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МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ

Petroleum products and lubricants — Petroleum oils and other fluids — Determination of rust-preventing characteristics in the presence of water

*Produits pétroliers et lubrifiants — Huiles de pétrole et autres fluides — Détermination des
caractéristiques anti-rouille en présence d'eau*

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Foreword

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International Standard ISO 7120 was prepared by Technical Committee ISO/TC 28, *Petroleum products and lubricants*.

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Petroleum products and lubricants — Petroleum oils and other fluids — Determination of rust-preventing characteristics in the presence of water

1 Scope and field of application

1.1 This International Standard specifies a method for evaluating petroleum oils and other fluids to indicate their effectiveness in preventing the rusting of ferrous parts should water become mixed with the oil/fluid. The method is for application to inhibited mineral oils including steam turbine oils, circulating oils and hydraulic oils and non-hydrocarbon fluids including fluids denser than water.

1.2 In many cases, such as in the gears of a steam-turbine, water can become mixed with the lubricant, and rusting of ferrous parts may occur. This test indicates the effectiveness of inhibited mineral oils aid in preventing this type of rusting.

2 References

ISO 3170, *Petroleum products — Liquid hydrocarbons — Manual sampling*.

ISO 3171, *Petroleum products — Liquid hydrocarbons — Automatic pipeline sampling*.

ISO 3448, *Industrial liquid lubricants — ISO viscosity classification*.

3 Principle

A mixture of 300 ml of the oil or fluid under test is stirred with 30 ml of distilled water or synthetic sea water, as required, at a temperature of 60 °C with a cylindrical steel specimen completely immersed therein. It is customary to run the test for 24 h; however, the test period may, at the discretion of the interested parties, be for a shorter or longer period. The specimen is then observed for signs of rusting and degree of rusting.

4 Reagents and materials

Unless otherwise stated, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

4.1 Cleaning and polishing fluid, *iso*-octane.

NOTE — For non-reference testing, petroleum spirit of satisfactory volatility may be used.

CAUTION — *iso*-octane is flammable. Do not use in an area near sparks or an open flame. Provide adequate ventilation.

4.2 Chromic acid cleaning solution or other equally effective glassware cleaning fluid.

CAUTION — Chromic acid is a highly corrosive and toxic substance. For environmental and/or toxicological reasons, its use is forbidden in many laboratories. In this case, use an alternative cleaning fluid which has been shown to produce chemically clean glassware.

Prepare chromic acid cleaning solution by dissolving 400 g of sodium dichromate ($\text{Na}_2\text{Cr}_2\text{O}_7$) in 100 ml of hot water. Allow the solution to cool and store in a glass-stoppered bottle. Place 35 ml of this saturated sodium dichromate solution in a 1 500 ml beaker. In a fume hood, while stirring, carefully add 1 litre of sulfuric acid (H_2SO_4). Stir for 5 min. Store in a glass-stoppered bottle.

4.3 Distilled water.

4.4 **Synthetic sea water**, for testing in accordance with procedure B, and conforming to the requirements given in 9.1 and 9.2.

4.5 Steel test specimen, as described in 7.1.

5 Apparatus

5.1 **Oil bath**, thermostatically controlled and capable of maintaining the test sample at a temperature of 60 ± 1 °C. An oil of viscosity grade ISO VG 32 is suitable for the bath. The bath shall have a cover with holes to accommodate the test beakers.

5.2 **Thermometer**, for checking the temperature of the test fluid with scale divisions of 0,5 °C with a specified immersion specification to allow measurement of the temperature in the test vessel at 60 °C with a precision at least equal to that of thermometer STL/0,5/ -35/ +115 of ISO 653.

NOTE — Thermometer conforming to ASTM 9C/IP 15C or ASTM 34C/IP 21C.

5.3 Beaker, 400 ml, tall-form heat-resistant borosilicate glass without pourout, as shown in figure 1, approximately 127 mm in height measured from the inside bottom centre and approximately 70 mm in inside diameter measured at the middle.

5.4 Beaker cover, flat, as shown in figure 2, of glass polymethyl methacrylate (PMMA) or chlorotrifluoroethylene, kept in position by suitable means such as a rim or groove.

Two holes shall be provided on any diameter of the cover; one for a stirrer approximately 12,0 mm in diameter with its centre approximately 6,4 mm from the centre of the cover; and the other, on the opposite side of the centre of the cover, for the test specimen assembly (see clause 7), approximately 18 mm in diameter with its centre approximately 16 mm from the centre of the cover. In addition, a third hole 12 mm in diameter shall be provided for a thermometer with its centre approximately 22 mm from the centre of the cover and on a diameter of the cover at right angles to the diameter through the other two holes. (Tolerances with respect to the dimensions are given in figure 2.)

NOTE — An inverted Petri dish makes a suitable cover, as the sides of the dish aid in keeping it in position. Figure 2 shows a PMMA cover for the beaker which has been found to be suitable. An optional feature is shown, consisting of a slot, 1,6 mm by 27,0 mm, which is centred on a diameter of the stirrer hole at right angles to the cover diameter through the specimen hole and stirrer hole. This feature allows withdrawal of the stirrer while the beaker cover is in place. If the test is used for other fluids such as synthetics, the beaker cover should be made from chemically resistant materials such as poly(chlorotrifluoroethylene) (PCTFE) and chlorotrifluoroethylene.

5.5 Stirrer, constructed entirely from 18/8 chromium nickel stainless steel in the form of an inverted T. A flat blade $25 \pm 1,0$ mm \times $6 \pm 0,5$ mm \times $0,6 \pm 0,1$ mm shall be attached to a 6 mm rod in such a way that the blade is symmetrical with the rod and has its flat surface in the vertical plane.

NOTE — If stainless steel is not obtainable, stirrers made of heat-resistant borosilicate glass and having the same dimensions as the stainless steel stirrers specified may be used.

5.6 Stirring apparatus, any convenient form, capable of maintaining a speed of $1\ 000 \pm 50$ r/min.

5.7 Grinding and polishing equipment.

5.7.1 Abrasive cloth, metalworking aluminum oxide, 97 μ m and 53 μ m (see the note) closed coat on a jeans backing.

NOTE — The corresponding grit sizes differ in Europe and the USA, the corresponding values being 280 grit and 240 grit respectively. A corresponding difference does not arise in respect of 150 grit.

5.7.2 Chuck, suitable for holding the specimen (see figure 4).

5.7.3 Rotation device, capable of rotating the specimen at a speed of 1 700 to 1 800 r/min.

5.8 Specimen assembly, as described in 7.1.

5.9 Oven, capable of maintaining a temperature of 64 ± 1 °C.

6 Sampling

Take a representative sample of the product to be tested according to ISO 3170 or ISO 3171 (or other relevant method).

7 Test specimen and its preparation

7.1 Requirements

The specimen assembly (5.8) shall consist of a round steel specimen fitted to a plastic holder. The plastic holder shall be made of PMMA resin in accordance with the dimensions shown in figure 3 (two types of holders are illustrated).

When testing non-hydrocarbon fluids, the plastic holder should be made from chemically resistant material such as polytetrafluoroethylene (PTFE). The round steel specimen when new shall be 12 to 13 mm in diameter and approximately 68 mm in length exclusive of the threaded portion which screws into the plastic holder and shall be tapered at one end as shown in figure 3. It shall be made of steel conforming to the specification given in annex A. If these steels are not available, other equivalent steels may be used, provided that they are found to be satisfactory by comparative test using this method. In cases of dispute, steel corresponding to the specification (annex A) shall be used for reference testing. The steel specimen, either new or from a previous test, shall be prepared as described in 7.2 and 7.3.

NOTE — When making a check test, the steel specimen that showed rust should not be re-used. Specimens that repeatedly show rust in tests of various oils may be imperfect. Such specimens should be used with oils known to pass the test. If rusting occurs in repeat tests, these specimens should be discarded.

7.2 Preliminary grinding

If the specimen has been used previously and is free from rust or other irregularities, the preliminary grinding may be omitted, and the specimen may be subjected only to final polishing as prescribed in 7.3.

If the specimen is new or if any part of its surface shows rust or other irregularities, clean it with cleaning solvent (4.1). Grind with 97 μ m aluminum oxide cloth (5.7.1) to remove all irregularities, pits, and scratches, as determined by visual inspection.

The specimen shall not be touched with the hands at any stage after cleaning with *iso*-octane (which precedes either preliminary grinding or final polishing) until the test is completed. Forceps or a clean, lintless cloth may be used. This precaution also applies to specimens that are to be re-used.

Perform the grinding by mounting the specimen in the chuck of the grinding and polishing apparatus, and turning it at a speed of 1 700 to 1 800 r/min while the 97 μ m aluminium oxide cloth (5.7.1) is applied. Old 97 μ m aluminum oxide cloth may be used to remove rust or major irregularities, but the grinding shall be completed with new cloth.

Proceed at once with the final polishing with 53 μm aluminum oxide cloth (5.7.1) or remove the specimen from the chuck and store in *iso*-octane until needed. Discard re-used specimens when the diameter is reduced to 10 mm.

7.3 Final polishing

7.3.1 Just before the test is to be made, subject the specimen to final polishing with 53 μm aluminum oxide cloth. If the preliminary grinding has just been completed, stop the motor which rotates the specimen. Otherwise, remove the specimen from the *iso*-octane (previously used unruled specimens shall be stored in this reagent), dry with a clean cloth and place in the chuck.

7.3.2 Rub a new piece of 53 μm aluminum oxide cloth longitudinally over the static specimen until the rounded end and the entire surface show visible scratches. Rotate the specimen at a speed of 1 700 to 1 800 r/min and polish with a strip of 53 μm aluminum oxide cloth by wrapping it halfway around the specimen, and applying a firm but gentle downward pull to the loose ends of the cloth for about 1 to 2 min so as to produce a uniform finely scratched surface free from longitudinal scratches. Carry out the final stages of the polishing with new cloth.

NOTE — To ensure that the flat shoulder (that portion of the specimen perpendicular to the threaded stem) is free from rust, this area should be polished. This may be done by holding a strip of 53 μm aluminum oxide cloth between the chuck and the shoulder while rotating the specimen for a brief period.

7.3.3 Remove the specimen from the chuck without touching with the fingers, wipe lightly with a clean, dry, lintless cloth or tissue (or brush the specimen lightly with a camelhair brush), attach to the plastic holder, and immediately immerse in the oil to be tested. This may be either the hot test portion (8.1) or a clean test tube containing a portion of the sample. The specimen may be removed later from this tube and allowed to drain briefly before being placed in the hot oil.

8 Procedure A for distilled water

8.1 The test shall be conducted in duplicate in order to report an oil as passing or failing. Clean the beaker (5.3) with chromic acid cleaning solution (4.2) or other equally effective glassware cleaning fluid (see 4.2, CAUTION), wash with distilled water to remove the acid completely. Dry in the oven (5.9). Clean a glass beaker cover (5.4) and a glass stirrer by the same procedure. To clean a stainless steel stirrer (5.5) and a PMMA cover (5.4), use cleaning solvent (4.1), wash thoroughly with hot water and finally with distilled water, and dry in the oven (5.9) at a temperature not over 65 $^{\circ}\text{C}$.

8.2 Pour 300 ml of the oil to be tested into the beaker and place the beaker in the oil bath (5.1) held at a temperature that will maintain the test portion at a temperature of 60 ± 1 $^{\circ}\text{C}$. The beaker shall be inserted into a hole of the bath cover and suspended in the hole with the beaker rim resting on the bath cover. The oil level in the bath shall not be below the oil level in the test beaker. Cover the beaker with the beaker cover with

the stirrer in position in the proper opening. Adjust the stirrer so that the shaft is 6 mm off centre in the beaker containing the test portion and the blade is within 2 mm of the bottom of the beaker. Suspend a thermometer (5.2) through the hole in the cover intended for that purpose so that it is immersed to a depth of 57 mm. Start the stirrer. When the thermometer reading reaches 60 ± 1 $^{\circ}\text{C}$, insert the steel specimen prepared in accordance with clause 7.

8.3 Insert the test specimen assembly through the specimen hole in the beaker cover and suspend so that its lower end is 13 to 15 mm from the bottom of the beaker. Either type of plastic specimen holder (see figure 3) may be used. The hole through which the specimen is suspended shall be unobstructed.

NOTE — Figure 1 shows the arrangement of the apparatus.

8.4 Continue stirring for 30 min to ensure complete wetting of the steel specimen. With the stirrer in motion, remove the thermometer temporarily and add 30 ml of distilled water through this hole, discharging the water from a pipette on to the bottom of the beaker, and replace the thermometer. Continue stirring at a speed of $1\,000 \pm 50$ r/min for 24 h from the time water was added, maintaining the temperature of the oil-water mixture at 60 ± 1 $^{\circ}\text{C}$.

8.5 Stop stirring at the end of the 24 h period, remove the specimen, allow to drain, and then wash with cleaning solvent. If desired, the specimen may be preserved by lacquering.

NOTE — In general, observations of rusting made at the end of 12 h testing time are indicative as to whether the oil will pass or fail. It is customary to run the test for 24 h; however, the test period may, at the discretion of the interested parties, be for a shorter or longer period.

9 Procedure B for synthetic sea water

9.1 The procedure for rust-preventing characteristics of inhibited mineral oils in the presence of synthetic sea water shall be identical with that described in 8.1, 8.2, 8.3, 8.4 and 8.5, except that synthetic sea water shall be used in place of distilled water in that portion of the procedure described in 8.4. The synthetic sea water shall have the following composition :

Salt	Concentration g/l
NaCl	24,54
$\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$	11,10
Na_2SO_4	4,09
CaCl_2	1,16
KCl	0,69
NaHCO_3	0,20
KBr	0,10
H_3BO_3	0,03
$\text{SrCl}_2 \cdot 6\text{H}_2\text{O}$	0,04
NaF	0,003

9.2 Preparation of synthetic sea water

9.2.1 The solution can be conveniently prepared as follows. This method avoids any precipitation in concentrated solutions with subsequent uncertainty of complete resolution. Using chemicals of recognized analytical grade and only distilled water or water of equivalent purity, prepare the following stock solutions :

Stock Solution No. 1 :

MgCl ₂ ·6H ₂ O	3 885 g
CaCl ₂ (anhydrous)	406 g
SrCl ₂ ·6H ₂ O	14 g

Dissolve and dilute to 7 l.

Stock Solution No. 2 :

KCl	483 g
NaHCO ₃	140 g
KBr	70 g
H ₃ BO ₃	21 g
NaF	2,1 g

Dissolve and dilute to 7 l.

9.2.2 Dissolve 245,4 g of NaCl and 40,94 g of Na₂SO₄ in a few litres of distilled water, add 200 ml of stock solution No. 1 and 100 ml of stock solution No. 2 and dilute to 10 litres. Stir the 10 l batch and add 0,5 mol/l Na₂CO₃ until the pH is between 7,8 and 8,2. One or two millilitres of the carbonate solution will be required.

10 Procedure C for heavier-than-water fluids

10.1 The procedure for testing the rust-preventive characteristics of heavier-than-water fluids is the same as described in clauses 8 and 9, but the stirrer action provided by the stirrer prescribed in 5.5 is not sufficient to mix thoroughly the water and test sample when the fluid under test is heavier-than-water. The changes relating to the apparatus in the method for testing heavier-than-water fluids are described in 10.2.

10.2 Apparatus

10.2.1 Beaker cover, as prescribed in 5.4. However, some heavier-than-water fluids may attack or dissolve PMMA covers and test specimen holders. It is, therefore, recommended that PTCFE beaker covers and PTFE specimen holders be used when testing heavier-than-water fluids.

10.2.2 Stirrer, as prescribed in 5.5 with an auxiliary blade attached to the stirrer shaft. The auxiliary blade shall be of stainless steel, 19 mm × 13 mm × 0,6 mm, as shown in figure 5. The auxiliary blade shall be positioned on the stirrer shaft so that the bottom edge of the auxiliary blade is 57 mm above the top edge of the fixed lower blade, and so that the flat surfaces of both blades are in the same vertical plane.

11 Expression of results

11.1 Express the results of the test as described below.

11.2 All inspections at the end of the test to determine the condition of specimens shall be performed without magnification under normal light. For the purpose of this test, normal light is considered to be illumination of about 650 lx. Within the meaning of this method, a rusted specimen is one on which any rust spot or streak is visible, by the inspection procedure described above.

11.3 An oil shall be reported as passing the test if both specimens are rust-free at the end of the test period. An oil shall be reported as failing the test if both specimens are rusted at the end of the test period.

An indication of the degree of rusting occurring in this test may be desired. For uniformity in such cases, use of the following classifications of rusting severity is recommended :

light rusting : rusting confined to not more than six spots, each of which is 1 mm or less, in diameter.

moderate rusting : rusting in excess of the above but confined to less than 5 % of the surface of the specimen.

severe rusting : rusting covering more than 5 % of the surface of the specimen.

If one specimen is rusted while the other is free of rust, tests on two additional specimens shall be made (see note following 7.1). If either of these latter specimens shows rusting, the oil shall be reported as not passing the test. If neither of these latter specimens shows rusting, the oil shall be reported as passing the test.

11.4 Test report

The test report shall contain at least the following information :

- the type and identification of the product tested;
- a reference to this International Standard, indicating whether procedure A, B or C was used and, in the case of procedure C, whether distilled or sea water was used;
- the period of time for which the test was conducted;
- the result of the test (see clause 11);
- any deviation, by agreement or otherwise, from the procedure specified;
- the date of the test.

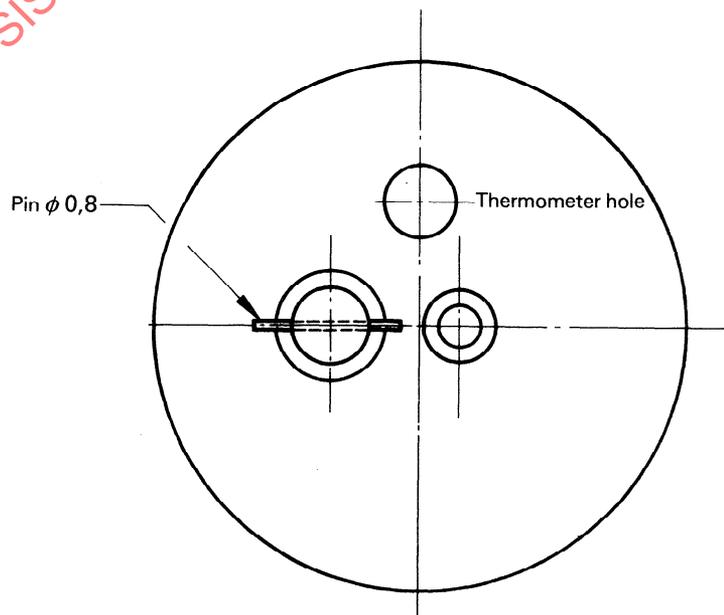
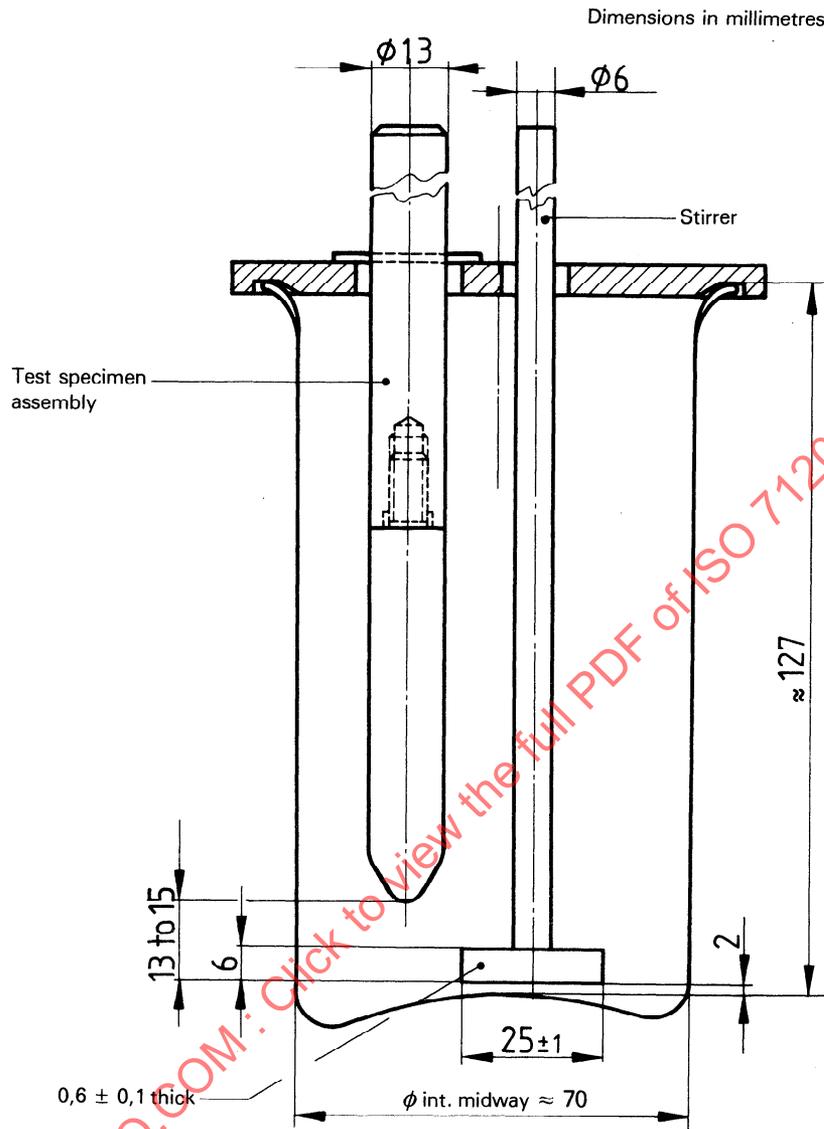


Figure 1 — Rusting test apparatus

Dimensions in millimetres

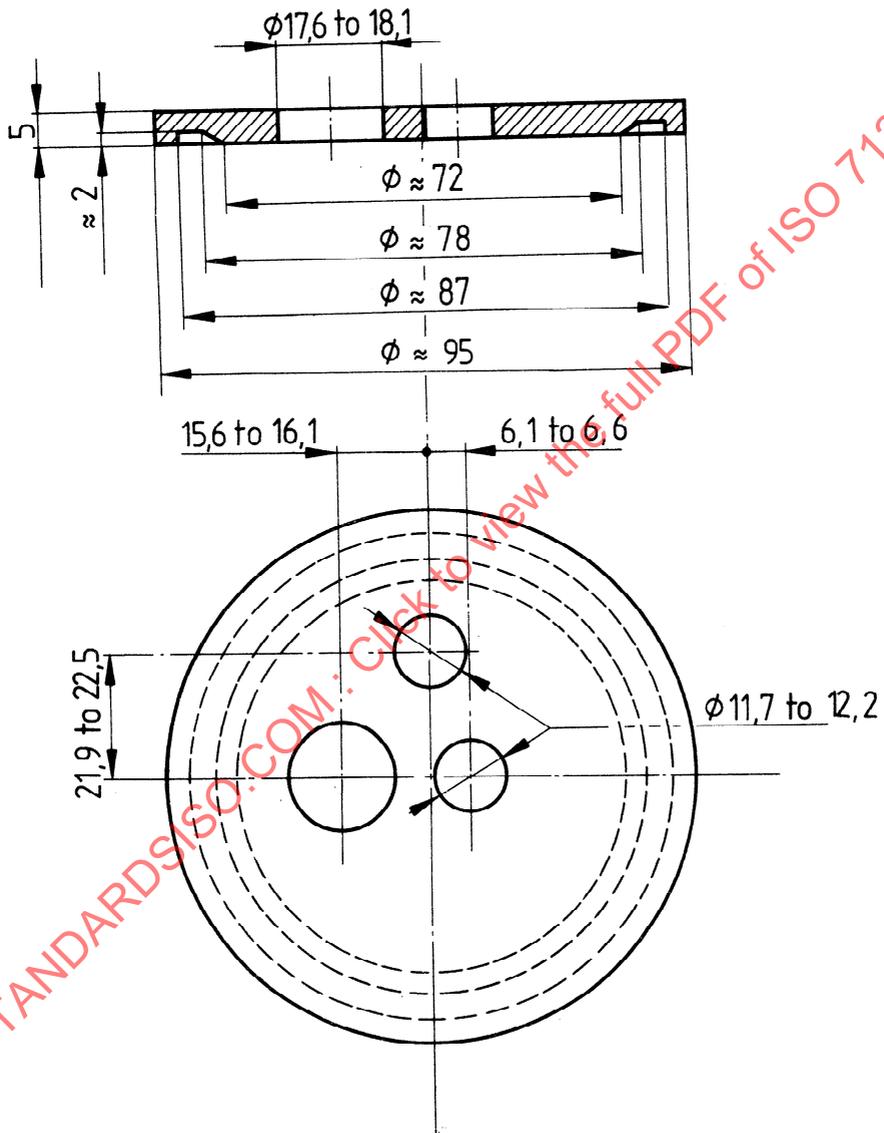
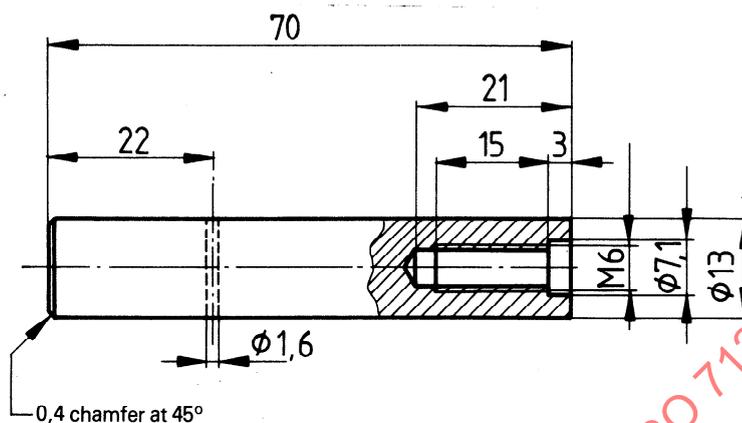


Figure 2 — Beaker cover

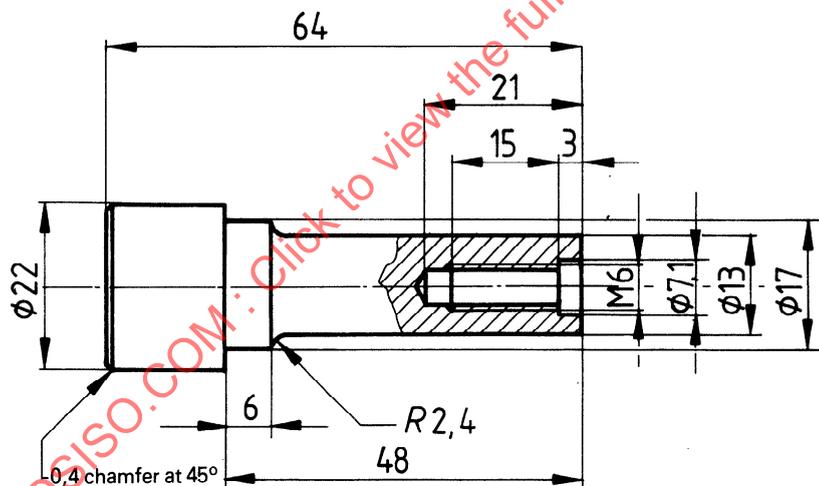
Dimensions in millimetres

Specimen holders

Type 1



Type 2



Test specimen

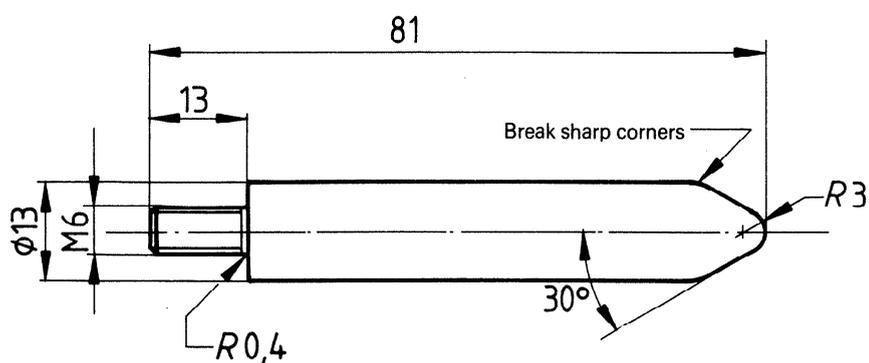


Figure 3 — Test specimen and holders