Brief guide to analytical methods for measuring lead in paint

IOMC
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1. Purpose and scope

This document provides a brief overview of analytical methods available for measuring lead in paint. It is primarily aimed at informing environmental health personnel and policy-makers who are not laboratory specialists but who may need to develop plans for lead paint testing and abatement programmes. This document lists well-established analytical methods for measuring lead in paint, briefly describes some of their characteristics, including their advantages and disadvantages, and highlights considerations for selecting the most appropriate method for various requirements and contexts. The document does not aim to provide an exhaustive description of analytical methods and protocols or to make specific recommendations regarding methodologies or specific instruments. Links to further information and reading are provided in section 6.

2. Background

Lead is a toxic metal whose widespread use has caused extensive environmental contamination and health problems in many parts of the world. Human exposure to lead is estimated to account for 143 000 deaths every year and 0.6% of the global burden of disease (*1*). Lead is a cumulative toxicant that affects multiple body systems, including the neurological, haematological, gastrointestinal, cardiovascular and renal systems. Children are particularly vulnerable to the neurotoxic effects of lead, and even low levels of exposure can cause serious and, in some cases, irreversible neurological damage. Childhood lead exposure is estimated to contribute to about 600 000 new cases of children with intellectual disabilities every year (*2*).

Despite recent reductions in the use of lead in petrol (gasoline), plumbing and solder, significant sources of exposure to lead still remain, including lead-based paint. Lead-based paint has been used, and is still in use in some countries, to paint the interiors and exteriors of homes and other buildings and to paint toys, furniture, playground equipment and other articles with which children can come into contact. Lead-based paint can be found in virtually every country. It is still available and in use in many parts of the world; even in countries where it has been banned for specific uses, leaded paint can be found in older houses and articles (*3*).

Non-occupational exposure to lead from paint occurs mainly in the home, from house paint that has flaked or chalked as it has aged or that has been disturbed during home maintenance and renovation. This results in the generation of lead-containing dust that can be inhaled or ingested. Soil around the home may also become contaminated. Lead-contaminated house dust has been recognized as a major contributor to the total body burden of lead in children (*4*). Young children are particularly at risk of ingestion of lead through normal hand-to-mouth activity. In addition, some children with pica persistently pick off and eat fragments of leaded paint. Young children can also be exposed through the mouthing of painted objects and toys. In some countries, lead-based paint may be the most widespread and dangerous high-dose source of lead exposure for children.

Removing existing leaded paint from housing and articles and eliminating the use of lead-based paint in residential areas and on furniture, equipment and toys are therefore important contributions to the primary prevention of lead poisoning globally. The ability to accurately determine lead concentrations in both new paint and dry paint on surfaces is a crucial requirement to guide these tasks.

3. Available analytical methods

There are three main approaches for determining the lead content in paint:

1) Test the paint for lead in situ using a chemical test kit.
2) Measure the amount of lead in paint in situ using a portable X-ray fluorescence (XRF) device.
3) Collect a paint sample and measure its lead content in a laboratory using various analytical techniques.

These approaches are described in the next sections and summarized in Table 1.

Table 1. Overview of analytical methods for lead in paint

Method	Strengths	Limitations
Chemical test kits	• Immediate results • Cheap • Relatively simple to use	• Limited accuracy • Qualitative or semiquantitative • Can test mainly top (surface) layers • May need to damage the paint surface • Difficult to observe colour change for dark paints
Portable X-ray fluorescence spectrometry	• Good accuracy • Immediate results • Low use costs • No damage to the paint surface • Rapid	• Potentially larger margin of error compared with laboratory analysis • Requires some training/certification • Relatively high purchase cost • Cannot measure lead in small objects or on highly curved or intricate surfaces, including many toys
Laboratory analysis[a]	• Most accurate method • Relatively affordable if laboratory is available and only few samples • No technical limitations • Can determine both the soluble and insoluble lead fractions • Can be used to analyse new (wet) paint	• Paint surface must be disturbed • Very expensive if equipment must be purchased and set up • Requires trained laboratory personnel and strict quality assurance measures • Results are not available immediately

[a] Various analytical methods exist to measure the concentration of lead in the sample, including flame atomic absorption spectrometry, graphite furnace atomic absorption spectrometry and inductively coupled plasma atomic emission spectrometry. Although these methods differ in their accuracy and limits of detection, all of them are usually adequate to determine lead in paint at commonly required detection limits and accuracy levels.

3.1 Chemical test kits

There is a range of chemical test kits, from simple qualitative tests to more sophisticated semiquantitative tests. Many rely upon a colour change to indicate the presence of lead above a certain concentration. In the simplest kits, the result is either positive (i.e. lead is present above a certain concentration) or negative (i.e. lead is absent above a certain concentration), according to whether a colour change occurred. The threshold concentration for the colour change depends upon the test kit used and may be regulated in the country where the test is marketed. In the USA, for example, test kits should detect concentrations above 0.5% lead by weight (5000 mg/kg). Depending on the context in which they are meant to be used, some chemical test kits may have lower limits of detection.

The simpler kits either test the paint using a swab soaked in a chemical reagent that is rubbed onto the painted surface or require the removal of a chip of paint of a specified area that is then mixed with reagents in a tube. More sophisticated spot test kits use fluorimetric or photometric methods on paint chips. The United States Environmental Protection Agency (USEPA) has evaluated a number of these kits, and additional information can be found on its web site (*5*).

Chemical test kits are relatively cheap and do not require specific training, although training of the user will give more reliable results. The results are immediate. These kits have a number of limitations, however. The kits can test exposed layers only; therefore, to test underlying layers that may be more likely to contain lead, it is necessary to score the paint surface or to remove a chip of paint (depending on the method). For some kits, the colour change may be difficult to observe, particularly when testing dark paint colours. In general, these kits cannot measure the amount of lead present in the paint; even the semiquantitative methods provide only concentration ranges. Finally, chemical test kits may have limited accuracy; in other words, they may indicate that lead is present above a certain concentration when it is not (false positive) or that lead is not present above a certain concentration when it is (false negative). The USEPA, for example, currently recognizes only three lead test kits as being compliant with the false negative criterion of the United States 2008 Renovation, Repair and Painting rule. These three tests are recommended only when used by a certified renovator for paint present on specific substrates, and none is recognized by the USEPA for the false positive criterion of the Renovation, Repair and Painting rule (*6*). For these reasons, methods other than chemical test kits are generally preferred for the accurate determination of lead in paint (*7*).

3.2 Portable X-ray fluorescence (XRF) spectrometry

XRF spectrometry is based on the fact that, when exposed to high-energy radiation, lead (like many other elements) emits X-rays at a characteristic frequency. The intensity of the rays can be measured and correlated to the amount of lead per unit area (usually in units of milligrams per square centimetre). As regulatory standards for lead in paint may be expressed in other units (e.g. parts per million or per cent concentration by weight), the XRF results may need to be converted. Portable XRF instruments can measure the total amount of lead in a painted surface in situ without damaging the paint or the substrate. Portable XRF

devices are very easy to use but, because of the radiation hazard, require special training. In some countries, operators must be certified.

The accuracy of portable XRF devices is good, although they have a larger margin of error compared with adequately executed laboratory analysis (this is particularly true of X-ray tube–based instruments). Borderline measurements (i.e. readings that are within the device's margin of error and are close to the established national limit for lead in paint) should therefore be confirmed by laboratory analysis. Depending on their size and characteristics, XRF devices require a relatively large and flat surface to conduct a measurement. This method is therefore particularly adapted to measuring flat paint surfaces, but is less applicable to testing highly curved or intricate surfaces, including many toys, because of safety concerns, poor reliability of results and the inability to determine the exact surface area measured (*7*, *8*). Some new devices may overcome these limitations.

Portable XRF devices are available from several manufacturers. They are relatively expensive, ranging in price from approximately US$ 10 000 to US$ 50 000, but require little maintenance and no consumables. Measurements are very rapid (about 1 minute, although less precise results can be obtained in a shorter time), allowing operators to measure many paint surfaces in a short period of time.

Guidelines for the determination of lead in paint using XRF devices are available from various sources, including manufacturers and national institutions (*8*).

3.3 Laboratory analysis

This approach requires that a sample of paint, either new paint or dry paint removed from a surface, be collected and analysed in a laboratory using various techniques. Laboratory analysis requires careful sample collection and preparation.

For dry paint on surfaces, it is generally recommended that:

- all layers of paint are collected, as the lower levels are more likely to contain lead;
- as little as possible of the underlying material (e.g. wood, plaster, metal) is included, as this might give erroneous results;
- within a house or building, several representative samples are collected from different areas;
- the location of each sample is recorded;
- surfaces from which paint samples have been collected are repaired to prevent future exposure in case the paint contains lead.

A minimum sample size of about 300 mg is usually required to conduct an analysis, although this depends on the concentration of lead, sample preparation and the method of analysis. Results can be provided in weight per cent if the weight of the sample is measured or in weight per unit surface area if the exact surface area of the sample can be determined.

In the laboratory, the paint sample must be processed before being analysed. For the measurement of total lead, the sample should undergo acid digestion. For the measurement of soluble lead (i.e. the lead likely to be available for absorption if a child mouths or swallows

the paint), an acid extraction process is used. Whether total lead or soluble lead is measured depends on the reason for the analysis and on the relevant national regulations (*9, 10*).

The actual analysis can be conducted using different techniques, including flame atomic absorption spectrometry (FAAS), graphite furnace atomic absorption spectrometry (GFAAS) and inductively coupled plasma atomic emission spectrometry (ICP-AES). Guidelines, recommendations and standard operating procedures for sample collection, preparation and analysis using these and other methods are available from numerous sources, including manufacturers, national institutions and international standardization agencies (*8–16*).

Laboratory analysis is considered the most accurate method for measuring lead in paint, provided adequate quality assurance (QA) principles are followed (see section 4.2) and, in the case of paint chip samples, all layers of paint are included, but not substrate material (*17*). This method requires, however, that a painted surface be disturbed and repaired. This is the only method that can be used for measuring lead in wet paint. It also requires significant time and financial resources for sample collection, transport and laboratory analysis. Analysis can be performed only by trained laboratory technicians in adequate laboratory settings.

The following sections briefly describe three of the most frequently used instrumental techniques for laboratory determination of lead in paint chips: FAAS, GFAAS and ICP-AES (*11, 18*). Although these methods differ in their levels of accuracy and their limits of detection, all of them are adequate to determine lead in paint at commonly required detection limits and accuracy levels. Other less commonly used instrumental methods that are not described here include inductively coupled plasma mass spectrometry, direct current plasma atomic emission spectrometry, dithizone spectrophotometry, anodic stripping voltammetry and potentiometric stripping voltammetry.

3.3.1 Flame atomic absorption spectrometry (FAAS)

Atomic absorption spectrometry (AAS) is based on the fact that free atoms absorb light at wavelengths characteristic of the element of interest: 283.3 nm in the case of ground-state atoms of lead. The amount of light absorbed can be correlated in a linear fashion to the concentration of the analyte in the sample. To conduct an AAS measurement, the lead-containing sample must first be processed by the instrument so as to generate ground-state atoms as a vapour within the light path of the instrument, a process known as atomization. FAAS uses an air–acetylene or a nitrous oxide–acetylene–air laminar flame to atomize lead at temperatures up to 2600 °C.

FAAS detection limits are moderate, but still sufficient for most cases. As direct sample aspiration is required, a minimum of about 5 ml of digest is needed for aspiration and measurement of a stable signal. FAAS measurements are subject to some interference from light scattering and molecular absorption by matrix components, which can be adequately corrected by various approaches. FAAS devices, which require some laboratory skills to operate, are widely available with or without autosamplers. The initial instrument cost is relatively low, and consumables, such as acetylene gas, are relatively inexpensive. Maintenance needs are relatively low, and sample throughput can be several samples per minute.

3.3.2 Graphite furnace atomic absorption spectrometry (GFAAS)

GFAAS is an AAS technique that uses an electrically heated graphite tube to vaporize and atomize the analyte at temperatures up to 3000 °C prior to its detection. GFAAS instruments give very low detection limits and require only very small digest volumes (about 20 µl).

GFAAS measurements are often subject to significant interference from light scattering and molecular absorption by matrix components, but this can be adequately corrected using various approaches, including the use of matrix modifiers. GFAAS devices must be operated by trained laboratory technicians.

GFAAS devices are widely available and require autosamplers to increase precision and throughput. Initial instrument cost is intermediate, and maintenance and consumable costs are significant. Sample throughput is approximately one sample every 2 to 3 minutes.

3.3.3 Inductively coupled plasma atomic emission spectrometry (ICP-AES)

ICP-AES uses an inductively coupled plasma (a very high temperature ionized gas composed of electrons and positively charged ions) source to dissociate the sample into its constituent atoms or ions. Under these high-energy conditions, lead (like many other elements) emits light at characteristic wavelengths. The amount of light emitted can be measured and correlated to the concentration of lead in the sample. ICP-AES instruments offer the advantage of being able to determine several elements simultaneously.

The detection limit for lead is intermediate, but still sufficient to measure lead accurately in paint at commonly observed concentrations. Sample volume requirements are moderate. Some spectral interferences are common, but can be corrected. ICP-AES instruments must be operated by trained laboratory personnel. The initial instrument cost is high, but the major consumable is only argon gas. Maintenance costs are relatively high because of the complicated design of ICP-AES instruments. Sample throughput is intermediate, typically about one sample per minute.

4. Important aspects of laboratory practice

In analytical chemistry, even the most sophisticated and accurate equipment will provide incorrect results if samples have not been appropriately collected and handled, if the equipment has not been used correctly or if analytical protocols have not been followed. Two concerns associated with measurements of lead in paint are unrecognized contamination and inadequate QA. These issues are briefly discussed in the following sections.

4.1 Preventing external contamination

Lead is pervasive and can contaminate samples in numerous ways, particularly in the case of paint chip laboratory analysis. Contamination can occur during sample collection, sample storage and transport, and sample manipulation. The quality of sample collection and handling is therefore a crucial aspect for the accurate determination of lead in paint. Sample handling within the laboratory entails as much risk of contamination as sample collection in the field. Laboratories should be as close to lead free as possible, and laboratory staff

should be properly trained to prevent sample contamination. Specific protocols are available for the different analytical methods, including from manufacturers and standardization agencies (*8–15*). These must be strictly adhered to. Contamination risks can be significantly reduced by the application of adequate QA measures.

4.2 Quality assurance (QA)

QA refers to all steps that must be taken to assure that laboratory results are reliable and reproducible. It covers the utilization of scientifically and technically sound practices for laboratory investigations, including the selection, collection, storage and transport of specimens and the recording, reporting and interpretation of results. It also refers to training and management designed to improve the reliability of investigations. From the point of view of an analysis, QA can be divided into two stages: 1) initial assessment of an analytical method as to its practicability and trueness, which includes linearity, specificity, recovery, calibration standards, blanks and interference; and 2) subsequent quality assessment.

Quality assessment refers to the quality of the analytical results. It has two components:

1) internal quality control, which is a set of procedures used by the staff of a laboratory for continuously assessing results as they are produced in order to determine whether they are reliable enough to be released;
2) external quality assessment (EQA), which is a system for objectively checking laboratory performance using an external agency.

Further information on laboratory QA and quality management and examples of EQA programmes specific for lead in environmental samples are available from various sources (*19–21*).

It is crucial that adequate QA measures be followed by the laboratory conducting the paint sample analysis, including, if possible, EQA. International and/or national accreditation should also be considered.

5. Method selection

The choice of method depends on numerous factors, including the level of accuracy required, the substrate to be tested (including whether it is new paint or a painted surface), the availability of trained personnel and analytical equipment, and financial considerations.

As a result of their limited accuracy and practical limitations and despite their ease of use, chemical test kits are usually not recommended for the accurate determination of lead in paint, with the exception of a few kits for specific applications and circumstances (*6, 7*).

The portable XRF lead-based paint analyser is the most commonly used primary analytical method for inspections in housing. This is because of its demonstrated ability to determine whether lead-based paint is present on many surfaces and to measure the lead content of the paint without destructive sampling or paint removal, as well as its high speed and low

cost per sample. This method, however, cannot be used to test small items and highly curved or intricate surfaces, including many toys.

It is generally considered that laboratory analysis is the most accurate method to measure lead in paint, provided all the requirements for adequate QA are met (*17*). However, this process requires significant skill and time for sample collection, transport and laboratory analysis. If analysis is conducted by a commercial laboratory, the cost of analysis per sample is relatively high. Damage from sampling is also an unavoidable consequence if dried paint is being tested. For these reasons, determination of lead in paint by the sole use of laboratory analysis is generally not recommended, and portable XRF analysis is often preferred (*8*). Laboratory analysis is, however, recommended in the following situations:

- for new paint;
- when a high accuracy and/or low limits of detection are needed;
- for small items, such as toys, and inaccessible areas or building components with irregular surfaces that cannot be tested using XRF instrumentation;
- to confirm borderline XRF results.

If laboratory analysis is preferred, numerous analytical methods are available, including FAAS, GFAAS and ICP-AES. All of these methods are adequate to determine lead in paint with commonly required limits of detection and accuracy as long as guidelines, standard operating procedures and strict QA measures are followed. Method choice will depend on numerous factors, such as:

- availability of operational equipment and laboratory;
- availability of adequately trained laboratory personnel;
- level of laboratory QA measures;
- number of samples and analysis turnaround time;
- analytical costs, including running and maintenance costs;
- equipment purchase and installation costs if new instrumentation must be purchased;
- availability of an external laboratory if analytical capacities are not available locally.

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