

Australian Standard™

Paints and related materials—Methods of test

Method 504.1: Metallic zinc content

PREFACE

This Standard was prepared by the Standards Australia Committee CH-003, Paints and Related Materials to supersede the 1993 edition.

METHOD

1 SCOPE

This method describes a procedure for determining the metallic zinc content in zinc dust and inorganic zinc silicate paints. The method is suitable for use with zinc dust and with solvent-borne or water-borne inorganic zinc silicate paints known by analysis to be free of other materials which react with acids to produce gaseous by-products.

2 REFERENCED DOCUMENTS

The following documents are referred to in this Standard:

AS

1152 Specification for test sieves

AS/NZS

3750 Paints for steel structures

3750.15 Part 15: Inorganic zinc silicate paint

ISO

383 Laboratory glassware—Interchangeable conical ground joints

3 PRINCIPLE

A finely ground sample of zinc dust or a dried film or zinc silicate paint is reacted with hydrochloric acid and the volume of hydrogen evolved is measured.

4 OCCUPATIONAL HEALTH AND SAFETY

During handling follow precautions for use outlined in suppliers Materials Safety Data Sheet (MSDS). Care should be exercised when handling acids.

5 REAGENTS

5.1 General

Unless otherwise specified, all reagents shall be of analytical quality. Distilled water or water of equivalent purity shall be used throughout.

5.2 Specific reagents

5.2.1 Hydrochloric acid/copper(I) chloride solution

Dissolve 0.05 ± 0.001 g of copper(I) chloride (Cu_2Cl_2) in 700 mL of hydrochloric acid (ρ_{20} 1160 kg/m³) and dilute to 1 L with water.

5.2.2 Levelling solution

The levelling solution shall consist of water acidified with several drops of hydrochloric acid (ρ_{20} 1160 kg/m³). Add methyl orange to colour the solution.

6 APPARATUS

The apparatus shall consist of the following:

- (a) *Burette* A water-jacketed gas burette, capacity 200 mL, graduated from 100 mL to 200 mL at intervals of 0.2 mL.
NOTE: A burette having a capacity of 400 mL, graduated from 300 mL to 400 mL, may also be used but this will necessitate changes in the size of the sample and the interval of graduation.
- (b) *Reaction flask* A water-jacketed reaction flask with a neck capable of taking a ground stopper of minimum size 24/29 complying with ISO 383.
NOTE: A flask with a capacity of about 250 mL is suitable for use with a 200 mL gas burette.
- (c) *Stopper* A conical ground glass stopper of minimum size 24/29 complying with ISO 383 with provision for being joined to the gas burette via rubber or plastic tubing.
- (d) *Levelling bottle*
- (e) *Boat* A boat, made from inert plastic materials, to hold the sample.
- (f) *Thermometer* A thermometer graduated in intervals of 0.1°C.
- (g) *Barometer* A barometer accurate to 1 mb.
NOTE: Suitable apparatus is shown in Figure 1.

7 PREPARATION OF SAMPLES

7.1 Zinc dust

Take sample of the zinc dust and, using an agate mortar and pestle, grind the zinc dust to pass a 75 µm sieve complying with AS 1152. Dry to constant mass in an oven at 105 C to 110 C.

7.2 Inorganic zinc silicate paint

The following method shall be used to prepare the test sample:

- (a) Apply the paint by a suitable method (see Note 1), as specified in the relevant product specification to a clean glass panel to provide a coating with a thickness of 65 µm to 85 µm (see Note 2).
NOTES:
 - 1 Suitable methods are brushing, spraying and pouring.
 - 2 A coating of the required thickness may be obtained by the application of 5 g to 6 g of paint to a panel of dimensions 150 mm x 100 mm.
- (b) Place the coated panel in one of the following environments, as applicable, for 24 h:
 - (i) Type 1 water-borne coating conforming to AS/NZS 3750.15, that requires heating in accordance with the manufacturer's specifications.

- (ii) Type 2 water-borne coating conforming to AS/NZS 3750.15, that requires a curing agent in accordance with the manufacturer's specifications.
 - (iii) Type 3 water-borne coating conforming to AS/NZS 3750.15, which self cures at $25 \pm 1^\circ\text{C}$, 50 ± 5 percent relative humidity.
 - (iv) Type 4 solvent-borne coating conforming to AS/NZS 3750.15, which self cures at $25 \pm 1^\circ\text{C}$, 50 ± 5 percent relative humidity.
- (c) Remove the cured coating from glass panel with a stainless steel spatula.
 - (d) Grind the coating in an agate mortar and pestle so that the particles pass through a $75 \mu\text{m}$ sieve.

8 TEST CONDITIONS

The test shall be performed at a temperature, which shall be maintained constant, to within $\pm 0.2^\circ\text{C}$ of the chosen temperature (see Table 1), by passing a steady flow of water through the apparatus.

9 PROCEDURE

The procedure shall be as follows:

- (a) Check the apparatus for leakage for air by connecting the empty reaction flask (A) (see Figure 1) to the burette (D) and, with the stopcock in a position to allow both (A) and (D) to be connected to the atmosphere, lower the level of the solution in the burette, using the levelling bottle, to the mid-point of the scale. Position the stopcock (E) to isolate both (A) and (D) from the atmosphere, and then raise the levelling bottle. If the system is airtight there should be no observable change in the position of the liquid in the burette after the initial displacement. If a change does occur, check for air leaks at (C) and (E).
- (b) Add 30 mL of the hydrochloric acid/copper(I) chloride solution (Clause 5.2.1) to the reaction flask (A).
- (c) Run water through the water jacket until the temperature is constant to $\pm 0.2^\circ\text{C}$.
- (d) Weigh $0.500 \pm 0.001 \text{ g}$ of the sample, prepared as in Clause 7, into the plastics boat. Transfer the boat and contents to the neck of the reaction flask.
NOTE: Care must be taken to ensure that the acid and the sample do not come into contact at this stage.
- (e) Connect the stopper (C) to the flask (A).
- (f) Position the 3-way stopcock (E) to open the gas burette (D) and the reaction flask (A) to the atmosphere. Adjust the fluid in the burette to zero by moving the levelling bottle (F) up or down. The level of the fluid in the burette and the levelling bottle should be equal.
- (g) Adjust the stopcock (E) to allow free flow of air between (A) and (D) and to isolate both from the atmosphere.
- (h) Measure the temperature of the water flowing through the water jackets of the flask and burette.
- (i) Tilt the reaction flask so that the sample comes into contact with the acid. When the initial vigorous reaction has subsided, periodically shake the flask so that all the sample reacts with the acid.

NOTE: Vigorous final shaking of the flask is essential.