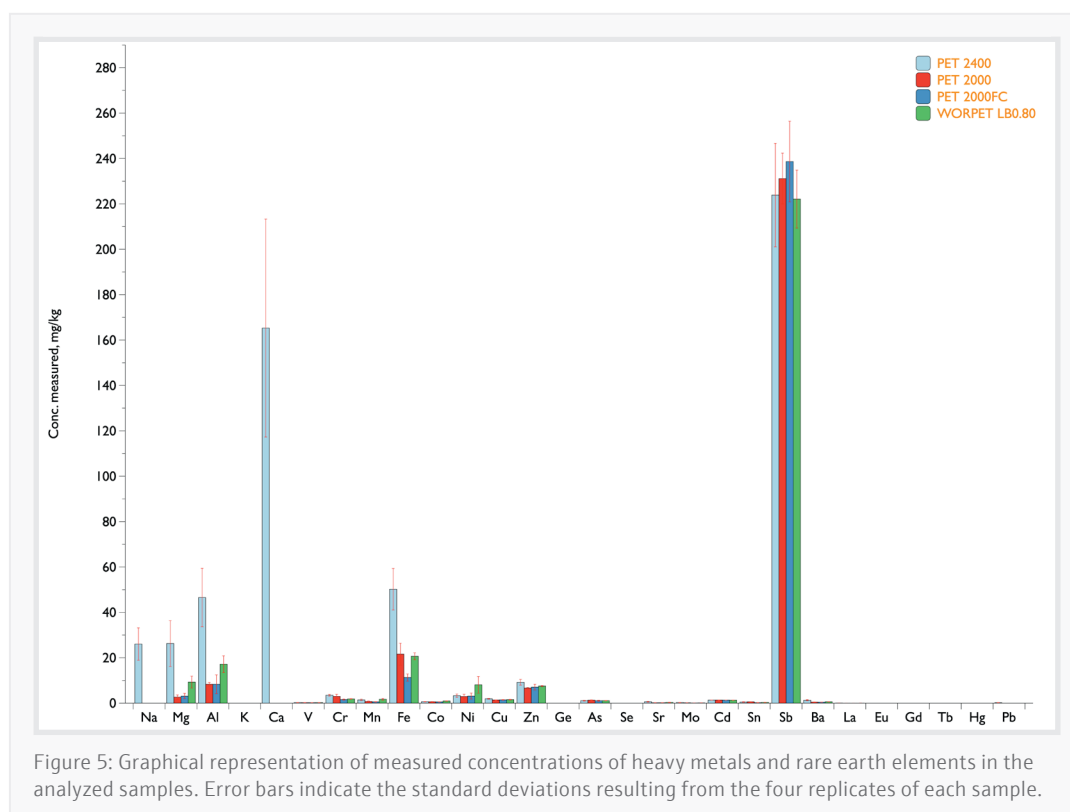


Figure 4: Z-score distribution of all elements measured in two CRMs.



Summary

In conclusion, the presence of lanthanum (La) in the PET and rPET samples serves as a potential indicator of rare earth element (REE) contamination from non-PET sources, such as e-waste, glass, and lenses. This correlation suggests possible contamination pathways arising from industrial applications, as these sources often contain La, supported by previous studies that reveal high concentrations of REEs in e-waste and glass products [5, 6]. The analytical method demonstrated excellent recoveries, achieving 86–105% for certified elements in two certified reference materials (CRMs). The method maintained high precision, with a relative standard deviation (RSD) of less than 10% across four replicates, even in the presence of random contamination.

Additionally, the initial washing process significantly reduced concentrations of sodium (Na), magnesium (Mg), aluminum (Al), calcium (Ca), and iron (Fe). Importantly, no significant changes in element concentrations were observed after further recycling, indicating stability in the recycling process. Nevertheless, leaching tests are necessary to assess compliance with EU migration limits for food safety.

Overall, the validated ICP-MS method for 30 elements in recycled PET not only supports quality control in recycling workflows but also contributes to ensuring the safety and



integrity of recycled materials in food contact applications. By effectively detecting trace heavy metals (HMs) and REEs, this method addresses the critical challenge of food safety compliance, providing food packaging manufacturers, recycling companies, regulatory bodies, and analytical laboratories with a reliable tool to safeguard consumer health and uphold industry standards.

Recommended device configuration

Table 6: Overview of devices, accessories, and consumables.

| Article | Article number | Description |
|-------------------------------------|----------------|---|
| PlasmaQuant MS | 818-08010-2 | PlasmaQuant MS is ideally suited for the elemental analysis of solid biofuels in line with ISO 16967 and ISO 16968. Its robust plasma and high sensitivity ensure accurate results even in complex matrices, making it the perfect tool for quality control and research in bioenergy applications. |
| Teledyne-Cetac ASX-560 autosampler | 810-88015-0 | The Teledyne CETAC Technologies ASX-560, next generation autosampler with integrated rinse function is sleek and durable by design. |
| Cetac ASXpress plus (or equivalent) | 810-88017-0 | The ASXpress plus is a Rapid sample introduction accessory which reduces sample uptake, stabilization, and rinse times, significantly cutting down sample run times while offering excellent washout behavior. |

Acknowledgement

This work was done in collaboration with Ecoibéria located in Famalicão, Portugal, which provided all the PET and rPET sample types and meticulously handled the sampling, preservation, and storage processes.



References

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