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METHOD STATEMENT METALS

INTRODUCTION

The performance of this method is validated in accordance with internationally recognised procedures.

The analytical procedure described herein outlines the determination of metals in soils, waters, filters, probe washes and impingers by Inductively Coupled Plasma – Optical Emission Spectrophotometry (ICP-OES) and Inductively Coupled Plasma – Mass Spectrometry (ICP-MS).

Soils are accredited to MCERTs for most common metals. Waters, filters and other matrices are ISO17025 (UKAS) accredited. The laboratory holds flexible scope for metals analysis, so please enquire about exact requirements.

PRINCIPLE

ICP-OES (LESS SENSITIVE, FOR SOIL DIGESTS, WASTE WATERS, TYPICALLY LOW MG/L, MG/KG FOR SOILS)

An acidified solution is aspirated into an argon plasma created by induction from a radio frequency coil around it. The high energy from the plasma (circa 6000 °C) is absorbed by the ions from the solution as it passes through. This causes the electrons contained in the ions to shift to higher energy states. When the electrons drop back to lower energy states, the ions emit radiation at wavelengths characteristic of the elements present. These emissions are focused onto a charge coupled detector (CCD), where their intensities are measured. The intensity for each element corresponds to the concentration present and is used to calibrate the instrument using standards of known concentration.

ICP-MS (HIGHER SENSITIVITY, FOR CLEAN WATERS, TYPICALLY µG/L LEVELS)

The same principle applies as in ICP-OES, except that the torch conditions are optimised to create ions with a single positive charge. These mono-positively charged ions are focused into a beam by a series of lenses. This ion beam then passes through a mass spectrometer that selects ions based on their mass to charge ratio (m/z). The selected ions then hit an electron multiplier, where



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positive ions displace electrons from the surface. These electrons then go on to disturb more electrons, creating a multiplication effect. The overall charge detected by the impact of the ions from a specific element corresponds to the concentration present and is used to calibrate the instrument using standards of known concentration. Kinetic energy displacement (KED) is used to minimise interferences from elements that form polyatomic molecules e.g. Ar-Cl at mass 75, the same as Arsenic.

Matrices

Soil samples and most filters are digested using nitric / hydrochloric acid. These are normally analysed by ICP-OES.

Water samples can be filtered through a 0.45 micron filter then acidified (filtered), or digested by heating with acid (unfiltered / total). For water samples to be classified as dissolved, they are required to be filtered and preserved in nitric acid on site. Water samples can be analysed by ICP-OES or ICP-MS depending on the source and the regulatory limits required by the customer. Leachates are tested as filtered waters.

Stack emission samples including filters, probe washes and impingers are tested by ICP-MS. Filters and probe washes are extracted using hydrofluoric acid and microwave digestion. Impingers are not extracted before analysis.

Performance Characteristics

Substances Determined include, but are not limited the following:-

Aluminium, Antimony, Arsenic, Barium, Beryllium, Cadmium, Calcium, Chromium, Cobalt, Copper, Iron, Lead, Magnesium, Manganese, Mercury, Molybdenum, Nickel, Potassium, Selenium, Sodium, Thallium, Tin, Vanadium, Zinc and Sulphur / Sulphate.

Range of Application (extendable by dilution)

- Soil samples 1 to 1000 mg/kg (depending upon specific element)
- Aqueous samples 0.001 to 200 mg/L (depending upon specific element)
- Filters and probe washes 1 to 1000 μ g (depending upon specific element)



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- Impingers 1 to 250 µg/L (depending upon specific element)

Limit of Detection

- Soil samples circa 1 mg/kg (depending upon specific element)
- Aqueous samples circa 1 µg/L (depending upon specific element)
- Filters and probe washes circa 1 µg (depending upon specific element)
- Impingers circa 1 µg/L (depending upon specific element)

Analytical Quality Control

Analytical quality control is maintained by a number of measures:

- Multi-point calibration with authentic standards (with defined minimum performance characteristics)
- Analysis of control samples within each analytical batch, such as independent standards, matrix spikes or reference materials
- Analysis of reagent / method blanks within each analytical batch
- Ongoing quality assured by the use of control charts in conjunction with warning and action limits for the QC sample data
- Participation in external proficiency testing and inter-laboratory schemes such as LGC CONTEST, AQUACHECK and HSE WASP

References

A Handbook of Inductively Coupled Plasma Spectrometry. M. Thompson, J.N. Walsh. 1983. ISBN 0-412-00371-6)

National Institute for Occupational Safety and Health, method 7300, trace metals by ICP-OES.