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Draft  
Jamaican Standard

Specification

for

**The Limit on Lead Content in Paints**

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**BUREAU OF STANDARDS JAMAICA**

**COMMENT PERIOD:  
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**Draft Jamaican Standard**  
**Specification**  
**For**  
**The Limit on Lead Content in Paints**

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Jamaican Standards establish requirements in relation to commodities, processes and practices, but do not purport to include all the necessary provisions of a contract.

The attention of those using this standard specification is called to the necessity of complying with any relevant legislation.

## Amendments

No.	Date of issue	Remarks	Entered by and date

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## Foreword

This standard was prepared by the Bureau of Standards Jamaica's Paint and Surface Coatings Technical Committee with the intention of setting a limit for the lead content in paints, varnishes and other surface coatings that are manufactured, imported, or traded nationally. The standard also provides requirements for sampling and analysis.

This standard is intended to be voluntary.

## Committee Representation

The preparation of this standard for the Standards Council, established under the Standards Act 1969, was carried out under the supervision of the BSJ's Paint and Surface Coatings Technical Committee which at the time comprised the following members:

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## Acknowledgement

Acknowledgement is made to the International Organization for Standardization (ISO) for the use of material taken from ISO and the Caribbean Community (CARICOM) Regional Organization for Standards and Quality (CROSQ) for the use of the material taken from the Caribbean Community Standard (CCS); the limit on lead in paints.

## Related Documents

This standard makes reference to the following:

ISO	Methods for sampling fluid paints and for the analysis for lead
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(ASTM)-D2369

Test method for Volatile Content of Coatings

CCS

The Limit on Lead Content in Paints

## **DJS 358: 2021 Draft Jamaican Standard Specification for the Limit on Lead Content in Paints**

### **1.0 SCOPE**

- 1.1** This standard prescribes a maximum limit on the lead content in paints and varnishes supplied in fluid form. It applies to paints and varnishes that are used in house-holds, on domestic furniture and appliances, in buildings and places open to the public, shops, offices, factories, or for commercial purposes.
- 1.2** This standard also applies to paints used for Industrial and Automotive applications.
- 1.3** This standard does not apply to paints supplied in the form of dried powders.
- 1.4** This standard includes methods of sampling and analysis.

### **2 NORMATIVE REFERENCES**

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 15528:2020	Paints, varnishes and raw materials for paints and varnishes — Sampling
ISO 1513:2010	Paints and varnishes — Examination and preparation of test samples
ISO 6503:1984	Paints and varnishes — Determination of total lead — Flame atomic absorption spectrometric method

ISO 3856-1:1984

Paints and varnishes — Determination of "soluble" metal content — Part 1: Determination of lead content — Flame atomic absorption spectrometric method and dithizone spectrophotometric method

(ASTM)-D2369

Test method for Volatile Content of Coatings

### 3.0 TERMS AND DEFINITIONS

**For the purpose of this standard, the following definitions apply:**

**3.1 Lead Content:** means the percentage by weight (mass) of lead (expressed as Pb) in a dried film of a paint or varnish, or in the total solids content of the paint or varnish.

### 4.0 MAXIMUM LEAD CONTENT OF PAINTS AND VARNISHES

**4.1** The maximum lead content of paints or varnishes shall be 0.009% (90 ppm) by weight, calculated on the basis of the total solids (non-volatile) content.

### 5.0 SAMPLING

**5.1** Sampling of paints or varnishes in fluid form shall be performed in accordance with Appendix A.

**5.2** Where the paint or varnish to be tested is in the form of a dried film on a surface, it should be removed from the surface with techniques which prevent or minimize contamination by the substrate material. Alternatively, a sample of substrate should be tested simultaneously, as a control.

### 6.0 ANALYSIS

**6.1** Where the paint or varnish is in fluid form, the total solids content shall be determined by the method given in Appendix B.

- 6.2** The lead content by weight of a paint or varnish is to be determined using one of the methods given in Appendices D and E, depending on the equipment and chemical reagents that are available, after extraction of the lead by the method given in Appendix C.
- 6.3** The lead content by weight of a paint or varnish is to be determined instrumentally using Appendix F.
- 6.4** The lead content by weight of a fluid paint or varnish is to be calculated as a percentage of the total solids (nonvolatile) content.

## APPENDIX A

### SAMPLING OF FLUID PAINTS

- A-0 SCOPE:** these procedures apply to varnishes and paints in fluid form. They do not apply to paints as dry powders.
- A-1** Paints may contain volatile, inflammable, or toxic solvents, and persons conducting sampling procedures should take appropriate precautions.
- A-2** Where the paint is prepackaged for distribution or sale, the following procedure shall be used.
- A-3** All paint containers of the same product found in one place or in one consignment shall be treated as one lot: however if paint from several batches can be identified, which can be separated, each batch shall be considered to be one lot.
- A-3.1** The number of containers (**N**) in the lot shall be ascertained and the number of containers, (**n**), to be selected at random to make up the sample should be the

whole number nearest to  $\mathbf{p(N/2)}$ .

For example:

Number in lot, N	Number in sample, n
2 - 10	2
11 - 20	3
21 - 35	4
36 - 50	5
51 - 70	6

Number in lot, N	Number in sample, n
71 - 90	7
91 - 125	8
126 - 160	9
161 - 200	10
over 200	10 + 1 for every additional 50

**A-3.2** No damaged or open containers shall be included in the sample

**A-3.3** Each container in the sample shall be opened and the contents examined for:

- a) presence of any surface skin (if any, remove it before proceeding);
- b) presence of any foreign matter;
- c) any separation of phases (liquid from solids);
- d) consistency, that is, if the paint has gelled (a thixotropic paint may appear to be gelled, but its consistency becomes more fluid on shaking or stirring);
- e) if the product is a varnish, its clarity and color should be noted; and
- f) settling, particularly if the material is deposited is hard and dry.

**A-3.4** If the paint in a container has gelled, or shows a hard and dry deposit, the container should be removed from the sample, and another drawn from the lot.

**A-3.5** For analysis, it is necessary that any skin should be dispersed and replaced in the container and any settled deposit re-incorporated in the medium (a palette knife or stirrer may be required). The container is then firmly closed, and the content thoroughly mixed by shaking, inverting and rolling.

**A-3.6** When all containers in the sample have been shaken etc., to homogenize the product, each shall be reopened and an equal volume removed from each and transferred to a clean, dry container, to form a “reduced sample”. The reduced sample should fill 95% of the container, which shall be closed tightly and labeled immediately with:

- a) manufacturer’s name, product description;
- b) manufacturing batch number or date of manufacture;
- c) details of consignment or location of lot;
- d) date of sampling, and place of sampling;
- e) number of ‘containers selected for the sample; and
- f) name of sampler.

**A-3.7** Reduced samples should be analyzed as soon as possible, but if any delay arises they should be stored in accordance with any recommendations given by the manufacturer.

**A-3.8** Any observations made in accordance with A-3.3 shall be included in the report of the analysis.

**(NOTE: These procedures are based on ISO 15528: 2020 and ISO 1513:2010).**

## APPENDIX B

### DETERMINATION OF THE TOTAL SOLIDS CONTENT BY WEIGHT OF A FLUID PAINT OR VARNISH

#### B-0 SCOPE

B-0.1 This method is to be used to determine the total solids content by weight of a sample of fluid paint or varnish.

#### B-1.0 PRINCIPLE

Test samples, after dispersion in a suitable solvent, are heated at  $110^{\circ}\text{C} \pm 5^{\circ}\text{C}$  for 60 minutes, and the percentage of solids remaining is found by weighing.

#### B-2.0 APPARATUS

The usual laboratory, apparatus, including:

- a) **Flat-bottomed Aluminum Foil Dishes**, about 60 mm in diameter, 20 mm deep; the dishes are to be dried for 30 minutes at  $110^{\circ}\text{C} \pm 5^{\circ}\text{C}$ , and cooled and stored in a desiccator before use;
- b) **Oven with Air Draft**, at  $110^{\circ}\text{C} \pm 5^{\circ}\text{C}$ ;
- c) **Weighing Containers**, either:
  - I. 5ml syringe;
  - II. test tube with stopper; or
  - III. weighing or dropping bottle.

#### B-3.0 REAGENTS

All reagents should be of analytical grade, except as specified. Distilled water shall be used, or its equivalent.

- a) Toluene - technical grade;
- b) 2-Ethoxyethyl Acetate - technical grade, ("Ethyl Cellosolve Acetate");

- c) if the paint cannot be dispersed in distilled water, toluene, or 2-ethoxyethyl acetate, use a solvent that is as volatile as the latter substance, (i.e., boiling range about 150°C to 160°C).

#### B-4.0 PROCEDURE

**B-4.1** Remix the reduced sample (**A-3.6**), if necessary on a mechanical shaker, or by stirring. Ensure that all air bubbles are removed.

**B-4.2** Using one of the weighing containers (**B-2.0 (c)** - preferably a syringe), weigh by difference a specimen of the paint or varnish into a tared aluminum dish, using:

- a)  $0 \pm 0.10$ g, if the volatile content is likely to be less than 40%;
- b)  $0.50 \pm 0.10$ g, if volatile content exceeds 40% by weight.

The aluminum dish should contain 2ml to 4ml of the chosen solvent, and the specimen is added dropwise, swirling the dish to disperse the specimen completely. If the material remains as a lump, try a different solvent (**B-3.0 (c)**). Prepare a duplicate specimen in the same way.

**B-4.3** Place the dishes and specimens in the oven at  $110^{\circ}\text{C} \pm 5^{\circ}\text{C}$  for 60 minutes, remove, and cool in the desiccator. Weigh to the nearest 0.1mg.

**NOTE:** if the material degrades or decomposes when heated as recommended, the temperatures and time recommended by the manufacturer for curing the produce should be used.

#### B-5.0 CALCULATION

The percentage of total solids in paint or varnish is given

by  $[(W2 - W1)/S] \times 100$

Where  $W1$  = weight of aluminum dish

$W2$  = weight of dish and specimen after heating;

and  $S$  = weight of specimen.

## APPENDIX C

### EXTRACTION OF LEAD FROM PAINTS FOR DETERMINATION OF 'TOTAL LEAD'

#### C-0 SCOPE

This method (dry ashing) is applicable to paints (as fluid, dried solids, or dried film) that contain lead in concentrations between 0.01% and 2% Pb by weight. The extracts may be used to determine the lead by flame atomic absorption spectrometry (Appendix D) or by a dithizone spectrophotometric method (Appendix E).

An alternative wet oxidation method, followed by EDTA extraction, is given in section 5 of ISO 6503 - 1984, but this uses concentrated sulphuric acid and hydrogen peroxide, or nitric acid, and is not recommended, as being somewhat hazardous.

#### C-1.0 DRY ASHING METHOD

**C-1.1 Principle** - a test portion of a reduced sample (Appendix A) or of a dried paint film is evaporated then ashed at 475°C, and any lead is dissolved in hydrochloric acid.

**C-1.2 Reagents** - all recognized and analytical purity; distilled water or its equivalent:

- a) Liquid paraffin;
- b) Hydrochloric acid. 180g/L, add 450mL 36% hydrochloric acid to 400ml distilled water, and dilute to 1L.
- c) Magnesium carbonate;
- d) Nitric acid, 315 g/L; add 1 volume of concentrated nitric acid, density 1.4 g/mL, to 2 volumes of distilled water;

- e) Sodium carbonate, anhydrous;
- f) Sodium sulphide solution, 10g/L;
- g) Sulphur.

**C-1.3 Apparatus** - basic laboratory apparatus:

- a) Analytical balance, accurate to 1mg;
- b) Hot plate, regulated; and
- c) Muffle furnace regulated to  $475^{\circ} \pm 25^{\circ}\text{C}$ .

**C-1.4 Preliminary** - Test the product for the presence of antimony (Sb) and cellulose nitrate, if its formulation is not known. If antimony is present, follow the procedure in **C-1.8**. If cellulose nitrate is present, note that liquid paraffin is to be added **C-1.5**.

**C-1.5 Test Samples**

Two test samples, taken from the reduced sample (or a dried film) are to be taken through the procedure, together with a 'blank' sample (including the reagents but omitting the test material).

Weigh about 5g (to the nearest 10mg) of reduced sample (or dried film) into a weighed silica crucible. If cellulose nitrate is present, add about 2g liquid paraffin in the crucible and mix thoroughly with the sample.

**C-1.6 Ashing**

Place the crucible and sample on the hot plate and heat up slowly in a fume cupboard to remove volatile solvents. Then cover the contents of the crucible with 2g magnesium carbonate and move the crucible slowly (during 10 minutes) into the muffle furnace at  $350^{\circ}\text{C}$ , and raise the temperature to  $475^{\circ}\text{C} \pm 25^{\circ}\text{C}$  over

60 minutes. Keep the crucible at this temperature until the paint is completely ashed. **DO NOT** let the sample ignite.

#### **C-1.7 Extraction (if antimony (Sb) not present)**

Let the crucible and ash cool, then place them in a 250mL beaker, add 100mL of hydrochloric acid (180g/L) and then boil gently on the hot plate for 15 minutes longer, followed by digestion for 15 minutes longer.

While still hot, filter the liquid by decanting it through a fine filter paper (Schleicher and Schull 589/3, 589/6, or Whatman 42,44) into a 250mL beaker. Wash the filter and residue with hot water, collecting the washings in the beaker.

Transfer quantitatively the filtrate and washings, when cool, to a 250mL volumetric flask, dilute to 250mL with water, and mix.

#### **C-1.8 Extraction (if antimony (Sb) present)**

Powder the ash (**C-1.6**) and add to it, in the same crucible, a mixture of 5g sodium carbonate and 5g sulphur. Cover the crucible, and heat over a moderate flame for 1 to 2 hours, until there is no odor of sulphur dioxide. Let the crucible cool, add a small amount of hot water, and digest till the mass breaks up. Filter, washing the entire residue on to the filter paper with sodium sulphide solution. Wash the residue with more sodium sulphide solution, and reject the filtrate.

Put the residue and the filter paper in a 250mL beaker, add, 15ml nitric acid (315 g/L) and boil gently for 15 minutes on the hot plate. Add 100ml hydrochloric acid (180 g/L) and digest for 30 minutes, then filter through a fine-texture filter paper into a 250mL beaker. Wash the residue and filter with hot water, collecting the washings in the

beaker. Transfer the filtrate and washings, when cool, to a 250mL volumetric flask dilute to 250mL with water, and mix.

**C-1.9 Test Solutions** - Aliquots of the extract solutions obtained at the end of the procedures in **C-1.7** and **C-1.8** are diluted to 100mL.

If the lead content is expected to be less than 0.4% take 25mL of extract solutions from **C- 1.8**, 5mL if between 0.4 to 1%; take 10mL if lead may be between 1 to 2%. Place the aliquots in a 100mL with water, and mix well.

#### **C-1.10 Standard lead solution**

A stock solution (1g Pb/L) is prepared, either from a commercial standardized sample, or by weighing 1.594 ( $\pm 0.001$ ) g of lead nitrate (dried at 105° for 2 hours) in some water in a 1000mL volumetric flask, adding 30mL nitric acid, diluting to 1000mL with water and mixing. This stock solution contains 1mg Pb/mL.

For use in analysis, 10mL of the stock solution are pipetted into a 100mL volumetric flask and diluted to 100ml with hydrochloric acid (18 g/L), and mixed.

This standard solution contain 100 $\mu$ g Pb/mL (0.1 mg/mL).

**(NOTE: This procedure is based on Section 4 of ISO 6503-1984).**

### APPENDIX D

#### **DETERMINATION OF LEAD BY FLAME ATOMIC ABSORPTION**

##### **SPECTROMETRY**

#### **D-0 SCOPE**

This procedure may be used to determine the concentration of solutions of lead in hydrochloric acids containing between 0 and 30 micrograms/mL of lead.

**D-1.0 PRINCIPLE:** The test solution is aspirated in an air/acetylene flame, and the absorbance of the 283.3nm spectral line emitted by a lead source is measured.

D-2.0 REAGENTS - All of recognized analytical grade; distilled water or its equivalent.

- a) Acetylene, commercial grade, in a steel cylinder;
- b) Compressed air;
- c) Standard lead solution, 100mg Pb/L (see **Appendix C-2.10**).

D-3.0 APPARATUS - Ordinary laboratory apparatus, and

- a) **Burette**, 50ml capacity, of certified accuracy;
- b) **Flame atomic absorption spectrometer**, burning air-acetylene, capable of absorbance measurements at wavelength 283.3nm;
- c) **Lead discharge lamp, or lead hollow-cathode lamp**;
- d) Volumetric flasks, 100mL, capacity, have certified accuracy.

D-4.0 PROCEDURE

**D-4.1 Calibration**

**D-4.1.1** On the day of use, prepare a series of standard matching solutions (Nos. 0 to 5), by adding to six 100mL volumetric flask the respective volumes (shown below) of the standard lead solution (100mg/L), using the burette. Add mL of hydrochloric acid (180g/L see **B-2.2 (b)**) to each flask, dilute to 100ml with water, and mix thoroughly.

Standard Matching solution (m.s.) Number	mL of standard lead solution used	concentration of Pb, µg/ml
0 (blank)	0	0
1	2	2
2	5	5
3	10	10
4	20	20
5	30	30

**D-4.1.2** Fit the lead lamp in the spectrometer, adjust the instrument as necessary. The monochromator should be adjusted for maximum absorbance in the region of 2.83mm. Adjust the flow of air and acetylene as required to operate the aspirator and burner, and ignite the flame.

**D-4.1.3** Aspirate into the flame, in increasing order of concentration, the six standard matching solutions and measure the absorbance of each. Between each measurement, aspirate water into the flame, and finally repeat the measurement with solution 4. The rate of aspiration shall be kept constant.

#### **D-4.1.4 Calibration graph**

Deduct the absorbances measured for Matching Solution 0 from the values measured for the other solutions. Plot a graph of these reduced absorbances against their concentrations of lead, mg Pb/mL.

#### **D-4.2 Determination**

- a) Repeat the measurement of the absorbance of Matching Solution 0, then measure the test solution (**C-1.9**) three times, followed by the Matching Solution 0 and M.S.4 (to verify that the response of the instrument is unchanged). Between each measurement aspirate water into the flame.
- b) If the absorbance of the test solution is greater than that of M.S.5, dilute by a known amount of water (giving a solution factor  $F = V_2/V_1$ ) and repeat the measurement.
- c) If the absorbance measurements for the test solution differ by more than 1% of their mean, repeat sequence (a).
- d) Take the mean of the absorbance measurements of the test solution, and deduct the value of M.S.0.

- e) From the calibration graph, determine the lead concentration of the test solution measured.

#### **D-4.3 Calculation**

The concentration of lead in the paint is given by

$$\% \text{ Pb, by weight} = (2.5' C' F) / M' V$$

Where **C** = Pb/ml of test solution, determined from calibration graph

**F** = dilution factor, if any (**D-4.2 (b)**);

**M** = mass of test sample in grams (**C-1.5**);

**V** = volume, in mL of aliquot of extract (**C-1.9**).

### APPENDIX E

#### **DETERMINATION OF LEAD IN EXTRACT FROM PAINT**

##### **DITHIZONE SPECTROPHOTOMETRIC METHOD**

#### **E-0 SCOPE**

This procedure can be used to estimate lead in concentrations between to 50 mg/L Pb, dissolved in hydrochloric acid (about 2.5 g/L) by measuring the color of the complex with dithizone at about 520mm wavelength, extracted with trichloroethane.

**E-1.0 Reagents** - Use distilled water or its equivalent, and

- a) Buffer Solution** - Dissolve 5g ammonium citrate, 3g potassium cyanide, 6g sodium metabisulphite in 200mL water, add 325ml concentrated ammonium hydroxide, and dilute to 1 litre with water.

**CAUTION: POTASSIUM CYANIDE IS EXTREMELY TOXIC.**

- b) Dithizone, Stock Solution** – 40mg dithizone dissolved in 100mL of 1,1,1, - trichloroethane.

- c) **Dithizone, Working Solution** - To be prepared on the day that it is to be used, by diluting 10mL of the stock solution **(b)** to 100mL with trichloroethane.
- d) Hydrochloric Acid - See **C-1.2 (b)**;
- e) **Hydroxylammonium Hydrochloride Solution** - Dissolve 20g of solid hydroxylammonium hydrochloride in 35mL water, and make up to 100mL.

**CAUTION: THIS SUBSTANCE IS IRRITANT AND TOXIC. KEEP AWAY  
FROM THE EYES AND SKIN.**

- f) **Lead, Standard Stock Solution** - See **C-1.10 (a)**;
- g) **Lead, Standard Solution** - See **C-1.10 (b)**;
- h) **1,1,1,-Trichloroethane** - analytical grade, free from inhibitors.

E-2.0 Apparatus - The usual laboratory apparatus, and

- a) **Burettes**, 10ml capacity, and 50ml capacity;
- b) **Pipette**, 10 ml capacity;
- c) **Separating Funnels**, 50ml capacity;
- d) **Spectrophotometer**, capable of measurements at about 520nm, using 5mm optical cells;
- e) **Volumetric Flasks**, 100mL capacity.

### E-3.0 Calibration

#### E-3.1 Standard Colorimetric Solutions

- a) Using the 50mL burette, place 15mL of the buffer solution in each of six 50mL separating funnels, then 1mL of the solution of hydroxylammonium chloride and 5mL of the dithizone solution, stopper the funnels, shake well for half a minute, and let the layers separate. Run off and discard the lower layers, and dry the insides of stems of the funnels with filter paper.
- b) Using the 10mL burette, add to the respective separating funnels the volumes of lead standard solution **(E-1.0 (g))** shown below

Colorimetric solution	Vol. lead standard solution, mL	concentration of lead, $\mu\text{m}/\text{mL}$
0 (blank)	0	0
1	1	1
2	2	2
3	3	3
4	4	4
5	5	5

Using the 10mL pipette, add 10mL 1,1,1,-trichloroethane to each funnel, stopper, shake well for half a minute, and let the layers separate. Run off the lower layers through filter paper (to collect water droplets) into six optical cells.

- c) Measure the absorbances of each of the dithizone extracts at a wavelength of 520nm against 1,1,1,-trichloroethane as reference.

Deduct the absorbances measured for the blank (colorimetric solution 0) from the values for the solutions. Plot a graph of these reduced absorbances readings against the Pb concentrations; this should be a straight line.

#### E-4.0 DETERMINATION

##### E-4.1

- a) In a 50mL separating funnel, prepare a buffered dithizone extraction solution following the procedure in **E-3 (a)**, running off the 1,1,1,-trichloroethane layer.
- b) Add to the separating funnel, using the 10mL burette, a volume of the test solution, prepared as in Appendix C, which will contain between 10 and 50 mg of Pb

(if the test solution contains more lead, dilute a known volume with hydrochloric acid (see **D-1.0 (d)**), to a known ratio, F, and add a measured amount to the separating funnel.

c) Mix the contents of the separating funnel, and let them stand a few minutes. Using the pipette, add 10mL of 1,1,1,-trichloroethane, stopper funnel, shake well for half a minute, and let the layers separate. Run off the lower layer through a filter paper, into an optical cell.

d) Measure the absorbance as in **E-3.1**

## E-5.0 CALCULATION

**E-5.1** The concentration of lead in the test solution is given by  $C = M \cdot F$ , where M is the lead concentration measured from the graph, and F is the dilution factor.

**E-5.2** The concentration of lead in the sample of paint is given by

$(2.5 C) / (M \cdot V)$ , where M is the mass in grams of the test sample (**C-1.5**) and V is the volume in ml of the aliquot of extract (**C-1.9**).

**NOTE:** This procedure is based on that given in **ISO 3856/1**.

## APPENDIX F

### DETERMINATION OF LEAD IN PAINT BY ED-XRF (Energy Dispersive x-ray fluorescence) METHOD

#### F-0 SCOPE

This test method is applicable for the products and materials with a lead (Pb) mass fraction range of 14 to 1200 mg/kg and is limited to paint and similar coatings. Metallic coatings are not covered by this test method.

#### F-1 PRINCIPLE

The test sample is irradiated using a source of x-rays resulting in the emission of characteristic

x-ray energies at 10.55 keV at the  $L\alpha$  line and 12.61 keV at the  $L\beta$  line, where the measured intensity of these energies allows for the quantification of lead.

## F-2 REAGENTS AND MATERIALS:

Isopropanol or ethanol

Nitric acid ( $HNO_3$ )

Hexane

Deionized water ( $H_2O$ )

Calibration Standards

## F-3 APPARATUS

ED-XRF Spectrometer

Designed for X-ray fluorescence analysis using multiple monochromatic excitation beams with an energy dispersive detector.

## F-4 PREPARATION AND PRESERVATION OF TEST SAMPLES AND TEST PIECES

### F-4.1 Paint

Two areas of the paint sample must be identified for this measurement as follows:

1. Identify an area of the sample that is coated by the target paint layer.
2. Identify an area of the sample that is not coated by the target paint layer and is representative of the substrate under the target paint layer. If such a portion of the substrate is not available, a small portion of the target paint layer must be removed without scraping the sample too vigorously as the substrate integrity may be affected, particularly in thin substrates.

Samples may be cleaned prior to measurement by removing any noticeable foreign substances.

Strong solvents that may affect the paint layer should not be used. Suitable solvents are:

- Isopropanol or ethanol (for removal grease)
- A solution of 5% HNO<sub>3</sub> in deionized water (for removal salts and mold release agents)

Special care must be taken in handling samples to prevent contamination of the portion of the sample to be analyzed by oils and salts from the skin.

#### F-4.2 Apparatus

Using the recommendations and requirements for the brand of the apparatus to be used:

1. Allow the apparatus to stabilize for normal operation
2. Set the X-ray tube conditions for each monochromatic beam
3. Set the correct detector parameters and calibrate the energy scale of the detector
4. Ensure that the spectral processing operates correctly according to manufacturer's guidelines

This method is non-destructive and as such the sample will be preserved.

#### F-5 PROCEDURE

Prepare the sample and the instrument according to the Preparation and Preservation of Test Samples and Test Piece Section

Place the paint layer sample in the X-ray beam path and perform the measurement selecting the paint layer/thin film mode.

Acquire spectra from the paint layer and substrate composite using the low energy and medium energy monochromatic beams or others.

Acquire spectra from the substrate only using low energy and medium energy monochromatic beams or others.

Process the spectrum.

## F-6 RESULTS

Using the net count rates for a sample and the instrument calibration data, the result is calculated by the instrument software in units of mg/kg or mg/cm<sup>2</sup>.

## F-7 REPORT

Report Pb content of the test sample calculated using units of mg/kg or mg/cm<sup>2</sup>, rounded to the nearest 0.1 mg for mass fractions  $\leq 100$  mg/kg or areic masses  $\leq 100$  mg/cm<sup>2</sup>, and rounded to the nearest 1 mg for mass fractions  $> 100$  mg/kg or areic masses  $> 100$  mg/cm<sup>2</sup>.

\* X-ray fluorescence spectrometers (particularly portable spectrometers) in paint layer/thin film modes typically report results in mass per area units (mg/cm<sup>2</sup> or  $\mu\text{g}/\text{cm}^2$ ).

## **Standards Council**

The Standards Council is the controlling body of the Bureau of Standards Jamaica and is responsible for the policy and general administration of the Bureau. The Council is appointed by the Minister in the manner provided for in the Standards Act, 1969. Using its powers in the Standards Act, the Council appoints committees for specified purposes. The Standards Act, 1969 sets out the duties of the Council and the steps to be followed for the formulation of a standard.

### **Preparation of standards documents**

The following is an outline of the procedure which must be followed in the preparation of documents:

1. The preparation of standards documents is undertaken upon the Standard Council's authorisation. This may arise out of representation from national organisations or existing Bureau of Standards' Committees of Bureau staff. If the project is approved it is referred to the appropriate sectional committee or if none exists a new committee is formed, or the project is allotted to the Bureau's staff.
2. If necessary, when the final draft of a standard is ready, the Council authorises an approach to the Minister in order to obtain the formal concurrence of any other Minister who may be responsible for any area which the standard may affect.
3. The draft document is made available to the general public for comments. All interested parties, by means of a notice in the Press, are invited to comment. In addition, copies are forwarded to those known, interested in the subject.
4. The Committee considers all the comments received and recommends a final document to the Standards Council
5. The Standards Council recommends the document to the Minister for publication.
6. The Minister approves the recommendation of the Standards Council.
7. The declaration of the standard is gazetted and copies placed on sale.
8. On the recommendation of the Standards Council the Minister may declare a standard compulsory.
9. Amendments to and revisions of standards normally require the same procedure as is applied to the preparation of the original standard.

### **Overseas standards documents**

The Bureau of Standards Jamaica maintains a reference library which includes the standards of many overseas standards organisations. These standards can be inspected upon request

The Bureau can supply on demand copies of standards produced by some national standards bodies and is the agency for the sale of standards produced by the International Organization for Standardization (ISO) members.

Application to use the reference library and to purchase Jamaican and other standards documents should be addressed to:

Bureau of Standards Jamaica

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