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Conservation of Cultural Heritage — Test methods — Determination of drying properties

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CEN-CENELEC Management Centre: Avenue Marnix 17, B-1000 Brussels

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Foreword

This document (EN 16322:2013) has been prepared by Technical Committee CEN/TC 346 “Conservation of Cultural Heritage”, the secretariat of which is held by UNI.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by April 2014, and conflicting national standards shall be withdrawn at the latest by April 2014.

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Introduction

This test method can be applied if it does not change the value of the cultural property according to the ethical code of conservation practice.

The drying properties of materials can be calculated from a curve that indicates the weight loss of the mass of water inside the sample, as a function of time, during a drying experiment. Usually the drying of specimens saturated with water consists of two phases.

The first drying phase is characterised by transport of liquid water to the surface followed by evaporation. The surface remains wet allowing evaporation at a constant rate, as water moves to the surface fast enough to compensate for the losses due to evaporation. The evaporation at the surface is determined to a large extent by the test boundary conditions. These are temperature, relative humidity and the flow velocity of the ambient air. The slope of the drying curve during the first drying phase therefore reflects these conditions.

The second drying phase starts when the amount of water brought to the surface becomes too small to keep the surface wetted and the rate of evaporation decreases. Transport of liquid water to the surface is no longer possible and only the less efficient vapour diffusion mechanism remains available.

Some materials, e.g. adobe or sandstones containing clay, do not dry in this typical two-phase drying curve. For example, in the case of material treated with water repellent, the first drying phase does not exist.

1 Scope

This European Standard specifies a method for the determination of the drying behaviour of porous inorganic materials used for and constituting cultural property. The method may be applied to porous inorganic materials either untreated or subjected to any treatment or ageing.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 15898, *Conservation of cultural property - Main general terms and definitions*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 15898 and the following apply.

3.1
porous inorganic material
material including natural stones as sandstone, limestone, marble, and others as well as artificial materials such as mortar, plaster, brick and others

3.2
drying rate
mass of water transported through the specimen per area and time

3.3
drying curve
graphical representation of water loss over time showing in most inorganic porous materials two distinct drying phases

3.4
first drying phase
characterised by transport of liquid water to the surface followed by evaporation

3.5
second drying phase
characterised by a decrease in liquid water transport and an increase in water vapour diffusion limited by hygric material properties

3.6
knick-point of the drying curve
time of transition between the first and the second drying phases shown on the drying curve

3.7
drying index
area under the curve derived by graphical or mathematical methods

4 Principle

Determination of the drying behaviour of porous inorganic materials saturated with water and subjected to drying in a controlled environment.

5 Symbols and abbreviations

m_{max} mass of the saturated sealed specimen, in kg;

m_i mass of the sealed specimen at time t_i, in kg;

m_f final mass of the sealed specimen at time t_f, in kg;

t_i time elapsed from the beginning of the test, in h;

t_k time at which the knick-point is reached in h;

t_f final time of the test, in h;

A area of the drying face, in m^2;

D_1 drying rate corresponding to the first drying phase, in kg/m^2 h;

D_2 drying rate corresponding to the second drying phase, in kg/m^2 $h^{1/2}$;

ID drying index;

M_i residual amount of water of the specimen at time t_i per unit area, in kg/m^2;

ß vapour transfer coefficient.

6 Test equipment

6.1 A chronometer with an accuracy of at least 1 s.

6.2 A ventilated oven which can maintain a temperature of (60 ± 2) °C.

6.3 An analytical balance with an accuracy of at least 0,01 g.

6.4 A linear measuring device (calliper) with an accuracy of at least 0,1 mm.

6.5 Climatic chamber with temperature of (23 ± 1) °C and relative humidity (50 ± 3) %.

6.6 Sand paper with grain size of 82 µm (corresponding to grit number P180 according to the FEPA [1)]) classification).

6.7 Desiccator filled with desiccant such as self-indicating silica gel or other drying agent.

7 Preparation of the specimens

7.1 Number and dimensions of the test specimens

The test specimens shall have a regular shape such as cubes or cylinders. They shall have minimum dimensions on any side of 10 mm. Large samples give greater experimental accuracy.

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1) FEPA – Federation of European Producers of Abrasives

The number and dimensions of specimens are dependent on the heterogeneity of the material. Each series shall consist of at least 3 specimens. In case of anisotropy, each series shall always be tested according to the same orientation, if any. All dimensions should not differ by ± 0,5 mm.

In case of non homogeneous materials such as mortars containing coarse aggregates, the dimensions shall be at least three times (and preferably five times) that of the largest grain size.

In cases where sampling constraints exist the number and dimensions of samples may need to vary from the requirements given above, however every effort should be made to ensure that the minimum requirements for reliability are satisfied.

7.2 Pre-conditioning of the specimens

The test surface shall be flat and wet or dry polished with sand paper (6.6). After polishing, the specimens shall be washed with water, gently brushed with a soft brush and immersed in deionised water for 30 min.

In case of water-sensitive materials, for example gypsum containing materials, only dry polishing and compressed air shall be used. The above procedure does not apply to treated specimens or specimens taken from exposed surfaces.

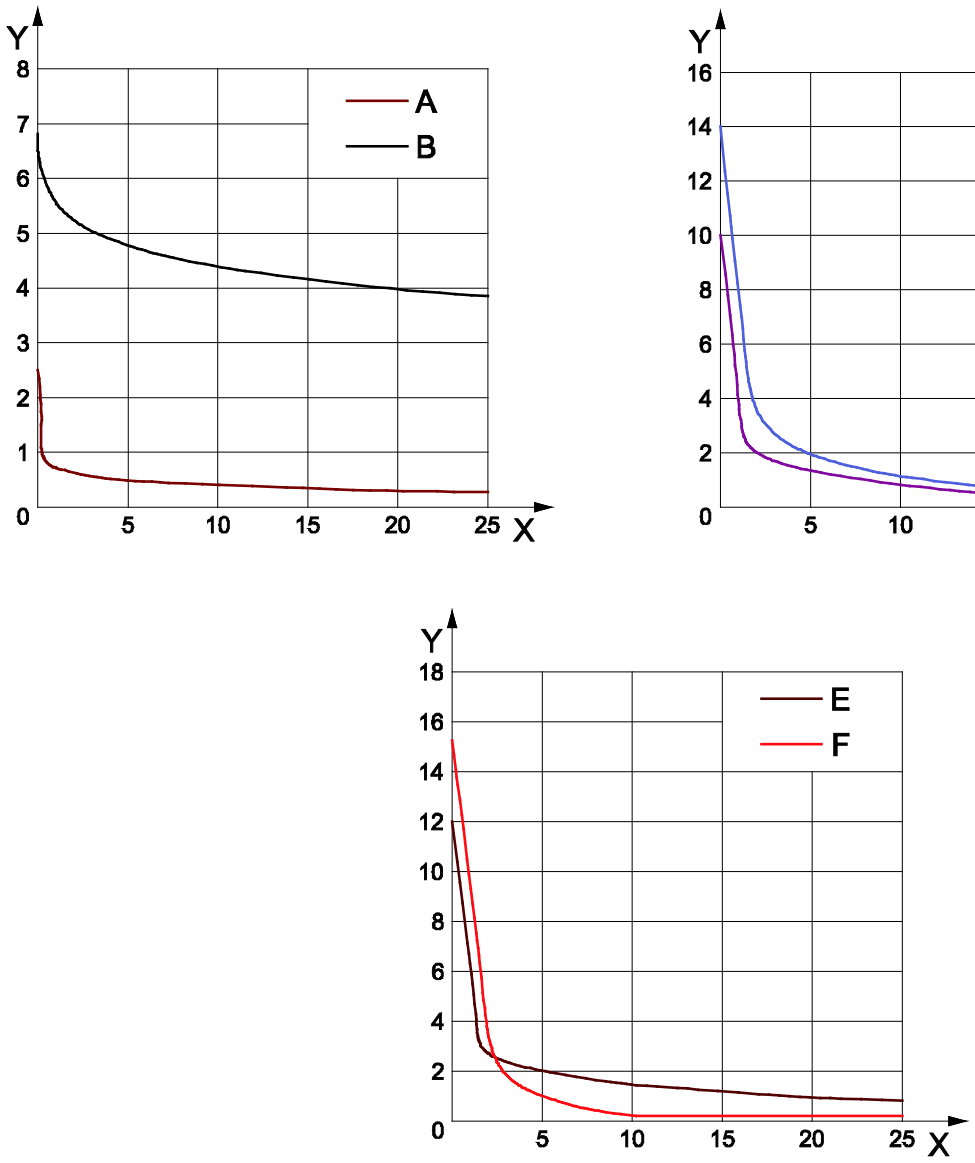
Specimens are saturated with water by capillary rising absorption for 24 h and then total immersion until constant mass in achieved. Constant mass is reached when the difference between two successive weightings at an interval of 24 h is not greater than 0,1 % of mass of the specimen. After this immersion the surface of the specimens is patted dry. All faces, except the test surface, are then sealed with a water impermeable (both in liquid and vapour form) material such as latex, aluminium foil, etc.

8 Test procedure

Specimens prepared according to Clause 7 are placed in a climatic chamber at temperature (23 ± 1) °C and relative humidity (50 ± 3)% in such a way that drying occurs through the upper side. The drying behaviour is recorded by periodic weighing.

As the air flow conditions have significant influence on the drying rate during the first drying phase, these conditions should be kept constant and reproducible. The influence is illustrated in Annex B.

The first weight reading at $t = 0$ is $m_{max.}$ In order to obtain enough data during the first drying phase, the measurement interval at the beginning of the drying shall be chosen in accordance with the specimen height and the materials under investigation. The following figure indicates this influence showing drying curves obtained under standard conditions for different materials.



Key

X time [d]

Y water content [kg/m^2]

A weathered granite

B concrete

C historic lime plaster

D sand stone

E clinker clay brick

F clay brick

Figure 1 — Drying curves for different materials with different length of the first drying phase; calculation results for specimens of 5 cm height dried under standard conditions

The above graphs indicate the following:

— materials with a low liquid conductivity (dense materials, e.g. concrete) generally show a short first drying phase

— materials with a high liquid conductivity (porous materials as clay brick) generally show a distinct and long first drying phase

In addition, a smaller specimen height can lead to a shorter first drying phase.

In the first hour of the experiment procedure a minimum of 5 measurements should be taken at set time intervals. After the first hour, measurements should be taken on an hourly basis for the next 7 h. Subsequent measurements should then be taken twice a day with at least 6 h between two successive weightings. The experiment is carried out until the final mass of the sealed specimen at time t_f (m_f) is reached.

9 Expression of results

9.1 Determination of the drying curve

The residual amount of water present in the specimen per unit area (expressed as kg/m^2) at time t_i is calculated as follows:

$$M_i = \frac{m_i - m_f}{A}$$

The calculated values of M_i are reported as a function of time expressed in h.

9.2 Calculation of the drying rate

9.2.1 Calculation of the drying rate corresponding to the first drying phase D_1

The drying rate corresponding to the first drying phase (see Figure A.1) is the negative slope of the initial linear part of the drying curve and shall be calculated by linear regression, using at least 5 successive aligned points.

9.2.2 Calculation of the drying rate corresponding to the second drying phase D_2

For the determination of the drying rate corresponding to the second drying phase the calculated values of M_i are reported in a graph as a function of the square root of time ($t_i^{1/2}$).

The drying rate corresponding to the second drying phase (see Figure A.2) is the negative slope of the linear section of the drying curve plotted against $t_i^{1/2}$ and shall be calculated by linear regression, using at least 5 successive aligned points.

9.2.3 Determination of the knick-point

While mathematically there are two intersection points between the curves, the knick-point (t_k) corresponds to the greatest of the two possible time values. A worked example of knick-point determination is illustrated in Annex A.

9.3 Calculation of drying index

The drying index is a valuable aid to characterise the drying properties for materials which do not exhibit a clear drying curve according to the mechanism of first and second drying phase.

It can be calculated by using the following formula:

$$ID = \int_0^{tf} \frac{M_i dt}{M_{\max} t_f}$$

The calculated drying index depends strongly on the duration of the experiment (t_f). It is therefore stressed that the drying index on its own is not a useful quantity to characterise the drying properties. The drying index should be used only when comparing drying behaviours, i.e. to compare the drying behaviour of a sample treated with a water repellent agent (measurement carried out during a certain time t_f), compared to the drying behaviour of the untreated sample (measured during exactly the same time t_f).

10 Test report

The test report shall include at least the following information:

a) shape and dimensions of the specimen, in m;

b) number of samples;

c) dimensions of the drying face, in m;

d) experimental data of the decrease in weight (kg) of the specimen over time during drying, expressed in h;

e) experimental data of the decrease in weight (kg) of the specimen over time during drying, expressed in $h^{1/2}$;

f) final time of the tests (t_f);

g) plot of the weight loss (kg/m^2) or the water content as a function of time (h);

h) plot of the weight loss (kg/m^2) as a function of square root of the time ($h^{1/2}$);

i) time at which the knick-point water content is reached, t_k (h);

j) drying rate of the first drying phase, D_1 (kg/m^2h);

k) rate of water vapour diffusion corresponding to the second drying phase, D_2 ($kg/m^2h^{1/2}$);

l) drying index ID (if calculated).

Annex A
(normative)

Numerical example

This test was carried out on a cubic sample of Belgian sandy limestone of side dimensions 5 cm.

Once saturated with water all faces except the test surface were sealed, as described in 7.2 of this standard. Then it was placed in a climatic chamber at 23° C and 50 % RH.

For reasons of clarity, not all measurements are given in Table A.1. The weight of the sample is given at hourly intervals for the first 8 h, and afterwards twice a day. However, all measurements have been taken into account during calculations of regression curves.

The formula used in the table is:

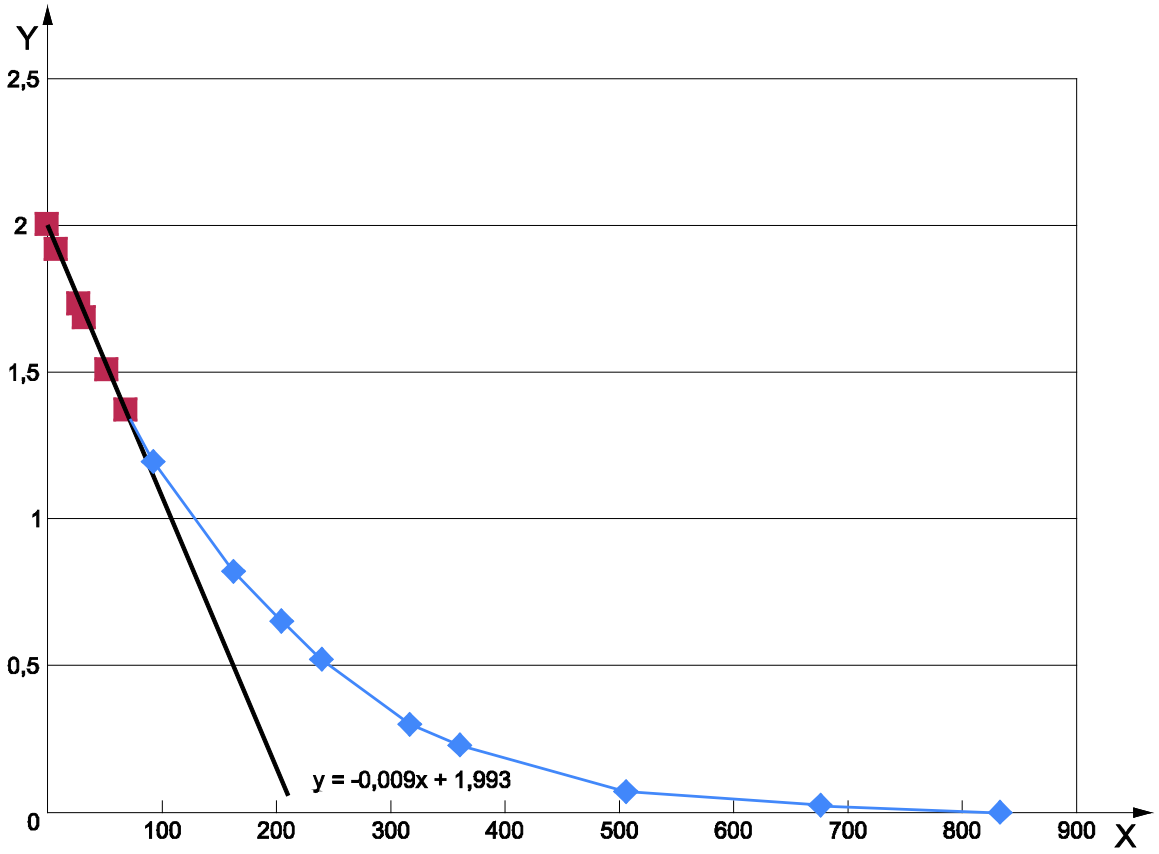
$$M_i = \frac{m_i - m_f}{A}$$

In this example M_i is 0,324984 kg and A is 0,0025 m^2

Table A.1 — Test results

time (h)	m_i (kg)	M_i (kg/m^2)
0	0,330000	2,0064
8	0,329782	1,9192
27,5	0,329318	1,7336
32	0,329202	1,6872
52	0,328765	1,5124
69	0,328408	1,3696
93	0,327969	1,1940
163	0,327034	0,8200
205	0,326611	0,6508
240	0,326284	0,5200
317	0,325731	0,2988
361	0,325552	0,2272
506	0,325158	0,0696
676	0,325042	0,0232
833	0,324984	0

Figure A.1 shows that the first 6 points (between 0 h and 69 h) are lying in a straight line. This corresponds to the first drying phase. The regression line through these 6 points, together with its formula, are included.



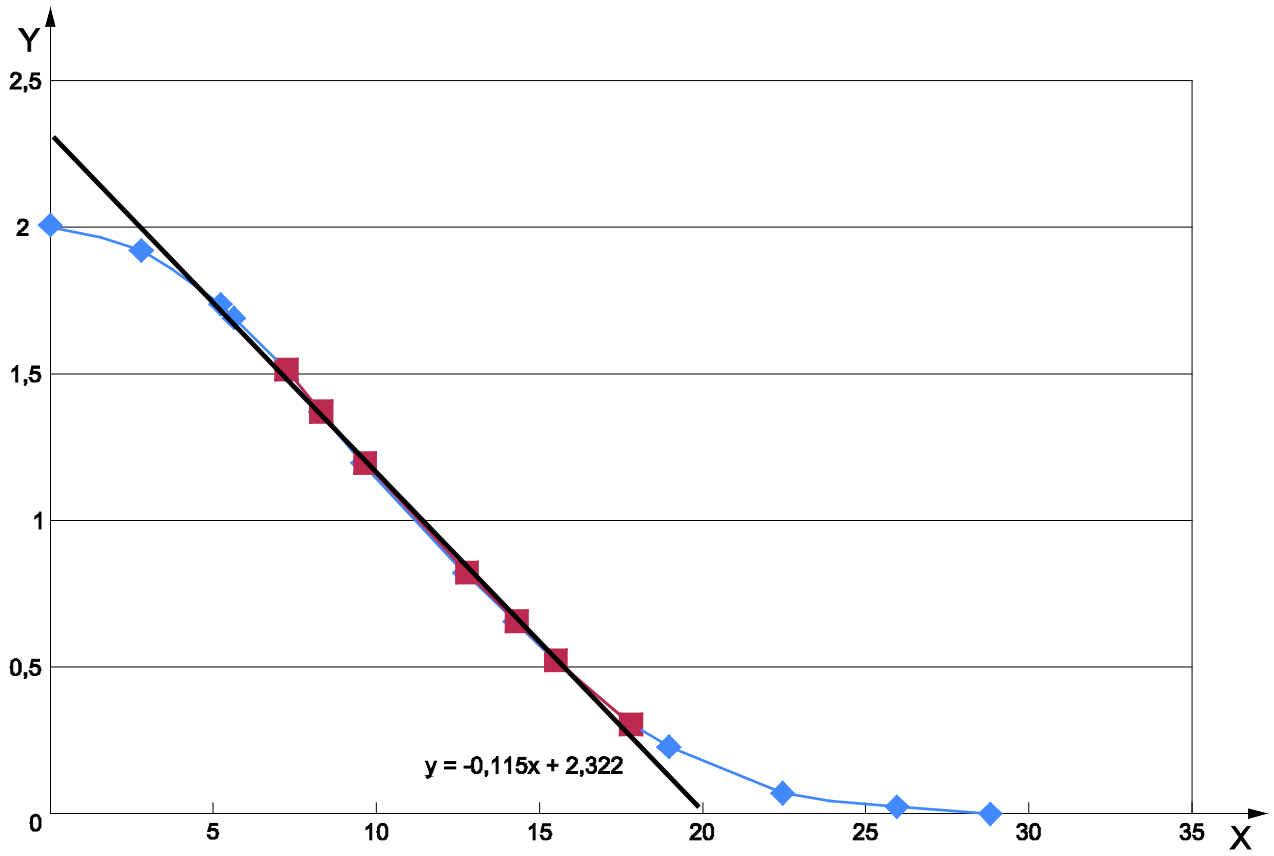
Key

X t (h)

Y M (kg/m^2)

Figure A.1 — The drying rate, corresponding to the first drying phase, equals D_1 = 0,009 kg/m^{2}h

When M is plotted in function of $t^{1/2}$, then a second linear part occurs, corresponding to the second drying phase. Figure A.2 shows this plot, together with the regression line of the linear part.



Key

X $t^{1/2}$ ($h^{1/2}$)

Y M (kg/m^2)

Figure A.2 — The drying rate, corresponding to the second drying phase, is therefore D_2 = 0,115 $kg/m^2h^{1/2}$

Formula of the first drying phase: M = −0,009 t + 1,993

Formula of the second drying phase: M = −0,115 $t^{1/2}$ + 2,322

The knick-point corresponds to the intersection of these two curves. There are two solutions for t: 18,8 h and 67,8 h. The real knick-point corresponds to the highest value, which corresponds in this example to 67,8 h.

It should be emphasised that the knick-point is the intersection of two curves that cross each other at a very sharp angle. This means that errors on the knick-point are relatively high. The knick-point should therefore be considered merely as an indication of the time of the transition between the first and second drying phase, instead of an exact time indication.

The drying index is calculated by calculating the integral under the curve of M in function of t (see 9.3).

This integral equals 343 kg h/m^2.

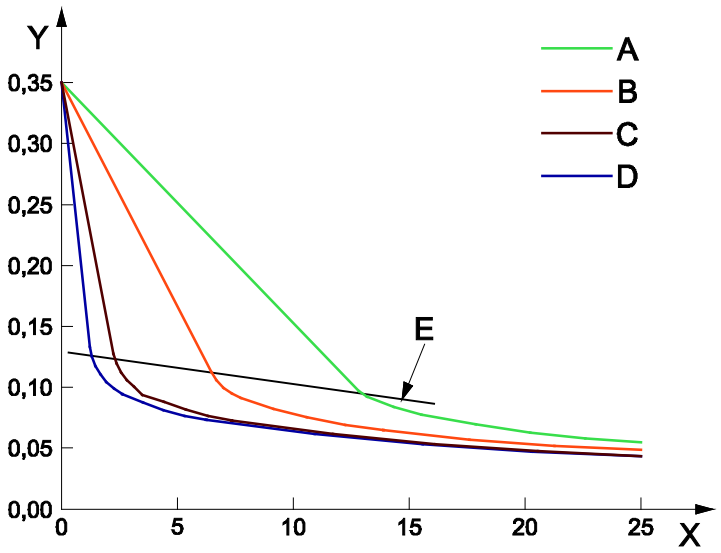
$M_{max}.t_f$ = 2,0064 kg/m^2 833 h = 1671 kg h/m^2.

The drying index is therefore 343/1671 = 0,21.

Annex B
(informative)

Influence of ventilation on the drying curve

The following graph shows drying curves of a clay brick. The data were obtained under the same drying conditions by varying only the air flow velocity at the drying surface which is indicated by ß being the vapour transfer coefficient.



Key

X drying time [d]

Y water content [m^3/m^3]

A ß small

B ß medium

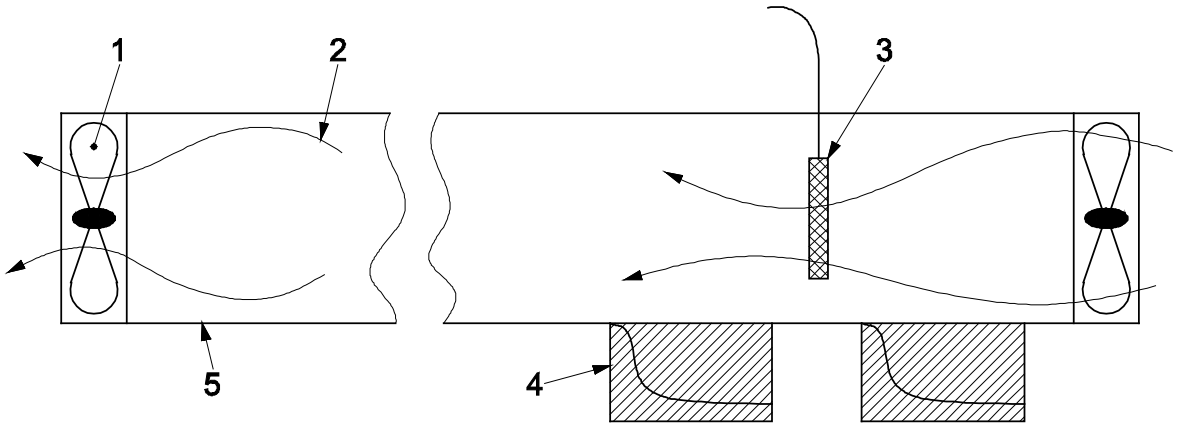
C ß large

D ß very large

E End of first drying phase

Figure B.1 — Influence of vapour transfer conditions at the drying surface on the measured drying behaviour; ß is the surface vapour transfer coefficient

A suitable measurement setup allowing to account for this influence is shown in the following figure.



Key

1 ventilator

2 controlled air flow velocity

3 sensor measuring temperature and relative humidity

4 material specimens, removable

5 flow channel

Figure B.2 — Schematic drawing of a suitable drying device

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