

42

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Photography — Films and papers — Determination of dimensional change

*Photographie — Films et papiers — Détermination des variations
dimensionnelles*

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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 6221 was prepared by Technical Committee ISO/TC 42, *Photography*.

This second edition cancels and replaces the first edition (ISO 6221:1980), which has been technically revised.

Annexes A, B and C of this International Standard are for information only.

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Introduction

This revision of ISO 6221:1980 includes additional information on photographic paper and expands the coding system for processing changes.

Photographic films and papers exhibit temporary or reversible dimensional changes as well as permanent dimensional changes. This International Standard is designed to provide uniform methods for treating the samples and for expressing the dimensional changes which occur with changes in atmospheric conditions and those which occur in processing and ageing.

Temporary or reversible dimensional changes are the result of changes in equilibrium moisture content (which is determined by the relative humidity of the surrounding atmosphere) or changes in temperature. Permanent dimensional changes occur as the result of processing and ageing. The rate of permanent shrinkage of film generally increases with temperature but decreases with time. The rate of shrinkage may also be greatest at either high or low relative humidity, depending on the type of film. Some materials, particularly photographic film on polyester base, can show a swelling after a high humidity exposure.

The increasing use of photographic films in recent years, in applications where dimensional stability is critical, has emphasized the importance of an accurate measure of dimensional properties. For example, in photomechanical reproductions a dimensional change of as little as 0,01 % may be of practical importance. In the case of aerial mapping, uniform shrinkage is not serious since it can be easily corrected by a change in magnification, but any difference in shrinkage in the two principal directions is a source of error. Any localized or non-uniform changes in dimension are of practical concern.

The dimensional change properties of any film or paper depend not only on their composition and method of manufacture but also on their thermal and moisture content history. Accurate evaluation of such properties requires some control over the sample history as well as very precise control over the conditioning and measuring procedures. Film and paper dimensions are also subject to hysteresis effects. These are relatively more important with the more stable materials such as polyester photographic base films.

Additional information on the dimensional characteristics of photographic films and papers and on methods of measurement may be found in the bibliography (see annex C).

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Photography — Films and papers — Determination of dimensional change

1 Scope

This International Standard gives a method for determining the dimensional change of photographic films and papers caused by

- a) variations in equilibrium moisture content due to change in the relative humidity of the atmosphere (humidity coefficient of expansion);
- b) change in temperature (thermal coefficient of expansion);
- c) processing;
- d) ageing.

This International Standard deals with the moisture content and thermal history of the samples before measurement, the atmospheric conditions during measurement, and the treatment of the data. This standard does not describe the various experimental techniques used to make the measurements.

This International Standard is not suitable for determining the dimensional change of instant photographic film.

2 Definitions

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

2.1 humidity expansion (or contraction): The dimensional change caused by the gain (or loss) of moisture following changes in the relative humidity of the ambient air at constant temperature.

1) This is an apparent thermal expansion since the moisture content of film varies slightly with temperature at constant relative humidity. However, the primary effect is thermal expansion. Thermal expansion is less important for paper because of the small changes involved, particularly compared to humidity effects.

2.2 humidity coefficient of expansion: The change in dimension per unit length per 1 % change in relative humidity.

2.3 thermal expansion (or contraction): The dimensional change caused by a rise (or fall) of temperature at constant relative humidity¹⁾.

2.4 thermal coefficient of expansion: The change in dimension per unit length per 1 °C change in temperature.

2.5 dimensional change due to processing: The permanent dimensional change caused by photographic processing. This may be the conventional wet chemical processing, vapour processing or heat processing. It is measured after conditioning at the same relative humidity and temperature as used for the original measurement and is expressed as a percentage.

2.6 dimensional change due to processing plus ageing: The permanent dimensional change which occurs as a result of processing plus ageing of the processed material. It is measured after conditioning of the processed, aged film or paper at the same relative humidity and temperature as used for the original measurement and is expressed as a percentage.

2.7 length direction: The direction of the film or paper parallel to its forward movement in the film- or paper-making machine. This is also termed "grain" or "machine direction" in the case of papers.

2.8 width direction: The direction of the film or paper at right angles to the length direction. This is also termed "cross direction".

2.9 differential dimensional change: The difference between the dimensional changes of the material in the two principal directions (length and width)²⁾.

2.10 conditioning: The exposure of a sample to air at a given relative humidity and temperature until equilibrium is reached.

2.11 preconditioning: The establishment of a moisture content history by conditioning the sample at a relative humidity above or below the conditioning relative humidity used for measurement. The purpose of preconditioning is to control the effects of hysteresis (see 2.12).

2.12 dimensional hysteresis: The difference in the absolute dimensions of a sample in equilibrium with air at a given relative humidity, when conditioned from a higher relative humidity and when conditioned from a lower relative humidity (see annex B).

3 Technique of measurement

There are a number of different techniques used for measuring the dimensional change of sensitized materials. Specifications of measuring equipment is beyond the scope of this International Standard, but several approaches are described in annex A.

4 Sampling

4.1 Selection of samples

Samples intended for dimensional stability tests should exhibit no obvious physical defects, be representative of the whole of the material being tested, be handled in the same manner as in actual use, and be treated uniformly. When different materials are to be compared, they should preferably have been subjected to the same conditioning history. The length direction should be indicated if known.

2) Polyester-based films frequently have the maximum and minimum dimensional changes in directions other than the length or width. These can be determined by rotating and viewing the uncoated base between a pair of crossed polarizers. When either the maximum or minimum direction is coincident with the optical axis of one polarizer, there is minimum light transmission through the base.

3) The room shall be vapour sealed and insulated on all six sides. The room shall be mechanically air-conditioned and the air circulated at a linear velocity of at least 15 cm/s. The number of personnel permitted in the room at any one time during testing shall be limited. The relative humidity of the room shall be checked regularly, preferably by means of an electric hygrometer calibrated by a dew point method.

4) A convenient size for a humidity cabinet is approximately 1 m in height and 0,5 m in width and depth. It should be constructed of materials which will ensure good insulation. Suitable provision shall be made for thermostatically controlling the temperature within the cabinet. Air shall be circulated throughout the cabinet at a linear velocity of at least 30 cm/s. The cabinet shall be equipped with ports filled with moisture impermeable (e.g. rubber or plastic) gloves for entrance of the operator's hands. The relative humidity of the cabinet shall be checked regularly, preferably by means of an electric hygrometer calibrated by a dew point method.

4.2 Handling of specimens

Specimens shall be prepared under controlled conditions and then separated into groups which are subjected to different atmospheric conditions. The operator shall take care not to breathe on the specimens and shall wear moisture-resistant gloves while handling them.

4.3 Processing of specimens

Specimens shall be exposed and processed by methods and equipment normal for the product. When the effects of processing machines, tensions, or drying conditions are being investigated, the film or paper should be processed in the sizes of practical interest. Specimens may be developed as negatives or as positives but this can affect the dimensional change properties of some materials. Silver-gelatin films generally show less dimensional change when they have low density rather than with high density.

5 Conditioning

5.1 Constant humidity chamber

Either a walk-in constant humidity room or a cabinet may be used.

5.1.1 Constant humidity room

The relative humidity shall be held constant to $\pm 1\%$ RH or better in areas of the room where specimens are measured³⁾.

5.1.2 Constant humidity cabinet

The relative humidity within the cabinet⁴⁾ should be controlled as closely as possible. Where the cabinet is mechanically air-conditioned, the relative humidity shall be constant to $\pm 1\%$ RH or better. Where a saturated salt solution is used for control, provision shall be made at the bottom of the cabinet for inserting suitable trays, which shall hold about

1 l of salt solution. A large surface area is needed and about 1000 cm² is suitable.

5.2 Standard temperature and humidity

The standard temperature shall be 23 °C ± 0,5 °C except for the test specified in clause 7. The relative humidity is specified in the respective test procedure subclauses and depends upon the property being measured.

5.3 Conditioning of specimens

Specimens may be suspended in the conditioning atmosphere by means of a hook or a rod through a hole in the middle of one end near the edge of the specimen. The specimens should be separated to prevent contact with each other. An alternative method of conditioning is to place specimens in racks spaced so that there is free circulation of the air on both sides of the material. The specimens shall not be removed from the conditioning atmosphere for measuring. Condition specimens until practical moisture equilibrium has been reached⁵⁾.

5.3.1 Film

The conditioning time for film will be about 4 h but will vary according to access of the conditioning air, the film type, base thickness, etc. Conditioning time should not exceed 24 h⁶⁾.

5.3.2 Paper

Double weight non-resin coated papers will require about 8 h of conditioning; resin coated papers require up to 14 d depending on resin coverage.

6 Test for humidity coefficient of expansion

6.1 Procedure

Five specimens shall be preconditioned⁷⁾ at 10 % to 15 % RH, then conditioned at 15 % to 25 % RH (but at least 5 % RH above the preconditioning relative humidity) and measured. They shall then be conditioned again at 50 % to 60 % RH and remeasured⁸⁾. The conditioning temperature shall be constant as specified in 5.2. The two conditioning

humidities shall be measured to a precision of ± 1 % RH in accordance with 5.1.1.

The test may be made on both unprocessed and processed specimens depending on the measuring method used (see annex A). The humidity coefficient of expansion of unprocessed and processed film is generally not the same.

6.2 Calculations

Since the dimensional change curve versus relative humidity is not always linear (see annex B), this test method gives only an average coefficient over the range measured. The dimensional change between the two measurements of five specimens shall be averaged and the humidity coefficients of expansion shall be calculated in accordance with the following formula:

$$H = \frac{l_2 - l_1}{l_1 \times \Delta RH}$$

where

H is the humidity coefficient of expansion per 1 % RH;

l_1 is the gauge distance or the dimension measured at the initial conditioning temperature and relative humidity;

l_2 is the gauge distance or the dimension measured at the final conditioning temperature and relative humidity;

ΔRH is the difference between the two conditioning relative humidities used, as a percentage.

6.3 Test report

The test report should contain the following:

- humidity coefficients of expansion for both the length and width directions;
- two conditioning relative humidities and temperature;
- a statement as to whether the samples were unprocessed, processed to high density, or processed clear;

5) The time required to achieve this shall be established by actual measurements on representative samples or based on prior experience.

6) At relative humidities of 60 % and above, films and papers sometimes undergo an irreversible change in size with time. For this reason, the conditioning time must be standardized for comparison purposes.

7) Preconditioning times of 1 h to 2 h are recommended for photographic film, 4 h for non-resin coated paper, and 7 d for resin coated paper

8) This range of relative humidity is selected because the dimensions with respect to the relative humidity curve for some materials is abnormal, i.e. above 60 % RH (see annex B).

- d) a statement as to the statistical validity of the measured coefficient.

7 Test for thermal coefficient of expansion⁹⁾

7.1 Procedure

Five specimens shall be conditioned, first at 45 °C to 50 °C and measured, and then conditioned at 10 °C to 25 °C and measured again¹⁰⁾. A conditioning time of 4 h at each temperature is recommended. Both the specimens and the measuring equipment shall be at thermal equilibrium. The relative humidity shall be the same at both temperatures and controlled as specified in 5.1. A low humidity is more practical for laboratory work, but other relative humidities may be used¹¹⁾. The test can be made on either unprocessed or processed material, depending on the measuring method used (see annex A). The measuring equipment shall not be affected by the thermal changes or else the measurements shall be corrected for thermal effects.

7.2 Calculations

The dimensional change between the two measurements of five specimens shall be averaged and the thermal coefficients of expansion shall be calculated in accordance with the following formula:

$$\alpha = \frac{l_4 - l_3}{l_3 \times \Delta T}$$

where

- α is the thermal coefficient of expansion per 1 °C;
- l_3 is the gauge distance or the dimension measured at the initial conditioning temperature and relative humidity;
- l_4 is the gauge distance or the dimension measured at the final conditioning temperature and relative humidity;
- ΔT is the difference between the two conditioning temperatures used, in degrees Celsius.

Depending on the type and composition of the measuring equipment, it may be necessary to correct for the thermal expansion of the gauge or of the reference standard.

9) Dimensional changes of photographic paper with reasonable changes in temperature (but at the same moisture content) are so small that measurement is very difficult and has little practical significance. This method is of importance for photographic film.

10) The high-temperature measurement is made first so that any permanent shrinkage which may occur during conditioning will not affect the result.

11) The thermal coefficient of expansion varies slightly with relative humidity for some materials.

7.3 Test report

The test report should contain the following:

- thermal coefficients of expansion for both the length and width direction;
- conditioning temperatures and relative humidity;
- a statement as to whether the samples were unprocessed, processed to high density, or processed clear;
- a statement as to the statistical validity of the measured coefficient.

8 Test for dimensional change due to processing

8.1 General

The dimensional change due to processing of photographic films and papers can be markedly affected by the following three variables in the test procedure:

8.1.1 Preconditioning relative humidity of raw sample

The dimension of the unprocessed specimen at a given relative humidity can be dependent to a large extent upon the preconditioning humidity history. This is due to the hysteresis and relaxation effects described in annex B. It is necessary to specify whether the sample had been preconditioned from a lower or from a higher humidity prior to the original measurement on the unprocessed material. This is indicated by the letter "L" for the former and "H" for the latter. When film is measured from the original box (or packaging) without preconditioning, the letter "B" is used.

8.1.2 Preconditioning relative humidity of processed sample

The preconditioning humidity history is as important for the dimension of the processed sample as for the raw sample described in 8.1.1. The letters "L" and "H" are also used to indicate the humidity history of the processed material. The letter "H" is also used when the sample is dried after processing at the measuring humidity.

Although the drying conditions after processing affect the dimensional change due to processing, the effect is erased when the processed film is preconditioned. If measurements are made on processed film that is not preconditioned after processing, the letter "M" is used.

8.1.3 Measuring relative humidity

Samples shall be conditioned to moisture equilibrium with the same relative humidity for both the raw and processed measurements. Otherwise a reversible humidity change will be included in the measured dimensional change. However, the dimensional change due to processing can be very dependent upon the relative humidity at which those measurements are made and this shall be indicated when reporting the results.

8.2 Coding system

A wide variety of test procedures can be used for the measurement of dimensional change due to processing by varying the preconditioning history of the raw and processed samples and the relative humidity at which these measurements are made. To characterize the procedure used, a coding system is used. For example, procedure LH-50 signifies that the raw sample had been preconditioned from a low humidity (L), the processed sample had been preconditioned from a high humidity (H), and all measurements were made at 50 % RH. Likewise, procedure LL-10 indicates that both the raw and processed sample had been preconditioned at a lower humidity than the 10 % RH at which the samples were measured. In practice, the range encompassed by LH-50 (tray processed and air dried) and HL-50 (machine processed and machine dried) generally reflects the expected variation in processing dimensional change. The procedure BM-50 indicates that no preconditioning was done for either the raw film from the original box (or packaging), or the processed film from the machine.

8.3 Procedure

Five specimens shall be preconditioned¹²⁾. The preconditioning relative humidity shall be chosen to provide the desired low (L) or high (H) humidity history and shall be at least 5 % RH below or above

the measuring humidity. The specimens shall then be conditioned to the measuring relative humidity and the specimen dimensions shall be measured (or exposed if a photographic image is to be used for measurement as described in annex A). The specimens shall be processed and dried as outlined in 4.3. The specimens shall subsequently be preconditioned to the desired low or high preconditioning humidity, conditioned at the measuring relative humidity and remeasured¹³⁾. The conditioning temperature shall be the same before and after processing and shall be controlled as specified in 5.2; the conditioning time shall be as specified in 5.3.

8.4 Calculations

The dimensional changes between the two measurements of five specimens shall be averaged and the processing dimensional change calculated according to the following formula:

$$\%P = \frac{l_6 - l_5}{l_5} \times 100$$

where

$\%P$ is the dimensional change due to processing, as a percentage. A negative sign indicates shrinkage and a positive sign shows expansion;

l_5 is the gauge distance or the dimension measured at the initial conditioning temperature and relative humidity;

l_6 is the gauge distance or the dimension measured at the final conditioning temperature and relative humidity.

8.5 Test report

The test report should contain the following:

- procedure used (for example LH-50);
- dimensional changes due to processing for both the length and width directions;
- processing conditions (including drying conditions) and whether processed to a high density or processed clear.

12) Preconditioning times of 1 h to 2 h are recommended for photographic film, 4 h for non-resin coated paper, and 7 d for resin coated paper.

13) When samples are dried at the measuring humidity the letter "H" is used without the need for a separate preconditioning step.

9 Test for dimensional change due to processing plus ageing

9.1 Ageing conditions

The ageing conditions¹⁴⁾ used shall be selected from the following¹⁵⁾:

- a) normal conditions, 23 °C ± 2 °C and (50 ± 5) % RH;
- b) summer conditions, 32 °C ± 2 °C and (50 ± 5) % RH;
- c) moist tropical conditions, 32 °C ± 2 °C and (90 ± 5) % RH;
- d) dry accelerated conditions, 50 °C ± 2 °C and (20 ± 5) % RH.

9.2 Ageing time

The ageing times used shall be selected from the following: one week, one month, three months, six months, one year or multiples thereof.

9.3 Procedure

Five specimens shall first be subjected to procedure LL-50 in accordance with 8.3. They shall be placed at least 6 mm apart on racks, or hung from clips, and stored under one or more of the ageing conditions listed in 9.1. After the ageing time (see 9.2) has elapsed, the specimens shall be preconditioned¹⁶⁾ at a low humidity at the standard temperature (see 5.2), then conditioned at 50 % RH, as specified in 5.3, and remeasured. The specimens may then be returned to the same ageing condition and stored for another time interval if desired.

9.4 Calculations

The dimensional changes between the initial measurement of the raw material and the final measurement of the processed and aged material for the five specimens shall be averaged and the dimensional change due to processing plus ageing calculated in accordance with the following formula:

$$\%A = \frac{l_8 - l_7}{l_8} \times 100$$

where

$\%A$ is the dimensional change due to processing plus ageing, as a percentage. A negative sign indicates shrinkage and a positive sign indicates expansion;

l_7 is the gauge distance or the dimension measured at the initial conditioning temperature and relative humidity;

l_8 is the gauge distance or the dimension measured at the final conditioning temperature and relative humidity.

9.5 Test report

The test report should include the following:

- a) dimensional change due to processing plus ageing for both the length (machine) and width (cross) directions;
- b) processing conditions (including drying conditions) and whether processed to a high density or processed clear;
- c) ageing conditions;
- d) ageing time.

14) Moist, accelerated conditions hasten shrinkage caused by loss of residual solvent in solvent-coated film base. Dry accelerated conditions hasten shrinkage caused by strain relaxation or plastic flow of film base. Photographic paper does not have solvent loss shrinkage but does change dimension with humidity cycling due to strain relaxation (see annex B).

15) The tolerances on temperature and relative humidity for ageing conditions do not need to be as critical as the tolerances on temperature and relative humidity for conditioning for measuring.

16) Preconditioning times of 1 h to 2 h are recommended for photographic film, 4 h for non-resin coated paper, and 7 d for resin coated paper.

Annex A

(informative)

Methods to measure dimensional change

Many different techniques are available today to measure the size change of photographic films and papers. The most suitable method depends upon the accuracy required, the dimensions of the sensitized material available for measurement, the relative importance of measuring the physical dimensions of the material or the displacement of the photographic image, whether dimensions shall be measured simultaneously in different directions, and the need to obtain a measure of size change uniformity. Several well-established approaches are briefly reviewed below.

One widely-used technique is the pin gauge method first described by Davis and Stovall [1]. With this technique, the photographic material is punched with two pairs of perforations which are a known distance apart. These perforations fit over corresponding pairs of pins in a mechanical gauge, and the distance between the pins is read on a dial indicator. Repeatability of $\pm 0,006\%$ can be obtained. Depending on gauge tension, this equipment may be less satisfactory with some thin photographic papers and thin base films since the perforations may not have sufficient strength to resist distortion. This procedure gives a dimensional change averaged over the gauge length, and localized areas of non-uniform behaviour will not be detected. Moreover, values are determined only in one direction of the test specimen and appropriate specimens shall be cut for each direction of interest. Within these limitations, this technique has proved to be extremely useful [2].

A variation of this method mounts the specimens on register pins, one of which is fixed at the top of an inclined plane while the other register pin is mounted on a steel block containing a dial gauge.

The block rests on bearings and is free to move up and down on the inclined plane [3].

Another useful device, which also measures the dimensions of strips, is an electronic gauge [4] using a strain gauge. Again, this method is limited to one dimension and a spring-loaded probe tends to deflect base materials.

Dimensional changes in several directions of photographic film have been simultaneously studied by exposing a réseau or grid on the film and subsequently comparing the intersections of this grid against those of the original master grid (on a glass plate) using an optical comparator. This approach can yield very precise values [5] [6] [7] and can be made on relatively large sheet sizes. However, the measurements are very laborious. A more sophisticated variation of this technique measures X and Y coordinates of a rectangular grid pattern [8]. A computer program converts these coordinates into more meaningful data.

Another optical method on large sheet sizes involves the principle of moiré interference patterns [9] to [12]. A fine half-tone chequered pattern on glass is exposed on the film and registration is subsequently made with the glass master of the chequered pattern. The resulting moiré patterns can be used to calculate absolute size change and also to illustrate readily any nonuniformities in size changes. Both this moiré technique and the grid-comparator method utilize the photographic image for measurement purposes and hence cannot be used on unprocessed materials.

Both grid and the moiré method also provide information on the uniformity of dimensional change.

Annex B (informative)

Dimensional hysteresis in photographic materials

Careful measurement of the dimensional properties of photographic materials, particularly those coated on low-shrink, moisture-resistant polyester photographic film base and on paper base, shall take into account the phenomenon of dimensional hysteresis. This is the failure of the material to reach the same dimensions when its equilibrium moisture content is approached from a lower or from a higher relative humidity.

Hydrophilic or moisture absorbing materials exhibit a moisture hysteresis and have a higher moisture content when equilibrium is approached from a higher relative humidity than when it is approached from a lower relative humidity. Paper, cellulose ester, and gelatin are typical of such materials. However, dimensional hysteresis in photographic materials is further complicated because mechanical factors, in addition to moisture absorption, are involved.

Dimensional hysteresis curves are shown in figure B.1 for an unprocessed cellulose ester base film. Figure B.2 shows the dimensional hysteresis for both unprocessed and processed film coated on hydrophobic polyester film base. Figure B.3 illustrates such curves for a typical photographic paper base. These curves illustrate the dimensional changes that occur with cycling relative humidity although there can be considerable variation in behaviour for different materials. The unprocessed cellulose ester base film illustrated in figure B.1 shows the normal type of hysteresis resulting from higher moisture content during desorption than during absorption. Film coated on the polyester photographic film base (see figure B.2) on the other hand, shows "hysteresis" but in the reversed direction; that is, the film has a smaller dimension when the relative humidity is approached from above. The reversed hysteresis observed here is due to mechanical and rheological effects related to the

gelatin-base interactions [13]. Thus, two competing forces may be present to cause dimensional hysteresis in photographic films, namely, normal moisture hysteresis in the base and a reversed hysteresis due to the mechanical effects of the gelatin. In fact, some types of cellulose ester base film also exhibit reversed hysteresis. In other words, the type of base and emulsion and the base-emulsion thickness ratio determine the dimensional hysteresis characteristics.

For photographic papers, hysteresis curves are complicated by the stretching which paper was given during the paper-making operation. As the humidity decreases from a high humidity condition, the machine direction follows a lower curve than it followed with increasing humidity due to release of dried-in strains. This strain relaxation results in permanent shrinkage. However, in the cross direction, the paper shows normal hysteresis behaviour and follows a desorption curve of greater dimensions.

It is apparent from the above that the dimensional properties can be accurately determined only by the most careful control of the sample history and the rigid specification of test conditions. In clause 6, measurement is specified over a range below 60 % RH because the curves sometimes show an inflection point at high humidity. In clause 8, different procedures may be used to take into account different combinations of moisture history in the raw and processed material that may occur in practice. This is relatively more important in the more stable materials coated on polyester photographic film base. Figure B.2 illustrates the variation in dimensional changes that can be caused by previous moisture history, even when the film is returned to the same relative humidity at the time of printing as existed at the time of camera exposure.

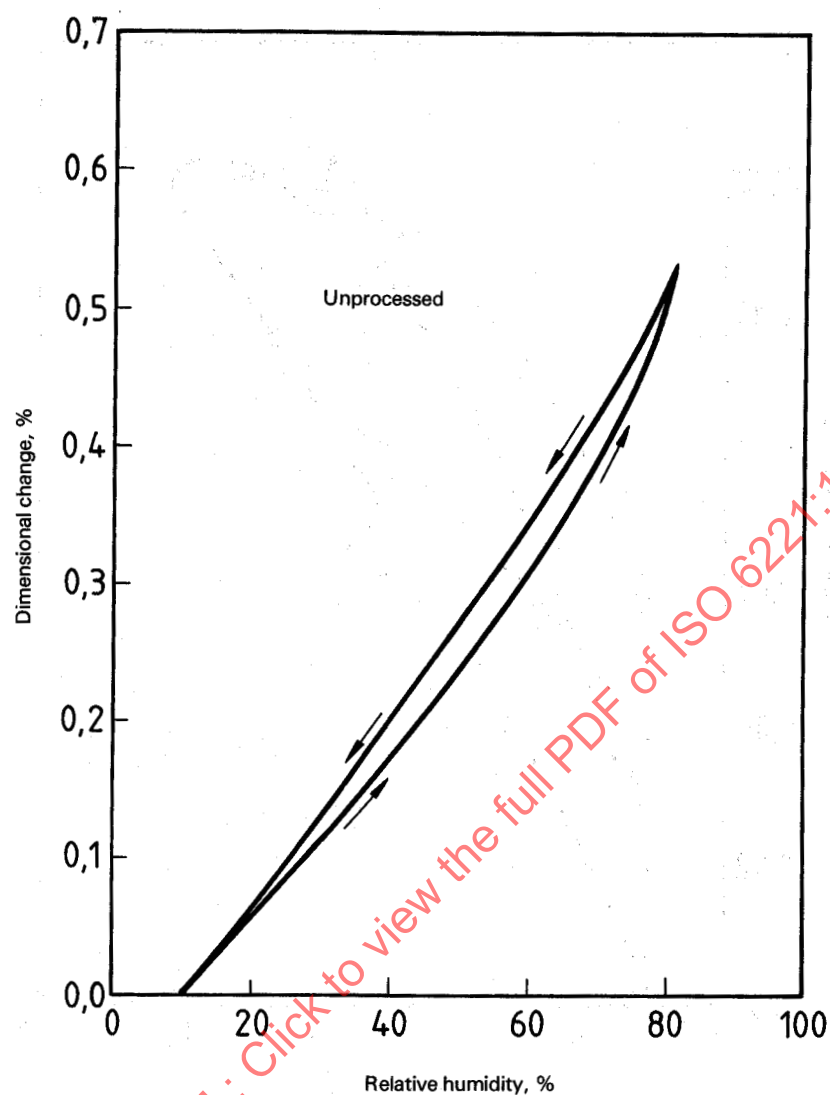


Figure B.1 — Dimensional hysteresis curve for a typical film (gelatin emulsion and backing) coated on cellulose ester base

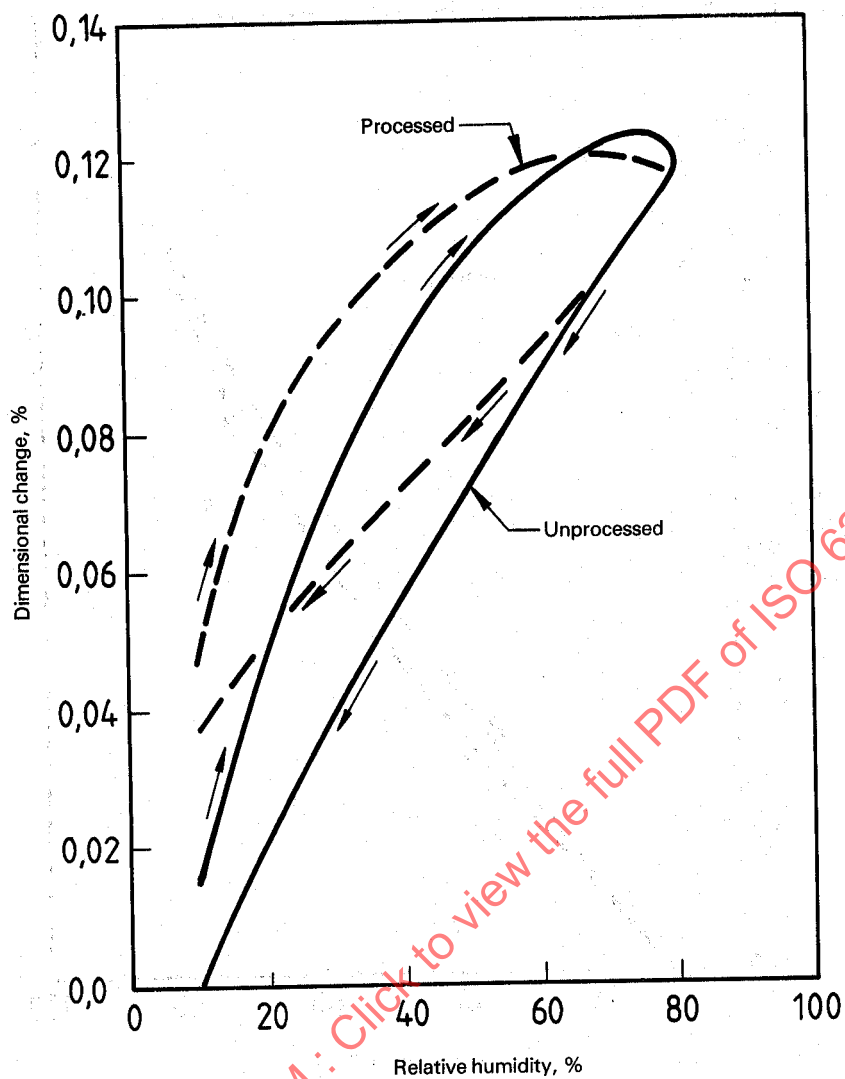


Figure B.2 — Dimensional hysteresis curves for a typical film (gelatin emulsion and backing) coated on polyester base

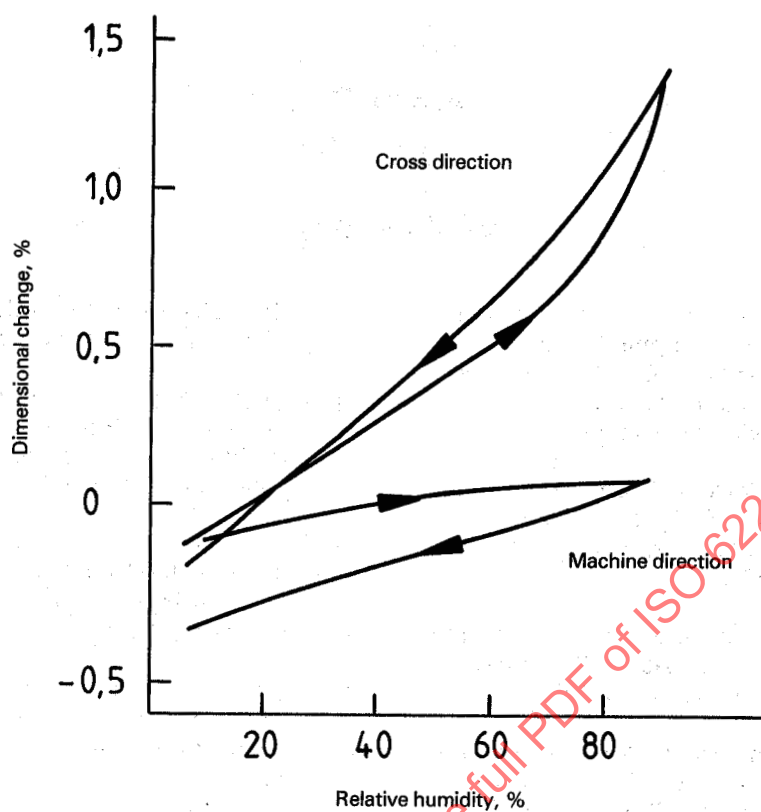


Figure B.3 — Dimensional hysteresis curves for a typical photographic paper base

Annex C (informative)

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