Procedures used by Queensland Department of Primary Industry (Animal Research Institute) for Trace Metal Analysis of Biological Samples

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Apparatus

All analyses were carried out on a standard Perkin-Elmer Sciex ELAN™ 5000 Inductively Coupled Plasma-Mass Spectrometer (ICP-MS).

Materials and Methods

All plastic-ware used for the preparation of standard solutions and for dilution and storage of sample digests was soaked in nitric acid (10%) for a minimum of 48 hours. All items were then rinsed three times using reverse osmosis (RO) prepared water followed by three further rinsings with polished reverse osmosis (ROP) prepared water (18M Ω).

All borosilicate glass volumetric flasks were fitted with PTFE stoppers. These flasks and the PTFE beakers were refluxed with HNO_3 (conc.) for eight hours, allowed to cool, rinsed three times with RO water and then soaked and cleaned as per plastic-ware.

Mixed multi-elemental standard solutions were prepared from 1000 ng/L stock solutions. Aluminium standards were prepared separately in TPX (polymethylpentane) volumetric flasks. The adsorption/release equilibriums of glass with aluminium in solution make low level determinations of this element in glass highly inaccurate. Tin standards were also prepared separately in glass, daily from concentrate.

HNO_3

Nitric acid was purified by sub-boiling double distillation of reagent grade feedstocks in quartz stills.

Sample Preparation

Sample dissolution was achieved using a HNO_3 microwave assisted digestion. The system used a was a Microwave Laboratory Systems MLS 1200 manufactured by MILESTONE, Italy.

Nitric acid was specially prepared by double distillation of AR grade acid in sub-boiling point quartz stills.

All samples were freeze-dried and ground to a fine powder to achieve an homogeneous final product. One hundred to two hundred milligrams of this material was accurately weighed into a TFM insert of the microwave digestion system. HNO_3 (4 millimetres) was added and the vessels sealed and placed in the microwave oven. The oven program used was:-

Step (1)	250 watts for 8 minutes
Step (2)	400 watts for 4 minutes
Step (3)	250 watts for 4 minutes

It should be noted that 250 watts power with this system is a continuous energy output which results in more even and controlled heating producing a gradual pressure increase to a maximum of 30 bar.

The vessels were then removed from the oven and cooled in an ice-bath for a minimum of one hour. This step is necessary to avoid losses of sample as an aerosol upon opening of the vessels. The sample solution is then transferred to a PTFE beaker and made up to 10.0 grams with ROP water. 3.0 grams of this solution is transferred to a polypropylene tube. This tube is set aside for mercury and tin determinations.

The remaining 7.0 grams of sample solution in the beaker is taken to near dryness on a ceramic hotplate at 90°C. A further 2 millimetres of HNO_3 and 2 millimetres of H_2O_2 is added dropwise and again taken to near dryness. This step is included to ensure complete digestion and to remove volatile interfering matrix components. The digestion solution is washed into a 50 millimetres polypropylene tube using 1% HNO_3 and accurately made up to 20.0 grams. This tube is used for solution nebulization ICP-MS.

Running Procedures

Each set of digestions contain 8 samples, a standard reference material (SRM) and either a blank or a duplicate sample. This is maintained for all samples that are digested using the above procedure.

The samples are run on the ICP-MS using solution nebulization. A set of calibration standards are run and then the digest solutions including SRM's, blanks and duplicate samples. For the purposes of the method all readings must lie between the lowest and highest standard concentration on the calibration curve. If readings are below the reading of the lowest standard concentration then a more concentrated solution must be run and if the readings are above the highest concentration a dilution of the sample must be run.

All standard concentrations must be within the linear dynamic range on the calibration curve. Calibration check solutions and blanks are also analysed in the run sequence and the instrument is recalibrated by reanalysing all standard solutions every 3 hours.

This method of analysis exceeds the guidelines set out in our quality control manual accredited by the National Association of Testing Authorities, Australia (NATA).