

Analytical Methods for Measuring Lead in Paint

Toolkit for establishing
laws to eliminate
lead paint

Second Edition

Module C-2



Global Alliance to
Eliminate Lead Paint

Outline

- Objectives of Lead Paint Testing
- Options for Measuring Lead Paint – New and Existing Paint
- New Paint: Options for Laboratory Analysis of Lead Content
- Existing Painted Surfaces: Options for Off-site and On-site Analysis of Lead Content
- Issues Around National Laboratory Capacity for Measuring Lead in Paint
- Using Lead Paint Analyses to Investigate New Paints on the Market
- Summary and References

Objectives of Lead Paint Testing

- To determine if paint meets the regulatory requirement for permitted lead content
- Paint manufacturers and importers – to obtain documentation of compliance with lead paint limit
 - Third-party laboratory testing: use a nationally or internationally accredited laboratory that can measure the lead content to the required limit (e.g. 90 ppm) to support a Declaration of Conformity
- Government – to test for compliance with lead paint limit
 - Use a nationally or internationally accredited laboratory or suitable portable analysis technology to test for compliance with regulatory limit

Reasons for Analysing the Lead Content of Paint

- New paint for sale:
 - Assess the availability of lead-containing paint in the market and the need for better government regulation and enforcement
 - Provide consumers with information so they can choose non-lead paint and can push for government controls on lead paint
 - Draw attention to companies that produce lead-containing paint and encourage them to reformulate their products voluntarily
- Existing paint on structures:
 - Assess potential sources of exposure to lead from existing paint on structures, e.g. in homes, schools and playgrounds, and the possible need for mitigation measures

Options for Measuring Lead in Paint

New paint for sale:

1. Laboratory analysis (three methods:FAAS, ETAAS, ICP-AES)
2. High-definition portable X-ray fluorescence analysis (HDXRF)

Existing painted surface:

1. Laboratory analysis (three methods)
2. Portable X-ray fluorescence (XRF) analysis (on-site)
3. Chemical test kits (on-site)

The choice of method depends on several factors e.g. the level of accuracy required, the substrate to be tested (new paint or painted surface), the analytical equipment, and the cost.

New Paint: Total Lead Content

- Measuring total lead content is preferred over 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 (1)*

See also Module E-1.

Total lead

- Measured by extracting all the lead present in the paint
- Used in almost all national regulatory standards
- Promotes harmonization for exports to countries with total lead standards for products
- Provides a more predictable test for manufacturers who have test results from ingredients
- Low-cost, routine laboratory methods are available and many laboratories can do the measurements

New Paint: Soluble Lead Content

- **Soluble lead** (migratable lead) – measuring total lead content is preferred over soluble lead content
- Content is assumed to simulate the uptake of lead from the gut when lead paint chips or coated objects are swallowed (1).

BUT:

- No scientific basis to support this assumption
- Does not take account of exposure to lead in dust from deteriorating paint and does not provide the best measure of potential health risks
 - More expensive, more complicated laboratory method is needed
 - Technical modifications to paint can hide dangerous lead content, e.g. paint shown to have >17 000 ppm total lead content not detected by soluble lead test (2)

Reporting Units for Measuring Lead in New Paint

- **New paint:** Lead paint formulations and regulatory standards are usually expressed as a percentage by weight (% wt) or as parts per million (ppm), though some regulatory standards use milligrams per kilogram (mg/kg)
 - Laboratory analysis: lead content can be reported in ppm, % or mg/kg
 - High-definition X-ray fluorescence: lead content can be reported in ppm
 - $90 \text{ ppm} = 0.009\% = 90 \mu\text{g/g} = 90 \text{ mg/kg}$

Note: When analyzing lead paint to meet a 90-ppm legal limit, the method of analysis must be able to report out in ppm

Reporting Units for Measuring Lead in Existing Paint Surfaces

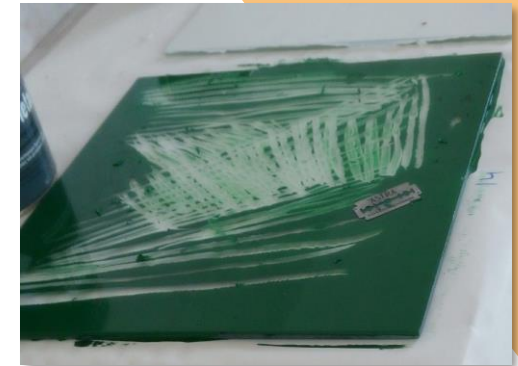
- **Existing painted surface:** Analysis of lead content may also be reported as the amount of lead per unit area: mg/cm^2 . There is no mathematical equivalence between ppm and mg/cm^2
 - Laboratory analysis: lead content can be reported in ppm, %, mg/kg or amount per unit area (mg/cm^2)
 - Portable X-ray fluorescence analysis (on-site): lead content is reported as mg/cm^2

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New Paint: Options for Laboratory Analysis of Total Lead Content

Sampling Methods to Test Paint

- Most common method is to apply paint to a homogeneous metal-free surface such as glass or wood (3)
- Paint is allowed to dry then a sample is scraped off for analysis in a laboratory
- Lead content may also be measured directly from surface using portable high-definition x-ray fluorescence and reported in ppm
- Measure lead in sample of wet paint using special sampling cup and high-definition x-ray fluorescence



Laboratory-Based and Portable Methods Available

- Laboratory-based instrumentation (various methods)
 - Samples are collected and sent to a laboratory, results available some time later
 - Destructive methods, e.g. atomic absorption spectrometry, require sample preparation stage
 - Non-destructive testing by high-definition X-ray fluorescence spectrometry (HDXRF)
- Portable methods
 - Hand-held X-ray fluorescence spectrometry (XRF)
 - Portable HDXRF devices can be used outside a laboratory e.g. at customs
 - Results are available immediately

Commonly Used Laboratory-Based Methods

- Three commonly used laboratory-based methods for measuring lead in paint are:
 - Flame atomic absorption spectrometry (FAAS)
 - Electrothermal or graphite furnace atomic absorption spectrometry (ETAAS/GFAAS)
 - Inductively-coupled plasma atomic emission spectrometry (ICP-AES)
- Some considerations:
 - Methods differ in complexity, limit of detection and cost
 - International standards exist for each

Method 1: Flame Atomic Absorption Spectrometry (FAAS)

- Relatively easy to use and moderate cost
- Can be fitted with auto-sampler so multiple samples can be processed
- Uses flammable gases so cannot be left to run unattended
- Limit of detection depends on sample preparation and method used – 100 ppm is typical

Note: 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 (*Howard Varner, personal communication, January 2020*).

Method 2: Electrothermal Atomic Absorption Spectrometry (ETAAS)

- Also known as graphite furnace atomic absorption spectrometry
- Can analyse very small samples
- Can be fitted with auto-sampler so large number of samples can be run
- Requires more staff training than FAAS
- Higher cost than FAAS
- Very low limit of detection



Method 3: Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)

- Can analyse very small samples
 - High purchase and maintenance costs but can be economical if used for large sample runs
 - Requires highly-skilled laboratory technician
 - Very low limit of detection
 - Can determine isotope ratio, which may help to identify the source of the lead



Laboratory Should Demonstrate Compliance with Quality Standards

- Trained personnel and good quality assurance procedures are essential to ensure accuracy and reliability of results
- Laboratory should have certification to show it works to an international standard e.g., ISO/ IEC 17025
- Laboratory should comply with national or international standards for sample preparation and analysis for lead in paint (*these standards are on the following slides*)
- Laboratory should be accredited to conduct analyses by a national or international accreditation program

International Standards for Sample Collection

- **ASTM E1729-16.** Standard practice for field collection of dried paint samples for subsequent lead determination
- **ISO 15528:2013.** Paints, varnishes and raw materials for paints and varnishes – sampling (available in English, French and Russian)

International Standards for Sample Preparation

- **ISO 1513:2010**, Paints and varnishes - examination and preparation of test samples
 - **ASTM E1645-16**, Standard practice for preparation of dried paint samples by hotplate or microwave digestion for subsequent lead analysis
 - **ASTM E1979-17**, Standard practice for ultrasonic extraction of paint, dust, soil, and air samples for subsequent determination of lead

International Standards for Test Methods

- **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)
- **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%)
- **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 (measurement of lead concentration differs according to analytical technique)
- **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

Note: ASTM has withdrawn E1613 but that it can be replaced by E3193 (as WK76000 - Main Addition to E3193-20 Analysis for Lead in Dust by Wipe, Paint, and Soil by FAAS - 11 Apr 2021) and E3203 (as WK76001 - Main Addition to E3203-19a Analysis for Lead in Dried Paint, Soil, and Wipes by ICP-OES - 11 Apr 2021), which are in the process of balloted modification for inclusion of standardized methods. (CPSC, personal communication, May 2021)

Considerations When Choosing a Laboratory

- Laboratory's experience in lead paint analysis
- If possible, accreditation through a recognized proficiency testing scheme
- Analytical methods used (e.g. FAAS, GFAAS/ETAAS, ICP-AES)
 - Limit of detection – is it adequate to confirm compliance?
- Sample requirements specified by laboratory
- Capacity to handle number of samples required
- Costs per sample, including any shipping costs
- Turn-around time

Environmental Lead Proficiency Analytical Testing (ELPAT) Program

- Operated by American Industrial Health Association
- ELPAT program assesses proficiency of laboratory in lead analysis of environmental samples, including for paint
- Laboratories shown to be proficient conduct lead paint testing to international standards
- Mostly in the US; non-US laboratories currently in Australia (2), Canada (6), France (1), Germany (1), Japan (2), Korea (1)
- Laboratories may work with clients in any country

Link: <https://www.aihapat.org/programs/environmental-lead-proficiency-analytical-testing-elpat-program>

X-Ray Fluorescence (XRF) Spectrometry (Portable Methods)

- Hand-held and portable conventional and high-definition XRF devices can be used in the field
 - Bench-top high-definition X-ray fluorescence (HDXRF) systems are available for use in the laboratory
 - Lead measurement results are available in minutes
 - XRF devices use ionizing radiation so specific health-and-safety and training needs

High-Definition XRF (HDXRF) (Portable Methods)

- Relatively new technology using optics to enable measurement of very low concentrations of lead
- Comparable results to conventional laboratory methods and suitable for compliance testing of new paints
- Approved as alternative to laboratory methods in the USA – ASTM F2853-10
- Sample painted on metal-free homogeneous surface and placed in front of device – lead concentration shown on screen within minutes
- Can accurately measure the lead content in liquid paint as well as in a dried paint film and can report results in ppm
- Expensive to buy but cheaper to operate than conventional laboratory methods

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Existing Painted Surfaces: Options for Off-Site and On-Site Analysis of Lead Content

Painted Surfaces: Options for Measuring Lead Content

1. Laboratory analysis (off-site)
2. Handheld conventional X-ray fluorescence (XRF) analysis
3. Handheld high-definition X-ray fluorescence (HDXRF) spectrometry
4. Chemical test kits (on-site)

Option 1: Laboratory Analysis for Existing Painted Surfaces

- Samples should be taken by trained personnel to ensure adequate quality
- When taking a paint chip sample from a painted surface:
 - paint must be removed from the underlying material, i.e. it is necessary to damage the painted surface
 - important to remove a precisely-measured area of paint
- Laboratory analysis involves multiple steps and takes additional time compared to on-site analysis

Note: See previous slides on new paint for information on laboratory test methods, standards, and considerations for choosing a laboratory

Option 2: Handheld Conventional X-Ray Fluorescence (XRF) Spectrometry

- Can be used at the site of the paint to be analysed
 - Paint surface does not need to be damaged
 - Good accuracy
 - Immediate results
 - Can measure many surfaces over a short period of time
 - Relatively high purchase cost, but cheaper to use than laboratory methods when many surfaces need to be tested
 - Requires training to ensure accurate results and observance of health and safety requirements: operator may need to be licensed and/or certified to use and transport the device.

Option 3: Handheld High-Definition X-Ray Fluorescence (HDXRF) Spectrometry

Same as for conventional XRF, in addition:

- Can report results as ppm
- Low detection limit (below 90 ppm)

Option 4: Chemical Test Kits

- Qualitative test for lead paint on walls or other surfaces
- Immediate results
- Low purchase and running costs and relatively simple to use
- Many limitations:
 - Cannot provide an accurate measurement
 - False positive and false negative results possible
 - Can test mainly top (surface) layers and may need to damage the paint surface to access lead paint
 - Special procedure may be needed for certain surfaces, e.g. plaster



Issues Around National Laboratory Capacity for Measuring Lead in Paint

Is In-Country Laboratory Capacity Essential for Compliance?

UNEP *Model Law and Guidance for Regulating Lead Paint* suggests:

“Current lack of in-country laboratory capacity need not be an impediment to a lead paint law going into effect, as industry can still comply with the law by sending paint samples to laboratories in other countries that are qualified to perform the required testing. Additionally, for imported paints, manufacturers and importers can rely on test results from qualified laboratories in the country of origin under the model law under certain circumstances.”

Increasing Demand for Laboratory Testing Creates a Market

- Regulations specifying a low limit on lead content of paint create a demand for laboratories to carry out compliance testing
- A laboratory can provide a service to manufacturers and regulatory authorities in multiple countries
- Establishing a laboratory service requires significant resources, therefore business case must be made
- May be possible to expand an existing laboratory service

Considerations for Building Laboratory Capacity

- Can lead paint analysis be added to an existing service:
 - Is the necessary equipment already available (e.g. ETAAS)?
 - What additional equipment (e.g. lamps), reagents and training are needed?
 - Is there a sufficient demand for the service to be sustainable (e.g. offering third-party testing to manufacturers, compliance testing for regulators)
- Is the service already available in a laboratory at home or abroad at a good price
- Large paint manufacturers have laboratory capacity and trained personnel that can be utilized to supplement testing by accredited labs



Using Lead Paint Analyses to Investigate the Paint Market

Conducting a Survey of Lead Content in New Paint for Sale

- Provides information about the extent of the problem and the need for regulatory or enforcement measures.
- Important to ensure that tested products are representative of all brands and include a range of colours:
 - Brightly coloured paints, e.g. yellow, red, orange, green, typically contain the highest levels of lead
 - Include low-lead colours such as white in the range



Linking Analytical Data to Paint Market Information

- Useful to link data about lead content of paints to information about the manufacturers selling paint on the national market
- Relevant information includes:
 - Available brands on the market, both locally-produced and imported products
 - Size of manufacturer and relative sales volume
 - Information on paint-can labels about ingredients, hazard warnings about lead, or statements indicating low lead content

Linking Analytical Data to Paint Market Information

- This information can be used:
 - as evidence when enacting and enforcing regulations on production, export / import, sales and use of paint with added lead
 - to show the need to include a labeling requirement in the regulation
 - in outreach to industry stakeholders for dialogue about regulatory controls on lead paint
- For additional information:
 - Description of how to conduct a market survey is in a 2013 UNEP/IPEN report (4)
 - Module C-3 of the toolkit provides paint study results in different countries

Summary

- Lead paint testing is a necessary part of enforcement and compliance activities of regulations to drive elimination of lead paint.
- Choice of analytical method to measure lead in paint depends on many factors, such as the reason for analysis, number of samples, cost limitations, need for precise measurement, etc
- For new paint, three good laboratory methods are available that vary in cost and limit of detection
- For existing painted surfaces, reliable measurement methods are off-site laboratory analysis or on-site, portable XRF (costs and limit of detection vary)
- International standards exist for laboratory competency, sampling and testing
- Market surveys of new paints for sale are used to determine the presence of lead paint and can provide evidence to justify regulation and to monitor compliance

References

1. Model law and guidance for regulating lead paint. Nairobi: United Nations Environment Programme; 2018 (<https://www.unep.org/resources/publication/model-law-and-guidance-regulating-lead-paint>, accessed 8 April 2021).
2. Brief guide to analytical methods for measuring lead in paint, 2nd ed. Geneva: World Health Organization: 2020 (<https://apps.who.int/iris/handle/10665/332932>, accessed 8 April 2021)
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4. Lead in enamel decorative paint: national paint testing results: a nine-country study (survey method described in pages 36-38). Nairobi: United Nations Environment Programme; 2013 (https://wedocs.unep.org/bitstream/handle/20.500.11822/22872/Lead_enamel_paint.pdf?sequence=1&isAllowed=y, accessed 8 April 2021)

Available WHO Resources: Brief Guide to Analytical Methods for Measuring Lead in Paint



<https://www.who.int/publications/i/item/9789240006058>

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