

Australian Standard™

Paints and related materials—Methods of test

Method 302.1: Pigment content

METHOD

1 SCOPE

This Standard sets out a method of determining the pigment content of solvent-borne and water-borne paints. It is not suitable for some paints whose resins exist as solvent-borne emulsions, such as polyvinylidene fluoride (PVDF), or for non-aqueous dispersions (NAD). The method may not be suitable for paints containing pigments and extenders of very fine particle size such as colloidal silica, carbon black and some organic pigments.

2 REFERENCED DOCUMENTS

The following documents are referred to in this Standard:

AS

1580 Paints and related materials—Methods of test

1580.102.1 Method 102.1: Sampling procedure

3 PRINCIPLE

The method involves the separation of the pigment from the vehicle by centrifugal means and is based on differences in density between the pigment and the dilute vehicle. The extracted pigment is then dried, weighed and calculated as a percentage by mass of the whole.

4 OCCUPATIONAL HEALTH AND SAFETY

During handling follow precautions for use outlined in suppliers Materials Safety Data Sheet (MSDS).

5 APPARATUS

5.1 Centrifuge

Capable of achieving an operational centrifugal force to allow pigment segmentation.

NOTE: It is recommended that the centrifuge be explosion-proof or, alternatively, that shatter proof tubes with close-fitting caps be used to avoid spillage or vapour explosion. A centrifugal force between 10 kN to 20 kN is suitable.

5.2 Centrifuge tubes

90 mL, heavy walled glass or metal.

NOTE: Centrifuge tubes down to 25 mL capacity may be used. Where the tubes are less than 90 mL capacity, the quantities of sample and extraction solvent should be reduced accordingly.

5.3 Laboratory oven

Fan-forced ventilated, capable of maintaining a temperature of $105 \pm 3^\circ\text{C}$.

5.4 Weighing balance

Capable of weighing up to at least 100 g, to an accuracy of ± 0.0001 g.

6 MATERIALS

6.1 Extraction Solvent A

A solvent for the vehicle in which the pigment is not soluble.

6.2 Extraction Solvent B

A solvent with similar solvation properties to Solvent A but of a relatively low boiling point to facilitate drying of the final extract. Examples are petroleum spirit or acetone.

7 TEST SAMPLE

A representative sample of the paint shall be taken in accordance with AS 1580.102.1.

8 PROCEDURE

8.1 General

All weighings are to be carried out to an accuracy of 0.001 g.

8.2 Method

The quantities stated in the following method are based on the use of 90 mL centrifuge tubes; for smaller size tubes, proportionally smaller quantities of sample and reagent should be used. The procedure shall be as follows:

- (a) Dry a centrifuge tube to constant mass (m_1) at $105 \pm 3^\circ\text{C}$.
- (b) Thoroughly mix the sample.
- (c) Place a 5 g to 10 g specimen of the sample into the centrifuge tube and immediately weigh (m_2).
- (d) Add approximately 25 mL of Solvent A and mix gently using a glass rod.
- (e) Wash the rod down with Solvent A and make the total volume up to approximately 40 mL.
- (f) Make up a counterbalance for the centrifuge. This may be done with a duplicate preparation.
- (g) Place the tube and the counterbalance in the centrifuge holder.
- (h) Centrifuge at the lowest speed needed to achieve pigment settlement. The low speed precludes excessively hard packing.
- (i) Decant the supernatant liquid.
- (j) Repeat Steps (d) to (i) at least twice, thoroughly mixing the settled pigment into Solvent A each time prior to centrifuging.
- (k) Make a final extraction (Steps (d) to (i)) using Solvent B.
- (l) Decant the upper layer and evaporate any remaining solvent in a safe manner away from open flames or sparks.
- (m) Dry the tube and contents to constant mass (m_3) at $105 \pm 3^\circ\text{C}$.

NOTES:

- 1 Drying can be facilitated by breaking up the pigment by lightly tapping the centrifuge tube on a cloth pad on a bench top.
- 2 Cooling before weighing should be done in a desiccator.