

# Elemental Analysis Manual

## for Food and Related Products

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## 3.6.3 Inductively Coupled Plasma-Atomic Emission Spectrometer

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This section provides information to assist the analyst on assuring analytical instrumentation is performing properly.

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GLOSSARY

### 3.6.3.1. Interferences<sup>1</sup>

Spectral interferences associated with inductively coupled plasma-atomic emission spectrometry (ICP-AES) are caused by background emission from continuous or recombination phenomena, stray light from line emission of high concentration elements, overlap of a spectral line from another element, or unresolved overlap of molecular band spectra. Subtracting background emission is usually necessary for most analytical emission lines. Spectral scans (wavelength verses intensity) in analyte wavelength region may indicate when alternate emission lines are desirable because of severe spectral interference. Spectral scans will also show whether the most appropriate estimate of background emission is provided by an interpolation from measurements on one or both sides of the analyte peak. Locations selected for background intensity measurements will be determined by the complexity of spectrum adjacent to a wavelength peak. Locations used for routine measurement must be free of off-line spectral interference (inter-element or molecular) or adequately corrected to reflect the same change in background intensity as occurs at the wavelength peak.

Spectral overlap may be avoided by using alternate wavelengths or can be compensated for by equations that correct for inter-element contributions, which involves measuring interfering elements. Extensive information on interferences at various wavelengths and resolutions is available in Boumans' Tables<sup>2</sup> and Winge's Atlas<sup>3</sup>. Users may apply inter-element correction factors determined on their instruments within tested concentration ranges to compensate (off-line or on-line) for effects of interfering elements. When inter-element corrections constitute a major portion of an emission signal results may not be accurate. Trace element levels typically found in foods do not cause significant spectral overlap for elements determined by this method<sup>2-4</sup>. Elements that might cause interference because of their potential levels in food and spectral characteristics include phosphorus, calcium, iron, zinc, aluminum and titanium. These six elements should be included in the analyte list even if quantitative results are not needed. This will enable inter-element correction factors to correct for spectral interference. Interference effects must be evaluated for each instrument. To determine appropriate location for off-line background correction, an analyst must scan on either side adjacent to the analytical wavelength and record apparent emission intensity from all other method analytes. On-line and off-line spectral interference effects must be determined and documented for all method analytes and correction on all analyses must be performed. Tests to determine spectral interference must be done using analyte concentrations that will adequately describe interference. Expansion of the scan's scaling may be necessary to observe the interference or ascertain its absence. For most elements, 100 mg/L single element solutions are sufficient although higher concentrations may be necessary for some mineral elements (*e.g.*, calcium). Failure to correct for spectral interference can result in false positive or false negative results. Uncorrected interfering peaks occurring on or very close to the analyte peak can result in false-positives or positive bias. Uncorrected interfering peaks occurring on or very close to a background correction wavelength can cause negative bias or even negative results.

Physical interferences are effects associated with sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies, especially in analytical solutions containing high dissolved solids or high acid concentrations. Physical interferences can be reduced by diluting the analytical solution.

Chemical interferences include molecular-compound formation, ionization effects, and solute-vaporization effects. Normally, these effects are not significant with ICP-AES. If observed, they can be minimized by careful selection of operating conditions, matrix matching, and using method of standard-additions. Chemical interferences are highly dependent on matrix type and specific analyte element determined.

Memory interferences occur when analytes from a previously measured analytical solution

contribute to analyte signals currently being measured in an analytical solution. Memory effects can result from analyte deposition on nebulizer uptake tubing or from build-up of material in the plasma torch and spray chamber. The site where these effects occur is element dependent and can be minimized by flushing with a standard blank between analytical solutions. Monitoring for memory interferences is performed during an analytical run and suitable rinse times are to be established to control their affect on analyte measurements. Rinse times necessary for a particular element must be determined before analysis. Determination of a sufficient rinse time may be achieved by aspirating a standard solution containing elements corresponding to either the upper end of their LDRs or concentrations ten times those usually encountered. A normal aspiration time should be used, followed by analysis of the standard blank at designated intervals. The length of time required to reduce an analyte's signal to within a factor of two of the ASDLs should be used as the rinse time if more than the minimum 60 sec is required. Until required rinse time is established, a 60 sec rinse period is recommended between analytical solutions and standards. If memory interference is suspected, analytical solutions should be re-analyzed using a longer rinse period.

#### 3.6.3.2. Instrument Setup

Each laboratory must determine optimum instrument parameters for radio frequency (RF) power, view height, argon flow rates and sample uptake rate. Analyst should be aware that small changes in RF power, view height and argon flow rates can greatly affect instrument performance and inter-element correction factors. Inspect sample introduction system including nebulizer, torch, injector tube and uptake tubing for salt deposits and dirt that would restrict solution flow and affect instrument performance. Inspection frequency will depend on work load and analytical solution composition. Inspect system at each use and clean as needed. Allow instrument to become thermally stable before standardization and analyses. This usually requires at least 20 to 30 minutes of operation. After instrument warm-up, perform optical profiling. Optical profiling is performed with a built-in mercury lamp, a 2 mg/L Mn solution, or a procedure recommended by instrument manufacturer. If laboratory has a sequential type ICP-AES instrument, perform wavelength calibration according to manufacturer's instruction.

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*Safety Note: Inductively coupled plasmas emit ultraviolet radiation during operation and must be viewed with proper eye protection.*

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#### 3.6.3.3. Pre-standardization Checks

Instrument sensitivity and precision check—Ensuring the instrument is operating correctly is essential before spending time standardizing or analyzing samples. Instrument sensitivity and short-term precision must be demonstrated before proceeding with standardization. Analyze one of the standard solutions, or a separate solution made for this check, for 5 replicate integrations. Monitor the emission counts (or emission ratio) of a selected element (*e.g.*, 2 mg/L Mn). Calculate the mean and RSD of the emission counts. The mean emission counts should be within 20% of the historical mean indicating good sensitivity. The RSD should be less than 5% indicating good precision. Failure of either the sensitivity or precision check usually indicates a solution introduction problem. Correct the problem before proceeding.

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*Note: A special solution dedicated to this daily task may be used routinely. The element used for this check can be different from analyte(s). The daily mean emission counts and RSD should be recorded for future reference.*

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#### 3.6.3.4. Standardization Verification

To ensure consistent instrument performance and accuracy, IDL and instrument standardization are verified initially. Instrument standardization is also verified during and after an analytical run.

- (1) IDL verification—immediately after standardization, determine IDLs. Analyze the standard blank 5 times (separate analyses with normal autosampler rinse in between). The IDLs must be within 3 times the normally obtained IDL values. Record IDLs for future reference.
- (2) Initial standardization verification—Analyze ICS and standard blank immediately following instrument standardization and IDL verification. Results for ICS recovery must be  $100 \pm 5\%$  of expected value. Analyze standard blank after ICS to check for carry over. Results for the standard blank must be less than the ASDL. If either of these conditions is not met, diagnose and correct the problem(s) and re-standardize ICP-AES instrument.

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*Note: If the fortification solution was used to prepare ICS and ICS is out of control, an error in fortification of the FAP should be suspected and may require the FAP to be re-prepared.*

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- (3) Continuing standardization verification—To verify lack of instrumental drift, analyze a check solution at a frequency of 10% of analytical solutions and at end of analytical run. Control limits for check solutions are  $100 \pm 10\%$  of expected concentrations. If control limits are not met analysis must be discontinued, cause of deviation determined and instrument re-standardized. All analytical solutions following the last acceptable check solution must be re-analyzed. This procedure ensures all groups of 10 or less analytical solution analyses are bracketed by valid standardization verification checks.

#### 3.6.3.5. Analysis Checks

- (1) Precision—All measurement results of analytical solutions, diluted analytical solutions, standard solutions, and quality control solutions shall be based on the mean of at least 3 replicate integrations. Precision of replicate integrations must be 7% RSD or less for analytes above ASQL in all analytical solutions. If control limits are not met then re-analyze the analytical solution. If the repeat analysis is still out of control then suspect either an instrument problem or matrix interference. Diagnose problem, make necessary adjustments and re-analyze analytical solution. There may be either a problem with the sample introduction system or a physical interference with the analytical solution. Flushing the sample introduction system for several minutes and diluting analytical solution by a factor of 2 may resolve the problem. If the RSD still fails then diagnose the problem and fix before proceeding.
- (2) Standard curve—The highest standard solution must be within the LDR. Values for correlation coefficients ( $r$ ) must be  $\geq 0.998$ . A value less than this control limit indicates

problem with preparation or standardization due to one or more standard solutions or the standard blank. If display of the standard curve (intensity vs. concentration) indicates which standard solution is bad, re-standardize that standard solution. Otherwise re-standardize ICP-AES instrument. If re-standardization does not fix the problem, then prepare new standard solutions and re-standardize instrument.

- (3) Alternate wavelength precision—If possible, use multiple alternate wavelengths for each analyte. For all measurement results >ASQL, the concentration found at the primary wavelength must agree within  $\pm 10\%$  relative difference of the concentration found at the secondary wavelengths for each element. A relative difference >10% can be due to instrument problems or matrix/spectral interference in the analytical solution. If check solutions are within control limits, dilute the analytical solution and re-analyze. If the diluted analytical solution is still out of control, an alternate analytical method must be used.
- (4) Wavelength scan—Each analytical solution is checked for spectral interference by performing a wavelength scan. An intensity (emission counts) versus wavelength scan is recorded for each element for each analytical solution. Depending on ICP-AES instrument software, these scans can be incorporated into the ICP-AES analytical run or performed in a separate "scan" run. An appropriate standard solution must be scanned and the result overlaid with the scan of the analytical solution. A standard solution close in element concentration to the analytical solution should be chosen. A broad or double peak indicates an unresolved peak that may result in a positive bias. Interfering peaks could be from elements not being quantified. Peaks in the area of the background correction point(s) may result in a negative bias. Background correction points must be in an area(s) free from other peaks.

## REFERENCES

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- (2) Boumans, P. W. J. M., (1984) *Line Coincidence Tables for Inductively Coupled Plasma Atomic Emission Spectrometry*, 2nd Ed., Pergamon Press, Oxford, United Kingdom.
- (3) Winge, R. K., Fassel, V. A., Peterson, V. J., and Floyd, M. A. (1985) *Inductively Coupled Plasma-Atomic Emission Spectroscopy: An Atlas of Spectral Information*, Physical Science Data 20. Elsevier Science Publishing, New York, New York.
- (4) Jones, J. W. (1988) in *Quantitative Trace Analysis of Biological Materials: Food Samples*, Chapter 20, Elsevier Science Publishing, New York, New York, pp. 353-365.