INTERNATIONAL STANDARD

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МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ

Decontamination of radioactively contaminated surfaces — Method for testing and assessing the ease of decontamination

Décontamination des surfaces contaminées par radioactivité — Méthode d'essai et de détermination de l'aptitude à la décontamination

Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 8690 was prepared by Technical Committee ISO/TC 85, *Nuclear energy*.

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

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0 Introduction

This International Standard aims to define objective conditions for testing the ease of decontamination of surfaces. The test method is designed to obtain data allowing the comparison of the ease of decontamination of different surface materials. The method can be used for comparative tests with any radio-nuclide in aqueous solution. An assessment of the results of a series of comparative tests can be made on the basis of the mean residual pulse rates. In order to allow the general qualifications of a surface material as a single product, this International Standard specifies a test and assessment method on the basis of contamination by solutions containing ^{60}Co and ^{137}Cs. These two radionuclides have been selected because they are the most important sources of contamination in the nuclear industry. The assessment of the result of a single test is made using an assessment table of final residual pulse rates based on round robin experiments.

Direct comparison of the results of this decontamination method with those obtained with other specifications or national standards is unlikely to be useful or relevant because of the different nature of the methods used.

Annexes A, B, C and D form part of this International Standard.

1 Scope and field of application

The specifications laid down in this International Standard apply to the testing of surfaces which may become contaminated by radioactive materials.

Decontaminability data obtained using this test method are not applicable to technical systems where layers of contaminating materials are formed as a result of long-term application of higher temperatures and pressures (for example primary circuits of nuclear reactors).

The purpose of the test is to assess the ease of decontamination of surfaces under laboratory conditions. In practical applications, it may be important to consider other qualities, such as chemical, mechanical and radiation resistance and long-term stability in the selection of the materials to be used. It should be recognized that further decontamination tests under simulated service conditions may be needed.

2 References

ISO 15, *Rolling bearings — Radial bearings — Boundary dimensions — General plan.*

ISO 273, *Fasteners — Clearance holes for bolts and screws.*

ISO 683-13, *Heat-treated steels, alloy steels and free-cutting steels — Part 13 : Wrought stainless steels.*

ISO 2009, *Slotted countersunk head screws (common head style) — Product grade A.*

ISO 2010, *Slotted raised countersunk head screws (common head style) — Product grade A.*

ISO 4762, *Hexagon socket head cap screws — Product grade A.*

3 Definitions

For the purposes of this International Standard, the following definitions apply.

3.1 contamination : The unwanted presence of radio-nuclides on or at a shallow depth below surfaces.

3.2 decontamination : The total or partial removal of contamination, preferably without changing the characteristics of the surface.

3.3 specific pulse rate : The pulse rate caused in the measuring apparatus under given geometrical conditions by 1 ml of a contaminant solution.

It is expressed in pulses per minute per millilitre. Pulse rates are derived from count rates applying dead time and background corrections.

3.4 residual pulse rate : The pulse rate caused in the measuring apparatus under given geometrical conditions by the residual radionuclide on the tested side of the specimen after decontamination.

It is expressed in pulses per minute.

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3.5 mean residual pulse rate : The arithmetic mean of the residual pulse rate values obtained for the five test specimens contaminated by the same radionuclide.

It is expressed in pulses per minute.

3.6 standardized mean residual pulse rate : A corrected value of the mean residual pulse rate. The correction factor is obtained by dividing a reference value of the specific pulse rate by the pulse rate of a contaminant solution used in the test.

It is expressed in pulses per minute.

The purpose of the correction factor is to compensate for variations in specific pulse rates of contaminant solutions used in different test laboratories.

3.7 final residual pulse rate : The arithmetic mean of the standardized mean residual pulse rate obtained for ^{60}Co and ^{137}Cs.

It is expressed in pulses per minute.

4 Principle

Preparation of separate contaminant solutions containing ^{60}Co and ^{137}Cs (carrier concentration : 10^{-5} molar; pH value : 4). Counting of 100 µl samples of these solutions using a large area radiation detector, and calculation of the specific pulse rates of contaminant solutions using the results from the count.

Contamination of specimens of the material under test over a defined area using the contaminant solutions and subsequent decontamination using demineralized water. Determination of the residual pulse rate by measuring the contaminated samples.

Calculation of standardized mean residual pulse rates for each radionuclide. Use of the arithmetic mean of the respective values for ^{60}Co and ^{137}Cs (final residual pulse rate) to assess the ease of decontamination by means of a classification which has been empirically compiled.

5 Apparatus

Ordinary laboratory apparatus, and

5.1 Two glass beakers, of the low-form type, having a capacity of 2 000 ml and complying with requirements laid down in ISO 3819.

5.2 Radiation detector and associated electronics for determining the pulse rate.

The minimum size of the sensitive area of the detector shall be a circle having a 30 mm diameter, but in practice the geometrical requirement specified will normally necessitate the use of a larger sensitive area. Suitable detectors include gas-flow proportional, scintillation and semi-conductor types.

To comply with geometrical requirements, the ratio $(l_{min} - 12,5 \text{ mm}) : h$ shall not be less than 3, where l_{min} is the smallest distance, in millimetres, from the centre point of the contaminated area, as projected onto the detector cross-section, to the edge of the sensitive detector cross-section, and h is the distance, in millimetres, of the contaminated test surface from the detector surface.

If the geometrical requirement specified [i.e. $(l_{min} - 12,5 \text{ mm}) : h$ shall not be less than 3] cannot be met, a detector having a circular sensitive area not less than 30 mm in diameter may be used, provided that

a) for the determination of the specific pulse rate (see 8.1), the 100 µl of contaminant solution is distributed as a series of individual droplets over a circular area 25 mm in diameter. i.e. the area over which the test specimens are contaminated;

b) the net pulse rate of 100 µl of contaminant solution measured under these geometrical conditions is not less than 200 000 pulses per minute (see 8.1).

CAUTION — For the apparatus described in 5.3 to 5.6, separate equipment shall be used for the two radionuclides to prevent cross-contamination.
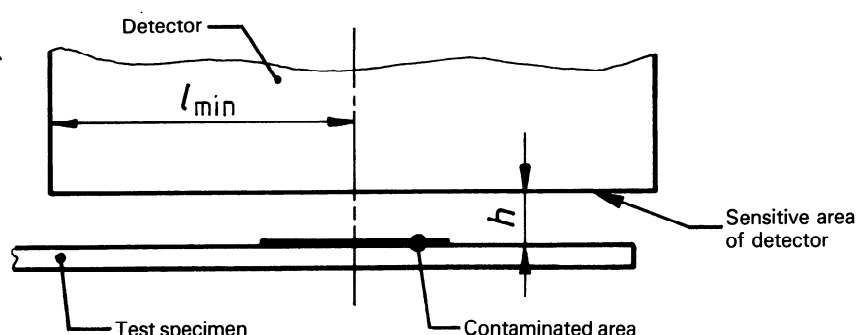

Figure 1 — Geometrical requirements (cross-section)

5.3   Two pipettes with disposable tips, having a capacity of 100 μl.

5.4   Two pipettes with disposable tips, having a capacity of 1 000 μl.

5.5   Two polytetrafluoroethylene (PTFE) beakers.

5.6   Two polytetrafluoroethylene (PTFE) storage bottles.

NOTE — Other fluorinated materials of similar chemical resistance are possible alternatives to polytetrafluoroethylene (PTFE), such as polytetrafluoroethylene/perfluoropropylene (PTFE/PFP), perfluoro alkoxyl alkane (PFA) and poly(vinylidene fluoride) (PVDF).

5.7   Five holders for test specimens, made of poly(methyl methacrylate) (PMMA), serving as positioning aids for the contamination step (see annex A).

Each holder shall contain a flat silicone rubber ring (45 mm × 25 mm × 2 mm) made of unfilled material having a Shore A hardness value of not more than 60.

NOTE — Unfilled, unpigmented, fluorinated silicone rubber has been found particularly suitable for this purpose.

Before being used for the first time, the rubber rings shall be cleaned using the organic solvent mixture used for cleaning the test specimens. The rings should only be re-used after careful decontamination.

NOTE — Ten holders, five for each radionuclide, will reduce the time needed to carry out the test and will help to prevent cross-contamination.

5.8   Cage-stirrer apparatus for six test specimens in accordance with annex B. The apparatus shall be equipped with a motor allowing the stirrer to be rotated at 100 r/min.

6   Contamination and decontamination agents

6.1   Contaminant solutions

6.1.1   Composition of contaminant solutions

The test specimens shall be contaminated by the radionuclides ^{137}Cs and ^{60}Co, contained in separate solutions.

The use of other radionuclides in aqueous solutions which may be more suitable in terms of type and chemical behaviour for the envisaged purpose of the surface material may be adopted, subject to consultation with the testing laboratory.

However, the contaminant solutions shall be chemically stable and shall not corrode the test specimens. The decontaminated samples shall be stable in order to allow the residual contamination to be measured. Special measurement techniques may be required in the case of radionuclides the emissions of which are subject to absorption.

The activity concentration of the contaminant solution shall be such that an evaporated 100 μl sample produces a pulse rate of not less than 200 000 pulses per minute in the measuring instrument, after correction for dead time and background.

NOTE — An activity concentration of 0,2 MBq/ml will usually be sufficient to fulfil the requirement.

The radionuclides shall be used with a carrier concentration of $(1 \pm 0,1) \times 10^{-5}$ mol/l in a solution of nitric acid with a pH-value of $4,0 \pm 0,2$. The pH-value of the contaminants shall be checked monthly. This shall be done using a sample of each contaminant solution which shall then be discarded.

6.1.2   Preparation of the contaminant solutions

6.1.2.1   Apart from Co^{2+} and Cs^+ ions and the corresponding nitrate ions, the radionuclide stock solutions shall not contain constituents which remain in the residue when the solutions are evaporated as described in 6.1.2.4. All reagents used shall be of analytical quality or better.

6.1.2.2   With the help of the data available for the activity concentrations of the ^{137}Cs and ^{60}Co stock solutions, the quantities of these solutions to be used for preparing the desired quantities of contaminant solutions can be calculated. Equations for the preparation of the contaminant solutions are given in annex C.

6.1.2.3   The next step is to calculate from these input quantities the carrier quantities transferred with the radionuclides, and from these in turn calculate the quantities of cobalt(II) nitrate [$Co(NO_3)_2$] or caesium nitrate ($CsNO_3$) solutions respectively, which need to be added to establish a carrier concentration of $(1 \pm 0,1) \times 10^{-5}$ mol/l in the individual solutions. Place these quantities of carrier solutions in polytetrafluoroethylene vessels of sufficient size to allow dilution of the solutions to their final volumes. In order to enhance the displacement of chloride ions which may be present in the radionuclide stock solutions, add 5 ml of nitric acid solution (high purity grade) [$c(HNO_3) = 1$ mol/l] per 90 ml of final volume of contaminant solution. Finally, add the calculated respective input quantities of ^{60}Co or ^{137}Cs stock solutions.

6.1.2.4   Evaporate the mixtures to dryness using infrared lamps until fume evolution stops. Then heat the vessels for another 2 h with the infrared lamps being moved to double the initial distance. After cooling, top the vessels up to the respective final volume by adding nitric acid with a pH-value of 4 [this is produced by diluting 7 μl of nitric acid ($\varrho = 1,4$ g/ml) to 1 l using double distilled water].

Check the specific pulse rates of the thoroughly homogenized solutions in accordance with 8.1.

The pH-value determinations shall be done at least 12 h after the dissolution of the dry residues.

6.1.2.5   In order to avoid wall effects which tend to alter the concentration, the individual solutions shall be kept in polytetrafluoroethylene containers which are tightly closed and which, in turn, are enclosed in glass containers of the smallest possible size as a protection against evaporation.

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6.1.2.6 A solution prepared in accordance with this procedure can be used as long as its pH-value lies within the specified range and the activity concentration has not changed by more than 5 % compared to its initial value (decay corrections being applied).

6.2 Decontamination agent

Demineralized water (maximum conductivity 3 μS/cm) shall be used as decontamination agent for the tests at a temperature of 23 ± 2 °C.

7 Test specimens

7.1 Preparation and preliminary testing

7.1.1 Resistance to cleaning solution

For the purposes of the preliminary testing, a test specimen having at least one flat surface of adequate size (see 7.2) and which may consist of any suitable material — such as non-metallic or metallic materials, coating systems, floor coverings — can be used. The test specimens shall have sufficient resistance to the cleaning solution; this shall be checked using the following procedure :

a) soak a small piece of cotton wool in the cleaning liquid, place it on the surface of a specimen and cover with a Petri dish;

b) after 10 min contact, remove the cotton wool and rinse the specimen using water;

c) then dry the specimen for 1 h at 40 ± 5 °C;

d) examine the specimen visually.

Specimens which show more than a slight change in coloration and lustre are unsuitable for testing.

7.1.2 Test specimens of non-metallic materials

Non-metallic materials to be tested — such as high molar mass polymers, glass, ceramic materials — shall have surfaces of a quality typical of those in actual usage.

If the backing of the test specimen is porous or is an uncoated metal, the back and edges shall be coated in a readily decontaminable manner (e.g. by using an epoxy, polyurethane or chlorinated rubber coating).

For preparing test specimens with coatings, a representative sample shall be taken from the coating material to be investigated, and prepared for testing in accordance with the relevant standard methods.

The coating materials shall be applied to the carriers or the backings in the manner customary in actual usage, and shall be suitably post-treated; any treatment going beyond this, such as additional thermal ageing, is not permissible.

The date of preparation of the test specimens shall be stated.

7.1.3 Test specimens of metallic materials

Test specimens of metal or with metallic surfaces shall be pretreated in a manner typical of actual usage. For coating of the reverse and edges, see 7.1.2.

The surface roughness (average roughness value) shall be stated by the manufacturer and shall be mentioned in the description of the sample material.

7.2 Number and dimensions

For the purposes of the test, 15 nominally identical test specimens shall be prepared and of these at least two groups of five shall be tested in two parallel tests.

NOTE — Remaining test specimens are used for the preliminary tests in accordance with 7.1 and as reference specimens following the test.

The test specimens should measure $50 \, {}^{+10}_{-2} \, mm \times 50 \, {}^{+10}_{-2} \, mm$. The thickness of the test specimen should be between 1 and 10 mm. One corner (having the best rectangular shape) shall be marked as the reference corner by means of a cross of fine scratches on the back of each test specimen.

Other test specimen dimensions and other conditions for preparation are subject to arrangement and shall be stated in the test report.

NOTE — Test specimens of dimensions not greater than 51 mm × 51 mm × 3,5 mm can be stored and transported conveniently using slide storage containers; this helps to avoid contact between the surfaces to be tested.

7.3 Conditioning and cleaning

The test specimens shall be kept in open containers at the testing laboratory in a clean and non-corrosive atmosphere at 20 ± 5 °C for not less than 14 days. This requirement does not apply to test specimens the surfaces of which to be tested consist of ceramic materials or glass.

For cleaning, wipe the test specimens using a soft cellulose tissue. First wipe the surface to be tested using a tissue heavily soaked in the cleaning mixture which should be composed of petrol ether (boiling range from 60 to 80 °C) and isopropanol (minimum content 99 %) in a mixing ratio of 1 : 1 by volume. Repeat the wiping using an "almost dry" tissue. Repeat the wiping operation a third time using a tissue heavily soaked in pure water.

In each case, carry out the wiping operation five times in a single direction over the sample using a different portion of the tissue for each stroke. The tissue shall only be used for one wiping operation (i.e. five strokes).

Finally, rinse the test specimens with pure water using a squeeze bottle to remove all fibres and particles from the surface to be tested. Then dry the test specimens in vertical position for 1 h at 40 ± 5 °C.

Test specimens coated all over with organic surface materials can, in addition, be tested without any prior cleaning when subject to special requirements. In such cases, the preparation

of the test specimens shall be carried out in accordance with the manufacturer's instructions by the centre performing the test in conditions which preclude any soiling of the surfaces to be investigated. If the cleaning stage is omitted, this shall be noted in the test report.

8 Procedure

8.1 Determining the specific pulse rate of each contaminant solution

Apply 100 μl of the contaminant solution on each of three sheets of window glass, measuring 50 mm × 50 mm, at that point which would coincide with the centre of the contamination spot when contaminating in accordance with the procedure laid down in 8.2. For this purpose, the positioning aid (5.7) or a similar device may be used. After the solution droplets have been allowed to dry at a maximum of 45 °C, measure the pulse rates of the three glass sheets using the equipment specified in 5.2.

CAUTION — In this connection it shall be ensured that the measurement geometry (particularly with regard to the distance of the contaminated glass surface from the detector) is the same as that planned for the measurement on the test specimens (see 8.3.2).

The measuring period shall be 1 min for each sheet. Apply corrections for background and dead time losses.

Multiply the arithmetic mean of the three results by a factor of 10 so that the result is expressed in terms of pulses per minute per millilitre.

Carry out the determination separately for both contaminant solutions.

8.2 Contamination

Contamination shall be performed using the assembly shown in figure 2.

Insert a test specimen into the positioning aid (5.7) with its reference corner R as shown in figure 3. Bring edges A and B of the test specimen into close contact with lines a and b of the positioning aid, the contact B-b having priority. Then press the test specimen against upper part U containing the silicone rubber ring by fastening the screws until the test specimen is in contact with upper part U along all its edges.

Contaminate the test specimens by applying 1 ml of the contaminant solution to the middle of the circle visible through the upper part U. Keep the holder in a horizontal position. If, in the case of highly hydrophobic surface materials, the contaminant does not immediately cover the test circle, the holder may be tilted to ensure that the liquid spreads sufficiently.

Place a small Petri dish over the test circle on top of part U to prevent evaporation of the contaminant solution.

After 120 ± 5 min, using a pipette (5.4) suck the contaminant solution off as completely as possible. For this purpose, it is necessary to tilt the holder. Then invert the holder. After

removing the nuts, lift the lower part L and then the test specimen vertically so that distribution of contaminant solution to uncontaminated parts of the test specimen is prevented.

In order to avoid the effects of evaporation, fix the test specimen immediately to the stirring cage and start the decontamination as soon as a set of specimens is completely fixed. The whole procedure of removing the specimens from their holders and fixing them to the cage shall not take more than 12 min.

8.3 Decontamination

8.3.1 Method

For decontamination, fix five of the test specimens to the windows of the cage-stirrer (5.8) using thin rubber bands or coil springs (made of acid-resistant steel).

In order to ensure that the contaminated area of a test specimen is positioned in the middle of a cage window, the reference corner of the test specimen shall cover the lower corner at the right-hand size of the cage window. Bring edges A and B of the test specimen into close contact with the positioning aid lines d and e, the contact B-e having priority (see figure 4).

A sheet of acid-resistant steel shall be used to close the sixth window. Steel sheets of this type shall also be used to support very thin test specimens, which otherwise would not have a sufficiently flat surface.

Immerse the cage in 900 ± 20 ml of demineralized water (6.2) in such a way that the cage touches the bottom of the beaker (5.1). If beakers other than those specified in 5.1 are used, they shall be filled to the minimum level indicated in figure 4. The initial temperature of the water shall be 23 ± 2 °C.

Run the stirrer for 2,5 min at a speed of 100 ± 5 r/min. Replace the beaker with another beaker containing water and run the stirrer for a further 10 s. Remove the test specimens from the cage and then dry them at 40 ± 5 °C in a drying cabinet.
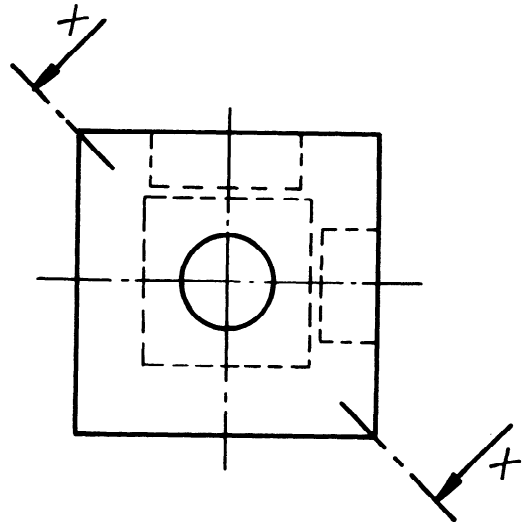
NOTE — If a check on recontamination from the decontaminant solution is deemed to be necessary, the steel sheet closing the sixth window may be replaced by an uncontaminated test specimen.

8.3.2 Determining the residual pulse rate

After drying, measure the pulse rate of the decontaminated test specimens using the equipment specified in 5.2. The measurement geometry, i.e. the distance and relative position of the test specimen and detector, shall be the same for all measurements (see 8.1).

Immediately before the measurement of the residual pulse rates of the test specimens, measure the background pulse rate with an uncontaminated test specimen present.

Each measurement shall be performed once, the background being deducted. Dead time losses shall be taken into account. Counting shall proceed for 5 000 counts above background or 10 min, whichever is the shorter time.

5

X–X (2:1)

Silicone rubber ring (8 × 4 × 2)
to keep screw in position

Flat silicone rubber ring (45 × 25 × 2)
in contact with surface to be tested

M 5 × 70

Upper part U

Test specimen

Lower part L
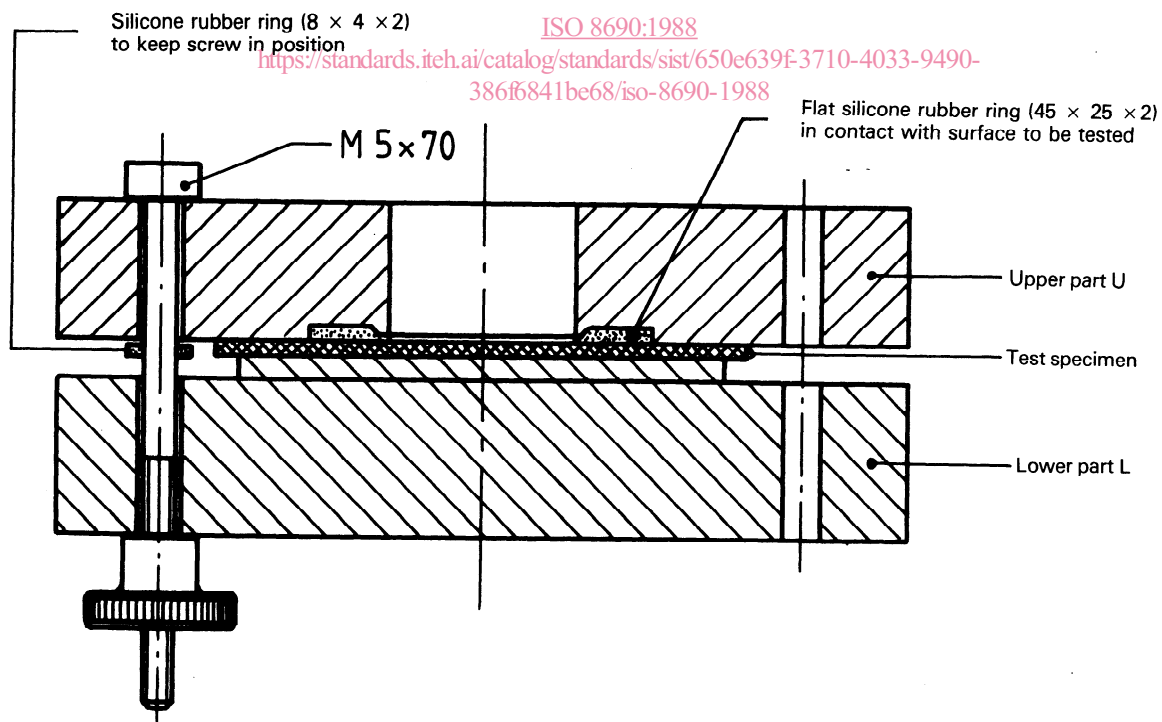
Figure 2 — Holder for contamination of test specimen
(see also figures 5 and 6)

Test specimen
(front)

A

R

B

a
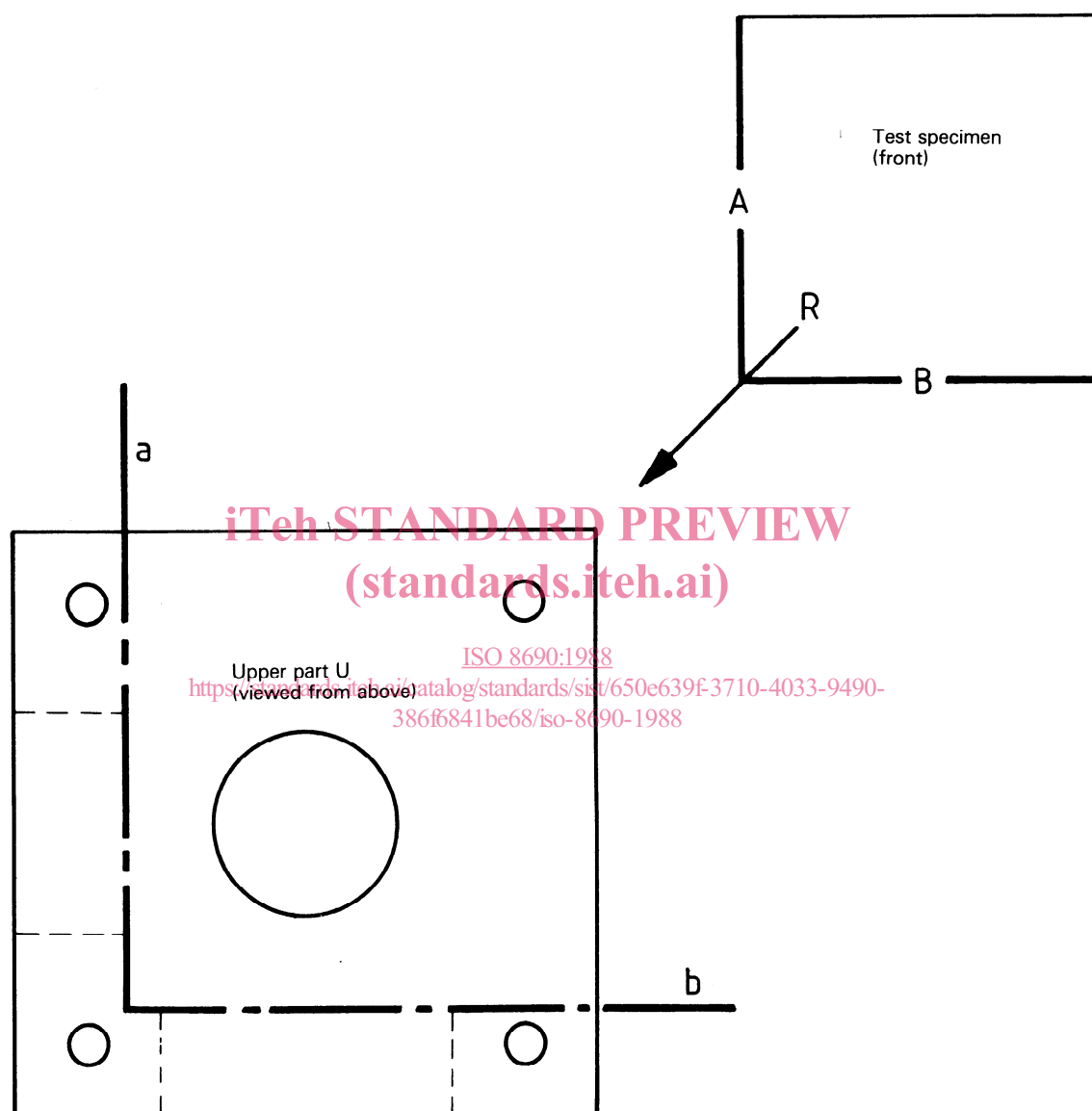
Upper part U
(viewed from above)

b

Figure 3 — Use of holder for contamination of test specimen
(insertion of test specimen)