
International Standard



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Plastics — Determination of environmental stress cracking (ESC) — Constant tensile stress method

Plastiques — Détermination de la fissuration sous contrainte dans un environnement donné (ESC) — Méthode sous contrainte de traction constante

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

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.

International Standard ISO 6252 was developed by Technical Committee ISO/TC 61, *Plastics*, and was circulated to the member bodies in May 1979.

It has been approved by the member bodies of the following countries :

<u>ISO 6252:1981</u>		
Belgium	Hungary	Romania
Brazil	India	South Africa, Rep. of
Canada	Israel	Spain
China	Italy	Sweden
Czechoslovakia	Japan	Switzerland
Egypt, Arab Rep. of	Korea, Rep. of	United Kingdom
Finland	Libyan Arab Jamahiriya	USA
France	Netherlands	USSR
Germany, F.R.	Poland	

No member body expressed disapproval of the document.

Plastics — Determination of environmental stress cracking (ESC) — Constant tensile stress method

0 Introduction

Environmental stress cracking is exhibited by many materials, including plastics. When a plastic material is stressed or strained in air below its yield point, stress cracking can occur after a period of time which may be very long. These stresses may be internal or external, or a combination of both. Exposure to a chemical environment simultaneously with the same stress or strain may result in a dramatic shortening of the time to failure. This phenomenon is referred to as environmental stress cracking (ESC).

The cracks produced may penetrate completely through the thickness of the material, separating it into two or more pieces, or they may be arrested on reaching regions of lower stress or different material morphology.

The determination of ESC is complex because it is influenced by many parameters, including :

- test specimen dimensions;
- test specimen state (orientation, structure, internal stresses);
- stress and strain;
- temperature of test;
- duration of test;
- chemical environment;
- test method;
- failure criterion.

By keeping all but one parameter constant, the influence of the variable parameter on ESC can be assessed. The main objective of ESC measurements is to determine the effect of chemicals media (environment) on plastics (test specimens and articles). The measurements may also be used to evaluate the influence of the moulding conditions upon the quality of an article when the failure mode corresponds to that obtained in actual service.

It may not be possible, however, to establish any direct correlation between the results of short-duration ESC measurements on test specimens and the actual service behaviour of articles, because the behaviour of the latter may be more complex than that of test specimens.

1 Scope and field of application

This International Standard specifies methods for the determination of environmental stress cracking (ESC) of plastics when they are subjected to a constant tensile force in the presence of chemical agents.

NOTE — A method for the determination of environmental stress cracking by means of a constant strain test is specified in ISO 4600.

2 References

- ISO 150, *Raw, refined and boiled linseed oil for paints and varnishes — Specifications and methods of test.*
- ISO 291, *Plastics — Standard atmospheres for conditioning and testing.*
- ISO 527, *Plastics — Determination of tensile properties.*¹⁾
- ISO 2557, *Plastics — Amorphous thermoplastic moulding materials — Preparation of test specimens with a defined level of shrinkage*
 - Part 1: *Test specimens in the form of parallelepipedic bars (Injection moulding and compression moulding).*
 - Part 2: *Test specimens in the form of rectangular plates (Injection moulding).*
- ISO 2818, *Plastics — Preparation of test specimens by machining.*
- ISO 4600, *Plastics — Determination of environmental stress cracking (ESC) — Ball or pin impression method.*

1) At present at the stage of draft. (Revision of ISO/R 527.)

3 Principle

A test specimen is subjected to a constant tensile force, corresponding to a stress lower than that at yield, while immersed in a specified liquid at the temperature selected for testing. The time and/or stress at which the specimen breaks is recorded.

The environmental stress cracking of the test specimens is determined by one of the following methods (A or B) depending upon the time to rupture :

- method A : Determination of the tensile stress leading to rupture at 100 h. This stress is obtained by interpolation of the graph of time to rupture versus applied tensile stress;
- method B : Determination of the time to rupture under a specified tensile stress. This method is used when the time to rupture exceeds 100 h.

4 Apparatus

4.1 Testing device, allowing test specimens to be submitted simultaneously to a tensile stress and to the chemical environment.

If the chemical is a liquid at the test temperature, the test specimen shall be completely immersed in it. If it is highly viscous at the test temperature, the specimens may be covered with a coating of the agent at least 2 mm thick (see clause 5).

The parts of the device that come into contact with the chemical shall be made of an inert material, for example stainless steel.

The constant tensile stress may be applied with weights (figures 1 and 2 are schematic diagrams of suitable apparatus). The force shall be accurate to $\pm 1\%$. Care shall be taken to ensure that there is no loss of stress in the system, for example by friction.

If the testing device has several test stations, means shall be provided to prevent the vibration occurring through failure at one station from being transmitted to the whole system.

4.2 A temperature-controlled bath, allowing the test system to be maintained at $23 \pm 1^\circ\text{C}$ or at a higher test temperature up to 105°C to within $\pm 0,5^\circ\text{C}$ (see clause 6).

4.3 An automatic timer, to measure the time to rupture of each specimen to the nearest 1 min.

4.4 Equipment for the preparation of test specimens by machining (see ISO 2818) or die cutting.

5 Chemical environments

The chemical environment used for the test shall be that specified in the relevant International Standard for the material tested. If nothing is specified, use either the agent to be in contact with the material in the expected application or a reference product agreed upon between the interested parties.

NOTE — Examples of reference products are :

- a) 95 % (V/V) ethanol — pharmaceutical quality;
- b) a 1 % (m/m) solution of nonylphenoxy poly(ethyleneoxy)ethanol¹⁾ in distilled water;
- c) refined linseed oil (see ISO 150).

6 Test temperature

The preferred test temperatures are $23 \pm 1^\circ\text{C}$ and $55 \pm 0,5^\circ\text{C}$. If required, other temperatures may be used, preferably selected from the following :

$40 \pm 0,5^\circ\text{C}$, $70 \pm 0,5^\circ\text{C}$, $85 \pm 0,5^\circ\text{C}$, $100 \pm 0,5^\circ\text{C}$.

7 Test stresses

7.1 Maximum permissible stress

The stress applied to the test specimen during the test shall be less than the tensile stress at yield of the material at the temperature of the test.

NOTE — As a general guide, the stress that produces an elongation of 2 % after 1 h can be taken as the maximum permissible stress. This stress can be determined by preliminary tests using several different stresses. After 1 h of application of the stress, remove the load and quickly measure the residual elongation of the calibrated part of the test specimen. See also the annex.

7.2 Method A

Determine the tensile stress required to cause rupture after 100 h by applying a series of stresses, the maximum stress being as defined in 7.1.

7.3 Method B

Determine the time to rupture under a single stress, the applied stress being the maximum permissible stress defined in 7.1. Other stresses below the maximum may be used if agreed between the interested parties.

1) This detergent is available commercially. Details may be obtained from the secretariat of ISO/TC 61 (ANSI) or from the ISO Central Secretariat.

8 Test specimens

8.1 Shape and dimensions

Use the type B specimen specified in ISO 527, with all dimensions scaled down by a ratio of 1:2 as shown in figure 4.

The preferred thickness is $2 \pm 0,2$ mm, but when the test specimens are prepared from products, the thickness may be that of the product.

8.2 Number of test specimens

A minimum of five specimens shall be tested at each tensile stress.

If the material is thought to be anisotropic, two sets of specimens shall be used, one set cut at right angles to the other.

8.3 Preparation

The specimens shall be prepared according to the appropriate International Standard. If nothing is stated, specimens shall be machined from sheet or from products by the methods specified in ISO 2818.

If sheets are prepared from moulding materials, they shall be moulded in accordance with the relevant material specification or as agreed between the interested parties. Specimens shall not be cut with a die unless machining is impossible, for example with soft materials.

NOTE — Environmental stress cracking of a specimen is influenced not only by the material, but also by the method of preparation. Materials should only be compared using specimens prepared in a similar manner and in the same state. Attention is drawn to ISO 2557 for the determination of level of shrinkage.

9 Conditioning

Unless otherwise specified in the relevant International Standard or by prior agreement, test specimens shall be conditioned prior to testing in the atmosphere specified in ISO 291 (23 ± 2 °C and relative humidity of 50 ± 5 %) for 24 h.

10 Procedure

10.1 Measure, to the nearest 0,01 mm, the thickness and the width of the central parallel portion of each specimen and calculate the force, P , in newtons, to be applied, from the formula

$$G \times A$$

where

G is the stress, in megapascals, selected for the test (as described in 7.1);

A is the area of the cross section of the central parallel portion of the specimen, in square millimetres.

NOTE — To determine the cross-sectional area it is recommended

- to measure thickness at each end of the central parallel portion and take the minimum value;
- to measure the width of each face at each end of the narrow parallel portion and to take the mean value.

10.2 Heat the temperature-controlled bath (4.2) to the selected test temperature.

10.3 Install the specimens in the jaws of the fixture (4.1) and immerse them in the test liquid or coat them with the chemical.

10.4 After 15 min, apply the load P to each specimen, without shock, in such a way that the loading time is preferably between 3 and 5 s and, in any case less than 10 s. Start the timer (4.3) as soon as the load is applied ($t = 0$). Record the time to rupture for each specimen and the type of break (brittle or ductile).

NOTE — If a liquid chemical environment is used, it should be renewed with liquid from the same batch for each test specimen (apparatus with one station) or each group of test specimens (apparatus with several stations).

10.5 Where method A is used, perform the test with a series of tensile stresses up to and including the maximum permissible stress as defined in 7.1.

NOTE — The 100 h stress is obtained by interpolation (see 11.1). It is necessary that the loads used give arithmetic means of times to rupture between 30 and 300 h.

10.6 Where method B is used, perform the test using the maximum permissible stress as defined in 7.1 or a specified or an agreed stress (see 7.3). If no break occurs after 1 000 h, terminate the test and record this fact in the test report.

10.7 If required, carry out a parallel series of tests, as described in 10.5 or 10.6, in air or another reference environment.

11 Calculation and expression of results

11.1 Method A

Calculate the arithmetic mean of times to rupture for each stress used and their standard deviations.

Plot the logarithm of each mean time to rupture (as abscissae) versus tensile stress (as ordinates) and determine by interpolation the stress, in megapascals, corresponding to a 100 h time to rupture.

11.2 Method B

Calculate the arithmetic mean of the times to rupture, in hours, obtained from the five specimens and the standard deviation.

NOTE — For some purposes the geometric, rather than the arithmetic, mean may be found useful because the logarithms of the times to rupture often show a better gaussian distribution than the times to rupture.

12 Test Report

The test report shall include the following particulars:

- a) a reference to this International Standard;
- b) complete identification of the material tested;
- c) the chemical environments used;
- d) the test temperature;
- e) the number of specimens tested (if applicable, in each direction of anisotropy) and their width and thickness;
- f) the procedure used for preparation of the specimens and, whenever possible, the elapsed time between their preparation and the beginning of the testing;
- g) the state of the specimens;
- h) conditioning duration and atmosphere;
- j) the stresses applied;
- k) the times to rupture, individual and mean values and standard deviations for each stress applied. If no rupture occurred after 1 000 h under the maximum permissible stress as defined in 7.1, report that fact;
- m) for method A, the stress corresponding to 100 h time to rupture;
- n) the type of break, i.e. brittle or ductile;
- p) any operational details not specified in this International Standard, and any circumstance liable to have influenced the results;
- q) results from parallel series of tests in air or another reference environment, if used.

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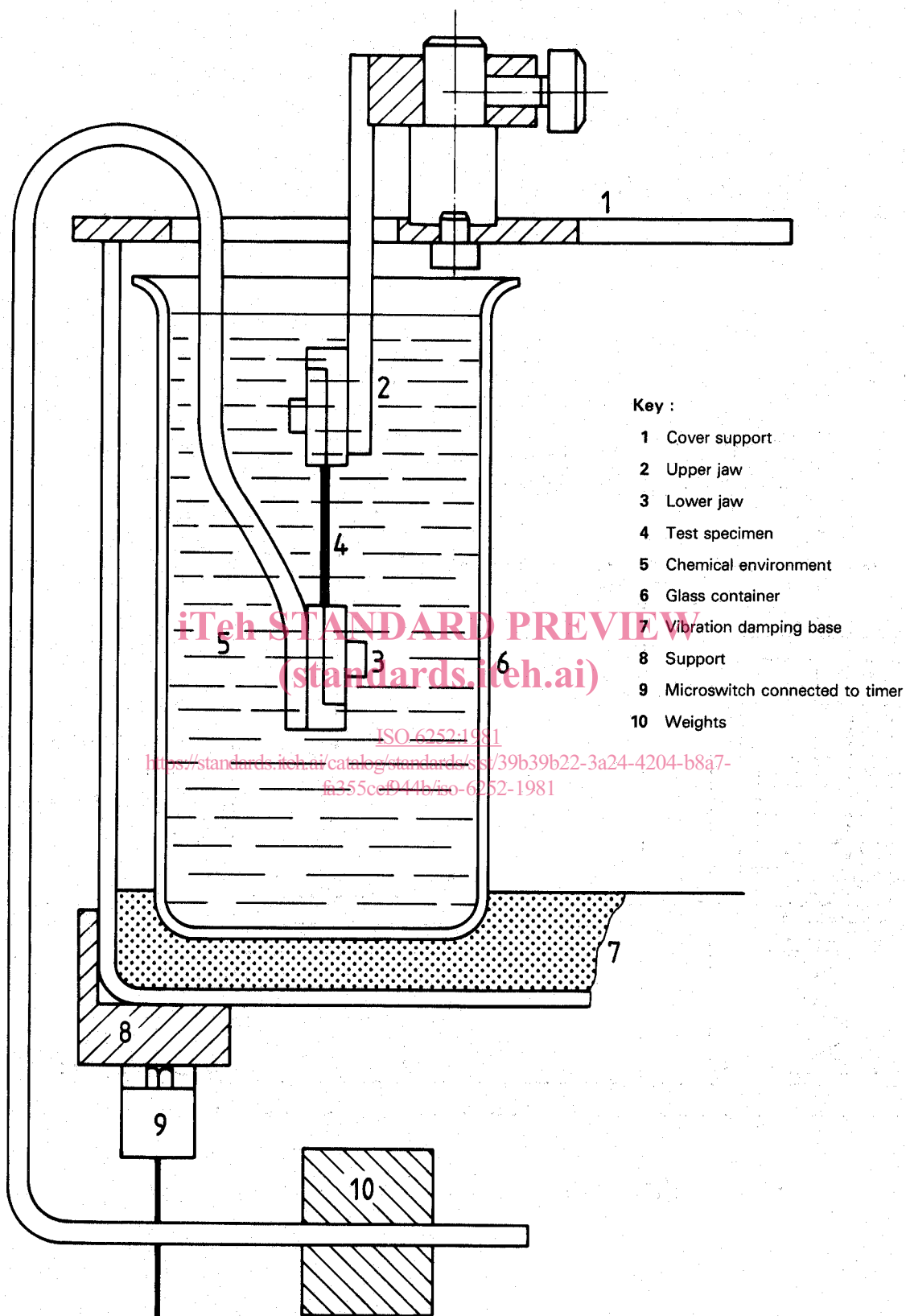


Figure 1 — One type of apparatus for measuring fracture under constant stress

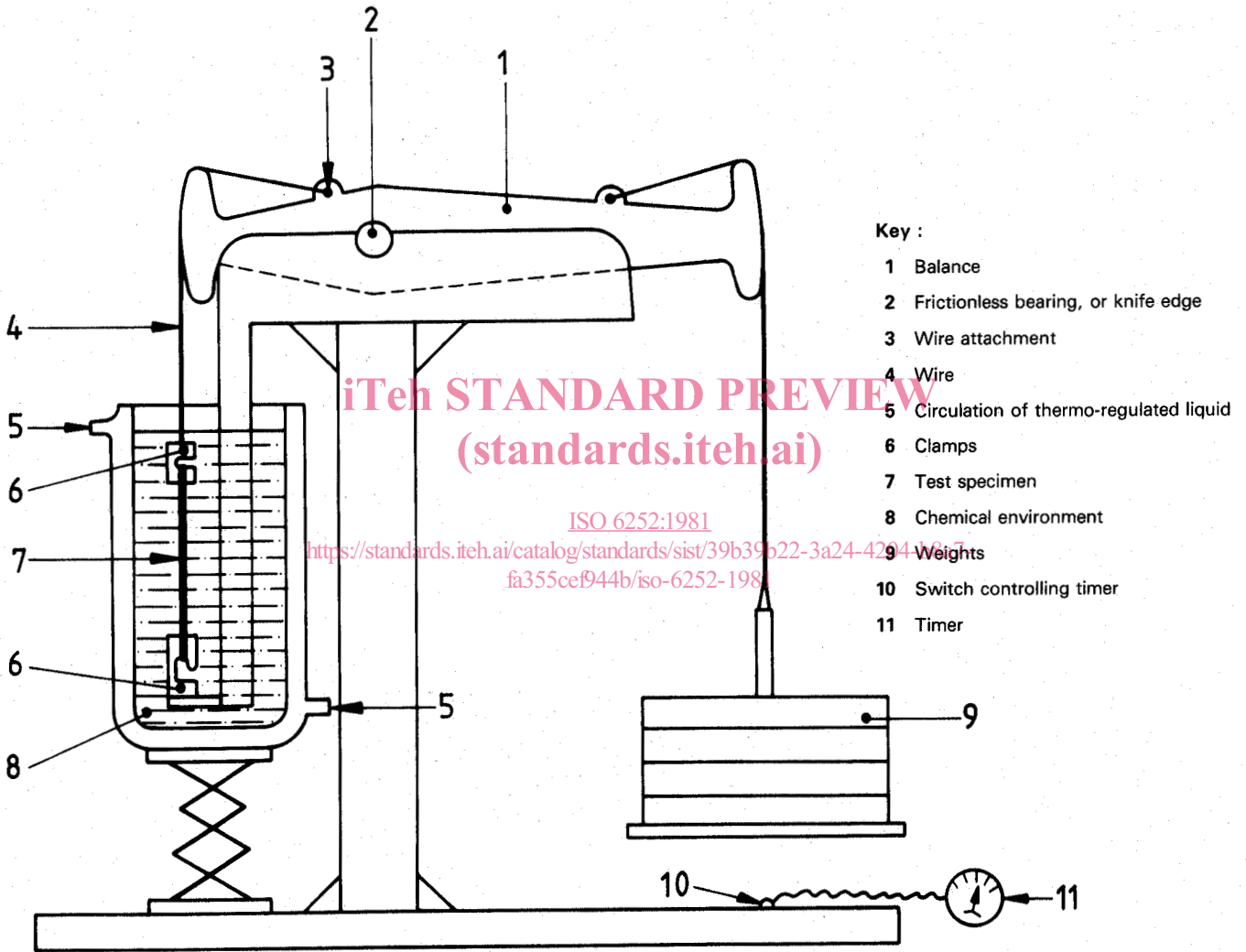


Figure 2 — Balance type of apparatus with 2 : 1 amplification of load

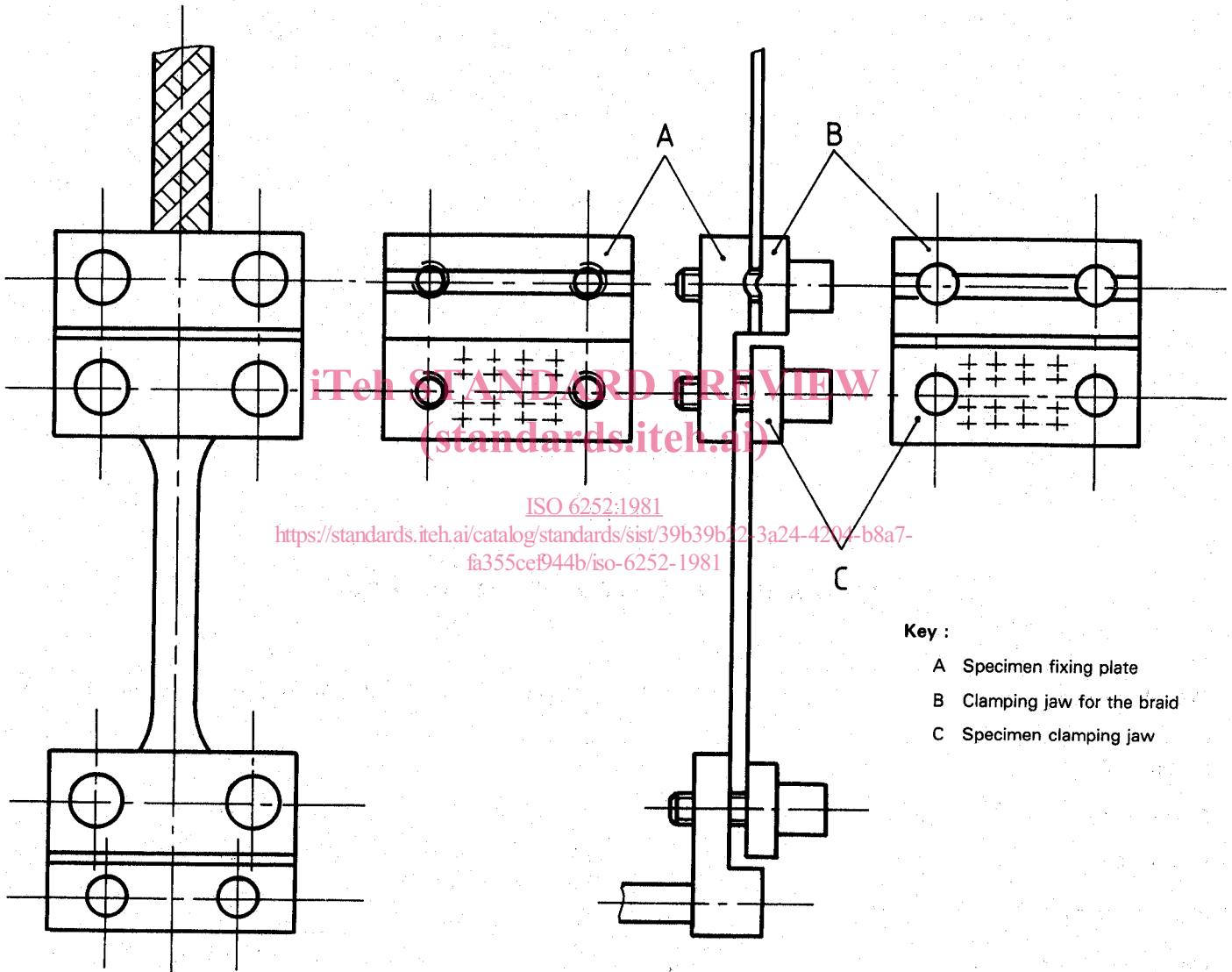


Figure 3 — Example of suitable jaw arrangement