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Second series

ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION

R 787

GENERAL METHODS OF TEST FOR PIGMENTS

SECOND SERIES

Parts 8 to 11

1st EDITION
June 1970

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BRIEF HISTORY

relating to the Second series

The ISO Recommendation R 787, *General methods of test for pigments – Second series*, was drawn up by Technical Committee ISO/TC 35, *Paints and varnishes*, the Secretariat of which is held by the Nederlands Normalisatie-Instituut (NNI).

Work on this question led to the adoption of Draft ISO Recommendation No. 1251, which was circulated to all the ISO Member Bodies for enquiry in November 1968. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Australia	Israel	Sweden
Austria	Italy	Switzerland
Denmark	Netherlands	Turkey
Germany	Peru	U.A.R.
Greece	Poland	United Kingdom
India	Portugal	U.S.S.R.
Iran	South Africa, Rep. of	
Ireland	Spain	

The following Member Body approved Parts VIII, IX, X and XI of the Draft and abstained from voting on Part XII :

France

This Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided to accept it as ISO Recommendation R 787, Second series.

FOREWORD

The purpose of this ISO Recommendation is to establish a series of general test methods for pigments which are suitable for all or many of the individual pigments for which specifications might be required. In such cases, a cross-reference to the general method should be included in the ISO Recommendation relating to that pigment, with a note of any detailed modifications which might be needed in view of the special properties of the pigment in question.

Committee ISO/TC 35 decided that all the general methods should be published in convenient groups as they become available, as parts of a single ISO Recommendation, in order to emphasize the relationship of each to the whole series.

The Committee also decided that, where two or more procedures were widely used for determining the same or a similar characteristic of a pigment, there would be no objection to including more than one of them in the ISO series. In such cases it will, however, be essential to state clearly in a specification which method is to be used, and in the test report, which method has been used.

ISO Recommendation R 787, *General methods of test for pigments – First series,* was published in 1968 and includes the following methods :

Part I : Comparison of colour

Part II : Determination of matter volatile at 105 °C

Part III : Determination of matter soluble in water (Hot extraction method)

Part IV : Determination of acidity or alkalinity of the aqueous extract

Part V : Determination of oil absorption value

Part VI : Determination of residue on sieve (Oil method)

Part VII : Determination of residue on sieve (Water method)

This ISO Recommendation R 787, *General methods of test for pigments – Second series,* includes the following methods :

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GENERAL METHODS OF TEST FOR PIGMENTS

PART VIII

DETERMINATION OF MATTER SOLUBLE IN WATER (COLD EXTRACTION METHOD)

INTRODUCTION

This document is a part of ISO Recommendation R 787, *General methods of test for pigments.*

1. SCOPE

1.1 Part VIII of this ISO Recommendation describes a general method of test for determining the percentage by mass of matter soluble in cold water, in a sample of pigment.

1.2 Part III of this ISO Recommendation describes a method for determining the percentage by mass of matter soluble in water after hot extraction. For most pigments, these two test methods will give different results, and it is therefore essential to state clearly in a specification which method is to be used, and in the test report which method has been used.

NOTE. – When this general method is applicable to a given pigment, a cross-reference to it should simply be included in the ISO Recommendation relating to that pigment, with a note of any detailed modification which may be needed in view of the special properties of the pigment in question. Only when neither of the general methods is applicable to a particular pigment should a special method for determination of water-soluble matter be specified.

2. REAGENT

Distilled water, freshly boiled and cooled, of pH 6 to 7, or water otherwise prepared of at least equal purity.

3. APPARATUS

3.1 *One-mark volumetric flask* of 250 ml.

3.2 *Filter paper,* fine-textured, or *diaphragm,* or *colloid filter.*

3.3 *Evaporating dish,* flat-bottomed, of glass, platinum, glazed porcelain or silica.

3.4 *Oven,* capable of being maintained at a temperature of 105 ± 2 °C.

3.5 *Balance,* sensitivity 1 mg or better.

3.6 *Desiccator.*

4. SAMPLING

The sample of pigment used for the test should be taken in accordance with the provisions of ISO Recommendation R 842, *Sampling raw materials for paints and varnishes.*

5. PROCEDURE

5.1 Test portion

Weigh 2 to 20 g of the sample, to the nearest 0.01 g, into a beaker.

NOTE. – The mass of pigment used should be chosen according to the amount of water-soluble matter in the pigment. This is particularly important for pigments that contain large amounts of water-soluble matter.

5.2 Determination

Wet the pigment in the beaker with a few millilitres of water or other suitable liquid.

NOTE. – If the pigment does not disperse easily in water, a wetting agent should be used. In the case of pigments not soluble in ethanol, 5 ml of ethanol may be used; in the case of pigments soluble in ethanol, a non-ionic wetting agent such as 10 ml of a 0.01 % solution of a polyethylene oxide condensate should be used. If the wetting agent is non-volatile under the conditions of test an appropriate correction should be made to the final water-soluble matter figure after carrying out a blank determination.

Add 200 ml of freshly distilled water (2) (cooled to room temperature) and stir continuously for 1 hour at room temperature. Transfer to the volumetric flask (3.1) and dilute to the mark with the distilled water. Mix thoroughly by shaking and inversion and filter through the filter paper (3.2) or, in the case of pigments which are difficult to filter, through a diaphragm or colloid filter (3.2), returning the filtrate to the filter until it runs clear. If these methods of obtaining a clear filtrate do not prove satisfactory, use a decantation, centrifugal or other method which will do so. Evaporate 100 ml of the perfectly clear filtrate to dryness in the previously weighed evaporating dish (3.3) on a water bath.

Dry the residue in the oven (3.4) at 105 ± 2 °C, cool in the desiccator (3.6) and weigh to the nearest 1 mg. Repeat the heating and cooling until the results of the two last weighings, at an interval including at least 30 minutes heating, do not differ by more than 10 % of the final figure obtained for the water-soluble matter.

6. EXPRESSION OF RESULTS

Calculate the water-soluble matter (cold extraction method), as a percentage by mass, by the following formula

$$\frac{250\, m_1}{m_0}$$

where

m_0 is the mass, in grammes, of the test portion;

m_1 is the mass, in grammes, of residue.

Take the mean of two determinations not differing by more than 10 % (of the mean) and report the result to one decimal place.

7. TEST REPORT

The test report should include the following information :

(*a*) a reference to ISO Recommendation R 787, Part VIII, or to a corresponding national standard;

(*b*) type and identification of the pigment under test;

(*c*) any deviation, by agreement or otherwise, from the test procedure described above;

(*d*) the result of the test as indicated in section 6;

(*e*) the date of the test.

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GENERAL METHODS OF TEST FOR PIGMENTS

PART IX

DETERMINATION OF pH VALUE OF AN AQUEOUS SUSPENSION

INTRODUCTION

This document is a part of ISO Recommendation R 787, *General methods of test for pigments.*

1. SCOPE

Part IX of this ISO Recommendation describes a general method of test for determining the pH value of an aqueous suspension of a sample of pigment.

NOTE. – When this general method is applicable to a given pigment, a cross-reference to it should simply be included in the ISO Recommendation relating to that pigment, with a note of any detailed modification which may be needed in view of the special properties of the pigment in question. Only when this general method is not applicable to a particular pigment should a special method for determination of pH value be specified.

2. REAGENT

Distilled water, freshly boiled and cooled, of pH 6 to 7, or water otherwise prepared of at least equal purity.

3. APPARATUS

3.1 *pH measuring device,* consisting of a glass electrode, a reference electrode and a suitable potentiometer, calibrated against a buffer solution of known pH value.

3.2 *Glass container,* 50 ml, made of chemically resistant glass, fitted with a rubber stopper.

NOTE. – Before using the container for the first time, it should be boiled out with dilute hydrochloric acid, and then thoroughly rinsed with distilled water.

4. SAMPLING

The sample of pigment used for the test should be taken in accordance with the provisions of ISO Recommendation R 842, *Sampling raw materials for paints and varnishes.*

5. PROCEDURE

Prepare a 10 % (m/m) suspension of the pigment in the distilled water (2) (see Notes 1 and 2 below) and place it in the clean container (3.2). Stopper the container and shake it vigorously for 1 minute. Allow it to stand for 5 minutes, remove the stopper and determine the pH value of the suspension.

Carry out the determination at room temperature and record the pH value and the temperature to which it is related.

NOTES

1. If the pigment does not disperse easily in water, a wetting agent should be used; in the case of pigments not soluble in ethanol, 5 ml of ethanol may be used but care should be taken to ensure that the minimum quantity is used and that it is neutral and free from pyridine. In the case of pigments soluble in ethanol a neutral non-ionic wetting agent such as 10 ml of a 0.01 % (m/m) solution of a polyethylene oxide condensate should be used. The neutrality of the wetting agent can be checked by making a blank determination.

2. For some pigments with low relative densities, it may be necessary to use less than the 10 % suspension specified. In such cases, the pigment to water ratio used should be stated when recording the results.

6. EXPRESSION OF RESULTS

Report the mean of duplicate determinations to the nearest 0.1 unit as the *pH value of the aqueous suspension.*

7. TEST REPORT

The test report should include the following information :

(*a*) a reference to ISO Recommendation R 787, Part IX, or to a corresponding national standard;

(*b*) type and identification of the pigment under test;

(*c*) any deviation, by agreement or otherwise, from the test procedure described above;

(*d*) the result of the test as indicated in section 6;

(*e*) the date of the test.

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GENERAL METHODS OF TEST FOR PIGMENTS

PART X

DETERMINATION OF DENSITY RELATIVE TO WATER AT 4 °C

INTRODUCTION

This document is a part of ISO Recommendation R 787, *General methods of test for pigments.*

1. SCOPE

Part X of this ISO Recommendation describes a general method of test for determining the density, relative to water at 4 °C, of a sample of pigment.

NOTE. – When this general method is applicable to a given pigment, a cross-reference to it should simply be included in the ISO Recommendation relating to that pigment, with a note of any detailed modification which may be needed in view of the special properties of the pigment in question. Only when this general method is not applicable to a particular pigment should a special method for determination of relative density be specified.

2. PRELIMINARY CONSIDERATIONS

2.1 Displacement liquid

A liquid should be selected in which the pigment is insoluble, and which has good pigment-wetting properties and a low evaporation rate under a vacuum. High-boiling aromatic or aliphatic hydrocarbon solvents with boiling point over 170 °C are suitable.

2.2 Temperature of determination

The temperature t °C at which the determination is carried out will affect the relative density of the displacement liquid used but not that of the pigment. In order that the determination may be carried out conveniently in the laboratory, the temperature of the determination should be at least 5 °C above room temperature.

3. APPARATUS

3.1 *Pyknometer,* Gay-Lussac type, 50 ml capacity (see Fig. 1) with loose-fitting cap. Alternatively, another type of pyknometer fitted with a capillary stopper may be used.

3.2 *Vacuum apparatus,* of suitable design, for example one consisting of :

(*a*) *Vacuum desiccator,* fitted with a stopper. A glass tube with a three-way stopcock passes through the stopper and connects the desiccator to evacuation equipment.

(*c*) *Vacuum pump,* or other apparatus which is capable of reducing the pressure to a value not greater than 30 mbar (25 mmHg).

3.3 *Water bath,* thermostatically controlled, capable of being maintained to within ± 0.1 °C at a temperature in the range 25 to 30 °C (or at an agreed temperature t °C).

3.4 *Sieve,* with nominal mesh aperture of 0.5 mm.

3.5 *Balance,* sensitivity 1 mg or better.

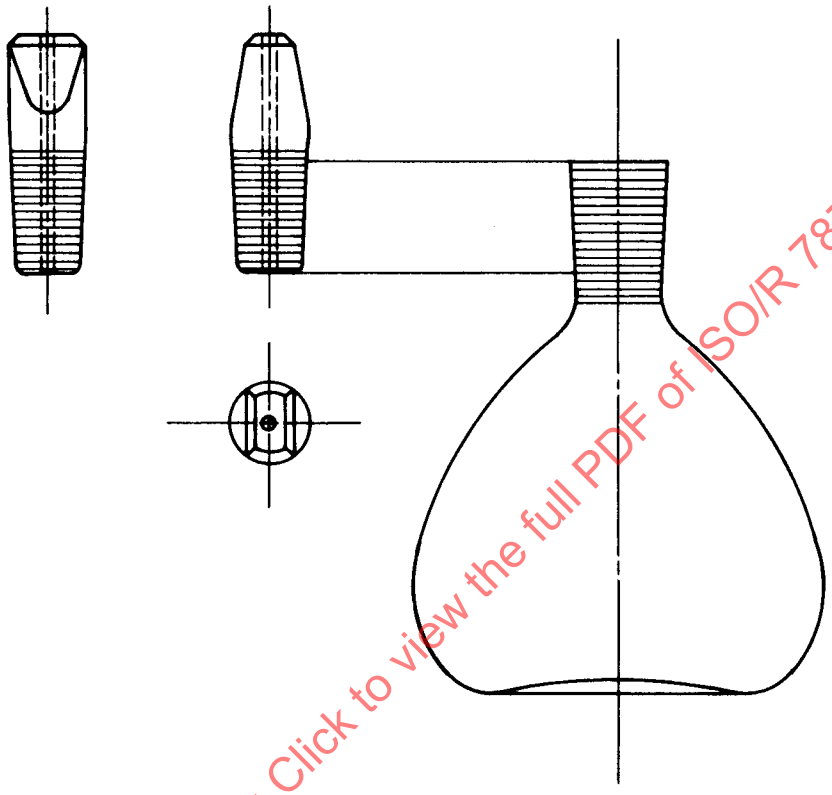

FIG. 1 – 50 ml Pyknometer, Gay-Lussac type

4. SAMPLING

The sample of pigment used for the test should be taken in accordance with the provisions of ISO Recommendation R 842, *Sampling raw materials for paints and varnishes.*

5. PROCEDURE

5.1 Test portion

Thoroughly mix the test sample and pass through the sieve (3.4). Dry a representative portion at 105 ± 2 °C* for 2 hours and allow to cool at room temperature in a desiccator.

Wash and dry the pyknometer (3.1), stopper, and cap and weigh to an accuracy of 0.001 g or better. Introduce into the pyknometer, by means of a dry funnel, a suitable quantity (2 to 20 g depending on the relative density) of the dried pigment so that the bottle is not more than half filled. Reweigh the stoppered pyknometer and cap.

5.2 Determination

Cover the pigment in the pyknometer with the displacement liquid (2) to a depth of about 13 mm and place the pyknometer in the vacuum desiccator (3.2 (*a*)). Close the three-way stopcock connecting the desiccator to the vacuum pump, start the pump and gradually open the three-way stopcock to the pump.

Allow the pyknometer to remain in the desiccator under reduced pressure (about 30 mbar) for about 4 hours or until all air bubbles have been removed from the liquid. Tap the desiccator occasionally to assist in removing entrained air. Stop the pump and gradually open the three-way stopcock to admit air into the desiccator until room pressure is restored.

Remove the pyknometer from the desiccator, fill it completely with the displacement liquid and place it in a water-bath (3.3) maintained at $t \pm 0.1$ °C. Allow the pyknometer to remain for 1 hour in the bath in order to attain the temperature of the bath, then carefully insert the stopper so that the excess liquid fills the capillary and wipe the liquid from the stopper. Remove the pyknometer from the bath, carefully wipe it dry and attach the cap. Transfer the pyknometer and cap to the balance case (3.5) and weigh after 15 minutes.

Empty, clean and dry the pyknometer and cap. Fill the pyknometer with the displacement liquid and, after allowing it to attain the temperature of the bath as previously mentioned, insert the stopper, wipe off the excess liquid, dry the pyknometer as in the previous operation, and attach the cap. Transfer the pyknometer and cap to the balance case and weigh after 15 minutes.

Finally again empty, clean and dry the pyknometer and cap and fill with distilled water. Carry out the same procedure as when using the displacement liquid.

6. EXPRESSION OF RESULTS

Calculate the relative density, d, of the displacement liquid at temperature t °C (relative to water at 4 °C), by the following formula :

$$\frac{m_4 - m_1}{f(m_5 - m_1)}$$

and the relative density of the pigment (relative to water at 4 °C), by the following formula :

$$\frac{d(m_2 - m_1)}{(m_4 - m_1) - (m_3 - m_2)}$$

* For pigments which decompose when dried under the conditions stated, the temperature and time should be adjusted to avoid decomposition.

where

d is the relative density of the displacement liquid at temperature t °C (relative to water at 4 °C);

m_1 is the mass, in grammes, of the pyknometer and cap;

m_2 is the mass, in grammes, of the pyknometer, cap and pigment;

m_3 is the mass, in grammes, of the pyknometer, cap, pigment and displacement liquid;

m_4 is the mass, in grammes, of the pyknometer with cap, filled with displacement liquid;

m_5 is the mass, in grammes, of the pyknometer with cap, filled with distilled water;

f is a factor for adjusting the density of water at t °C to density at 4 °C (see Table below for values of this factor at different temperatures).

TABLE

Temperature of water °C	f
15	1.0009
20	1.0018
25	1.0029
30	1.0043

Take the mean of two determinations and report the result to two decimal places as the *relative density* of the pigment (relative to water at 4 °C) at the temperature of the determination.

7. TEST REPORT

The test report should include the following information :

(*a*) a reference to ISO Recommendation R 787, Part X, or to a corresponding national standard;

(*b*) type and identification of the pigment under test;

(*c*) any deviation, by agreement or otherwise, from the test procedure described above;

(*d*) the result of the test as indicated in section 6;

(*e*) the displacement liquid used;

(*f*) the date of the test.

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GENERAL METHODS OF TEST FOR PIGMENTS

PART XI

DETERMINATION OF TAMPED VOLUME

INTRODUCTION

This document is a part of ISO Recommendation R 787, *General methods of test for pigments.*

1. SCOPE

Part XI of this ISO Recommendation describes a general method of test for determining the tamped volume of a sample of pigment.

NOTE. – When this general method is applicable to a given pigment, a cross-reference to it should simply be included in the ISO Recommendation relating to that pigment, with a note of any detailed modification which may be needed in view of the special properties of the pigment in question. Only when this general method is not applicable to a particular pigment should a special method for determination of tamped volume be specified.

2. APPARATUS

2.1 *Sieve,* 200 mm diameter, with a nominal mesh aperture of 0.5 mm.

2.2 *Tamping volumeter* (see Fig. 2), composed of :

(*a*) *Graduated measuring cylinder,* glass, capacity 250 ml, mass 220 ± 40 g.

NOTE. – If convenient, a measuring cylinder of different mass may be used provided that the total mass of cylinder and holder is 670 ± 45 g.

(*b*) *Holder for measuring cylinder,* with shaft, mass 450 ± 5 g.

(*c*) *Cam,* which lifts the shaft pestle and measuring cylinder once per revolution and revolves at a rate of 250 ± 15 rev/min.

(*d*) *Anvil,* on which the raised shaft falls from a height of 3 ± 0.1 mm.

(*e*) *Revolution counter,* to count the number of revolutions of the cam.

(*f*) *Sleeve,* to guide the shaft, constructed of a suitable material to give minimum friction.

NOTE. – The apparatus should be so constructed that, without undue free play, the friction between the shaft and the sleeve is as low as possible without the use of a lubricant.

2.3 *Oven,* capable of being maintained at a temperature of 105 ± 2 °C.

3. SAMPLING

The sample of pigment used for the test should be taken in accordance with the provisions of ISO Recommendation R 842, *Sampling raw materials for paints and varnishes.*

4. PROCEDURE

4.1 Take sufficient of the sample to carry out two determinations (i.e. about 500 ml), dry it in the oven (2.3) at 105 ± 2 °C for 2 hours and allow it to cool in a desiccator. Pass it through the sieve (2.1) to avoid agglomerations and add it to the graduated measuring cylinder (2.2 (*a*)) (previously weighed empty to the nearest 0.5 g) so that no air pockets are formed. Carry out this operation by holding the cylinder on the slant and turning it on its long axis while adding the pigment to a volume of approximately 200 ml. Weigh the cylinder and pigment to the nearest 0.5 g. Adjust the surface of the pigment so that it is horizontal, by carefully tilting the cylinder.

4.2 Place the cylinder in the holder of the tamping volumeter (2.2) and tamp it approximately 1250 times. Read off the volume of the pigment to the nearest 1 ml.

NOTE. – If the pigment surface is no longer horizontal after tamping, it should nevertheless be possible to estimate the volume to the nearest 1 ml.

Continue tamping in steps of approximately 1250 times, reading off the volume of the pigment after each step, until the difference between the volume at the end of two successive steps of 1250 tampings is less than 2 % of the larger figure. Record the latter figure as the volume of the pigment after tamping.

5. EXPRESSION OF RESULTS

Calculate the tamped volume, in millilitres per 100 g, by the following formula:

$$\frac{100\,V}{m_1 - m_0}$$

where

m_0 is the mass, in grammes, of the empty cylinder;

m_1 is the mass, in grammes, of the cylinder and pigment;

V is the volume, in millilitres, of the pigment after tamping.

Report the result, as the mean of two determinations, to the nearest 1 ml.

6. TEST REPORT

The test report should include the following information :

(*a*) a reference to ISO Recommendation R 787, Part XI, or to a corresponding national standard;

(*b*) type and identification of the pigment under test;

(*c*) any deviation, by agreement or otherwise, from the test procedure described above;

(*d*) the result of the test as indicated in section 5;

(*e*) the date of the test.

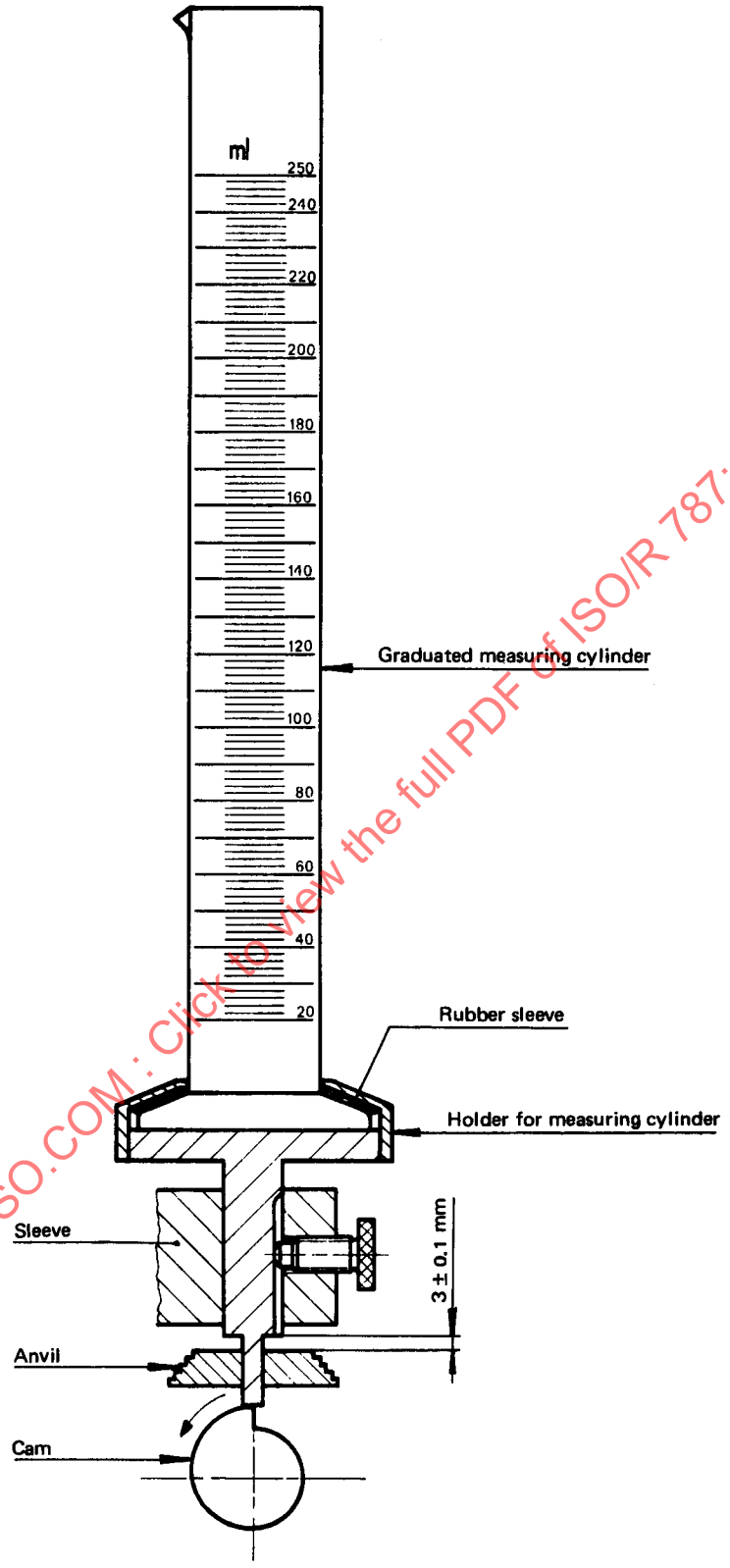

FIG. 2 – **Tamping volumeter**