K4 Elemental Analysis of Hair and Tissue by Open Vessel Digestion and Inductively Coupled Plasma/Mass Spectrometry (ICP/MS) Analysis

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Learning Overview: After attending this presentation, attendees will have gained knowledge about a newly developed sample preparation method for the analysis of hair and tissue samples using an open vessel acid digestion method followed by ICP/MS analysis.

Impact on the Forensic Science Community: This presentation will impact the forensic science community by introducing a method of acid digestion that provides the opportunity for greater sample throughput and the potential for less contamination compared to closed vessel microwave systems.

Trace metal analysis is highly important in both forensic and clinical toxicology. Metals analysis can play a key role in death and poisoning investigations as well as in environmental and workplace exposure monitoring. Blood, serum, and urine are typical matrices used for analysis. However, in some cases, it is necessary to analyze non-aqueous matrices, such as hair, nails, and tissues. This study introduces a method in which non-aqueous samples are digested into an aqueous solution utilizing a series of chemicals and a heating block, and then analyzed by ICP/MS. Approximately 0.3–1.0 grams of solid sample is weighed into pre-cleaned disposable tubes for the digestion process. Minimization of contamination is key in trace metals analysis for proper identification of toxicological results without the influence of pre-analytical variables and, in this method, digestion tubes are pre-cleaned to remove possible contaminants. These digestion vessels are not recycled as in microwave procedures. An acid digestion mixture composed of 4% hydrochloric, 23% nitric, and 1% hydrofluoric acids is added to each tube, followed by the addition of hydrogen peroxide. The sample is then placed into the heating block that has a 47-sample capacity for digestion, which runs 3 hours and 35 minutes. Upon completion, the sample is diluted to a final volume of 25 milliliters with 2.0% nitric acid. A quantitative method using quadrupole ICP/MS with collision cell technology is used to detect 14 elements. These elements are chromium, manganese, cobalt, nickel, arsenic, selenium, molybdenum, cadmium, tin, antimony, barium, thallium, lead, and bismuth. This presentation will focus solely on barium, arsenic, lead, and cadmium in hair.

Precision and bias estimates were tested for all elements using certified reference materials, including hair, mussel, scallion, celery, dogfish liver, and synthetic urine and blood. Precision as % Coefficient of Variation (CV) for the total digest procedure was less than 15% for all certified reference materials. The accuracy for the total digestion procedure was determined to be within +/-15%. The method uses aqueous standard calibration curves for each analyte of interest. The curves for all elements pass an acceptance criteria in which the slope of the back-calculated concentrations to the target concentrations during validation fell between 0.85 to 1.15 and the bias was within 20%. The relationship between the measured and expected values was also plotted and established to be linear for all elements within the analytical measurement range.

Cases where this method was used include hair analyses over a period of one year from individuals with unknown histories of metals exposures. The findings were: for cadmium (n=10), results ranged from 0.018mcg/g to 0.57mcg/g with a median of 0.10mcg/g and average of 0.20mcg/g; for barium (n=13), results ranged from 1.6mcg/g to 8.4mcg/g with a median of 4.9mcg/g and average of 4.8mcg/g; for arsenic (n=5), results ranged from 0.22mcg/g to 1.2mcg/g with a median of 0.85mcg/g and average of 0.78mcg/g; and for lead (n=36), results ranged from 0.14mcg/g to 3.8mcg/g with a median of 0.68mcg/g and average of 0.90mcg/g. Results from hair analysis can help differentiate between baseline levels of trace metals and overexposure or poisoning situations.

In conclusion, open vessel hot block digestion provides a suitable process in which non-aqueous matrices are prepared for ICP/MS analysis. The results from these analyses may assist with the assessment of trace metal determinations in alternative specimens of toxicological interest.

Metals Analysis, Digestion, ICP/MS

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