Challenge
Matrix effects, sample digestion, calibration, quality control, method validation, element interactions and instrument performance in soil analysis for HJ 766 and ISO 14869 compliance

Solution
An effective microwave digestion procedure with an accurate and precise ICP-MS determination to overcome matrix effects

Intended audience
Environmental consultants, agricultural industry, mining companies, soil testing labs, research institutions

Multielemental Analysis of Soils using ICP-MS after Microwave-assisted Digestion in Compliance with HJ 766 and ISO 14869

Introduction

Accurate determination of elements in soil analysis presents a challenge due to the complex nature of soil matrices. This application note focuses on addressing this challenge by utilizing microwave digestion and ICP-MS in accordance with HJ 766[1] and ISO 14869[2] standards. The objective is to achieve reliable and accurate quantification of elements in soil samples, essential for environmental monitoring, agricultural practices, and geological studies. The samples used in this study include diverse soil types, representing various geographical locations and potential sources of contamination. Key analytical parameters such as low detection limits, high accuracy, and the ability to handle a wide range of elements with varying concentrations are crucial for effective soil analysis.

The analytical challenge was addressed by utilizing microwave-assisted digestion by means of the speedwave XPERT and multielemental analysis by means of the PlasmaQuant MS.

The PlasmaQuant MS system offers exceptional sensitivity, allowing for accurate quantification of trace elements even at low concentrations. It provides a wide dynamic range, ensuring reliable results across a wide concentration range. For sample digestion, the speedwave XPERT system offers rapid and efficient microwave-assisted digestion, enabling complete decomposition of soil samples. The methodology combines the capabilities of the PlasmaQuant MS with the speedwave XPERT systems to deliver robust and accurate element determination in soil samples, meeting the specific requirements of HJ 766 and ISO 14869.

Due to the high sensitivity of the PlasmaQuant MS low detection limits can be achieved, ensuring accurate assessment of soil contamination even at trace levels. The speedwave XPERT microwave digestion system facilitated efficient sample preparation, reduced digestion times, and minimizing matrix interferences.

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The combined solution met the specific requirements of HJ 766 and ISO 14869, providing reliable and compliant results.

The main objective of this work is to provide a validated method for sediments/soils analysis covering the elements mentioned in HJ 766 and ISO14869 regulations using Certified Reference Materials (CRMs) which can be used routinely by any laboratory e.g., in environmental monitoring, soil fertility, contamination detection, geological exploration, or land restoration activities.

Materials and Methods

Instrument settings

The digestion of the soil samples was carried out by the closed-vessel microwave-assisted speedwave XPERT system. The multielement analysis was performed using the PlasmaQuant MS featuring the integrated collision/reaction cell (iCRC) technology to remove polyatomic interferences formed in the plasma for accurate analysis. The ICP-MS system was coupled to a CETAC ASX-560 autosampler and a CETAC ASXPress Plus injection valve and equipped with a Scott type spray chamber with Peltier cooling and a SeaSpray® nebulizer. Since H_3BO_3 was used to mask remaining HF during digestion, the application of the expensive "inert" (HF resistant) sample introduction kit was not required. As a result, the standard glass/quartz sample introduction system was used.

All experiments were carried out in a routine analytical laboratory and not under 'clean room' conditions. Instrument operating conditions are summarized in Table 1, including iCRC modes using helium and hydrogen gases to remove polyatomic interferences on the first-row transition metals.

Table 1: Instrument settings – PlasmaQuant MS

Parameter	Specification	
Plasma Gas Flow	9.0 L/min	
Auxiliary Gas Flow	1.50 L/min	
Sheath Gas Flow	0.00 L/min	
Nebulizer Gas Flow	1.07 L/min	
Sampling depth	6.0 mm	
Plasma RF Power	1.40 kW	
Rump Rate	20 rpm – black/black PVC pump tubing (< 1mL/min)	
Stabilization delay	10 s	
iCRC Gas Setting	He 120 mL/min	^{31}P
	He 150 mL/min	^{27}Al, ^{44}Ca, ^{51}V, ^{52}Cr, ^{55}Mn, ^{59}Co, ^{60}Ni, ^{65}Cu, ^{66}Zn
	No Gas	^{7}Li, ^{9}Be, ^{23}Na, ^{24}Mg, ^{39}K, ^{86}Sr, ^{98}Mo, ^{107}Ag, ^{111}Cd, ^{121}Sb, ^{133}Cs, ^{137}Ba, ^{201}Hg, ^{205}Tl, $^{206+207+208}$Pb
	H_2 150 mL/min	^{57}Fe, ^{75}As, ^{78}Se
Dwell Time	10 ms (No Gas) and 30 ms (iCRC)	
Scan per Replicate	20 (peak hopping, 1 pt/peak)	
No. of replicates	3	
Sample uptake time	0 s – ASXPress Plus Sample Introduction system used	
Internal Standards	^{103}Rh and ^{193}Ir at 25 µg/L, interpolate correction	

Samples and reagents

For digestion purposes the following high purity reagents were used:

- Deionized water (> 18.2 MΩ/cm, Millipore MiliQ)
- Nitric acid sub-boiled 69% (AnalytikJena GmbH+Co. KG)
- Hydrochloric acid Supra quality 35% (ROTIPURAN® Supra)
- Hydrofluoric acid Supra quality 48% (ROTIPURAN® Supra)
- Boric acid 99.9999% (Supelco Suprapur®)

Sample preparation

In total, six Certified Reference Materials of soil and sediments were prepared in triplicate for method validation purpose. Two from the National Institute of Standards & Technology, Gaithersburg, MD 20899, USA (NIST 1646a and NIST 2711a); two from the China National Analysis Center for Iron and Steel, Beijing, China, (NCS DC 73319a and NCS DC 73325), one from Supelco® Laramie, WY 82070, USA (SQC001) and one from nsi lab solutions, Raleigh, NC 27617, USA (SQCI-001). Each solid sample was weighed into the pre-cleaned speedwave XPERT digestion system vessel and mixed with the reagent(s) according to Table 2.

Table 2: Digestion methods parameters used by speedwave XPERT microwave digestion system

Parameter	Specification
Sample amount	0.1 g dried and sieved
H_2O	Few drops
HCl	4 mL
HNO_3	8 mL
HF	1 mL
H_2O_2	1 mL
Vessel	PM60
Temp. / Ramp / Hold	200 °C / 15 min / 20 min at 1200 W
Cooling / Time	Room / 30 min
Boric acid	10 mL of 4% (w/v)
Temp. / Ramp / Hold	170 °C / 25 min / 20 min at 1000 W
Cooling / Time	Room / 30 min
Final volume	Fill up to 50 mL with 0.5 mol/L HNO_3
Centrifuged	3500 rpm / 5 min
ICP-MS prior dilution	1:2 with 1% (v/v) HNO_3

The solutions were made up to 50 mL with 0.5 mol/L HNO_3 following the two-step digestion. They were then diluted twofold with DI H_2O before undergoing ICP-MS analysis. The final acid concentrations were as follows: 8% (v/v) HNO_3, 4% (v/v) HCl, 1% (v/v) H_2O_2, and 0.4% (w/v) H_3BO_3. The 2% (v/v) HF included in the digestion mix was neutralized by the addition of H_3BO_3. The concentration of total dissolved solids (TDS) in the final digest analyzed, was approximately 0.17% (1700 ppm), which included sediment matrix

(~0.1%) and boron (0.07%). It's worth noting that samples containing up to 0.3% TDS are easily within the limit that the PlasmaQuant MS can handle routinely, without requiring further aerosol dilution.

Each soil/sediment CRM was also spiked after the two-step microwave digestion procedure. The final concentration of each spiked element ranged from 0.25 up to 250 µg/L depending on the expected concentration of each element.

Calibration

Standard solutions for external calibration were matrix-matched using 8% (v/v) HNO_3, 4% (v/v) HCl and 0.4% (w/v) H_3BO_3 by appropriate dilution in 50 mL VWR® Metal-Free Centrifuge Tubes. Standard multielemental solution containing Li, Be, V, Cr, Mn, Co, Ni, Cu, Zn, As, Se, Sr, Ag, Cd, Cs, Ba, Tl and Pb (10 mg/L, 10% HNO_3, traces of HF, Periodic table mix 1 for ICP, 33 elements, TraceCERT®, Supelco®), single element solutions for Mo, Sb and Hg (CertiPUR® 1000 mg/L, in 2−3% HNO_3), and single element solution for Na, Mg, Al, P, K, Ca and Fe (CertiPUR® 10 000 mg/L, in 2−3% HNO_3) were used to prepare

five-point concentration curves as follows:
- 0.01 up to 2.5 µg/L for Hg;
- 0.01 up to 50 µg/L for Ag;
- 0.5 up to 125 µg/L for Mo;
- 0.1 up to 250 µg/L for Li, Be, V, Cr, Mn, Co, Ni, Cu, Zn, As, Se, Sr, Cd, Cs, Ba, Tl, and Pb;
- 1 up to 250 µg/L for Sb;
- 2 up to 450 µg/L for Al;
- 10 up to 1250 µg/L for Na, Mg, and P;
- 20 up to 2250 µg/L for K, Ca, and Fe.

Excellent linearity was achieved for major elements and trace elements, as indicated by the four representative calibration curves shown in Figure 1. In all cases, the R value exceeded the method requirement and was greater than 0.9997.
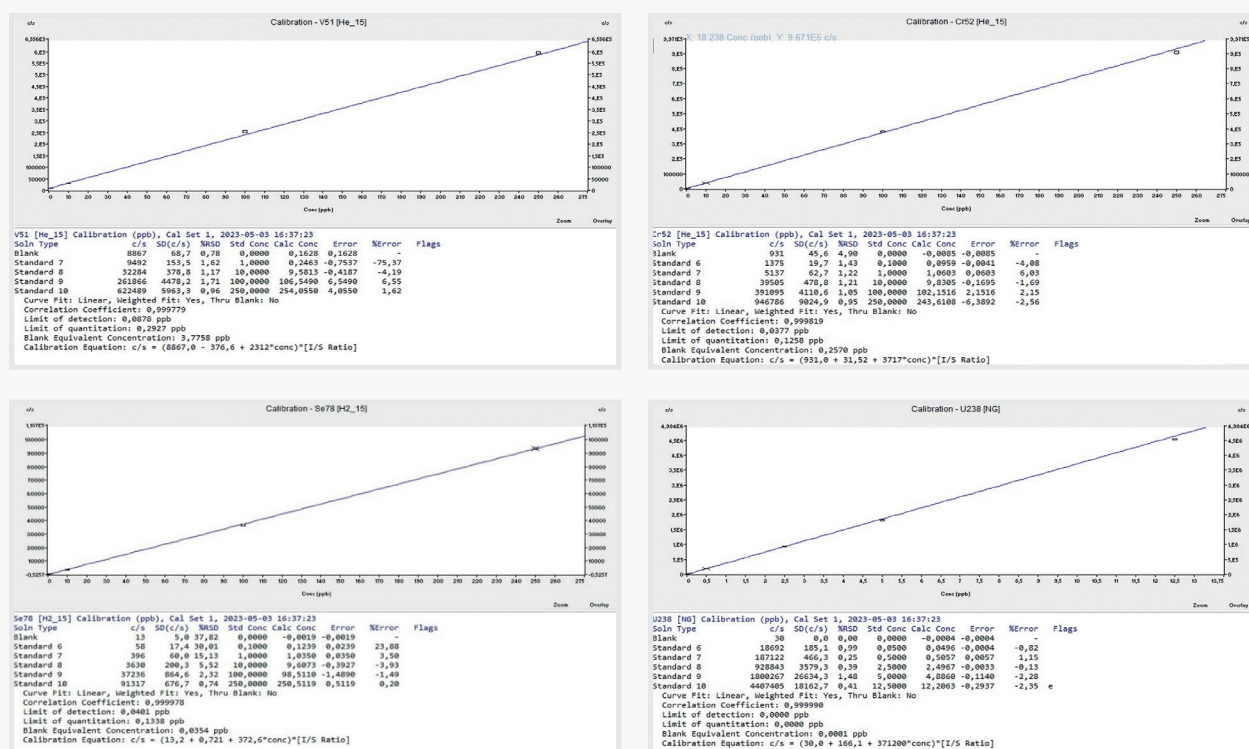

Figure 1: Calibration graphs for some investigated major and minor elements

Evaluation

According to the expected interferences on certain mass/charge ratios, different isotopes were measured by using collision gas (He) or a reactive gas (H_2) utilizing the iCRC technology. Besides the two available gases, PlasmaQuant MS allows the users to enhance analytes signals after collisions with the reaction gas took place on iCRC through the Boost patented technology applied on skimmer cone, which was not needed in this application. Therefore, for each measurement, and to achieve the best performance in terms of precision and accuracy, four

condition sets (Helium 120 and 150 mL/min, No Gas and Hydrogen 150 mL/min) were selected with the respective isotopes in the appropriate set (see Table 3). Within one measurement, all condition sets were executed with switching times of 10 seconds. For data recording, three average values were calculated from twenty scans each, which were used for the calculation of one average value including standard deviation. The total sampling time, including rinsing and sample uptake, was approximately three minutes per sample.

Table 3: Expected interferences and used iCRC gases

Isotope	Expected interferences	iCRC mode	Isotope	Expected interferences	iCRC mode
^{7}Li	-	No Gas	^{65}Cu	^{49}Ti^{16}O; ^{25}Mg^{40}Ar	He_150
^{9}Be	-	No Gas	^{66}Zn	^{26}Mg^{40}Ar; ^{31}P^{35}Cl; ^{50}Ti^{16}O; ^{40}Ar^{26}Mg	He_150
^{23}Na	-	No Gas	^{75}As	^{40}Ar^{35}Cl; ^{40}Ca^{35}Cl	H$_2$_150
^{24}Mg	^{12}C^{12}C (Negligible)	No Gas	^{78}Se	^{62}Ni^{16}O; ^{40}Ar^{38}Ar; ^{64}Ni^{14}N; ^{64}Zn^{14}N; ^{43}Ca^{35}Cl	H$_2$_150
^{27}Al	^{11}B^{16}O	He_150	^{86}Sr	^{70}Zn^{16}O; ^{70}Ge^{16}O; ^{75}As^{11}B (Negligible)	No Gas
^{31}P	^{15}N^{16}O	He_120	^{98}Mo	^{58}Fe^{40}Ar (Negligible)	No Gas
^{39}K	^{23}Na^{16}O (Negligible)	No Gas	^{107}Ag	^{91}Zr^{16}O; ^{67}Zn^{40}Ar (Negligible)	No Gas
^{44}Ca	^{28}Si^{16}O	He_150	^{111}Cd	^{95}Mo^{16}O; ^{97}Mo^{14}N; ^{71}Ga^{40}Ar (Negligible)	No Gas
^{51}V	^{35}Cl^{16}O	He_150	^{121}Sb	^{105}Pd^{16}O; ^{81}Br^{40}Ar (Negligible)	No Gas
^{52}Cr	^{40}Ar^{12}C; ^{35}Cl^{16}O^{1}H	He_150	^{133}Cs	^{117}Sn^{16}O; ^{119}Sn^{14}N; ^{93}Nb^{40}Ar (Negligible)	No Gas
^{55}Mn	^{39}K^{16}O	He_150	^{137}Ba	^{121}Sb^{16}O; ^{123}Sb^{14}N; ^{97}Mo^{40}Ar (Negligible)	No Gas
^{57}Fe	^{40}Ar^{16}O^{1}H; ^{41}K^{16}O; ^{43}Ca^{14}N	H$_2$_150	^{201}Hg	^{185}Re^{16}O; ^{184}W^{16}O^{1}H; ^{161}Dy^{40}Ar (Negligible)	No Gas
^{59}Co	^{58}Fe^{1}H; ^{43}Ca^{16}O; ^{24}Mg^{35}Cl	He_150	^{205}Tl	^{165}Ho^{40}Ar; ^{191}Ir^{14}N (Negligible)	No Gas
^{60}Ni	^{44}Ca^{16}O; ^{46}Ca^{14}O	He_150	$^{206+207+208}$Pb	-	No Gas

Results and Discussion

Digested blank solutions and method detection limits

Digested blank solutions were prepared in the same way as sample solutions; equal volumes of acid mixture, which have been used for sample digestion, and the same microwave-assisted digestion program was applied for the soil samples digestion. The measured concentrations of the elements in the procedural blank were subtracted from the concentration values measured in the soil CRM solutions.

The method detection limits (MDL) shown in Table 4 were determined by running a full calibration and were calculated as three times the standard deviation of the calibration blank's measured intensity, divided by the slope (3σ) and multiplied by the dilution factor (1000).

Table 4: MDL of metals in soil samples obtained by PQMS after microwave-assisted digestion of samples in closed-vessels

Element	MDL (mg/kg)	Element	MDL (mg/kg)	Element	MDL (mg/kg)	Element	MDL (mg/kg)
Li	0.05	Cu	0.03	Cr	0.04	Cs	0.001
Be	0.003	Zn	0.12	Mn	0.09	Ba	0.01
Na	25	As	0.20	Fe	11	Hg	0.004
Mg	0.01	Se	0.04	Co	0.003	Tl	0.001
Al	0.37	Sr	0.16	Ni	0.06	Pb	0.002
P	46	Mo	0.002				
K	11	Ag	0.01				
Ca	5.2	Cd	0.001				
V	0.09	Sb	0.01				

Spike recoveries, CRM recoveries and Precision

Tables 5a, 5b, 5c, 5d, 5e and 5f show the results of 28 major and trace elements determined in the six Certified Reference Materials tested, including information of spike recoveries, concentration mean recoveries obtained compared to the certified values and the precision. The spike level of some elements/CRMs were not high enough compared to the concentrations in the un-spiked samples and are not shown. The overall spike recoveries were within 75 to 128% excluding two values due to potential contamination in the NIST 2711a (K – 135% and Na – 160%). The mean concentration values of three digested replicates were in good agreement compared to the certified ones with overall recoveries of 79 to 135% and RSDs < 10% for the majority of the results, despite potential contamination or even a heterogeneous CRM sample (SQCI-NSI).

Table 5a: Metals and semi-metals concentration, recoveries, precision, and spike recoveries achieved in the NIST 1646a (sediment) after microwave-assisted digestion (n=3)

Isotope	Certified value (mg/kg)	Conc. Found (mg/kg)	Recovery (%)	RSD (%)	Spike Recovery (%)
Li	18*	22	122	6.9	105
Be	< 1*	0.79	-	2.5	100
Na	7410 ± 170	7616	103	1.4	111
Mg	3880 ± 90	4006	103	1.7	120
Al	22970 ± 180	22506	98	3.3	-
P	270 ± 10	302	112	5.7	105
K	8640 ± 160	8939	103	0.8	96
Ca	5190 ± 200	4986	96	3.5	100
V	44.8 ± 0.8	44	97	2.4	101
Cr	40.9 ± 1.9	34	84	8.7	100
Mn	234.5 ± 2.8	237	101	3.2	103
Fe	20080 ± 390	21028	105	4.0	117
Co	5*	5.0	99	11.0	99
Ni	23*	27	115	9.4	99
Cu	10 ± 0.3	11.7	117	7.3	99
Zn	48.9 ± 1.6	55	113	3.6	99
As	6.2 ± 0.2	6.54	105	0.9	97
Se	0.19 ± 0.03	0.19	101	12.7	99
Sr	68*	69	101	4.6	95
Mo	1.8*	1.974	110	0.2	94
Ag	< 0.3*	0.20	-	9.1	102
Cd	0.15 ± 0.01	0.16	104	3.7	100
Sb	0.3*	0.32	105	5.2	95
Cs	-	1.23	-	3.9	104
Ba	210*	195	93	7.2	98
Hg	0.04*	0.049	123	12.5	102
Tl	< 0.5*	0.207	-	2.7	107
Pb	11.7 ± 1.2	11.0	94	4.8	99

*Noncertified values

Table 5b: Metals and semi-metals concentration, recoveries, precision, and spike recoveries achieved in the NIST 2711a (Montana II Soil) after microwave-assisted digestion (n=3)

Isotope	Certified value (mg/kg)	Conc. Found (mg/kg)	Recovery (%)	RSD (%)	Spike Recovery (%)
Li	-	32.0	-	2.1	104
Be	-	3.0	-	11.0	102
Na	12000 ± 100	12478	104	1.1	160**
Mg	10700 ± 600	12574	118	0.7	122
Al	67200 ± 600	68643	102	8.9	-
P	842 ± 11	867	103	8.0	96
K	25300 ± 1000	26051	103	0.8	135**
Ca	24200 ± 600	22593	93	7.5	100
V	81 ± 6	82	101	9.1	101
Cr	52 ± 3	47	90	4.3	100
Mn	675 ± 18	691	102	5.7	105
Fe	28200 ± 400	30969	110	5.5	84
Co	9.9 ± 0.2	11	109	9.5	100
Ni	22 ± 1	22.1	100	0.5	101
Cu	140 ± 2	139	100	4.2	98
Zn	414 ± 11	422	102	3.6	92
As	107 ± 5	106.4	99	2.8	98
Se	2	2.02	101	3.2	102
Sr	242 ± 10	233	96	2.8	104
Mo	-	2.025	-	0.04	95
Ag	6*	5.5	92	4.8	102
Cd	54 ± 1	53.2	99	1.2	100
Sb	24 ± 1	21.4	89	2.6	95
Cs	6.7 ± 0.2	6.6	98	3.4	102
Ba	730 ± 15	744	102	1.5	105
Hg	7.4 ± 0.2	7.13	96	0.6	101
Tl	3*	2.70	90	3.0	107
Pb	1400 ± 10	1440	103	5.1	99

*Noncertified
** potential contamination

Table 5c: Metals and semi-metals concentration, recoveries, precision, and spike recoveries achieved in the NCS DC 73319a (Soil) after microwave-assisted digestion (n=3)

Isotope	Certified value (mg/kg)	Conc. Found (mg/kg)	Recovery (%)	RSD (%)	Spike Recovery (%)
Li	28 ± 2	37.9	135	10.2	104
Be	3.3 ± 0.3	3.74	113	1.7	105
Na	12240 ± 519	12675	104	1.8	106
Mg	7056 ± 241	7564	107	8.7	-
Al	68378 ± 1111	67157	98	3.0	-
P	2300 ± 200	2226	97	8.0	80
K	23659 ± 664	24428	103	1.4	76
Ca	19868 ± 786	18780	95	7.1	-
V	61 ± 4	62.3	102	0.4	99
Cr	44 ± 3	43.0	98	0.5	99
Mn	1310 ± 60	1324	101	3.4	78
Fe	30846 ± 1399	31200	101	4.5	-
Co	10.3 ± 0.6	10	101	10.0	100
Ni	16.9 ± 1.5	18.2	108	3.4	98
Cu	42 ± 5	51	122	4.2	95
Zn	475 ± 30	496	104	5.2	118
As	33 ± 3	36.0	109	2.7	100
Se	0.22*	0.27	123	16.6	102
Sr	192 ± 9	191	99	3.9	91
Mo	2 ± 0.2	2.34	117	2.0	94
Ag	0.81 ± 0.04	1.37***	-	3.3	102
Cd	2.5 ± 0.2	2.61	104	2.1	99
Sb	2.4 ± 0.3	2.79	116	1.0	95
Cs	7.2 ± 0.5	6.7	94	2.8	105
Ba	700 ± 40	679	97	2.3	96
Hg	0.31 ± 0.03	0.39	126	3.5	103
Tl	1.2 ± 0.1	1.14	95	3.6	102
Pb	339 ± 12	348	103	2.3	102

*Noncertified
*** Contaminated

Table 5d: Metals and semi-metals concentration, recoveries, precision, and spike recoveries achieved in the NCS DC 73325 (Soil) after microwave-assisted digestion (n=3)

Isotope	Certified value (mg/kg)	Conc. Found (mg/kg)	Recovery (%)	RSD (%)	Spike Recovery (%)
Li	19.5 ± 0.9	21.9	112	3.6	95
Be	2.8 ± 0.6	2.5	91	4.2	100
Na	593.5 ± 148	468	79	0.3	91
Mg	1568 ± 181	1341	86	3.0	98
Al	154896 ± 1800	138428	89	5.0	-
P	1150 ± 39	1153	100	11.3	97
K	1660 ± 166	1423	86	1.2	88
Ca	1144 ± 143	988	86	7.4	97
V	245 ± 21	244	99	3.5	97
Cr	410 ± 23	408	99	2.7	101
Mn	1780 ± 113	1811	102	2.2	105
Fe	131216 ± 2308	127990	98	1.8	114
Co	97 ± 6	99	102	3.7	96
Ni	276 ± 15	281	102	1.8	99
Cu	97 ± 6	97	100	2.9	95
Zn	142 ± 11	147	104	3.4	95
As	4.8 ± 1.3	7.02***	-	0.6	99
Se	0.32 ± 0.05	0.35	108	8.1	100
Sr	26 ± 4	27	105	5.5	94
Mo	2.9 ± 0.3	3.15	109	2.1	93
Ag	0.057 ± 0.011	0.96***	-	9.5	101
Cd	0.08 ± 0.02	0.138	172	1.5	100
Sb	0.42 ± 0.09	0.47	112	7.3	96
Cs	2.7 ± 0.8	2.40	89	2.8	105
Ba	180 ± 27	173	96	5.1	92
Hg	0.061 ± 0.006	0.095***	-	5.8	97
Tl	0.21 ± 0.06	0.187	89	2.8	97
Pb	14 ± 3	12.99	93	0.1	91

*** Contaminated

Table 5e: Metals and semi-metals concentration, recoveries, precision, and spike recoveries achieved in the SQCI (NSI) after microwave-assisted digestion (n=3)

Isotope	Certified value (mg/kg)	Conc. Found (mg/kg)	Recovery (%)	RSD (%)	Spike Recovery (%)
Li	711 ± 6.62	741	104	5.2	104
Be	190 ± 1.77	189	99	4.5	102
Na	3640 ± 33.9	3682	101	5.1	127
Mg	5260 ± 49	5975	114	8.5	102
Al	3620 ± 33.7 (2550–8730)[a]	18140*	-	21.0*	-
P	-	-	-	-	95
K	6040 ± 56.2	6881	114	4.2	104
Ca	3320 ± 30.9	3185	96	4.5	105
V	118 ± 1.1	123	104	7.8	97
Cr	290 ± 2.7	322	111	6.0	99
Mn	1920 ± 17.9	1860	97	2.2	105
Fe	19100 ± 178	18849	99	2.4	76
Co	165 ± 1.54	153	93	1.9	96
Ni	475 ± 4.42	475	100	3.8	96
Cu	358 ± 3.33	306	85	3.3	97
Zn	610 ± 5.68	587	96	4.2	90
As	320 ± 2.98	296	92	2.7	99
Se	310 ± 2.89	300	97	5.8	92
Sr	105 ± 0.978	122	116	5.1	104
Mo	105 ± 0.978	102	98	4.5	92
Ag	95 ± 0.884	90	94	2.5	102
Cd	70 ± 0.652	66	94	2.7	101
Sb	245 ± 2.28	243	99	3.9	106
Cs	-	0.90	-	3.1	105
Ba	399 ± 3.71	428	107	5.2	108
Hg	16 ± 0.149 (3.96–17.6)[a]	6.2*	-	6.1	86
Tl	380 ± 3.54	390	103	7.0	119
Pb	123 ± 1.15	125	102	8.7	100

*Heterogeneous sample/contamination;
[a] acceptance limits

Table 5f: Metals and semi-metals concentration, recoveries, precision, and spike recoveries achieved in the SQCI (Supelco) after microwave-assisted digestion (n=3)

Isotope	Certified value (mg/kg)	Conc. Found (mg/kg)	Recovery (%)	RSD (%)	Spike Recovery (%)
Li	123 ± 3	141	115	5.2	104
Be	72 ± 3	86	119	2.5	97
Na	1690 ± 90	1913	113	5.6	92
Mg	1840 ± 34	2192	119	0.2	96
Al	3085 ± 63	3366	109	1.1	128
P	-	146	-	4.0	95
K	4530 ± 133	5065	112	0.3	83
Ca	2650 ± 52	2535	96	2.2	95
V	95 ± 3	90	95	5.3	100
Cr	159 ± 4	159	100	4.9	101
Mn	435 ± 14	439	101	8.0	103
Fe	5752 ± 84	5841	102	10.5	91
Co	257 ± 4	249.6	97	0.2	100
Ni	183 ± 4	171	93	1.2	98
Cu	151 ± 3	152	100	2.0	99
Zn	375 ± 5	354	94	8.4	100
As	65 ± 2	72.8	112	0.8	96
Se	56 ± 1	60	108	3.0	101
Sr	116 ± 8	136	117	2.5	97
Mo	155 ± 4	177	114	4.8	115
Ag	46 ± 5	52	112	3.9	107
Cd	118 ± 3	134	113	2.6	100
Sb	59 ± 6	119***	-	8.0	112
Cs	-	0.008	-	10.6	103
Ba	626 ± 33	762	122	2.4	126
Hg	28 ± 1	26.7	95	2.7	75
Tl	83 ± 3	103	124	2.2	104
Pb	230 ± 5	250	109	3.6	97

*** Contaminated

Internal Standards stability

A total of 145 solutions were analyzed over a period of 7.5 hours. For this study, the isotopes [103]Rh and [193]Ir were used from the list of internal standard elements and all the ISTD recovery measurements, as shown in Figure 2, were within ± 30%. Most of the ISTD recoveries were within 20% which revealed a high matrix tolerance of the PlasmaQuant MS without the need of aerosol dilution approach. This resulted in minimal variability in the ISTD signals from sample to sample throughout the run, with no significant matrix deposition on the interface.
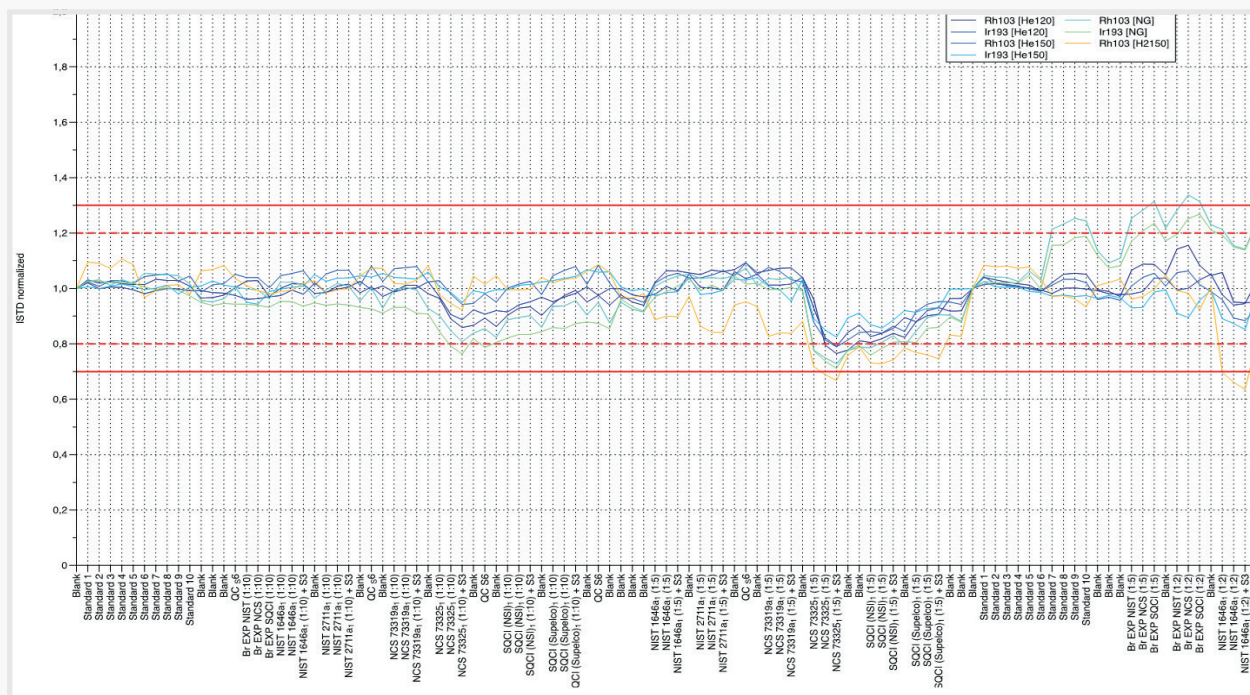
Figure 2: Internal standards stability during the analysis of a total of 109 solutions measured over a 7.5 hours run. ISTD for all samples have been normalized to the calibration blank.

Summary

Based on the CRM recoveries obtained in this study, it can be concluded that the method demonstrates good accuracy in analyzing high and trace concentration elements in the same dilution without the need for aerosol dilution feature.

The optimized microwave digestion sample preparation method using closed vessels during the digestion process proved to be an effective procedure for soil and sediment digestion, ensuring accurate measurement of all elements, including volatile elements such as Hg, As, Sb, Se, Cd, and even Zn and Pb, which showed good recoveries.
This highlights the robustness of the method and its ability to accurately quantify a wide range of analyte concentrations typically found in soils and sediments, covering the HJ 766 and ISO 14689 elements.

Figure 3: PlasmQuant MS

Liquid dilution was used, despite the potential risks of contamination or human errors. When controlled and minimized, the method can provide accurate and precise data. The method detection limits (MDLs) were not compromised since the PlasmaQuant MS can provide up to 5 times more sensitivity than any other Single Quad in the market.

In summary, the PlasmaQuant MS, along with the optimized sample preparation method, enables reliable routine analysis of targeted environmental samples according to the HJ 766 and ISO 14689 standards. The system offers quick and easy analysis, ensuring accurate quantification of a wide range of elements and elemental concentrations.

Recommended device configuration

Table 6: Overview of devices, accessories, and consumables

Article	Article number	Description
PlasmaQuant MS - high sensitive, robust and reliable ICP-MS Instrument	818-08010-2	PlasmaQuant MS is optimized for the analysis of tough samples such as seawater, soil, sludge among others.
speedwave XPERT	819-5005000-2	Universally applicable microwave digestion system for the preparation of organic and inorganic sample materials that impress with its reliability, safety, and economy.
Teledyne-Cetac ASX-560 autosampler for ICP-OES and ICP-MS	810-88015-0	Autosampler designed to simplify analysis and save time
Cetac ASXPress Plus for PQMS	810-88017-0	The ASXpress plus Rapid Sample Introduction Accessory reduces time required for autosampler movement, sample uptake, stabilization, and rinse operations, thereby reducing sample run times significantly

References

[1]   HJ 766-2015: Solid Waste. Determination of metals. Inductively coupled plasma mass spectrometry (ICP-MS), National Environmental Protection Standard of the People's Republic of China
[2]   ISO 14869-3:2017: Soil Quality – Dissolution for the determination of total element content – Part 3: Dissolution with hydrofluoric, hydrochloric, and nitric acids using pressurized microwave technique.