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LABORATORY PREPARATION OF ARTIFICIAL
SEA AND SALT ICE

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PREFACE

This report was prepared by Dr. W.F. Weeks, Glaciologist, of the Research Division, U.S. Army Cold Regions Research and Engineering Laboratory (USA CRREL), and G.F.N. Cox, of the Earth Sciences Department, Dartmouth College.

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LABORATORY PREPARATION OF ARTIFICIAL SEA AND SALT ICE

by

W.F. Weeks and G.F.N. Cox

INTRODUCTION

Our understanding of the details of the variations in the physical and chemical properties of sea ice is largely based on studies of artificially grown sea and salt ice. There are several reasons for this, the most important of which is that natural sea ice is very difficult to store without the occurrence of significant brine drainage. This brine drainage results in changes in the original ice properties that are usually undesirable. Additional complicating effects that may occur during natural sea ice growth, such as the formation of slush ice, infiltrated snow ice and underwater ice, can be avoided in the laboratory. Furthermore, with the laboratory preparation of sea ice, one can control variations in the growth conditions in such a way as to enhance the particular aspect of sea ice that is under study. Finally, it is expensive and many times logistically difficult to send investigators to the field to study sea ice. This is particularly true during the initial period of ice growth when offshore operations usually require either ship or aircraft support. Therefore, it is not surprising that the production of artificial sea and salt ice in coldrooms, where the temperature, composition and structure of the ice can be partially controlled, is attractive.

The following is a discussion of several successful techniques that have been used to grow artificial sea and salt ice. In addition some of the difficulties associated with developing suitable experimental designs for investigating a variety of specific sea ice problems are described.

FREEZING ARRANGEMENTS

The first requirement in simulating the growth of natural sea ice is to achieve one-directional freezing. Fortunately this is rather easy to accomplish. Figures 1 and 2 show freezing chambers that have worked quite successfully. The first was used to produce large salt ice samples for mechanical testing (Weeks 1962). After the tank was filled with an NaCl solution it was placed in a coldroom set at a constant temperature (usually -20°C) where it remained until the desired ice thickness was achieved. Both the sides and bottom of the tank were insulated and resistance heating elements were placed within the insulation to cancel out the bottom and side heat losses.

The freezing chamber shown in Figure 2 was used to produce small sea and salt ice samples for structural and chemical studies (Weeks and Lofgren 1967, Lofgren and Weeks 1969). The cold source was a copper cold plate through which a coolant was circulated from a constant temperature bath. Although the sides of the plastic freezing tube were insulated, the exact nature of the insulation was not critical because the ambient coldroom temperature could be set just above the freezing temperature of the sea water. Any significant deviation from one-dimensional freezing could easily

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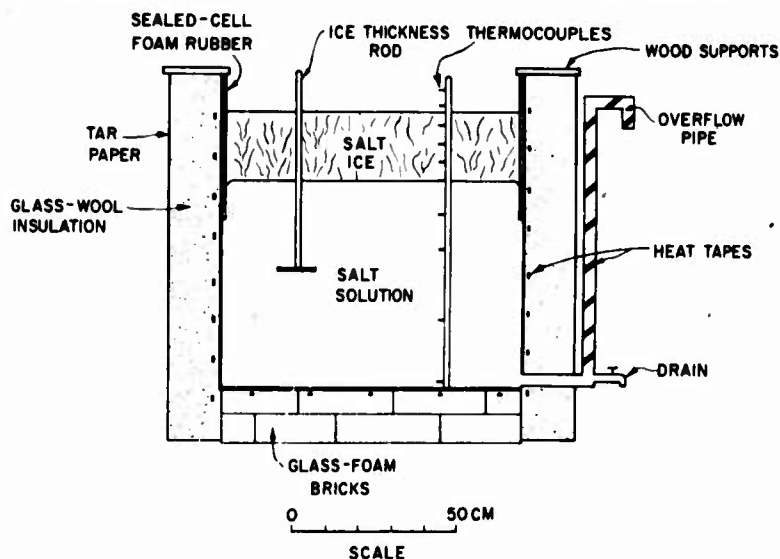


Figure 1. Freezing tank used to produce NaCl ice for mechanical testing (Weeks 1961, 1962).

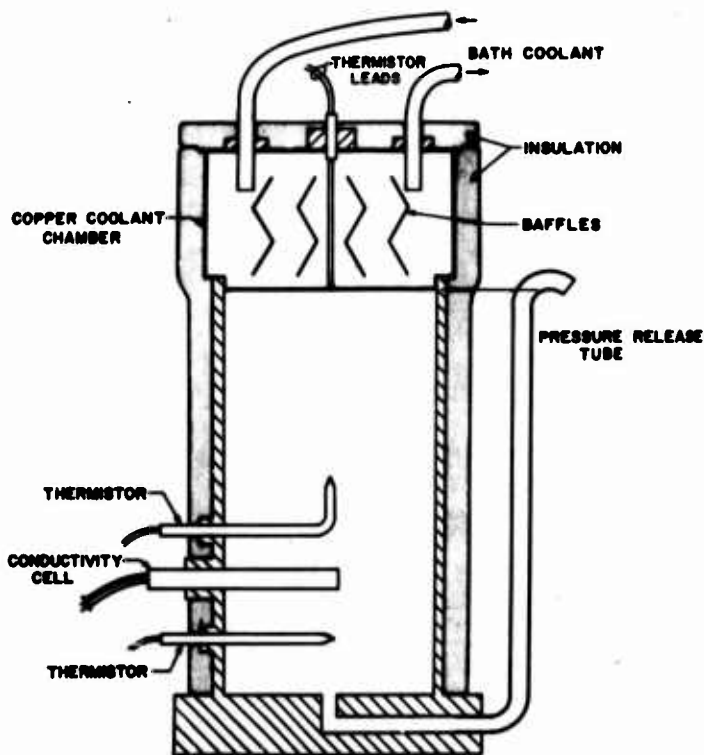


Figure 2. Freezing container used to produce sea and NaCl ice for structural and chemical studies. The diameter of the inner plastic tube was 15 cm (Weeks and Loigren 1967, Loigren and Weeks 1969).

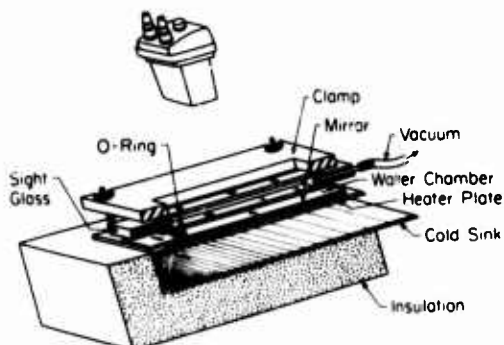


Figure 3. Freezing apparatus used by Harrison and Tiller (1963) to study the structural characteristics of growing salt ice.

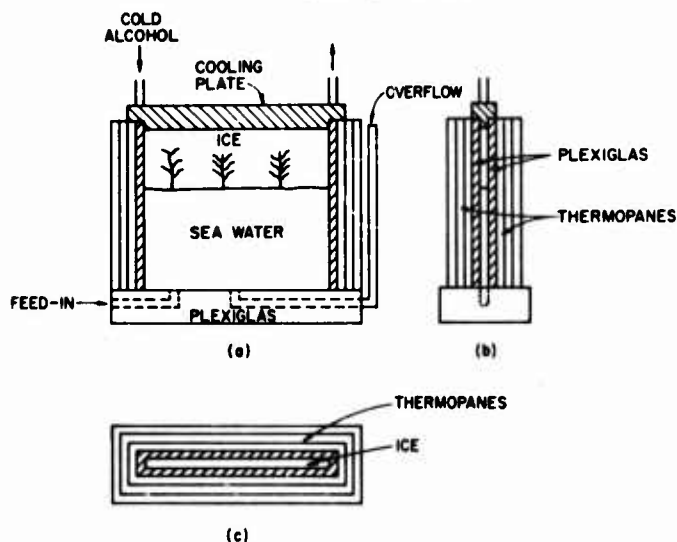


Figure 4. Freezing apparatus used by Elde and Martin (1974) to investigate brine drainage from growing sea ice.

be detected by examining the shape of the solid/liquid interface. Ideally the interface should be planar. Appreciable heat losses or gains through the container walls will cause the interface to be concave downward or upward respectively. In most freezing runs we have tried to adjust the insulation and the coldroom temperature so that the ice at the edge of the freezing container is slightly thicker than that in the center of the container. The ice around the edge can then be discarded when samples are collected.

An interesting small freezing compartment, which allows a detailed examination of the morphology of the solid/liquid interface during freezing, has been used by Harrison and Tiller (1963 a, b). In their experimental arrangement (Fig. 3) the growth direction was horizontal, similar to experimental arrangements commonly used with metals and ceramics. In addition, Elde and Martin (1974) have successfully grown sea ice between narrowly separated sheets of insulated glass (Fig. 4). The purpose of this design was to allow visual observation of the brine drainage tubes.

When clear plastic containers are used, ice growth information can be obtained by visually measuring the location of the ice/water interface as a function of time. If visual observations are not possible, ice thickness can easily be determined by using a thin, hollow metal tube with a crossbar at the end (Fig. 1). Inside the tube is an electric resistance heater. When an ice thickness is required, the heater is turned on until the bond between the ice and the metal surface of the rod is broken. Then the rod is pulled up until the crossbar encounters the lower surface of the growing ice sheet. To obtain growth velocities at any vertical location in the ice sheet, the empirical equation

$$t = a_1 h + a_2 h^2 + a_3 h^3 + a_4 h^4$$

where t is time and h is ice thickness can be fitted to the data by least-squares. In the freezing runs studied by Lofgren and Weeks (1969) the lowest correlation coefficient obtained on their fitted curves was 0.9991. Growth velocities at any h are then obtained by differentiation. A discussion of the theoretical basis for this general procedure is given in Lofgren and Weeks (1969). Recent work by the authors indicates that this relationship breaks down at lower growth velocities ($< 10^{-3}$ cm/sec) where the ice thickness approaches a constant value. Under these circumstances, the growth velocity is best determined directly from the ice thickness-time curve.

In the freezing apparatus shown in Figures 1 and 2, the volume expansion on freezing was allowed for by constructing a pressure release tube. Gold (1963) has also used air-filled balloons (or a small inner tube) positioned on the bottom of the freezing chamber and attached to a tube submerged in a column of antifreeze that connects to the outside of the freezing chamber. As the ice grows, the pressure build-up caused by the volume expansion is countered by the release of air at a fixed depth in the antifreeze solution. Although this latter method does not appear to have been used in sea ice experiments, it has an advantage in that solute would not be lost through the pressure release tube.

In some experiments, such as those by Rohatgi and Adams (1967a, b, c), Kvajfc et al. (1971) and Kvajfc and Brajovic (1973), the cold source has been located on the bottom of the liquid column and the ice grown from the bottom upward. For many purposes such arrangements are excellent in that they greatly simplify the analysis of the solute distribution in the liquid phase during ice growth. When ice grows upward the solute distribution in the liquid produces a stable vertical density distribution (the colder, more saline solution near the interface is denser than the warmer, less saline solution above it). Therefore, concentration changes are controlled by simple diffusion, a physical situation that has been extensively analyzed (Elbaum 1959, Tiller 1963). If, however, one wishes to simulate the conditions that occur during the growth of natural sea ice, freezing should proceed from the top downward. Convective mixing will then occur in the liquid and quite different amounts of salt will be trapped within the ice (Kvajfc and Brajovic 1970).

In the design of a freezing apparatus the length of the liquid column below the ice is small compared to most natural situations, where it effectively may be considered infinite. Therefore, during artificial freezing runs there is a gradual increase in the salinity of the underlying solution as ice growth proceeds. This increase, of course, also occurs in nature but the effect is so small that for most purposes it can be neglected. During artificial freezing it is probably a good rule of thumb to never let the ice thickness exceed half the length of the freezing tube. A rough estimate of the salinity of the solution at any stage of the growth of ice can be obtained from the salinity profile by using the relations given in Weeks and Lofgren (1967). This approach assumes that during the period of ice growth brine drainage is negligible. A detailed analysis of this general problem, referred to as the "terminal transient" in the crystal growth literature, is given in Smith et al. (1955) for the case where the solute transport in the liquid is diffusion-limited.

We know of no simple method for keeping the composition of the freezing solution constant. Martin (1972) has utilized an experimental set-up in which the freezing solution was continuously replaced by a new solution of the desired initial composition. This, of course, requires the artificial circulation of the freezing solution. Unfortunately, unless considerable care is taken such an arrangement could easily introduce large amounts of heat into the system. Such effects may or may not be desirable. In the future it might be possible to utilize salt exchange through a membrane to maintain a constant solution composition below growing ice.

Whatever the details of the freezing arrangement, it is important to describe the growth conditions accurately by measuring temperature profiles in both the ice and the underlying solution. This can easily be done by positioning closely spaced thermistors or thermocouples within the freezing chamber. The composition of the freezing solution should also be measured at several different locations by the use of conductivity cells or probes. A special microprobe capable of detailed salinity measurements near the freezing interface has been described by Terwilliger and Dizio (1970). Unfortunately these devices are not capable of measuring salinities within the ice. At present, the only way to measure ice salinities without destroying the sample during the analysis is by utilizing radioactive tracers such as ^{22}Na . However, the procedures and instrumentation required for such measurements are so involved that these techniques can only be used in special studies. The addition of tracers may also make utilization of the ice quite difficult because of the radiation hazard. If the solution beneath the growing ice is well mixed compositionally because of convection, then knowing the temperature of the solution is equivalent to knowing its composition because from the phase rule the system has only one degree of freedom (assuming constant pressure). In addition, if we are dealing with a closed system and we know the initial composition of the freezing solution and the amount of ice and solution concentration at a given time, the average ice salinity can easily be calculated for that time. For many purposes this information is more than adequate.

THE FREEZING SOLUTION

The simplest way to prepare standard sea ice would appear to be by freezing "standard" sea water. Inasmuch as "standard" sea water costs roughly \$15/liter, this procedure is unduly expensive when significant volumes of water are required. If the laboratory is near the ocean, sea water can be trucked in at a reasonable cost. However, coastal surface water is generally less saline than water from the open ocean because of the diluting effect of freshwater runoff. Because surface water salinities from polar areas lie in the range 30.0 to 33.5 ‰, this is probably not a serious problem, assuming that the salinity of the sample is specified.

A discussion of the phase relations and brine volume variation in standard sea ice is given by Assur (1958). Simplified relations for computing brine volume from temperature and salinity based on Assur's brine volume table are given by Frankenstein and Garner (1967). In certain physiochemical studies of sea water, it may be advantageous to utilize a simplified reproducible solution of a known composition in place of true sea water. A recent recipe for preparing artificial sea water with a salinity of 35.00 ‰ is given by Kester et al. (1967). This recipe is essentially an update of the commonly used recipe of Lyman and Fleming (1940). In calculating brine volumes for sea ice grown from artificial sea water, one must assume that the phase relations discussed by Assur (1958) still apply. At the present time, we have no reason to believe that this assumption is significantly in error.

One-component solutions have also been used to produce ices that are structurally similar but chemically simpler than sea ice. The most obvious example is NaCl ice (Weeks 1962, Weeks and

Lofgren 1967, Lofgren and Weeks 1969). This ice has the advantage of being a simple ice + brine mixture between the freezing temperature and -21.2°C , and at lower temperatures, a mixture of ice + solid salt ($\text{NaCl}\cdot 2\text{H}_2\text{O}$). The phase relations and brine volume tables for NaCl ice are given by Weeks (1961). In addition KCl, LiCl and HCl solutions have been used (Rohatgi et al. 1969, 1974).

ICE CHARACTERISTICS

Ideally, one would like to be able to grow sea ice with any desired combination of properties. However, the independent variation of some specific characteristics of sea ice may prove to be impossible, simply because many physical properties are coupled to certain growth conditions. For instance, high growth velocities cause high brine entrapment, high air entrapment, small substructure spacing and probably small grain size. In the following discussion we will briefly describe how one could control certain of the more important characteristics of artificially grown sea ice.

Salinity

The most extensive study of salt entrapment (NaCl ice) is by Weeks and Lofgren (1967). Other works of interest are Tsurikov (1965), Kvajčić and Brajović (1970) and Fertuck et al. (1972). The basic conclusions of these studies are similar: the salinity of the ice produced increases with the salinity of the freezing solution and with higher growth velocities. The relations suggested by the different authors vary and, with the exception of the work of Kvajčić and Brajović (1970), who used a tracer to make direct measurements of the effective distribution coefficients, the observed coefficients are probably lower than the true values because of brine drainage. Nevertheless, the relations should allow one to make a good estimate of the possible combinations of growth velocity and solution composition that will produce ice of a desired salinity. It should be noted that, at the present, the majority of the experimental observations on solute partitioning as a function of growth conditions are based on freezing NaCl solutions, not sea water. In the one study that used sea water (Tsurikov 1965) the major ions in the ice were found to be present in the same proportions as in the water, with the exception of Ca^{++} which was concentrated in the ice.

Grain size and orientation

There are no detailed studies of grain size variations in artificial sea ice and only a limited amount of data are available for natural sea ice. Observations suggest that, in general, the principal variable controlling the mean grain size is the grain size of the initial ice skim. Once the initial skim has formed, the mean grain size increases linearly with depth (Weeks 1968). The initial grain size may certainly vary widely under natural conditions and we would be rather surprised if similar variations did not occur during artificial freezing runs. Thus, if reproducible grain size is important in artificial sea ice samples, one should consider seeding the surface water layer at the time of first skim formation (Gold 1963, 1965), thereby controlling the grain size of columnar-grained freshwater ice. The water is allowed to cool until a first skim forms, this ice is then removed and the water surface is seeded with a liberal amount of finely crushed ice. This seeding procedure produces a random crystallographic orientation in the initial skim. This is compared to many growth situations where the major portion of the skim is composed of crystals oriented with their c-axes vertical. An initial skim with a random c-axis orientation will probably make the transition zone* both thinner and of a more uniform thickness from freezing run to freezing run. A thin transition zone is desirable so that more of the specimen will consist of ice with a c-axis

* The layer just below the initial skim where geometric selection produces the c-axis horizontal fabric so characteristic of sea ice (Perey and Pounder 1968, Weeks and Assur 1968).

horizontal orientation. In many types of studies, the upper portion of the sample that includes the initial skim and the transition zone should be discarded and tests performed only on the underlying columnar zone ice. If the upper portion of the specimen is used, consideration should be given to documenting its fabric.

Substructure

The only aspect of the sea ice substructure that has been studied as a function of growth conditions is the plate width a_0 (the distance between the midpoints of the intercellular grooves measured parallel to the c-crystallographic axis). Rohatgi and Adams (1967a, b, c) examined the variations of a_0 as a function of growth velocity v and solute concentration C in simple salt solutions frozen from the bottom upward. They found that a_0 consistently decreased linearly as a function of the distance from the constant temperature cold plate. This corresponds to an inverse linear relation between a_0 and v . They also observed an increase in a_0 as a linear function of C .

In contrast, Lofgren and Weeks (1969) froze their samples from the top downward in a manner similar to the growth of normal sea ice. They obtained rather different results depending upon whether or not convection occurred in the liquid. When convection occurred, the slope of log-log plots of $\bar{a}_0$ vs v showed a change from $\frac{1}{2}$ at high values of v to approximately 0 at low velocities. This variation was believed to be the result of convection reducing the effective value of C at the growing interface. The variation of $\bar{a}_0$ with C appeared to be quite complex and showed a minimum in the composition range 9 to 25 ‰. These results suggest that until the variation of a_0 with growth conditions is better understood during downward freezing, investigators who wish to control a_0 should design their experiments to freeze from the bottom upward.

Other aspects of the sea ice substructure include the geometry and spacing of air bubbles and brine pockets as well as the overall geometry of the intercellular grooves. However, there is currently no information available on these subjects

Single crystals

There have been no documented attempts to grow large single crystals of sea ice. However, if one starts with a seed crystal of freshwater ice and continues one-dimensional growth from the seed into either sea or salt water, the new ice will have the same orientation as the seed but will possess a typical sea ice substructure. This fact could presumably either be used directly, or indirectly via a modified Bridgman method (Chalmers 1964), to produce large sea ice single crystals of any desired orientation. In approaching a somewhat similar problem Kvajčić and Brajović (1973) utilized growth from single and bicrystal seeds of fresh ice to study growth selection in the sea ice single and bicrystals that formed through subsequent growth.

Underwater ice

The formation of underwater ice has not been studied experimentally in any detail. This ice type commonly forms late in the growth season, and in certain localities in the Antarctic comprises a major portion of the ice sheet. A review of our current understanding of the underwater ice problem can be found in Lewis and Lake (1971) or Lewis and Weeks (1971). The ice type is a mixture of dendritic plates, apparently formed as a growth extension of the overlying congelation ice, and ice spicules and plates that formed deeper in the water column and subsequently floated into position at the bottom of the ice sheet. It is very fragile and difficult to transport. Its occurrence appears to require the removal of all sensible heat from the underlying water column plus the convective transfer of cold brine plumes from brine drainage channels (Lake and Lewis 1970, Dayton and Martin 1971). Lewis and Lake (1971) have reported the formation of underwater ice in a circular insulated tank of sea water after the water column under the congelation ice had reached the

freezing point. They suggest that if heat extraction is sufficiently slow so that all the sensible heat is removed from the water column before the ice cover forms, underwater ice will grow immediately at the water surface. Therefore, to grow underwater ice great care should be taken in the initial cooling of the freezing solution.

If it is desired to relate crystal formation in the lower portion of the water column to the location of brine drainage tubes in the ice, then a fairly wide freezing container should be used. Currently available information (Lake and Lewis 1970) from natural sea ice indicates that, on the average, one large brine drainage channel occurs per 180 cm² of interface, giving a mean distance of 13.4 cm between channel centers. Therefore a sufficiently large container should be used to assure that there will be a reasonable number of drainage channels active in the system under study.

CONCLUSION

In this report we have given the reader a number of suggestions on how to grow artificial sea and salt ice. We believe that the techniques and problems that have been discussed will assist investigators in circumventing a few of the problems that must be faced when one attempts to simulate the growth of natural sea ice in the laboratory.

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