

INTERNATIONAL STANDARD

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6077**

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Photography — Photographic films and papers — Wedge test for brittleness

*Photographie — Films et papiers photographiques — Détermination de la
méthode dite "du coin"*



Reference number
ISO 6077:1993(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 6077 was prepared by Technical Committee ISO/TC 42, *Photography*.

This second edition cancels and replaces the first edition (ISO 6077:1980), of which it constitutes a technical revision.

Annex A of this International Standard is for information only.

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International Organization for Standardization

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Introduction

Brittleness is a property of many materials under specific atmospheric conditions, which causes them to break or crack when deformed by bending. In the case of photographic films and papers it is an undesirable property since satisfactory performance in equipment or in handling requires sufficient flexibility (or lack of brittleness) to ensure good tolerances to bending stresses under the atmospheric conditions likely to be encountered in practice.

Photographic films and papers are laminates of two or more different materials, usually a plastic or paper support and firmly bonded gelatin layers that contain image forming and other substances. These laminate structures must have sufficient flexibility to withstand the stresses imposed by bending and flexing during use. However, under low relative humidity and low temperature conditions, the gelatin layers and/or support tend to become brittle. This can cause the formation of cracks and/or breaks when the laminate is stressed beyond its diminished capacity to withstand bending and flexing.

Different types of brittle failure occur when film and paper are subjected to stress in different ways. For example, failure can consist of very fine cracks in the emulsion (without a break in the support) which are objectionable when the photograph is viewed. Under different conditions or with different materials, failure can consist of cracks in the support or a complete break of all components of the laminate.

Brittleness is not an absolute physical property. The apparent brittleness of photographic material is very dependent upon the manner in which it is mechanically treated as well as the ambient conditions of temperature and relative humidity. As a result, no one test for brittleness is capable of ranking a variety of types of photographic material as they would behave under widely different practical conditions. The wedge test for brittleness specified in this International Standard subjects the photographic material to a high rate of strain in a simple folding action. It has been an accepted method of rating brittleness for many years and has correlated with product behaviour under many practical applications. It is a widely accepted method in the photographic industry. Other methods used have consisted of flex tests in which the specimen is subjected to a repeated folding action until it breaks or the bending over mandrels of different diameters. There are a considerable number of flexibility tests available and standardization is not possible at this time.

Brittleness is affected adversely by both reduced temperature and reduced relative humidity. Brittleness at low relative humidity is encountered more frequently than brittleness at low temperature in most applications. Moreover, a marked change in brittleness can occur with only a very small change in relative humidity below a level of about 25 %. Consequently brittleness tests are carried out only in an atmosphere which is very accurately controlled with respect to both temperature and relative humidity.

Brittleness is also very dependent on the sample thickness, increasing with increasing thickness of either support or emulsion. For this reason, the thickness of the layers must be considered when comparing the behaviour of different materials. The thermal and moisture exposure history of the material between manufacture and testing can also affect the brittleness, even though the sample is reconditioned to a standard temperature and humidity. Gelatin is generally more brittle than the support, so that photographic products having a gelatin layer on only one side are usually more brittle when bent with the gelatin side out (that is, gelatin under tension). Brittleness can vary with the bend axis depending upon the orientation of the support. There is generally no directional effect in the brittleness of the emulsion.

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Photography — Photographic films and papers — Wedge test for brittleness

1 Scope

This International Standard specifies a method for determining and expressing quantitatively the brittleness of photographic film and papers. It does not, however, specify a universal brittleness test for all types of stress encountered for which special tests can be required to correctly rate the material for brittleness.

The method is applicable to films with or without a gelatin backing and to fibre-based or resin-coated (RC) photographic papers. It may also be applied to either raw or processed materials although the brittleness level can be quite different after processing from that before processing.

2 Normative reference

The following standard contains provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the edition indicated was 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 edition of the standard indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 483:1988, *Plastics — Small enclosures for conditioning and testing using aqueous solutions to maintain relative humidity at constant value.*

3 Principle

Measurement of the opening of a wedge at which a specimen loop fails when subjected to high strains.

4 Apparatus

4.1 Wedge tester for brittleness

The wedge tester for brittleness consists of two non-parallel metal plates or jaws, which form a wedge or V as shown in figures 1 and 2. A clamp is attached to one jaw at the narrow end of the wedge to hold one end of the specimen. A scale is provided which gives the wedge separation between the two jaws at any point. [1]

The dimensions for the wedge tester are standardized for both a narrow and a wide tester. The narrow wedge (see figure 1) has a wedge angle of $9,0^\circ$ and a maximum wedge opening of 25 mm. It is generally satisfactory for most films. For more brittle films and papers, a wide wedge (see figure 2) is recommended. It has a wedge angle of $28,0^\circ$ and a maximum wedge opening of 74 mm.

4.2 Test chamber

An air-conditioned box or walk-in room shall be used for both conditioning and testing. The temperature shall be controlled within $\pm 1^\circ\text{C}$ and the relative humidity to within $\pm 1\%$ ¹⁾ of the specified values. The linear air velocity shall be at least 15 cm/s. If a walk-in conditioned room is used, the air velocity shall be adequate to maintain the conditions specified. The

1) To control the relative humidity to this accuracy usually requires a dewpoint method.

number of personnel permitted in the room during testing shall be limited and precautions shall be taken to prevent the operator's breath reaching the material.

5 Sampling

5.1 Preparation of specimens

Specimens shall be cut in an atmosphere of 20 °C to 23 °C and from 40 % to 55 % relative humidity²⁾. The cutter shall be of a precision type and shall be kept sharp so that the specimens' edges are smooth and free of nicks. Rubber gloves shall be worn by the operator in handling the specimens, both in their preparation and testing.

5.2 Selection of specimens

A set of ten specimens shall be prepared for each test. If there is sufficient material, one set of specimens should be cut in the machine direction and a second set in the transverse (cross) direction. If the film has a discrete backing layer, a separate set of specimens shall be cut in at least one of the two principal directions for testing with the backing convex.

5.3 Size of specimens

The standard specimen size shall be 350 mm long by 15 mm to 16 mm wide. If the sample size available does not permit a 350 mm specimen length, the length can be effectively increased by taping a film or paper leader to the specimen and placing the specimen end in the clamp. Although not recommended, 16 mm perforated film may be tested for comparative purposes only, by cutting off the perforated edges and testing a specimen 9 mm wide. Similarly, 35 mm film may be tested, but the results are not necessarily comparable to the standard 15 mm to 16 mm width.

6 Conditioning and test conditions

6.1 Conditioning of specimens

Specimens shall be conditioned in the test chamber until practicable moisture equilibrium has been

reached. This may be determined by weighing specimens at regular intervals and determining the time at which further conditioning does not appreciably change the mass. In many instances, this time will be in the vicinity of 4 h for photographic film, 1 d for fibre-based papers and 7 d for RC papers. However, actual times will vary due to access of the conditioning air and the type and thickness of the material. The specimens shall be held in racks permitting free circulation of air.

6.2 Test conditions

The standard relative humidity for testing shall be $(15 \pm 1) \%$. However, other relative humidities such as 10 %, 20 % and 35 % or approximations thereof, utilizing stable saturated solutions in a closed box can prove useful.³⁾ The standard temperature for testing shall be $23 \text{ °C} \pm 1 \text{ °C}$. However, other temperatures may be used where the effect of temperature is to be investigated.

The specimen shall not be removed from the conditioning atmosphere for testing except at a temperature of 0 °C. For testing film at 0 °C or below, the specimens shall be conditioned at the desired relative humidity at 23 °C, sealed in small, taped cans, cooled long enough to reach the test temperature and then removed, one at a time, for testing⁴⁾.

7 Procedure

Fasten one end of the specimen in the clamp at the narrow end of the wedge. Loop the material, emulsion out, within the wedge, with a sufficient length at the free end, extending through the narrow opening of the wedge, to allow it to be pulled. Make a mark on the clamped end of the specimen at the point where the narrow end of the wedge starts (at the 1,5 mm scale marking). Pull the specimen by the free end completely through the narrow opening of the wedge using a smooth, rapid motion completed within 1 s.⁵⁾ Remove the specimen from the clamp and lay it on the scale with the mark aligned at the end corresponding to 1,5 mm. Read the wedge separation at the point of failure of the specimen directly from the scale to the nearest 0,5 mm.

2) If the specimens are cut at relative humidities lower than 40 %, it can be difficult to obtain smooth edges. Handling under these conditions can also cause emulsion cracking which will affect the subsequent brittleness results. Exposure to relative humidities greater than 55 % can permanently alter the subsequent brittleness behaviour of some films and photographic papers.

3) See ISO 483.

4) Direct control of relative humidity at temperatures of 0 °C or below is impracticable, but once photographic material is conditioned, the rate of gain or loss of moisture is much lower at low temperatures.

5) The speed of testing in the range from 1 s to 6 s had relatively little effect on the brittleness values obtained with several films investigated.

If emulsion cracks occur⁶⁾, record the wedge separation for both the first emulsion crack and the complete material break. The former can also be found from the broken specimen, it being necessary to examine both pieces of the material.

Films which have emulsion on both sides or with a discrete backing layer shall be tested in duplicate, testing each side separately.

Photographic film and paper shall have satisfactory adhesion between the emulsion and support for the wedge separation to reflect the brittleness. If the adhesion is not satisfactory, the emulsion will strip from the support during the test and the wedge separation at stripping will be very dependent upon the emulsion-base adhesion and will not reflect brittleness of the complete laminate.

8 Test report

8.1 Test data

Specimens which pass through the narrowest part of the wedge (1,5 mm) without failure shall be arbitrarily assigned a value of 1 mm to enable an arithmetic mean to be calculated with the values for those specimens which fail by breaking.

The following values shall be reported for each manner in which the material was tested, for example lengthwise, widthwise, emulsion convex and backing convex:

- a) the mean wedge separation for the first emulsion crack for those specimens which exhibit cracks;
- b) the percentage of specimens showing emulsion cracks;

- c) the mean wedge separation for complete breaks (including values of 1 mm for those specimens which did not break);
- d) the percentage of specimens showing complete breaks;
- e) the mean wedge separation for first failure, whether a crack or break (including values of 1 mm for those specimens which did not fail);
- f) the temperature and relative humidity at which the test was carried out;
- g) the average thickness of the specimen (and width, if not 15 mm to 16 mm);
- h) whether the narrow or wide wedge was used.

8.2 Significance

It is important to note whether the sample fails by emulsion cracks or by a complete break, since this can give information about the reasons for brittleness. Emulsion cracks are influenced by the brittleness of the emulsion layer and the emulsion base adhesion. Breaks are affected by these two factors together with the brittleness of the base. First-failure measurements give a single value for the brittleness of the laminate, but no information as to the cause of the brittleness.

A difference in the mean wedge test brittleness between two materials of less than 1 mm is not believed to be significant.

A qualitative judgement of the acceptable wedge test brittleness of a material is very dependent upon its nature and use. For example, a motion-picture film requires a low level of brittleness as it is transported and flexed over small diameter rollers and sprockets when in use. However, a thick-base photographic paper can tolerate much higher wedge values as it is generally not flexed during use.

6) Emulsion cracks can best be seen under strong illumination by a direct (parallel, collimated) light beam. Frequently, examination of emulsion cracks can most easily be detected from the back of the specimen, provided it does not have a backing layer. Holding the material under a slight tension or viewing with transmitted light is sometimes helpful.

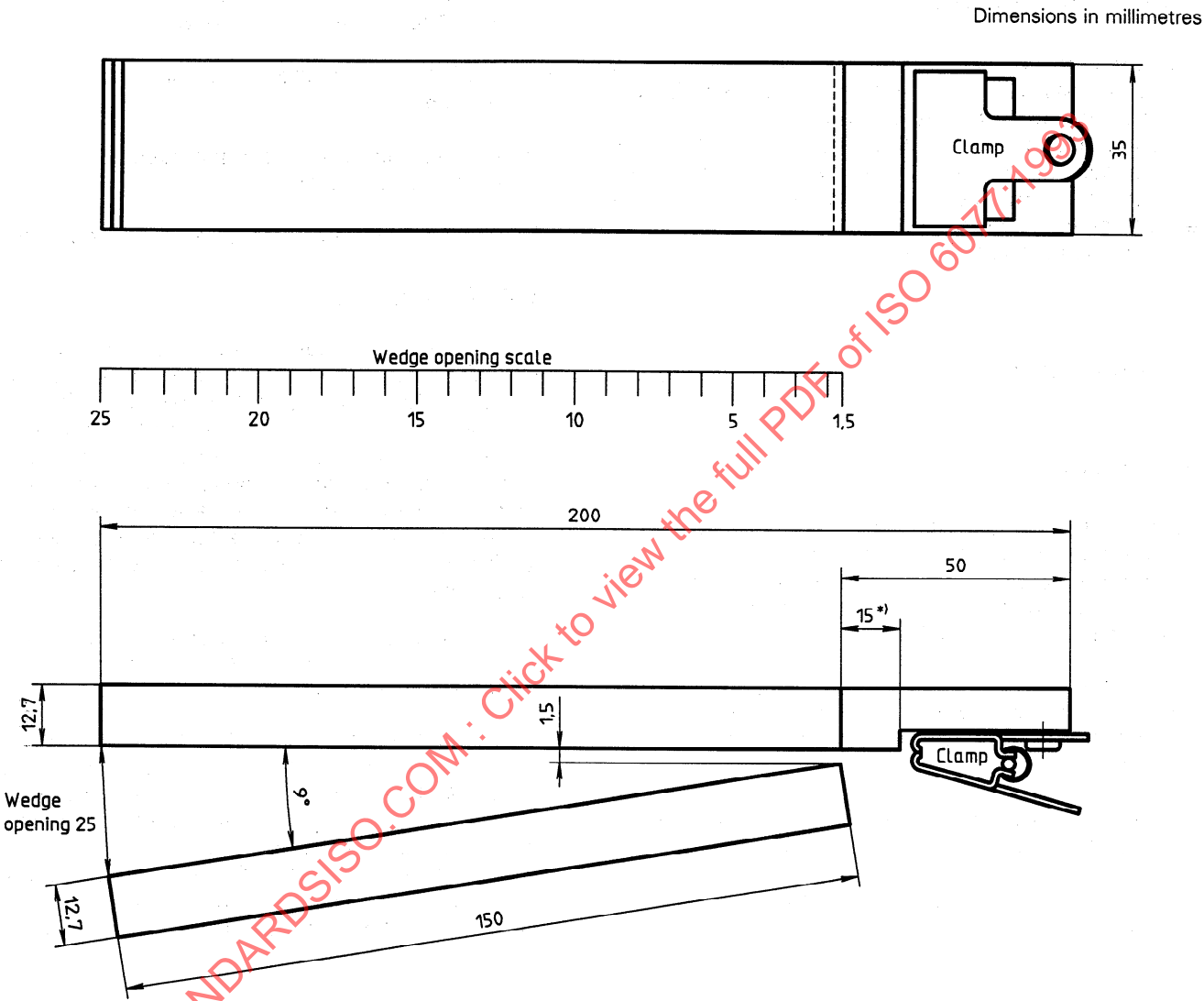


Figure 1 — Narrow wedge tester for brittleness

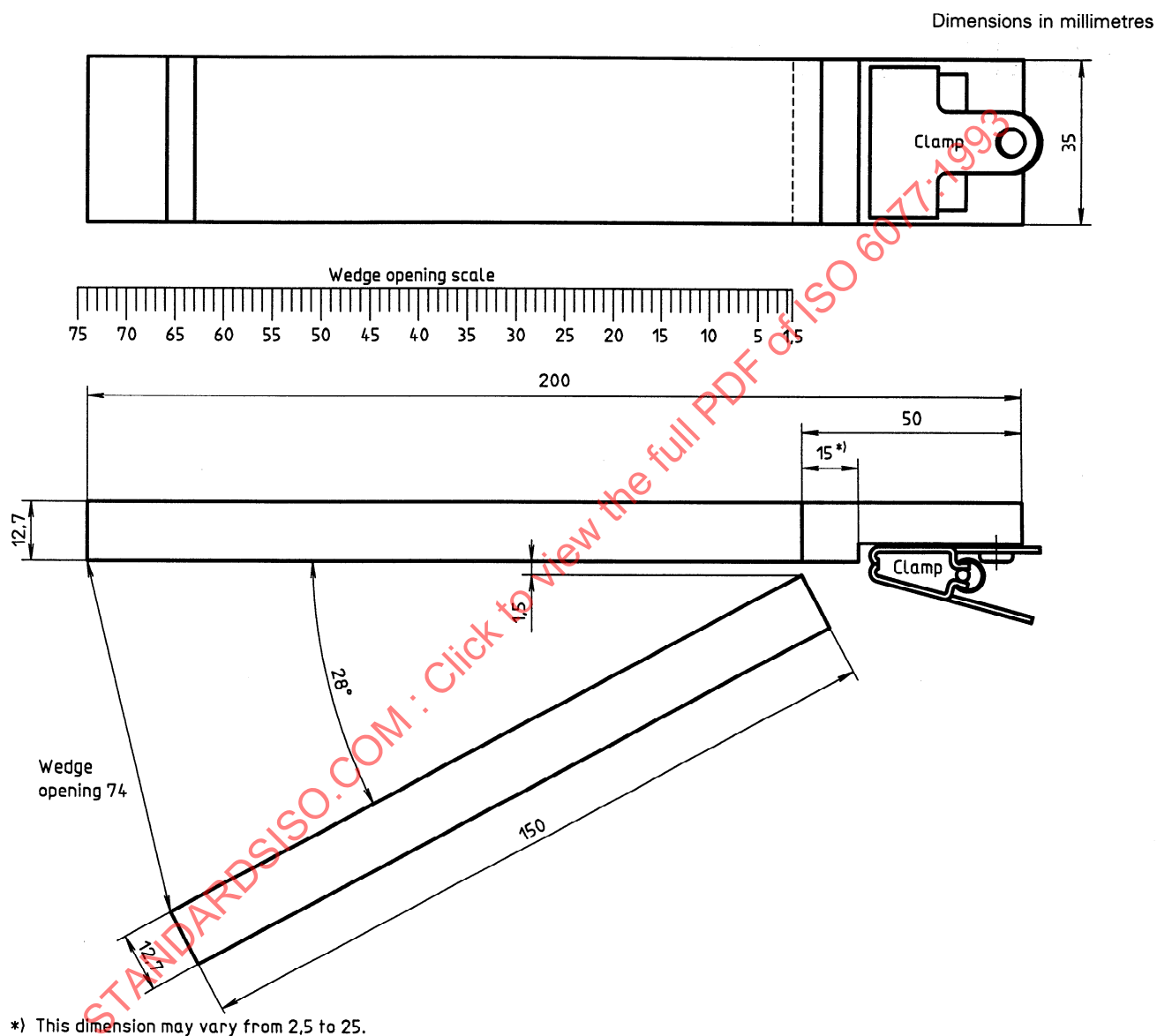


Figure 2 — Wide wedge tester for brittleness