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Decontamination of radioactively contaminated surfaces — Testing of decontamination agents for textiles

*Décontamination des surfaces contaminées par la radioactivité — Essai
des agents de décontamination pour les textiles*



Reference number
ISO 9271:1992(E)

Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 9271 was prepared by Technical Committee ISO/TC 85, *Nuclear energy*, Sub-Committee SC 2, *Radiation protection*.

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

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Introduction

The purpose of this International Standard is to define objective conditions for testing the effectiveness of decontamination agents for textiles. The test method is designed to obtain data which permits the comparison of the effectiveness of different decontamination agents.

Comparative tests can be carried out with all possible combinations of textile materials and radionuclides in homogeneous solutions. Inorganic or organic solutions can be used and they shall be based on a solvent which evaporates at room temperature. An assessment of the results of a series of comparative tests is made on the basis of the mean residual pulse rates.

In order to permit the general qualification of a decontamination agent as a single product, this International Standard specifies a test and assessment method on the basis of ^{60}Co and ^{137}Cs applied to internationally standardized cotton fabric. These two radionuclides were selected because they are the most important sources of contamination in the nuclear industry. The cotton fabric selected is the only reference material available in this field. The assessment of the result of a single test is made using an assessment table of final residual pulse rates based on inter-laboratory experiments.

Information obtained from the test method will enable the optimization of the choice of decontamination agents for textiles. This should result in lower demands for materials and water in laundry systems, with consequent savings in the cost of radioactive waste processing operations such as filtration, evaporation, solidification and disposal.

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Decontamination of radioactively contaminated surfaces — Testing of decontamination agents for textiles

1 Scope

This International Standard specifies a test method to determine the effectiveness of decontamination agents in removing radionuclides from a textile.

It applies to the testing of detergents, which may be used in aqueous solutions for the purpose of cleaning radioactively contaminated textiles. It does not apply to the testing of the ability of detergents to remove non-radioactive dirt; this is considered to be satisfactory.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 15:1981, *Rolling bearings — Radial bearings — Boundary dimensions — General plan.*

ISO 273:1979, *Fasteners — Clearance holes for bolts and screws.*

ISO 1302:1978, *Technical drawings — Method of indicating surface texture on drawings.*

ISO 2009:1983, *Slotted countersunk head screws (common head style) — Product grade A.*

ISO 2010:1983, *Slotted raised countersunk head screws (common head style) — Product grade A.*

ISO 2174:1990, *Surface active agents — Preparation of water with known calcium hardness.*

ISO 2267:1986, *Surface active agents — Evaluation of certain effects of laundering — Methods of preparation and use of unsoiled cotton control cloth.*

ISO 3819:1985, *Laboratory glassware — Beakers.*

ISO 4762:1989, *Hexagon socket head cap screws — Product grade A.*

ISO 6330:1984, *Textiles — Domestic washing and drying procedures for textile testing.*

3 Definitions

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

3.1 contamination: Pollution of textiles with radioactive materials.

3.2 contaminated textile specimen: Pieces of textile reference materials which are contaminated in a specified manner and which are used to determine the efficiency of decontamination agents.

3.3 decontamination: Total or partial removal of radioactive contamination.

3.4 specific pulse rate: Pulse rate created in the measuring apparatus by 1 ml of a contaminant solution under given geometrical conditions. It is derived from the measurement of 50 µl of contaminant solution evaporated on a textile carrier and is expressed in pulses per minute.

3.5 residual pulse rate: Pulse rate caused in the measuring apparatus under given geometrical conditions by the residual radionuclide on the contamination carrier after decontamination. It is expressed in pulses per minute.

3.6 mean residual pulse rate: 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.

3.7 standardized mean residual pulse rate: Corrected value of the mean residual pulse rate, expressed in pulses per minute. The correction factor is obtained by dividing the aimed for value of the specific pulse rate ($3 \times 10^6 \text{ min}^{-1} \cdot \text{ml}^{-1}$) by the specific pulse rate of the actual contaminant solution used in the test. The purpose of the correction factor is to compensate for variations in specific pulse rates of contaminant solutions used in different test laboratories.

3.8 final residual pulse rate, FRP: Arithmetic mean of the standardized mean residual pulse rates 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 . Counting of 50 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.

Application of the contaminant solutions to specimens made from textile reference materials and subsequent decontamination at 60 °C using a solution of the decontamination agent under test. Determination of the residual pulse rate by measuring the activity remaining on the textile specimens.

Calculation of standardized 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), for assessing the decontamination efficiency by means of a classification which has been empirically compiled.

5 Apparatus

Ordinary laboratory apparatus and the following should be used.

5.1 Two squat glass beakers, of capacity 2 000 ml, as specified in ISO 3819.

5.2 Thermostat, for setting and maintaining the test temperature at 60 °C.

5.3 Drying cabinet.

5.4 Two polytetrafluoroethylene (PTFE) beakers, for preparing the contaminant solution.

5.5 Two polytetrafluoroethylene (PTFE) flasks, for storing the contaminant solutions.

5.6 Cage-stirrer apparatus (see annex A).

5.7 Six clamp specimen holders (see annex B).

5.8 Plastic support block, of dimensions 38 mm x 38 mm x 5 mm.

5.9 Two pipettes with disposable tips, of capacity 50 μl .

5.10 Radiation detector and associated electronics, for determining the pulse rate.

The geometrical requirements for a radiation detector are illustrated in figure 1.

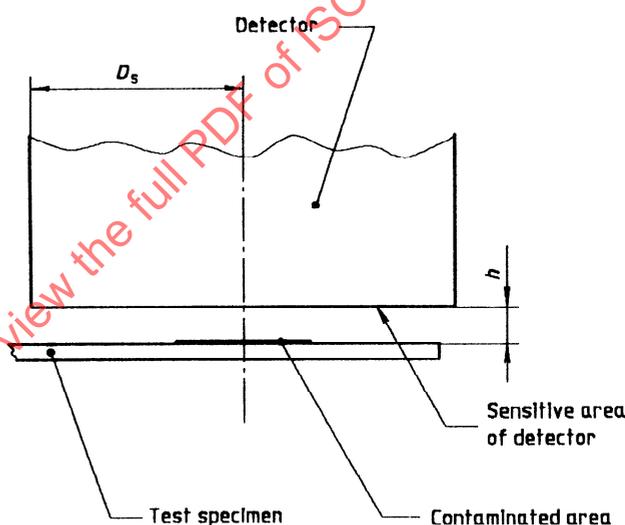


Figure 1 — Geometrical requirements for a radiation detector (cross-section)

The ratio $(D_s - 1,5)/h$ shall not be less than 3 where D_s is the smallest distance, expressed in centimetres, from the centre point of the contaminated zone projected onto the detector cross-section, to the edge of the cross-section of the sensitive area of the detector, and h is the distance, expressed in centimetres, between the contaminated textile specimen surface and the detector surface.

NOTES

1 The minimum size of the sensitive area of the detector should be a circle of diameter 3 cm, but in practice the geometrical requirement for h will normally necessitate the use of a larger sensitive area. Suitable detectors include gas-flow proportional, scintillation and semiconductor types. The same instruments, with the same settings, should be used throughout the test.

2 If the requirement that $(D_s - 1,5)/h$ should not be less than 3 cannot be met, then a detector of circular sensitive

area not less than 3 cm in diameter may be used, provided the net pulse rate of 50 μ l of contaminant solution on a textile specimen measured under these geometrical conditions is not less than 100 000 pulses/min (see 7.1.3).

6 Contaminated textile specimen

6.1 Reference materials

Standard cotton fabric conforming to the specifications in ISO 2267 shall be used as reference material. This fabric shall be pretreated in a washing machine of rotating drum type in the following manner.

Programme: Hot wash three times at 60 °C (coloured wash) without pre-wash; followed by a complete rinsing programme

Wash load: 1 m² standard cotton fabric (no additional load)

Detergent: 80 g ECE¹⁾ reference detergent described in ISO 6330, free of optical brighteners

Water hardness: 2,5 mmol/l complying with ISO 2174

Pressing: Ironing between pieces of fabric which are free of optical brighteners (to be tested using an UV-lamp)

NOTE 3 Information on where the reference detergent and pretreated fabric may be purchased can be obtained from the secretariat of ISO/TC 85 (DIN) or from the ISO Central Secretariat.

If requested, the use of other textile specimen materials is permissible subject to agreement. These other materials include textiles impregnated with defined quantities of dirt and textiles which have been subjected to pretreatment not complying with the specifications of this International Standard. In this case, an assessment in accordance with table 1 is not permissible, and other assessment tables shall be established.

6.2 Number and dimensions of contaminated textile specimens

Twelve square-shaped pieces of pretreated reference material shall be used.

The contaminated textile specimens should measure (50 ± 2) mm \times (50 ± 2) mm.

Table 1 — Assessment of decontamination efficiency

Final residual pulse rate (FRP) pulses/min	Assessment
FRP < 1 000	excellent
1 000 \leq FRP < 3 000	good
3 000 \leq FRP < 10 000	fair
FRP \geq 10 000	poor

NOTE — In inter-laboratory tests using pure water (deionized water of conductivity less than 3 μ S/cm) as the decontamination liquid, a mean FRP-value of approximately 30 000 was obtained.

7 Test agents

7.1 Contamination agents

7.1.1 Composition

Use only separate solutions of ¹³⁷Cs and ⁶⁰Co to contaminate the textile specimens.

An activity concentration of the contamination solutions of 0,2 MBq/ml is sufficient for most types of detection system. However, the concentration shall be such that an evaporated 50 μ l sample of solution produces a pulse rate of not less than 100 000 pulses/min under the geometric measurement conditions used throughout the test. This corresponds to a minimum value of 2×10^6 pulses/(min·ml) as assumed in the calculations in annex C.

The radionuclides shall be used with a carrier concentration of 10^{-5} mol/l in a solution of nitric acid of pH $4,0 \pm 0,2$.

The use of other radionuclides, which may be more suitable for the required purpose in terms of type and chemical behaviour is permissible in the form of an additional test and is subject to consultation with the testing laboratory. In this case, an assessment in accordance with table 1 is not permissible, and other assessment tables shall be established.

If radionuclides other than ¹³⁷Cs and ⁶⁰Co are used, the contaminant solutions shall be chemically stable and shall not attack the contaminated textile specimen. The decontaminated carrier shall be stable, in order to allow measurement of the residual contamination. Special measurement techniques may be required in the case of radionuclides whose emissions are subject to absorption.

1) ECE: European Colourfastness Establishment.

7.1.2 Preparation

Apart from Co^{2+} and Cs^+ ions and the corresponding NO_3^- ions, the radionuclide stock solutions shall not contain constituents which remain in the residue when the solutions are evaporated.

Calculate the quantities of the solutions to be used for preparing the desired quantities of contaminant solutions, using the data available for the activity concentrations of the ^{137}Cs and ^{60}Co stock solutions.

Calculate, from these input quantities, the carrier quantities transferred with the radionuclides, and from these calculate the quantities of standardized $\text{Co}(\text{NO}_3)_2$ or CsNO_3 solutions which must respectively be added to establish a carrier concentration of 10^{-5} mol/l in the final product. These quantities of carrier solutions shall be placed in PTFE vessels which are sufficiently large to allow dilution of the solutions to their final volumes. To enhance the displacement of chloride ions which may be present in the radionuclide stock solutions, 5 ml of 1 mol/l nitric acid (high purity grade) shall be added per 90 ml of final volume of contaminant solution. Finally, the calculated respective input quantities of ^{137}Cs or ^{60}Co stock solutions shall be added. (Equations for the preparation of the contaminant solutions are given in annex C.)

The mixtures shall be gently evaporated to dryness in the PTFE vessels using infrared lamps until fume evolution stops. The vessels shall then be heated for another 2 h with the infrared lamps moved to double the initial distance. After cooling, the vessels shall be filled to their respective final volumes by adding HNO_3 of pH 4. This is obtained by diluting 7 μl of HNO_3 (ρ 1,4 g/cm³) to 1 litre using double-distilled water.

The specific pulse rates of the thoroughly homogenized solutions shall be checked by evaporating a 50 μl aliquot of each solution on a textile specimen and measuring them, using the activity-measuring apparatus. (See also 7.1.1, second paragraph, and 7.1.3.)

The pH-values of the solutions should not be measured until at least 12 h after dissolution of the dry residues.

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

Solutions made in this way can be used as long as their pH-values lie within the specified range and the activity concentration has not changed by more than 5 % compared to its initial value (with decay corrections taken into account).

7.1.3 Determination of the specific pulse rate of each contaminant solution

Apply 50 μl of the contaminant solution on each of three textile specimens prepared in accordance with 8.3.1, at the point which would coincide with the centre of the contamination spot when contaminating in accordance with 8.3.2. After the solution has been allowed to dry at a maximum temperature of 45 °C, remove the textile specimens from their frames and measure their count rates with a radiation detector (5.10). It shall be ensured that the measurement geometry (particularly with regard to the distance of the contaminated textile specimen from the detector) is the same as that planned for the measurement on the decontaminated textile specimen.

The measuring period shall be such that at least 10 000 counts are collected for each textile specimen. Corrections for background and dead time losses shall be applied to the count rate to yield the pulse rate.

The arithmetic mean of the three results shall be multiplied by 20, to express the result in terms of pulses per minute per millilitre.

The determination shall be done separately for both contaminant solutions.

7.2 Decontamination agents

For the purpose of the test, the decontamination agents shall be used in solutions with a concentration of 7,5 g/l. If the producer recommends a lower concentration for optimum performance, a test at this lower concentration may be carried out, either

- as an additional test; or
- instead of the test under standard concentration conditions.

In case b), an assessment in accordance with table 1 may be carried out but it shall be stated in the test report that it is

"not fully comparable with standard test results, due to the deviating test concentration of the decontamination agent being, for example, 3 g/l instead of 7,5 g/l".

If the producer recommends a higher concentration for optimum performance, an additional test with the recommended concentration may be carried out.

Deionized water with a maximum conductivity of 3 $\mu\text{S}/\text{cm}$ shall be used as the solvent. The solutions shall be prepared not more than 1 h before each application.

8 Procedure for testing decontamination efficiency

8.1 General

Each test shall comprise contamination, drying, measurement and decontamination of the textile specimens in direct succession.

8.2 Preparation of the textile specimens

The textile specimens shall be cut to size parallel to the warp or the weft of the fabric, taking care not to stretch the material.

Care should be taken to protect the material against soiling, for example by wearing polyethylene gloves or by using tweezers.

8.3 Contamination

8.3.1 Preparation

For each radionuclide solution, six textile specimens shall be clamped in their respective steel specimen holders. In order to prevent sagging of the textile fabric, the plastic support block (5.8) shall be inserted into the lower part of the holder (see figure 2). Care should be taken to avoid creasing of the fabric when screwing the holder tight.

The corners of the textile specimens shall be cut off slightly, in order to prevent their coming into contact with the screws (see figure 2).

Once the textile specimens are clamped into the holder, the plastic support block shall be removed and the holder shall be inverted for contamination.

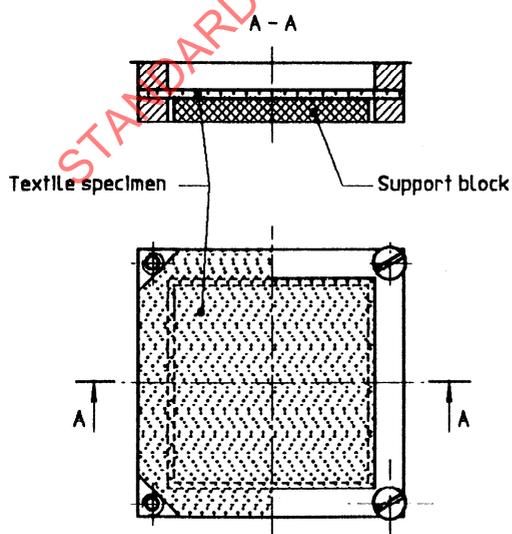


Figure 2 — Use of specimen holder

8.3.2 Procedure

Apply, drop by drop, using a pipette, 50 μ l of the contamination agents (as specified in clause 5) to the centre of the textile specimens (see figure 3 showing a diagonal cut of the holder).

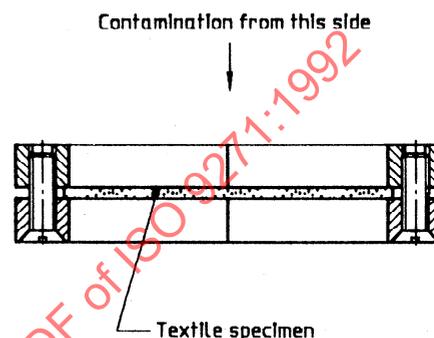


Figure 3 — Position of specimen holder during contamination

The textile specimens, still in their respective holders, shall then be dried in a drying cabinet at $40\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$ for 2 h. In this way, five textile specimens shall be contaminated with each contaminant solution.

8.4 Decontamination

8.4.1 Preparation

For the purpose of decontamination, six textile specimens, five of which are contaminated and one of which is not, are necessary.

The specimen holders containing the textile specimens shall be fastened to the windows of the cage-stirrer apparatus. Stainless steel coil springs or disposable rubber bands may be used to fasten the holders onto the cage-stirrer. Care should be taken to ensure that the side of the fabric on which the contaminant solution was applied faces the inside of the cage.

8.4.2 Procedure

Decontamination shall be carried out at a speed of 100 r/min in 900 ml of the decontamination agent (see 7.2) maintained at a temperature of $60\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$. If the producer recommends a lower temperature for optimum performance, a test at this lower temperature may be carried out, either

- a) as an additional test; or
- b) instead of the test under standard temperature conditions.

In case b), an assessment in accordance with table 1 may be carried out, but it shall be stated in the test report that it is

“not fully comparable with standard test results, due to the deviating test temperature (for example 30 °C instead of 60 °C)”.

Care shall be taken that the cage-stirrer is positioned vertically and centrally, and that it touches the bottom of the glass beaker. The stirrer shall first be run for 10 min in one direction, and then for an additional 10 min in the other direction.

The specimens shall then be rinsed at the same rotational frequency in 900 ml of deionized water for 5 min at room temperature, with the stirrer running in one direction. The deionized water shall be replaced and the rinsing repeated with the stirrer running in the opposite direction. The contaminated textile specimens shall be removed from the cage-stirrer and dried in their respective holders at 100 °C ± 5 °C for 30 min.

8.4.3 Residual pulse rate

After cooling to room temperature, the contamination carriers shall be removed from their holders and their residual pulse rates determined. At least 5 000 counts should be measured, after allowance is made for background. The measurement geometry shall be the same as when determining the specific pulse rates (see 7.1.3). Care should be taken to ensure that the side of the contaminated textile specimen on which the contamination was applied faces the detector.

9 Calculation of results

After decontamination, the arithmetic mean of the residual pulse rates of the five originally contaminated test specimens of each group shall be calculated separately for ⁶⁰Co and ¹³⁷Cs. The results shall be expressed in pulses per minute and shall be used to calculate the standardized mean residual pulse rate, which is the product of the mean residual pulse rate and

$$\frac{3 \times 10^6}{\text{Specific pulse rate}}$$

NOTE 4 The figure of 3 × 10⁶ pulses/(min·ml) is the aimed for value of the specific pulse rate of the contaminant solutions. The assessment table is based on this reference value for the specific pulse rate.

The final residual pulse rate is calculated as the arithmetic mean of the standardized mean residual pulse rates for ⁶⁰Co and ¹³⁷Cs.

The pulse rate measured on the uncontaminated textile specimen may be used to assess recontamination from the solution.

10 Assessment of decontamination efficiency

The efficiency of the decontamination agent under test shall be assessed on the basis of the final residual pulse rate value in accordance with table A.1.

11 Test report

The test report shall include the following information:

- a) standardized mean residual pulse rates for ⁶⁰Co and ¹³⁷Cs;
- b) final residual pulse rate;
- c) assessment of decontamination efficiency;
- d) parameters of any additional tests.

12 Consideration of other properties

When assessing the suitability of a decontamination agent, in addition to the decontamination efficiency, other properties have to be taken into account depending on the technical conditions prevailing in the waste treatment plant and the solidification plant.

The main properties to be considered are:

- a) the foaming ability;
- b) the thermal stability;
- c) the solubility in and miscibility with water;
- d) the pH-value;
- e) the acid/basic strength;
- f) the presence of steam-volatile components;
- g) the halogen content;
- h) the content of complexing agents;
- i) the content of filling substances increasing the waste volume;
- j) the reaction with heavy metal and calcium ions;

- k) the reducing power;
- l) the degreasing ability;
- m) the flashpoint;
- n) the general handling risks of the material, such as
 - the need for masks, gloves, etc., during handling of concentrates,
 - the consequence of gross leakage of detergent.

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Annex A
(normative)

Cage-stirrer apparatus for decontamination

A general view of the cage-stirrer apparatus is shown in figure A.1. Figures A.2 to A.9 illustrate the

components of the cage-stirrer. Table A.1 lists the components indicated by numbers in figure A.1.

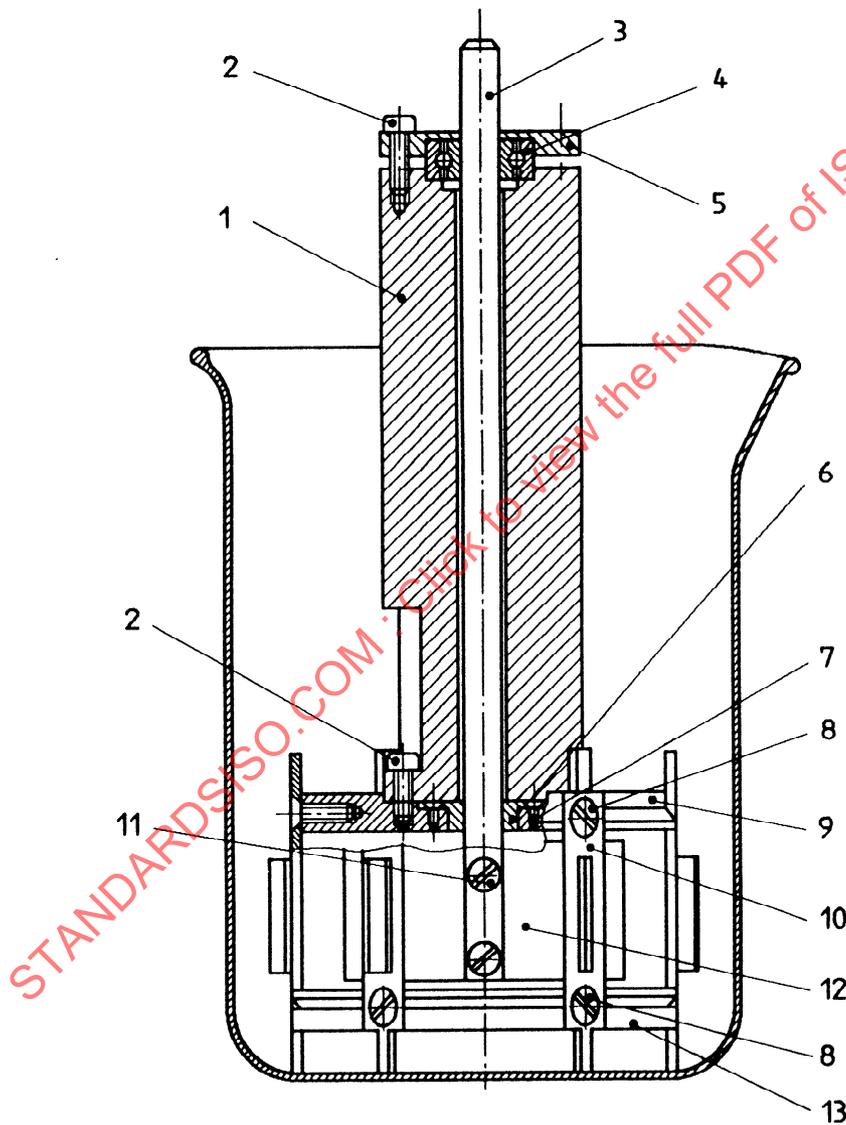


Figure A.1 — Cage-stirrer apparatus

Table A.1 — Components of the cage-stirrer apparatus

Reference number on figure A.1	Number of items	Description	Material	Figure
1	1	Stirrer support column	Acrylic glass	A.2
2	6	Hexagon socket head cap screws, M3 x 12, in accordance with ISO 4762	Acid-resistant stainless steel	—
3	1	Stirrer axle	Acid-resistant stainless steel	A.3
4	1	Radial deep-groove ball bearing with washers in accordance with ISO 15 (Dimensions: $d = 10$, $D = 26$, $B = 8$)		—
5	1	Mounting support for ball bearing	Acrylic glass	A.4
6	1	Bearing of stirrer shaft Bores: $\varnothing 3,4$ Clearance in accordance with ISO 273 Countersunk in accordance with ISO 2009	Polytetrafluoroethylene (PTFE)	A.5
7	2	Slotted countersunk head screws, M3 x 5, in accordance with ISO 2009	Acid-resistant stainless steel	—
8	12	Slotted countersunk head screws, M4 x 10, in accordance with ISO 2009	Acid-resistant stainless steel	—
9	1	Upper hexagonal disc	Acrylic glass	A.6
10	6	Connecting part Bores: $\varnothing 4,5$ Clearance in accordance with ISO 273 Countersunk in accordance with ISO 2009	Acid-resistant stainless steel	A.7
11	2	Slotted raised countersunk head screws, M3 x 10, in accordance with ISO 2010	Acid-resistant stainless steel	—
12	1	Stirrer blade	Acid-resistant stainless steel	A.8
13	1	Lower hexagonal disc	Acrylic glass	A.9

NOTE — Non-metric equivalents for the screws may be used.

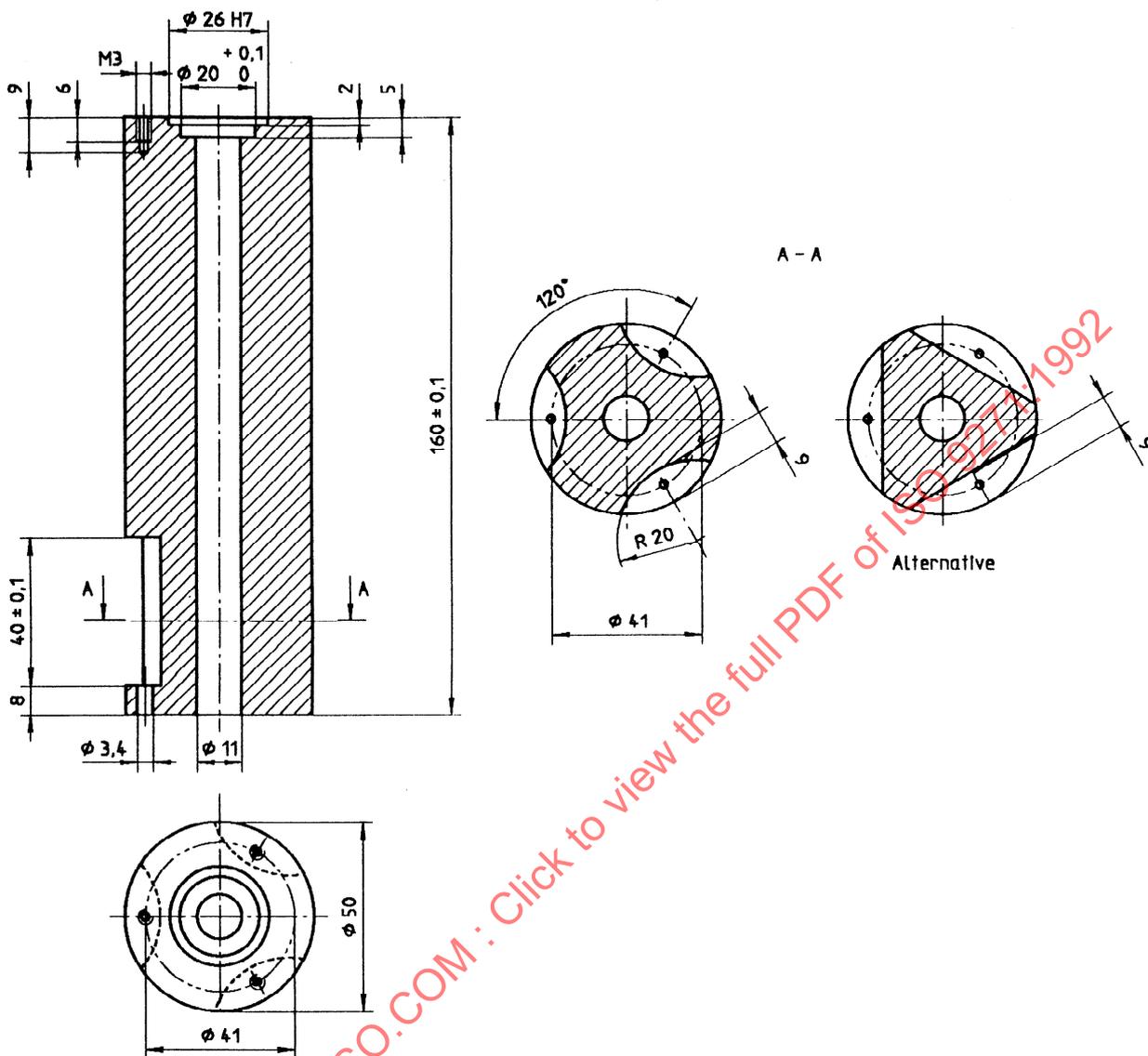


Figure A.2 — Stirrer support column

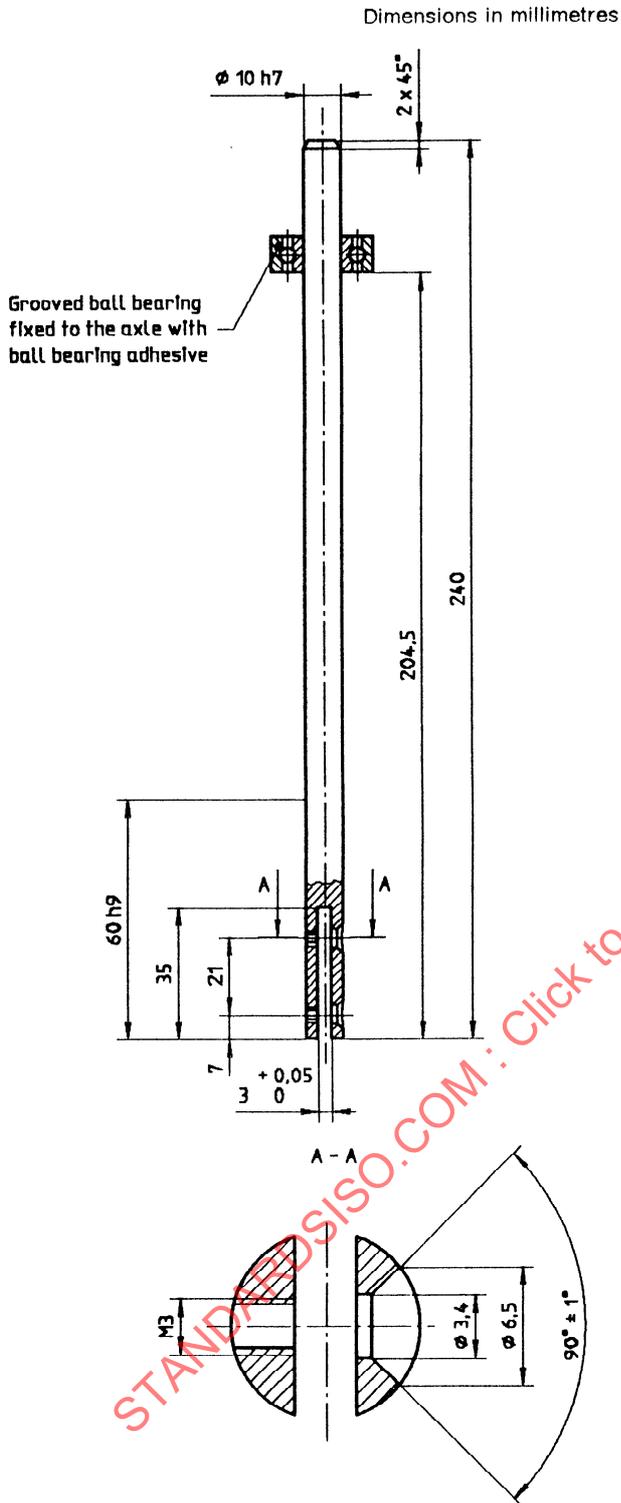


Figure A.3 — Stirrer axle

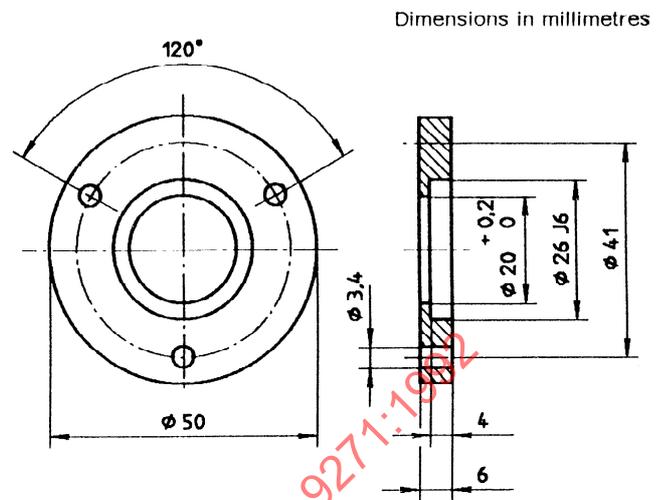


Figure A.4 — Mounding support for ball bearing

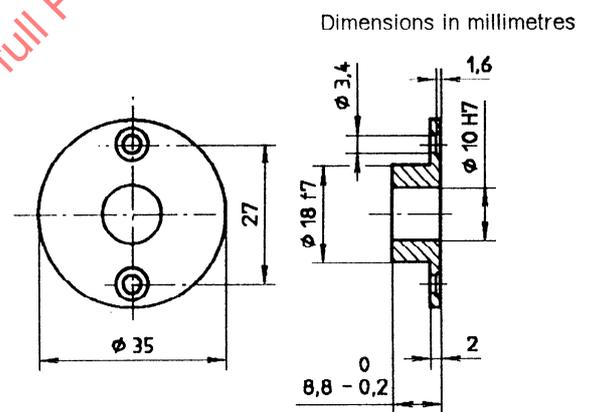


Figure A.5 — Bearing of stirrer shaft

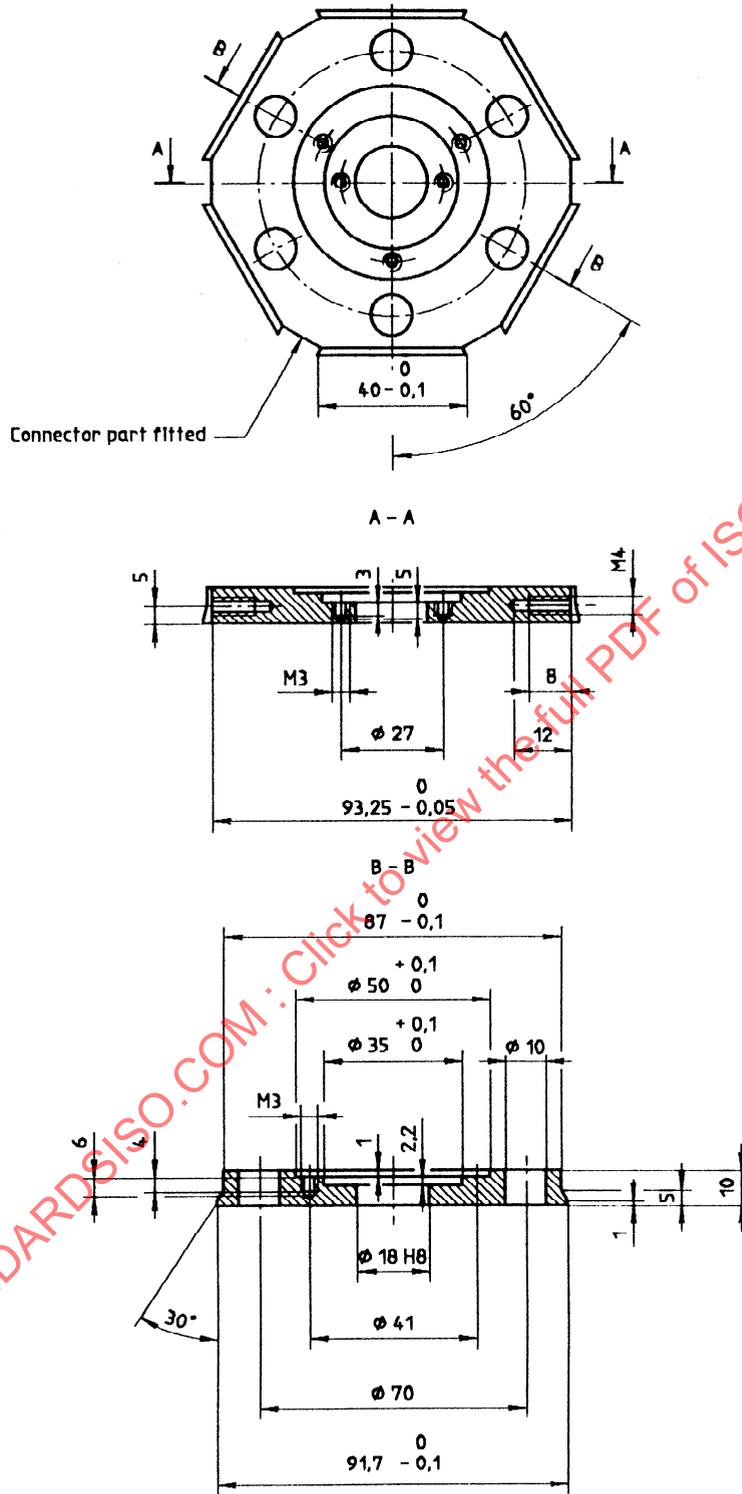


Figure A.6 — Upper hexagonal disc

Dimensions in millimetres

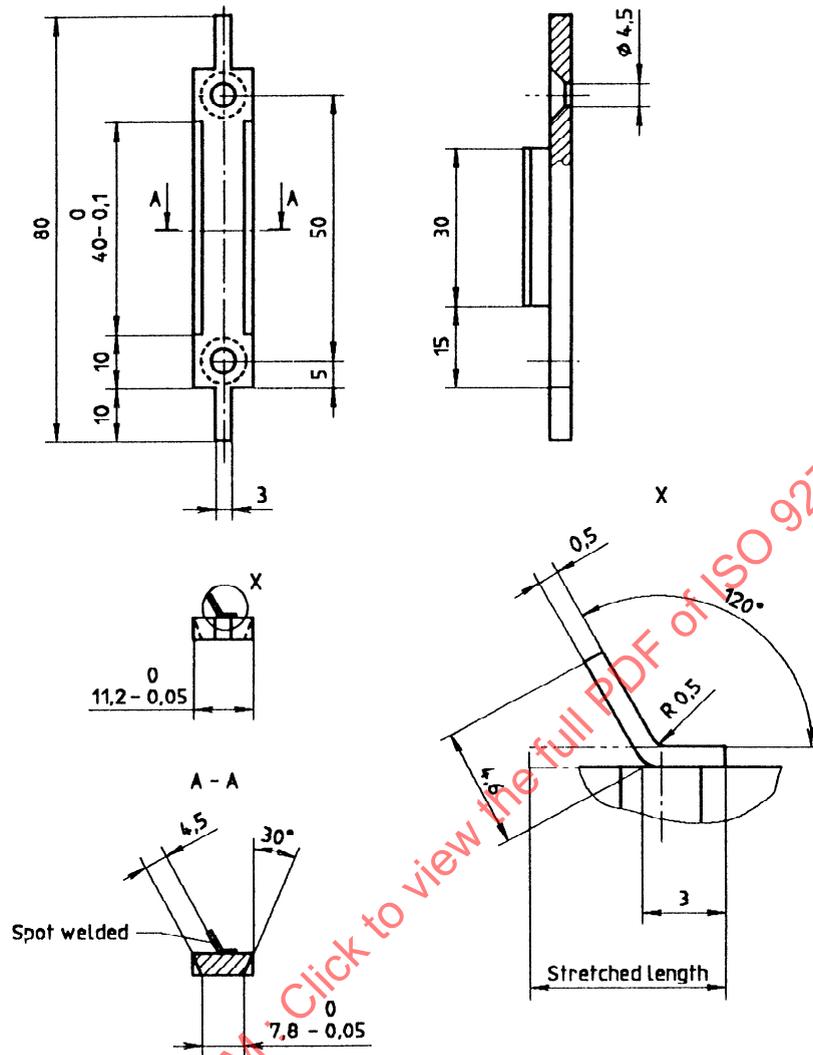
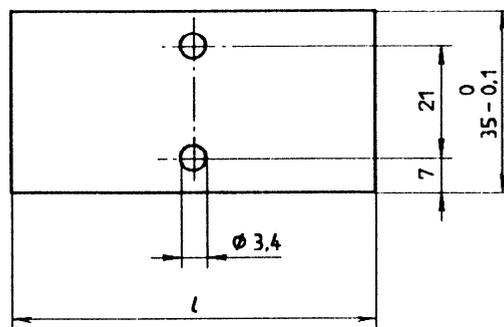


Figure A.7 — Connecting part

Dimensions in millimetres



$l - 1$	50	60	70
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Thickness = 3

Figure A.8 — Stirrer blade

Dimensions in millimetres

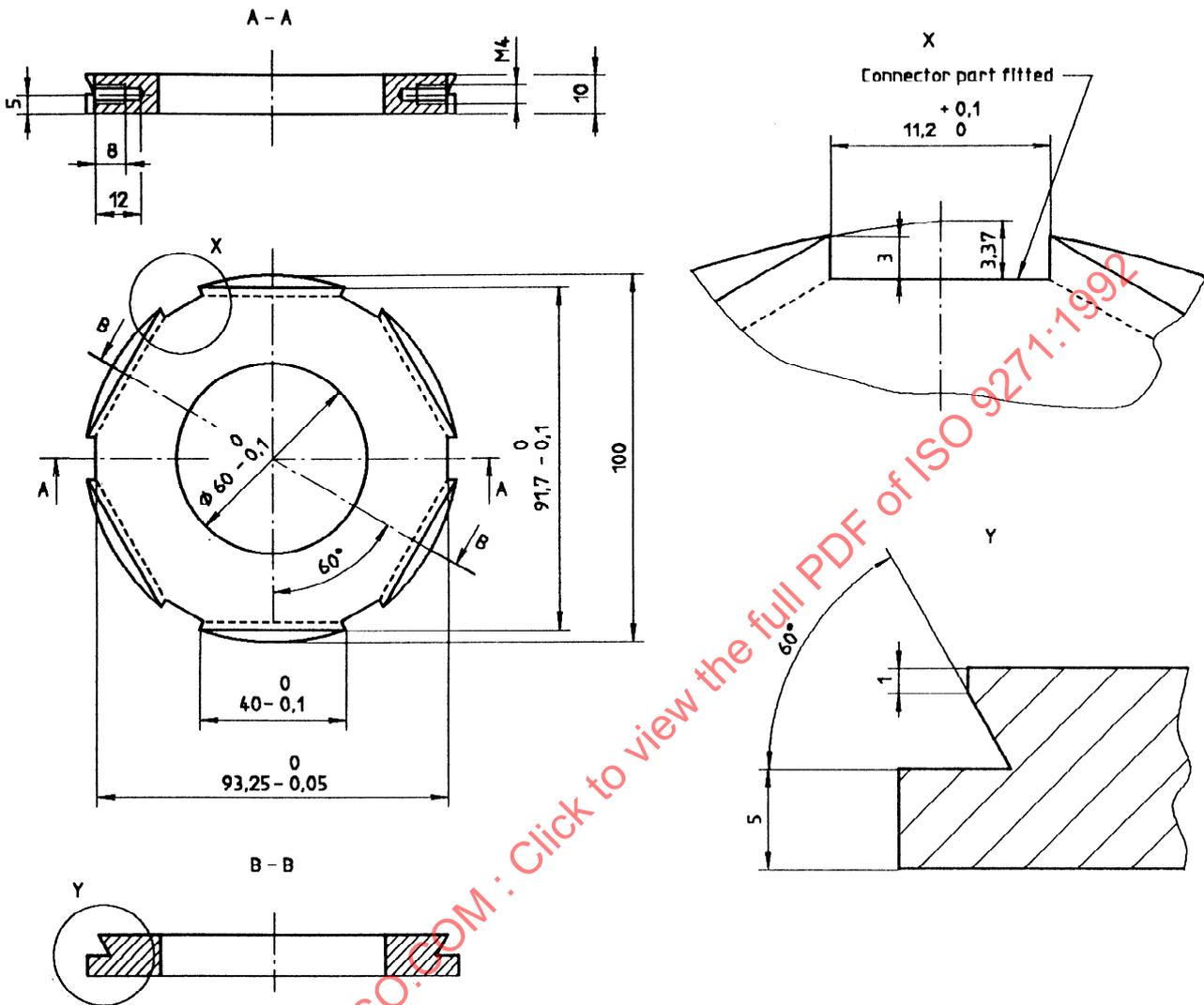


Figure A.9 — Lower hexagonal disc

Annex B (normative)

Clamp specimen holder

The clamp specimen holder is shown in figures B.1 and B.2.

The top and bottom parts are screwed together with four M3 × 8 countersunk screws complying with ISO 2009.

The roughness dimensions comply with ISO 1302.

Material: acid-resistant steel.

Dimensions in millimetres

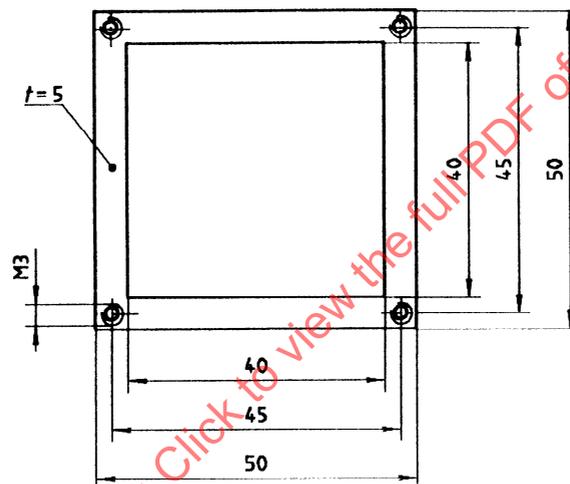


Figure B.1 — Clamp specimen holder — Bottom part

Dimensions in millimetres

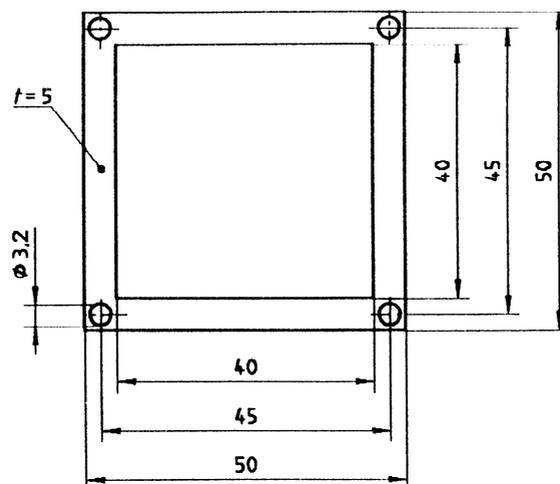


Figure B.2 — Clamp specimen holder — Top part