INTERNATIONAL STANDARD

ISO 15361

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Pulps — Determination of zero-span tensile strength, wet or dry

Pâtes — Détermination de la résistance à la traction à mâchoires jointives, à l'état humide ou sec

Reference number
ISO 15361:2000(E)

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established 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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

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.

Attention is drawn to the possibility that some of the elements of this International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 15361 was prepared by Technical Committee ISO/TC 6, *Paper, board and pulps*, Subcommittee SC 5, *Test methods and quality specifications for pulp*.

Annex A forms a normative part of this International Standard.

Introduction

Tensile strength data at a span length of zero may be used to assess the retention of fibre strength through the entire fibre-processing chain, providing opportunities to optimize fibre characteristics and utilization in various paper grades. Tensile strength values determined at a span length of zero contribute to our understanding of finished sheet strength and are of increasing importance in measuring the impact of new pulping, bleaching and papermaking processes on fibre characteristics.

The zero-span tensile test may be used to determine the strength of pulp fibres when beaten under laboratory conditions, regardless of the laboratory beating procedure used. Measurement of zero-span tensile strength, in conjunction with tensile strength as well as other physical properties, is useful in optimizing new fibre-processing techniques and maximizing utilization of new fibre sources such as recycled fibres. Papers referenced in the bibliography give further information on the use of zero-span tensile measurements.

The clamping pressure utilized in zero-span testing ensures a maximum clamping effect but cannot totally prevent micro-slippage, whereby the tensile load transmitted in the clamped fibres is dissipated by frictional shear into the clamping jaws. This micro-slippage means that the ends of some fibres will slip out from beneath the clamping jaw, thereby diminishing the number of fibres carrying the load at tensile failure. In addition, if kinks in fibres are not removed in the beating process, test results may be diminished. For these reasons, careful interpretation of the zero-span tensile strength value should be exercised in order to separate effects due to the relative number of fibres which are carrying the load at failure, and the effects due to the tensile strength of the individual fibres present in the aggregate.

The zero-span strength values may be different if the samples are tested dry and conditioned, rewetted or wet (never dried).

Pulps — Determination of zero-span tensile strength, wet or dry

1 Scope

This International Standard specifies the procedure for determining the tensile strength of laboratory sheets at a test span which is initially zero. It is applicable to all kinds of fibres, including recycled fibres. The laboratory sheets can be tested either dry, rewetted, or never dried.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 187, *Paper, board and pulps — Standard atmosphere for conditioning and testing and procedure for monitoring the atmosphere and conditioning of samples.*

ISO 287, *Paper and board — Determination of moisture content — Oven-drying method.*

ISO 536, *Paper and board — Determination of grammage.*

ISO 1924-2, *Paper and board — Determination of tensile properties — Part 2: Constant rate of elongation method.*

ISO 5263, *Pulps — Laboratory wet disintegration.*

ISO 5264-1, *Pulps — Laboratory beating — Part 1: Valley beater method.*

ISO 5264-2, *Pulps — Laboratory beating — Part 2: PFI mill method.*

ISO 5269-1, *Pulps — Preparation of laboratory sheets for physical testing — Part 1: Conventional sheet-former method.*

ISO 5269-2, *Pulps — Preparation of laboratory sheets for physical testing — Part 2: Rapid-Köthen method.*

ISO 7213, *Pulps — Sampling for testing.*

3 Terms and definitions

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

3.1
tensile strength
maximum force per unit width that a test piece of the sample will withstand before breaking under the conditions defined in ISO 1924-2

3.2
zero-span
shortest possible span between the clamps that hold the sample; when clamps are adjusted to zero-span, a beam of light aimed between the two clamps is completely interrupted

3.3
zero-span tensile strength
tensile strength value measured using an appropriate instrument, with the clamps adjusted to zero-span, under conditions specified in this International Standard

3.4
zero-span tensile index
zero-span tensile strength divided by the grammage

NOTE Either conditioned or oven-dry grammage may be used in the calculation, and should be reported.

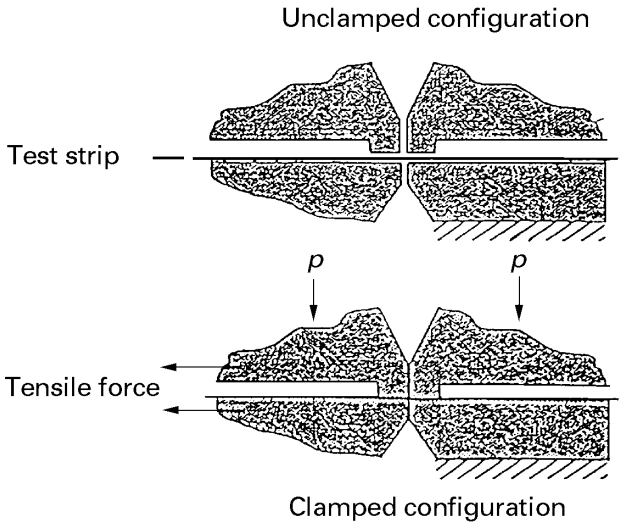
4 Principle

From a pulp suspension, laboratory sheets or wet test pieces are formed. They can be tested wet (i.e. never dried), dried (as conditioned) or rewetted. The test pieces are clamped in a tensile-testing instrument where the clamps are adjusted to zero-span and the test pieces are strained to break. The maximum force at rupture is measured and the zero-span tensile strength is calculated.

5 Apparatus

5.1 Tensile strength tester, complying with the following requirements.

5.1.1 Clamping device, incorporating two clamps with jaws for holding the test piece (see Figure 1). In each clamp, the lower jaw is planar, and is sometimes referred to as the anvil. The upper jaw is similar to the lower jaw, except that it includes a tip extending across the entire front width of the jaw and having a minimum dimension perpendicular to the width of at least 0,6 mm. The width of the jaws shall be between 15 mm and 25 mm, however, the exact width used is not critical, but shall be known to a certainty of ± 0,01 mm. The jaws shall be of identical width to ± 0,01 mm (see Figure 2).



p is the pressure applied to the test piece by the clamps in the pressurized state.

Figure 1 — Essential elements for any zero-span tensile tester

Dimensions in millimetres

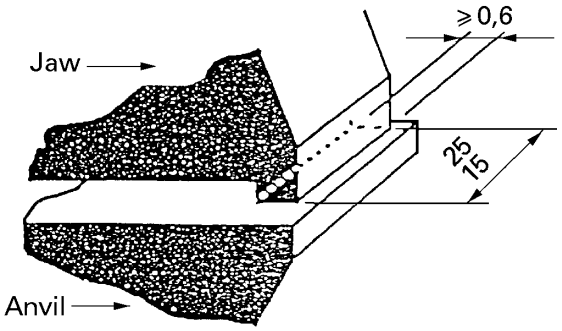


Figure 2 — Suitable arrangement for the clamps

There shall be a means for applying a clamping pressure by the clamps on the test piece. The clamping pressure shall be uniform to 0,1 % across the width of the jaw. The clamping pressure shall be variable between 250 kPa and 1 000 kPa.

The clamps shall be in alignment in both the horizontal direction, A, and vertical direction, B (see Figure 3). When the clamps are in the closed position with no test piece in place, a beam of light aimed to pass between the clamps, is completely interrupted. The alignment of the clamps is generally set by the manufacturer, and is not user-adjustable. In use, however, adherence of one or more fibres to one of the jaws is possible, in which case a light beam may not be completely interrupted. This matter shall be dealt with as described in 8.1.2.

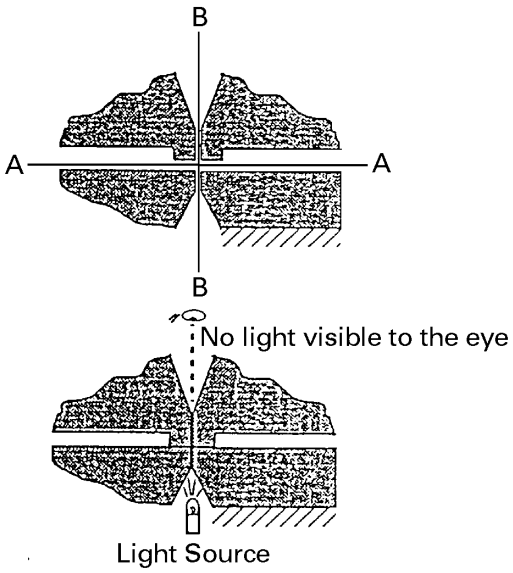


Figure 3 — Spatial alignment of the two clamps

5.1.2 Means of applying an in-plane tensile force within the fibre aggregate in the free span between the clamps, and of measuring the force at failure, complying with the following requirements.

The force shall be applied by tending to cause one clamp to move away from the other in such a way that the rate of increase of the force is (25 ± 2) N/s per 10 mm of jaw width. The accuracy of the means used to measure the applied force shall be ± 0,5 % of the measuring range of the tensile strength tester.

5.2 Laboratory sheet former, capable of producing homogeneous isotropic sheets with an oven-dry grammage of 60 g/m^2 ± 3 g/m^2.

5.3 Blotters, couch weight and plane press, for use in production of laboratory sheets and test pieces, as required in the standard for the sheet former used. (See ISO 5269.)

5.4 Device for cutting test pieces, see 7.2.

5.5 Immersion dish or **laboratory spray bottle** or **other suitable device for wetting of test pieces**. These are required only if the test piece is to be wetted before testing.

5.6 Sponge roller, for removal of excess water from the wetted test piece, or alternatively a rectangular block of sponge with a smooth, flat surface and without obvious surface imperfections. This is required only if the test piece is to be wetted before testing.

5.7 Sample inserter, made of stainless steel or another noncorrosive material, 0,5 mm or less in thickness. This may be used to transport a wet test piece into the jaws of the tester.

5.8 Mould, made of stainless steel or another noncorrosive material, for preparing test pieces when testing never-dried laboratory sheets. For dimensions see 7.3.

5.9 Various items of common laboratory equipment, for determination of grammage of the never-dried samples. See ISO 536.

5.10 Disintegrator, in accordance with ISO 5263. This is needed only when a sample of dry or semi-dry pulp is to be tested.

5.11 Device for mechanical treatment of fibres, to reduce fibre curl or kinks, if required, such as a laboratory beater or pulper, for example a Valley beater (ISO 5264-1), a PFI mill (ISO 5264-2) or a high-speed blender as described in annex A.

6 Sampling

If the result is intended to reflect the quality of a lot of pulp, sampling shall be carried out in accordance with ISO 7213.

7 Preparation of test pieces

7.1 Introduction

The test method requires a random aggregate of fibres in sheet form for testing. Even when the sample to be tested is obtained in sheet form, the fibres shall be reduced to a slurry and then reformed into a randomly oriented sheet, unless they are in the form of a laboratory sheet complying with 7.3. If desired, the fibre slurry may be treated to remove fibre curls or kinks prior to sheet forming, but this shall be reported together with the results.

7.2 Pretreatment of sample

For pulps received in a dry or semi-dry form, disintegrate the pulp in accordance with ISO 5263. Pulp slurries may also be disintegrated as required or agreed.

Remove from the slurry the quantity of pulp fibres required for preparation of the laboratory sheets. The exact amount of fibre required will depend upon the sheet-forming procedure.

If the concentration of fibres in the slurry is greater than that required in the following steps, reduce the concentration by adding water.

Fibres in mill-produced pulp or mixed/kneaded laboratory pulp are often deformed (curled or kinked). This deformation influences the measured zero-span tensile strength value. If the deformations need to be eliminated, soak the pulp as described in ISO 5263, disintegrate if necessary, and mechanically treat the fibres. An example of a treatment, which may be suitable for straightening deformed fibres so that their maximum zero-span tensile strength may be measured, may be 15 min in a Valley beater (ISO 5264-1), 3 000 revolutions in a PFI mill (ISO 5264-2), or 5 min at 0,2 % consistency using the high-speed mixer described in annex A. Other treatments may also be possible.

7.3 Preparation of test pieces to be tested as dry or rewetted

Prepare laboratory sheets using equipment that will produce homogeneous isotropic sheets with an oven-dry grammage of 60 g/m^2 ± 3 g/m^2, for example by the procedures described in ISO 5269.

Determine the grammage of the sheets as described in ISO 536.

If the pulp is to be tested dry, condition the laboratory sheet in accordance with the requirements of ISO 187.

From the laboratory sheet, cut test pieces with a width exceeding the jaw width of the clamp used by at least 2 mm, and with a length exceeding the total length of the tips of the two jaws by 1,2 mm. A convenient test piece length may be 25 mm to 50 mm, but will depend on the instrument.

7.4 Rewetting of test pieces

7.4.1 General

It is important to thoroughly saturate the test piece with water, and then to remove water to result a in dry-matter content between 20 % and 50 %. This can be done by the procedures given in 7.4.2 or 7.4.3, or by other means consistent with the stated intent and which do not damage the test piece.

7.4.2 Spraying

Place the test piece on a prewetted sponge block (see 5.6) and spray with distilled or deionized water at a temperature of 23 °C ± 5 °C using a common laboratory spray bottle, until the test piece is saturated. Saturation can generally be judged as complete when the test piece is uniform in appearance. Remove any free water from the test piece using another piece of sponge block or a sponge roller, removing water until the dry matter content of the test piece is between 20 % and 50 %. Proceed as described in 8.2.1.

NOTE It might be helpful to carry out a pretest in order to find out how to achieve a dry matter content of 20 % to 50 %. The approximate dry matter content in the range stated may be confirmed by use of a rapid moisture method of choice, or ISO 638, *Pulp — Determination of dry matter content.*

7.4.3 Immersion

Immerse the test piece in distilled or deionized water having a temperature of 23 °C ± 5 °C. Normally, the test piece will be immediately wetted, but some test pieces may require soaking for a period of time to be completely saturated with water. Saturation can generally be judged as complete when the test piece is uniform in appearance. Check the required soaking period for various types of fibres. Include the soak time in the test report. Place the wet test piece on a prewetted sponge block and remove excess water with another sponge or a sponge roller until the dry matter content of the test piece is between 20 % and 50 %. See note to 7.4.2. Proceed as described in 8.2.1.

7.5 Preparation of never-dried test pieces

7.5.1 General

Use the equipment and procedures specified in ISO 5269-1 or ISO 5269-2 modified as in 7.5.2, 7.5.3 or 7.5.4. Alternatively, use some other sheet-forming device that will produce homogeneous isotropic test pieces with a dry matter content of 20 % to 50 % and an oven-dry grammage of 60 g/m^2 ± 3 g/m^2. Specific details of preparation will depend upon the equipment and procedure used. When forming is accomplished manually using ISO 5269-1 or ISO 5269-2, or using some other manual sheet-forming device, the user will find that the procedures in 7.5.2 to 7.5.4 provide useful guidance, although the procedures used shall be consistent with the sheet former used. Automated sheet formers may also be used, providing they can produce sheets complying with the requirements of this subclause, in which case 7.5.2 to 7.5.4 may not apply or may apply only with appropriate modification.

7.5.2 Forming

Place the mould (5.8) on the wire of the sheet former (5.2). With the mould in place, prepare the test pieces using the equipment and procedure specified in ISO 5269-1 or ISO 5269-2, or using some other sheet-forming device that can produce homogeneous, isotropic test pieces of the required grammage.

7.5.3 Couching

Remove the mould carefully. Place two blotters (5.3) on the test pieces. Place the couch weight (5.3) on top of the blotters and remove it after a period long enough for the pieces to adhere to the blotter touching it. Lift the blotters and the adhering test pieces from the wire of the sheet former (5.2) used.

7.5.4 Pressing

Remove the blotter which is not touching the test pieces and place it on the exposed surface of the pieces, so that the pieces are contained between two blotters in the form of a "sandwich". Place additional blotters on each side of the sandwich and place the pad of pieces and blotters in a flat plane press (5.3). Raise the pressure on the pad at a constant increasing rate over a period of 25 s to 410 kPa $\pm$ 10 kPa. Maintain this pressure for 2 min. A stack containing several pads may be pressed in one operation. The number of backing sheets on each side of the wet pieces shall be adjusted by trial and error to give pieces having a dry-matter content of 20 % to 50 % at the end of the 2 min pressing time.

8 Procedure

8.1 Calibration

For calibration of the zero-span tester, use the calibration procedure specified by the manufacturer.

8.1.1 Force values

In the absence of the manufacturer's instructions, use a calibrated force gauge with a relative tolerance of $\pm$ 0,5 % or better to verify the force values being measured by the instrument. Make measurements at a minimum of six points over the usable-load measuring range of the instrument. Values on the calibrated force gauge should agree within 1 % of the values measured by the instrument.

8.1.2 Vertical alignment

Verify that the vertical surfaces, which are in contact at zero-span, shall, when clamped (see 5.1.1), conform to Plane B in Figure 3 with a tolerance such that a beam of light is completely interrupted when the jaws are in clamped zero-span contact. As an alternative, insert a piece of aluminium foil between the clamps and close the jaw. By using a microscope, check from the imprint if there is vertical alignment. The vertical alignment is adjusted to comply with this requirement by the manufacturer, but contamination of the jaw tips by small fibre bits can allow light to pass between the jaws in the vertical plane. In some cases, these fibre bits may be removed by opening and closing the jaws several times on a piece of writing paper or slightly thicker paper stock used for making cards or files. In other cases, gently cleaning the tips of the jaw surfaces with a non-linting material such as cotton on the end of a small wooden stick may be useful. Examination of the jaw tips using a low-power magnifying glass will generally identify the area of contamination, and aid in its elimination. If, after following the above procedures, and verifying the absence of fibre bits by examination with a magnifying glass, jaw alignment in Plane B of Figure 3 is not achieved, consult the instrument manual or contact the manufacturer to determine additional steps which may be taken to identify the cause of the apparent jaw misalignment.

8.1.3 Clamping pressure

Choose the optimal clamping pressure. The optimal clamping pressure is determined by testing at clamping pressures increased in steps of 150 kPa between about 250 kPa and 1 000 kPa. The measured zero-span tensile strength will increase to a constant value until an additional increase in clamping pressure will cause damage to the

fibres, at which the test value will begin to decrease. The optimal clamping pressure for use is that giving the highest measured value at break. Frequently, little change in the test result will be seen as the clamping pressure is varied between about 450 kPa and about 650 kPa, in which case a clamping pressure for testing of about 550 kPa is recommended.

8.2 Determination

8.2.1 Insertion of test pieces

Place a test piece between the instrument jaws (see 5.1.1). If desired, based upon the operational characteristics of the testing instrument, use a sample inserter (5.7) to make it easier to insert test pieces that have been wetted or never dried. In this case, the following procedure is recommended.

Lightly press the sample inserter on the wet test piece, aligning a long edge of the test piece with one edge of the inserter, and taking care not to handle the test piece in the region to be tested. Gently raise the inserter away from the sponge block (see 5.6). The test piece will remain on the inserter. If there is an excess of free water on the test piece, the test piece may be held so firmly to the sample inserter that it is damaged during insertion into the instrument clamps. Alternatively, the wet test piece may be peeled from the sponge block and placed on the sample inserter or directly into the instrument grips. Handling of wet test pieces shall be done with great care, as they are easily damaged.

Close the jaws of the zero-span tensile strength tester.

8.2.2 Measurement

Carry out at least 10 determinations.

Start the test and record the force (Z_B) when the test piece breaks.

8.2.3 Dry-matter content and grammage

Immediately after performing the test on wet test pieces, determine the dry-matter content of the test pieces according to ISO 287, and then calculate the oven-dry grammage of the laboratory sheets. Adjust the grammage to a conditioned grammage (23 °C and 50 % RH) based on knowledge of the typical equilibrium moisture of the type of fibres being tested. For example, if the typical equilibrium moisture level of the type of fibres is 5 %, multiply the oven-dry grammage by 1,05 to obtain the conditioned grammage; for a moisture level of 7 %, multiply by 1,07.

8.3 Test on reference material

Perform the test on a reference pulp or a reference paper for routine control of the instrument. Suitable pulps for use as an ongoing reference are available from The Pulp and Paper Research Institute of Canada, and National Institute for Science and Technology (USA). Users of the instrument may establish their own internal reference pulp or paper materials for the specified purpose of routine control.

9 Expression of results

9.1 Reporting of results

Report separately the results obtained for testing done using dry (conditioned), rewetted or wet (never-dried) test pieces, as applicable.

9.2 Zero-span tensile strength

For each test piece, calculate the zero-span tensile strength from the expression

$$Z_T = \frac{Z_B}{b}$$

where

Z_T is the calculated zero-span tensile strength, in kilonewtons per metre;

Z_B is the zero-span tensile force from 8.2.2, in kilonewtons;

b is the width of the instrument jaws, in metres.

Report the mean zero-span tensile strength to three significant figures. Calculate the standard deviation of the result.

Alternatively, the mean and standard deviation of the zero-span tensile force may be obtained first and then divided by the width of the instrument jaws.

9.3 Zero-span tensile index

Calculate the zero-span tensile index from the expression

$$Z_I = \frac{Z_T}{G}$$

where

Z_I is the calculated zero-span tensile index, in kilonewton metres per gram;

G is the oven-dry or conditioned grammage, in grams per square metre.

NOTE Either conditioned or oven-dry grammage may be used in the calculation, and should be stated in the test report.

Report the zero-span tensile index to three significant figures. If required, calculate the standard deviation of the results.

10 Precision

In a precision study published by ASTM, 98 determinations of wet zero-span strength were made in a single laboratory. The study included samples of bleached hardwood pulp, bleached softwood pulp and neutral sulfite semi-chemical pulp. The results ranged from 6,0 kN/m to 8,8 kN/m.

In the same study, precision data for dry zero-span strength were based on determinations of over 30 samples of various types of pulp tested in three laboratories. The results ranged from 7,5 kN/m to 9,5 kN/m.

The data in Table 1 were calculated from the above results.

Table 1 — Calculated coefficient of variation

Property	Coefficient of variation within laboratory %	Coefficient of variation between laboratories %
Wet zero-span strength (mean value 7,5 kN/m)	1,5	—
Dry zero-span strength (mean value 8,8 kN/m)	1,0	1,8

11 Test report

The test report shall include the following particulars:

a) reference to this International Standard;

b) date and place of testing;

c) complete identification of the sample tested;

d) the grammage of the laboratory sheets, oven-dry or as conditioned;

e) the sheet former and sheet-forming procedure used;

f) the type of samples tested: wet, dry (conditioned), rewetted;

g) the soaking time of rewetted samples, if applicable;

h) the laboratory beating method, if any was used;

i) the zero-span tensile strength;

j) the zero-span tensile index, whether calculated on oven-dry or conditioned basis;

k) the standard deviation of the zero-span tensile strength;

l) the clamping pressure chosen in 8.1.3;

m) any departure from the standard procedure or any circumstances that may have affected the results.

Annex A
(normative)

Alternative equipment for straightening fibres

In an alternative procedure for straightening fibres, a high-speed laboratory blender is used. The blades of the blender shall be dulled as shown in Figure A.1.

The mixing chamber shall be cylindrical of a style designed to fit the mixing head, and have a nominal capacity of 1 litre. The mixing head shall be motor driven and have a nominal speed of at least 16 000 r/min under load.

The radius of the leading edge of the ends of the blades when viewed from above shall be 1,65 mm to 1,78 mm.

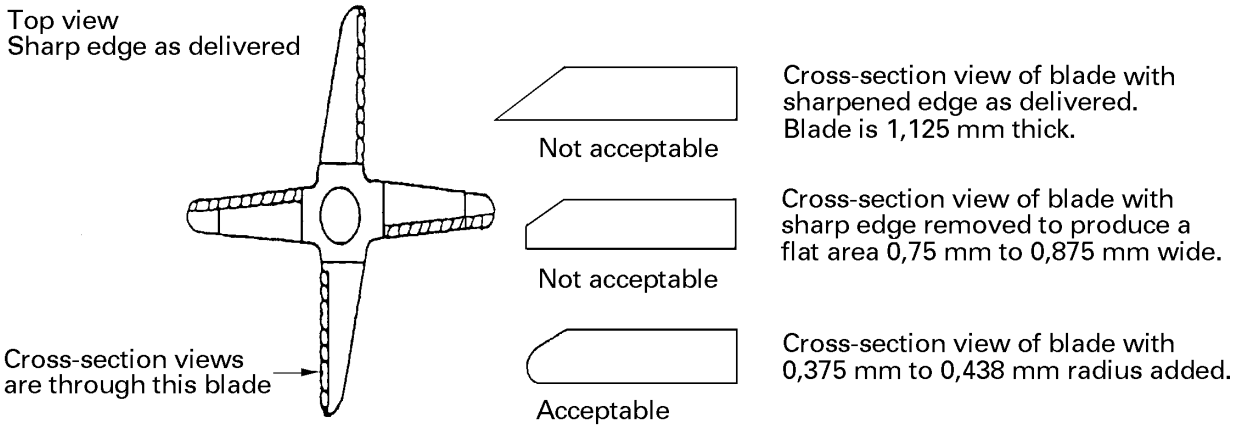


Figure A.1 — Key elements of the blender-blade configuration

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