

Trace and Major Elements Analysis of Environmental Samples Using EPA 6020B

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Introduction

Solid and liquid wastes from different commercial and industrial facilities contaminate our water and land and affect the environment. EPA method 6020B is a guidance method for the analysis of water and waste samples using inductively coupled plasma-mass spectroscopy (ICP-MS) included in the SW-846 (Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods Compendium).



Here, we present the performance demonstration of the Shimadzu ICPMS-2030 equipped with CETAC autosampler for the analysis of 19 elements in soil samples in accordance with method 6020B. This configuration provides high capacity and throughput for automatic analysis of hundreds of samples. The obtained results demonstrate that the Shimadzu ICPMS-2030 provides excellent sensitivity, precision, accuracy, tolerance, and fast response to meet and exceed quality assurance criteria outlined in EPA 6020B.

Experimental

Instrumentation

A Shimadzu ICPMS-2030 equipped with a CETAC ASX-280 Autosampler and a Glass Expansion in-line internal standard addition kit was used for all analyses. Operational parameters of the Shimadzu ICPMS-2030 are listed in Table 1.

Table 1. Operating conditions of Shimadzu ICPMS-2030

Parameter	Setting	Parameter	Setting
Radio Freq. Power	1.20 kW	Plasma Gas	8.0 L/min
Sampling Depth	5.0 mm	Auxiliary Gas	1.10 L/min
Chamber Temp.	5 °C	Carrier Gas	0.70 L/min
Total Integration Time	2 sec	Number of Repetition	3

Sample Preparation, Tuning, Calibration and Internal Standards

Two Certified soil samples from Inorganic Ventures (CRM-Soil-A and CRM-Soil-B) were diluted 10 times with 2% nitric acid before analysis.

Calibration standards were prepared by diluting the Inorganic Ventures multi-elements standards with 2% nitric acid.

The instrument was tuned to adjust instrument parameters prior to analysis using Shimadzu's ICP-MS tuning solution to ensure proper torch position, lens voltages, and mass resolution.

Appropriate calibration ranges were selected for each analyte to cover the required analysis range. All the calibration curves show excellent linearity across the respective calibration range. R values are >0.999, as shown in Table 2.

Table 2. Analytical elements and their corresponding measurement parameters

Element	Mass	Internal Standard	Calibration Range (ppb)	Calibration R	Scan Time (Sec)	Num. of Scans	Collision Cell
Al	27	Li (6)	0 - 10000	0.99973	0.2	10	ON
As	75	Y (89)	0-100	0.99990	0.2	10	ON
Ba	137	Tb (159)	0-10000	0.99990	0.2	10	OFF
Ca	44	Sc (45)	0-10000	0.99996	0.2	10	ON
Cd	111	In (115)	0-100	0.99968	0.2	10	ON
Co	59	Sc (45)	0-100	0.99997	0.2	10	ON
Cr	52	Sc (45)	0-100	0.99996	0.2	10	ON
Cu	63	Sc (45)	0 - 100	0.99935	0.2	10	ON
Fe	56	Sc (45)	0 - 10000	0.99963	0.2	10	ON
K	39	Sc (45)	0 - 10000	0.99942	0.2	10	ON
Mg	24	Li (6)	0 - 100	0.99973	0.2	10	ON
Mn	55	Sc (45)	0 - 100	0.99985	0.2	10	OFF
Na	23	Li (6)	0 - 100	0.99906	0.2	10	ON
Ni	60	Sc (45)	0 - 200	1.00000	0.2	10	ON
Pb	208	Bi (209)	0 - 5000	0.99999	0.2	10	OFF
Sb	121	In (115)	0 - 10000	0.99977	0.2	10	OFF
Se	78	Y (89)	0 - 100	0.99968	0.2	10	ON
V	51	Sc (45)	0 - 100	0.99960	0.2	10	ON
Zn	66	Sc (45)	0 - 10000	0.99945	0.2	10	ON

Internal standard containing Yttrium, scandium, indium, bismuth, terbium and lithium were added to all samples using the internal standard addition kit, with dilution factor 1:10. The final concentration of the internal standards was 100 ppb.

Results and Discussion

To validate the instrument's analytical capabilities, two certified samples with known concentrations of the analyte elements were analyzed.

Table 3. Measured and expected concentrations of elements in certified soil samples and recovery yields

Element	Mass	Certified Soil Sample A			Certified Soil Sample B		
		Certified Conc. (ppb)	Measured Conc. (ppb)	Recovery %	Certified Conc. (ppb)	Measured Conc. (ppb)	Recovery %
Al	27	500000	453000	90.6	700000	653000	93.2
As	75	200	194	97.0	6000	5490	91.5
Ba	137	5000	5110	102.2	7000	6810	97.3
Ca	44	350000	356000	101.7	125000	121000	96.8
Cd	111	NA	NA	NA	200	199	99.5
Co	59	NA	NA	NA	100	106	106
Cr	52	NA	NA	NA	400	416	104
Cu	63	300	271	90.3	3000	2760	92.0
Fe	56	200000	180000	90.0	350000	348000	99.4
K	39	200000	205000	102.5	210000	207000	98.5
Mg	24	70000	63800	91.1	80000	82000	102.5
Mn	55	100	91.5	91.5	100000	90100	90.1
Na	23	70000	66600	95.14	100000	102000	102.0
Ni	60	300	283	94.3	200	217	108.5
Pb	208	NA	NA	NA	60000	61800	103.0
Sb	121	NA	NA	NA	300	291	97
Se	78	NA	NA	NA	10	9.2	92
V	51	100	96.8	96.8	800	814	101.7
Zn	66	1000	1080	108.0	70000	648000	92.5

Table 3 shows the concentrations of 19 elements in certified soil samples and their respective recoveries. The measured values are compared to the certified values to calculate the recoveries. If the sample was not certified for the certain elements, the values for those elements were indicated as NA in Table 3.

The recoveries for all analytes fall within the acceptable range of 90 to 110% demonstrating the methodology and the high accuracy.

The recoveries for the certified samples were also displayed in Figure 2; ($\pm 10\%$ of the certified concentration range showed with green color).

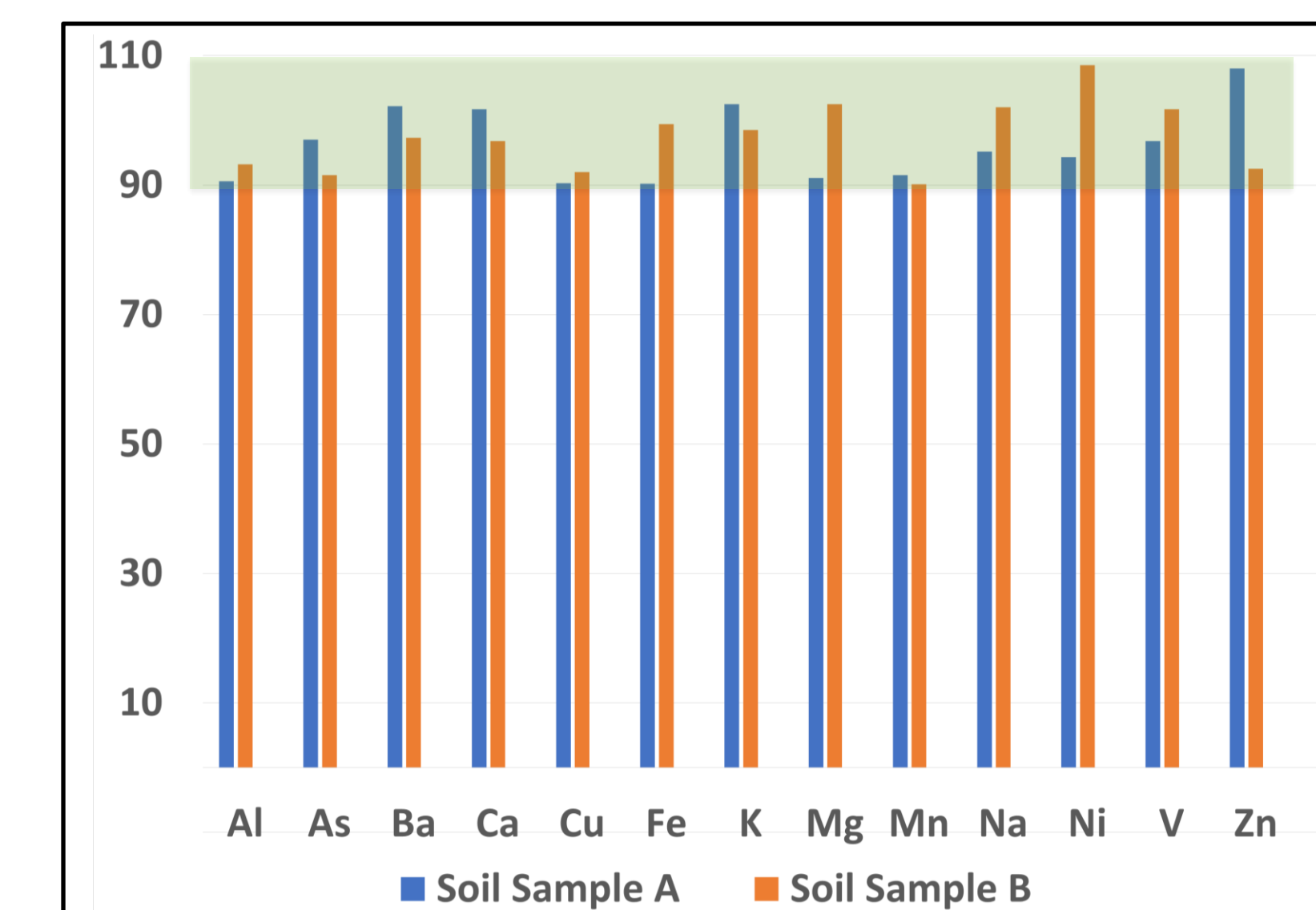


Figure 2. Recovery for certified soil samples

Conclusions

The environmental samples were successfully analyzed with the Shimadzu ICPMS-2030 according to method EPA 6020B. The obtained recoveries were in the acceptable range between 90 to 110% and all RSDs are below 5% demonstrating method performance and accuracy. The Shimadzu ICPMS-2030 provides excellent sensitivity, precision, accuracy, tolerance and fast time response to meet and exceed compliance with regulations on trace elements analysis in water, waste, and soil samples.

References

- "Analysis of River Water by ICPMS-2030", Shimadzu Application News, No.J118.