EnviroMail™

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Important Changes to the Australian Standard Leaching Procedures (AS4439.2 & AS4439.3)

Introduction

Standards Australia have published updated ASLP procedures for leaching waste sediments and contaminated soils – AS4439.2 (zero headspace procedure) and AS4439.3 (bottle leaching procedure). The main changes include specific considerations and protocols for leaching of samples for PFAS analysis and the provision for reduced sample amount where limited sample mass is submitted. This Enviromail provides clarification of the procedural updates together with the response of ALS to the changes.

Centrifugation versus Pressure Filtration for removal of particulates

The standard approach to removing particulates from the leachate is pressure filtration through glass fibre filter papers of effective pore size 0.6 to 0.8 µm. The issue raised in the standard around pressure filtration is the potential for the filter material to retain PFAS. ALS has assessed glass fibre filter papers and demonstrated retention of long-chain acids and particular sulfonamides.

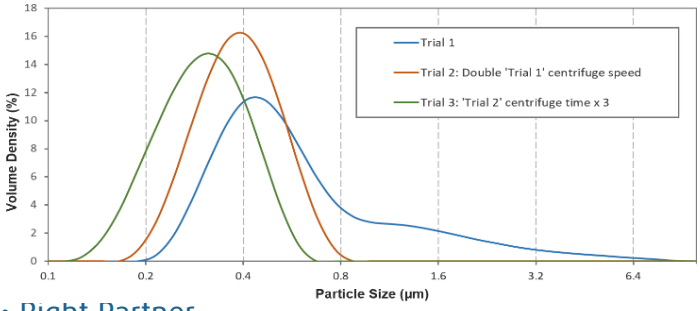
Pressure filtration devices can also represent a source of cross contamination even where rigorous cleaning protocols are adopted between samples. Centrifugation eliminates this process as a potential source of contamination by employing single use centrifuge vessels. **ALS has adopted centrifugation over pressure filtration for leaches requiring PFAS analysis.**

Centrifuge Conditions

A shortfall in the standards is their failure to prescribe or provide guidance for centrifugation. Particle settling by centrifugation is dependent on the speed of rotation (dictating the exerted g-force) and time. Selection of these parameters will have a substantial impact on the concentration of analytes of interest in the analysed solution, particularly where particle-bound concentrations are significant. The below figure demonstrates how centrifuging the same leachate under different conditions can result in prominent differences in particle size distribution in the resulting solution.

This highlights the importance of not merely selecting arbitrary centrifugation conditions, but targeting conditions to best approximate the outcome of filtering, thereby eliminating either negative or positive bias in the corresponding analytical data.

ALS has invested significant effort in deriving centrifuge conditions using Stoke's Law. Stoke's Law describes the settling velocities of spherical particles in a fluid medium. Using this approach, ALS has developed a means of determining the centrifuge conditions (rpm and time) aimed at targeting the settling of particles down to 0.7 µm (representing the midpoint of the nominated pore size for pressure filtration).



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Separate Leaches for Semi-Volatile Organics and PFAS – Incompatible Materials

Discrepancies between materials considered compliant for semi-volatile organics versus PFAS means these tests must be leached separately. The compliant material for sampling containers and leaching vessels for semi-volatile organics is glass with PTFE-lined lids (to avoid contact with plastic lids). Conversely, glass is discouraged for PFAS due to adsorption concerns (WA DER, PFAS NEMP 2.0, AS4439.2/AS4439.3, etc). As a result, the new standard stipulates PFAS be leached in a separate fit-for-purpose container (AS4439.3, Section 6.3).

Where semi-volatile organics and PFAS are requested on the same sample, ALS will perform two separate leaches, taken from compliant ALS sample containers. Whilst this approach will represent a second leach charge per sample, it is viewed as the appropriate approach to provide quality analytical data. Where samples require leaching for metals and PFAS OR metals and semi-volatile organics, a single leach procedure is appropriate as metals analysis can be undertaken from either glass or plastic sample workflows.

Minimal "Limited Sample Mass" Submissions

The minimum sample mass considered to be a representative subsample according to the bottle leaching procedure (AS4439.3) is 100 g solid material, where particle size is reduced to <2.4 mm (as required). The new standard provides provision for reduced sample mass leachates where limited sample mass is submitted based on further reduced particle size. **It is however important to note this option is not discretionary for labs to justify scaled down leaches as a default approach.** The standard stipulates particle size reduction "shall not be undertaken to facilitate a limited sample mass solids leachate" and that the "sample mass for solids leachate preparation shall always be the maximum mass possible" (AS4439.3, section 7.4.3)". ALS sample containers are appropriately sized to ensure the maximum mass is always collected.

Relation to the Toxicity Characteristic Leaching Procedure (TCLP)

The PFAS NEMP (2.0) refers to ASLP as the leaching procedure for PFAS. The US EPA's Toxicity Leaching Characteristic Leaching Procedure (TCLP) has yet to be updated with specific instructions for leaching PFAS. Given the updates to the Australian Standards (AS4439.2/AS4439.3) represent best practice with regards to leaching for PFAS, ALS will adopt these procedures (namely the use of plastic leaching vessels and centrifugation rather than pressure filtration) where the TCLP is requested for PFAS.

References

AS4439.2-2019: Wastes, sediments and contaminated soils. Part 2: Preparation of leachates – Zero headspace procedure.

AS4439.3-2019: Wastes, sediments and contaminated soils. Part 3: Preparation of leachates – Bottle leaching procedure.

PFAS National Environmental Management Plan (NEMP), Heads of EPAs Australian & New Zealand, January 2020 (Version 2.0).

Toxicity Characteristic Leaching Procedure (TCLP), US EPA Method 1311.

WA Department of Environment Regulation (DER) – Interim Guideline on the Assessment and Management of Perfluoroalkyl and Polyfluoroalkyl Substances (PFAS), January 2017 (Version 2.1).