

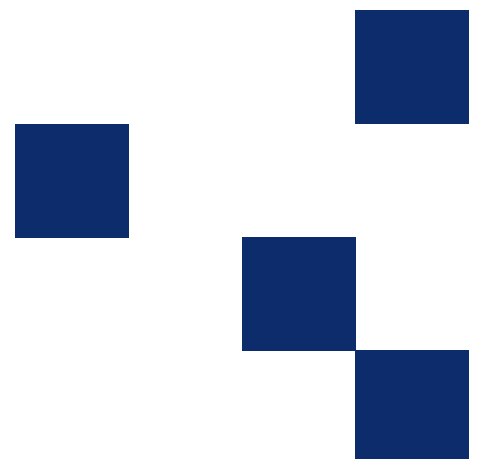


Transport
Roads & Maritime
Services

Test method T817

Determination of low concentrations of
lead in paint

JULY 2012



Revision Summary

Ed/Rev Number	Clause Number	Description of Revision	Authorisation	Date
Ed 1/ Rev 1		Reformatted and Revision Summary Added	D.Dash	June 2001
Ed 1/ Rev 2	All	Reformatted and Revision Summary added	D.Dash	July 2012

Note that Roads and Maritime Services is hereafter referred to as 'RMS'.

The most recent revision to Test method T817 (other than minor editorial changes) are indicated by a vertical line in the margin as shown here.

Test method T817

Determination of low concentrations of lead in paint

1. Scope

This test method sets out the procedure for determining the percentage of lead in paint when the lead is not a major constituent (less than 5 percent when based on the dry film). The method is specifically designed to include lead from both pigment and vehicle and is identical to the method set out in the American Society for Testing and Materials Designation D 2088-68.

2. Apparatus

- (a) A muffle furnace with a suitable temperature range, with temperature control within the range of $\pm 5^{\circ}\text{C}$ and preferably having suitable apertures at the front and rear to allow a slow natural draught of air to pass through.
- (b) A suitable electrolytic analysing apparatus fitted with platinum gauze anodes.
- (c) Laboratory glassware.

3. Reagents

Note: All reagents used are to be of AR grade and all water to be distilled.

- (a) Ammonium nitrate solution. Dissolve 200 g of ammonium nitrate (NH_4NO_3) in water and dilute to 1 litre.
- (b) Copper sulphate solution. Dissolve 1 g of copper sulphate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) in 1 litre of water.
- (c) Nitric acid (Density 1.42 g/mL).

4. Preparation of Sample

- (a) Transfer 10-15 g of well mixed paint to a tared evaporating dish and determine the mass.
- (b) Place the evaporating dish under a 250 W infrared heat lamp until the surface begins to char.
- (c) Transfer to a muffle furnace and submit to the following heating cycle:

20 minutes at 315°C

40 minutes at 425°C

1 hour at 538°C

Cool the sample in a desiccator and determine the mass of ash formed.

5. Determination of Lead

- (a) Weigh accurately 1 g of ash into a 400 ml beaker and moisten the sample with 2 mL of water.
- (b) Add 20 mL of concentrated HNO_3 and cover the beaker. Place the beaker in an aluminium cup or other device to prevent spattering and bake on a hot-plate to a paste or nearly to dryness. Remove from the hotplate and cool.
- (c) Take up the residue in 10 mL of concentrated HNO_3 and add 80 mL of water and bring to the boil.
- (d) Filter through a fine textured paper to remove insoluble residue and wash the residue several times with hot water.
- (e) Dilute the filtrate to approximately 300 mL and add 20 mL of NH_4NO_3 solution and 10 mL of CuSO_4 solution.
- (f) Determine the mass of the platinum gauze anodes (M_1).
- (g) Heat nearly to boiling and electrolyze immediately.

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- (h) Electrolyze for three periods of 15 minutes at 1, 2 and 3 amp current respectively. Rinse the electrodes three times in water with the current still on.
- (i) Remove the electrodes, rinse in alcohol in an oven at 105°C-110°C. Cool and determine the mass (M_2) of the electrodes.

6. Calculation and Reporting

Calculate the percentage of lead in the ash as follows:

$$\text{Lead percent} = A \times 0.86623$$

$$\text{Where } A = M_2 - M_1$$

= grams of lead oxide (PbO_2) found.

Calculate the percentage of lead on the weight of the original paint sample taken and report.