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मानक

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Jawaharlal Nehru

“Step Out From the Old to the New”

IS 101-2-1 (1988): Methods of sampling and test for paints, varnishes and related products, Part 2: Test on liquid paints (chemical examination), Section 1: Water content [CHD 20: Paints, Varnishes and Related Products]



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Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

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Indian Standard

**METHODS OF SAMPLING AND TEST FOR PAINTS,
VARNISHES AND RELATED PRODUCTS**
PART 2 TESTS ON LIQUID PAINTS (CHEMICAL EXAMINATION)
Section 1 Water Content
(Third Revision)

1. Scope — Prescribes the Dean and Stark method for the determination of water content in paints. This method shall be used if the percentage of water content is more than 0.5 percent.

2. Principle — The material is refluxed with organic solvent which is immiscible with water. The carrier liquid distils into a graduated receiver carrying water with it which then separates to form the lower layer, the excess carrier liquid overflowing from the trap and returning to the still.

3. Apparatus

3.1 The Dean and Stark apparatus used for determination of water content has the following essential features.

3.1.1 Flask — 500 ml capacity, as shown in Fig. 1, and made of hard resistance glass, well annealed and as free as possible from striae and similar defects. Alternatively, a metal flask may be used.

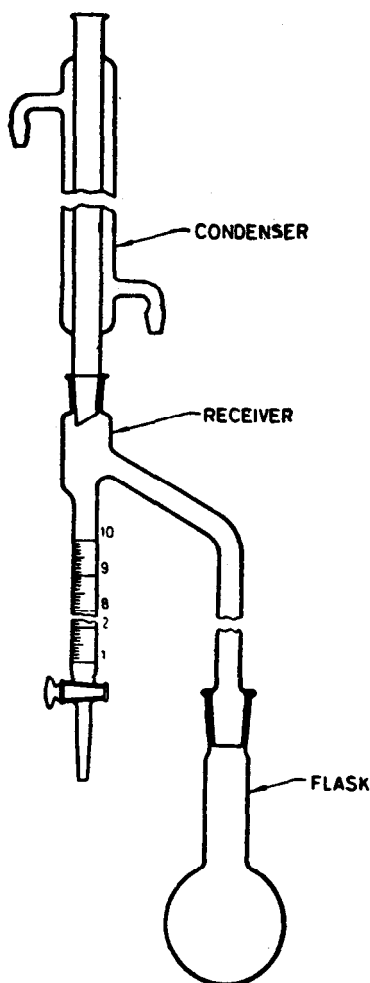


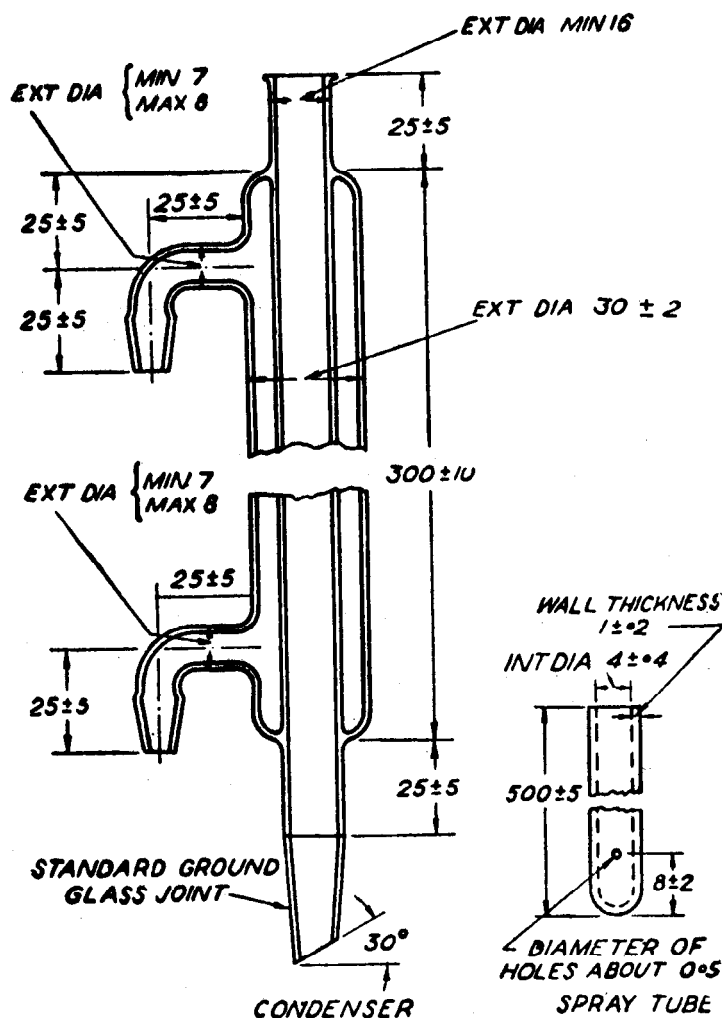
FIG. 1 DEAN AND STARK ASSEMBLY (WITH 10-ml RECEIVER)

3.1.2 Condenser — Made of hard resistance glass, well annealed and as free as possible from striae and similar defects, with shape and dimensions as shown in Fig. 2.

Adopted 30 March 1988

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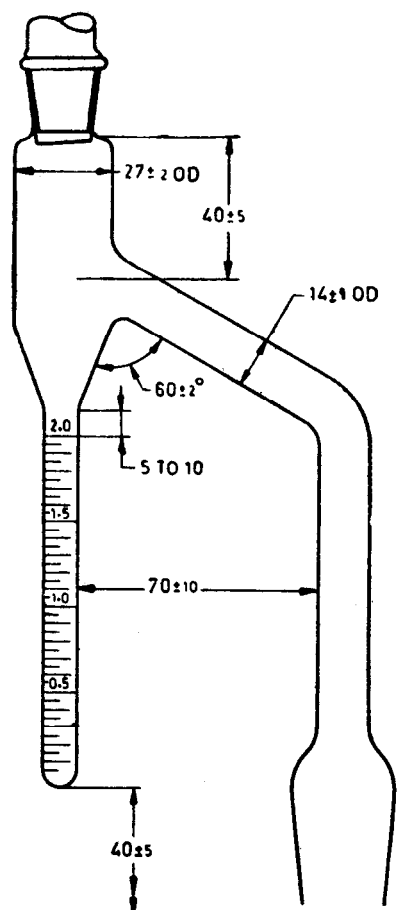
All dimensions in millimetres.

FIG. 2 CONDENSER AND SPRAY TUBE (DEAN AND STARK APPARATUS)

3.1.3 Spray tube — made of glass, sealed at one end, having four small holes equidistantly placed around the wall near the closed end of the tube, with the shape and dimensions as shown in Fig. 2.

3.1.4 Two-millilitre receiver — made of hard resistance glass, well annealed and as free as possible from striae and similar defects, provided with ground glass joints, and of shape and dimensions as given in Fig. 3. It consists essentially of the upper chamber together with the tube and ground joint leading to the flask and the graduated tube. When a metal flask is used, care shall be taken to provide an air-tight connection between the flask and the receiver. The graduated portion shall have a capacity of 2 ml at 20°C when filled to the highest graduation mark. The scale shall cover the range of 0.1-2 ml and shall be divided into intervals each of 0.05 ml. The graduation marks corresponding to 0.5 ml, 1.0 ml, 1.5 ml and 2.0 ml shall be numbered. The numbered graduation marks shall be carried completely round the tube. The graduation marks corresponding to 0.15 ml, 0.25 ml, 0.35 ml and so on up to and including 1.95 ml, shall be carried half way round the tube. The remaining graduation marks shall be intermediate in length and shall project equally at each end beyond the shortest graduation marks. The error at any point on the scale shall not exceed ± 0.03 ml and the difference between the errors at any two points shall not exceed 0.03 ml.

3.1.5 Graduated cylinder — 100 ml capacity.



All dimensions in millimetres.

FIG. 3 2-ml RECEIVER

3.2 Procedure — Weigh 100 g of the material in the flask, add 100 ml of dry toluene (see IS : 1839-1961 Toluene, reagent grade) and 1 ml of dry ethyl acetate [see IS : 229-1972 Ethyl acetate (*second revision*)] or amyl acetate (see IS : 231-1957 Amyl acetate) and thoroughly mix the contents of the flask. Pour toluene solvent into the receiver up to the level of the side tube. Attach the flask to the Dean and Stark condensing and collecting system and heat the flask at such a rate that the condensate falls from the end of the condenser at a rate of two-five drops per second. Continue the distillation until condensed water is no longer visible in any part of the apparatus except at the bottom of the graduated tube and until the volume of water collected remains constant. Remove the persistent ring of condensed water in the condenser tube, if any, by increasing the rate of distillation by a few drops per second. Wash droplets of water which adhere to the lower end of the condenser tube into the receiver with toluene solvent, using the spray tube.

3.3 Note the quantity of water in the receiver in ml and also the temperature at which the sample was measured. Assuming the density as 1.000 g/ml for the water collected in the receiver, calculate the percentage of water (by mass) in the material.

EXPLANATORY NOTE

This standard is one of a series of Indian Standards dealing with methods of sampling and tests for paints, varnishes and related products. This standard supersedes 14 of IS : 101-1964 'Methods of test for ready mixed paints and enamels (*second revision*)'.