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Imaging materials — Test method for Arrhenius-type predictions

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National foreword

This British Standard is the UK implementation of ISO 18924:2013. It supersedes BS ISO 18924:2000, which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee CPW/42, Photography.

A list of organizations represented on this committee can be obtained on request to its secretary.

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Imaging materials — Test method for Arrhenius-type predictions

Matériaux d'image — Méthode d'essai pour les prédictions de type Arrhenius

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Foreword

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The main task of technical committees is to prepare International Standards. 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 document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 18924 was prepared by Technical Committee ISO/TC 42, *Photography*.

This second edition cancels and replaces the first edition (ISO 18924:2000), of which it constitutes a minor revision with the following changes:

— Clause 2 has been removed;

— Annex A has been removed.

Imaging materials — Test method for Arrhenius-type predictions

1 Scope

This International Standard specifies a test method for the prediction of certain physical or chemical property changes of imaging materials.

This International Standard is applicable to the Arrhenius test portion of ISO 18901, ISO 18905, ISO 18909, ISO 18912, and ISO 18919.

This International Standard is applicable to the prediction of the optical-density (D) loss or gain of imaging materials. Photographic dye images may be produced by chromogenic processing, by formation of diazo dyes, or by non-chromogenic methods such as dye diffusion and silver dye-bleaching processing. This International Standard also covers density changes caused by

— residual coupler changes in dye images,

— excess residual processing chemicals in silver black-and-white materials,

— temperature effects on thermally processed silver images.

This International Standard is applicable to the prediction of support degradation. One such example is the generation of acetic acid by degradation of cellulose acetate film support. Another example is the change in tensile energy absorption of black-and-white paper support.

2 Terms and definitions

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

2.1
Arrhenius plot
plot of the logarithm of the time for a given change in a characteristic proportional to the reaction rate (dye loss, tensile strength change, D_{min} yellowing, etc.) versus the reciprocal of the temperature, in kelvins

Note 1 to entry: The Arrhenius plot may be used to predict behaviour at a temperature lower than those at which the tests are run.

2.2
glass transition
reversible change in an amorphous polymer from, or to, a viscous or rubbery condition to, or from, a hard and relatively brittle one

2.3
glass transition temperature
$\boldsymbol{T_{\mathrm{g}}}$
approximate mid-point of the temperature range over which glass transition takes place

Note 1 to entry: T_{g} can be determined readily only by observing the temperature at which a significant change takes place in a specific electrical, mechanical, or other physical property.[1]

Note 2 to entry: T_{g} can also be sensitive to the moisture content of the polymer (see 4.4, Annex A, and B.3 of Annex B for information).

Note 3 to entry: For imaging materials containing gelatin, T_{g} is very humidity dependent.

2.4
irrelevant physical or chemical reactions

chemical or physical reactions that take place only at high temperatures and/or humidities and do not take place at the temperatures at which the Arrhenius predictions are to be made

Note 1 to entry: Such reactions may nevertheless affect the quality of the image, binder, or support.

2.5
morphological changes

changes in the physical structure of the association of the molecules

2.6
thermodynamic temperature

temperature measured on the absolute scale which is based on absolute zero (–273,15 °C) and having an interval of measurement that is equivalent to degrees Celsius

Note 1 to entry: The temperature unit in the absolute scale is the kelvin.

3 Background and theory

3.1 Background

In the 1890s, Svante Arrhenius discovered that the rate of some chemical reactions is proportional to the reciprocal of the absolute temperature. This relationship has been used with phenomena related to a chemical change, such as the loss of a particular physical property or the change in the optical density of film. If a linear relationship exists between the logarithm of the time for a change of a particular property and the reciprocal of the temperature, then this plot can be extrapolated to lower temperatures than those used in laboratory studies. This allows the prediction of the time required for the change to happen at room temperature or lower.

This relationship was first used for the rates of chemical reactions[2] and was later applied to paper materials.[3,4] This theory became the basis for TAPPI Standard 453.[5] The approach was also applied to textiles[6] and to physical properties of photographic film supports.[7,8] More recently, it has been used to predict the fading of both chromogenic and non-chromogenic photographic dyes.[9-11]

Predictions based on the Arrhenius equation require the reactions to be run under a series of temperatures at either constant relative humidity (free-hanging) or constant moisture content inside the enclosure. The investigator shall determine which of the above conditions is more relevant to the system being tested.

There may also be cases where elevated temperatures cause different reaction pathways from those occurring at ambient or sub-ambient conditions. In these cases, the plot of the logarithm of time versus the reciprocal of the absolute temperature will be nonlinear and great caution shall be taken in drawing conclusions. Only the linear and lower temperature portion of the plot can be extrapolated to ambient conditions or below.

The drawback to elimination of higher temperature data is that the experiment will then take longer because of the slow reaction rate at lower temperatures. Patience is the only solution for getting the correct answer when this happens. When incubations are limited to a few of the higher temperatures, this can lead to incorrect or misleading results and shall be done with extreme caution.

Confidence in the Arrhenius methodology is obtained when predictions for phenomena with reasonably short lifetimes correspond to the real-time results. Such data do exist for the fading of photographic dyes[12,13] and the stability of cellulose ester film supports.[8,14]

3.2 Theory

The basic relationship in the study of chemical reaction rates is the Arrhenius equation:

$$\log k = \frac{-E}{2{,}30\ RT} + C \tag{1}$$

where

k is the rate of reaction (change per time);

E is the energy of activation for a specific reaction;

R is the gas constant;

T is the temperature (in kelvins);

C is a constant for the specific reaction.

By combining all the constant terms ($E/2{,}30\ R$) into a constant “a” and measuring the time for a given change, this equation can be rewritten as:

$$\log\,(\text{time}) = \frac{a}{T} + C \tag{2}$$

Consequently, when the logarithm of the time is plotted against the reciprocal of the absolute temperature, a straight line is produced. This relationship can be used to predict the time required for a given change to occur at lower temperatures where the reaction might require hundreds of years. This is done experimentally by determining the time required for a given change at a number of elevated temperatures (where the times required are reasonable), plotting these points, and extending the straight-line graph to the lower temperatures of interest. This “Arrhenius method” of predicting long-term ageing behaviour is widely used and accepted by experts in the photographic industry.

In the chemical literature, the equation has been widely applied to relatively simple, chemical reactions where both reactants and products have been identified. However, there may be circumstances in which the fit of the Arrhenius prediction line is less than perfect. In these cases, there may be more than one reaction occurring and this may result in nonlinear behaviour or two distinct linear portions to the prediction line. In other situations, physical properties are measured, although the changes are the result of chemical reactions. More details of these phenomena are given for information in Annex B. However, despite the complex reactions involved, this equation applies very well to many complicated reactions that occur with photographic materials.

3.3 Effects of relative humidity

The Arrhenius method is run at either constant relative humidity or constant moisture content in the enclosure. It should be noted that many of the responses evaluated by the Arrhenius method are humidity dependent and that rates can change quite drastically as a function of relative humidity.[15]

The effect of moisture may be determined by several separate experiments at multiple temperatures, with each experiment at a constant relative humidity or moisture content.

4 Experimental procedures

4.1 Outline of Arrhenius test

An Arrhenius test should have the following steps that are explained in more detail in several of the references.[16,17]

a) Prepare specimens; this may include exposing, processing, cutting, trimming, etc.

b) Take initial readings of the property of interest on the non-incubated specimens.

c) Incubate the specimens at a minimum of four temperatures, using either the free-hanging or the sealed-bag technique (see 4.3).

d) Measure the property of interest on the incubated specimens after different incubation times.

e) Determine the incubation time at each incubation temperature for the property of interest to reach a predetermined level.

f) Plot the log of the incubation time determined in e) against the reciprocal of the thermodynamic temperature to obtain an Arrhenius plot.

g) Predict the time for the property of interest to change the desired amount at the desired temperature by extrapolation of the Arrhenius plot.

h) Examples of Arrhenius plots are given in Annex C.

4.2 Requirements for a meaningful Arrhenius test

Although a straight line can be drawn between two points and an Arrhenius prediction may be made by plotting the results of two different incubation temperatures, there can be no evaluation of the statistical significance of this experiment unless three or more temperatures are used. Because a smaller number of data points is apt to lead to a strongly biased prediction, a minimum of four temperatures shall be run for each prediction.

If the effect of relative humidity needs to be considered, experiments at different relative humidities shall be studied. The relative humidities shall be at least 10 % RH apart and preferably should be 20 % RH apart. The tests shall be run at a humidity range representing the anticipated storage of the material.

4.3 Sealed-bag versus free-hanging testing

Two test methods, known as the “sealed-bag” and the “free-hanging” methods, are available for accelerated stability testing. These kinds of test conditions tend to give somewhat different results.

In the sealed-bag method, the photographic material is stored in a sealed container with very little air. Pre-equilibration of the samples to a constant relative humidity is necessary before they are sealed. This test simulates real-life storage conditions in which substances released by the photographic material are trapped inside the container and can interact with the image or support layers. For example, with cellulose-acetate-base motion picture films stored in sealed bags or closed metal or plastic cans, the fading of the dye images may be accelerated by the presence of acetic acid generated by the degradation of the acetate film base.

In the free-hanging method, the specimens are suspended in a relatively large humidity-controlled test chamber at a sufficient distance from each other to ensure free access of circulating air to all surfaces. This test would be appropriate to simulate storage conditions in which photographic materials are stored in vented containers and circulating air is used to remove any released substances.

The advantages and disadvantages of each method are discussed in informative Annex A.

4.4 Effect of heating on sealed bags containing photographic film or paper

If photographic paper or film containing significant amounts of gelatin is incubated in a sealed foil bag, the relative humidity (RH) of the environment will increase with increasing temperature, even though the moisture content of the overall system remains constant. In some systems, this increase of humidity can also cause physical changes such as the transformation of the gelatin conformation to the above T_g state (see 2.3).

Experimental evidence has shown that this increase of relative humidity results in more staining on paper and, in some cases, more dye fading in colour photographs. For example, if a processed black-and-white paper is incubated in the free-hanging mode, thermal yellowing will be considerably less than if it is incubated in a sealed foil bag. Thus, in order to establish the same environmental conditions for both the sealed bag and free-hanging test methods, one shall choose pre-equilibration RH levels for the bagged samples which compensate for the RH increase that occurs at the elevated test temperature.

The amount of RH compensation required may be sample dependent, since different materials can have different moisture-sinking properties.[15] From a practical standpoint, the determination of correct RH values for sealed-bag incubations may require a few trial runs where RH measurements are taken inside bags containing representative materials prior to commencement of the actual tests.

4.5 Determination of test increments

If the free-hanging method is used, time increments of treatment can be accumulated upon the same samples and they can be returned to the oven until the failure point has been reached. However, foil-bag testing requires that the increments be determined in advance, since all the bags are pre-equilibrated and prepared at the same time.

It is strongly recommended that the initial increments for foil-bag testing be somewhat conservative so as not to overshoot the failure point, and that subsequent increments be adjusted once data have been obtained on the first few increments. Adjusting the schedule in this way increases the probability that the failure point is neither underreached nor overreached.

5 Calculations

Figure 1 illustrates the dye density loss of a generic yellow dye, at a starting optical density of 1,0, incubated at 40 % RH. The top plot shows the rate of fade curves for each incubation temperature. The time at which a 10 % loss of density occurs is translated down to the lower plot where the log of this time is plotted versus the reciprocal of the temperature for that particular incubation. The prediction line connecting these latter data points is then extrapolated to the desired keeping temperature to determine the length of time required for the 10 % blue density loss to occur. In this example, it is approximately 30 years at 24 °C.[9]

6 Test report

The following shall be reported:

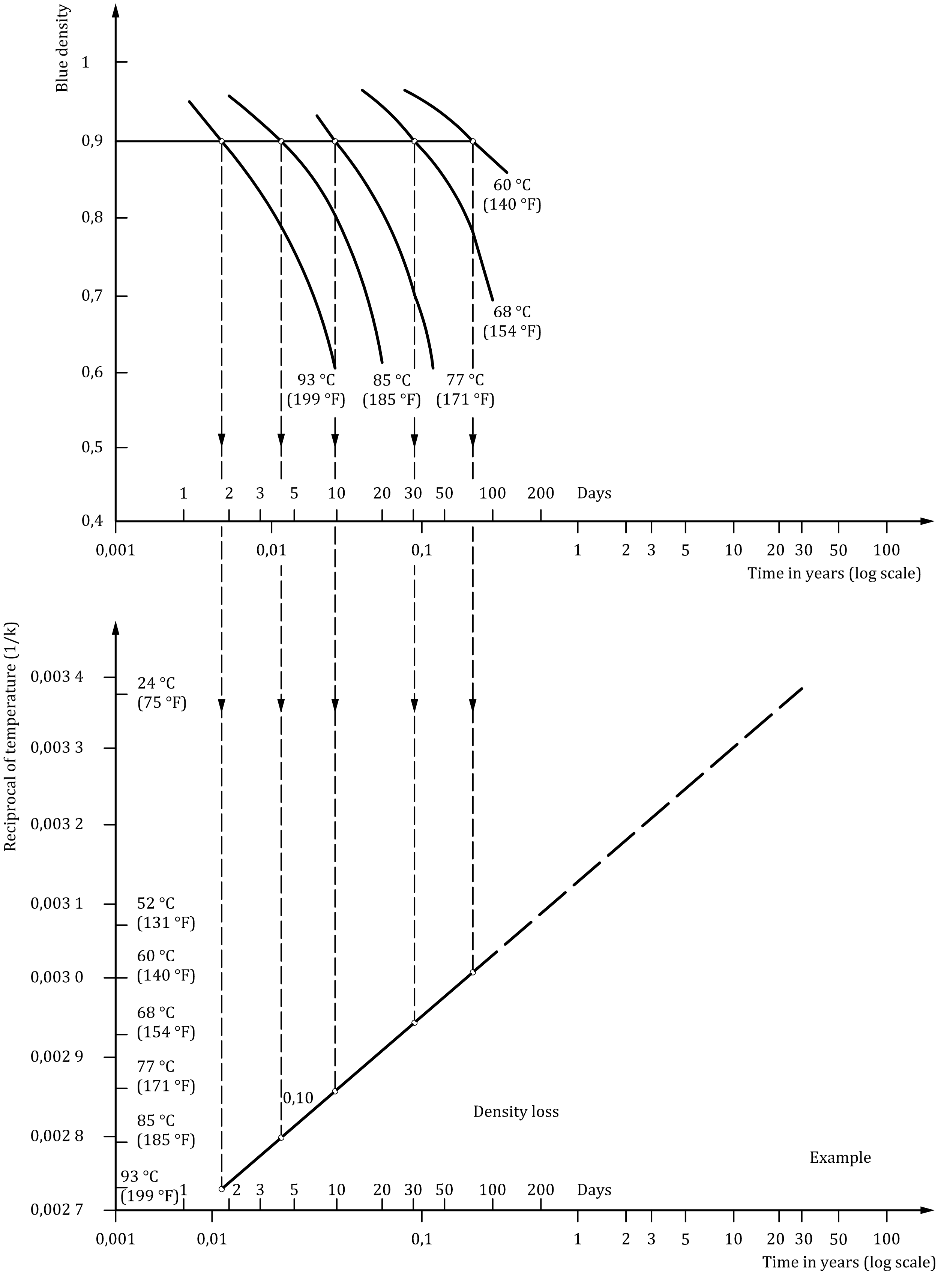
a) property being measured, along with its starting value (e.g. colour of the patch, dye density loss, density 1,0 above D_{min}, etc.);

b) percent of property change which determines the end-point;

c) whether the incubation was free hanging or was in a sealed container (foil bag, film can, etc.);

d) range of temperatures and the specific RHs at which the test was run;

e) extrapolated time for the end-point to occur at a specified temperature and relative humidity.


Blue density
1
0,9
0,8
0,7
0,6
0,5
0,4
60 °C
(140 °F)
68 °C
(154 °F)
93 °C
(199 °F)
85 °C
(185 °F)
77 °C
(171 °F)
1
2
3
5
10
20
30
50
100
200
Days
0,001
0,01
0,1
1
2
3
5
10
20
30
50
100
Time in years (log scale)
Reciprocal of temperature (1/k)
0,003 4
0,003 3
0,003 2
0,003 1
0,003 0
0,002 9
0,002 8
0,002 7
24 °C
(75 °F)
52 °C
(131 °F)
60 °C
(140 °F)
68 °C
(154 °F)
77 °C
(171 °F)
85 °C
(185 °F)
93 °C
(199 °F)
0,10
Density loss
Example
1
2
3
5
10
20
30
50
100
200
Days
0,001
0,01
0,1
1
2
3
5
10
20
30
50
100
Time in years (log scale)

X time, in years (log scale)

Y blue density

Y′ reciprocal of temperature (1/k)

NOTE See Reference[9].

Figure 1 — Illustration of a prediction method (blue density change for 1,0 yellow dye)

Annex A
(informative)

Advantages and disadvantages of sealed-bag and free-hanging incubations

The choice of test method should be based on the known properties of the photographic materials being evaluated and the expected storage conditions of these materials. For example, motion-picture films are normally stored in closed metal or plastic cans and, therefore, should be evaluated with the “sealed-bag” method. This method also provides better simulation of the storage environment of still-camera colour films placed into plastic sleeves in strip or roll form. However, the “free-hanging” method is usually more representative of the keeping conditions for mounted 35-mm colour slides and reflection prints.

The “free-hanging” method is an incubation at constant relative humidity. It provides an essentially unlimited supply of the required oxygen and moisture to fuel chemical reactions. Multiple time intervals can be accumulated on the same samples in a free-hanging mode, providing there are no physical changes that affect the results. This reduces the amount of material needed for testing. However, the environmental chambers required for free-hanging incubations are expensive because of the need to accurately control both temperature and relative humidity.

The “sealed-bag” method is used for constant moisture content of the system, but the relative humidity within the bag may be different at different incubation temperatures. Oxygen and moisture may become depleted components in this form of incubation. Because the relative humidity inside the bag may increase with an increase in incubation temperature, the sealed-bag method is more likely to cause some of the incubations to be above the glass transition temperature T_g (especially in the case of gelatin) while others are below it.[15] This may produce Arrhenius lines with greater variability or with distinct breaks in the prediction lines. More specimens are required with the sealed-bag method, but the incubation chambers need to control only temperature and are therefore less expensive.

Annex B
(informative)

Limitations of the Arrhenius method

NOTE See Reference [16].

B.1 Physical changes

The methodology of accelerated testing can lead to physical or chemical changes that misrepresent the degree of change that takes place at moderate temperature. An example of this might be a dye-fading reaction that is complicated by a morphological change in the dye clouds (e.g. spreading out of the clumps of dye that form during photographic development) and is therefore accompanied by an increase in covering power. One indication of this behaviour is a dye-density increase for the initial times followed by a decrease in density as the dye starts to fade.[17] The difficulty here is that, since two phenomena are working against each other, the net result at early stages of the incubation may not be an increase in density. In these cases, a way to determine that covering power changes are not happening is through the use of microscopic examination before and after incubation to see if the dye clouds have spread. An alternative method is to measure the remaining dye concentration by an analytical chemical method (such as liquid chromatography) after incubation. A plot of density versus dye concentration should be linear if morphological changes are not happening.

This last statement is not precisely true for reflection density, but is a close enough approximation that it should still be applicable to check for covering power changes.

B.2 Irrelevant physical or chemical reactions

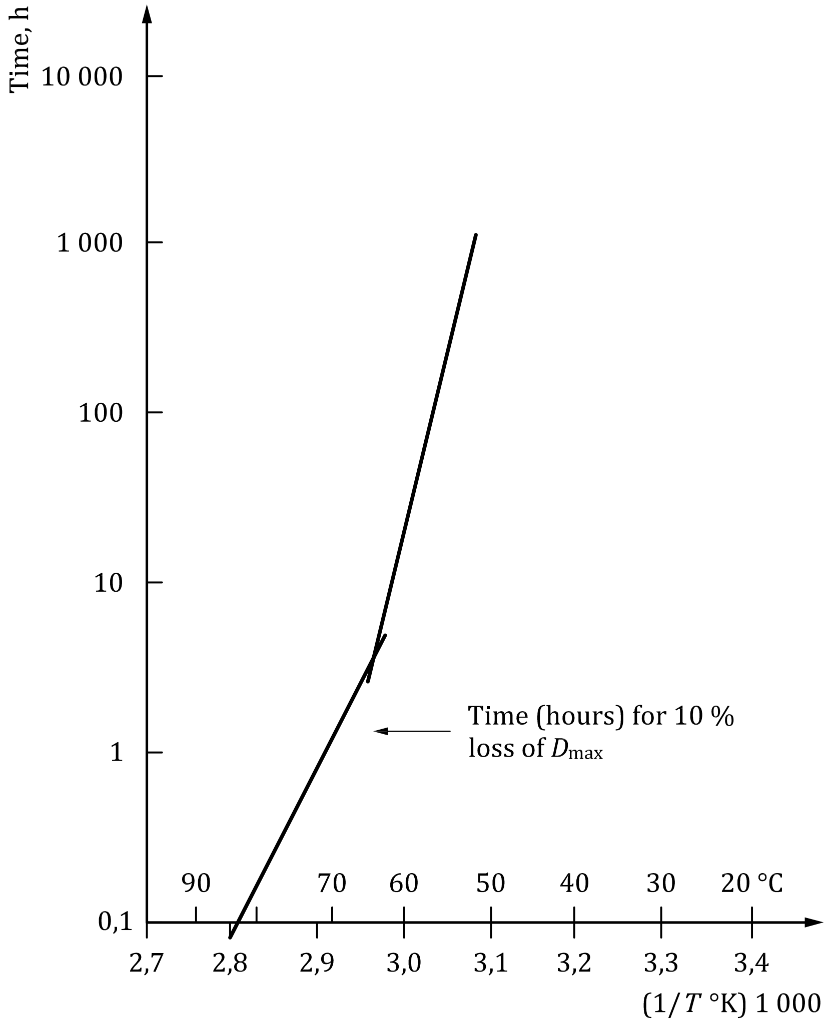
An Arrhenius test is intended to measure the product of a single chemical reaction. If other physical or chemical reactions occur at high temperatures or high humidities, the Arrhenius plot may either have a nonlinear high-temperature end or there will be a discontinuity of slope with two separate Arrhenius lines, one where the high-temperature reaction predominates and one where the low-temperature reaction predominates (see Figure B.1).

In extreme cases of dye-fading reactions, the minimum density of a film containing coloured masking couplers can show a loss of density at some temperatures and an increase in density at others. A similar phenomenon occurs when the initial incubations show a loss of density followed by gradual build-up of stain.

B.3 Exceeding the T_g of the binder (see 2.3)

Figure B.1 illustrates the density loss of a vesicular film.[20] In this graph, the higher temperatures indicate a different Arrhenius line than the one for the lower temperatures. Different reaction rates occur when the T_g of the binder has been exceeded. Use of these higher temperatures in the Arrhenius analysis will lead to incorrect results.

Studies of emulsion properties using the sealed-bag technique can create situations where the T_g of the gelatin binder can be exceeded at elevated temperatures. It has been observed that the equilibrium relative humidity in a sealed bag increases with increasing temperature.[15] At higher temperatures, the T_g of the gelatin will be exceeded.



X (1/*T* K) 1 000

Y time (hours)

[a] Time (hours) for 10 % loss of D_{max}.

NOTE See Reference [20].

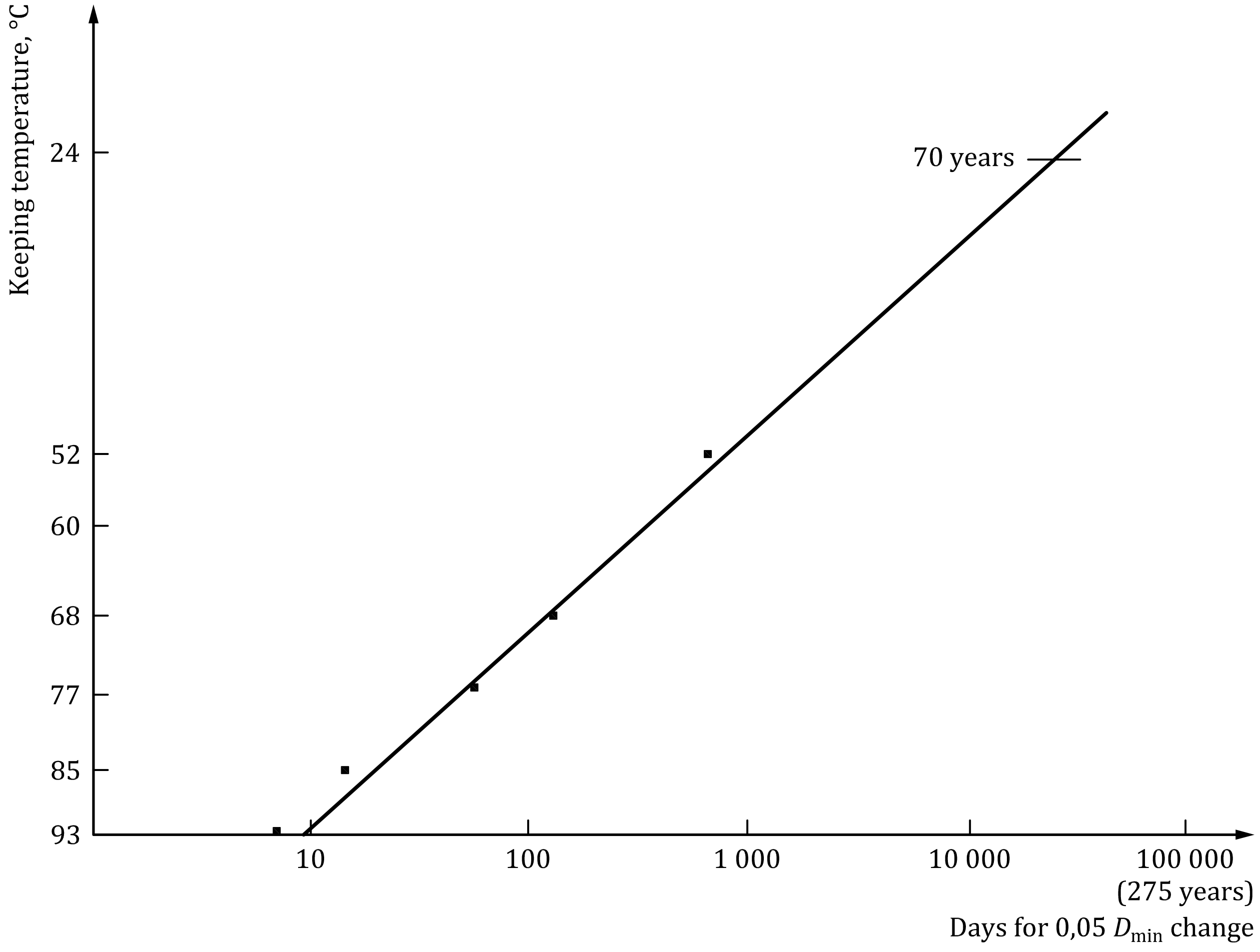
Figure B.1 — Arrhenius plot of D_{max} loss of a vesicular film

Annex C
(informative)

Examples of Arrhenius relationships

Figure C.1 illustrates the Arrhenius prediction line for residual thiosulfate ions and shows the time for the blue D_{min} to rise 0,05 density units for a radiographic film.[18]

Figure C.2 shows the prediction line for the loss of 20 % of the intrinsic viscosity of degraded cellulose triacetate and polyester film supports.[19] This is a case where the higher temperature data rank the two supports opposite to that predicted by the Arrhenius lines at room temperature because the lines cross at about 80 °C.



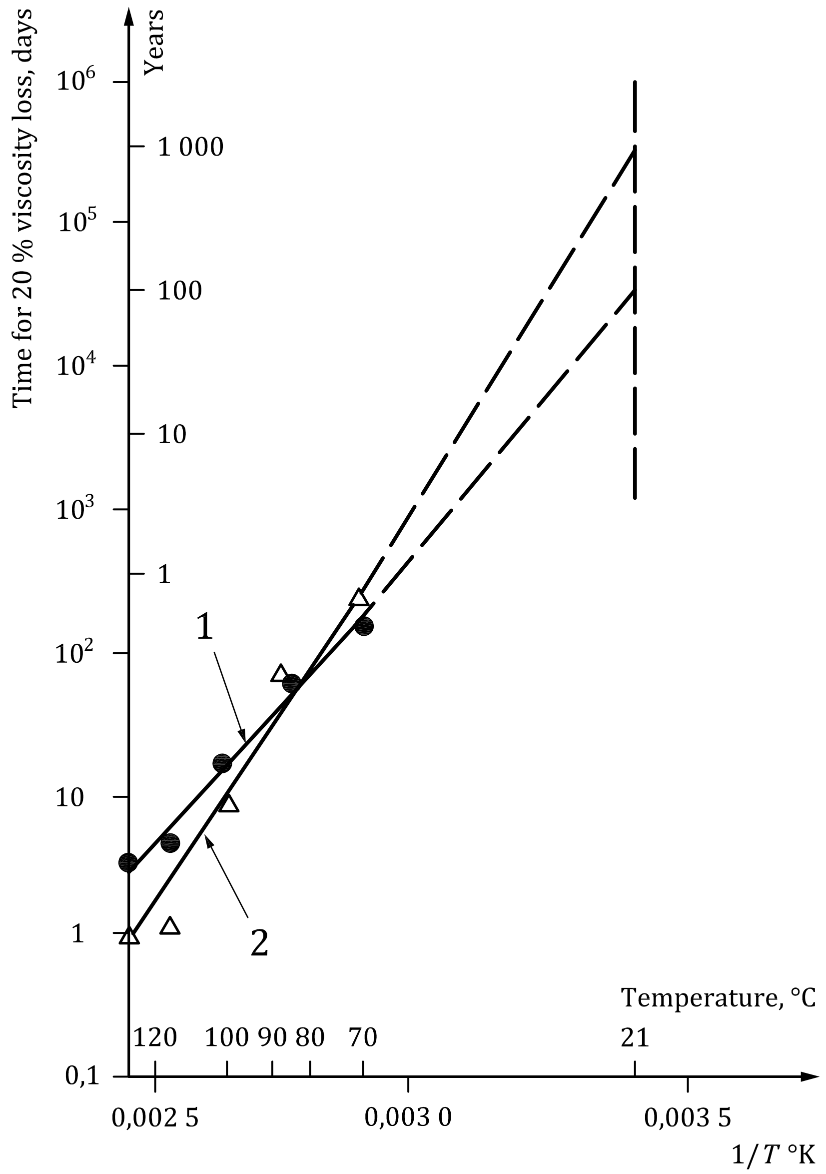
X days for 0,05 D_{min} change

Y keeping temperature (°C)

[a] 70 years.

NOTE See Reference [18].

Figure C.1 — Days for 0,05 D_{min} change versus temperature for radiographic film



Key

X	1/*T* K
Y	time for 20% viscosity loss (days)
1	cellulose triacetate-base film
2	polyester-base film

NOTE See Reference [19].

Figure C.2 — Arrhenius plot of time for 20 % intrinsic viscosity loss of two film supports

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