Simple and Reliable Determination of Heavy Metals in Soil with the PinAAcle 900H AA Spectrometer

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1 Introduction

Soil, as the root of the food chain, plays a central role in food safety and human health. It is vitally important to routinely monitor heavy metals in soil to protect soil quality and food safety. To prevent and control soil pollution, the Chinese government implemented the 'Ten Action Plan' and conducted a nationwide soil quality sampling analysis starting in 2016 and continuing to the present. For analytical labs, the determination of heavy metal elements in soils has been a challenge because of the large variety of soil samples and stringent requirements for accuracy and throughput. A rapid and accurate solution for inorganic contaminants in soil was established and verified in this work.
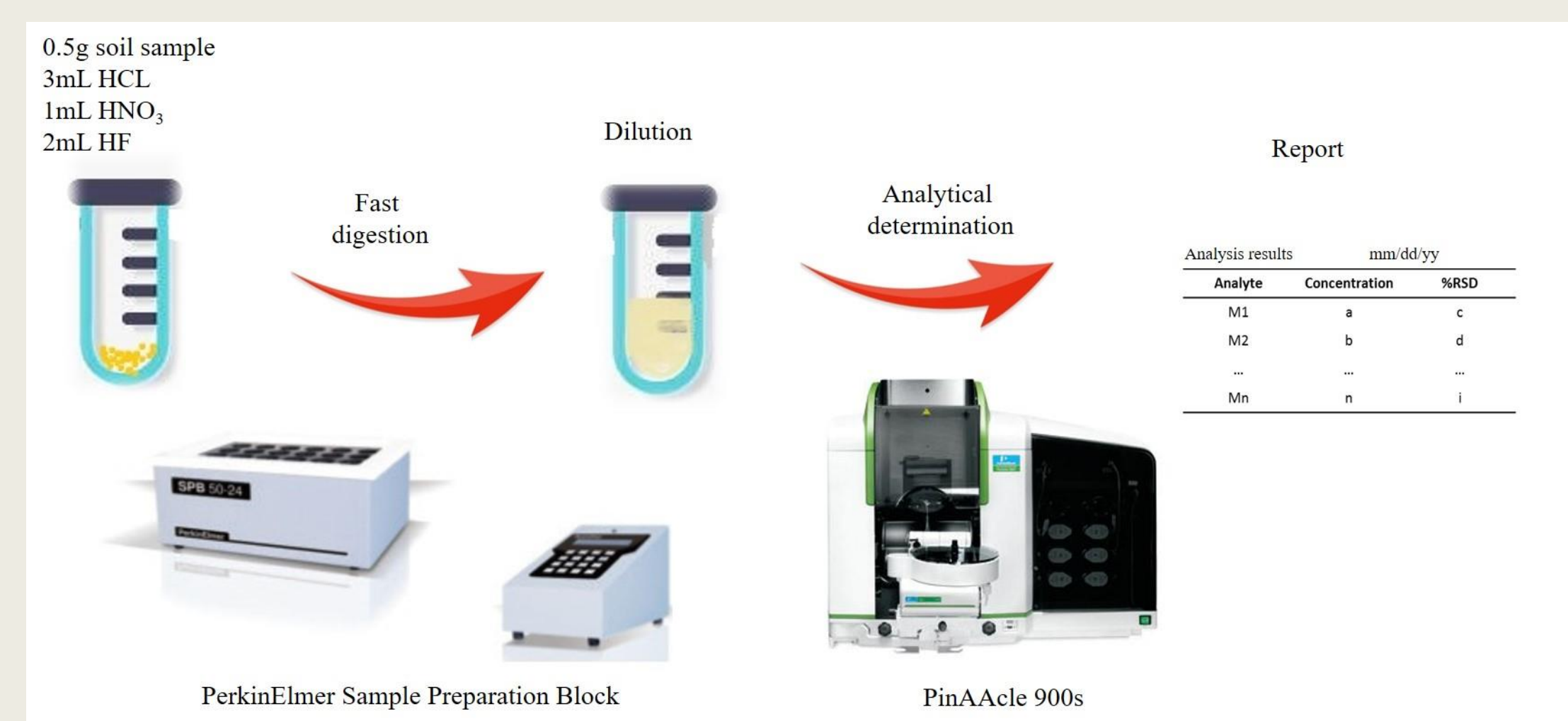
Schematic of inorganics determination in soil by PerkinElmer

2 Experiments

1. Soil samples
Four soil Certified Reference Materials:
GSS 2, GSS 8, GSS 13, GSS 14.
Elements of interest:
Lead (Pb), Cadmium (Cd), Copper (Cu), Zinc (Zn), Chromium (Cr) and Nickel (Ni).

2. Soil digestion
A SPB 50-48 Sample Preparation Block system (PerkinElmer Inc., USA) was used for sample fast digestion. The samples were digested at 120 °C for 1h with the acids of HCl, HNO_3 and HF. After being diluted with D.I. water and then centrifuged, the supernatant solution was instrumentation-ready.
The PerkinElmer fast digestion method is superior in many aspects compared to conventional techniques: high efficiency, simple operation, less reagent consumption, minimized potential contamination and lower cost.

Digestion Method	Reference reg.	Time (hr)	Acid volume*	Vessel
Microwave	EPA 3051A; EPA 3052	2	~10 mL	PTFE digestion vessel, PP volumetric tube
Heating Plate	EPA 3050B; GB 15618	2+	~20 mL	PTFE beaker, PP volumetric tube
SPB Fast Digestion	PerkinElmer solution	1	6 mL	PP volumetric tubes

* Acid volume based on the digestion of 0.2-0.5g sample

3. Instrumentation
All measurements were performed using a PerkinElmer PinAAcle™ 900 H AA Spectrometer (PerkinElmer Inc., Shelton, CT USA) equipped with Syngistix™ for AA Express software, which provides a simplified workflow and only three steps to acquire results. PerkinElmer Lumina single-element hollow cathode lamps were used as light source. Air-acetylene was used for flame analysis. PerkinElmer graphite furnace autosampler AS 900 was used for Pb and Cd analysis.

In accordance with Chinese analytical standards, Cu, Zn, Ni and Cr were determined by Flame AA (GB/T 17138, GB/T 17139 and HJ 491), while Pb and Cd were analyzed on Graphite Furnace AA (GB/T 17141).

FLAA operation conditions	Cu	Ni	Zn	Cr
Wavelength (nm)	324.75	232.0 Alternatives: 352.4	213.86	357.87
Slit Width (nm)	0.7	0.2	0.7	0.7
Mode	AA	AA-BG*	AA- BG	AA
Calibration standards (mg/L)	0.1,0.2, 0.6,0.8	0.1, 0.2, 0.4, 0.8	0.05,0.1, 0.2,0.3	0.2,0.4, 0.6,0.8
Read Time (sec)	3	3	3	3
Acetylene Flow (L/min)	2.5	2.5	2.5	4
Air Flow (L/min)	10	10	10	10

| GFAA operation conditions	Pb	Cd
Wavelength (nm)	283.31	228.80
Slit Width (nm)	0.7	0.7
Mode	AA-BG	AA-BG
Signal Measurement	Area	Area
Calibration standards (ug/L)	12.5, 25, 37.5, 50.0	0.5, 1.0, 1.5, 2.0
Read Time (sec)	4	5
Matrix Modifier	$NH_4H_2PO_4$	$NH_4H_2PO_4$
Sample Volume (µL)	16	16
Matrix Modifier Volume (µL)	5	5

*= Atomic absorption with background correction
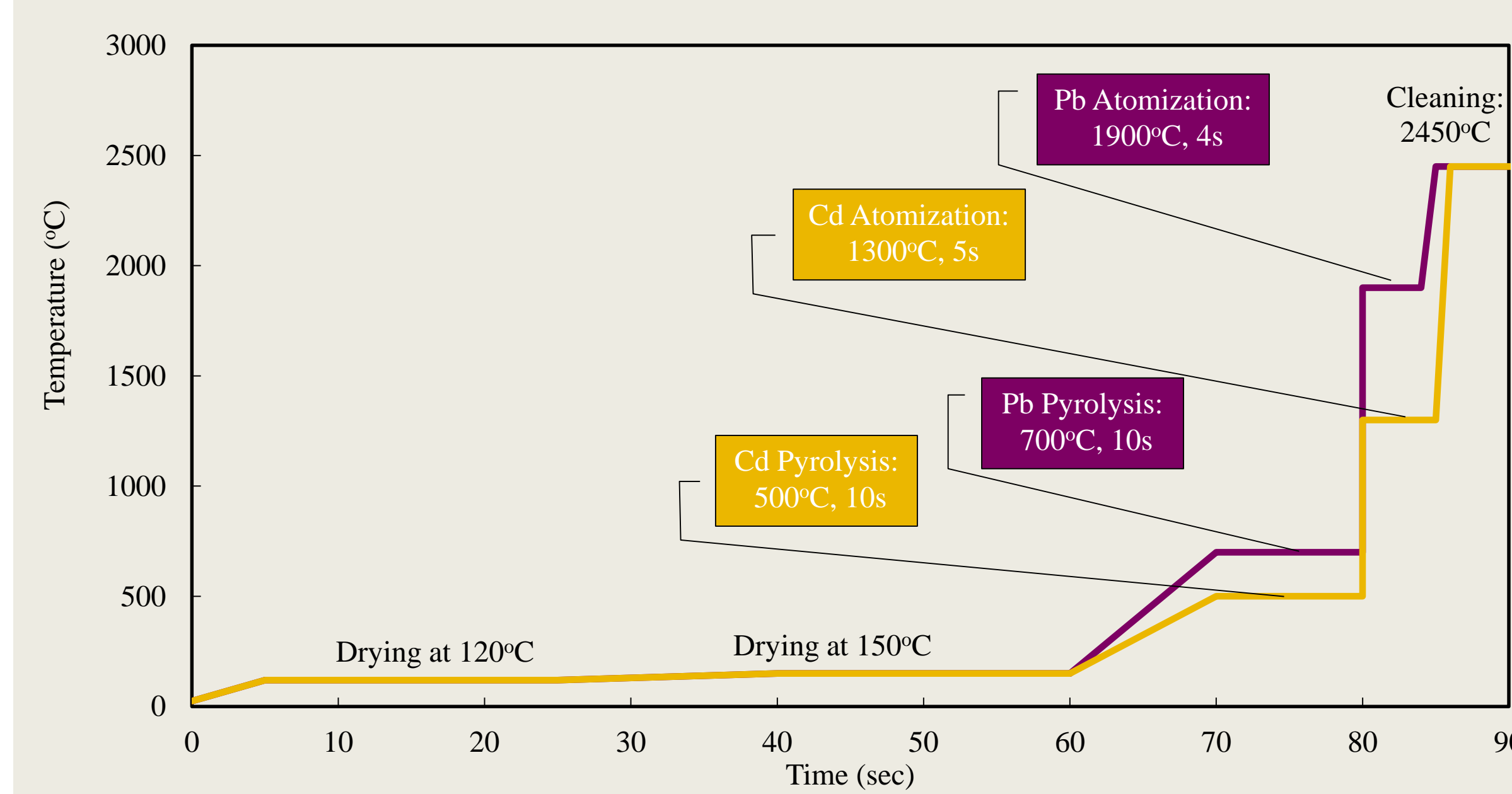
GFAA Temperature Programs for Pb and Cd

3 Results and discussion

Calibration curves of Cu, Zn, Cr, Ni, Pb and Cd by method of addition, correlation coefficients of better than 0.999 were obtained

All Method Detection Limits (MDL) obtained fully meet the required MDLs in the China National Standard and are far below the limiting values specified in the soil quality standards of China, Germany, Holland, Australia and Canada.
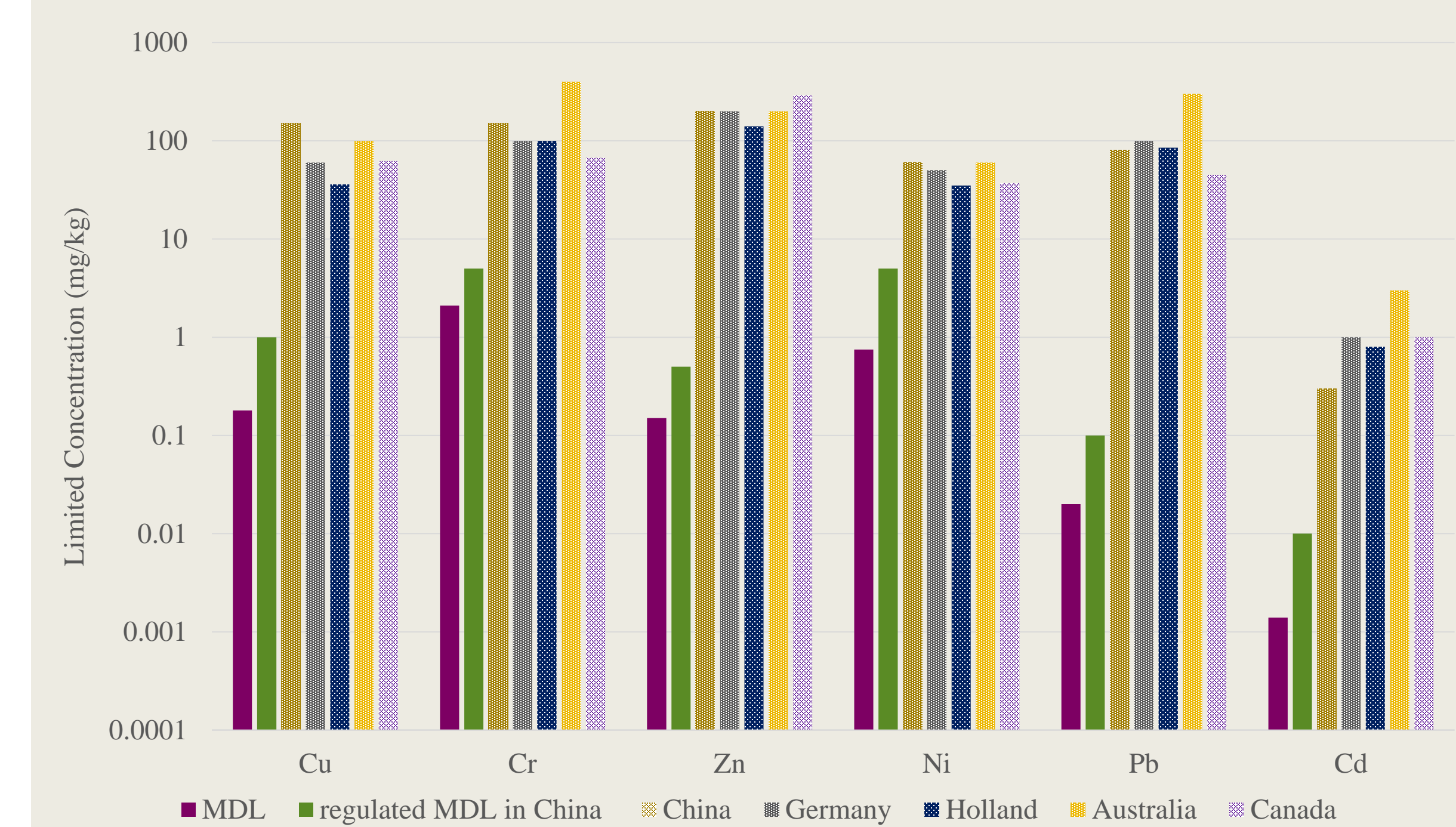
Experimental MDLs compared to regulated levels in the China standard and the maximum allowable concentrations in China, Germany, Holland, Australia, and Canada

The accuracy of the method and effectiveness of sample pretreatment were validated by analyzing four representative soil Reference Materials GSS 2, GSS 8, GSS 13, and GSS 14. The measured and certified results are in great agreement. All the recoveries were within the range of 90% -110%.

Recoveries of four soil CRMs: well agree in confidence limit
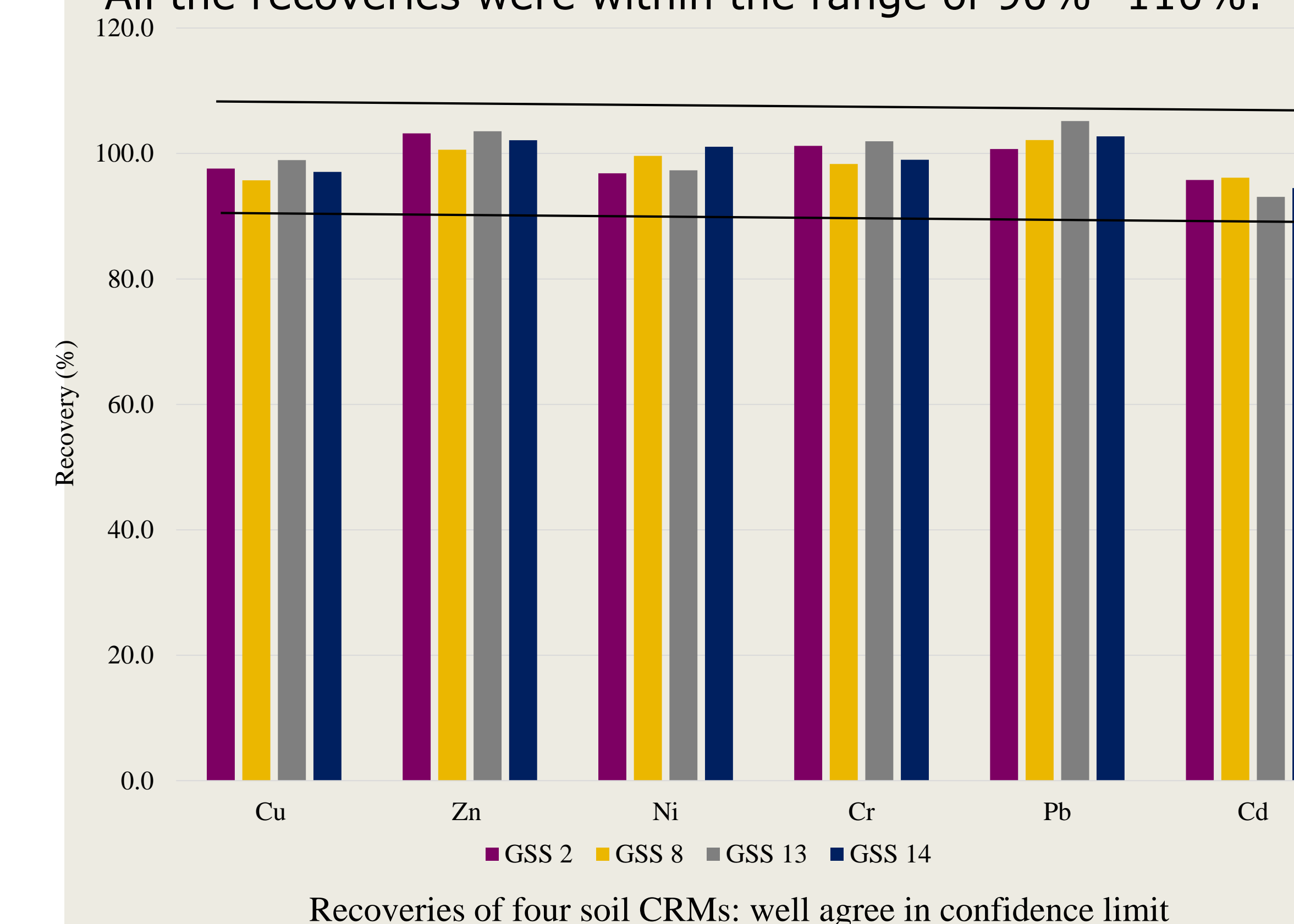
4 Summary

The PinAAcle™ 900 H AA spectrometer, equipped with both a flame and HGA graphite furnace, provides outstanding performance for analyzing a variety of soil samples over a wide range of concentrations. Excellent correlation coefficients of better than 0.999 were obtained and the measured method detection limits met requirements in regulations; The powerful capability of background correction in PinAAcle ™ 900 H AA spectrometer significantly eliminated matrix interference and guaranteed accurate results.

The simple and rapid fast digestion technique ensures greater sample throughput which effectively increases efficiency in environmental labs. Meanwhile, less acid consumption leads to lower cost and the potential of less contamination.

Fast digestion followed by PinAAcle 900 AA analysis provides a new solution for determination of inorganic contaminants in soil.