

ICP - Mass Spectrometry

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The Analysis of Drinking Waters by U.S. EPA Method 200.8 Using the NexION 300Q/350Q ICP-MS in Standard Mode

Introduction

Method 200.8 is a well-established method promulgated by the U.S. Environmental Protection Agency (EPA) for the analysis of ground waters, surface waters, drinking waters, and wastewaters by inductively coupled plasma mass spectrometry (ICP-MS). The method was first published in 1990 to support the National Primary Drinking Water Regulations (NPDWR), which specified

maximum contaminant levels (MCL) for 12 primary elemental contaminants in public water systems as part of the Safe Drinking Water Act (SDWA) of 1986. There have been many iterations of Method 200.8, including the addition of 9 secondary contaminants under the National Secondary Drinking Water Regulations (NSDWR). These 21 elements, along with suggested analytical masses, are shown in Table 1. The version in use today is Revision 5.4 of the Method, which was approved for drinking water in 1994 and became effective in January, 1995.³ In addition, Method 200.8 was also recommended in 1992 for the monitoring of wastewaters under the National Pollutant Discharge Elimination System (NPDES) permit program to control the discharge of pollutants into navigable water systems, as part of the amended Clean Water Act (CWA) of 1977.⁴ It was approved on a nation-wide basis for this matrix in 2007.

Application Note 1 of 3 – Why?

This is the first of a series of application notes describing the use of an innovative, next-generation ICP mass spectrometer – the NexION 300 – for the determination of 21 primary and secondary contaminants in drinking water samples using U.S. EPA Method 200.8. An overview of this technology will be given, and in particular, how the instrument's Universal Cell Technology™ (UCT) can be used in either the Standard mode, Collision mode using Kinetic Energy Discrimination (KED), or the Reaction mode (Dynamic Reaction Cell – DRC), depending on the severity of interferences presented by the sample matrices and the needs of the user.

The study is separated into three different application notes. Because collision/reaction cell technology is not currently permitted for the analysis of drinking water using Method 200.8, this application note focuses on the use of the NexION 300Q in the Standard mode using only the correction equations described in the Method to minimize matrix-based polyatomic interferences. In anticipation of the Method eventually allowing the use of collision/reaction cell technology for drinking water, the second application note (The Analysis of Drinking Waters by U.S. EPA Method 200.8 Using the NexION 300X ICP-MS in Standard and Collision Modes) describes a method using both the Standard and Collision (KED) modes with a NexION 300X,¹ while the third application note (The Analysis of Drinking Waters by U.S. EPA Method 200.8 Using the NexION 300D ICP-MS in Standard, Collision and Reaction Modes) provides an overview of how all three modes – Standard, Collision (KED) and Reaction (DRC) – can be utilized in one multielement method to achieve the optimum performance for this analysis, using a NexION 300D.²



Table 1. The complete list of elements defined in Method 200.8 with recommended analytical masses.

Analyte	Symbol	Recommended Mass (amu)
Beryllium	Be	9
Aluminum	Al	27
Vanadium	V	51
Chromium	Cr	52
Manganese	Mn	55
Cobalt	Co	59
Nickel	Ni	60
Copper	Cu	65
Zinc	Zn	66
Arsenic	As	75
Selenium	Se	82
Molybdenum	Mo	98
Silver	Ag	107
Cadmium	Cd	111
Antimony	Sb	121
Barium	Ba	137
Mercury	Hg	202
Thallium	Tl	205
Lead	Pb	206, 207, 208
Thorium	Th	232
Uranium	U	238
Sodium*	Na	23
Magnesium*	Mg	24
Potassium*	K	39
Calcium*	Ca	44
Iron*	Fe	54

Note: Elements marked with an asterisk () are included for information only.*

Several additional masses must be monitored, but not quantified, according to the U.S. EPA Method 200.8.³

Method 200.8 is a general analytical method that not only recommends procedures for selecting analyte masses, instrument tuning and calibration, and interference corrections, but also provides specific instructions concerning sample collection, preservation, and treatment. In addition, Method 200.8 also requires a specific set of quality assurance/control protocols to be run along with samples to ensure the quality of the data obtained. Method 200.8 is an exhaustive compendium of information which has been successfully used for the determination of trace elements in environmental samples for the past 17 years.

It is therefore important that new instrumentation demonstrate the ability to meet the requirements of existing and potential new methodology, through the initial demonstration of performance and on-going quality control procedure requirements. With this in mind, the NexION 300, a next-generation ICP-MS system, was evaluated for the analysis of drinking water samples, and in particular to get a better understanding of how it performs under the extreme demands of a high sample workload, which is typical of many laboratories carrying out environmental monitoring, both in the U.S. and around the world.

Instrumentation

For this study, the NexION® 300Q (PerkinElmer Inc., Shelton, CT) was used for the determination of all the elements defined in Method 200.8. The instrument has been described in the literature,⁵ but it's worth explaining that this version (300Q) does not have collision or reaction cell capabilities. It can be upgraded with a Universal Cell at a later date, but for the purpose of this application note, it was used in the Standard mode configuration. For those not familiar with the Universal Cell Technology, a brief description of the three modes of analysis is given in the call-out box on this page.

The objective of this particular application note was therefore to evaluate the capability of the instrument in the Standard mode for the analysis of drinking water, using the operating parameters shown in Table 2.

Table 2. ICP-MS instrumental operating conditions.

Component/Parameter	Type/Value/Mode
Nebulizer	Meinhard® glass microconcentric
Spray Chamber	Glass cyclonic
Triple Cone Interface Material	Nickel/Aluminum
Plasma Gas Flow	16.0 L/min
Auxiliary Gas Flow	1.2 L/min
Nebulizer Gas Flow	1.00 – 1.05 L/min
Sample Uptake Rate	250 µL/min
RF Power	1600 W
Integration Time	500 – 1500 ms
Replicates per Sample	3
Mode of Operation	Standard

NexION 300 ICP-MS Configurations and Capabilities

The NexION 300 ICP-MS is available in four different instrumental configurations. The 300Q version does not contain a collision/reaction cell and can only be used in the Standard mode, while the NexION 300X, 300D and 300S instruments all come with the Universal Cell installed. The 300X version has the ability to use one reaction/collision gas, while the 300D and 300S versions have the flexibility to use two reaction/collision gases. The 300S version is also intended for applications that require the highest performance and sensitivity, such as those found in the semiconductor industry. The NexION's three modes of operation include:

Standard Mode: The cell is actively vented to remove residual gases. This enables the instrument to be run in true Standard mode, with the cell conditions turned off. This means the instrument doesn't suffer from decreased sensitivity encountered with passively vented systems, which are forced to use KED even in Standard mode because of potential interferences, and as a result, are limited to using only a single collision gas. In this mode, the instrument is effectively a standard ICP-MS system with the collision cell turned off.

Collision Mode using KED: In this mode, the instrument offers conventional collision cell capability using Kinetic Energy Discrimination (KED). By using a non-reactive gas, such as helium, Collision mode removes many of the simple solvent- and argon-based polyatomic spectral interferences, which makes it ideal for semi-quantitative analyses, environmental sample monitoring, and the testing of unknown samples. It offers the ideal combination of performance and simplicity for straight-forward sample matrices.

Reaction Mode using DRC: Universally recognized as the technique that offers the ultimate detection capability, the instrument's true Reaction mode, using Dynamic Reaction Cell (DRC) technology, removes any and all interferences with little or no loss of analyte sensitivity. Unlike collision cells, DRC technology features a scanning quadrupole that removes all interferences created by reactions in the Universal Cell, which enables any reactive gas to be used. And by optimizing the cell's quadrupole conditions, only the element of interest is allowed to pass through to the analyzing quadrupole. It is well recognized that ion-molecule reaction chemistry offers the very best performance for the reduction of complex polyatomic spectral interference.⁶

200.8 Methodology

It is not the intent of this investigation to go into great detail about Method 200.8 and what is involved in carrying out the full analytical procedure, because an application note describing this method using previous ICP-MS technology has been available since 2008.⁷ However, before a suite of samples can be analyzed, a set of protocols must be followed to ensure the instrument is working at its optimum performance. A summary of Method 200.8's analytical protocol is shown below.

- Ignite plasma, allow 30 minutes to warm up
- Optimize instrument, as per instrument manufacturer's instructions
- Perform mass calibration check – adjust if change is > 0.1 amu
- Perform resolution check – adjust if > 0.75 amu at 5% peak height
- Calibrate using blank and standards
- Monitor all masses necessary for interference correction
- Screen new samples for relative levels and presence of internal standards
- Run instrument performance evaluation tests:
 - Instrument detection limits (IDLs)
 - Method detection limits (MDLs)
 - Linear dynamic range (LDR)
 - Spike recoveries
 - Long-term stability

- Run quality control samples (QCSs) or standard reference materials (SRMs) such as:
 - High-Purity Standards Trace Metals in Drinking Water (TMDW), both neat and diluted
 - NIST® 1643e
- Run unknown samples (e.g. local tap water)
- Review results of quality control samples for pass/fail criteria

It can be seen that once the instrument has been tuned and optimized for use, results from the performance evaluation tests and the successful analysis of the known reference samples are critical to achieve good accuracy and precision of unknown samples using Method 200.8. For this reason, it is important to compare the performance of the different modes of analysis, especially its polyatomic spectral interference reduction capabilities. Although not being presented here, this is the focus of the other two application notes in this series.^{1,2}

Calibration Standards

The multielement calibration standards made up in 1% nitric acid (HNO₃) used for the evaluation are shown in Table 3.

All tuning and monitoring of internal standards, together with the measurement of additional isotopes for interference correction purposes, were carried out exactly as defined in Method 200.8. Once completed, all the performance tests mentioned in the Method protocol shown earlier were carried out.

Table 3. Calibration standards.

Analytes	Standard 1 (µg/L)	Standard 2 (µg/L)	Standard 3 (µg/L)	Standard 4 (µg/L)
Be, Al, V, Cr, Mn, Co, Ni, Cu, Zn, As, Se, Mo, Ag, Cd, Sb, Ba, Tl, Pb, Th, U, Fe*	1	10	50	100
Hg	0.1	1	5	10
Na*, Mg*, K*, Ca*	100	1000	5000	10000
Internal Standards – ⁴⁵ Sc, ⁷² Ge, ¹⁰³ Rh, ¹¹⁵ In, ¹⁵⁹ Tb	20 – 200 µg/L			

Note: Elements marked with an asterisk () are not included in Method 200.8 – they are for information only.*

Performance Tests

A list of the most important performance tests described in Method 200.8, are listed below:

- Instrument detection limits (IDLs)
- Method detection limits (MDLs)
- Linear dynamic range (LDR)
- Analysis of standard reference materials (SRMs) or quality control samples (QCSs)
- Spike recoveries in typical samples
- Long-term stability of quality control samples

Each of these tests is described in greater detail below.

Instrument Detection Limits

Instrument detection limits (IDLs) were determined using the procedure referenced in Section 3.5 of Method 200.8, which defines the IDL as the concentration of analyte signal which is equal to three times the standard deviation of a series of ten replicate measurements of the calibration blank (1% HNO₃) signal at the selected analytical masses. Results should be in close approximation or better than estimated IDLs stated in Method 200.8.

Method Detection Limits

MDLs were determined using the protocol outlined in Section 9.2.4 of Method 200.8, which states that seven replicate aliquots of the spiked calibration blank (2% HNO₃) should be analyzed, and the MDLs calculated with the following equation:

$$\text{MDL} = (t) X (S)$$

Where:

t = Student's t value for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom

(t = 3.14 for seven replicates)

S = standard deviation (n-1 degrees of freedom) of the replicate measurements

Different spike concentrations were used to obtain the MDLs, since Method 200.8 specifies that MDL determinations should be made on spikes at levels 2 – 5 times the estimated MDL. Results should be equal to or better than estimated MDLs stated in Method 200.8.

Linear Dynamic Range

Linear calibration ranges were established for each isotope measured according to Section 9.2.2 of Method 200.8. After calibrating the instrument with a blank and the standards listed in Table 3, a series of standards of increasing concentration were analyzed. The LDR was defined as the concentration for which the results (in concentration units) were within ±10% of the true value of the standard. According to Method 200.8, the upper LDR limit should be an observed signal no more than 10% below the level extrapolated from the lower calibration standards.

Analysis of Standard Reference/Quality Control Samples

Accuracy and precision of the method was tested by analyzing two standard reference/quality control samples. For this test, the reference samples analyzed were High-Purity Standards Trace Metals in Drinking Water (TMDW), where spiked additions were analyzed in both the neat and a 10-fold dilution of the sample. In addition, NIST® 1643e "Trace Elements in Water" SRM was analyzed and compared to the certificate values.

Spike Recoveries

Spike recoveries were assessed by making spiked additions of 20 µg/L to a tap water sample from our laboratory. Spike recoveries should be within the Method 200.8 acceptance limits of ±30%.

Long-Term Stability of Quality Control Samples

The QC sample can also be used as the calibration check standard throughout the course of the run, which will allow the long-term stability of the instrument to be assessed over a "typical" working day of over nine hours.

Quality control procedures specified in Section 9.2.3 of Method 200.8 state that a quality control sample (QCS) must be run immediately after calibration and after every ten samples throughout the entire analytical batch of samples. The measured results for the QCS are compared to the true values to determine the percent recoveries. If QCS results are not within ±10% of the true value, the analysis should be terminated, the problem corrected, and the analysis repeated.

Results

The analysis was performed in Standard mode of the instrument with elemental equations mandated by the Method 200.8 and listed in Table 4.

Table 4. Isotopes measured and correction equations used for Method 200.8 using the NexION 300Q ICP-MS.

Analyte	Symbol	Isotopes Monitored (amu)	Correction Equations
Beryllium	Be	9	
Aluminum	Al	27	
Vanadium	V	51	- 3.127 * [ClO 53 - (0.113 * Cr 52)]
Chromium	Cr	52	
Manganese	Mn	55	
Cobalt	Co	59	
Nickel	Ni	60	
Copper	Cu	65	
Zinc	Zn	66	
Arsenic	As	75	- 3.127 * [ArCl 77 - (0.815 * Se 82)]
Selenium	Se	82	- 1.0078 * Kr 83
Molybdenum	Mo	98	- 0.1096 * Ru 101
Silver	Ag	107	
Cadmium	Cd	111	- 1.073 * [MoO 108 - (0.712 * Pd 106)]
Antimony	Sb	121	
Barium	Ba	137	
Mercury	Hg	202	
Thallium	Tl	205	
Lead	Pb	208	+ 1 * Pb 206 + 1 * Pb 207
Thorium	Th	232	
Uranium	U	238	
Sodium*	Na	23	
Magnesium*	Mg	24	
Potassium*	K	39	
Calcium*	Ca	44	
Iron*	Fe	54	- 0.0282 * Cr 52

Note: Elements marked with an asterisk (*) are included for information only.

The IDLs, MDLs, and LDR were measured and the results are shown in Table 5. All analytes in this table are well within the Method guidelines using integration times of 0.5 – 1.5 seconds per mass. The total measurement time was 1 min 30 sec per sample for all 21 analytes, 5 internal standards, plus the additional isotopes mandated by the Method.

Table 5. Method 200.8 instrument detection limits (IDLs) and method detection limits (MDLs), together with the linear dynamic range (LDR) using the NexION 300Q ICP-MS in Standard Mode.

Analyte	IDL (µg/L)	MDL Spike (µg/L)	MDL (µg/L)	Linear Range (mg/L)
Be	0.0005	0.002	0.0008	50
Al	0.01	0.05	0.03	100
V	0.006	0.01	0.01	100
Cr	0.02	0.05	0.02	100
Mn	0.002	0.01	0.004	100
Co	0.0005	0.002	0.0008	100
Ni	0.002	0.01	0.002	100
Cu	0.0007	0.002	0.0009	100
Zn	0.008	0.05	0.03	100
As	0.006	0.01	0.009	100
Se	0.03	0.05	0.03	50
Mo	0.0006	0.002	0.001	100
Ag	0.0006	0.002	0.001	100
Cd	0.003	0.01	0.003	100
Sb	0.0009	0.002	0.001	100
Ba	0.0009	0.002	0.002	50
Hg	0.0003	0.002	0.002	0.05
Tl	0.0002	0.002	0.0008	50
Pb	0.0003	0.002	0.0004	50
Th	0.0002	0.002	0.0006	20
U	0.0001	0.002	0.0004	20
Na*	0.03	0.05	0.03	50
Mg*	0.002	0.01	0.002	50
K*	0.8	5.0	1.0	30
Ca*	0.06	0.5	0.1	100
Fe*	1.3	5.0	2.6	100

Note: Elements marked with an asterisk (*) are included for information only.

The results from a spike recovery study of an undiluted and diluted drinking water standard reference material (High-Purity Standards TMDW) sample are shown in Table 6, while the analysis of the NIST® 1643e standard reference material is shown in Table 7. Finally, spike recovery data for a typical drinking water from our laboratory are shown in Table 8. It can be seen that almost all CRM values are within 10% of the certified values, and all spike recoveries are well within the Method 200.8 criteria of ±30%.

Table 6. Measured values and spike recoveries of trace elements in the High-Purity Standards Trace Metals in Drinking Water (TMDW) SRM using the NexION 300Q ICP-MS (data for the neat sample are shown on the left and a 10x dilution of the sample is shown on the right).

Analyte	High-Purity Standards Trace Metals in Drinking Water (Neat)			High-Purity Standards Trace Metals in Drinking Water (10x dilution)		
	Certified Value (µg/L)	Average Measured Value (µg/L)	Recovery (%)	Certified Value (µg/L)	Average Measured Value (µg/L)	Recovery (%)
Be	20.0	17.44	87.2	20.0	19.31	96.6
Al	120	114.3	95.2	120	114.9	95.7
V	30.0	28.37	94.6	30.0	26.62	88.7
Cr	20.0	19.40	97.0	20.0	18.88	94.4
Mn	40.0	39.51	98.8	40.0	37.03	92.6
Co	25.0	23.64	94.6	25.0	22.80	91.2
Ni	60.0	57.86	96.4	60.0	56.24	93.7
Cu	20.0	19.22	96.1	20.0	19.71	98.6
Zn	70.0	68.07	97.2	70.0	68.15	97.4
As	80.0	78.68	98.4	80.0	76.81	96.0
Se	10.0	9.66	96.6	10.0	9.59	95.9
Mo	100	97.14	97.1	100	91.56	91.6
Ag	N/C	1.80		N/C	1.31	
Cd	10.0	9.80	98.0	10.0	9.74	97.4
Sb	10.0	10.02	100.2	10.0	9.69	96.9
Ba	50.0	49.27	98.5	50.0	47.22	94.4
Hg	N/C	<0.002		N/C	<0.02	
Tl	10.0	9.46	94.6	10.0	9.34	93.4
Pb	40.0	38.66	96.6	40.0	39.42	98.6
Th	N/C	0.002		N/C	0.004	
U	10.0	9.73	97.3	10.0	9.70	97.0
Na*	6000	5897	98.3	6000	5988	99.8
Mg*	9000	8781	97.6	9000	8987	99.9
K*	2500	2431	97.3	2500	2393	95.7
Ca*	35000	33715	96.3	35000	33689	96.3
Fe*	100	91.47	91.5	100	91.06	91.1

Note: Elements marked with an asterisk (*) are included for information only.

N/C = Not Certified

Table 7. Analysis of NIST® 1643e Drinking Water SRM using the NexION 300Q ICP-MS.

Analyte	NIST® SRM 1643e Trace Elements in Water		
	Certified Value (µg/L)	Average Measured Value (µg/L)	Recovery (%)
Be	13.98	13.24	94.7
Al	141.8	137.7	97.1
V	37.86	36.70	96.9
Cr	20.40	20.00	98.0
Mn	38.97	37.37	95.9
Co	27.06	25.25	93.3
Ni	62.41	59.42	95.2
Cu	22.76	21.30	93.6
Zn	78.50	73.86	94.1
As	60.45	57.25	94.7
Se	11.97	11.30	94.4
Mo	121.4	117.3	96.7
Ag	1.06	0.99	93.1
Cd	6.57	6.26	95.4
Sb	58.30	55.38	95.0
Ba	544.2	511.0	93.9
Hg	N/C	0.01	
Tl	7.445	6.67	89.5
Pb	19.63	18.05	91.9
Th	N/C	<0.0006	
U	N/C	<0.0004	
Na*	20740	20272	97.7
Mg*	8037	7561	94.1
K*	2034	1919	94.3
Ca*	32300	29750	92.1
Fe*	98.1	98.23	100.1

Note: Elements marked with an asterisk (*) are included for information only.

N/C = Not Certified

Table 8. Analysis of drinking water found in our laboratory using the NexION 300Q ICP-MS.

Analyte	Local Drinking Water Spike Recovery			
	Sample Value (µg/L)	Spike Level (µg/L)	Spiked Sample Value (µg/L)	Spike Recovery (%)
Be	0.01	20.00	20.86	104.3
Al	37.10	20.00	57.75	103.2
V	0.19	20.00	18.96	93.8
Cr	0.10	20.00	19.47	96.9
Mn	2.14	20.00	21.41	96.4
Co	0.02	20.00	19.53	97.6
Ni	0.17	20.00	19.94	98.9
Cu	111.5	20.00	130.9	96.7
Zn	3.65	20.00	24.22	102.9
As	0.66	20.00	20.89	101.1
Se	0.02	20.00	20.52	102.5
Mo	0.28	20.00	20.02	98.7
Ag	0.05	20.00	20.13	100.4
Cd	<0.003	20.00	20.33	101.7
Sb	0.03	20.00	20.26	101.2
Ba	3.66	20.00	23.87	101.1
Hg	0.02	2.00	2.01	99.7
Tl	0.04	20.00	20.15	100.5
Pb	0.29	20.00	20.55	101.3
Th	0.06	20.00	20.07	100.0
U	0.14	20.00	20.16	100.1
Na*	3478	2000	5490	100.6
Mg*	267	2000	2268	100.0
K*	193	2000	2137	97.2
Ca*	3390	2000	5377	99.3
Fe*	57.35	20.00	76.49	95.7

Note: Elements marked with an asterisk (*) are included for information only.

With the accuracy of the instrument established, the long-term stability was assessed by measuring the TMDW QCS sample after every 10 samples throughout a 12-hour analysis. The results are shown in Figure 1. The recovery for all elements is < ±10% of the true value over the 12 hours, demonstrating excellent instrumental stability and well within the Method 200.8 guidelines.

Conclusions

The evaluation has clearly demonstrated that the NexION 300Q ICP-MS system is well-equipped to handle the real-world demands of U.S. EPA Method 200.8 for the analysis of drinking water samples. It has shown that by using the instrument's Standard mode, the method IDLs and MDLs can be met with ease. It has also been shown that the analytical accuracy of reference materials is excellent, together with the spike recoveries in real-world drinking water samples. However, probably the most impressive of all the Method 200.8 performance tests is the instrument's

drift characteristics. It shows that for a 12-hour stability test, analyte signals change by less than $\pm 9\%$ from their original concentrations, which is absolutely critical to meet the long-term stability requirements of environmental labs carrying out the high-throughput analysis of drinking water samples. This study will be completed with an evaluation of the instrument's Collision and Reaction mode capabilities in the other two application notes in the series.^{1,2}

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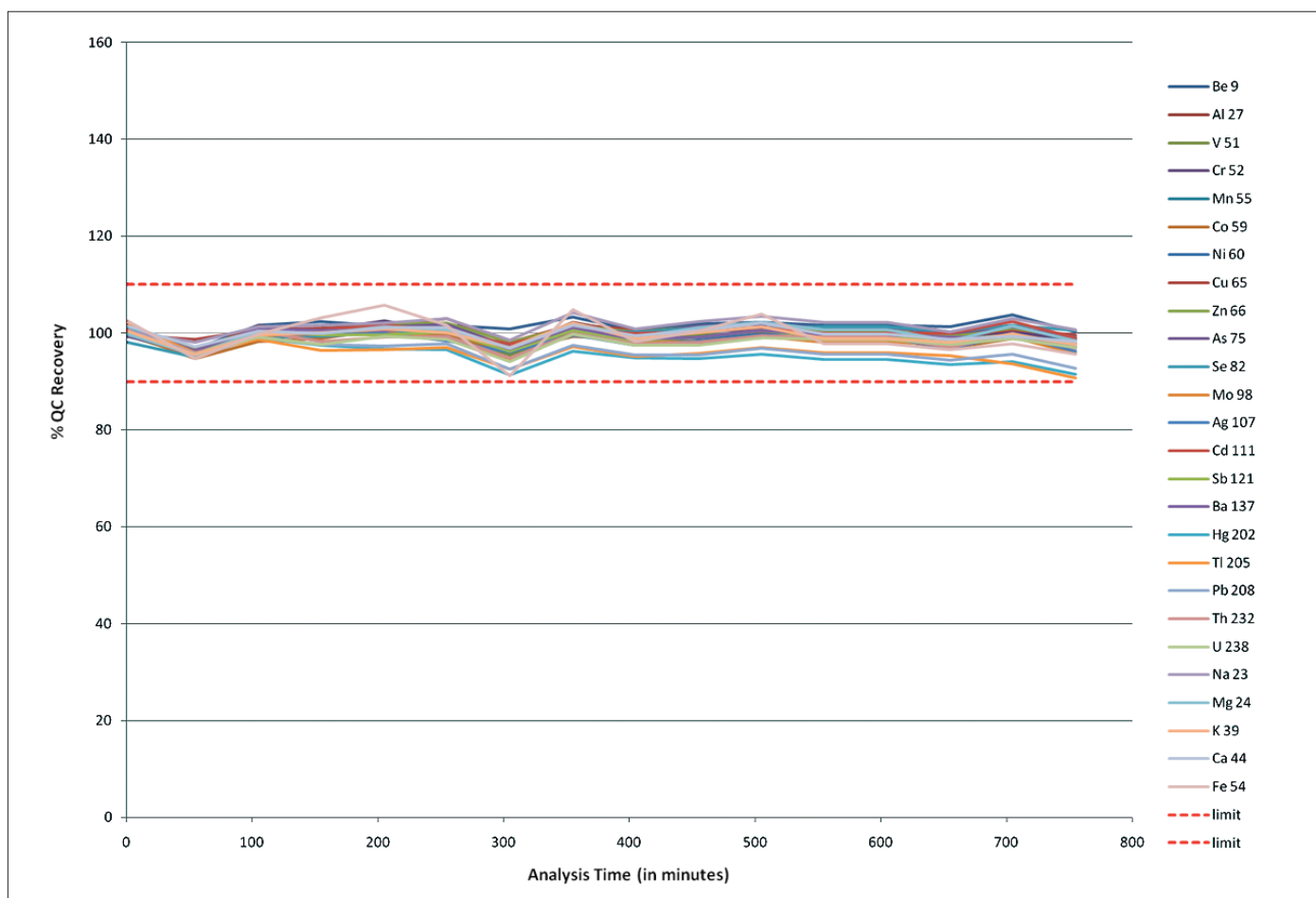


Figure 1. Long-term stability of the TMDW QCS sample measured every ten samples throughout a 12-hour analysis using the NexION 300Q ICP-MS.

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