Universal drying chamber

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It appeared, following the floods in 2002, that there is no facility in the Czech Republic where larger amount of artefacts, documents, books and alike materials of historical and artistic value could be dried. As evidence the fact that even after four years there are still around 1,300 frozen books bound in leather or parchment left, which could not be dried as the common library collections by means of the thermal method organised by the National Library of the Czech Republic utilising wood drying facilities.

Each material, when dried, requires specific conditions, which shall ensure it is damaged as minimum as possible. Therefore in our considerations on a drying facility it was clear if some would be constructed it would be very appropriate the facility is of universal type, which would enable to dry given materials in accordance with up-to-date findings and knowledge. There was an informal team of experts formed under the leadership of the National Library of the Czech Republic and the result of its work is the prototype of a universal drying chamber, which was put under operation in August 2006 following its approval procedure. In the lines here below I will strive to introduce this equipment.

Potential use of the drying chamber

The chamber is designed to enable operating regimes as follows:

- vacuum drying at temperature above the freezing point;
- vacuum freeze drying (lyophylisation);
- drying under controlled atmosphere (temperature and relative humidity);
- book conditioning; and
- disinfection of books.

Design of the chamber

The drying chamber, which scheme can be seen in Figure 1, comprises of the chamber itself, the system of heating tiles, oil vacuum pump, freezing unit, air-conditioning loop, evaporator for a disinfecting agent, and a nitrogen tank. Of the units listed here, only some are engaged in every operating regime of the chamber. In the chamber system there are temperature, relative humidity, pressure, and equipment state sensors providing input data for the control computer. In order to monitor the drying process independent temperature and humidity sensors located right in the books and, furthermore, a tensometric balance with the scale up to 5000 g are employed. The sensors and the tensometric balance were developed especially for this application.

The chamber is made as a horizontally mounted cylinder of stainless steel equipped with two lids. Its volume is 3.3 m^3, diameter 1.4 m and is equipped with a special internal structure enabling individual fixing and heating of books by means of the heating tiles in order to minimise their distortions and overdrying (see Figures A, B, C, D, E, F).

The heating tiles are made as a doublet of unglazed ceramic tiles mounted together by means of glued distance cubes creating a ventilation gap in between of them. Each of the glued doublet tiles is, on the side of the gap, equipped with a heating wire, temperature sensor, and a control circuit maintaining tile temperature at the required value within the range -6°C to +60°C. Tiles are available in three sizes as follows: 20 x 25, 25 x 33, and 25 x 45 cm. Inside the chamber each tile doublet has its own connector with the control and heating circuit.

The oil vacuum pump enables to attain inside the chamber pressure below 1 mbar and is designed for exhausting of spaces with the high water vapour content.
The freezing unit is a steel cylinder equipped with an internal cooling coil and connected to the chamber by means of two closable holes and also connected to the vacuum pump through valves mounted on lids. Surface temperature of the cooling coil is lower than minus 60°C. It is, of course, equipped with a compressor for the coolant and an external heat exchanger for cooling. On the cooling coil surface there is a heating wire wound, which accelerates melting of accumulated ice in the defrosting period.

The conditioning loop is air-conditioning equipment, which is connected to the chamber by means of two closable inlets and outlets. The equipment consists of units for heating, cooling /dewatering, moisturising, filtration, tangential mixer, and blowers and provides for intensive circulation of air of defined parameters (temperature and relative humidity) in the chamber.

The disinfecting agent dosing can be carried out either from the *evaporator,* which is a closable glass vessel approx. 3 litres in volume equipped with external heating and connected through a valve to the internal space of the chamber, or by means of a piston feeder (large syringe), which applies the liquid disinfecting agent through a valve onto the chamber bottom.

The nitrogen tank contains nitrogen pressurised cylinders connected through a valve to the chamber and enables to fill the chamber with nitrogen to pre-defined pressure.

Independent sensors are especially developed thin temperature and relative humidity sensors, which can be inserted into various locations in the book or in its vicinity, and, furthermore, the tensometric balance, which enables to monitor weight decrement of the book selected. Data of these sensors are transmitted over the Internet and enable continuous supervision of the drying process.

Principle of the book fixing

On the basis of positive experience obtained in the course of the book drying following the floods in 2002 the books for drying are assembled into columns comprising of sandwich units (Figure 2).

Every sandwich unit consists of (from the top):

- heating tiles doublet (position 1);
- absorption paper (filter paper, old newspapers, etc.) (position 2);
- non-woven textile (based on polypropylene or polyethylene) ;
- THE BOOK (position 3);
- non-woven textile (position 4); and
- absorption paper.

In the bottom section of the column, below the last sandwich, there is the doublet of heating tiles put. At the top of the column there is a ballast weight set preventing the book distortion in the course of the drying process. Every column is located in a replaceable container, which moves on rails inside the chamber. The container size corresponds to the tile size and its major function is to fix the column in its vertical position and avoid it from falling down. The vertical arrangement with the gravitational weight advantage is that the shrinking of books during the drying has no influence on the weight effect. The book column shrinkage is up to 20%.

Independent measurement

The already mentioned thin sensors of temperature and relative humidity can be located onto various locations inside the column of the books dried and then monitor the course of the drying and depending on it to set drying parameters, or potentially monitor effects of drying conditions on the rate and quality of drying up and to prevent useless overdrying of the book. Figure 2 schematically depicts the locations of sensors as well.

Sensors' positions:

a. free in space;
b. in between the tiles;
c. on the tile surface;
d. on the book cover;
e. underneath the book cover;
f. in the book at various distance from its edge.

It is not realistic, of course, to have sensors of temperature and relative humidity in each book dried. Therefore we use so-called *signal books*, which are equipped with the required sensors. The signal books shall be characteristic types of the books dried, are soaked and dried simultaneously with the aimed books. After acquiring certain experience one can deduce the state of the group of books dried from the data transmitted from the signal book.

Water in the wet book is present in several forms as follows:

- water adsorbed on the internal surface area of fibre, which is in equilibrium with the relative humidity of its surrounding environment (this water causes paper swelling);
- free water in the interfibre space of paper (makes interfibre bonds weaker); and
- free water in between of the book block sheets.

The purpose of the book state checking is to find out as exactly as possible the moment of the right drying state and thus prevent overdrying. It holds for every drying method that the water evaporation absorbs heat from the book and the book mass has then lower temperature than its surrounding environment. The higher the temperature difference the more intensive is the evaporation under the given conditions. This depends on the water amount in the given book and on the surface, which water is evaporated from. Furthermore, it depends on the gradient of the water vapour concentration at the surface (slope of the gradient of the water vapour concentration drop outward from the book). Respective drying methods differ in the way they create the concentration gradient – vapour exhausting. The measurement of relative humidity is related to the water vapour concentration. If the space is saturated with water vapour then relative humidity is 100%. This is the state on the surface or inside the wet book. If there is the aforementioned gradient then relative humidity decreases outwards the surface. As the book is being dried water disappears from the book surface, relative humidity in its vicinity drops and this decrease advances into the book inside. Simultaneously, temperature on the dry surface is equalised with that of the surrounding environment.

This process can be documented on records from temperature and moisture sensors located at various positions in the book and in its vicinity. The record is made in the course of the ten-day-long drying by means of the vacuum freeze drying (lyophylisation) method. In vacuum drying or in the drying under controlled atmosphere records are of similar quality and differ solely in absolute values, namely of temperature.

Figure 3 demonstrates the course of temperature in the room around the book, in between the book and the tile, underneath the book cover, and inside the book. At the beginning of the drying process we can see great temperature differences in between the positions measured, which vanish at the drying process termination. For the first four days temperature inside the book is constant even if temperature in the surrounding environment is increased due to the heating in tiles. This indicates the fact that there is still free water– or ice, in the case of lyophylisation, respectively in the sensor location. As soon as this form of ice disappears temperature in the given location starts to increase and approximate to the temperature of the surrounding environment. Fluctuations of values, which occur in parallel on all curves relate to the pressure regime in the chamber that depends on the defrosting cycles and on the setting of the vacuum pump pressure regime. Figure 4 shows the record of the course of relative humidity in the wet (frozen) signal book and relative humidity at its edge (the same book as in Figure 3), furthermore, then in dry book for the sake of comparison. At the beginning of the drying process relative humidity at the edge and inside the book is 100%. Free water vanishes relatively early from the immediate vicinity of the sensors because the sensors are placed in a hole drilled in the frozen book in parallel to the sheet plane and the hole was not closed which enables faster evaporation of water from the hole walls. Therefore in this case the sensors indicate the value of relative humidity lower than the actual moisture value in between sheets inside the book. (As long as there is free water in the vicinity of the sensor the value of relative humidity is equal to 100%.) The end of drying process, similarly as in the case of temperature, occurs when relative humidity values at the book edge and inside are close to each other and reach values close to those of a dry book or values in the chamber space. Abrupt fluctuations on the record curves relate again to the pressure regime in the chamber (increasing pressure reduced the water vapour concentration gradient – relative humidity value is increasing).

Respective drying methods

Each of the three drying methods, which can be applied in this chamber, has its advantages and pitfalls and this chamber should allow acquiring detailed knowledge of their behaviour in concrete applications and potentially a useful combination thereof.

The drying process generally consists in shifting of saturated partial pressure of water vapour at the surface of the book dried and respective drying methods differ in the way of achieving this shift. Equilibrium partial pressure depends on temperature; the higher the temperature, the higher the partial pressure. As soon as the equilibrium partial pressure exceeds ambient pressure due to temperature increase the spontaneous evaporation occurs in the whole volume of the book dried. Under atmospheric pressure (1013 mbar) this conditions occur when temperature 100°C is attained. Under pressure 6.1 mbar it is attained at temperature 0°C. If pressure is lower than 6.1 mbar there is spontaneous vapour evaporation right from ice (sublimation).

Vacuum drying

This is the drying method, which utilises the aforementioned physical phenomenon of spontaneous evaporation that occurs in the moment when ambient pressure drops below the equilibrium value of the water vapour partial pressure at the given temperature. The intensive evaporation (boiling) occurs at low temperature and therefore there is no threat of thermal damage to the material dried. In the case of very wet material attention shall be paid to avoiding damage to the internal paper structure by intensive vapour evolution (boiling). On the other hand, too deep drop of pressure below triple point (6.1 mbar) may cause undesirable repeated freezing of water in the book. Figure 5 shows the water vapour equilibrium value depending on temperature (blue curve) and actual dependence of pressure in the chamber on temperature in the books. At the given temperature actual pressure is lower than the theoretical value because it is not equilibrium pressure, what is measured in the chamber, yet pressure reduced by the vacuum pump action.

This method advantage is simple equipment – the chamber and vacuum pump. At low pressure there is no danger of mould growths (due to the lack of oxygen). Water is present in liquid phase and once removed the disturbed interfibre bonds get re-established. The drawback is the need for a special quality vacuum pump, which enables to exhaust virtually pure water vapour from the chamber, which can be at the amount of up to 100 kg per one chamber charge. Furthermore, if conditions in the chamber are not monitored there is still threat of the material overdrying. There is one undesirable fact with this drying process that the migrating water transports soluble compounds applied in the paper and book production process to the book surface. These are binders, glues, adhesives, and dyes, which create a hard, dark-coloured film namely on the book edges, which is often mistakenly considered as the sign of high temperature effects.

Commercial vacuum chambers dedicated to the document drying are equipped with heated shelves. In this chamber instead of the heated shelf each book is fixed in between heating tiles, while every book column can have its temperature set individually. Therefore it is suitable to sort the dried books the way each column contains similar books concerning their dimensions and potentially their degree of wetting. The drying process is computer controlled preventing too intensive evolving of vapour at the beginning of the drying, which could damage the materials dried. The fixing prevents the distortions of the books dried.

Vacuum freeze drying (lyophylisation)

This drying method is carried out under pressure below 6.1 mbar, under which frozen water in the books is transferred right into gaseous phase (ice sublimates). The main advantage of this method is that the absence of liquid phase minimises further diffusion of inks and dyes and there is no aforementioned transport of soluble compounds onto the surface of the book cover and edges; lower pressure also does not allow moulds to grow. Its drawbacks are as follows:
a) need to have a freezing unit, which is capable to reach temperature deep below freezing point (-60°C);
b) overdrying is easily attained;
c) because no water in liquid phase is present the interrupted inter-fibre bonds in paper are not restored and thus especially highly absorbing paper grades have distorted structure after the drying up.
The similar facts hold for leather and parchment. The structure distortion is clearly visible in certain tracing paper grades, which loose their transparency following the lyophylisation drying (see here below).

The method is also demanding for the control process algorithm because the freezing unit must be regularly defrosted that means the accumulated ice must be removed. During this stage water vapour is taken into the vacuum pump suction because the freezer is set out of operation. Because of the real transfer of radiant heat from the chamber jacket into the books the water vapour evolution at the process beginning is really substantial and it may happen that the vacuum pump is not able to maintain pressure below triple point temperature if the chamber is completely full and ambient temperature is high. As soon as temperature is above triple point risk of the frozen water thaw on the book surface occurs. This is especially acute at the beginning of the drying process and in summer months. Therefore the chamber room is equipped with an air-conditioning unit, which can maintain room temperature below the critical value. Details of the relation in between pressure in the chamber and temperature in the book (heating tiles are switched on) can be seen in Figure 6.

Drying under controlled atmosphere and conditioning of books

This way of drying is the most complicated concerning the automatic control thereof. The chamber arrangement scheme (used components) is shown in Figure 7. Because of the chamber huge weight (toll for its universality because it must withstand deep vacuum) as well as of the books dried the system has a large thermal inertia. In the course of the pilot operation optimum locations for measuring of values of temperature and relative humidity, which the system shall maintain in the chamber, were sought. The outlet pipeline from the chamber was selected as the most appropriate location. In this location properties of circulating air are closest to those of the air flowing in the chamber.

When controlling the process properties of inlet air are changed the way outlet air has parameters required for the drying. Because of the aforementioned system high thermal inertia it was necessary to set the maximum allowed rate of change of inlet air temperature and its relative humidity value bound to temperature in the coolest location of the chamber the way that dew point temperature must be lower than temperature in the coolest location in the chamber. In other words relative humidity of inlet air must have such value that when inlet air is cooled down to temperature in the coolest location of the chamber no water condensation occurs.

The coolest location in the chamber is its jacket and lids, which also have largest thermal capacity. There is a temperature sensor in the jacket, which can be employed for the control. The temperature distribution at various locations in the chamber if drying temperature is set to 62°C and relative humidity is set to 45% can be clearly seen Figure 8. The record interruption relates to the chamber opening and inspection of the chamber charge. In its continuation the ramp of the chamber regime transition into the original state before the interruption is seen and at the termination the course of temperature at lower temperature set. The aforementioned criteria, which prevent condensation in the cool locations in the chamber, will cause that the chamber heating period from ambient temperature to pre-set 62°C lasts for almost 10 hours.

When drying under controlled atmosphere, conditions of drying can be set within the range of the natural drying in air up to temperature 60°C and relative humidity up to 60%. The last parameters mentioned are suitable to prevent continuing ion mould growth of contaminated books, which were frozen late. Common moulds are killed under these conditions. The two previous drying methods merely stop the mould growth during the drying process /1/. High temperature at drying, causes, of course, accelerated ageing of paper and our experience can be summarised into the statement that every day of the drying under the aforementioned conditions will reduce mechanical properties of paper, expressed as folding endurance, by about 1%. In order to prevent mould growth it is not necessary the books are under these conditions for the whole period of drying yet these conditions are applied for a certain minimum period, which can be preset in the chamber. Upon the period expiry temperature could be reduced and the drying can be completed under more moderate conditions. This way the worsening of the paper mechanical strength could be minimised. The undoubtable advantage of the drying under controlled atmosphere is that this allows to completely eliminate overdrying.

Book conditioning is the process for attaining such values of temperature and moisture in the whole book volume corresponding to equilibrium ones in the environment where the books are located. Technically it is a special case of "drying" under controlled atmosphere. Under static conditions of storage in a room it is time-demanding process as it can be seen in Figures 9 and 10, which demonstrate approaching of relative moisture content inside the overdried, or underdried book, respectively to the values in the environment. Under conditions of intensive air circulation in the chamber this process could be accelerated. At the time when this paper was written information from appropriate experiments have not been available yet.

Effects of drying methods on the structure of paper and books

As it was mentioned in introduction water present in wet books causes swelling of paper and loosening of interfibre bonds. The following freezing fixes these changes, or makes them even more pronounced due to the ice volume expansion. This fact is clearly seen in Table 1.

Table 1: Effects of the drying method on the book thickness in %

	ORIGINAL	WET	FROZEN	DRIED UP	
				Under controlled atmosphere	Lyophylisation
Book	100%	121%	133%	114.5%	125%
Book block	100%	118%	---	111%	122.5%

At the drying under temperature above freezing point the leaving liquid water restores inter-fibre bonds by the influence of surface tension yet does not restore the effects of pressing and calendering in the paper production, which were dismissed by the soaking. In the vacuum freeze drying (lyophylisation) the interrupted interfibre bonds, because the effects of surface forces in between liquid water and paper fibre are missing, are not restored and the loosen structure will remain. This is the main reason why the book thickness rise is twice as high after the drying by lyophylisation as in the case of the drying under controlled atmosphere. Table 1 compares two identical books dried by various methods following the previous soaking (by submersion) for 24 hours.

The aforementioned fact is significantly pronounced in the drying of tracing paper. If it is drying by vacuum freeze drying the water sublimated from the interfibre space is replaced with air, which makes the paper to cease being transparent. In the left part of Figure 11 there are two halves of a cut tracing paper strip, which were dried by vacuum freezing and are matched together again. The right half was, before matched, rewetted and dried in air. Comparing the halves we can see by how much the size of the lyophylised sample (on the left) is larger than that of the sample, which was rewetted and dried in air and water in it was in liquid phase when dried. The shrinking is the consequence of the restoration of interfibre bonds and to the elimination of air-filled gaps between fibres. This led to the re-establishing of transparency, which can be partly seen in Figure where the left half is whiter (reflects more light). In the right part of Figure 11 there are the two halves in cross section – the upper sample underwent lyophylisation only. It can be stated, even if the photograph is not of high quality, that the upper sample has larger volume.

Energy demand of the drying process

Respective drying methods utilise different pieces of equipment, which form the chamber accessory. The energy-demanding processes of the respective drying methods are given in Table 2.

Table 2

Drying method	Energy-demanding processes employed
Vacuum freeze drying (lyophylisation)	Vacuum pump, freezer compressor, tile heating
Vacuum drying	Vacuum pump, tile heating
Drying under controlled atmosphere	Air heating, fans, moisturiser

Because of the specific usage of the chamber any application of heat recovery would be technically very complex and therefore it is not applied. Therefore the lyophylisation method is the most energy–demanding one. In Table 3 respective methods are compared at comparable volume charge of books and newspapers dried. It follows from the experiments carried out so far that the most energy efficient drying method is the drying under controlled atmosphere. The composition of books – quality of paper, types of covers, etc., which affect the water vapour diffusion through the book mass, will also play a certain role. This is seen in the difference of specific energy values in the drying of newspapers and books, that means energy in kWh required for the removal of 1 kg water from the given material.

Table 3: Specific energy consumption for the drying up of 1 kg water

Drying method	Lyophylisation *newspaper*	Lyophylisation *books*	Vacuum drying *books*	Drying under controlled atmosphere *books*
Specific energy consumption for the drying up (kWh/kg water)	13.0	16.8	11.8	9.5

Let us take, for the sake of illustration, take the weight of a medium-sized book of around 0.5 kg, which, if completely soaked, absorbs the same amount of water, then the price for energy required for the book drying up, at the price 2.00 CZK/kWh (about 0.1 USD/kWh), falls in between of 9.50 and 17 CZK (about USD 0.45 and 0.82) depending on the method selected.

Conclusions

Following roughly ten months of pilot operation, which was necessary for fixing of certain issues that accompany any prototype and for debugging and optimisation of software under real conditions of the chamber full of wet books, the approval procedure was performed and the chamber was approved and released for common operation. It is the unique facility in the world. Thanks to it all leather and parchment bound books of the Municipal Library of the City of Prague, mentioned in introduction, are dried up in it at present time (Feb. 2007). In the future also potential drying of large-format documents (180 x 120 cm) and bulky artefacts is considered due to the option of easy adjustment of the internal structure thereof.

References

/1/ *Studie vlivu různých sušicích metod na mechanické vlastnosti papíru a životaschopnost plísní (Study on effects of various drying methods on mechanical properties of paper and vitality of moulds)*. Kolektiv autorů NK ČR a NA ČR (Collective of authors from the National Library of the Czech Republic and the National Archives of the Czech Republic), (Praha / Prague 2002). The results are also published on the Internet pages of the National Archives of the Czech Republic and of the National Library of the Czech Republic.

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Figure 1: Scheme of the chamber

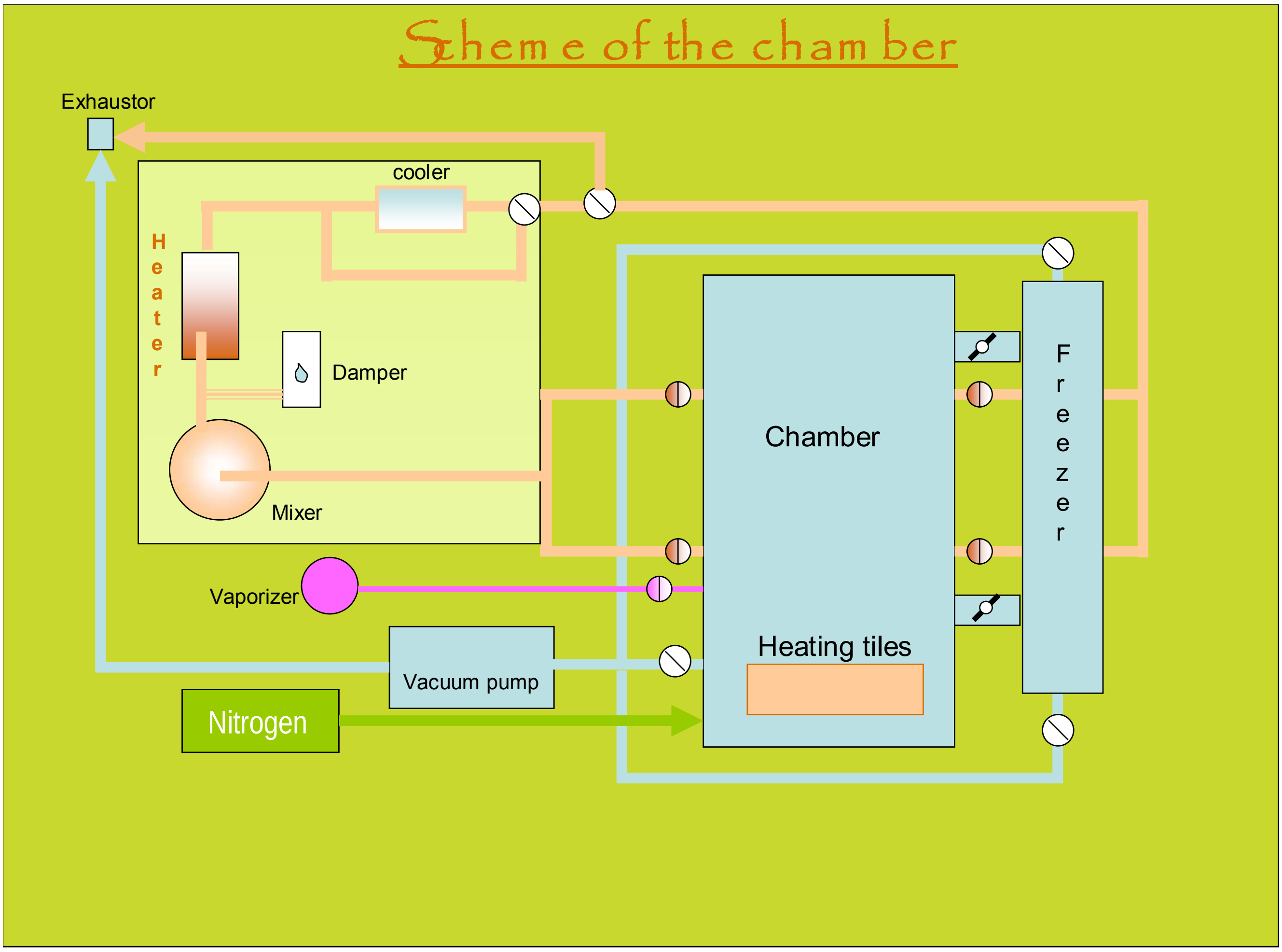

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Figure 2: Composition of the sandwich and scheme of the sensor locations.

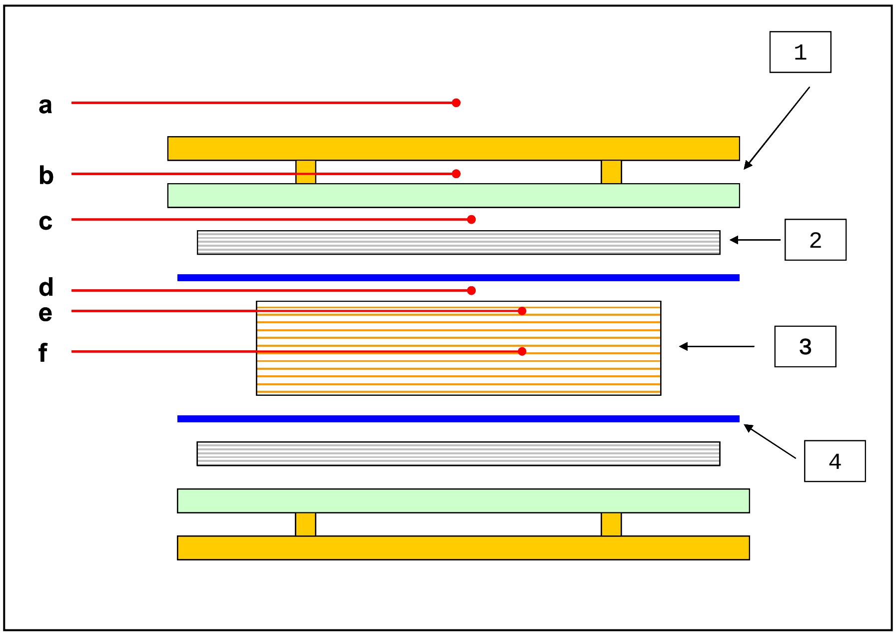

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Figure 3: Record of temperature sensors in the signal book.

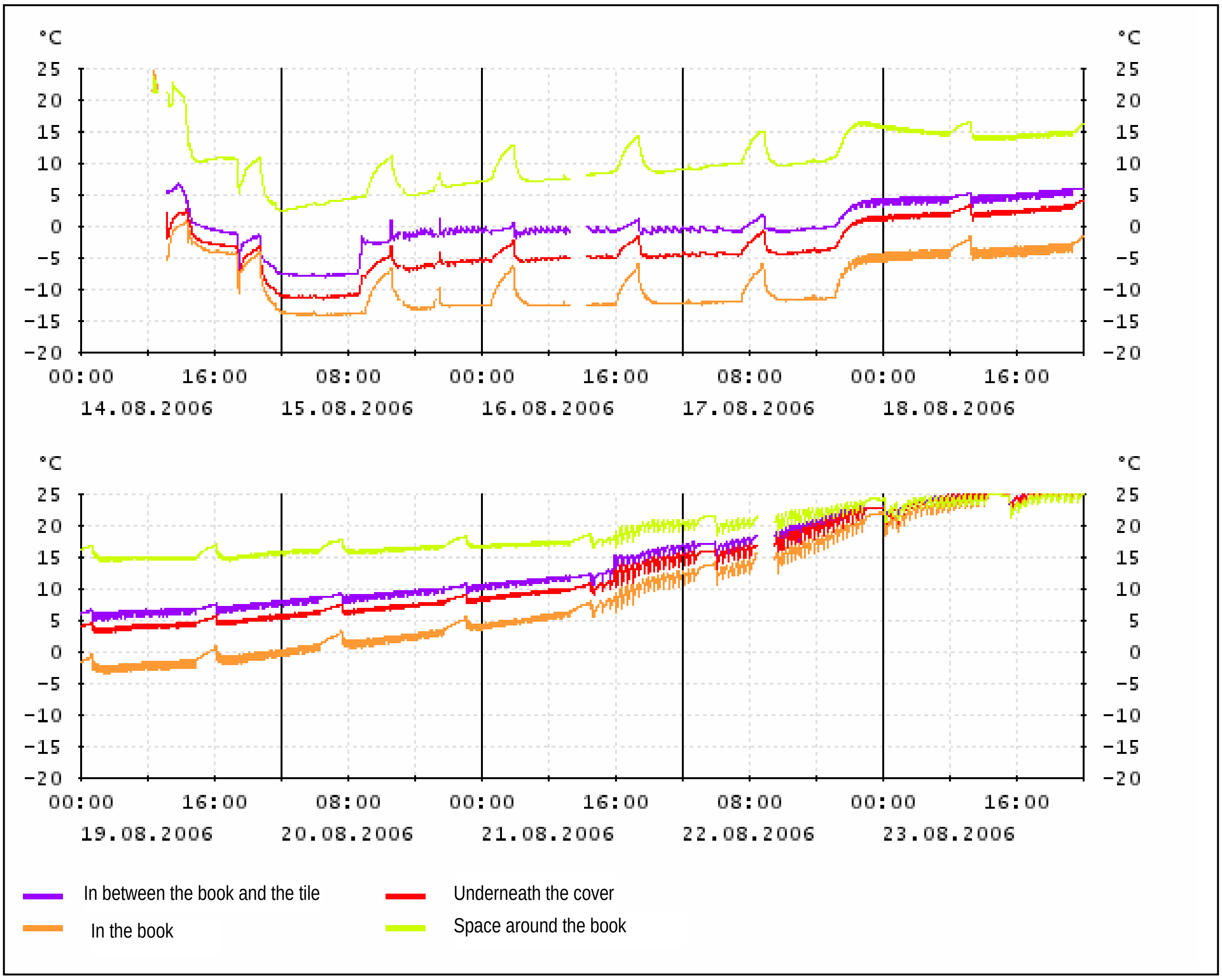

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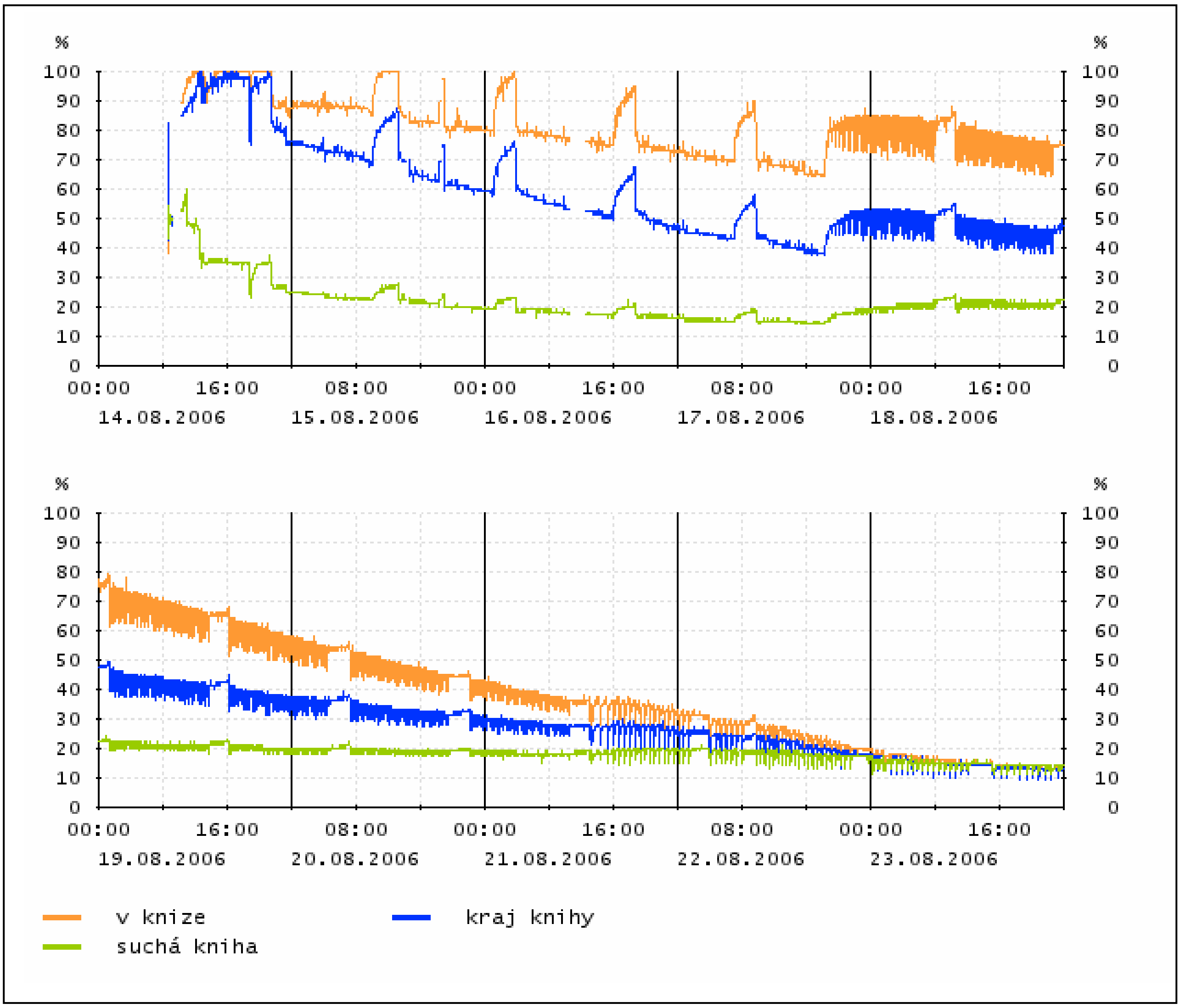
Figure 4: Record of relative humidity sensors in the signal book and in the dry one.



orange line: in the book
blue line: book edge
green line: in the dry book

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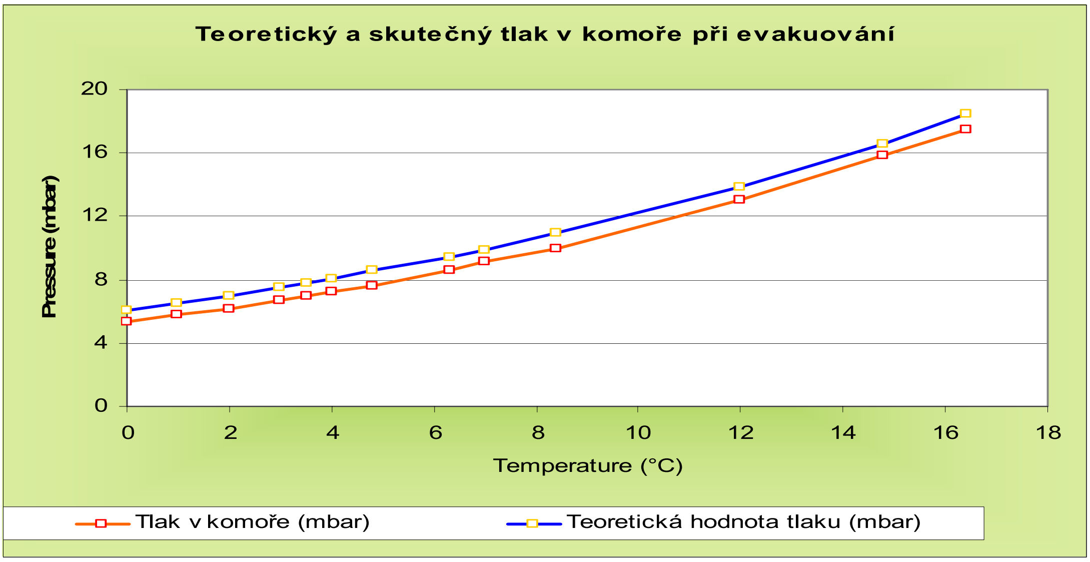
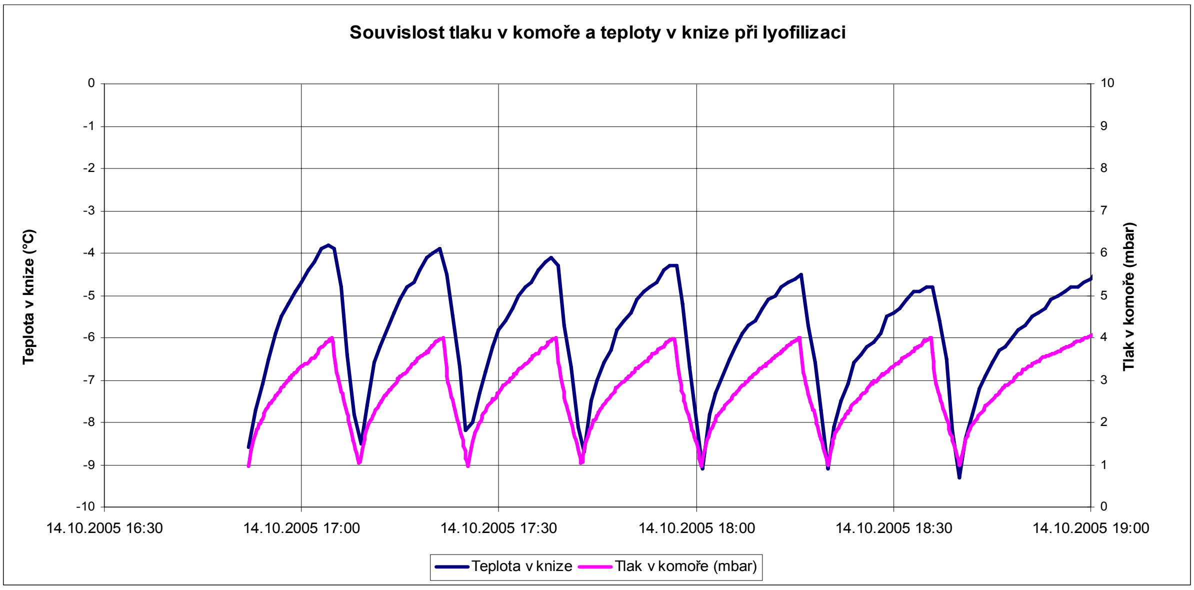
Figure 5: Relation in between temperature in wet books and pressure in the chamber.



pressure in the chamber (mbar) theoretical pressure value (mbar)

Figure 6: Relation in between temperature in the book and momentary pressure in the chamber.



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temperature in the book pressure in the chamber (mbar)

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Figure 7: The arrangement for the drying under controlled atmosphere
(red arrows point to locations of sensors for temperature and relative humidity)

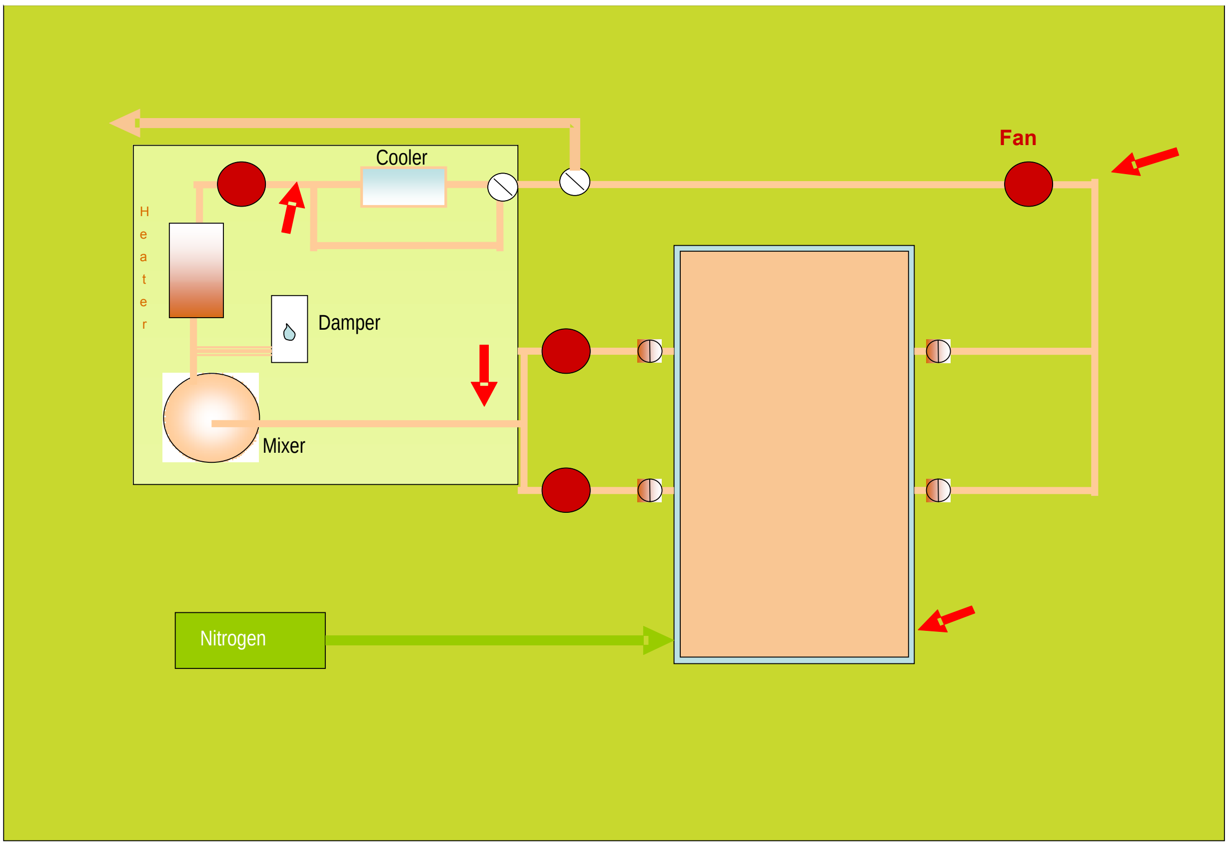

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Figure 8: Air temperature in respective chamber parts.

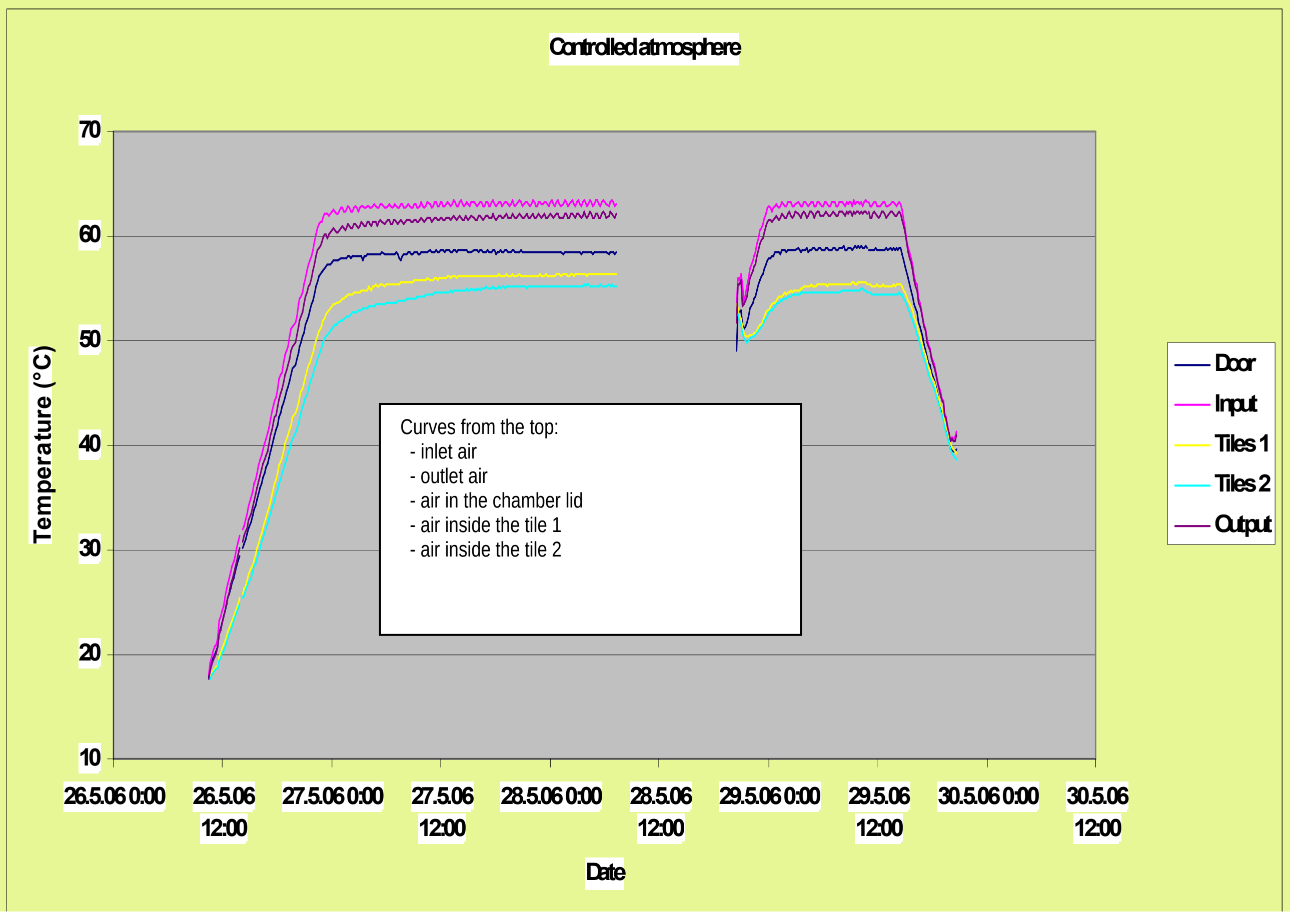

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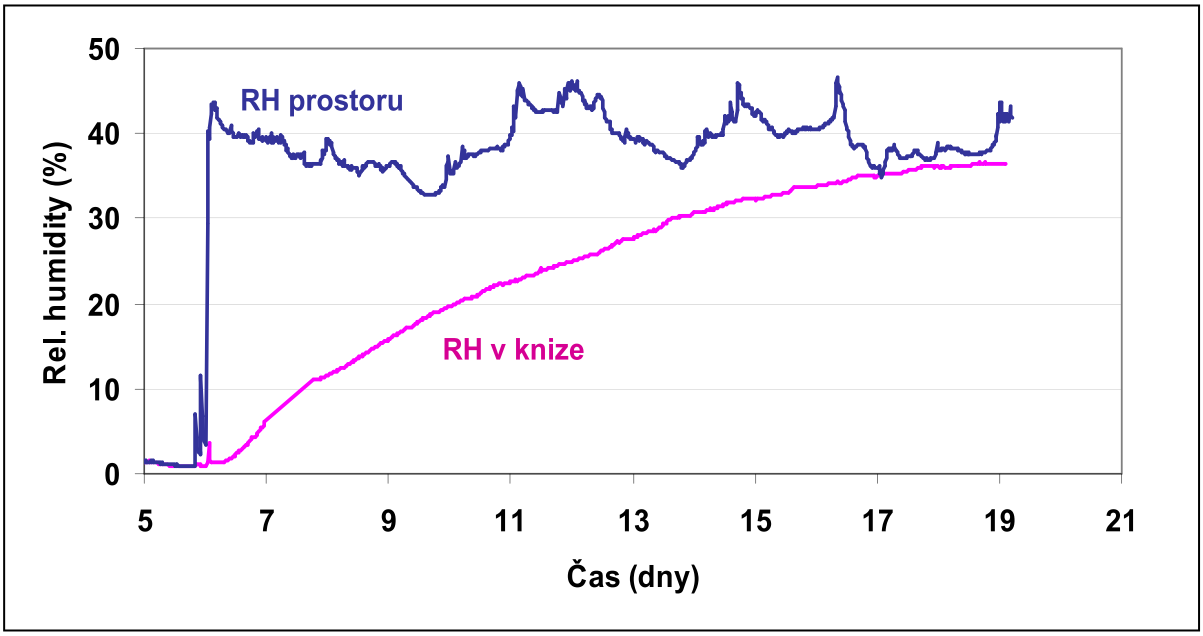
Figure 9: Conditioning of the overdried book.



blue line: rel.humidity in the room
pink line: rel.humidity in the book
abcissa: time (days)

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Figure 10: Relative humidity in the book and the book weight when moist book is conditioned; demonstration of the data transmission over the Internet.

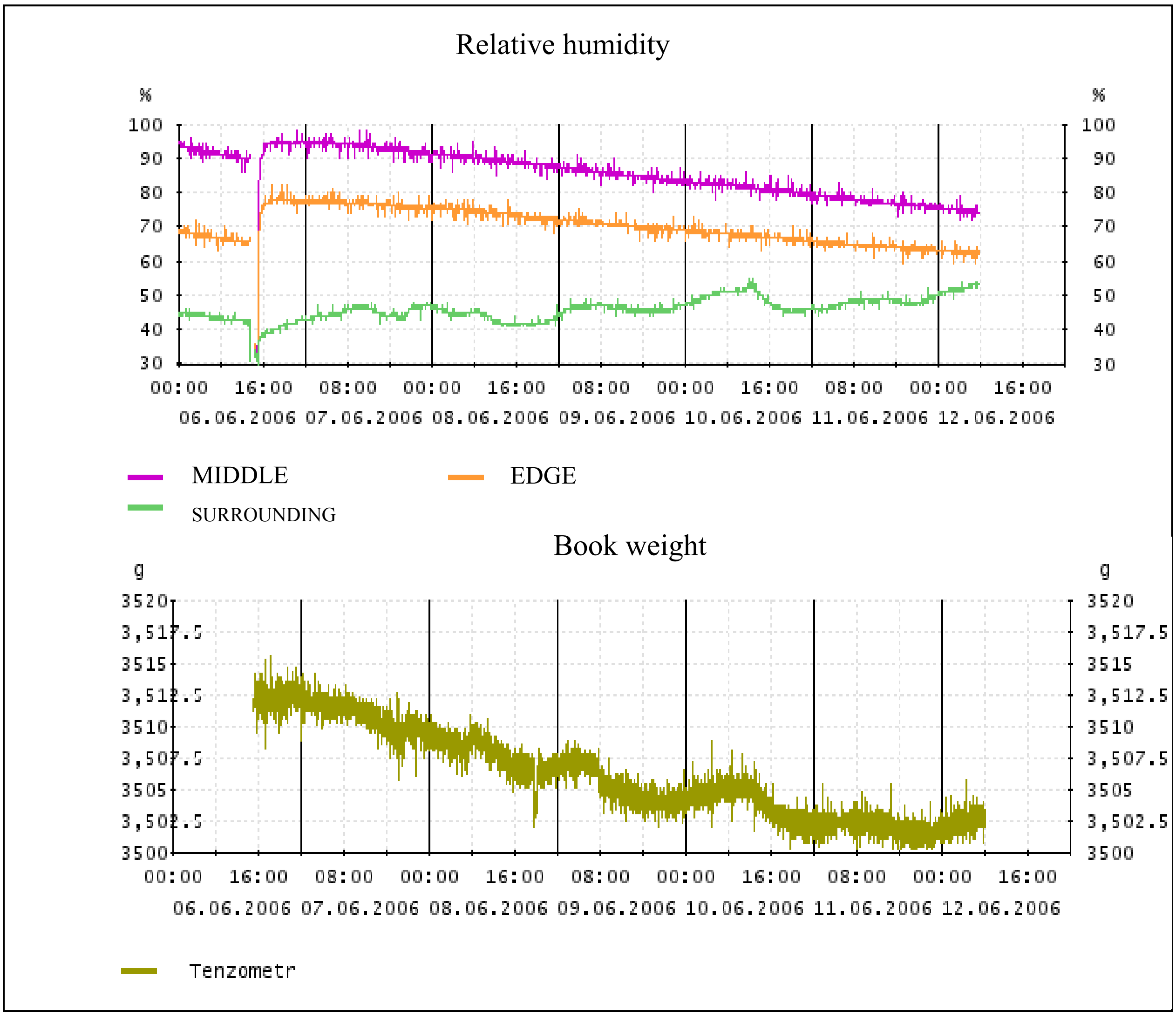

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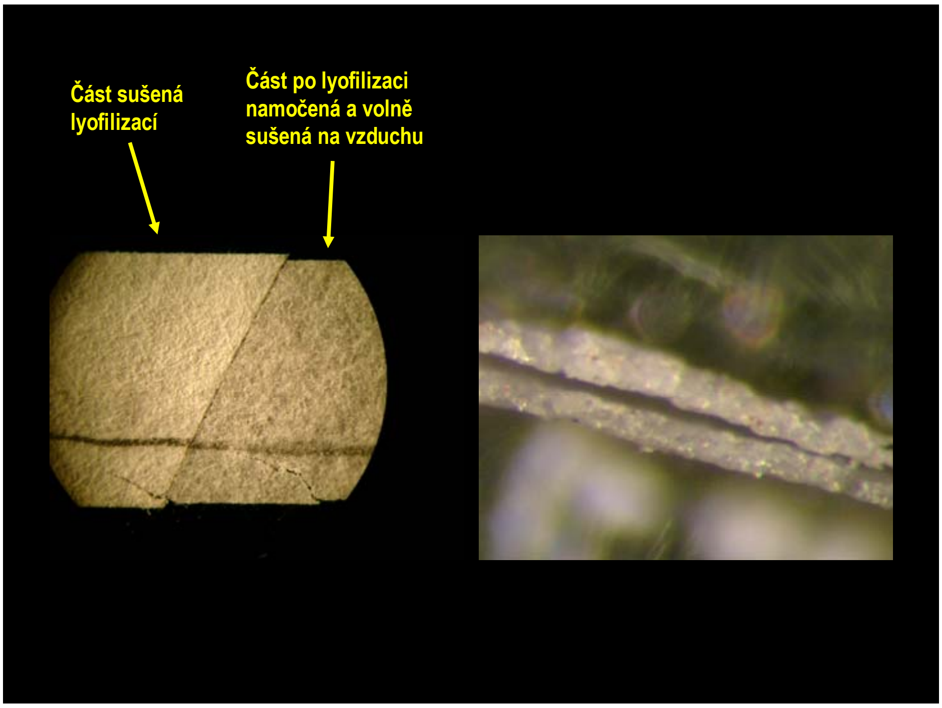
Figure 11: Expansion of tracing paper following the lyophylisation



Část sušená lyofilizací = The part dried by vacuum freeze drying (lyophylisation).

Část po lyofilizaci namočená a volně sušená na vzduchu = The part, which was wetted after being vacuum freeze dried (lyophylised) and then air dried.