



Heavy Metal Analysis in Cocoa Powder Using ICP-MS: Implications for Food Safety and Metrology in South Africa

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Introduction

Cocoa powder is a popular product for its unique flavour and health benefits [1]. However, the presence of heavy metals and other trace elements, such as Cd, B, Cr, Mn, Fe, Co, Ni, Cu, Zn, Mo, Al, P, S, K, As, and Ca, has raised concerns regarding food safety and regulatory compliance [2, 3]. South Africa relies on imported cocoa powder, mainly sourced from cocoa-growing regions in West Africa and South America [4]. Toxic elements like cadmium (Cd) and arsenic (As) are of particular concern due to their potential health risks, such as kidney damage [5, 6].

On the contrary, essential elements like potassium (K) and iron (Fe) contribute to the nutritional value of cocoa products [7]. Accurately quantifying these elements is important for ensuring compliance with international food safety standards, such as those set by the Codex Alimentarius Commission and the European Union (EU) [8, 9].

This study highlights the key role of metrology in ensuring the reliability, accuracy, and metrological traceability of elemental analysis using ICP-MS [10]. The involvement of South Africa's National Metrology Institute (NMISA) ensures that measurements conducted by analytical laboratories are traceable to the International System of Units (SI), encouraging global comparability and confidence in the results [11].

Materials and Methods

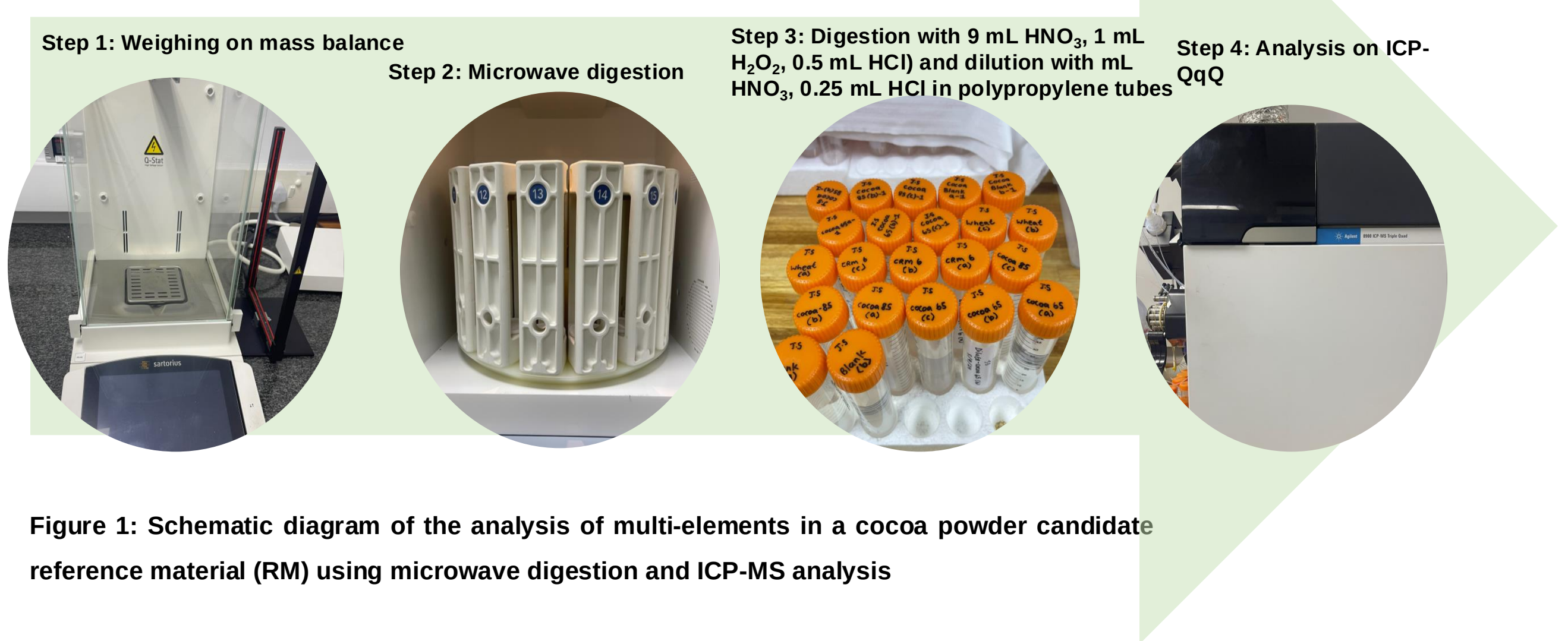


Figure 1: Schematic diagram of the analysis of multi-elements in a cocoa powder candidate reference material (RM) using microwave digestion and ICP-MS analysis

Results and Discussion

The analysis of heavy metals and nutritional elements in cocoa powder candidate reference material was conducted using external calibration with gravimetric preparation of the samples and standards with gravimetric addition of the internal standard and analysis with ICP-QqQ. The results provided quantitative concentrations (µg/kg and mg/kg) for each element, ensuring reliability and high accuracy with established metrological traceability (see Figure 2).

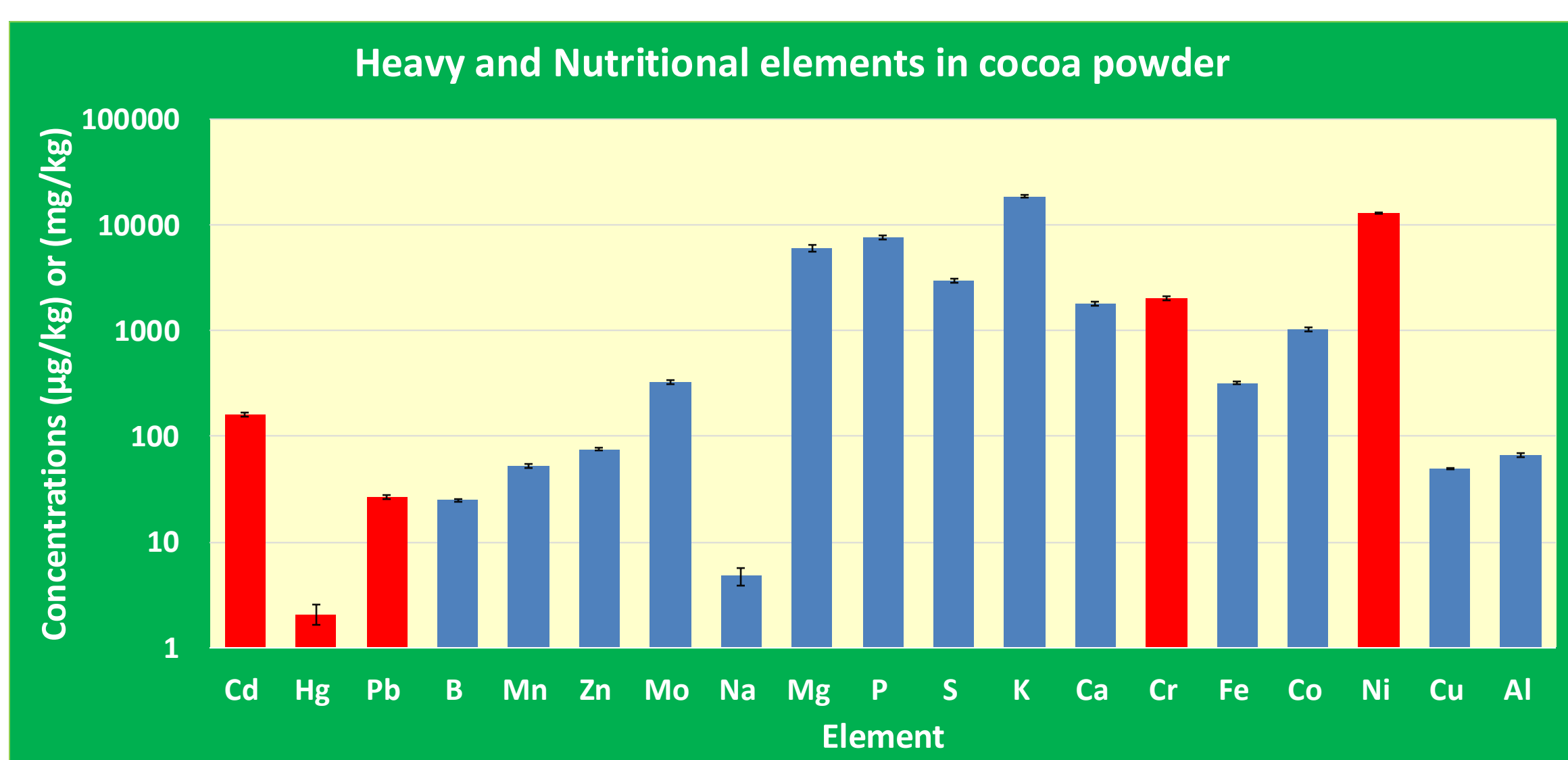


Figure 2: Illustration of the concentration levels with its associated uncertainty values of heavy metal elements (in red), and the nutritional elements (in blue) in the cocoa powder samples

Heavy Metal Analysis

The Cd concentration is 200 times the EU limit for cocoa powder, posing a significant risk. Hg is well below the EU limit, and Pb is within the acceptable range. While no strict limits exist for Cr and Ni in cocoa, their levels are high based on general food safety guidelines, requiring further risk assessment. International limits for Cd, Pb (cocoa powder), and Hg (food) are 0.8 mg/kg, 0.5 mg/kg, and 0.1 mg/kg, respectively. Cd levels in this study are extremely high, while Cr and Ni also require further evaluation. Pb and Hg remain within safe limits.

Nutritional element analysis

The cocoa powder contains essential minerals (Mn, Zn, Mo, Na, Mg, P, S, K, Ca, Fe, Cu) within acceptable ranges, supporting a balanced diet. Co and Al levels are high but not toxic, while Cu is at its maximum limit and may pose a toxicity risk.

Method Validation Using Wheat Flour CRMs

This study assesses elemental measurement accuracy using two wheat flour CRMs (NIST SRM 1567b & NMISA CRM-0006). While most elements performed well, some had high En-scores, indicating potential bias. In the NIST SRM, Zn (3.81), Na (4.09), K (2.59), and Ca (2.32) were elevated, likely due to matrix effects or calibration bias, while Mn (1.10), Fe (1.25), and Mo (1.03) were near the warning threshold. In the NMISA CRM, Zn (4.89), Pb (3.78), and Na (4.09) exceeded limits, possibly due to certification or instrumental issues. Cr (0.75), Mn (0.12), Fe (0.28), and K (0.70) were within acceptable accuracy.

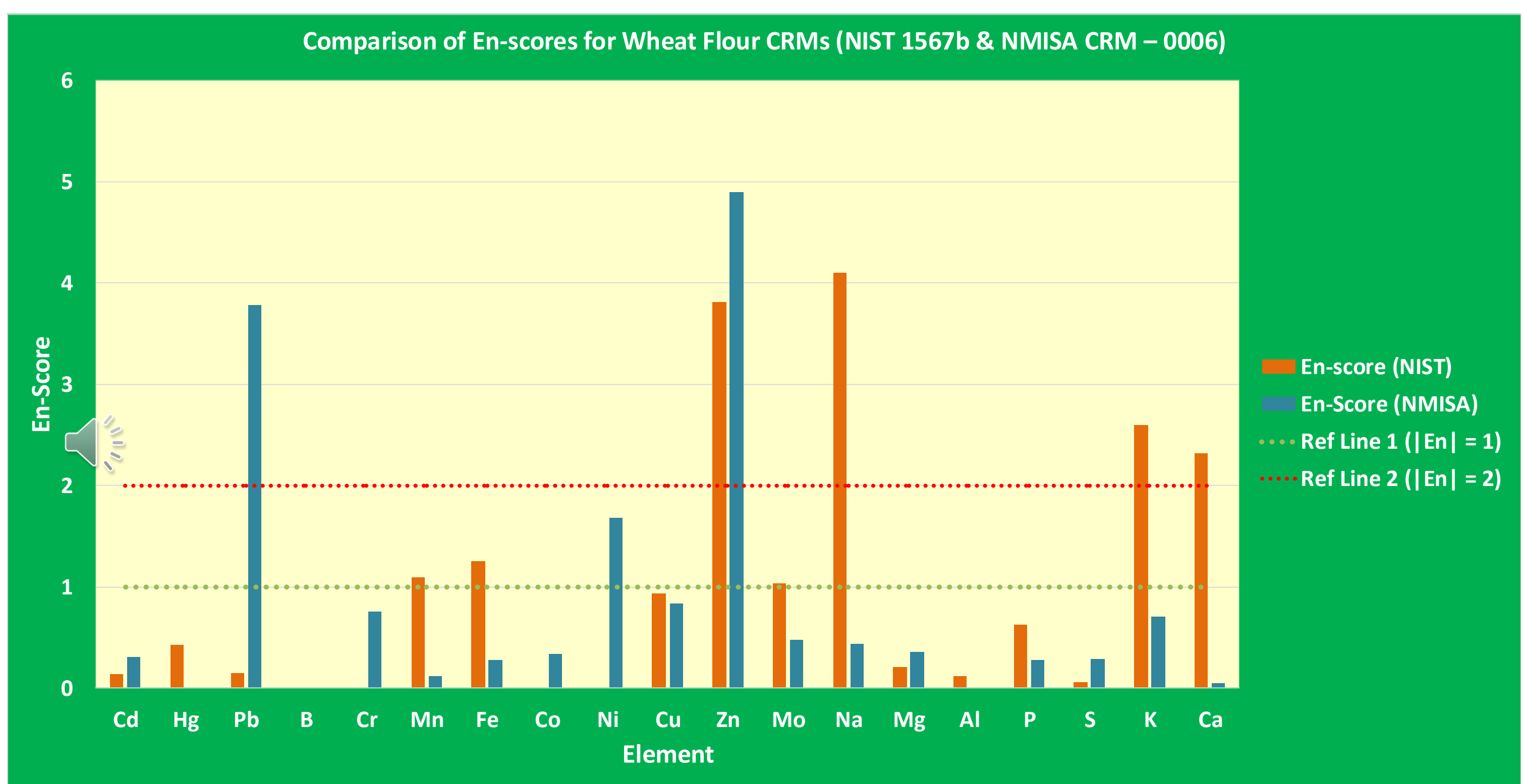


Figure 3: E_n -scores for Wheat Flour Certified Reference Materials (CRMs): NIST SRM 1567b and NMISA CRM-006. The E_n -scores evaluate the accuracy of measurement results, with reference lines at $|E_n| = 1$ (acceptable range)

Conclusion

Hg and Pb levels in the cocoa powder meet South African and international safety standards, but Cd, Cr, and Ni levels are high, raising concerns about safety. While most nutritional elements comply with regulations, Co, Al, and Cu should be monitored for long-term risks. The method showed good accuracy, though Zn, Na, K, Ca, and Pb need further evaluation. Future work will address matrix effects and calibration issues.

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