

# Multi-element analysis of surface and waste waters using triple quadrupole ICP-MS with prepFAST autodilution

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## Goal

To demonstrate routine automated multi-element analysis of environmental samples using triple quadrupole ICP-MS.

## Introduction

The adverse effects of increased concentrations of heavy metals in the environment and their potential impact on human, animal and plant health are of key concern. Consequently, monitoring of the content of these elements in a variety of environmental matrices (ground and surface waters, drinking water, waste waters, soils and sediments) is

one of the most frequently performed analyses in the context of environmental legislation. Some of the most common guidelines for different regions are summarized in Table 1.

Elemental analysis of environmental waters usually requires a number of different analyses to cover the different elements depending on element chemistry, interferences and linear range. Ideally, a single technique that could measure all environmentally relevant elements from a single sample, based on an easy sample preparation procedure (e.g. dilution or digestion), and with potential for automated and unattended analysis is desirable.

**Table 1. Overview of applicable regulatory guidelines for the analysis of drinking waters and waste waters globally**

Regulation	Region	Applicable for	Comment
EPA method 200.8	USA	Drinking waters and waste waters	Use of collision cell is only approved for waste waters
EPA method 6020 B	USA	Solid waste	Use of collision cell technology approved
CEN/TS 17200:2018	Europe	Digests and eluates of construction products	Aqua regia and nitric acid digests
FprEN 16171:2015	Europe	Sludge, treated biowaste and soil	Use of collision cell or high resolution technology approved
EN ISO 17294	Europe	Drinking waters, surface waters, ground waters, waste waters	
GB3838-2002	China	Surface waters	
GB5749-2006		Drinking waters	
IS 10500:2012	India	Drinking waters	Mentions ICP-OES primarily, methods for bottled drinking water require ICP-MS for analysis

Following the introduction of triple quadrupole ICP-MS, the routine removal of all types of spectral interferences is now possible (including doubly charged ions interfering for example on arsenic and selenium). Used in combination with an online dilution system, this kind of instrumentation can offer the possibility of sensitive and robust, quantitative analysis for all relevant matrix and trace elements in a wide variety of environmental sample matrices. Triple quadrupole ICP-MS systems such as the Thermo Scientific™ iCAP™ TQ ICP-MS, allow the use of reactive gases following a mass filtration step in a quadrupole mass filter situated axially in front of the collision/reaction cell (CRC). Therefore, more confidence in the results for a number of critical elements (for example cadmium, mercury, arsenic, selenium, sulphur or silicon) can be achieved compared to the single quadrupole ICP-MS instruments present in most environmental laboratories. In combination with the use of an automated inline dilution system, such as the Elemental Scientific prepFAST, the iCAP TQ ICP-MS offers the potential to determine all relevant matrix and trace elements in a single analysis.

A feasibility study was carried out in collaboration with the Flemish reference laboratory for environmental monitoring (Vito), and Flanders Environment Agency (VMM) on 25 waste waters and 25 surface waters. The aim of this study was to evaluate the performance of the iCAP TQ ICP-MS for the quantitative multi-element determination of 68 elements in aqueous environmental samples, in accordance with the performance requirements included in the compendium for sampling, measurement and analysis of water (Compendium voor analyse van water, commonly abbreviated as WAC)<sup>1</sup>. The results obtained were verified using a combination of quality control standards and certified reference materials.

## Instrumentation

All measurements were performed using an iCAP TQ ICP-MS. The instrument was operated using the Thermo Scientific™ Qtegra™ Intelligent Scientific Data Solution™ (ISDS) Software and was initially optimized using the supplied tune solution to optimize the interface parameters for maximum sensitivity. Modes using reactive gases were also tuned using the supplied autotune procedures to determine optimum gas flows and potentials for the CRC and the analysing quadrupole. Typical operating conditions are summarized in Table 2.

**Table 2. Typical instrumental parameters**

Parameter	Value		
Nebulizer	PFA-ST nebulizer pumped at 40 rpm		
Spraychamber	Quartz cyclonic spraychamber cooled at 2.7 °C		
Injector	2.5 mm i.d., Quartz		
Interface	High Matrix (3.5 mm) insert, Ni cones		
RF power	1,550 W		
Nebulizer gas flow	1.00 L·min <sup>-1</sup>		
Interface	High Matrix		
<b>QCell settings</b>	<b>SQ-NA</b>	<b>SQ-KED</b>	<b>TQ-O<sub>2</sub></b>
Gas flow	N/A	4.7 mL·min <sup>-1</sup>	0.42 mL·min <sup>-1</sup>
CR bias	-2.0 V	-21 V	-7.2 V
Q3 bias	-1.0 V	-18 V	-12 V

The calibration of the system was carried out daily using a mixed multi-elemental standard solution. The concentration levels for the individual calibration standards were as shown in Table 3.

**Table 3. Concentration levels for calibration standards**

Element	Standard concentrations
Na	5, 25, 50 mg·L <sup>-1</sup>
Mg	1, 5, 10 mg·L <sup>-1</sup>
Si	0.5, 2.5, 5 mg·L <sup>-1</sup>
P	0.2, 1, 2 mg·L <sup>-1</sup>
S	2.5, 12.5, 25 mg·L <sup>-1</sup>
K	0.1, 5, 10 mg·L <sup>-1</sup>
All other elements	0, 10, 50, 100 µg·L <sup>-1</sup>
Ca	2, 10, 20 mg·L <sup>-1</sup>

The digested samples were automatically diluted 5 times by the prepFAST syringe-driven inline dilution system, by defining a prescriptive dilution factor 5 in the sample list. The internal standard (Rh 20 µg·L<sup>-1</sup> in 1% HNO<sub>3</sub>/5% butanol) was added on-line, again utilizing the prepFAST system.

In order to select the appropriate instrument settings (e.g. choice of reaction gas and the mass to be transmitted in each quadrupole (Q1 and Q3)), the Reaction Finder method development assistant was used. Reaction Finder allows analytical methods to be set up without prior detailed knowledge of potential reaction pathways caused by other components in the sample. It also determines the applied resolution setting for Q1 automatically (intelligent Mass Selection (IMS) vs.  $\leq 1$  amu). Further optimization of the method parameters, such as for example the use of different reactive gases for some analytes (e.g. NH<sub>3</sub> for the analysis of Ti and platinum group elements), can also be accomplished. For this work, different measurement modes were selected outside the default Reaction Finder settings and were automatically applied to scan all elements in each sample using a single aspiration.

- O<sub>2</sub> was used for the analysis of <sup>28</sup>Si, <sup>31</sup>P, <sup>32</sup>S, <sup>75</sup>As, <sup>80</sup>Se, <sup>111</sup>Cd and <sup>202</sup>Hg in TQ mode
- Single quadrupole no gas mode was selected for <sup>7</sup>Li, <sup>9</sup>Be and <sup>11</sup>B
- All other isotopes measured in single quadrupole KED mode

The performance characteristics included in this note are indicative and additional optimization of the multi-element method is necessary, including the use of multiple internal standards in addition to Rh (e.g. <sup>6</sup>Li, Sc, Ge, Ir).

### Sample preparation

All 50 samples (25 waste waters and 25 surface waters collected at different sites in the Flanders region of Belgium) were digested in accordance with NBN-EN-ISO 15587-1. In brief, 6 mL of HCl + 2 mL of HNO<sub>3</sub> were added to a 25 mL aliquot of each sample, and digested at 105 °C for 2 hours using a hot block digestion system. After cooling, the samples were diluted to a final volume of 50 mL with ultra-pure water.

The list of elements to be determined is summarized in Table 4, which contains the following three subcategories:

- NBN EN ISO 17294: 2016 “Water – Application of mass spectrometry with inductively coupled plasma – Part 2: Determination of selected elements including uranium isotopes”: 63 elements are included in this standard method
- Monitoring: 33 elements that are currently being monitored in the context of Flemish environmental monitoring
- List of priority substances: 23 elements for which a hazardous substance classification criterion (HS) has been defined in the Flemish Environmental Permitting Regulations (VLAREM). The hazardous substances classification criterion (GS) determines from which concentration a waste water must be regarded as “Industrial waste water with hazardous substances”. If the concentration of hazardous substances is higher than the GS classification criterion, an environmental permit with emission limit values can be issued per sector. In VLAREM II Appendix 2.3.1. (Basic environmental quality standards for surface water) a GS classification criterion has been defined for 23 elements

Table 4. List of elements to be determined

Element	EN ISO 17294	Monitoring	Priority substances limit [ $\mu\text{g}\cdot\text{L}^{-1}$ ]	Element	EN ISO 17294	Monitoring	Priority substances limit [ $\mu\text{g}\cdot\text{L}^{-1}$ ]
Ag	X	X	0.4	Nb			
Al	X	X		Nd	X		
As	X	X	0.5	Ni	X	X	30
Au	X			Os			
B	X	X	700	P	X	X	1,000
Ba	X	X	70	Pb	X	X	50
Be	X	X	0.1	Pd	X		
Bi	X			Pr	X		
Ca	X	X		Pt	X		
Cd	X	X	0.8	Rb	X		
Ce	X			Re	X		
Co	X	X	0.6	Rh	X		
Cr	X	X	50	Ru	X		
Cs	X			S		X	
Cu	X	X	50	Sb	X	X	100
Dy	X			Sc	X		
Er	X			Se	X	X	3
Eu				Si		X	
Fe	X	X		Sm	X		
Ga	X			Sn	X	X	40
Gd	X			Sr	X		
Ge	X			Ta			
Hf	X			Tb	X		
Hg	X	X	0.3	Te	X	X	100
Ho	X			Th	X		
In	X			Ti		X	100
Ir	X			Tl	X	X	0.2
K	X	X		Tm	X		
La	X			U	X	X	1
Li	X	X		V	X	X	5
Lu	X			W	X		
Mg	X	X		Y	X		
Mn	X	X		Yb	X		

Table 5 provides an overview of environmental quality standards and reporting limit requirements of elements in groundwater, drinking water, surface water and waste water as included in the Flemish Environmental Permitting Regulations. 30% of the standard value is currently used as a criterion for the maximum Limit of Quantification (LOQmax, i.e. reporting limit requirement).

**Table 5. Environmental quality standards and reporting limit requirements of elements in groundwater, drinking water, surface water and waste water as included in the Flemish Environmental Permitting Regulations**

Element	Concentration	Waste water	Surface water				Groundwater					Drinking water	
			Reference	2	3	4	5	6	7	8	9	10	11
Aluminium	[ $\mu\text{g}\cdot\text{L}^{-1}$ ]	100					200	60		20	20	200	60
Antimony	[ $\mu\text{g}\cdot\text{L}^{-1}$ ]	30	100	30	30	10			3	3	5	1.5	
Arsenic	[ $\mu\text{g}\cdot\text{L}^{-1}$ ]	5	5	1.5	2.5	20	6	10	3	3	10	3	
Barium	[ $\mu\text{g}\cdot\text{L}^{-1}$ ]	21	70	21	21	1,000			300	300			
Beryllium	[ $\mu\text{g}\cdot\text{L}^{-1}$ ]	1	0.1	0.03	0.5								
Boron	[ $\mu\text{g}\cdot\text{L}^{-1}$ ]	210	700	210	210	1,000	39		300	300	1,000	300	
Cadmium	[ $\mu\text{g}\cdot\text{L}^{-1}$ ]	0.8	0.8	0.24	0.4	5		2.5	0.75	0.75	5	1.5	
Cerium	[ $\mu\text{g}\cdot\text{L}^{-1}$ ]	100											
Chromium	[ $\mu\text{g}\cdot\text{L}^{-1}$ ]	15	50	15	15	50	12		15	15	50	15	
Phosphorous	[ $\mu\text{g}\cdot\text{L}^{-1}$ ]	300	140	42									
Iron	[ $\mu\text{g}\cdot\text{L}^{-1}$ ]	100				20,000	1100		6,000	6,000	200	60	
Cobalt	[ $\mu\text{g}\cdot\text{L}^{-1}$ ]	0.6	0.6	0.18	0.3					0.3			
Copper	[ $\mu\text{g}\cdot\text{L}^{-1}$ ]	15	50	15	15	100	5		30	30	2,000	600	
Mercury	[ $\mu\text{g}\cdot\text{L}^{-1}$ ]	0.15	0.3	0.09	0.15	1			0.3	0.3	1	0.3	
Iodine	[ $\mu\text{g}\cdot\text{L}^{-1}$ ]	15	50	15	15	20		10	3	3	10	3	
Manganese	[ $\mu\text{g}\cdot\text{L}^{-1}$ ]	20				1,000	70		300	300	50	15	
Molybdenum	[ $\mu\text{g}\cdot\text{L}^{-1}$ ]	105	350	105	105								
Nickel	[ $\mu\text{g}\cdot\text{L}^{-1}$ ]	9	30	9	9	40	6	20	6	6	20	6	
Selenium	[ $\mu\text{g}\cdot\text{L}^{-1}$ ]	5	3	0.9	3	10			3	3	10	3	
Tellurium	[ $\mu\text{g}\cdot\text{L}^{-1}$ ]	30	100	30	30								
Thallium	[ $\mu\text{g}\cdot\text{L}^{-1}$ ]	1	0.2	0.06	0.5								
Tin	[ $\mu\text{g}\cdot\text{L}^{-1}$ ]	12	40	12	12								
Titanium	[ $\mu\text{g}\cdot\text{L}^{-1}$ ]	30	100	30	30								
Uranium	[ $\mu\text{g}\cdot\text{L}^{-1}$ ]	0.5	1	0.3	0.3								
Vanadium	[ $\mu\text{g}\cdot\text{L}^{-1}$ ]	5	5	1.5	2.5								
Silver	[ $\mu\text{g}\cdot\text{L}^{-1}$ ]	1	0.4	0.12	0.4								
Zinc	[ $\text{mg}\cdot\text{L}^{-1}$ ]	60	200	60	60	500	17	260	78	78			
Calcium	[ $\text{mg}\cdot\text{L}^{-1}$ ]					270	51		15	15			
Magnesium	[ $\text{mg}\cdot\text{L}^{-1}$ ]					50	8.3		2.5	2.5			
Sodium	[ $\text{mg}\cdot\text{L}^{-1}$ ]					150	18		6	6	200	60	
Potassium	[ $\text{mg}\cdot\text{L}^{-1}$ ]					12	4.1	8	1.2	1.2			
Sulfate	[ $\text{mg}\cdot\text{L}^{-1}$ ] $\text{SO}_4^{2-}$	25			4.5	250	51		15	15	250	75	
Phosphate	[ $\text{mg}\cdot\text{L}^{-1}$ ] $\text{PO}_4^{3-}$	0.15				1.34	0.31		0.25	0.25			

## Results

A number of quality control samples as defined in the reference method for the analysis of water (WAC) and reference materials were analyzed to verify the method. Table 6 shows the recoveries and the relative standard deviations (RSD) for the LCS AW (laboratory control samples – waste water) control sample. These control samples were analyzed approximately every 10 samples in the measurement series (n = 6). In accordance with WAC / III / B / 011, the criterion for recovery is 80–120%. The iCAP TQ ICP-MS meets the requirement with analyte recoveries in the range 89–114%.

**Table 6. Recoveries and RSD for the waste water laboratory control standard**

Element	LCS AW		
	Expected value $\mu\text{g}\cdot\text{L}^{-1}$	Recovery %	RSD %
Ag	3	103	± 2
Al	300	105	± 2
As	15	99	± 1
B	300	99	± 5
Ba	30	104	± 2
Be	3	98	± 7
Ca	200	93	± 2
Cd	2.4	99	± 2
Ce	30	104	± 1
Co	1.8	103	± 6
Cr	30	105	± 3
Cu	30	103	± 2
Fe	300	104	± 1
K	10	97	± 2
Mg	20	95	± 3
Mn	30	101	± 4
Mo	30	97	± 2
Na	50	95	± 3
Ni	30	102	± 3
Pb	30	104	± 2
S	50	89	± 5
Sb	30	99	± 5
Se	15	100	± 3
Sn	30	104	± 2
Te	30	101	± 5
Ti	30	114	± 4
Tl	3	103	± 2
U	1.5	104	± 2
V	15	105	± 3
Zn	150	100	± 2

In addition, SRM 1640a (Trace Elements in Natural Water – NIST) and SPS-SW2 Batch 135 (elements in surface water – Spectrapure Standards) were also directly analysed. The recoveries and relative standard deviations are shown in Table 7. The combination of both reference materials allowed the determination of at least one value for the recovery for 48 out of the 68 elements included in this study, and hence allows the verification of the accuracy of the method. Generally, good agreement with certified values was found in both reference materials. In all cases, the measurement modes provided by the Reaction Finder method development assistant allowed complete removal of all the interferences. In some cases, manual addition of the TQ-O<sub>2</sub> mode and subsequent comparison of the results indicated improved interference removal over KED, for example to remove MO<sup>+</sup> interferences from unreactive analytes, such as e.g. <sup>95</sup>Mo<sup>16</sup>O<sup>+</sup> on <sup>111</sup>Cd<sup>+</sup>, or <sup>186</sup>W<sup>16</sup>O<sup>+</sup> on <sup>202</sup>Hg<sup>+</sup>. Matrix elements, such as sodium, potassium or calcium, were analyzed in He KED mode to reduce sensitivity and thereby extend the dynamic range of the method, rather than interference removal. In addition, different resolution settings for Q3 (limiting ion transmission for highly abundant elements) can further decrease count rate and hence further extend both dynamic range and detector lifetime.

Table 7. Recoveries and RSD for SRM 1640a and SPS-SW2 determined with iCAP TQ ICP-MS

Element	Units	SRM 1640a			SPS-SW 2		
		Reference value	Recovery %	RSD %	Reference value	Recovery %	RSD %
Ag	µg·L <sup>-1</sup>	8.08	100	± 1			
Al	µg·L <sup>-1</sup>	53	94	± 4	250	96	± 2
As	µg·L <sup>-1</sup>	8.07	95	± 1	50	96	± 1
B	µg·L <sup>-1</sup>	303	90	3	250	90	± 4
Ba	µg·L <sup>-1</sup>	151.8	100	± 2	250	99	± 2
Be	µg·L <sup>-1</sup>	3.03	90	± 4			
Ca	mg·L <sup>-1</sup>	5.6	108	± 2	10	111	± 2
Cd	µg·L <sup>-1</sup>	3.99	97	± 2	2.5	98	± 1
Ce	µg·L <sup>-1</sup>				2.5	103	± 2
Co	µg·L <sup>-1</sup>	20.24	97	± 2	10	98	± 2
Cr	µg·L <sup>-1</sup>	40.54	95	± 1	10	95	± 2
Cs	µg·L <sup>-1</sup>				10	103	± 3
Cu	µg·L <sup>-1</sup>	85.75	98	± 2	100	97	± 1
Dy	µg·L <sup>-1</sup>				2.5	103	± 3
Er	µg·L <sup>-1</sup>				2.5	103	± 2
Eu	µg·L <sup>-1</sup>				2.5	105	± 2
Fe	µg·L <sup>-1</sup>	36.8	97	± 1	100	97	± 2
Gd	µg·L <sup>-1</sup>				2.5	101	± 2
Ho	µg·L <sup>-1</sup>				2.5	103	± 2
K	mg·L <sup>-1</sup>	0.58	92	± 1	1	92	± 2
La	µg·L <sup>-1</sup>				2.5	104	± 2
Lu	µg·L <sup>-1</sup>				2.5	104	± 2
Mg	mg·L <sup>-1</sup>	1.06	89	± 2	2	87	± 2
Mn	µg·L <sup>-1</sup>	40.39	97	± 2	50	99	± 1
Mo	µg·L <sup>-1</sup>	45.6	92	± 1	50	91	± 1
Na	mg·L <sup>-1</sup>	3.14	85	± 2	10	87	± 2
Nd	µg·L <sup>-1</sup>				2.5	102	± 3
Ni	µg·L <sup>-1</sup>	25.32	96	± 3	50	97	± 2
P	mg·L <sup>-1</sup>				0.5	87	± 5
Pb	µg·L <sup>-1</sup>	12.1	104	± 2	25	102	± 2
Pr	µg·L <sup>-1</sup>				2.5	106	± 2
Rb	µg·L <sup>-1</sup>	1.198	102	± 12	50	101	± 2
S	mg·L <sup>-1</sup>				10	78	± 4
Sb	µg·L <sup>-1</sup>	5.1	77	± 9			
Sc	µg·L <sup>-1</sup>				2.5	85	± 6
Se	µg·L <sup>-1</sup>	20.13	98	± 2	10	99	± 3
Si	mg·L <sup>-1</sup>	5.21	86	± 3	5	86	± 3
Sm	µg·L <sup>-1</sup>				2.5	102	± 3
Sr	µg·L <sup>-1</sup>	126	101	± 2	250	102	± 2
Tb	µg·L <sup>-1</sup>				2.5	103	± 1
Th	µg·L <sup>-1</sup>				2.5	102	± 2
Tl	µg·L <sup>-1</sup>	1.619	103	± 1	2.5	102	± 2
Tm	µg·L <sup>-1</sup>				2.5	102	± 2
U	µg·L <sup>-1</sup>	25.35	101	± 3	2.5	101	± 1
V	µg·L <sup>-1</sup>	15.05	94	± 2	50	97	± 2
Y	µg·L <sup>-1</sup>				2.5	100	± 3
Yb	µg·L <sup>-1</sup>				2.5	103	± 2
Zn	µg·L <sup>-1</sup>	55.64	100	± 2	100	101	± 2

In order to investigate whether the proposed method is capable of achieving the required reporting limits, Limits of Quantification were calculated based on 6 \* standard deviation of a surface water control sample (LOQ SW). Table 8 summarizes the recoveries and the resulting LOQmax values. The control sample LOQ SW was analyzed every 10 samples in the measurement series (n = 6). The table also shows the environmental quality standards (EQS), it's subsequent 30% concentrations again correlated with the LOQmax for surface water (WAC / VI / A / 001 Performance characteristics, version 11/2018). The calculated LOQ values are shown and can be compared to the 30% EQS and LOQmax. The recovery is calculated based on the expected concentration levels of the surface water control sample (LOQ SW).

Based on the above summarized results, it can be stated that the iCAP TQ ICP-MS in multi-element mode generally meets the legally required LOQmax values for surface water samples. In cases where the limit was exceeded (for example cobalt), slight modifications to the method could significantly improve the results. In this particular case, very short dwell time were used, which may limit the relative signal stability and hence affect the LOQmax. Increasing the measurement times for cobalt (for example, by using longer dwell time) will lead to a lower LOQ value. The total measurement time for 68 elements, including stabilization time for the use of the different gases, can be limited to less than 3 minutes per sample, assuring highest throughput.

**Table 8. Recoveries and LOQ for LOQ SW control sample with iCAP TQ ICP-MS**

Element	EQS $\mu\text{g}\cdot\text{L}^{-1}$	30% EQS $\mu\text{g}\cdot\text{L}^{-1}$	LOQmax $\mu\text{g}\cdot\text{L}^{-1}$	LOQ SW $\mu\text{g}\cdot\text{L}^{-1}$	Results iCAP TQ ICP-MS	
					LOQ $\mu\text{g}\cdot\text{L}^{-1}$	Recovery %
Ag	0.4	0.12	0.4	0.4	0.19	110
As	5	1.5	2.5	2.5	0.38	105
B	700	210	210	100	10.3	111
Ba	70	21	21	10	0.93	117
Be	0.1	0.03	0.5	0.5	0.29	99
Cd	0.8	0.24	0.4	0.4	0.11	108
Co	0.6	0.18	0.3	0.3	0.32	113
Cr	50	15	15	10	2.12	110
Cu	50	15	15	10	2.17	110
Mo	350	105	105	10	0.93	108
Ni	30	9	9	10	1.91	112
Pb	50	15	15	10	0.50	113
Sb	100	30	30	3	7.92	342
Se	3	0.9	3	3	0.78	113
Sn	40	12	12	10	1.68	110
Te	100	30	30	10	8.79	105
Ti	100	30	30	10	10.8	140
Tl	0.2	0.06	0.5	0.5	0.10	114
U	1	0.3	0.3	0.3	0.09	112
V	5	1.5	2.5	2.5	1.69	107
Zn	200	60	60	50	6.18	108
				<b>mg·L<sup>-1</sup></b>		
Ca				100		96
K				5		100
Mg				10		99
Na				25		95
S				25		86

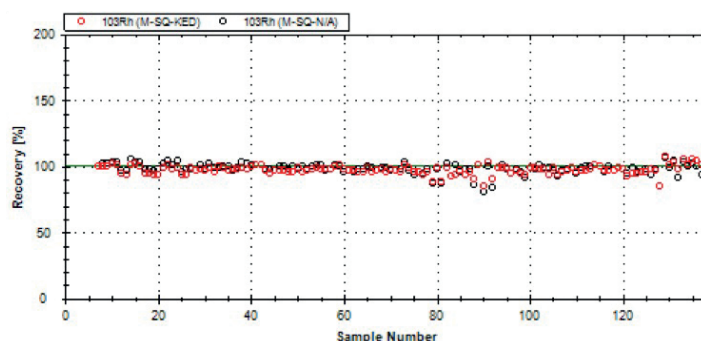


In addition, for all elements it can be stated that the iCAP TQ ICP-MS in multi-element mode met the 30% of the EQS requirement, with the exception of silver, beryllium and thallium. For beryllium and thallium this can largely be explained by the concentration level of the LOQ sample that is approximately more than a factor of 10 higher than 30% of the EQS. If the LOQ values would be determined at a concentration level similar to 30% of the EQS, a lower (and more realistic) picture of sensitivity and attainable quantification limits would be obtained. For silver, it is expected that with additional optimization of the instrument / method a LOQ value could also be obtained that is <30% of the EQS. The variation in the determination of Sb is expected to be due to the in sample stability of this element.

Overall, the data shows very good precision for the analysis of the surface water and waste water samples for the various elements, with a dynamic range of more than 8 orders of magnitude (concentration ranges from 0.01 to 1,000,000  $\mu\text{g}\cdot\text{L}^{-1}$ ).

In this study, only a single internal standard, rhodium, is used. Although the obtained results are generally in good agreement with certified values, the method can be improved significantly in terms of robustness and accuracy if multiple internal standards, covering the entire mass range, would be used. Based on the analysis of the different waste and surface waters, it would be relevant to consider common alternatives, such as lithium-6, scandium, germanium or iridium as additional internal standards, in particular for the determination of the lighter elements.

Ultimately, the robustness of the method was evaluated using the stability of the internal standard response as an indicator. Generally, considering the variability of the sample matrices in this study (surface waters and waste waters all run in the same sequence), some variability particularly in the concentration of major elements (especially iron) may cause internal standard variation, which may be critical in a high throughput laboratory. Most regulated methods for environmental waters allow for an internal standard recovery between 70–130%. Figure 1 shows the internal standard response for a complete sequence containing in total 130 samples, analysed over a period of approximately 7 hours.



**Figure 1. Internal standard stability during the measurement series (approx. 130 analyses)**

As can be seen from the plot, the internal standard recovery is well within the applicable limits, with variation between 80–110%. This points to the robust measurement conditions of the iCAP TQ ICP-MS and confirms the practicality of high throughput analysis.

## Conclusion

Triple quadrupole ICP-MS is a viable tool for the routine analysis of environmental water samples, such as ground water, surface water and waste water. The Reaction Finder method development assistant enables fast method development for interference free analysis even for inexperienced users. In combination with the fully integrated prepFAST in-line dilution system, automatic dilution of all samples effectively minimizes operator interaction and hence frees up valuable resources and helps to avoid interruption through human errors. Triple quadrupole technology can help to tackle difficult sample matrices, where significant contributions from matrix components or unexpected presence of other elements may lead to the formation of severe polyatomic interferences on the target analyte. In this work, the fast and accurate analysis of environmental water samples has been demonstrated. Excellent analyte recoveries, low LOQs and robust instrument performance make the iCAP TQ ICP-MS ideal for routine environmental applications.

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4. 30% of the standard value (see 2009/90 / EC)
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8. Vlarem II Appendix 2.4.1, minimum threshold value
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11. Drinking water guideline standard value
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