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Indian Standard

METHODS OF SAMPLING AND TEST FOR PAINTS, VARNISHES AND RELATED PRODUCTS

PART 3 TESTS ON PAINT FILM FORMATION

Section 2 Film Thickness

(Third Revision)

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FOREWORD

This Indian Standard (Part 3/Sec 2) (Third Revision) was adopted by the Bureau of Indian Standards on 25 July 1989, after the draft finalized by the Paints and Allied Products Sectional Committee had been approved by the Chemical Division Council.

This standard (Part 3/Sec 2) is one of a series of standards on methods of sampling and test for paint, varnishes and related products.

In the preparation of this standard considerable assistance has been derived from ISO 2808 : 1974 "Paints and varnishes — Determination of film thickness", published by the International Organization for Standardization (ISO)".

Indian Standard

METHODS OF SAMPLING AND TEST FOR PAINTS, VARNISHES AND RELATED PRODUCTS

PART 3 TESTS ON PAINT FILM FORMATION

Section 2 Film Thickness

(Third Revision)

1 GENERAL

1.1 This standard (Part 3/Sec 2) prescribes methods for measuring the film thickness of paints and related materials. It is not intended to cover all methods available and seeks only to specify methods for determining film thickness under certain conditions.

1.2 Table 1 gives details on methods, their field of application and precision. Suggestions have been made in the test methods concerning the number and location of the test areas to be adopted for typical test panels. On other test panels and on painted articles the number and location of test areas shall be such as to provide a representative picture of the thickness of the paint film and shall be the subject of agreement between the interested parties.

2 DETERMINATION OF DRY FILM THICKNESS BY RELATING DRY FILM MASS TO DRY FILM THICKNESS

2.1 Scope

This specifies a method for checking that the thickness of a dried film of paint on a test panel lies within the limits specified to the relevant tests. The measurement is obtained by reference to a graph showing the relationship between film thickness and film mass of the material under test. This method is intended for use with airdrying paints which produce films that require several days before they are sufficiently hard to permit thickness measurements by instrumental methods.

2.2 Apparatus

2.2.1 Foil

Foil should be of plastics, resistance to a temperature of $105 \pm 2^{\circ}$ C and unaffected by paint solvents. Polyester films of 25 μ m thick has been found to be suitable.

2.2.2 Film Spreading Device

Film spreading device shall be capable of producing uniform wet films approximately 50 μ m and 100 μ m thick.

2.2.3 Glass Plates

Glass plates should not be less than 250 mm in length, 100 mm in width and about 6 mm thick, a size suitable for use with the film spreading devices.

2.2.4 Dial Gauge

Dial gauge should be capable of measuring accurately to $2 \mu m$, mounted on a rigid support.

2.2.5 Oven

Oven shall be capable of being controlled at $105 \pm 2^{\circ}$ C.

2.2.6 Metal Template, 80 mm square.

2.2.7 Analytical Balance, with accuracy of 1 mg or better.

2.3 Procedure

2.3.1 Cut a number of sheets of the plastics foil to the size of the glass plates.

Select six sheets and weigh each to the nearest milligram. Select four sheets whose masses do not differ by more than 3 mg.

2.3.2 Wet the surface of a glass plate with a solvent conforming to Type A of IS 1745: 1978 'Specification for petroleum hydrocarbon solvents (*second revision*)' and squeeze one of the selected sheets of foil into intimate contact with the surface of the glass plate, taking care to avoid air bubbles or any other surface irregularities.

2.3.3 Place a suitable quantity of the paint on one end of the sheet of foil and distribute it evenly over the foil using the 50 μ m film spreading device.

2.3.4 Repeat procedures 2.3.2 and 2.3.3 on a second sheet of foil using the 100 μ m film spreading device.

2.3.5 Remove the painted sheets of foil from the glass plates and after 15 minutes dry them in an oven, together with the two unpainted sheets, for 2 hours at $105 \pm 2^{\circ}$ C, maintaining the sheets in a horizontal position throughout the operation.

IS 101 (Part 3/Sec 2) : 1989

Table 1 Methods, Their Applications and Remarks

(Clause 1.2)

Number and Description	Applications	Remarks
Method 1 Determination of dry film thickness by relating dry film mass to dry film thickness	For use on films too soft to be measured by instrumental methods, for example, test panels of an air drying paint in early stages of hardening	Measurements are not precise but pro- vide a check that the mean thickness lies between specified limits. The test film remains undamaged
Method 2 Measurement of dry film thickness by micrometer method	Test panels or painted surfaces which are substantially flat	The film must be hard enough to resist indentation on closing the micrometer jaws. Accuracy is $\pm 5 \ \mu$ m. The method is not suitable for films less than 25 μ m thick. The film is damaged in test
Method 3 Measurement of dry film thickness by the dial gauge method	Test panels or painted surfaces which are substantially flat	The film must be hard enough to resist indentation on lowering the gauge presser foot. Accuracy is $\pm 2 \mu m$. The film is damaged in the test
Method 4 Measurement of dry film thickness. Microscope Methods A and B	 A) Measurement of film thickness to an accuracy of ±2.5 μm or better B) Measurement of film thickness to an accuracy of 1 μm 	A portion of the painted panel or article is cut out and mounted in resin. Recommended as a referee method and for films on substrates of varying prc- file, for example, grit-blasted metal. A special microscope is used to exa- mine the profile of the film from which a small portion is removed down to the substrate
Method 5		
Non-destructive instrumental me- thods β-ray backscatter method	For magnetic metallic sub- stances For non-magnetic metallic sub- stances	Instruments operate on: a) magnetic flux principle, or b) eddy current principle, or c) magnetic pull-off principle Instruments operate on an eddy cur- rent principle. Highly specialized instrument employing radio active sources. Paint films must be homo- geneous for measurement to be accurate
Method 6 Determination of wet film thick- ness	 a) Wheel gauge — For measurement of wet-film thickness on laboratory test panels or freshly painted surfaces b) Comb gauge — For measurement of wet film thickness during painting operations on use 	Measurements are not precise but enable an estimate to be made of the approximate thickness of the film when dry Measurements give a rough indication of thickness of the wet film NOTE - Dry film thickness should in both cases be checked by method No. 5

NOTE — In case where appreciable decomposition may occur under these drying conditions, other more suitable conditions may be used by agreement between the interested parties.

2.3.6 Remove all four sheets from the oven and allow them to cool for 1 hour at room temperature.

2.3.7 Using the template, cut two squares from the central area of each sheet.

Weigh each square to the nearest milligram.

Calculate the mean mass of the four unpainted squares

Calculate the mass of paint on each of the four painted squares by subtracting the mean

mass of the unpainted square from the mass of the painted square. Calculate the mass of paint film in grams per square metre.

2.3.8 Measure the thickness of each painted square with the dial gauge in six places and calculate the mean thickness for each square.

Measure the thickness of each unpainted square in six places with the dial gauge and so calculate the mean thickness of the plastics foil.

Calculate the thickness of the paint film on each painted square by subtracting the mean thickness of the unpainted squares from the thickness of the painted square. 2.3.9 Construct a graph showing the relation between the film thickness and film mass on the four painted squares, drawing the best straight line passing through the origin and between the plotted points.

2.4 Procedure for determining the dry film thickness on test panels. Use a weighed test panel prepared in accordance with the requirements of IS 101 (Part 1/Sec 3): 1986 'Methods' of sampling and test for paints, varnishes and related products: Part 1 Tests on liquid paints (general and physical), Section 3 Preparation of panels (*third revision*)'. Coat the panel with the material under test by the appropriate method. Allow the panel to dry for 24 hours at a temperature of $27 \pm 2^{\circ}$ C and a relative humidity of 65 ± 5 percent. Other ambient temperatures and relative humidities may be used by agreement between the interested parties. Weigh the panel and calculate the mass of the dry film in grams per square metre. Determine the equivalent film thickness by reference to the graph.

3 MEASUREMENT OF DRY FILM THICKNESS BY MICROMETER METHOD

3.1 Scope

Specifies a method for measuring the thickness of a dried paint film on a painted article or test panel to an accuracy of $\pm 5 \ \mu m$. The measurement is made after the film has dried to a condition such that the closure of the jaws of the micrometer does not produce any visible indentation of the film. The method is only suitable for flat surfaces such as sheet metal or similar material.

3.2 Apparatus

A suitable micrometer capable of measuring accurately to 5 μ m, fitted with a ratchet.

3.3 Procedure

Select the positions at which readings are to be taken. They shall be free from surface irregularities and shall be not less than 20 mm from any paint film edge and approximately 50 mm apart.

As a guide, suitable positions on a 150 mm \times 100 mm test panel are shown in Fig. 1, but for larger areas the number and distribution of the test areas shall be such as to give a representative indication of the film thickness.

Mark an area round each test position by lightly drawing a circle approximately 10 mm in diameter and add a distinctive number alongside. Support the painted specimen rigidly in a manner such that all the test positions are accessible to the micrometer. Position the micrometer with the fixed jaw in plane contact with the underside of the test specimen and immediately opposite the first test area. Gently screw



All dimensions in millimetres.

FIG. 1 MEASUREMENT OF DRY FILM THICKNESS: Selection of Positions for Measurement of Film Thickness on Panel 150 mm \times 100 mm

home the movable jaw until a resistance is felt and no further movement of the jaw occurs on turning the ratchet. Note the reading on the micrometer, using a mirror if necessary to read the vernier scale. Record the reading and position reference number on a test record sheet. Release the micrometer and repeat the whole procedure in each of the other test positions. Record the results as before. Remove the test specimen and carefully remove the paint film from within the circle at each test area with a suitable solvent or paint remover, taking care not to obliterate the distinctive number. For example, this may be done by covering the test area with a small circle of thick filter paper and applying a few drops of a suitable solvent. Repeat procedures as above at each test area and thus measure the thickness of the substrate.

3.3.1 Calculation

Calculate the film thickness at each test area by subtracting the second reading from the first. Calculate the mean value for the test panel, rounding the result to the nearest multiple of $5 \ \mu m$.

4 MEASUREMENT OF DRY FILM THICK-NESS BY THE DIAL GAUGE METHOD

4.1 This section specifies a general method for measuring the thickness of a dried paint film on a painted article or test panel to an accuracy of $\pm 2 \mu m$. The measurement is made after the film has dried to a condition such that the lowering of the presser foot of the instrument does not produce any detectable indentation of the film. The method is only suitable for painted specimens that are substantially flat.

4.2 Apparatus

A suitable dial gauge, capable of measuring accurately to 2 μ m, mounted on a rigid support.

NOTE — Some instruments have facilities for applying a load on the presser foot during operation. The load applied shall be such that no indentation of the paint film occurs during test.

4.3 Procedure

Select the positions at which readings are to be taken. They shall be free from surface irregularities and shall be not less than 20 mm from any paint film edge and approximately 50 mm apart. As a guide, suitable positions on a 150 mm \times 100 mm test panel are shown in Fig. 1, but for larger areas the number and distribution of tear areas shall be such as to give a representative indication of the film thickness. Mark each test position by lightly drawing a circle approximately 10 mm diameter and add a distinctive number alongside.

Set the reading on the dial to zero. Raise the presser foot and place the test specimen, paint film uppermost, so that the presser foot is immediately above the centre of the first test area. Support the specimen in such a way that no movement occurs during the taking of a reading. Carefully lower the presser foot until it is in good contact with the paint film. If, after making contact with the paint film the dial pointer does not remain steady, select a new test position and repeat the procedures. If the pointer again shows movement after making contact with the surface the paint film is not sufficiently dry and readings shall be discontinued until such time as a steady reading is obtained on lowering the presser foot. Record the reading and position reference number on a test record sheet. Repeat the procedure at each test position. Raise the presser foot and carefully remove the paint film from within the circle of the test area with a suitable solvent or paint remover, taking care not to obliterate the distinctive number. For example, this may be done by covering the test area with a small circle of thick filter paper and applying a few drops of a suitable solvent. Carefully lower the presser foot until it is in good contact with the cleaned surface. Record the readings and the position reference number on the test record sheet. Repeat the procedure at each test position.

4.4 Calculation

Calculate the film thickness at each position by subtracting the second reading from the first. Calculate the mean value for the test panel, rounding the result to the nearest multiple of 2 μ m.

5 MEASUREMENT OF DRY FILM THICK-NESS BY MICROSCOPE METHODS

5.1 This section specifies two methods in which microscopes are used for measuring the dry film

thickness of paint films on a variety of substrates.

Method A is a general method for measuring the thickness of a dried film of paint on a section cut from a test panel or painted article. It is recommended for use as a referee method in any dispute concerning the thickness of the paint film on a painted specimen. It is particularly useful in measuring variations in thickness such as occur due to unevenness of the substrate, for example, grit-blasted steel.

Method B employs apparatus by means of which an image of the surface profile of the test specimen is viewed in a special microscope. It does not involve cutting out a section of the specimen as described in Method A. The number of specimens prepared by either method shall be such as to be representative of the painted article or test panel.

5.2 Method A

5.2.1 Apparatus

5.2.1.1 *Microscope*, with an eyepiece capable of reading to an accuracy of 2.5 microns or better.

5.2.1.2 Abrasive paper, of water proof, silicon carbide abrasive paper.

5.2.2 Reagents

5.2.2.1 Cast resin

Cold setting potting or cast resin shall have no deleterious effect on the paint film. The colour of the mounting resin shall be such as to distinguish it clearly from the paint film under test. This may be achieved by the incorporation of suitable dyes or pigments into the resin.

5.2.3 Procedure

Cut test sections from the painted specimen with a sharp hacksaw. The painted area shall be approximately 25 mm square. Remove any burrs with abrasive paper. Cover a flat metal plate with a sheet of polyethylene film and place it in a horizontal position. Construct a small cylindrical cell from thin waxed cardboard of a size sufficient to contain the cut section. Fix the cell to the polyethylene film with molten parafin wax and allow the wax to cool. Support the cut section within the cell with a straight cut edge resting on the polyethylene film and the painted surface in a strictly vertical position.

NOTE — A suitable means of supporting the specimen is shown in Fig. 2.

Mix sufficient of the potting resin to cover the section, allow it to stand a few minutes to release air bubbles and carefully pour it into the cell, taking precautions to see that the painted surface remains in a vertical position. Allow the potted section to stand for 24 hours at room temperature. Remove the resin block from the polyethylene foil and rub down the face which was



FIG. 2 METHOD FOR PREPARING SPECIMEN FOR MEASUREMENT OF DRY FILM THICKNESS

in contact with the foil on the coarsest grade of abrasive paper using plenty of water as a lubricant. Support the paper on a flat glass plate. Continue abrading until the edge of the cut section is free from resin and the thickness of the paint film is fully exposed. Continue abrading until the edge of the cut section is free from resin and the thickness of the paint film is fully exposed. Continue abrading on the next finest grade of paper. Throughout abrading take great care to maintain the painted surface of the section at right angles to the plane of the abrasive paper, so as to avoid bevelling the cut edge. Examine the abraded surface periodically under the microscope to see if it is sufficiently smooth for reading to be taken. Finally polish the specimen on the finest grade of abrasive paper, rinse it under the tap and dry it with a clean soft rag. Mount the ported section on a microscope slide with the polished face upper-most and parallel to the plane of the side. (This is readily done by placing a piece of soft putty between section and slide and levelling the polished, surface with a spirit level.) Place the slide under the microscope and measure the thickness of the paint film by the scale on the eyepiece. Take measurements at seven places along the edge of the paint film and calculate the mean thickness. Where the film thickness is markedly variable along the specimen it is often useful to supplement the readings by pictorial illustrations such as photomicrographs or drawings.

5.3 Method B

5.3.1 Apparatus

5.3.1.1 Profile measuring microscope, consisting of an illuminator projecting a flat parallel bundle

of light on the surface at an angle of 45° and an objective viewing the reflected light bundle so that an image of the surface profile is seen in the microscope.

One instrument employs a special objective combining the illuminator and a reflected beam receptor. The eyepiece carries cross wires for focussing on the images of the portion of the beam reflected from the upper surface of the paint film and of the portion reflected from the exposed substrate. A vernier attachment measures the distance between the two portions of the reflected beam and thus enables the thickness of the film to be calculated.

NOTE — In favourable circumstances it is possible to make readings to an accuracy of $1 \mu m$.

5.3.2 Procedure

Using a sharp cutting tool, carefully remove a small portion of the paint film in such a manner as to completely expose a small area of the substrate, but taking care not to cut into the substrate (see Fig. 3). Reading d in eyepiece scale divisions is converted into the corresponding film thickness s (see Fig. 3) in micrometres. Direct the beam from the illuminator on to the area at an angle of 45° and along the length of incision. View the reflected image in the profile measuring microscope. The thickness of the paint film is determined by measuring the distance between the image of the paint film upper surface and the image of the exposed surface of the substrate using the scale of the measuring eyepiece or the vernier attachment fitted in the microscope. Using the calibration factor convert the reading in scale divisions into the

corresponing reading in micrometers. Figure 4 shows the appearance of a typical specimen as seen in the viewing microscope.



FIG. 3 SECTIONED VIEW OF INCISED PAINTS SPECIMEN



FIG. 4 TYPICAL IMAGE AS SEEN IN THE MICROSCOPE

6 NON-DESTRUCTIVE INSTRUMENTAL METHODS

6.1 Scope

This section specifies non-destructive methods for determining the thickness of dry paint films on metallic substrates. A variety of instruments is available for this purpose and they are classified under the following headings:

- a) nature of the substrate for which they are applicable that is non-magnetic metals, magnetic metals; and
- b) principle on which the instrument operates.

The methods described in this section are primarily intended for use in checking the thickness of paint films on painted articles; they may also be used for test panels by agreement between the interested parties. The thickness and contour of some substrates may render some instruments unsuitable for measuring the thickness of the surface coating and the instrument manufacturer's instructions in such cases shall be strictly observed. This is particularly the case with paint films on grit-blasted metal and the use of Method A (see 5.2) is recommended in such instances. Several readings shall be taken to obtain representative results over the painted area. The maker's instructions shall be strictly observed when making measurements with all instruments.

6.2 Measurement on Magnetic Metallic Substrates

6.2.1 Principle

A variety of instruments is available and they are classified according to the principle on which they operate.

6.2.1.1 Magnetic flux principle

Instruments in this class operate on the principle that the magnetic flux between a magnet and magnetic substrate varies according to the size of the air gap between the two or, in the case of painted substrates, the thickness of the nonmagnetic layer of paint film between magnet and substrate. The magnet may be either an electromagnet or a permanent magnet.

6.2.1.2 Eddy current principle

Instruments are available which operate on a similar principle to that described in **6.3** using a probe specially designed for use on magnetic substrates.

6.2.2 Apparatus

6.2.2.1 Electromagnet

This type of instrument requires a supply of electrical power and incorporates means of stabilizing the supply to an electromagnetic head.

The head is placed on an unpainted metal surface similar in nature to that bearing the paint film under test. A reading is taken and the operation repeated on the painted surface. The scale on the instrument is calibrated to indicate the thickness of the paint film shown by the difference between the two readings.

6.2.2.2 Permanent magnet

This instrument incorporates a permanent magnet with one or more poles in the form of spherical contacts which are placed on the painted surface. The dial reading is first adjusted to zero by placing the contracts on an unpainted substrate similar in nature to that of the test piece. Calibration is usually effected by the use of non-magnetic shims of known thickness supplied with the instrument, which are placed on the reference substrate and the reading adjusted by means of a control knob to indicate the thickness of the standard shim.

After calibration the instrument is placed on the painted surface and the dial reading noted. In order to avoid the effects of any magnetism in the substrate the instrument is turned through 180° and a second reading taken on the same area of film. If the second reading is different from the first the mean is taken.

Several readings are taken in a similar manner to obtain representative results over the area of the painted articles.

6.2.2.3 Magnetic pull off instruments

These instruments measure the force required to overcome the magnetic force of attraction between a permanent magnet and the magnetic substrate.

The force of attraction varies with the thickness of the non-magnetic paint film between magnet and substrate and is measured by the force applied by the tension in a spring coil connected to the permanent magnet.

6.2.3 Precautions to be Observed

No pole piece of the instrument shall be less than 25 mm from any edge of the test piece or other positions where the magnetic intensity of the instrument's field will not be uniform. The instrument shall be checked frequently against standard shims and the reference surface to ensure that the calibration is still correct. Additional checks may be made by placing the shims on the test surface and checking that the increase in readings obtained corresponds to the known thickness of the shims within the stated limits of accuracy of the instrument. Test specimens may readily become magnetised during mechanical handling and fabrication and this will affect the accuracy of the readings obtained. For this reason the control specimens used as reference surfaces shall be as similar as possible to the test specimens in respect of composition handling prior to measurement, etc.

6.3 Measurement on Non-magnetic Metallic Substrates

6.3.1 Principle

Two instruments are specified which operate on an eddy current principle.

Instruments in this class operate on the principle that changes occur in the apparent impedance of a probe coil due to the cddy currents which the alternating magnetic flux of the coil induces in the non-magnetic metallic substrate of the object under test. This in turn alters the amplitude of the alternating current flowing in the probe coil, and the change can be measured by means of a sensitive instrument to which the probe is connected. The magnitude of the

induced eddy current varies with the distance of the probe coil from the base metal, that is, the thickness of the dry paint film with which the coil is placed in contact. The measuring instrument is calibrated in such a manner as to indicate directly in thickness of the paint film.

Paint films containing metallic pigments can provide difficulties because of eddy current induced in these pigments.

6.3.2 Apparatus and Procedure

Two types of instrument are available, one requiring a supply power from the mains, the other being transistorized and powered by a selfcontained battery. The latter type is quite small and easily portable.

Both types require careful setting in the zero position on a reference surface similar in nature to that of the article under test. Calibration is then carried out using shims of special plastics foil supplied with the instrument in the case of the transistorized model; calibration is not normally necessary after zeroing the mains model.

Readings are taken by placing the probe of the instrument on the painted article and following the maker's instruction for taking a reading.

Several readings are taken in a similar manner to obtain representative results over the area of the painted article.

6.4 Other Methods

6.4.1 The use of other suitable methods is not precluded by this document but where used they shall be subject to special agreement between the interested parties.

An example of such a method is the β -ray backscatter method, which is a highly specialized method of measuring film thickness and employs radio active materials. Although rarely used in the laboratory, it is used in production control in some industries, for example, coating of steel, oil.

7 DETERMINATION OF WET FILM THICKNESS

7.1 Scope

This section specifies two methods of measuring the thickness of wet paint films. By using the correlation procedure described in 3, it is possible to estimate the approximate thickness of a dry film, by measuring its thickness immediately after application.

The methods specified are suitable for use on rigid substrates of suitable profile.

Both methods give only an approximate indication of the thickness of the film after drying and when suitably dry, the thickness of the film should be measured, using Method No. 6. The wheel gauge may be used both in the laboratory and in production control. The comb gauge gives only a rough indication of wet film thickness and consequently is used mainly during painting operations to check that major deviations from specified thicknesses are not occurring.

7.2 Wheel Gauge

7.2.1 Apparatus

The gauge consists of a wheel of which the perimeter has three equally spaced rims, the central one of which is smaller than and eccentric to the outer ones. When the gauge is rolled over a wet film, the eccentric central rim shows a position at which it just touches the wet paint surface, and a calibrated scale engraved on the outer wheel enables the wet paint thickness at this point to be noted. A range of gauges is available.

Several readings are taken in a similar manner to obtain representative results over the painted area.

7.2.2 Procedure

Immediately after the application of the paint, place the wheel gauge into the paint film so that the two outer rims are in a contact with the substrate at the point of maximum gap (that is, the largest reading on the calibrated scale). Rotate the wheel through at least 180° along the surface and remove. Examine the centre rim to determine the position at which contact was first made with the wet film surface. The calibrated scale shows the wet film thickness at this point.

Record the film thickness as the nearest lower scale division.

Take at least two further readings in different places in a similar manner to obtain representative results over the painted area.

7.3 Comb Gauge

7.3.1 The gauge consists of a comb the outer teeth of which form a base line. The inner teeth are progressively shorter so as to present a range of gaps between the teeth and the base line, and the size of each gap can be read from a scale on the gauge.

7.3.2 Procedure

Immediately after the application of the paint, place the comb gauge firmly onto the substrate in such a way that the teeth are normal to the plane of the surface. Remove the gauge and examine the teeth to determine which is the shortest one to touch the wet paint film. The film thickness is recorded as laying between the last 'touching' tooth and first 'non-touching' tooth as shown on the tooth calibrations marked on the gauge.

Take at least two further readings in different places in a similar manner to obtain representative results over the painted area.

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Review of Indian Standards

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