Laboratory analysis of lead

Bioavailability in soil and total lead in paint

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Lead Bioavailability in Soil - Outline

- Background
- How Lead Bioavailability is determined in the lab
- Different method options
- Example data

Background

- Why is Lead Bioavailability in soil important? NES assumes 100% bioavailability of contaminants. What is the real risk?
- Bioavailability defined as “the proportion of a substance that is absorbed from soil in the digestive system into the body.”
  - Determined in vivo (within the living)
  - Not popular due to ethical issues
  - Very expensive time-consuming and complicated
- Bioaccessibility defined as “the proportion of a contaminant that can be extracted under simulated digestive conditions.”
  - Determined in vitro (within the glass)
  - Bioaccessibility = $\frac{\text{(Gastric extraction result, mg/kg)} \times 100}{\text{(Total Recoverable result, mg/kg)}}$
  - NOTE: both results from <250µm soil fraction!
- Bioavailability can be estimated from in vitro bioaccessibility data using a calibration model

Lead Bioaccessibility determination

- Reference Method is Solubility/Bioavailability Research Consortium (SBRC) Stomach Phase Extraction (SBRC-G, Kelly et al, 2002).
- Note that there is also an SBRC-Intestinal phase method but we don't have this set up (different simulated gastric fluid, pH and time, but still at 37°C).
- IANZ accreditation achieved Feb 2017.

Summary of Method

1. Soil samples are dried and sieve to <2mm, and then further sieved to <250µm.

   (The <250um fraction is used as this is the portion of soil that adheres to children's hands, and is then potentially ingested through hand to mouth activities).

1. 1.00 ± 0.05g of the dried <250µm sample is weighed into a container
2. 100 ± 0.5mL of gastric extraction fluid is added (glycine in water solution adjusted to pH 1.5 with HCl at a temperature of 37°C).
3. Shake on orbital shaker for 60±5 minutes at 200±2rpm at a temperature of 37°C

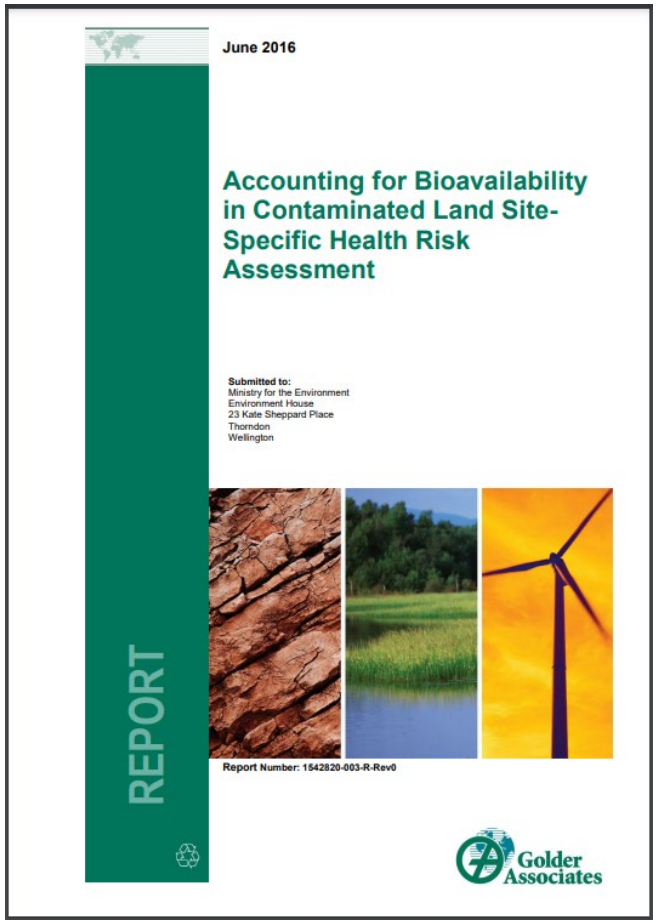
   (note that the reference method uses an end-over-end shaker but our validation data shows the orbital shaker works just as well).

Summary of Method (continued)

5. Filter through 0.45µm
   (must be completed within 1hr30min otherwise we have to start again).
5. Measure pH, if the fluid pH is not within ±0.5 pH units of the starting pH of the fluid then the test must be discarded and the sample re-analysed.
6. Analyse by ICPMS.

Different method options

- Golder Associates identified ten different in vitro bioaccessibility methods available (MfE, June 2016).
- Many are complex and simulate bioaccessibility from saliva, gastric, and intestinal fluid.
- Golder concluded: “the gastric phase test developed by the Solubility/Bioavailability Research Consortium (SBRC-G) meets relevant scientific, economic and social assessment criteria.”



Example Data

Gastric Extractable Arsenic mg/kg dry wt (<250µm fraction)	Total Recoverable Arsenic mg/kg dry wt (<250µm fraction)	In Vitro BioAccessibility (%)
1.2	33	3.6
< 1.0	38	< 2.6
1.1	39	2.8
1.1	37	3.0

Lead in Paint - Outline

- Hazardous Lead Paint - Background
- The Method - How is lead tested in the laboratory.
- Considerations.
- Setting the Standard - Future Improvements by Hill-Labs.

Background

- Management of hazardous lead in paint (AS/NZS 4361.2:2017).
- MOH – Guidelines for the Management of Lead-based Paint.
- Extensive use of lead based paints in the 1960s.
- Buildings containing lead paint or older layers of lead paint.
- Lead paint is a primary cause of lead poisoning cases.
- Specifically for children and pets.
- Identification – (XRF, Kits and Lab testing).

Method

- Based on a modified US.EPA method.
- Weigh 0.1g -0.5g of sample in to a digestion vessel.
- Acid is added - mixture of nitric and hydrochloric acid.
- Place on a hot block at an elevated temperature for 50mins (Digestion).
- Large dilution with dilute acid.
- Analyse on an inductively coupled plasma mass spectrometer (ICP-MS).

Method (continued)

- Quality Controls. (procedural blanks, matrix QC, reference material).
- QC performance is assessed (approval procedure).
- Report is then generated to be sent to client.
- Report is based on AS/NZS 4361.2:2017

Example Report

Lead in paint				
Total Recoverable Lead	mg/kg dry wt	11,500	1,340	490
Total Recoverable Lead	% w/w	1.15	0.134	0.049
Paint classification		Lead Paint	Lead Paint	Lead-free Paint

Comment:
The accuracy of the paint classification may be affected by the sampling strategy and procedure employed. Please ensure that the sampling strategy and procedure outlined in section A4 and section A3.2.2, respectively of AS/NZS 4361.2:2017 have been followed before interpreting these results. The classification of the paint has been determined using a decision rule that treats all values as fixed, with no consideration of the Uncertainty of Measurement (UoM) of the analysis performed. A paint that contains greater than 0.1% lead by mass in the dry film is defined as 'Lead Paint'.

Setting the Standard

- Current methods used by NZ labs - Soil based digestion.
- Hill-Labs are pursuing IANZ accreditation.
- Method development - Labs internationally are using NIOSH based methods.
- Homogenisation of samples is a critical point that NZ labs have not adopted extensively.
- Hill - labs are looking to set a standard in lead paint testing.
- Offer IANZ accredited results.
- Provide reports that differentiate lead paints from bulk material containing lead paints
- Use appropriate international methods such as NIOSH.