Guidelines for Determining IEC's on iCAP 6000/7000 Series Running Qtegra

Table of Contents

Optimize System / Method 1
Determine Analytical Range 1
Perform IEC Experiment 2
Sample Analysis 3

Optimize System / Method

1. Clean all glassware and sample introduction components. *(Don't forget Radial View Window and POP Window.)*

2. Light plasma and allow the system to thermally stabilize for 15-20 minutes.

3. Optimize the Nebulizer Flow rate by observing the Yttrium bullet position

4. Perform a Torch Alignment.

5. Perform a Spectrometer Optimization.

6. Perform an Auto Peak adjustment for wavelengths that have not been previously Auto Peaked.

Determine Analytical Range

7. Set all Background Points to ***Yes*** within the method, **Do not use "Auto"**.

8. Calibrate the system and calculate detection limits, ensure they are well below required reporting limits for each wavelength *(3-5X at a minimum).*

9. View Subarray of a standard and adjust the number of center read pixels. For the wavelengths of analytes of interest, 2 or 3 center pixels should be used. *(see Technical Note "Determining Number of Center Pixels in Qtegra for ICP-OES")*

10. Define IEC *(Interfering Element Correction)* Pass/Fail Criteria for each wavelength.
    (For example: If the RL (Reporting Limit) is less then 10ppb use +/- 2X the RL, if the RL is greater than 10ppb use +/- 1X the RL) ***Note:*** *this is only an example. Please refer to any methodology in place from the regulatory body governing your application for exact criteria on how to setup the Pass/Fail criteria for the method you are running.*
    Note: This document refers to Reporting Limit (RL) which is also known as LOQ (Limit of Quantification).

11. Calibrate the system and perform a Linear Dynamic Range Study to determine the upper end of the analytical range for each element.

Perform IEC Experiment

12. Create **contaminate free** single element solutions at concentrations that are typically seen in real customer samples. This concentration should be within the linear range for the wavelength used for any given element and preferably not at the linear range value. The ICSA *(Interfering Check Solution A)* elements *(Al, Mg, Fe, Ca)* should be analyzed at the same levels that are in the ICSA and ICSAB *(Interfering Check Solution AB).*
    Note: *This document references the EPA methodology which is where the terms ICSA and ICSAB originate but in principle the guidelines listed in this document will apply to any application ran by ICP-OES.*

13. Calibrate the system and run all single element solutions as Unknowns.

14. Overlay all single element solutions, calibration blanks, and calibration standards to view image data in the subarray.

15. Determine the appropriate position and number of Background Points to be used for each wavelength. Compromises will have to be made in some circumstances based upon which interfering element is more prevalent in customer samples.
    - Avoid placing Background Points beneath interfering peaks
    - Avoid placing Background Points on last pixel column on either end of the subarray *(far left or far right.)*
    - It may be necessary to widen some subarrays to find an appropriate Background Point location. If so, change the subarray width in the method, save the method, recalibrate the system, re-analyze the single element solutions, and position Background Points accordingly for that wavelength.
    - Once the Background Point positions have been established, save the LabBook.

16. Analyze the results of the single element solution analysis to see if any of the analytes of interest are reporting a value which is outside of the range determined for that element in step 10 thus generating a failure action and requiring further investigation. This would potentially indicate that the element from which this single element solution was made has interfered with the analyte of interest.

17. View the image data for each failed result within each single element standard and make sure the failure was not caused by contamination. *(If contamination is suspected do not calculate an IEC for that element. Better to re-make the standard cleanly and re-run)*

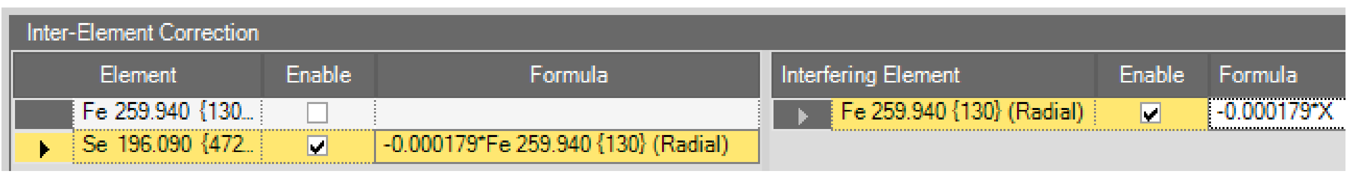
Note: *Also look for spectral contributions that do not fail the IEC Pass/Fail Criteria but may be border line within range. An IEC Calculation should be considered on an element by element basis.*

18. Calculate the IEC factors by performing one of the following two methods:

    a) Auto Calculate the IEC factors for an interfering element
    Note: *Please reference the video "How to Setup Inter Element Correction" (https://youtu.be/FXjFgkBWJ5c) which will demonstrate how to input the correction equations either manually or automatically within Qtegra*

    b) Manually calculate the IEC factors using the example below but substituting your interfering element and analyte of interest (Fe and Se respectively in the example below)

    For example: An analysis of a 200ppm Fe standard gives results of 201 ppm Fe and 0.036 ppm Se. k1 = 0.036 ppm Se / 201 ppm Fe = +0.000179. In this example, 0.000179 would be entered as the correction factor for Fe on Se in Qtegra as seen below.



Note: *Please reference the technical note "Manually Entering Inter-Element Correction Equations into Qtegra for ICP-OES" for detailed instructions on how to manually input IEC's into Qtegra*

19. Repeat this process for each interferent.

<u>Sample Analysis</u>

20. A spectrometer optimization should be performed every day after the system has had time to thermally equilibrate *(15-20min)* and before running samples.

21. The sample introduction components should be cleaned (torch, center tube, nebulizer, POP window, and Radial viewing window) or replaced (pump tubing, capillary tubing, autosampler probe) on a regular basis as a preventative measure in order to maintain optimal performance. A record of this should be documented. *(Example: If the radial view bucket becomes dirty and suppresses the radial signal, the ICSA results may be affected. The ICSA elements are viewed radially while many of the very sensitive wavelengths that these interfere with are viewed axially.)*

22. If your ICSA fails, re-run the ICSA single element solutions for the element that failed.

(Example: if Lead fails and the only IECs you have on Lead from the ICSA solution are Al and Fe then you only need to re-run the Al and Fe solutions.)

23. IECs can then be recalculated and inputted into LabBook (see step 18). Associated sample results will then be automatically updated with the new IEC factors.

24. If the location or number of Background Points or Center Pixels are modified for any wavelength which has an IEC factor applied to it or has been used in the calculation of an IEC, an IEC study will have to be performed again. This is because the IEC, Background Points, and Center Pixels all interact with one another. The accuracy of your IEC factor is dependent upon these variables remaining constant.