

Australian/New Zealand Standard®

Paints and related materials—Methods of test

Method 503.1: Determination of water by the Dean and Stark method

PREFACE

This Standard was prepared by the Joint Australia/New Zealand Standards Committee CH/3 on Paints and Related Materials, under the direction of the Multitechnics Standards Policy Board.

The objective of this Standard is to revise the 1973 edition of AS 1580.503.1. Although the method is identical with AS 1580.503.1—1973, the Standard has been reformatted and refers to current Standards.

METHOD

1 SCOPE This method describes a procedure for determining water in paints or paint materials, other than water-based paints, using a Dean and Stark apparatus complying with AS R25.

2 REFERENCED DOCUMENT The following document is referred to in this Standard:

AS

R25 Dean and Stark apparatus

3 PRINCIPLE A sample of the test paint is mixed with toluene and a small volume of amyl or ethyl acetate and boiled in a flask connected to the Dean and Stark apparatus. Distillation is continued until water recovery ceases. The volume of water collected is then measured and reported as a percentage of the mass of the test sample.

4 APPARATUS AND REAGENTS

4.1 Reagents

4.1.1 Acetate—either reagent grade amyl acetate (ρ_{20} 870 kg/m³) or ethyl acetate (ρ_{20} 900 kg/m³).

4.1.2 Toluene (ρ_{20} 867 kg/m³).

4.2 Apparatus

4.2.1 Dean and Stark apparatus—of glass, employing a flask of 1 L capacity and a receiver of 2 mL capacity, meeting the requirements of AS R25.

4.2.2 Heating apparatus—consisting of an oil bath or a rheostat-controlled electric heating mantle, capable of accommodating a 1 L flask.

4.2.3 Boiling chips—non-reacting ceramic chips.

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5 PROCEDURE The procedure shall be as follows:

- (a) Ensure that the flask, receiver and condenser are clean and dry.

NOTE: It is important that the receiver and condenser should be thoroughly clean. They should, therefore, be treated with a mixture of potassium dichromate and sulphuric acid or other suitable cleansing agent prior to use. When the apparatus is in constant use it is usually sufficient to clean it after every third determination.

- (b) Weigh into the flask 200 g of the material under test, add 400 mL of the toluene and 1 mL of the amyl or ethyl acetate, a few boiling chips, and mix the contents of the flask well.
- (c) Pour toluene into the receiver up to the level of the side tube.
- (d) Connect the flask to the apparatus and place it in an oil bath or a heating mantle. Heat to a temperature that keeps the contents of the flask boiling briskly. Continue the distillation until no further water collects in the receiver.
- (e) At the conclusion of the determination, release any water still clinging to the condenser tube, causing it to fall into the receiver, by pushing a wire, or a small camel-hair brush (wet with toluene) attached to a glass rod, through the condenser tube from the top. Dislodge any condensed water on the sides of the receiver after dismantling the top part of the apparatus.
- (f) Read off the volume of water collected in the receiver to the nearest 0.05 mL.

6 CALCULATION Calculate the water content using the following equation:

$$\text{Water \% (v/m)} = \frac{V}{M} \times 100 \quad \dots 6(1)$$

where

V = volume of water collected, in millilitres

M = mass of sample, in grams

7 PRECISION The difference between two single results found on identical test material by one operator using the same apparatus within a short time-interval will not exceed the repeatability on average by more than once in 20 cases in the normal and correct operation of the method.

The difference between two single and independent results found by two operators working in different laboratories on identical test material will not exceed the reproducibility on average by more than once in 20 cases in the normal and correct operation of the method.

Results obtained within a laboratory or between laboratories shall be considered suspect if the values shown below of repeatability (r) and reproducibility (R), as appropriate, are exceeded:

Repeatability (r)

0.05 mL

Reproducibility (R)

0.1 mL

8 REPORT The report shall include the following information:

- (a) Name of testing authority.
- (b) Report number and date of issue.
- (c) Identification of paint under test.
- (d) The calculated percentage water in the paint sample, expressed to the nearest 0.01 percent.
- (e) Reference to this Standard, i.e. AS/NZS 1580.503.1.