


Article

Theoretical and Experimental Analysis for Cleaning Ice Cores from Estisol™ 140 Drill Liquid

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Featured Application: This work gives indications for cleaning and preservation of ice cores, which will be drilled in Antarctica during the EU project Beyond EPICA Oldest Ice and provides general guidelines for ice drilling activities and preservation of ice cores.

Abstract: To reconstruct climate history of the past 1.5 Million years, the project: Beyond EPICA Oldest Ice (BEOI) will drill about 2700 m of ice core in East Antarctica (2021–2025). As drilling fluid, an aliphatic ester fluid, Estisol™ 140, will be used. Newly drilled ice cores will be retrieved from the drill soaked in fluid, and this fluid should be removed from the cores. Most of it will be vacuum-cleaned off in a Fluid Extraction Device and wiped off with paper towels. Based on our experiences in Greenland deep ice coring, most of the residual fluid can be removed by storing the cores openly on shelves in a ventilated room. After a week of “drying”, the cores have a dry feel, handling them do not give “wet” gloves and they can easily be marked with lead pencils. This paper presents a theoretical investigation and some simple testing on the “drying” process. The rates of sublimation of ice and evaporation of fluid have been calculated at different temperatures. The calculations show that sublimation of the ice core should not occur, and that evaporation of fluid should be almost negligible. Our test results support these calculations, but also revealed significant fluid run-off and dripping, resulting in the removal of most of the fluid in a couple of days, independent of temperature and ventilation conditions. Finally, we discuss crucial factors that ensure optimal long-term ice core preservation in storage, such as temperature stability, defrosting cycles of freezers and open core storage versus storage of cores in insulated crates.

Keywords: ice cores; drilling; Estisol; Beyond EPICA

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1. Introduction

The European project: Beyond EPICA Oldest Ice (BEOI) aims at drilling an approximately 2700 m long continuous ice core to bedrock in East Antarctica, covering the climate history of the last 1.5 Million years. This will be a significant extension of the climate record of the previous EPICA project, which covers the past 800,000 years, and the hope is to better understand Antarctic climate and greenhouse gases during the Middle Pleistocene Transition (MPT), which occurred between 900,000 and 1.2 Million years ago. To guarantee the best conservation of the ice core material and the integrity of the gas record in the ice, the cores will be kept at -50 °C. Deep ice core drilling can only be done using “wet” drilling techniques, i.e., the ice cores are drilled in a fluid-filled borehole. In the literature,

many different fluids have been proposed and studied for ice drilling [1–5], discussing their properties and comparing advantages and disadvantages. For the project BEOI, an aliphatic ester called Estisol™ 140, has been selected as it meets many criteria, such as density, freezing point, no toxicity (to nature and humans), non-polarity, low pour point (−90 °C), and low viscosity. To limit contamination of the core by the fluid, the Estisol™ 140 should be removed from the ice cores after drilling. Most of the fluid will be mechanically removed from the ice core surface by a special vacuum-cleaner unit and wiped down with paper towels immediately after drilling. Prompted by experiences in Greenland deep drilling projects, where it was observed that the cores after one week of storage on open shelves in a snow cave appeared much “drier” than fresh cores, the BEOI project planned to store the fresh cores on open shelves in a ventilated room. In order to speed up the “drying” process by evaporation of drilling fluid, a ventilation air flux of 40 m³/h air flux has been suggested.

The main aim of this paper is to discuss how storage time, degree of ventilation and temperature influence the removal of residual fluid from the cores, i.e., the “drying” process. We will discuss: (1) the physical processes of ice sublimation and drilling fluid evaporation under ventilation conditions; (2) calculations of how much ice could be lost and how much drilling fluid could be removed at maximum ventilation; (3) simple tests of fluid removal, and (4) how to optimize the storage conditions of ice cores to preserve their scientific usefulness even after long time in storage. We were most curious about the rate between the evaporation of Estisol™ 140 and the sublimation of the ice. Evaporation and sublimation processes will be discussed theoretically first, followed by an analysis of the particular properties of ice and the chosen drilling fluid. The evaluation of ice losses and fluid evaporation will be finally compared with specific tests on real ice cores, in order to get an evaluation of the theoretical findings. The overall aim is to provide input to the logistical planning and engineering of the drill site under development for the BEOI.

2. Theoretical Section and Calculations

2.1. General Theoretical Aspects

The state diagram of water shows that ice and snow do not evaporate (sublimate) below the freezing temperature at standard pressure. Indeed, the direct solid-vapor transition can happen only below 611.73 Pa (which is 6×10^{-3} atm) at temperatures below 0.01 °C. Yet, it is a common observation that ice and snow can disappear rapidly in external environments especially when exposed to wind and sunlight. An analysis of ice sublimation was presented by Jambon-Puillet et al. [6] and provides a quantitative description for the diffusion of ice molecules at the ice-air boundaries. Other theoretical analyses are available on the analogous problem of water evaporation in the coexistence of liquid and gas phases [7,8], an issue that was investigated for long [9–11]. The first pioneering studies of Hertz on the evaporation of mercury [9], further developed by Knudsen [10] set the basis for understanding the physics of evaporation (and sublimation). The partial pressure of water vapor over water (or ice) plays a major role as well as the relative ambient humidity, determining a dynamic equilibrium between the rate of evaporation (or sublimation) and the rate of condensation. In the framework of the classical kinetic theory, the calculations are based on a statistical approach, considering a Maxwellian distribution function for the molecular velocities. The Hertz–Knudsen formula for the net evaporation rate, corrected for mass transportation across the interface in non-equilibrium conditions [12], is expressed by the following formula:

$$\mathcal{M} = \frac{2}{2 - \sigma_c} \sqrt{\frac{m}{2\pi k_B}} \left(\sigma_e \frac{P_{sat}}{\sqrt{T_L}} - \sigma_c \frac{P_V}{\sqrt{T_V}} \right) \quad (1)$$

where M is the net mass flux, m is the molecular mass, k_B is the Boltzmann constant, P_{sat} and P_V are the saturation vapour pressure and the partial pressure of the vapor phase, respectively, T_L and T_V are the temperatures of the liquid and the vapor. Further developments were proposed by Labuntsov [13], Ytrehus [14], and Rose [15,16], but Equation (1)

represents the concept well. A major difficulty is knowing the values of the evaporation and condensation coefficients and the conditions that exist at the interface. These coefficients must be determined experimentally, but values obtained in several experiments show large discrepancies. For experimental and theoretical purposes, σ_e and σ_c are often assumed as equal ($\sigma_e = \sigma_c$). An additional critical element is the effective temperature in the liquid/vapor phase at the interface, where mass transfer takes place. Indeed, P_{sat} and P_V are dependent on the temperature. Moreover, evaporative cooling reduces the local liquid temperature below that in the bulk, with a minimum temperature observed at the liquid-vapor interface, where a discontinuity can be predicted. Direct experimental measurements have demonstrated that the temperature gap at the interface can be as large as 4 °C [17]. The equations can be directly transferred to the ice-vapor system for studying the case of sublimation.

A different approach to describe mass-transport phenomena is based on the diffusion process. The paper by Jambon-Puillet et al. [6] is straightforward for ice sublimation. The diffusion equation at the interface allows calculating the local evaporative flux. It is shown that this equation is a Laplace equation; therefore, there is a strong analogy with electrostatics. The strong curvature dependence is analogous to the known electrostatic tip effect, indicating that the flux of sublimated material is maximum at the edges. Few mathematical steps lead to the mass transportation equation:

$$\mathcal{M} = -H \left(\frac{P_{sat}}{T_{ice}} - \frac{P_V}{T_V} \right) \quad (2)$$

with H a mass transfer coefficient and T_{ice} the ice temperature. Equations (1) and (2) are mathematically similar.

Vapor concentration through the interface is not a continuous function, but can be obtained by equilibrium correlations, where we have the boundary condition that at the interface the vapor density equals the saturation density. If the gas phase is composed of many components, the molar fraction of water vapor alone (χ_v) can be calculated by using Raoult law:

$$\chi_v \cong \frac{P_{sat}(T)}{P} \quad (\text{Raoult law}) \quad (3)$$

This formula also indicates the maximum molar fraction of vapor in air at a certain pressure and will be used in the following calculations for estimating the maximum amount of ice sublimation.

2.2. Properties of Ice and Drilling Fluids

The aim of storing ice cores in a ventilated chamber is to enhance the removal of drilling fluid without the risk of significant ice sublimation. A crucial point in the processes of evaporation or sublimation is the partial pressures of the gas phases of the involved substances, in our case of ice/water and EstisolTM 140.

A detailed review on vapor partial pressure over ice and other properties as a function of temperature, including numerical data and parametrizations, was published in 2005 by Murphy and Koop [18], providing the following equation:

For $T > 111$ K (−162.15 °C):

$$P_{ice} = \exp(9.550426 - 5723.265/T + 3.53068 \ln(T) - 0.00728332T) \quad (4)$$

This fits the numerical solution to within 0.025% from 111 K to the triple point. The plot of this function is shown in Figure 1, using a linear-log scale, indicating a fast-growing variation with the temperature. The value obtained at the temperature of −50 °C is $P_{ice}(-50 \text{ °C}) = 3.939 \text{ Pa} = 0.0389 \text{ atm}$, which is less than 4% of the external atmospheric pressure surrounding the ice core. According to Raoult law in Equation (3), the molar fraction of vapor (χ_v) in the total atmospheric composition at −50 °C is less than 4%.

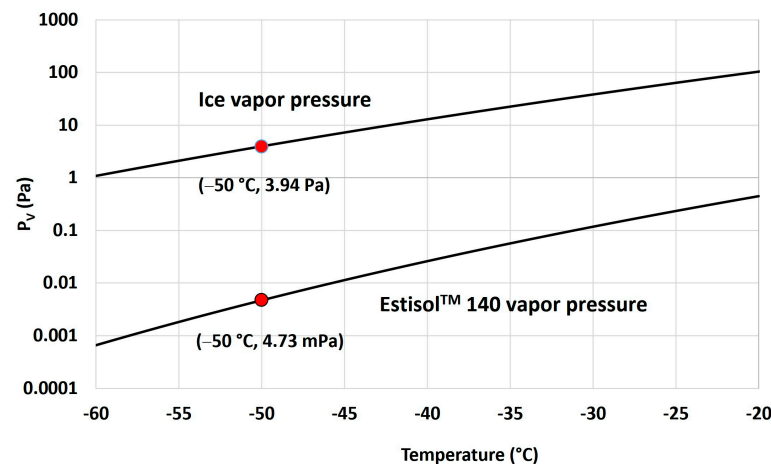


Figure 1. Vapor pressure over ice and Estisol™ 140 as a function of temperature. The plot is in linear-log scales, indicating a fast-growing variation.

The vapor pressure for Estisol™ 140 is shown in Figure 1. It was calculated by the Antoine equation, reported in Equation (5), provided by the supplier Esti Chem A/S. These values are about 1000 times lower than the ice vapor pressure; therefore, cleaning the ice cores from the drilling fluid by evaporation alone could be very difficult.

$$\ln P = A - \frac{B}{C + T} \quad \left(P \text{ in bar, } T \text{ in } ^\circ\text{C} \right) \quad (5)$$

with $A = 9.9539$; $B = 3932.58$; $C = 196.63$.

The functions $P_{sat}(T)/\sqrt{T}$ and $P_{sat}(T)/T$, which control Equations (1) and (2) both increase with temperature, due to the exponential trend that dominates the growing of $P_{sat}(T)$. Since phase change requires heat, the surface temperature will decrease during this process, providing a negative feedback and consequently reducing the sublimation and evaporation fluxes.

2.3. Evaluation of Ice Sublimation and Fluid Evaporation

At the drilling site of BEOI, a ventilated container is planned for storing fresh ice cores for one week at $-50\text{ }^\circ\text{C}$ with a suggested air flux of $40\text{ m}^3/\text{h}$. The aim is to remove the drilling fluid from the ice by drying. There are, however, two constraints: loss of ice should be small (the best would be nothing) and removal of drilling fluid should be large (the target would be all of it). Considering Equations (1) and (2), the main driving force for material diffusion is the difference between the vapor partial pressures. We may assume that initially the ice is covered by the drilling fluid, but after some time it may become exposed. Sublimation of exposed ice is driven by the relative humidity of the air surrounding the core. Under static conditions (no air is exchanged) an equilibrium is eventually reached, and sublimation stops when $P_V = P_{ice} = P_{sat}$; $\text{RH}\% = 100\%$. In our system, the air for ventilation is taken from a deep hole in the firn pack at the temperature of $-50\text{ }^\circ\text{C}$, which is the annual mean temperature at the site. As this air is passing through only snow and firn before entering the container, the equilibrium conditions of the air match those of the air inside the container. This condition prevents the sublimation of ice from the core: $M = 0$. Moreover, since sublimation requires heat (latent heat of sublimation is about $675\text{ Cal/g} = 2824\text{ J/g}$), the surface material of the ice core will cool from any small amount of sublimation, in agreement with similar studies on liquids [17], resulting $T_{ice} < T_{air}$. If the temperature of the ice decreases, the driving force for vapor transfer will be further diminished, according to Equations (1) and (2). The numbers simply indicate that ice sublimation is not a significant issue in a ventilated chamber.

To establish an absolute upper limit on the maximum amount of ice loss, we assume a quasi-static condition. We also assume that ice sublimation will provide vapor to the

saturation point before the ventilated air exits and we neglect ice cooling. In each hour, 40 m^3 of air at $-50 \text{ }^\circ\text{C}$ is replaced in the ventilation chamber. Using the ideal gas law and remembering Raoult law in Equation (3), we can calculate the maximum ice weight loss as a function of temperature for different values of RH%, as shown in Figure 2.

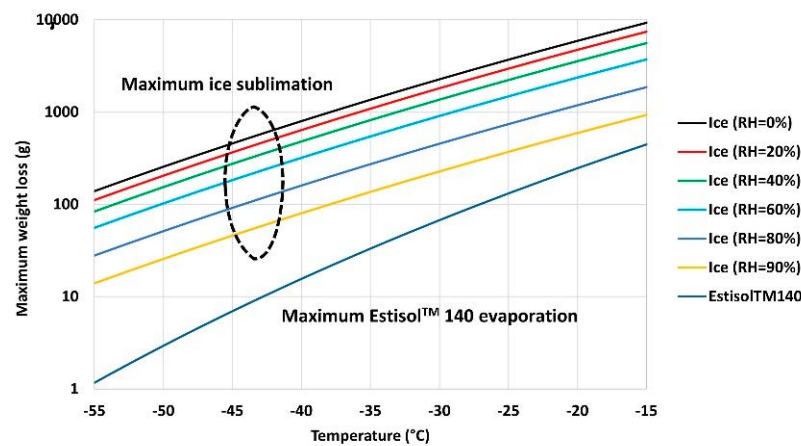


Figure 2. Maximum ice sublimation and Estisol™ 140 evaporation after one week ventilation at $40 \text{ m}^3/\text{h}$ as a function of the temperature and relative humidity of the inlet air. If, as assumed, the air is collected from a hole in the ice the relative humidity is 100% and the ice loss goes to zero.

Using saturated air coming from a deep hole in the firn pack we do not expect any losses in the ice core. Anyway, at $-50 \text{ }^\circ\text{C}$, even in dry air (RH = 0%), the maximum loss (257 g) is negligible, considering the typical size of an ice core (diameter $\sim 10 \text{ cm}$, length $\sim 1 \text{ m}$, weight $\sim 7.26 \text{ kg}$) and that the ventilation container is planned to have space for 200 ice cores (1452 kg).

A similar analysis can be done to calculate the evaporation of the exposed drilling fluid. Again, this is an upper limit since part of the fluid could be trapped inside the pores of the core and not exposed to the air flux. The maximum amount of Estisol™ 140 that could be removed as a function of temperature is reported in Figure 2. In particular, at the working temperature of $-50 \text{ }^\circ\text{C}$, after one week in the ventilation container this value is only 2.9 g. Given the density of Estisol™ 140 as $930 \text{ Kg}/\text{m}^3$ [3], the liquid loss corresponds to a volume of only 3.2 mL of fluid for the whole ventilation chamber, which is a negligible amount, and this is even an overestimation of the real expected value.

3. Experimental Testing

3.1. Ice Core Cleaning

The theoretical values discussed in Section 2 are at odds with practical experience handling ice cores at drilling sites. It is a common observation, that over the course of a few days, fresh ice cores do in fact loose fluid. We, therefore, decided to perform a few tests. The tests were done using real 10 cm full ice cores, 55 cm long (Figure 3), taken from a depth of 400 m at the NEEM “North Greenland Eemian Ice Drilling” deep drilling site in Greenland.

The ice cores were initially weighed as dry cores. The average weights were about 3800 g, in agreement with the ice density, accounting for some bubbles and porosities. The scale used for weighing, had an uncertainty 0.3 %, which for a full core means $\pm 1.1 \text{ g}$. The cores were then soaked in Estisol™ 140 and wiped with paper towels to mimic the fluid removal during drilling by the drillers with vacuum-cleaning and wiping down. Then the cores were weighed again to measure the initial amount of drilling liquid. The weighing was repeated after 8.5, 39.5, 46.0, 95.0, 121.0, and 145.5 h. We assumed that the difference in weight represented the residual amount of drilling liquid. Between the measurements, some cores were kept in a ventilation chamber, some in a closed box and some were positioned in a core trough and some were resting on open supports. Additionally, tests

were done at different temperatures: $-18\text{ }^{\circ}\text{C}$, $-30\text{ }^{\circ}\text{C}$, $-50\text{ }^{\circ}\text{C}$. A sketch of the experimental configurations is shown in Figure 3.



Figure 3. Configurations for the experimental tests on real 10 cm ice cores, 55 cm long.

Figure 4 shows the residual amount of EstisolTM 140 as a function of time for the different drying conditions. The reason for the high initial value of 14 g residual fluid of the $-50\text{ }^{\circ}\text{C}$ test is that the fluid at this temperature has significantly higher viscosity and, therefore, the ice core is able to retain more fluid at the initial soaking. We note, that after 46 h all the curves reach a plateau, and from then on the variations fall well within the uncertainty on the single measurements, which we estimate to about 1 g, which is shown as error bars in the figure. For the samples kept at $-18\text{ }^{\circ}\text{C}$ and $-30\text{ }^{\circ}\text{C}$, we compared a sample stored in a ventilated chamber resting in a trough with a sample stored in a closed box and resting on open supports. In contrast with the previous theoretical discussion, we find that the rate of fluid loss and the final amount of residual fluid does not show a strong temperature dependence. We also see that the influence of ventilation is less than the influence from the choice of core trough or not in removing the residual amount of fluid. The fluid loss is much faster when the ice cores are on open supports, as shown by the much higher lapse rate for open supports in both the $-18\text{ }^{\circ}\text{C}$ and the $-30\text{ }^{\circ}\text{C}$ experiments. As we examined the samples in the core troughs, we noticed that fluid had collected in the bottom of the troughs. We took that as an indication of fluid loss by run-off and dripping, which is highly probable as the fluid has low viscosity and has low surface tension, cf. bio-diesel or low viscosity vegetable oils. We also think we can explain the different plateaus for troughs and open supports. Along each side, the core rests against the trough. The lines of contact act as gutters for the fluid running off the top of the core, and we observed two stripes of excess fluid along the lines of contact as we removed the cores. Therefore, in agreement with theoretical calculations, we can infer that evaporation of the drilling liquid is not a first order effect. Gravity driven run-off is the most significant process in fluid loss. Due to the oily nature of the fluid, drip-drying continues for three days and leaves behind a residual film of approximately 2 g per core section. Drip-drying does not require expensive ventilation systems or higher temperatures, which could compromise the scientific quality of the ice cores. However, if cores are placed in troughs, we suggest placing the troughs in a shelf system with a slope to allow for the fluid to run off along the axis of the core. Although the tests have been simple, they clearly show that the effect of gravitational run-off is the most dominant in fluid loss even after several days in storage. Our tests were not designed to capture such small fluid losses by evaporation or of ice by sublimation as was calculated in Section 2, but our tests confirm that these effects indeed fall well below the one-gram sensitivity of the tests. Experience with ice cores from Greenland drilled with the same fluid has shown us that after one year storage in an open shelf in a snow cave, the cores appear “dry” but for a thin film of fluid. Therefore, some mechanical

decontamination will have to be performed to avoid undesired interferences in chemical analysis, as reported by Warming et al. [19].

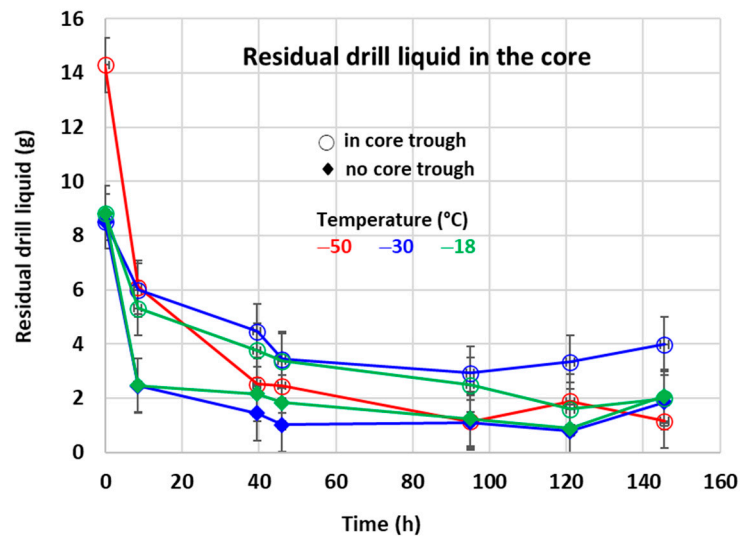


Figure 4. Amount of drilling liquid in the ice cores as a function of time.

3.2. Longlasting Preservation

The conservation of ice cores is fundamental for preserving their precious information for future studies. Ice cores are stored in refrigerated rooms. In some storage facilities the cores are placed on shelves in cardboard “tubes”, in others on shelves in cardboard boxes and in others inside insulated crates on shelves. From previous analysis, the only driver for ice sublimation is the different partial pressure of water vapor at the ice surface and in the ambient air. If RH is 100%, then a temperature difference between the ice and the air is needed to drive sublimation. The air in a freezer/cold-room is close to saturated (RH 100%) because when the door is opened, warm moist air is introduced. The excess moisture condenses in the freezer units and most of it is subsequently physically removed through drainpipes by the de-frost cycle of the freezer. However, some of the vapor is re-introduced into the freezer air during de-frosting because the units are heated. So, when de-frost is over and freezing resumes, that air becomes supersaturated and the surplus vapor condensates again in the freezer units. RH of close to 100% is quickly restored. In reality, the air in the freezer is slightly under-saturated, because for the cooling units to work, they are typically $-35\text{ }^{\circ}\text{C}$ in a $-30\text{ }^{\circ}\text{C}$ cold room.

So why do we see sublimation of ice cores in cold storage, particularly when they are forgotten on worktables? The answer is the transient temperature chock of the de-frost cycle. Depending on the freezer and season (moist summer air or dry winter air) defrosting is required at rates from twice a day to several times a week. The cooling is stopped and the air is heated up to melt the ice in the evaporators. This creates a temperature difference between ice and air, and the ice core warms up while some condensation frost is formed on the surface. When cooling resumes, the core is now warmer than the air, so there will be sublimation. However, the ice that is sublimating is not the same as was condensed. Observations are in agreement with what is predicted by a diffusion approach [6] with stronger sublimation at the sharp edges which become rounded.

If the exposed ice core is stored inside a plastic bag, the net transport of vapor through the bag is zero. However, we still observe that the edges of the core become rounded and we see a significant amount of frost forming on the inside of the bag, indicating a net transport of ice from the core to the inside surface of the plastic. Over time, this process significantly decays the quality of ice cores.

That is why ice cores should never be exposed to defrost cycles (or any changes in temperature) by storing them in, e.g., cardboard boxes or tubes. EPICA and BEOI have chosen to store all cores inside the same insulated boxes that are used for shipping ice cores

from the field to the freezer and around the world. Because of the heavy insulation, the cores are not experiencing the defrost cycles and the temperature is almost constant. Just recently, in the Copenhagen storage, we observed perfect conservation of ice cores 40 years after drilling. The samples were in mint condition with sharp edges and no frost on the inside of the plastic, providing a strong and clear experimental confirmation that the only driver of sublimation in a cold room is temperature oscillations.

Of course, if the cores are stored in a snow cave (very stable temperature), the snow walls will ensure RH of 100%, so no sublimation will occur as soon as the ice cores reach the same temperature as the cave. When people enter the cave, they bring in some outside summer air and they also emit water vapor through breathing. This will condensate as frost on both ice cores and walls. However, if a slow and gentle breeze of ventilated air is maintained through the cave to keep a $-50\text{ }^{\circ}\text{C}$ temperature of the ice cores, then all excess water vapour will condensate on the snow walls (at $-55\text{ }^{\circ}\text{C}$) and the cores will be unaffected. The advice is to maintain an ice core temperature that is slightly higher than the cooling surface and keeping it constant.

4. Conclusions and Perspectives

The present analysis was focused on the evaluation of a suggested ventilation process at the drill site for BEOI for removing EstisolTM 140 drilling fluid from ice cores, while preserving the ice quality and avoiding ice losses. We have discussed: (1) the physical processes of ice sublimation and drilling fluid evaporation under ventilation conditions; (2) calculations of how much ice could be lost and how much drilling fluid could be removed at maximum ventilation; (3) simple tests of fluid removal, and (4) how to optimize the storage conditions of ice cores to preserve their scientific usefulness even after long time in storage. By going through a theoretical physical description of ice sublimation and fluid evaporation as well as kinetic effects and diffusion, we obtained some quantitative estimates on loss of fluid as functions of temperature, partial vapor pressure and ventilation. Our estimates indicate that, under the suggested operating conditions in the ventilated room, no significant weight loss of ice is expected, particularly if inlet air is taken from a reservoir in the firm so that the air is already saturated by water vapor. Our calculations also indicate that even if the ventilated air has lower relative humidity, the ice losses (during one week in storage) remain negligible relative to the total mass of ice core material. Our estimates on EstisolTM 140 fluid also indicate a limited effect on fluid removal in the ventilated room. Even after a full week of $40\text{ m}^3/\text{h}$ ventilation, the maximum amount removed by evaporation is estimated to be less than 3 g (about 3 mL) for all the planned 200 ice cores stored in the room. To check that our theoretical estimates were not severely flawed, and that we did not leave out important processes, some simple experimental tests were done. Our one-week tests failed to reveal different patterns at different temperatures, so temperature appears not to be a prime factor in affecting the rates of fluid removal and the final amount of residual liquid. Our tests also failed to reveal differences between ventilated and non-ventilated cores. However, we did discover that most of the liquid is removed by gravitational run-off or dripping, and that storage in core troughs slows down the run-off process. Based on this, we recommend that cores and core troughs in the drying room at BEOI site are stored with a slight inclination along core axes. Moreover, we do not recommend ventilation as a means to speed up the drying process as ventilation both in calculations and in our tests has very little effect. Heating up the storage room to speed up the drying process is also not recommended, as the effects of temperature are small and potential consequences for ice core quality are large.

Finally, for long-term storage of the BEOI ice cores, it is highly recommended to avoid exposing the cores to temperature oscillations such as in defrosting cycles of freezer units, otherwise sublimation effects will occur. Sublimation effects are particularly significant at sharp edges, in agreement with practical experience and theoretical predictions obtained by a diffusion approach. Sublimation effects can be almost completely avoided by keeping the cores inside insulated crates in the freezer. The insulation of the crate and the heat capacity

of 40–60 kg ice in combination prevent the ice from experiencing short term temperature pulses such as de-frosting cycles or human activity. This approach has retained the scientific quality of ice core samples, even after 40 and 55 years in storage.

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