REPORT OF THE

LEAD IN SOIL AND DUST

WORKING GROUP

Prepared for the

NSW GOVERNMENT LEAD TASKFORCE

January 1994

REPORT BY THE SUB-COMMITTEE ON ANALYTICAL METHODOLOGY

LEAD IN SOIL AND DUST WORKING GROUP

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INTRODUCTION

At the first meeting of the Lead in Soil and Dust Working Group, it was established that, for the determination of lead in soil and dust, there is a need for standardisation of procedures for:-

- sampling,
- sample preparation and
- analytical methodology.

This standardisation is necessary if the results are to be:-

- comparable,
- reliable,
- helpful in protecting the health of young children, and,
- the results are to withstand the scrutiny of regulating authorities.

Australian Standards (AS) Committee CH/28 (Analysis of Soils and Biota) was convened and is presently preparing a standard which concerns the sampling and analysis of soil for lead. The findings of this committee will be of great importance to the NSW Lead Strategy.

SOIL ISSUES

Design of Sampling Program

- For a meaningful assessment of lead contamination at a site, an appropriate sampling pattern producing a statistically adequate number of samples will be required.

- Statistics have to be adequate for the expected heterogeneity of the sampling site.

- Design of sampling programs will probably be site specific. For example, in the case where lead containing paints are identified as being a problem or where historical data indicate probable existence of "hot spots".

- It is recognised that the approach to sampling a site for contaminated site assessment will be quite different to the sampling of a site for health risk assessment in regard to exposure of children to lead.

- Residential sites will generally be sampled in a different manner to industrial sites.

- The Queensland Government Chemical Laboratory provides a guide to sampling strategy in their publication "Guidelines for Soil Sampling". In the absence of more specific advice and where the contaminant distribution is uniform, for example in the case of areas up to 0.1 ha, a grid size of 15 m and 6 samples is recommended.

The foregoing suggests that it will be difficult to recommend blanket sampling patterns and appropriate sample numbers for site assessment purposes.

It was decided to assess the design used in other Australian studies (Port Pirie, Broken Hill, Wollongong and the Hunter region).

Three common methods for selecting sample locations are:-

- **Judgemental selection** of sample points is based on a validated site history and also on the basis of a site inspection with respect for example to the area where children play.

- **Grid sampling** is used to cover the whole site and when there is little site history to guide the judgmental approach.

- **Random sampling** may lead to a clustering of points with consequent inadequate coverage of the whole site. Stratified random sampling, where the site is divided into cells with randomly chosen locations within each cell, is therefore preferred.

- Often a combination of **judgmental** and *either* **grid** or **random** sampling produces the most efficient sampling pattern for the site.

- AS CH/28 at their meeting on 17th September 1993 formed a working group to "produce a more succinct document as a base document for the soil quality sampling....This new draft should include guidance to an analysis of risk assessment.". This working group includes a statistician from CSIRO.

The LSDWG acknowledges that there are different sampling programs appropriate for assessment of lead in soils and recommends that the sampling program design follow recommended procedures to be produced by Australian Standards CH/28.

What sample to take?

- It was recognised that in terms of risk to human health it is the metals contained in the first few centimetres of the soil that people are largely exposed to by way of direct soil ingestion (particularly by children) and potential soil dust inhalation.

- It was recognised that for health assessment purposes, the upper layers of the soil should be sampled.

- Studies have used various sampling depths up to 200mm.

  * "A core sample is taken to a depth of 15 - 20 cm and the top 2 cm are used to produce a sample of nominally 10 g" - USEPA.

  * O - 50 mm, 50 - 100 mm and 100 - 200 mm layers have been sampled (Port Pirie).

  * Studies at Wollongong have taken soil from upper 0 - 100 mm, with sub-sampling of the top 20 mm.

  * 0 - 50 mm was used for studies at Newcastle

- Either the upper 20 mm or 50 mm was seen as an appropriate sampling depth.

- It is recognised that the upper 50 mm sample is easier to take and thus can be expected to yield more reproductable results.

- However, AS CH/28 probably favours the upper 20 mm sample rather than the upper 50 mm.

- In cases of children's health assessment there may be obvious samples to take, e.g. sand pit samples.

It was resolved that it was appropriate for AS CH/28 to continue to deliberate on the matter of sampling depth and that their eventual recommendation be accepted for the NSW Lead Strategy.

What to do with the sample (sample preparation)?

- Once a soil sample is taken, it seemed the most appropriate treatment was firstly to dry and sieve the sample to less than 2 mm (to obtain the "fine earth fraction" of the soil sample). 2 mm particles attach to clothing etc.

- It was felt that for a meaningful health assessment the separation and analysis of a fine particle faction of the soil sample (the 'ingestible' and 'inhalable' size fraction), - this size may be less than 200, 150 or 100 um. Less than 200 um represents soil "stickability" to fingers etc.

- It was also recognised that the coarser material of the less than 2 mm sample may contain significant lead as for example paint flakes so it was thought the less than 2 mm sample should be pulverised and analysed as well.

- Suggested sample preparation scheme - after drying and sieving to less than 2 mm, a sub-sample is pulverised for analysis and a further sub-sample is sieved to separate a finer fraction, say 100 micron, and this sample is analysed as well.

- There was considerable debate at one of the AS CH/28 meetings regarding the particle size which should be analysed as part of a health risk assessment - health experts favour - 100 um because this fraction is ingestible.

- Dr P Mushak (US expert) recommended the less than 100 um size fraction for soil and dust.

- The scheme suggested at the last LSDWG meeting (see above) is similar to that under consideration by AS CH/28.

It was resolved that it was appropriate for AS CH/28 to continue to deliberate on the matter of sample preparation and that their eventual recommendation be accepted for the NSW Lead Agency.

Analysis of Samples

- "Total" lead - using either a decomposition procedure that dissolves the silicate matrix of a soil (procedures using hydrofluoric acid or fluxes) or an X-ray based technique.

- "Extractive" lead is that lead solubilised using a specific digestion technique e.g. aqua regia, nitric acid/perchloric acid, nitric acid/hydrogen peroxide, dilute hydrochloric acid.

- The proportion of the total lead content extracted is a function of the digestion technique. This is the basis of predictive and quantitative tests for lead content.

Note: It is recognised that many of the "extractive" procedures (nitric/perchloric or aqua regia) give results that in many cases that are very similar to those obtained by the more rigorous (and time consuming and more expensive) "total" extraction procedure. For example, in the nitric acid/perchloric acid, the aqua regia and the nitric acid/hydrogen peroxide extraction procedures, the lead content measure is the

total lead except for variable proportions that bound in the silicate matrix. It is recognised that the silicate bound lead content is probably not "bioavailable" and so these mineral acid digestions are adequate for the determination of lead in soil and dust for assessment purposes.

Any of the extraction methods involving nitric acid (i.e. nitric acid, aqua regia, nitric acid and perchloric acid mixtures, or nitric acid and hydrogen peroxide mixtures) appear to be suitable for dissolution of the lead content in soils except for silicate bound lead content.

. Extraction methods will be specified by standard produced by AS CH/28

. The standard from the AS CH/28 is expected to be available during 1994.

Note: It is also important to consider the potential non-homogenous nature of some soil and dust samples. The size of the aliquots taken for analysis and the number of replicate assay's must be such that the inconsistent distribution of lead in some soil samples does not lead to inaccurate and misleading results.

Bioavailability

. It is recognised that this is probably the most important parameter that should be measured.

. Although there may be suitable methods which are good for predictive purposes, there appears to be no general agreement and so a suitable standard is not available.

. Methods for "Bioavailable" lead vary from country to country and from laboratory to laboratory.

. USEPA recognises that "further animal feeding studies and epidemiological studies are needed to assess the bioavailability of lead from soil and dust".

. It is recognised that, from the outset, the bioavailability is probably a function of surface area (among other things) and so the lead content of fine material may be of high toxicological significance.

. There is a need to correlate lead source with bioavailability and its pathway and design appropriate rehabilitation methods.

. Further research should be directed in this direction.

. Bioavailability will not be addressed by AS CH/28.

DUST ISSUES

Lead in house dust is recognised as one of the best predictors of childhood lead poisoning, however techniques for house dust sampling have not been standardised, because quantitative, meaningful sampling is not straightforward.

There are presently more than fifteen unstandardised house dust sampling techniques described in the literature. USEPA has recognised these problems and has commissioned a study to help USEPA to proceed towards standardising lead in dust sampling techniques.

Two measurements of lead are generally used when dust is considered. They are lead loading (micrograms of lead per square metre) and lead concentration (micrograms of lead per gram of dust). It is generally thought that measures of dust lead loadings (i.e. the amount of lead on a given surface that may come in contact with a child), more directly measure lead available for a child to ingest and so better predict children's blood levels than do dust lead concentrations (the amount of lead in the dust).

Unfortunately, lead loadings are often difficult to measure, either accurately or precisely. Dust lead concentration measurements are generally more accurate and precise than lead loading measurements, and while lead concentration may not predict a child's blood lead level, high concentration values may indicate that sources of lead (e.g. contaminated soil deteriorating lead paint) may be present.

The LSDWG decided that only settled dust (i.e. dust deposited on a surface) is considered. Entrained dust in household air is another issue, which has been referred to the Lead in Air Working Group.

Other points to note:

- The USEPA recommends sampling all hard surfaces, carpets, upholstered furniture, play areas and the entry way (for collection of exterior dust).

- While dust loading (i.e. grams of dust per square metre of surface per day/week/month) is more toxicologically significant, dust deposition rate (i.e, grams of dust per square metre per day/week/month) is also a valuable parameter for assessment purposes.

Port Pirie Dust Sampling Techniques

Simple brushing of accumulated dust - In the Port Pirie study, dust samples were collected for surfaces of furniture, floors, and windowsills where there was sufficient for analysis (approx. 10 g). In the instances where there was little accumulated dust, it was first brushed together and scooped with stainless steel spatula.

Plastic cup sampling (Port Pirie study) - cups left uncovered for 1 - 2 months at height of 1 - 2 m (on top of bookcases, refrigerators, wardrobes). This procedure can measure the mass of dust as well as the lead content.

"AH" stick sampling technique (Port Pirie study) - clear adhesive tape fixed to wooden tongue depressor is rubbed over surface to collect sample for Scanning Electron Microscopy (SEM) analysis. Specialised for SEM analysis - probably able to be used for lead determination in adhered dust after nitric/perchloric digestion.

Ceiling dusts (Port Pirie study) - 250 ml plastic container (100 g) of dust collected by scaping container across the surface.

Methods of Dust Sampling

Three broad categories of dust sampling are used, namely wipe methods, dust fall methods and powered vacuum methods.

1. Wipe Methods

These methods consist of wiping over a known surface area using a medium which collects deposited dust particles. The wipes used by various researchers including paper towels, filter paper, gauze pads, polythene filters, adhesive paper labels and adhesive cloth. Various methods of wiping over surfaces have been proposed, however, these methods can generally only be used for lead loading measurements and are normally imprecise and inaccurate.

Wet wipe sampling (mixed cellulose ester MCE filter bag moistened with 2% nitric acid) is preferred. However, a wet wipe is ineffective for sampling dust from carpets and upholstered furniture, and the procedure does not measure the mass of dust.

2. Dustfall Methods

These methods consist of capturing deposited dust in a suitable container over a period of time. Both lead loading and concentration measurements can be made. Good reproductibility and accuracy can be attained, however, the time taken for deposition (e.g. one month) is a disadvantage.

3. Powered Vacuum Methods

It is recognised that use of normal domestic vacuum cleaners to obtain samples of household dust is a problem in as much as entrained dust lead levels may be increased.

Commercial vacuum cleaners have been used in many studies. Some difficulties have been found in recovering of fine particles (e.g. <150 um) using some commercial vacuum cleaners. There are added problems such as uncollected fine dust particles are made airborne, and decontamination of vacuum cleaners is difficult.

It is recognised that a suitably designed vacuum cleaner with adequate filters could be of use in sampling dust. Personal air monitoring pumps have been used to collect samples.

It should be noted that all present sampling techniques have limitations and that results obtained from one sampling technique are not applicable to another technique for comparison purposes.

Analysis of Dust Samples

Analytical procedures are generally the same as those for soil - there is a Australian Standard which is probably suitable for analysis of dust samples, namely,

. AS 2800 - 1985 Ambient Air - Determination of Particulate Lead - HIGH VOLUME SAMPLER GRAVIMETRIC COLLECTION - FLAME ATOMIC ABSORPTION SPECTROMETRIC METHOD

Key Dust Issues

. No standardised house dust lead sampling techniques are available, although many house dust sampling techniques are described in the literature.

. Given that house dust lead is one of the best predictors of childhood lead poisoning, a standardised dust sampling method is a high priority objective. USEPA is presently assessing these methods with the object of providing a standardised method.

Dust and the Household Vacuum Cleaner

. Less than 5 um dust goes through the household vacuum cleaner filter. This fraction is relevant to leaded dust sampling.

. Current vacuum cleaners in general do not have filters capable of removing lead in dust. There is a need to research filters such as the High Efficiency Particulate Accumulator (HEPA) filter. This traps 0.3 um or greater at 98% efficiency. Standard vacuums won't currently handle these filters. However there are special HEPA vacuums available.

. As a general observation, a reduction of lead in petrol won't necessarily result in an immediate reduction in lead levels in the short term due, in part, to the accumulated lead in soil and dust.

. Regular vacuuming of houses with household vacuum cleaners should not be recommended as a means of reducing the risk of childrens exposure to household dust. Two findings of work in Broken Hill are that the best correlation with

children's blood lead was the total lead content of the sub - 60 um soil fraction; the second best correlation with children's blood lead was with the frequency of vacuuming. The higher the frequency the higher the blood lead.