

Elemental Analysis

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Analysis of Toxic Elements in Foodstuffs and Pharmaceuticals by ICP Emission Spectroscopy

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Introduction

The safe production and monitoring in food and ancillary industries, such as herbal medicines and dietary supplements, is ever more prevalent with the increase in homeopathic and organic traditional remedies and diets re-emerging into world culture. There is increasing public health concern with regards to chemical contamination of food, dietary supplements and pharmaceuticals by certain toxic elements. Long term exposure to lead, for example, can damage nervous connections and cause blood and brain disorders. Manganese can be toxic in excess, resulting in impaired motor skills and cognitive disorders. In addition, excessive iron can damage DNA, proteins, lipids and other cellular components in the heart and liver and can cause long-term organ damage and even death.

The ever increasing public health concerns have triggered the emergence and enforcement of many strict regulations. An appropriate method capable of providing accurate measurements of toxic elements in foodstuffs and pharmaceuticals is required to assist manufacturers to comply with the stringent regulations and ensure they are offering consumers safe and healthy products. ICP emission spectroscopy is the ideal solution for trace element analysis providing excellent detection capability and fast and accurate analyses.

Regulatory Framework

The food industry standards are largely based upon ISO FDIS 22000, which specifies requirements for a food safety management system that allows control of food safety hazards in order to ensure that food is safe at the time of human consumption. The HACCP (Hazard Analysis and Critical Control Point) is a more popular control system used by food and drug organizations to identify potential hazards, define the production points at which hazards can be controlled or eliminated and establish preventive measures and actions needed to correct potential problems. The World Health Organization (WHO) has adopted many HACCP systems as the international standards for food safety into its own Codex regulations. According to these regulations, each region regulates the process from the ground up, with normative documents for soils analysis, harvesting and storage and then onto more stringent controls when it comes to the manufacturing of foodstuffs.

In the US, food and pharmaceutical manufacturers must pass CGMP (Current Good Manufacturing Practice) certification, in line with the Food and Drug Administration (FDA) Rule 21 CFR Parts 111 and 112. These establish the minimum CGMPs necessary for activities related to the manufacturing of dietary supplements to ensure the quality of the products. On the other hand, the 21 CFR Parts 210 and 211 regulations affect the pharmaceutical industry, with Part 210 dealing with the manufacturing and processing, of drugs while Part 211 relates to finished pharmaceuticals.

The need for strict regulatory frameworks in the US is further strengthened by the growing trend in international trade for worldwide equivalence of food products. As China exports a large proportion of their traditional medicines to the US, they must conform with FDA and ISO standards in addition to their own specific 2005 version Chinese Codex which specifies the maximum permissible limits of lead (Pb), cadmium (Cd), mercury (Hg), arsenic (As) and copper (Cu) in Chinese medicine. In India, a growing market for traditional herbal medicines, the Government of India, the Ministry of Health and Welfare, the Department of Ayurveda, Yoga and Naturopathy, Unani, Siddha and Homoeopathy (AYUSH) have produced a notification confirming that testing of heavy metals such as As, Pb, Hg and Cd is mandatory for export purposes.

There is also an additional requirement to control electronic records under the FDA 21 CFR Part 11 rule. An electronic record is any text, graphic or numerical data that is created, modified or stored in an electronic format. It permits more efficient monitoring of a company's compliance with food and drug safety regulations over an entire period rather than on any given day. The rule requires that any electronic record must have a full audit trail associated with it and any changes that occur to the record must be recorded.

When selecting a technology for the regulatory analysis of food and drugs, it is extremely important to take into consideration both the analytical performance requirements the instrument must meet and its effective record keeping capabilities. The appropriate instrument will be capable of fulfilling all specifications in order to help manufacturers adhere to the strict global regulations outlined above. Inductively Coupled Plasma (ICP) emission spectroscopy is the ideal method for accurate trace element analysis of toxic elements in food and pharmaceutical products ensuring regulatory compliance. Coupled to a Charge Injection Device (CID) detector, ICP emission spectroscopy achieves non-blooming operation and a wide dynamic range of +8 orders of magnitude. This makes it possible to read both high and low concentrations within the same analytical run. For example, the analysis of sodium in food products, which is normally at low percent levels, can be performed at the same time as the analysis of lead, which is present at sub parts per million (ppt) concentrations.

Experimental

A Thermo Scientific iCAP 6500 Duo ICP emission spectrometer was chosen for the analysis of toxic elements in foodstuffs. This ICP model features full Mass Flow Control of all gases for enhanced stability and advanced software features including source optimization. A standard sample introduction kit was utilized for this application. Sample digestion was processed using a microwave lab station with high-pressure rotor, temperature and pressure monitoring. The instrument parameters employed during the analysis are listed in Table 1.

Parameter	Setting
Sample Pump Winding	Orange/white tygon
Pump rate	50 rpm
Nebulizer	Standard concentric
Nebulizer Argon Pressure	0.6 L/min
Spray Chamber	Standard cyclonic
Centre tube	2.0 mm
Torch Orientation	Duo
RF Forward Power	1150 W
Purge Gas	Argon
Coolant flow	12 L/min
Auxiliary flow	0.5 L/min

Table 1: Instrument Parameters

Nitric Acid (HNO₃), sg 1.42, AnalaR grade and 1000 ppm single element standards for Cu, iron (Fe), manganese (Mn), Pb, tin (Sn), strontium (Sr) and zinc (Zn) were used. 1000 ppm yttrium (Y)

single element standard was used to prepare the Internal Standard. Standard reference materials which included skim milk powder (ARC/CL), Japanese diet (NIES CRM, no.27), poultry feed (LGC7173) and tomato paste (ERM – BC084a).

Samples were weighed out and allowed to dry demonstrating a final weight of around 0.5g. 5 ml of HNO₃ was then added before samples were microwave digested. The samples were allowed to cool and made up to 25 ml with deionized water. High purity standards were used to prepare the calibration standards for this method. They were then acid matched to the samples (20% HNO₃). Table 2 indicates the concentration of each of the standards, which was selected to cover the linear range of the samples. All samples and standards were analyzed with an Internal Standard of 5 ppm Y solution. This was added automatically using the Internal Standard Mixing Kit, p/n 8423 120 51551. By connecting an additional pump tube and adding the Internal Standard on-line, continuous accurate dilution of the sample was assured.

Element	Concentration in ppb
Cu, Fe, Mn, Sr	0, 10, 50, 100
Fe	0, 50, 250, 500
Pb, Sn	0, 10, 50, 500
Zn	0, 250, 500, 1000

Table 2: Standard concentrations

As the samples comprised different matrices, an Internal Standard was employed to act as a dynamic correction for suppression/enhancement of signals due to viscosity, matrix or sample transport differences. iTEVA Security analytical software (Thermo Fisher Scientific) was set up to automatically correct all signals by referencing appropriate Y lines to those method wavelengths demonstrated in Figure 1. All low method wavelengths were referenced to Y 224.306 nm and all high method wavelengths were referenced to Y 324.228 nm.

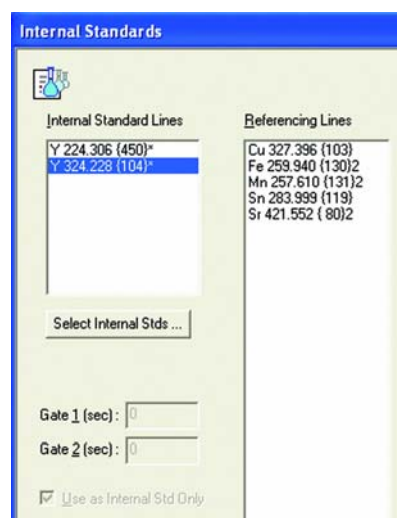


Figure 1: Internal Standard referencing screenshot from iTEVA

The method was then calibrated and all samples were analyzed in a single run, with a calibration blank analyzed using 10 replicates and standard method settings (15 second integration for low wavelengths and 5 second integration for high wavelengths) to determine a Detection Limit. The sub-array peaks for each element were examined to ensure that they were interferent free and the correct background points were employed (Figure 2). By overlaying all the sub-arrays, it was possible to determine if there were any overlaps/interferents and identify any differences between samples and calibration standards.

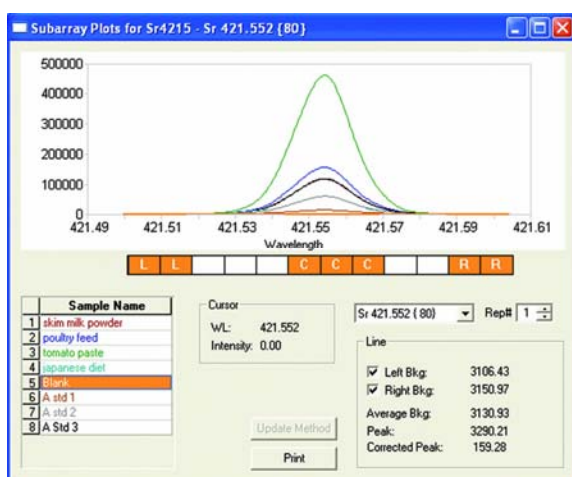


Figure 2: Strontium 421.552 nm sub-arrays for all samples

Results

A Fullframe was performed on the poultry feed sample which displayed a graphical presentation of the spectrum recorded by the CID detector and was able to identify all the elements present in the sample. The bright spots on the image are indicative of the presence of an element while the position of the spots shows the wavelength of the element. The brighter the spot, the more intense the concentration. Fullframe is very useful for true unknown analysis as it can be used for qualitative analysis and also for semi quantitative analysis with results being typically within 15% of the full quantitative result. From the Fullframe shown in Figure 3, the method elements are clearly identified with the colored boxes that relate to the legend on the right hand side of the image. Any other elements can be identified using the Wavelength Finder function, which is available by clicking on the element of interest. From the poultry feed Fullframe, the presence of calcium (Ca) at % levels was obvious (circled in red) in addition to the method elements.

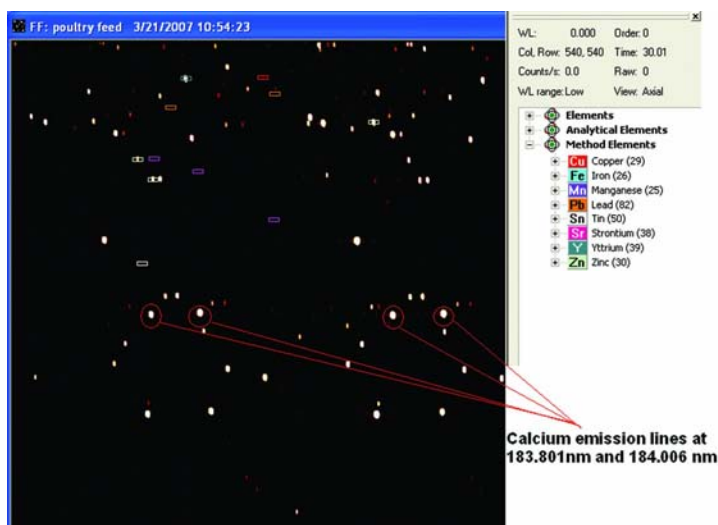


Figure 3: Fullframe of poultry feed sample

The detection limits determined show that ICP emission spectroscopy is ideal for most food and medicine testing requirements. As an example, the Indian Standards set by AYUSH for Pb is 10 ppb, which is more than ten times above the detection limit of 0.722 ppb gained through this experiment. Furthermore, the iTEVA Security analytical software, which is FDA 21 CFR part 11 compliant, enabled recording of the analysis and modification of all electronic records with a time, date and user stamp.

Conclusions

The presence of toxic elements in foodstuffs, dietary supplements and pharmaceuticals is strictly regulated worldwide with industry standards being largely based upon ISO FDIS 22000 and with the HACCP (Hazard Analysis and Critical Control Point) being a popular control system used by food and drug organizations. ICP emission spectroscopy coupled to a CID detector provides a powerful, accurate and efficient tool for trace element analysis of toxic elements in a variety of foodstuffs and related products. The use of a microwave provides easy sample preparation with a common technique applicable to many different matrices. Method development is made easy with the use of an automatic Internal Standard correction to overcome the effects of different matrices.

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