

ICP-MS

## Thermo Scientific iCAP MSX ICP-MS: Typical limits of detection

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### Introduction

Inductively coupled plasma – mass spectrometry (ICP-MS) is known to be a powerful technique for the analysis of trace elements in many application areas, such as environmental analysis, food safety testing, and clinical analysis. Some of the reasons to choose ICP-MS as an analytical tool are its low limits of detection combined with an extended linear dynamic range and the ability to analyze varying sample matrices without suffering from extensive matrix effects. The Thermo Scientific™ iCAP™ MSX ICP-MS has been designed to deliver these characteristics while also offering the greatest ease of use and the simplest method development.

Instrumental detection limits are a set of key performance indicators detailing an instrument's capabilities and are useful as an aid in determining that instrument's suitability to a chosen task. They demonstrate the lowest level of analyte distinguishable from the background noise under optimal conditions and are typically determined several times for statistical accuracy. Method detection limits (MDL), in comparison, determine the lowest level of analyte that can be detected in a sample matrix using the proposed method of analysis and considers sample preparation steps. This is usually determined on a multi-element basis by diluting standard solutions to concentration levels that can no longer be accurately read, or by adding a low concentration spike into a real sample. There are numerous procedures for determining and calculating the MDL, but most methods require numerous analytical runs, over several days, to ensure a realistic determination of instrument performance.

The instrumental detection limit typically depends on two factors: sensitivity observed for the analyte of choice, as well as low fluctuation of the background signal. To accomplish both, the iCAP MSX ICP-MS instruments feature a series of innovations. To improve sensitivity, IntelliLens™ Signal Optimization dynamically adjusts the voltage applied to selected lenses in the ion optics of the system to yield optimum transmission for every  $m/z$  ratio.\* Backgrounds can be effectively controlled due to the novel interface design in conjunction with the proven performance of the RAPID lens.\* Both features allow for an additional filtering of the ion beam prior to the mass filtration.

ICP-MS provides the highest sensitivity in the so-called standard mode, where the collision reaction cell (CRC) is bypassed. For analytes in the low mass range (i.e., lithium and beryllium) as well as in the high mass range, this mode of operation allows the lowest limits of detection to be achieved, if spectral interferences can be excluded.

To accurately detect and quantify an element of interest, it must be ensured that the measured isotope can be detected free from spectral interferences. While isobaric interferences can be avoided by adequate selection of the isotope to be measured for a given analyte, other types of interferences can be of more concern. The most observed interferences are so-called polyatomic interferences, which are formed through recombination of ions after leaving the plasma or in the interface.

For effective interference removal in single quadrupole ICP-MS instruments, collision/reaction cells (CRCs) are most often used. CRCs may be operated in different ways depending on the nature of the interference to be eliminated; however, the most comprehensive approach is the use of an inert gas, such as He, combined with kinetic energy discrimination (KED).

In KED, unwanted polyatomic interferences are filtered out based on the difference in collision cross-section sizes of the analyte and polyatomic interferences. In addition, the proprietary design of the Thermo Scientific™ QCell™ CRC adds an automatically configured low mass cut-off to the interference removal toolbox. The combination of KED with a low mass cut-off allows unwanted precursor ions to be filtered out along with the polyatomic interferences formed in the plasma. These ions are then unable to recombine later in the QCell CRC so that backgrounds are further reduced and detection limits are improved.

Detection limits themselves can be defined as either the Instrument Detection Limit (IDL) or the Method Detection Limit (MDL) and are accompanied by the Background Equivalent

Concentration (BEC). The IDL defines the lowest concentration of an analyte that can be detected under ideal conditions by the instrument and is normally measured on a single element basis using a clean sample, e.g., using ultrapure 2% nitric acid matrix.

## Method

### Sample preparation

Blank solutions and calibration standards at 1, 5, and 10  $\mu\text{g}\cdot\text{L}^{-1}$  were prepared gravimetrically by adding the appropriate quantity of a multi-elemental stock solution (SPEX™ CertiPrep™) directly to a solution containing 2% m/m  $\text{HNO}_3$  (65%  $\text{HNO}_3$  Optima™ grade, Fisher Chemical™) for measurements in standard mode and 2% m/m  $\text{HNO}_3$  and 0.5% m/m HCl (32–35% HCl Optima™ grade, Fisher Chemical™) for measurements in KED mode. All solutions were prepared in freshly rinsed vials. This acid matrix was selected to reflect a sample preparation procedure that is broadly appropriate to a wide variety of industries and applications.

### Instrument configuration

An iCAP MSX ICP-MS with a Thermo Scientific™ iSC-65 Autosampler was used for all tests. The instrument was equipped with the Thermo Scientific™ iCAP™ MX Series ICP-MS PFA nebulizer, glass spray chamber (Peltier cooled to operate at 2.7 °C), a 2.5 mm i.d. quartz injector, and a demountable quartz torch, as well as nickel-based sample and skimmer cones. The detection limits were determined in the high sensitivity mode.

To test the performance of the KED interference correction, a relatively high concentration of HCl in the acid matrix was used in this study (0.5% m/m), which subsequently leads to the observance of a high number of complex Cl-based polyatomic interferences.

**Table 1. Instrument configuration and operating parameters**

Parameter	Value
Spray chamber	Glass spray chamber, cooled at 2.7 °C
Nebulizer	iCAP MX Series ICP-MS PFA nebulizer
Pump tubing	40 rpm, 0.581 mm i.d., PVC orange-yellow pump tubing
Torch	Quartz torch
Injector	2.5 mm i.d., quartz injector
Interface	Nickel sampler and nickel skimmer
Plasma power	1,550 W
Nebulizer gas flow	1.00 $\text{L}\cdot\text{min}^{-1}$
CRC gas flow	He, 4.2 $\text{mL}\cdot\text{min}^{-1}$
KED	3 V
Scan setting	0.1 s dwell time, 5 sweeps, 3 main runs

\* Patent pending

## Results

The typical performance of the iCAP MSX ICP-MS using STD and He KED mode is shown in Table 2, where detection limits were calculated using the raw intensity data from the standard and the blank, as per the following equation:

$$IDL = 3SD_{\text{blk}} \frac{STD_{\text{conc}}}{STD_x - BLK_x}$$

Where:

IDL is the instrument detection limit

$SD_{\text{blk}}$  is the standard deviation of the intensities of the multiple blank measurements

$STD_x$  is the mean signal for the standard

$BLK_x$  is the mean signal for the blank

$STD_{\text{conc}}$  is the concentration of the standard

Table 2. Typical LODs achieved with the iCAP MSX ICP-MS in STD and He KED mode. All results are reported in ng·L<sup>-1</sup>.

	Standard mode	KED mode
	2% HNO <sub>3</sub>	2% HNO <sub>3</sub> , 0.5% HCl
<sup>7</sup> Li	0.6	19.1
<sup>9</sup> Be	0.10	9.0
<sup>11</sup> B	1.7	64.1
<sup>23</sup> Na	19.7	46.4
<sup>24</sup> Mg	1.2	7.2
<sup>27</sup> Al	3.4	43.1
<sup>39</sup> K	-	280
<sup>44</sup> Ca	61.2	18.2
<sup>45</sup> Sc	5.8	0.4
<sup>48</sup> Ti	0.3	0.8
<sup>51</sup> V	0.12	0.2
<sup>52</sup> Cr	11.1	2.4
<sup>55</sup> Mn	0.2	0.8
<sup>56</sup> Fe	-	5.22
<sup>57</sup> Fe	616	17.0
<sup>59</sup> Co	0.2	0.2
<sup>60</sup> Ni	0.7	2.0
<sup>63</sup> Cu	0.4	0.5
<sup>66</sup> Zn	1.5	5.3
<sup>71</sup> Ga	0.1	0.21
<sup>73</sup> Ge	0.5	1.5
<sup>75</sup> As	0.5	0.5
<sup>77</sup> Se	60	6.7
<sup>85</sup> Rb	0.2	0.5
<sup>88</sup> Sr	0.05	0.3
<sup>89</sup> Y	0.04	0.07
<sup>90</sup> Zr	0.07	0.1
<sup>93</sup> Nb	0.04	0.03
<sup>95</sup> Mo	0.13	0.12
<sup>101</sup> Ru	0.1	0.3
<sup>103</sup> Rh	0.03	0.04
<sup>105</sup> Pd	0.07	0.2

	Standard mode	KED mode
	2% HNO <sub>3</sub>	2% HNO <sub>3</sub> , 0.5% HCl
<sup>107</sup> Ag	0.1	0.05
<sup>111</sup> Cd	0.1	0.13
<sup>115</sup> In	0.02	0.03
<sup>118</sup> Sn	0.1	0.2
<sup>121</sup> Sb	0.06	0.2
<sup>125</sup> Te	0.61	0.64
<sup>133</sup> Cs	0.04	0.08
<sup>137</sup> Ba	0.11	0.43
<sup>139</sup> La	0.01	0.01
<sup>140</sup> Ce	0.02	0.02
<sup>141</sup> Pr	< 0.01	0.01
<sup>146</sup> Nd	0.04	0.02
<sup>147</sup> Sm	0.04	0.01
<sup>153</sup> Eu	0.01	0.01
<sup>157</sup> Gd	0.03	0.01
<sup>159</sup> Tb	0.01	0.01
<sup>163</sup> Dy	0.02	0.01
<sup>165</sup> Ho	0.01	0.01
<sup>168</sup> Er	0.02	< 0.01
<sup>172</sup> Yb	0.02	0.01
<sup>175</sup> Lu	< 0.01	< 0.01
<sup>178</sup> Hf	0.05	0.12
<sup>181</sup> Ta	0.06	0.11
<sup>182</sup> W	0.4	0.40
<sup>185</sup> Re	0.02	0.01
<sup>193</sup> Ir	0.07	0.2
<sup>195</sup> Pt	0.08	0.2
<sup>197</sup> Au	0.23	0.4
<sup>202</sup> Hg	0.31	0.8
<sup>205</sup> Tl	0.06	0.04
<sup>208</sup> Pb	0.05	0.20
<sup>209</sup> Bi	0.03	0.03
<sup>232</sup> Th	0.01	0.01
<sup>238</sup> U	0.01	0.01

The Thermo Scientific™ iCAP™ MSX ICP-MS offers two principal modes of operation, Sensitivity and Matrix, intended to balance the fundamental requirement of sensitivity with the ability to effectively handle potential matrix load from the samples. While the Sensitivity mode delivers the highest signals, the Matrix mode is typically used for the analysis of real samples. The data summarized in Table 2 refers to the Sensitivity mode, typically offering a 30% increase in sensitivity. However, the achievable detection limits in Matrix mode will often be highly comparable.

### Conclusions

The iCAP MSX ICP-MS system with its integrated Thermo Scientific™ Qtegra™ Intelligent Scientific Data Solution™ (ISDS) Software demonstrates excellent performance and provides a significant improvement to analytical laboratory capabilities through simplicity, productivity, and robustness. The flexible configuration delivers a complete solution for a wide range of trace elemental analysis in both research and analytical testing applications.

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