# Real-time adjustment of ICP-MS elemental equations†

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An MS Excel calculation procedure was set up to calculate the correction factors that are part of elemental equations used to correct for polyatomic and doubly charged ion interferences in the ICP-MS analysis of complex samples. The factors are calculated after the measurement of an interference check solution. For typical environmental and biomedical applications, a set of 5–6 check solutions is needed to be able to distinguish where the interference finds its origin. The calculation procedure compares the result for the quantitative isotope with the result for a control isotope (when feasible). Three cases for calculation are distinguished. The procedure also yields the right correction factors when there is some analyte contamination present in the check solution. The procedure lends itself to automation.

In the analysis of complex samples using inductively coupled plasma mass spectrometry (ICP-MS), polyatomic and doubly charged ion interferences (PAI/DCI interferences) can play an important role.<sup>1-4</sup>

The basic approach to overcome problems related to these ions is the use of high-resolution ICP-MS (HR-ICP-MS), where the analyte and interferent peaks are separated.<sup>5</sup> However, the decrease in sensitivity related to the high-resolution mode compromises the detection limits to a certain extent. A new and promising approach is the use of reaction cells inside the vacuum system of the spectrometer to reduce the levels of argon related PAI interferences before these ions can reach the detector.<sup>6,7</sup> Also, the levels of other PAI interferences can be reduced by the careful selection of the reaction gas.

The classical but not fully optimized approach to reduce the effects of PAI/DCI interferences is the use of elemental equations (EEs).  $^{8-11}$  In the EEs approach, the signal of a particular interference (e.g., CIO<sup>+</sup>) is related to the signal of the parent ion (here Cl<sup>+</sup>) via a correction factor (CF): signal of CIO<sup>+</sup> = CF × signal of Cl<sup>+</sup>. By measuring Cl<sup>+</sup> at mass 35, the gross signals at mass 51 and 53 can be corrected for the interferences of CIO<sup>+</sup> to obtain the net signals for  $^{51}$ V<sup>+</sup> and  $^{53}$ Cr<sup>+</sup>. The right value of the CFs in the EEs is checked by aspirating an interference check solution (ICS) containing the particular interferent, but no analyte. The final result of the measurement should be 'zero' concentration for the analyte. If not, the CF in the EE is adjusted until a zero output is obtained after reprocessing. Hence, by trial-and-error, which can be very time consuming, the right CF is obtained.

Further, interferents acting upon the same isotope of the same element should be present in different ICSs to be able to decide which CF should be adjusted. Therefore, a set of interference check solutions should be used to check for the correctness of the CFs and not just one such as in EPA methods 6020 and 6020 CLP.

Finally, in the approach mentioned above, it is essential that the ICS contains no analyte at all, which is sometimes difficult to achieve, because of the limited availability of chemicals of the required purity. This is the case, for instance, in the analysis of rare earth elements, suffering from both PAI interferences<sup>12–14</sup> and standard contaminations.<sup>8</sup>

In the present study, a calculation procedure using MS Excel

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was set up to calculate the CFs after the measurement of a particular ICS. The calculation procedure also yields the correct CFs when the ICS contains some analyte (except for monoisotopic elements). Ways were explored for automatic performance of this task (including updating of the CFs in the method). Attention was focused on analyte and matrix elements relevant in the analysis of environmental and biomedical samples, but the range of both type of elements can easily be extended to other fields.

# **Theory**

The total approach is based on the measurement of two isotopes (in one case three) for each analyte element (if possible, of course). In principle, one isotope is used for quantification purposes, while the other serves as a control isotope in the measurement of the ICS.

For the different elements, three cases can be distinguished:

- (1) No analyte contamination is present in the ICS. This is the requirement for monoisotopic elements such as V and As. Fortunately, these elements are mostly absent in the test chemicals used to prepare ICSs.
- (2) Analyte contamination is (possibly) present in the ICS and a not-interfered control isotope is available.
- (3) Analyte contamination is (possibly) present in ICS and all (relevant) isotopes are subject to interference.

For the different cases, the calculation of the CFs in the EEs is as follows.

# Case 1

The basic equation to calculate a CF is:

$$CF = -\frac{\text{net signal of interference}}{\text{net signal of interferent}}$$

$$= -\frac{\text{signal of ICS at analyte mass} -}{\text{signal of blank at same mass}}$$

$$= \frac{\text{signal of ICS at mass of interferent} -}{\text{signal of blank at same mass}}$$
(1)

In the above equation and all equations to follow the word 'signal' means the signal ratio, *i.e.*, the analyte signal divided by the signal of the internal standard.

For the situation of ClO<sup>+</sup> interference on V<sup>+</sup>, the equation becomes (an example has been chosen for easier understanding of the equations and the symbols used).

J. Anal. At. Spectrom., 2000, 15, 1157–1160 1157

$$\begin{aligned} \text{CF} &= -\frac{\text{signal of ICS at mass } 51 - \text{signal of blank at mass } 51}{\text{signal of ICS at mass } 35 - \text{signal of blank at mass } 35} \\ &= -\frac{\text{ICS}51 - \text{B}151}{\text{ICS}35 - \text{B}135} \end{aligned}$$

(2)

### Case 2

The calculation of the CF for the Case 2 situation will be demonstrated for  ${}^{32}S^{16}O_2{}^{1}H^+$  interference on  ${}^{65}Cu^+$ . The notinterfered  ${}^{63}Cu$  isotope can serve as a control isotope. When there is Cu contamination present in the ICS, containing sulfur for instance as sulfuric acid, the final concentration result for the 65 isotope using the right CF has to be the same as the result for the 63 isotope. The calculation of the CF is as follows:

signal of 
$$^{65}$$
Cu<sup>+</sup> in ICS =  $\frac{\text{abundance of }^{65}\text{Cu}}{\text{abundance of }^{63}\text{Cu}}$   
× net signal of  $^{63}$ Cu<sup>+</sup> in ICS =  $(\text{ab}^{65}\text{Cu}/\text{ab}^{63}\text{Cu})(\text{ICS}63 - \text{Bl}63)$ 

The abundance ratio of <sup>65</sup>Cu and <sup>63</sup>Cu can be calculated from theoretical values or from the measurement of a Cu-containing standard solution. The latter approach gives more accurate results. Then eqn. (2) is modified to give

$$CF = -\frac{ICS65 - (ab^{65}Cu/ab^{63}Cu)(ICS63 - Bl63) - Bl65}{ICS34 - Bl34}$$
 (4)

For this CF the signal of the interferent S is measured at mass 34.

## Case 3

For this situation the calculations will be demonstrated for Ca interference on Ni. The two relevant Ni isotopes (58 and 60) are both subject to interference by Ca containing PAIs ( $^{42}$ Ca $^{16}$ O $^+$  and  $^{44}$ Ca $^{16}$ O $^+$ ). Other Ni isotopes are 61, 62 and 64, but they are either also subject to interference by a Ca PAI or of too low abundance.

Similarly to the Case 2 situation, the signal of the ICS at the interfered masses should be corrected for the (possible) presence of Ni in the ICS:

$$CF^{58}Ni = -\frac{ICS58 - (Ni\ contamination)(slope\ ^{58}Ni) - Bl58}{ICS43 - Bl43}$$
(5)

$$CF^{60}Ni = -\frac{ICS60 - (Ni\ contamination)(slope\ ^{60}Ni) - Bl60}{ICS43 - Bl43}$$
(6)

where the slope is defined as signal/concentration for the specified isotope and the signal of the interferent Ca is measured at mass 43.

CF <sup>58</sup>Ni and CF <sup>60</sup>Ni are related as follows:

$$\frac{\text{CF}^{58}\text{Ni}}{\text{CF}^{60}\text{Ni}} = \frac{\text{abundance}^{42}\text{Ca}}{\text{abundance}^{44}\text{Ca}} = \frac{\text{ab}^{42}\text{Ca}}{\text{ab}^{44}\text{Ca}}$$
(7)

Dividing eqn. (5) by eqn. (6) gives

$$\frac{\text{CF}^{58}\text{Ni}}{\text{CF}^{60}\text{Ni}} = \frac{\text{ICS58} - (\text{Ni contamination})(\text{slope}^{58}\text{Ni}) - \text{Bl58}}{\text{ICS60} - (\text{Ni contamination})(\text{slope}^{60}\text{Ni}) - \text{Bl60}}$$
(8)

Combining eqns. (7) and (8) gives

$$\frac{ICS58 - (Ni\ contamination)(slope\ ^{58}Ni) - B158}{ICS60 - (Ni\ contamination)(slope\ ^{60}Ni) - B160} = \frac{ab\ ^{42}Ca}{ab\ ^{44}Ca}$$

After rearrangement:

1158

J. Anal. At. Spectrom., 2000, 15, 1157-1160

Ni contamination =

$$\frac{ICS58 - B158 - (ab^{42}Ca/ab^{44}Ca)(ICS60 - B160)}{slope^{58}Ni - (ab^{42}Ca/ab^{44}Ca)(slope^{60}Ni)}$$
(9)

Insertion of this result in eqns. (5) and (6) yields the CFs sought. The slopes for <sup>58</sup>Ni and <sup>60</sup>Ni can be calculated from the results of the measurement of an Ni containing standard solution.

For improved accuracy, it is better for some elements or isotopes to calculate the CF from the CF of another element or isotope. This is mostly the case for isotopes with low abundances and elements with low sensitivities. Hence, it is better to calculate the chloride CF of <sup>53</sup>Cr (abundance 9.5%) from the CF of <sup>51</sup>V and the CF of <sup>77</sup>Se (abundance 7.6%) from the corresponding CF of <sup>75</sup>As using the abundance ratio of <sup>35</sup>Cl to <sup>37</sup>Cl of 3.13. This is considered as a Case 4 situation.

# **Experimental**

### Instrumentation, measurement conditions and software

The measurements were performed on a Hewlett-Packard (Avondale, PA, USA) HP 4500 plus instrument equipped with an HP V-groove nebulizer, a double pass Scott-type spray chamber cooled to 2 °C and a Cetac ASX-500 autosampler. An internal standard solution containing  $20 \, \mu g \, l^{-1}$  of Sc, Rh and Bi was mixed on-line (1+5) with the samples and standard solutions *via* separate tubing on the peristaltic pump. The measurement conditions are given in Table 1.

Using the Intelligent Sequence Software of the instrument, a new QC configuration was set up allowing the use of more than one ICS. This new configuration was based on the 6020 CLP QC configuration. An MS Excel calculation program, named Correct.xls, using the equations in the Theory section was set up to calculate the CFs. The raw data (uncorrected counts s<sup>-1</sup>) obtained for the blank, a multi-element standard solution and the ICSs were pasted into the spreadsheet, yielding the CFs. The resulting CFs were brought into the method. To be able to carry out this step, it is essential that the method file containing the CFs is open/accessible to the user.

# **Experiments**

In the experiments, the analyte elements considered were V, Cr, Fe, Ni, Cu, Zn, As, Se and Cd. For these elements an inventory of possible PAI/DCI interferences was made for matrix elements relevant in the analysis of environmental and biomedical samples. Following this, a selection was made of analyte isotopes for quantification purposes (high abundance, low level of PAI/DCI interferences) and of control isotopes. Also, the composition and the number of the ICSs was chosen based on the results of the above measurements. After measuring the ICSs, the CFs were calculated using Correct.xls.

# Results and discussion

Matrix elements giving significant interferences on quantification isotopes are C, Mg, S, Cl, Ca, Br, Sr and Ba. Small interferences, only relevant for high concentration levels of interferents, are caused by Mo and Sn. Matrix elements giving significant interferences on control isotopes are Na, Si and Fe.

Table 1 Measurement conditions

R.f. power	1330 W
Sampling depth	7.5 mm
Nebulizer gas flow rate	$1.191\mathrm{min}^{-1}$
Aux. gas flow rate	$1.01\mathrm{min}^{-1}$
Plasma gas flow rate	$15.01\mathrm{min}^{-1}$
Pump speed	Sample $0.07 \text{ ml min}^{-1} + \text{IS } 0.35 \text{ ml min}^{-1}$

Table 2 Composition of ICSs, interfered isotopes and control isotopes. Quatification isotopes are shown in bold

	Interferent (measured at mass)	Concentration <sup>a</sup>	Interfering ion <sup>b</sup>	Acting on	Possible control isotope <sup>c</sup>
ICS1	Mg (26)	100 mg l <sup>-1</sup>	$^{25}{ m Mg}^{16}{ m O_2}^+$	<sup>57</sup> Fe	( <sup>54</sup> Fe)
	- , ,		$^{40}\text{Ar}^{24}\text{Mg}^+, ^{40}\text{Ar}^{26}\text{Mg}^+$	<sup>64</sup> Zn, <sup>66</sup> Zn	<sup>68</sup> Zn
		1	$^{40}\text{Ar}^{25}\text{Mg}^{+}$	<sup>65</sup> Cu	<sup>63</sup> Cu
ICS2	Ca (43)	$400 \text{ mg } 1^{-1}$	$^{40}\text{Ca}^{16}0^{1}\text{H}^{+}$	<sup>57</sup> Fe	$(^{54}\text{Fe})$
			$^{42}\text{Ca}^{16}\text{O}^{+},  ^{44}\text{Ca}^{16}\text{O}^{+}$	<sup>58</sup> Ni, <sup>60</sup> Ni <sup>64</sup> Zn	<sup>66</sup> Zn
			$^{48}\text{Ca}^{16}\text{O}^{1}\text{H}^{+}$	65 <b>Cu</b>	<sup>63</sup> Cu
			$^{40}Ar^{42}Ca^{+}$	82Se	( <sup>77</sup> Se)
	Mo (95)	$100 \ \mu g \ l^{-1}$	<sup>95</sup> Mo <sup>16</sup> O <sup>+</sup> , <sup>98</sup> Mo <sup>16</sup> O <sup>+</sup>	<sup>111</sup> Cd, <sup>114</sup> Cd	
ICS3	S (34)	$400 \text{ mg } 1^{-1}$	95Mo <sup>16</sup> O <sup>+</sup> , 98Mo <sup>16</sup> O <sup>+</sup> 32S <sup>16</sup> O <sub>2</sub> <sup>+</sup> , 34S <sup>16</sup> O <sub>2</sub> <sup>+</sup>	<sup>64</sup> Zn, <sup>66</sup> Zn	$^{68}$ Zn
			$^{32}S^{16}O_{2}^{1}H^{+}$	<sup>65</sup> Cu	<sup>63</sup> Cu
		=1	$^{34}S^{16}O_3^{2} +$	<sup>82</sup> Se	$(^{77}Se)$
ICS4 <sup>d</sup>	Cl (as NaCl)	$1000 \text{ mg } 1^{-1}$	<sup>35</sup> Cl <sup>16</sup> O <sup>+</sup> <sup>35</sup> Cl <sup>16</sup> O <sup>1</sup> H <sup>+</sup> , <sup>37</sup> Cl <sup>16</sup> O <sup>+</sup>	<sup>51</sup> V <sup>52</sup> Cr, <sup>53</sup> Cr	
	(35)		<sup>40</sup> Ar <sup>35</sup> Cl <sup>+</sup>	75 <b>As</b>	
			$^{40}\text{Ar}^{37}\text{Cl}^{+}$	77Se	( <sup>82</sup> Se)
	Si (29)	$40 \text{ mg } 1^{-1}$	$^{40}\text{Ar}^{28}\text{Si}^{+}$	<sup>68</sup> Zn	<sup>66</sup> Zn
ICS5	Fe (54)	$20 \text{ mg } 1^{-1}$	<sup>58</sup> Fe <sup>+</sup>	<sup>58</sup> Ni	<sup>60</sup> Ni
	Br (81)	$8 \text{ mg } 1^{-1}$	$^{1}H^{81}Br^{+}$	<sup>82</sup> Se	$\binom{77}{3}$ Se)
	Ba (69)	$1 \text{ mg } 1^{-1}$	$^{130}$ Ba $^{2+}$	<sup>65</sup> Cu	<sup>63</sup> Cu
	G (00)	2 1=1	$^{132}\text{Ba}^{2+}$ , $^{136}\text{Ba}^{2+}$ $^{86}\text{Sr}^{2+}$ , $^{88}\text{Sr}^{2+}$	66 <b>Zn</b> , 68 <b>Z</b> n	<sup>64</sup> Zn
1006	Sr (88)	$2 \text{ mg l}^{-1}$ 500 mg l <sup>-1</sup>	$^{40}\text{Ar}^{12}\text{C}^{+}$	<sup>43</sup> Ca, <sup>44</sup> Ca <sup>52</sup> Cr <sup>e</sup>	<sup>53</sup> Cr
ICS6	C (13) Na (23)	100  mg  1	$^{40}\text{Ar}^{23}\text{Na}^{+}$	<sup>63</sup> Cu	<sup>65</sup> Cu
	Sn (118)	20 μg l <sup>-1</sup>	114Sn+	114Cd	( <sup>111</sup> Cd)

<sup>a</sup>For sixfold on-line dilution (see text). <sup>b</sup>The first interfering ion acts on the first analyte isotope and the second ion on the second isotope. <sup>c</sup>Control isotopes in parentheses not very suitable for calculation of CF because of too variable results (too high detection limits). Case 1 calculation is then preferred. <sup>d</sup>See text (variation of CFs with matrix composition). <sup>e</sup>For real samples with high C concentration and low Cl concentration, it can be advantageous to use the 53 isotope.

No significant interference related to Al and P was observed. Interference of TiO<sup>+</sup> on Zn and Cu was typically small because of the low Ti concentrations in 'real-life' samples. PAI/DCI interferences related to Li, B and F were not investigated, but are most probably not relevant. Hence, in fact, all matrix elements up to Sr (mass 88) were investigated with respect to their potential for formation of PAIs/DCIs, and also some interferents with higher mass (Mo, Sn and Ba).

The composition of the ICSs chosen is given in Table 2, together with the interfered analyte isotopes. When feasible, nitrates were used for the preparation of the ICSs. Sulfuric acid

**Table 3** Typical concentration output of Correct.xls in  $\mu$ g I<sup>-1</sup>. Values in bold are concentrations corrected for interference by the interferents of the particular ICS. Non-bold values are (in most cases) control values

		ICS1 Mg	ICS2 Ca, Mo	ICS3 S	ICS4 Cl, Si	ICS5 Fe, Ba, Br, Sr	ICS6 C, Na, Sn
Ca	43					8	
	44					28	
V	51				0.006		
Cr	52			-0.15	-0.07		0.38
	53			0.12	0.00		0.35
Fe	54	25	17				
	57	1	- <b>2</b>				
Ni	58		0.26			-0.04	
	60		0.35			0.16	
Co	59		0.00				
Cu	63	0.15	0.05	0.18		0.12	0.13
	65	0.16	0.05	0.18		0.14	0.13
Zn	64	0.3	1.1	0.3	3.6	0.4	
	66	0.4	0.9	0.3	3.2	0.4	
	68	0.4	1.1	0.2	3.2	0.4	
As	$75^{a}$		0.03	0.02	0.00	0.03	
	$75^{b}$		-0.01	-0.08	-0.09	0.09	
Se	77		0.2	0.4	0.1	-0.3	
	82		0.0	0.0	-0.3	0.0	
Cd	111		-0.001				0.003
	114		-0.001				-0.001
a <sub>C</sub>	rrooti	n boso	d an 35C1	bCommo	ation bo	sed on 82Se 77Se	340 4300

"Correction based on <sup>35</sup>Cl. <sup>b</sup>Correction based on <sup>82</sup>Se, <sup>77</sup>Se, <sup>34</sup>S, <sup>43</sup>Ca and <sup>81</sup>Br. <sup>11</sup>

was used as a source of S in ICS3 and galactose as a source of C in ICS6. A typical output of Correct.xls is given in Table 3. Correct.xls is also capable of calculating concentrations in addition to the calculation of CFs. As mentioned in the Theory section, results obtained for different isotopes of the same element when measuring a particular ICS should be the same, indicating that the CF(s) is (are) calculated correctly. This is usually the case. The deviation observed for Ni in ICS2 is caused by variation of the signal measured at mass 54 for the blank and for ICS2, as was found out by numerical adjustment of the values. As <sup>58</sup>Ni is also corrected for Fe interference using this mass, this caused a slightly deviating result for this Ni isotope (the result for <sup>60</sup>Ni equals the contamination calculated). The deviation for Ca in ICS5 is caused by the fact that the actual Sr CF ratio for <sup>43</sup>Ca and <sup>44</sup>Ca deviates slightly from the applied ratio due to mass bias effects. Adjustment of this factor (related to Sr isotope abundances) by 1-2% equalizes the results for <sup>43</sup>Ca and <sup>44</sup>Ca. However, the deviation is small compared with the detection limits of the two Ca isotopes (60 and  $30 \text{ g l}^{-1}$ , respectively, for the six-fold dilution applied). The deviations found for Fe in ICS1 and ICS2 are caused by the fact that for 57Fe a Case 1 approach was used (making the result equal to zero) because of the high detection limit for <sup>54</sup>Fe (see Table 2). Finally, the negative results in Table 3 will be related to the fact that, similarly to the situation for Ni, a particular isotope is corrected for more than one PAI/DCI interference, the level of which may also vary.

The magnitude of the Cl CFs for V, Cr, As and Se is related to the composition of the ICS, particularly the presence of Na. An increase in the Na concentration results in a smaller Cl CF for <sup>51</sup>V and <sup>53</sup>Cr (less interference of ClO<sup>+</sup> on <sup>51</sup>V and <sup>53</sup>Cr) and a higher Cl CF for <sup>52</sup>Cr, <sup>75</sup>As and <sup>77</sup>Se (more interference for these isotopes). No effect was observed of a varying concentration of Ca. Hence it is best to determine the Cl CFs in the presence of an equimolar amount of Na, because this represents the samples the best in most cases (see ICS4 in Table 2). However, when HCl is the dominant matrix component, the NaCl in ICS4 should be replaced by HCl.

The variation of the magnitude of the CFs with time was

usually limited. The largest variation was observed for the CaO CF of <sup>60</sup>Ni: a relative standard deviation (RSD) of the average CF of 27% over a period of 1 month was observed (n=8). For other cases the RSDs ranged from 7 to 15% over the same period. The variation of the CaO CF of <sup>60</sup>Ni is most probably related to the condition of the cones. At the end of the test period mentioned above the sample cone had to be cleaned because of low sensitivity. Also, the CaO interference is relatively small, resulting in more variation of the CF. In general, it is advisable to condition the instrument with a suitable matrix solution prior to the measurement of the ICSs.

Future developments should include the following. Within the software of the instrument all information is available: the raw data of the measurements and the file containing the CFs. Hence automation of the procedure and (semi-)real-time adjustment of elemental equations is feasible and within reach (adjustment within the measurement sequence = automatic interference correction, AIC). At the beginning of the sequence the CFs should be updated following the measurement of the ICSs and at the end of sequence they should be checked for drift by a new measurement of the ICSs. Ideally, CFs valid for each measurement should be interpolated from the results of the first and last measurement of the ICSs in a measurement sequence.

To conclude, in practice not all the ICSs have to be measured in all measurement series. Depending on the concentration levels in the samples of the elements requested and the required detection limits, matrix component concentration levels and specific instrumental performance with respect to interference levels (variable from instrument to instrument), the measurement of some ICSs might be omitted, speeding up the analysis procedure.

# **Conclusions**

The calculation procedure developed to calculate the elemental equation correction factors to correct for polyatomic and doubly charged ion interferences in the ICP-MS analysis of complex samples also yielded the right correction factors even when there is some analyte contamination present in the interference check solution. For typical environmental and biomedical applications a set of 5–6 check solutions are needed to be able to distinguish where the interference finds its origin. Three cases for calculation were distinguished and the procedure lends itself to automation.

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