Analytical methods used to measure lead in paint

Brief guide to analytical methods for measuring lead in paint

Second edition

World Health Organization

GLOBAL ENVIRONMENT FACILITY
Brief guide to analytical methods for measuring lead in paint

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<table>
<thead>
<tr>
<th>Abbreviation</th>
<th>Full Form</th>
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<tr>
<td>AAS</td>
<td>Atomic absorption spectrometry</td>
</tr>
<tr>
<td>AIHA</td>
<td>American Industrial Hygiene Association</td>
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<tr>
<td>ASTM</td>
<td>American Society for Testing and Materials</td>
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<tr>
<td>cm</td>
<td>centimetre</td>
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<tr>
<td>DALY</td>
<td>Disability adjusted life years</td>
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<tr>
<td>EDXRF</td>
<td>Energy Dispersive X-ray fluorescence</td>
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<tr>
<td>ELLAP</td>
<td>Environmental Lead Laboratory Accreditation Program</td>
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<td>ELPAT</td>
<td>Environmental Lead Proficiency Analytical Testing Program</td>
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<tr>
<td>EQA</td>
<td>External Quality Assurance</td>
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<tr>
<td>ETAAS</td>
<td>Electrothermal atomic absorption spectrometry</td>
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<tr>
<td>FAAS</td>
<td>Flame atomic absorption spectrometry</td>
</tr>
<tr>
<td>GFAAS</td>
<td>Graphite furnace atomic absorption spectrometry</td>
</tr>
<tr>
<td>HDXRF</td>
<td>High definition X-ray fluorescence</td>
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<tr>
<td>ICP-AES</td>
<td>Inductively coupled plasma atomic emission spectrometry</td>
</tr>
<tr>
<td>ICP-MS</td>
<td>Inductively coupled plasma mass spectrometry</td>
</tr>
<tr>
<td>ILAC</td>
<td>International Laboratory Accreditation Cooperation</td>
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<tr>
<td>ISO</td>
<td>International Standards Organization</td>
</tr>
<tr>
<td>kg</td>
<td>kilogram</td>
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<tr>
<td>mg</td>
<td>milligram</td>
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<tr>
<td>mL</td>
<td>millilitre</td>
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<tr>
<td>μL</td>
<td>microlitre</td>
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<tr>
<td>ppm</td>
<td>parts per million</td>
</tr>
<tr>
<td>XRF</td>
<td>X-ray fluorescence</td>
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</tbody>
</table>
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 public health personnel, scientific institutions and policy-makers who are not laboratory specialists but who may need to develop plans for measuring the concentration of lead in new paint and for studies on the availability of lead paint\(^1\) on the market for purchase by consumers.

The document lists well-established analytical methods for measuring the lead content of paint and briefly describes some of the characteristics of these methods, including their strengths and limitations. It also highlights, for various types of applications and scenarios, the considerations when deciding whether to use a laboratory-based method or portable technology, and whether to establish a laboratory service for lead measurement or to buy in services from another laboratory. This document does not aim to provide a description of analytical methods and protocols or to make specific recommendations regarding methods or specific instruments. Detailed technical information on this subject is available elsewhere, and links to further information are provided in the references section and the annex.

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 in 2017 was estimated to account for 1.06 million deaths and the loss of 24.4 million disability-adjusted-life-years (DALYs) from long-term health effects, with the highest burden falling on low- and middle-income countries (1).

Lead is a cumulative toxicant that affects multiple body systems, including the neurological, haematological, gastrointestinal, cardiovascular and renal systems. Long-term effects include increased risk of hypertension, ischaemic heart disease and renal disease. Children are particularly vulnerable to the neurotoxic effects of lead, and even low levels of exposure can impair cognitive development and cause behavioural disorders. These effects can be lifelong (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 paint. Lead paint may be used to paint the interiors and exteriors of homes, schools and other buildings, to paint toys, furniture, playground equipment and other articles with

\(^1\)“Lead paint” is defined by the Global Alliance to Eliminate Lead Paint Model Law and Guidance for Regulating Lead Paint as any paint with levels higher than a maximum limit, for example, 90 ppm.
which children can come into contact, and to
paint road-markings, bridges and other external
structures. Paint with high levels of lead is still
available and in use in many parts of the world
(3, 4). Even in countries where it has been banned
for specific uses, lead paint can be found in older
houses and on articles (5).

People can become exposed to lead from paint
from occupational and environmental sources,
mainly through ingestion and inhalation.
Occupational exposure can occur during the
manufacture of paints, when paints are being
applied or removed, and when buildings and
structures painted with lead paint are being
renovated or demolished. Removal of lead paint
by burning produces lead fumes and particles.
Abrasive paint removal methods, such as sanding,
produce large amounts of lead dust that can be
inhaled and ingested. While intact lead paint in
good condition does not present a health hazard,
this paint breaks down over time, fragmenting
into flakes and dust that can contaminate the
domestic environment (6).

Lead-contaminated house dust is a major
contributor to the total body burden of lead in
children (7). Young children are particularly
vulnerable to lead exposure because they absorb
around 40–50% of the ingested amount (8).
Moreover, children’s innate curiosity and their
age-appropriate hand-to-mouth behaviour result
in their mouthing and swallowing lead-containing
or lead-coated objects, such as contaminated soil
or dust and flakes from decaying lead paint (8). In
addition, children with pica may persistently eat
flakes of lead paint or lead-contaminated soil.

3. Measurement of lead in paint

There are two main reasons for measuring the lead content in paint:
a) to assess new paints, either to check for compliance with regulatory or legal
restrictions, or as part of a market survey to find out if lead paints are being sold;
b) to find out whether existing paint on surfaces in homes or other settings, on furniture,
playground equipment, or toys, contains lead in order to determine the need for risk
management measures.

Compliance testing to enforce a legal limit on lead
in new paint takes two forms. A manufacturer (or
importer or distributor) can send paint samples to
a third-party laboratory that will analyse the paint
and issue a declaration of conformity, provided
the lead content of the paint is within the required
limits. A regulatory or enforcement authority can
also carry out inspections and analyse paints to
see if they conform to the required lead limit, with
the possibility of sanctions in case of failure (9).

The results from market surveys of new paints
can be used to provide evidence of the need for
better government regulation and enforcement.
The studies can also provide consumers with
information so they can make informed purchase
decisions, and lobby for government controls
on lead paint. Such studies may also encourage
manufacturers to reformulate their products
voluntarily.
where existing paint that has been applied to walls or other surfaces is analysed and is found to contain high levels of lead then risk management measures might include stabilization or removal of lead paint. Note that when lead paint is being removed special procedures must be followed to minimize the release of lead and the exposure of workers and others. In the case of toys or other products typical risk management measures would include stopping the goods at customs, withdrawal of the products from the market and issuing consumer alerts.

3.1 Measurement of total lead versus soluble lead

The lead concentration in paint can be measured using methods that quantify the total lead content or the soluble lead content. Most regulatory standards for new paint are based on the total lead content, and this is the recommended measurement described in the Model law and guidance for regulating lead paint (9).

Total lead is measured by extracting all the lead present in the paint sample. It represents the total amount of lead that could be absorbed from ingestion or inhalation of flakes and dust from decaying or abraded paint, or fumes produced by burning off the paint.

The soluble lead content is the amount of lead that can be extracted using a standard laboratory test that involves incubating a sample of paint film with dilute acid. The soluble lead content is sometimes measured on the assumption that it represents the amount of lead that is bioaccessible and potentially available for absorption when lead paint chips or coated objects are mouthed or swallowed (10, 11). When assessing lead paint, however, this measurement does not accurately represent all of the lead that can be absorbed (12). Furthermore, paints with a low soluble lead content can have a high total lead content (10). For example, a recent study carried out in China found that more than half of paints that were measured as having a soluble lead content below 90 parts per million (ppm) had a total lead content higher than 90 ppm, ranging up to 17 400 ppm (11).

3.2 Measurement units

The lead content in paint can be expressed as a mass concentration or a concentration per unit area on a painted surface (also known as lead loading). The mass concentration is used for new paint and most regulatory standards refer to the total amount of lead in the dried paint film expressed as a percentage, as ppm, or as milligrams per kilogram (mg/kg). In the case of existing paint on surfaces, the lead content may be expressed as the mass concentration (for example as ppm) or as the amount per unit area in milligrams per square centimetre (mg/cm²).

It is important to note that mass concentration and lead loading measurements are not interchangeable. There is no precise relationship between laboratory measurements of paint on surfaces expressed as ppm and as mg/cm² because of variations in the composition and thickness of the paint and the possible inclusion of substrate material during the analysis.

Table 1 summarizes the units with which different methods report results.
Table 1. Units for reporting results using different methods

<table>
<thead>
<tr>
<th>Method</th>
<th>New paint</th>
<th>Existing paint surfaces</th>
</tr>
</thead>
<tbody>
<tr>
<td>Laboratory analysis</td>
<td>ppm, % or mg/kg</td>
<td>ppm, %, mg/kg or mg/cm²</td>
</tr>
<tr>
<td>Handheld conventional X-ray fluorescence (XRF)</td>
<td>ppm or %</td>
<td>mg/cm², ppm (but see footnote c)</td>
</tr>
<tr>
<td>Handheld high-definition X-ray fluorescence (HDXRF)</td>
<td>ppm</td>
<td>mg/cm², ppm</td>
</tr>
</tbody>
</table>

Notes:
(a) No mathematical equivalence between ppm and mg/cm²;
(b) 0.009% = 90 ppm = 90 mg/kg;
(c) Some XRF devices have an option to calculate and display units in ppm, but the density and thickness of the paint must be entered to enable this calculation (Olaf Haupt, Thermo Scientific Field and Safety Instruments, personal communication November 2019).

3.3 Sampling methods

The method of sample collection depends on whether new or existing paint is being analysed, and on the analytical method to be used.

3.3.1 New paint

For analysis of new paint (for example from paint in a can), the usual method is to apply a thin layer of paint to a non-metal, homogenous surface, such as a glass slide or a flat, smooth piece of wood and allow it to dry. In the laboratory the paint is then scraped off, prepared and analysed using one of the instrumental techniques described in section 4.1 below (13). The lead content in the dried paint film can also be measured directly using high-definition X-ray fluorescence (HDXRF), without the need to scrape the paint off the test surface.

Liquid samples of paint can be analysed by laboratory-based HDXRF instruments using special sample cups (see sub-section 4.2.1).

3.3.2 Existing paint

For laboratory analysis the collection of samples of existing paint should follow a pre-determined standard operating procedure. It is generally recommended that (6, 14, 15):

- all layers of paint are collected, as the lower levels are more likely to contain lead;
- as little as possible of the underlying material (for example wood, plaster, metal) is included, as this might give erroneous results if reported as mass concentration;
- within a house or building, several representative samples are collected from different areas and 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.

It is advisable to consult with the selected laboratory ahead of time about the minimum sample size, sample packaging and other sampling requirements. Results can be provided as a mass concentration if the weight of the sample is measured, or as mass per unit surface area if the exact surface area of the sample can be determined.

The lead concentration in the paint on a surface such as a wall can also be measured directly using portable XRF or HDXRF instruments and the results are generally reported as mg/cm². This method is commonly used for home inspections for lead-based paint because it enables the rapid measurement of multiple samples and does not require the destruction of any of the paint surfaces (6, 15, 16).
4. Analytical methods used to measure lead in paint

As mentioned above, the lead content in paint can be measured using laboratory methods, benchtop or portable XRF devices, and test kits. These methods are briefly described in this section.

4.1 Laboratory methods

The most frequently used instrumental techniques for laboratory determination of lead in paint are flame atomic absorption spectrometry (FAAS), electrothermal atomic absorption spectrometry (ETAAS) and inductively coupled plasma atomic emission spectrometry (ICP-AES). Although these methods have differing levels of accuracy and limits of detection, they are all adequate to determine lead in paint at commonly required detection limits and accuracy levels. 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 (6, 13, 14, 17–19). Additional information is provided in the annex.

Lead measurements can also be carried out using free-standing or benchtop HDXRF instruments, described in section 4.2.

Other less commonly used instrumental methods that are not described here include inductively coupled plasma mass spectrometry (ICP-MS), direct current plasma atomic emission spectrometry, dithizone spectrophotometry, anodic stripping voltammetry and potentiometric stripping voltammetry.

4.1.1 Flame atomic absorption spectrometry (FAAS)

The principle behind atomic absorption spectrometry (AAS) is the interaction between outer-shell electrons of free, gaseous, uncharged atoms and ultraviolet or visible light generated from the element to be measured. In the case of lead, for example, a cathode lamp containing lead emits light from excited atoms of lead, which has characteristic wavelengths, and which can be absorbed by lead atoms in the sample. The light passes through the atomized sample and some energy is absorbed by the lead atoms, reducing the amount transmitted to the detector. The amount of light absorbed (or absorbance) is related in a linear fashion to the concentration of the analyte in the sample (20). To conduct an AAS measurement, the lead-containing sample must be introduced into the instrument so as to generate ground-state atoms in the gas phase within the optical path of the instrument, a process known as atomization.

FAAS typically uses an air–acetylene flame to atomize lead at temperatures in the order of 2100–2400°C.

Analysis by FAAS is applicable for lead concentrations at a lower limit of 100 ppm (21). Although some laboratories have developed methods that can measure concentrations as low as 40 ppm, FAAS is not the best method for confirming compliance with a 90-ppm limit.² As

direct sample aspiration is required, a minimum of about 5 millilitres (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 (17).

4.1.2. Electrothermal atomic absorption spectrometry (ETAAS) or Graphite Furnace Atomic Absorption Spectrometry (GFAAS)

Most ETAAS systems use an electrically heated graphite tube to pyrolyse and atomize the analyte, so this method is also known as graphite furnace atomic absorption spectrophotometry (GFAAS) (20). The pyrolyzed sample is heated to temperature of around 1700 °C to atomize lead (19). ETAAS instruments give very low detection limits and require only very small digest volumes — about 20 microlitres (µL) (17). The method is applicable for lead concentrations at a lower limit of 0.1 ppm (17).³

ETAAS measurements can be subject to significant interferences from light scattering and molecular absorption by matrix components, but this can be mitigated by using various approaches, including the use of chemical modifiers and Zeeman background correction techniques (17, 20). ETAAS devices must be operated by trained laboratory technicians.

ETAAS 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 two to three minutes (17).

4.1.3. Inductively coupled plasma atomic emission spectrometry (ICP-AES)

ICP-AES uses a source of inductively coupled plasma (a very high-temperature ionized gas composed of electrons and positively charged ions) 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.

ICP-AES can measure lead concentrations in paint as low as 2 ppm.⁴ Sample volume requirements are moderate. Some spectral interferences are common but can be corrected (17). 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 (17, 20).

4.2 X-ray fluorescence (XRF) spectrometry

XRF spectrometry provides a means for the rapid, non-destructive testing of paint. The principle of the method is that when exposed to high-energy radiation, lead (like many other elements) emits X-rays at a characteristic frequency (X-ray fluorescence). These X-rays are the result of the displacement of an electron from the K or L shells of the atom (6, 16). The radiation source can be an X-ray tube or a gamma radiation source. Devices using the former can detect L

³ Ibid.
Analytical methods used to measure lead in paint

shell fluorescence, while those using a gamma radiation source can detect both K and L shell fluorescence (6, 16, 22).

The intensity of the rays can be measured and correlated to the concentration of lead in the sample. As with other analytical methods it is necessary to first calibrate the instrument using standard reference materials.

There are two main types of XRF: energy-dispersive XRF (EDXRF) and wavelength-dispersive XRF (23). XRF devices are available for use in the laboratory and there are also portable and hand-held versions that can be used in the field. Laboratory EDXRF systems have higher power, better resolution and lower detection limits than portable and hand-held versions (23).

A category of EDXRF is high-definition X-ray fluorescence (HDXRF). This uses EDXRF combined with doubly curved crystal X-ray optics that direct an intense focused beam of X-rays onto the surface of the test material (23, 24). This optical system enables high-sensitivity, compact, low-power analysers. HDXRF systems can accurately measure the lead content in liquid paint as well as in a dried paint film and can report results in ppm.

4.2.1 Laboratory-based HDXRF
Free-standing and benchtop versions of HDXRF are available. Some benchtop versions are portable and can be used in a non-laboratory setting for compliance testing.

Laboratory HDXRF can be used to measure the lead content in a paint film directly without the need to scrape off and digest the paint. It has been shown to be as accurate and precise as the laboratory methods described above and a standard test method has been developed (25, 26).

For analysing liquid paint the sample is placed in a special sample cup, sealed with a film, which is then put against the detector. While sample preparation is considerably simpler than for the laboratory methods described above, it is important that the correct sample cup and film are used and that the sample cup is correctly assembled; training is therefore required (27).

By comparison with atomic absorption methods and ICP-AES, laboratory XRF is cheaper and simpler to operate. A typical analytical range is 1 ppm to 100%.

4.2.2 Handheld X-ray fluorescence (XRF) spectrometry
There are a range of handheld XRF instruments. They allow the measurement of lead in paint to be carried out in situ without the need to damage the paint or the substrate. Two types of field-portable devices are described here: conventional XRF and high-definition XRF.

As these devices contain a radiation source, training in their safe use is essential (6, 16). In some countries, operators must be licensed and certified (6).

Conventional X-ray fluorescence (XRF) spectrometry
Most handheld XRF devices detect K shell fluorescence, some detect L shell fluorescence, and some detect both. A comparison of handheld XRF devices found that those that only detected L shell fluorescence could give false negative results when the lead paint was covered with another layer because the L X-rays may not penetrate through the layers to reach the detector. This was less of a problem with devices that measured K-shell fluorescence, as K X-rays have higher energy (16). On the other hand, L X-rays are less likely to be influenced by the effects of the substrate, while substrate correction techniques may be needed for K shell fluorescence devices (6, 22). It is important, therefore, to select the appropriate instrument for the testing required, and advice should be sought from the manufacturer.

Conventional XRF instruments report results as mass per unit area (i.e. mg/cm²). Some devices give the option to calculate and display units in ppm for painted products, but the density and
thickness of the paint must be entered and the range is limited (23).\textsuperscript{5}

The limit of detection of XRF devices for specific purposes can be as low as 5 ppm, but this is a function of the testing time, sample matrix, and the presence of interfering elements.\textsuperscript{6} For paint on housing surfaces a measurement range of 0.1 to 10 mg/cm\textsuperscript{2} can be obtained.\textsuperscript{7} The range can vary depending on the substrate beneath the paint, for example iron-containing substrates can significantly degrade the limit of detection for lead.\textsuperscript{8}

Most devices are calibrated to measure lead in paint on a variety of substrates, for example wood, metal, cement, plasterboard (drywall) and plaster, and it is important that the correct calibration setting is selected when using the device. While results can be obtained within a few seconds, the limit of detection and the precision increase with longer measuring times.

The accuracy of conventional XRF devices is good, provided they are used by a trained operative who follows the calibration procedures and ensures that the detector is correctly held against the test surface.

Conventional XRF devices suitable for measuring lead in paint are available from several manufacturers. They are relatively expensive, ranging in price from approximately US$ 10 000 to US 50 000. They require little maintenance, though in devices that use a radioisotope source this will need periodic replacement. The short measurement time allows operators to rapidly assess multiple paint surfaces.

Guidelines for the determination of lead in paint using conventional XRF devices are available from various sources, including manufacturers and national institutions (6, 16).

\textbf{High-definition X-ray fluorescence (HDXRF) spectrometry}

Unlike conventional XRF, handheld HDXRF devices can make separate measurements of the surface and the substrate and can report results as ppm. In the United States of America, HDXRF analysis has been deemed suitable by the Consumer Product Safety Commission for compliance measurements of new paints as well as existing painted surfaces (28). A method for lead in paint and coating materials has been published, which describes an analytical range of 30—450 ppm (26). The main limitation of this method is the cost of the equipment, which can be higher than the cost of conventional XRF equipment. There are the same requirements for training operators as for conventional XRF analysers.

\section*{4.3 Chemical test kits}

A number of chemical test kits are available. The kits cannot measure the lead content of paint with the same accuracy and precision as laboratory and XRF measurements, but information is provided here for the sake of completeness.

Chemical test kits may be simple qualitative tests or more sophisticated semi-quantitative 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 (lead is present above a certain concentration) or negative (lead is not present 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 United States, for example, test kits should detect concentrations above 1 mg/cm\textsuperscript{2} (6). Depending on the context in

\begin{itemize}
\item \textsuperscript{5} Olaf Haupt, Thermo Scientific Field and Safety Instruments, personal communication, November 2019.
\item \textsuperscript{6} Diego Tschuor, CONTROLTECH AG, personal communication, November 2019.
\item \textsuperscript{7} Olaf Haupt, Thermo Scientific Field and Safety Instruments, personal communication, November 2019.
\item \textsuperscript{8} Olaf Haupt, Thermo Scientific Field and Safety Instruments, personal communication, November 2019.
\end{itemize}
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 from a specified area which is then mixed with reagents in a tube. More sophisticated spot test kits use fluorimetric or photometric methods on paint chips (29, 30). The United States Environmental Protection Agency has evaluated a number of these kits, and additional information can be found on its website (31).

These kits have a number of limitations (6, 30, 32). 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) (32). For these reasons, methods other than chemical test kits are generally preferred for the accurate determination of lead in paint.

The various characteristics of the different analytical methods are summarized in Table 2.
Table 2. Overview of analytical methods for lead in paint measurement

<table>
<thead>
<tr>
<th>Method</th>
<th>Strengths</th>
<th>Limitations</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flame atomic absorption spectrometry (FAAS)</td>
<td>• Relatively easy to use and moderate cost</td>
<td>• Needs special gases</td>
</tr>
<tr>
<td></td>
<td>• Can be fitted with auto-sampler so multiple samples can be processed</td>
<td>• Limit of detection depends on sample preparation and method used</td>
</tr>
<tr>
<td></td>
<td></td>
<td>• Requires skilled laboratory technician</td>
</tr>
<tr>
<td>Electrothermal atomic absorption spectrometry (ETAAS)</td>
<td>• Can analyse very small samples</td>
<td>• Needs special gases</td>
</tr>
<tr>
<td></td>
<td>• Can be fitted with auto-sampler so large number of samples can be run</td>
<td>• Requires skilled laboratory technician</td>
</tr>
<tr>
<td>Inductively-coupled plasma atomic emission spectrometry (ICP-AES)</td>
<td>• Can be economical if used for large sample runs</td>
<td>• Expensive, with high running costs</td>
</tr>
<tr>
<td></td>
<td>• Can analyse very small samples</td>
<td>• Requires highly-skilled laboratory technician</td>
</tr>
<tr>
<td></td>
<td>• Can determine isotope ratio, which may help to identify the source of the lead</td>
<td></td>
</tr>
<tr>
<td></td>
<td>• Very low limit of detection</td>
<td></td>
</tr>
<tr>
<td>Laboratory-based high-definition X-ray fluorescence (HDXRF) spectrometry</td>
<td>• Low running and operating costs</td>
<td>• High purchase cost</td>
</tr>
<tr>
<td></td>
<td>• Can measure lead in a liquid paint sample</td>
<td>• Requires training to ensure accurate results and observance of health and safety requirements</td>
</tr>
<tr>
<td></td>
<td>• Sample preparation is simple</td>
<td></td>
</tr>
<tr>
<td></td>
<td>• Low limit of detection</td>
<td></td>
</tr>
<tr>
<td>Handheld conventional X-ray fluorescence (XRF) spectrometry</td>
<td>• Can be used at the site of the paint to be analysed</td>
<td>• Requires training to ensure accurate results and observance of health and safety requirements</td>
</tr>
<tr>
<td></td>
<td>• Paint surface does not need to be damaged</td>
<td>• Operator may need to be licensed and/or certified</td>
</tr>
<tr>
<td></td>
<td>• Good accuracy</td>
<td>• Relatively high purchase cost</td>
</tr>
<tr>
<td></td>
<td>• Immediate results</td>
<td></td>
</tr>
<tr>
<td></td>
<td>• Can measure many surfaces over a short period of time</td>
<td></td>
</tr>
<tr>
<td></td>
<td>• Cheaper to use than laboratory methods when many surfaces need to be tested</td>
<td></td>
</tr>
<tr>
<td>Handheld high-definition X-ray fluorescence (HDXRF) spectrometry</td>
<td>Same as for conventional XRF, in addition:</td>
<td>Same as for conventional XRF</td>
</tr>
<tr>
<td></td>
<td>• Can report results as ppm</td>
<td></td>
</tr>
<tr>
<td></td>
<td>• Low detection limit (below 90 ppm)</td>
<td></td>
</tr>
<tr>
<td></td>
<td>• Can be used on dried paint film to measure lead in new paint for compliance testing</td>
<td></td>
</tr>
<tr>
<td>Chemical test kits (on site)</td>
<td>• Qualitative test for lead paint on walls or other surfaces</td>
<td>• Cannot provide an accurate measurement</td>
</tr>
<tr>
<td></td>
<td>• Immediate results</td>
<td>• False positive and false negative results possible</td>
</tr>
<tr>
<td></td>
<td>• Low purchase and running costs</td>
<td>• Can test mainly top (surface) layers</td>
</tr>
<tr>
<td></td>
<td>• Relatively simple to use</td>
<td>• May need to damage the paint surface</td>
</tr>
<tr>
<td></td>
<td></td>
<td>• May be difficult to observe colour change for dark paints</td>
</tr>
<tr>
<td></td>
<td></td>
<td>• Special procedure may be needed for certain surfaces, e.g. plaster</td>
</tr>
</tbody>
</table>
5. Choosing the most appropriate method

The choice of method depends on numerous factors, including the reason for analysis (compliance, risk assessment or screening), the number of samples to be tested, the substrate to be tested (new paint, existing painted surface, size and shape of surface), cost limitations, the need for precise measurement, the limit of quantification required, and the availability of trained personnel and analytical equipment.

With regard to the limit of quantification, a suggested value is equal to or less than 20% of the regulatory limit (33). In the case of a 90-ppm limit, therefore, a method would be needed that could report to at least 18 ppm. Table 3 summarizes the analyses that can be used for different purposes.

Table 3. Selecting the most appropriate analysis for the purpose

<table>
<thead>
<tr>
<th>Purpose of test</th>
<th>Laboratory methods</th>
<th>X-ray fluorescence spectrometry</th>
<th>Chemical test kit</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Laboratory</td>
<td>Handheld conventional XRF</td>
<td>Handheld HDXRF</td>
</tr>
<tr>
<td></td>
<td>Laboratory</td>
<td>Laboratory</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Yes</td>
<td>Yes – readings can be taken on</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>dried paint film or liquid paint</td>
<td></td>
</tr>
<tr>
<td>Compliance testing for new paint</td>
<td>Yes</td>
<td>No – results only available in</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>mg/cm²</td>
<td></td>
</tr>
<tr>
<td>Market survey of new paint</td>
<td>Yes</td>
<td>Yes – results only available in</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>mg/cm²</td>
<td></td>
</tr>
<tr>
<td>Testing existing paint in homes,</td>
<td>Yes – some</td>
<td>Yes – results only available in</td>
<td></td>
</tr>
<tr>
<td>schools and other sites</td>
<td>destruction of</td>
<td>ppm; readings can be taken on</td>
<td></td>
</tr>
<tr>
<td></td>
<td>paint surface</td>
<td>dried paint film</td>
<td></td>
</tr>
<tr>
<td></td>
<td>needed</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Testing paint on toys or other</td>
<td>Yes</td>
<td>Yes</td>
<td></td>
</tr>
<tr>
<td>irregularly shaped objects</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

11
Laboratory analysis can provide accurate measurements of lead in paint (existing or new paint), as long as appropriate samples are collected and adequate quality assurance principles are followed (see section 6.2). Significant skill and time are needed for sample collection, transport and, in the case of FAAS, ETAAS and ICP-AES, laboratory analysis (laboratory HDXRF is a faster and simpler method). The cost of dispatch and laboratory analysis may be significant, depending on the location of the laboratory, the number of samples to be measured and the analytical method used. If existing paint is being tested, the paint surface will have to be damaged to collect the sample.

Handheld XRF is an alternative for measuring lead in existing paint as it does not require destructive sampling or paint removal, and because of its high speed and low cost per sample. For these reasons, it is the preferred method when inspecting a home for the presence of lead paint (6, 15).

Laboratory analysis is, however, recommended in the following situations:

- when a high accuracy or low limits of detection are needed;
- for inaccessible areas or building components with irregular surfaces that cannot readily be tested using XRF instrumentation;
- to confirm borderline handheld XRF results.

While chemical test kits also give immediate results, they provide limited information on the lead concentration and are the least accurate and reliable method.

5.1 Choosing a laboratory

There are a number of considerations when selecting a laboratory to test paint, and it is recommended that the laboratory is contacted beforehand to verify its suitability for the desired analyses. The quality of the service provided is of key importance. Using a laboratory that is certified by a recognized body and accredited to carry out the specific tests required will give confidence in the accuracy and reliability of the analytical results obtained. This is particularly important when testing paints to obtain a declaration of conformity with the lead paint law. More information on laboratory quality, including accreditation, is provided in section 6.

Other factors are:
- the experience of the laboratory in testing paints
- the analytical methods used and the limit of detection
- the sampling requirements
- the capacity to handle the number of samples required
- the costs of analyses, including shipping costs
- the turnaround time.

5.2 Finding a laboratory

Information on laboratories accredited for the measurement of lead in paint can usually be found on the websites of national accreditation bodies. Contact details of national accreditation bodies are given on the International Laboratory Accreditation Cooperation (ILAC) website,9 which lists those bodies that are signatories to the ILAC Mutual Recognition Arrangement. The website also gives information on regional accreditation cooperation bodies.

Another source of information is the website of the American Industrial Hygiene Association (AIHA) proficiency testing programme.10 AIHA runs the Environmental Lead Proficiency Analytical Testing (ELPAT) Program (34) and lists over 250 laboratories in the United States and other countries.

9 https://ilac.org/signatory-search
10 https://online.aihapat.org/patssa/f?p=AIHASSA:17800; search for ELPAT Laboratories Program
countries that have passed the most recent proficiency test for lead in paint.

The United States Consumer Product Safety Commission\textsuperscript{11} maintains a list of laboratories certified to conduct third-party testing for compliance with the applicable United States federal safety requirements for children’s products.\textsuperscript{(35)} This list includes certified laboratories in the United States and in other countries that can test for lead in paint.

5.3 Establishing a laboratory service for lead in paint

Establishing a laboratory service requires a significant investment of resources, and some points to consider when deciding whether to go ahead are given below.

- Is there a sufficient workload to justify setting up the service?
- Is there another laboratory in the country or abroad that is already providing this service at a reasonable cost and within an acceptable time frame?
- Is there an existing laboratory that can add the testing of paint to its services?
- What type of instrumentation is needed for the purposes of the laboratory (HDXRF, GFAAS, ETAAS, ICP-MS)?
- Is the necessary analytical equipment already available or must it be purchased?
- Are there sufficient funds for the purchase of equipment, its installation, maintenance and operating costs, including purchase of certified reference materials and replacement of consumables such as lamps, tubes and gases?
- Are there suitable premises for the laboratory with a reliable and consistent power and water supply? Can an existing building be modified or is it necessary to build a new laboratory?
- Are there sufficient numbers of adequately trained laboratory personnel to operate the selected instrumentation?
- Will the laboratory seek accreditation for the measurement of lead in paint and are resources available to support this?

\textsuperscript{11} https://www.cpsc.gov/cgi-bin/labsearch/
6. 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 and maintained periodically, or if analytical protocols have not been followed. Two concerns associated with the measurement of lead in paint are unrecognized contamination and inadequate quality assurance and control. These issues are briefly discussed in the following sections.

6.1 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. These activities should be carried out following a standard operating procedure that includes measures to prevent contamination, such as the use of new sampling equipment for each sample (17).

Sample handling within the laboratory also carries a risk of contamination. Laboratories should be as close to being free of lead contaminants 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, and these should be strictly followed (13, 17–19). Contamination risks can be significantly reduced by the application of adequate quality assurance measures (33).

6.2 Quality assurance and quality control

Quality assurance and quality control are components of a quality management system. Quality management involves the integration of all aspects of laboratory operation, including the organizational structure, processes, procedures and resources in order to ensure that the service provided to users is of high quality and laboratory results are reliable and reproducible (33, 36).

Quality assurance is concerned with processes and procedures. 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. Quality assurance includes the initial assessment of an analytical method as to its practicability and trueness, which includes linearity, specificity, recovery, calibration standards, blanks, limits of detection and limit of quantitation and robustness (17).

Quality control refers to the control of errors in the performance of tests and verification of the test results. It has two components: internal quality control and external quality assessment.
Internal quality control: This 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 accurate, precise and, therefore, reliable enough to be released (17, 20). An example of a quality control measure is analysis of control samples of paint containing a well characterized lead content to check the performance of the analytical method. In case proper (certified) reference materials are available in terms of concentration and matrix match, these are highly recommended for the validation step and regular quality control. The United States Environmental Protection Agency has developed quality control procedures for the measurement of lead in paint (17, 33). The standard operating procedure for a sample analysis should normally include a description of quality control measures (33).

External quality assessment (EQA): This is a system for objectively checking laboratory performance using an external agency. It involves the laboratory being sent “blind” test samples in which the quantity of lead is unknown. The analytical results are then compared against the actual lead concentrations, which are not revealed until after the analyses have been completed. The results are also compared against those of other laboratories participating in the scheme. External quality assessment schemes normally involve a number of test cycles each year. The ELPAT scheme mentioned above is an example of an EQA scheme for lead in paint chips (also soil and dust). The assessments are carried out quarterly.

6.2.1 In situ testing
The need for quality assurance and quality control measures also applies to analyses carried out away from the laboratory, for example, when XRF devices are used to assess the lead content of paint in buildings. Measures include ensuring operatives are adequately trained and the use of calibration checks and control tests (6).

6.3 Standards, certification and accreditation

Standards, certification and accreditation are important measures for ensuring and demonstrating laboratory quality.

A standard document is a document, established by consensus and approved by a recognized body, that provides, for common and repeated use, guidelines or characteristics for activities or their results, aimed at the achievement of the optimum degree of order in a given context (36). Standards may be developed nationally or internationally. Examples of international standards pertaining to the measurement of lead in paint are those developed by the International Organization for Standardization (ISO) and by ASTM International. These are listed in the annex.

Certification is the procedure by which an independent body gives written assurance that a process or service conforms to specific requirements. This involves inspection of the laboratory by representatives from a certification body, who are looking for evidence of compliance with standards, policies, procedures, requirements, and regulations. The main assessment is for the physical presence of procedures and documents (36).

Accreditation is the procedure by which an authoritative body gives formal recognition that the laboratory is competent to carry out specific tasks, for example quantification of the lead content in paint. In this case the laboratory is inspected by representatives from an accreditation body who, in addition to looking for evidence of compliance with standards, policies, procedures, requirements and regulations, also assess competency by observing the laboratory staff. The accreditation body may also set the standards with which the laboratory must comply (36). An example of an accreditation standard is ISO/IEC 17025 (37). In the United States, participation in ELPAT is a prerequisite for a laboratory to qualify under the Environmental Lead Laboratory Accreditation Program (ELLAP).
7. Conclusions

Lead paint is an important source of exposure to lead, in particular for children and workers. As paints with the desired colours and properties can be made without the use of lead additives, this is a source of exposure that is preventable. However, even where a country has restricted the use of lead in paint there may be a legacy of lead paint in buildings and structures that will continue to be a source of exposure for many years.

The primary action to prevent lead exposure involves the implementation of legally binding control measures such as laws, regulations or standards to stop the addition of lead-based ingredients to new paints. In countries where there are already buildings and other structures painted with lead paint, prevention may also involve the use of appropriate risk management measures, such as lead paint abatement. For both primary and secondary prevention purposes it is necessary to have the means to measure the lead content in paints.

This booklet outlines commonly used methods for measuring lead in paint, both in the laboratory and in situ where lead paint has been used. A range of different methods, involving varying costs and levels of complexity, are available and public health authorities, environmental agencies and others should choose the methods that best suit their needs.
8. References


# Annex

## International standards and methods for sample collection, preparation and analysis of lead in paint

<table>
<thead>
<tr>
<th>Procedure</th>
<th>Name of standard</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>International standards for sample collection</strong></td>
<td>ASTM E1729-16. Standard practice for field collection of dried paint samples for subsequent lead determination</td>
</tr>
<tr>
<td></td>
<td>ISO 15528:2013. Paints, varnishes and raw materials for paints and varnishes – sampling (available in English, French and Russian)</td>
</tr>
<tr>
<td></td>
<td>ASTM E1645-16. Standard practice for preparation of dried paint samples by hotplate or microwave digestion for subsequent lead analysis</td>
</tr>
<tr>
<td></td>
<td>ASTM E1979-17. Standard practice for ultrasonic extraction of paint, dust, soil and air samples for subsequent determination of lead</td>
</tr>
<tr>
<td><strong>International standards for test methods</strong></td>
<td>ISO 6503:1984. Paints and varnishes – determination of total lead – flame atomic absorption spectrometric method (for measurement of lead concentration of 0.01% to 2.0%) (available in English and French)</td>
</tr>
<tr>
<td></td>
<td>ASTM D3335-85a (2014). Standard test method for low concentrations of lead, cadmium, and cobalt in paint by atomic absorption spectroscopy (for measurement of lead concentration of 0.01% to 5.0%)</td>
</tr>
<tr>
<td></td>
<td>ASTM E1613-12. Standard test method for determination of lead by inductively coupled plasma atomic emission spectrometry (ICP-AES), flame atomic absorption spectrometry (FAAS), or graphite furnace atomic absorption spectrometry (GFAAS) techniques</td>
</tr>
<tr>
<td></td>
<td>ASTM F2853-10 (2015). Standard test method for determination of lead in paint layers and similar coatings or in substrates and homogenous materials by energy-dispersive X-ray fluorescence spectrometry using multiple monochromatic excitation beams</td>
</tr>
</tbody>
</table>

ISO standards can be obtained from [https://www.iso.org](https://www.iso.org)
ASTM standards can be obtained from [https://www.astm.org](https://www.astm.org)