Model K29iX

Direct Expansion Immersion Cooler

for Triple Point of Water Cells

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This report documents the operation and maintenance procedures for the Pond Engineering Model Number K29iX Direct Expansion Immersion Cooler. The Immersion Cooler is designed to form even, long-lasting ice mantles in TPW Cells for the realization and maintenance of the “TPW temperature” for extended periods of time. It is suitable for use with virtually all commercially produced TPW cells.

Information contained in this manual is considered by Pond Engineering Laboratories to be proprietary and is provided for use exclusively by the purchaser for instructional and maintenance purposes relative to the hardware. Any other use is prohibited. The apparatus described in this manual is in the process of obtaining utility patent protection.

1 Background Information

1.1 System General Layout

Figure 1, below, shows the general configuration of the Direct Expansion Immersion Cooler. Also shown are the locations of major components discussed later in this manual.

As shown in Figure 1, the Immersion Cooler consists of a base unit with a probe connected by a flexible tube. The Probe and connecting tube are intended to be stored in pockets formed in the back section of the base unit.

The probe section of the system is designed to uniformly remove heat from the cell along its entire length, ensuring even ice mantle formation along the length of the probe. At the lower end of the probe is a removable contact-enhancing brass tip. This produces added thickness at the bottom of the center well, lengthening mantle life.

1.2 Theory of Operation

In operation, the base unit is fitted with a refillable carbon dioxide cylinder with an integral pin valve assembly. The cylinder is used in the inverted orientation (valve end down) so that liquid is drawn from the cylinder. As the mechanism in the expander base is actuated to OPEN, the pin valve is actuated and liquid carbon dioxide is allowed to flow through a stainless steel tube contained in the flexible plastic jacket to the probe section where it is allowed to expand and absorb heat as it vaporizes. Gaseous carbon dioxide is then vented through a second tube contained in the flexible plastic jacket back to the base unit where the gas is vented to atmosphere. Since there is a fixed amount of carbon dioxide available to the system, only a fixed amount of ice can be formed by the system with a single cylinder of carbon dioxide. Use of a 20 ounce cylinder is recommended to form ice mantles of good thickness in Type A or Type B cells.

1.3 General Operating Considerations

The density of water as a function of temperature passes through a maximum at about 4°C. As a result, the warmest water in a cell is often at the bottom when the temperature is reduced below 4°C. (This effect is commonly observed as lakes always freeze at the surface first.) Since the water in TPW cells lacks impurities necessary for easy crystal nucleation, the water can be subcooled (below 0°C) without producing any ice crystals. When the temperature is reduced to approximately -10°C, crystals will nucleate spontaneously. However, the cell can be maintained at –3 to -5°C until the user initiates crystal formation. Starting with a subcooled cell reduces the amount of heat which must be removed from the water to form an ice mantle and significantly improves temperature uniformity within the cell.

Ice crystal formation in a subcooled cell is self limiting. As the crystals form, the heat of fusion (approximately 80 cal/gm) is released into the water causing the water temperature to rise. As a result, crystal formation automatically stops when the temperature rises above the triple point temperature. The resulting crystals form a loosely-connected, relatively open network and will not damage the cell. Despite its appearance, less than ten percent of the water is typically crystallized. The advantage these ice crystals or slush provide is primarily in their ability to locally absorb or release heat anywhere in the boy of the cell. The crystals ensure that the cell water is uniform in temperature and therefore is of uniform density. When heat is absorbed into the cell, the "slush" in that area melts and then redistributes itself, virtually eliminating temperature gradients.

Alcohol can be used to enhance thermal contact between the immersion cooler probe and the center well of the TPW cell. Methyl Alcohol is recommended because it remains liquid at low temperatures (liquid carbon dioxide and dry ice temperatures). Alcohol can also be used, to some extent, to direct the ice formation. A few drops in the bottom of the well results in ice formation primarily around the end of the well. Uniform ice formation along the entire length of the Cooler shaft can be accomplished by filling the well with alcohol to just below the water level in the cell. Ensuring that the alcohol level is below the water level minimizes the risk of ice bridging at the top of the cell.

1.4 Protecting your cell from breakage

It is possible that, during the formation of the mantle, an ice bridge may form between the thermowell and the cell outer wall. This is unusual under normal operating procedures. If liquid water can be seen between the cell wall and the bridge, or if water can move through the ice bridge (it is not solid enough to hold the water in), this is not a dangerous condition. However, if a solid bridge does form, and significant pressure builds in the cell body due to expansion as the ice mantle forms, the bridge must be melted immediately. The heat from one's hands, along with a continued gentle agitation, should be enough to melt the bridge. The user should be aware of the possibility of breakage if the Cell is not properly attended.

2 Building a Mantle

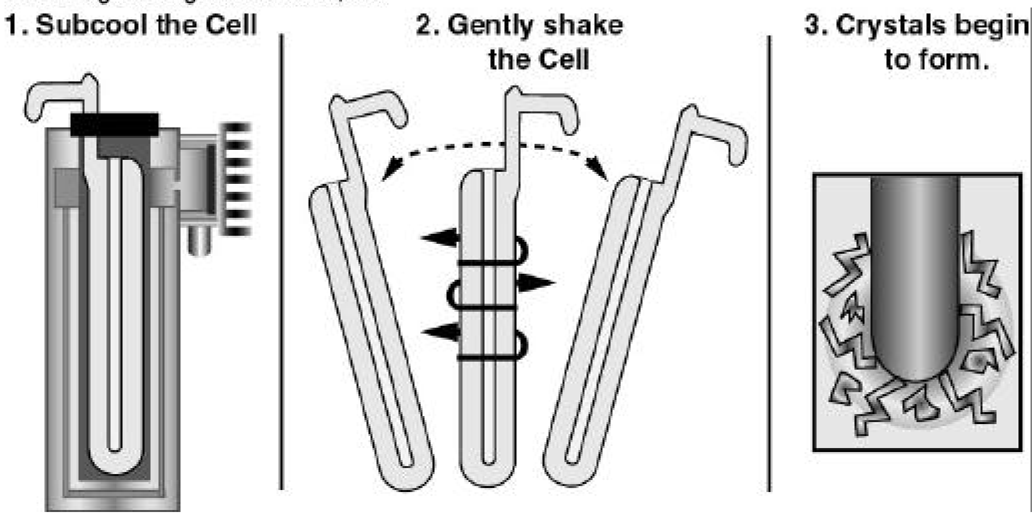
The Triple Point of Water is realized when a mantle of ice is surrounded on all sides by liquid water and above by water vapor. Calibrations can only be precise when the mantle is uniform and intact. Thus, a good ice mantle is essential in realizing and maintaining a uniform phase change plateau with any water triple point maintenance system.

There are several methods and numerous variations that can be successfully used to form a mantle. The following is a simple, straight-forward procedure which produces excellent results when used in a K29M maintenance system. Several other methods and variations are discussed later in this manual. Users may need to try several variations to determine which method is most suitable for their particular set of equipment and lab conditions.

2.1 Forming First Ice crystals

The first step in mantle formation is to initiate the crystal nucleation. First, the Water Triple Point Cell should be subcooled to approximately -3.0 to -5.0°C. This can be easily achieved using the Pond Engineering Model K29M Water Triple Point Maintenance System or any other comparable system. In a Pond Engineering K29M Maintenance System, a room temperature cell can be completely subcooled in approximately 24 hours. Once the cell stabilizes in the above mentioned temperature range, the crystals can be formed at any time.

Crystal formation can be initiated in numerous ways. The subcooled cell can be tipped on its side and returned to an upright position semi-abruptly, creating a localized disturbance to nucleate the crystals. Or, the cell can simply be shaken gently, side-to-side, until crystals begin to form (see Figure 3). This method perhaps entails less risk of cell breakage. Once nucleation occurs, the crystal formation occurs quite rapidly and, despite its appearance, will not damage the cell. The ice is formed in a very open, loose network and is self limiting.



Agitation Method

2.2 Building the Ice Mantle

Once the first ice crystals have been formed, the mantle is ready to be built. To begin, insert the Immersion Cooler probe fully into the center well of the cell and add the appropriate amount of alcohol. In order to build a mantle of uniform thickness along the full length of the probe, the well should be filled to just below the level of the water.

IMPORTANT NOTE
The alcohol provides improved thermal contact along the entire length of the probe. Without the alcohol, mantle formation may be very irregular and may result in significant ice build up in a limited area (typically the bottom of the well). If the mantle becomes large enough to touch the outer walls, or if an ice bridge forms from the inner well to the outer walls of the cell, the force of the expanding ice may break the cell.

The next step in mantle formation is to fit a full 20 oz. cylinder of carbon dioxide to the expander base (be sure that the FLOW CONTROL lever to the CLOSE position when fitting the cylinder to the base) and start the flow by moving the FLOW CONTROL lever to the OPEN position. The ice crystals formed earlier (See Section 2.1) will allow formation of a solid ice mantle adjacent to the thermometer well as the probe removes heat from the cell. The ice crystals will also act as a heat buffer during mantle formation as they melt and absorb heat migrating into the cell from the surroundings. Best results are achieved if the mantle is allowed to form in a K29M (set to approximately -0.25°C) or equivalent maintenance system.

At this point, the mantle should begin to form and should require no particular supervision. The ideal mantle is between 4 and 8 mm in thickness. Normally, one full cylinder will provide a mantle thickness within the desired range when it is expended. Additional thickness can be formed easily but should be done with care. To build the mantle further, the empty cylinder may be removed, the lever moved to CLOSED position and another cylinder fitted to the expander base and the process continued. It is recommended that smaller cylinders (7 oz. or smaller) be used in this operation so that small quantities of ice are formed by the time the cylinder is expended and the possibility of damaging the cell is minimized. Until the user has gained some experience with the rate of mantle growth and carbon dioxide usage, caution is encouraged.

NOTE

If the mantle becomes large enough to touch the outer walls, or if a solid ice bridge forms from the thermowell to the outer walls of the cell, the pressure generated by the expanding ice may break the cell. Bridging is most likely to occur at the top of the cell, but is possible in any area along the length of the thermowell. The cylindrical shape of the cell's exterior wall creates an optical distortion, making the mantle appear slightly thicker than it actually is. The mantle size and shape can be viewed with more accuracy by submerging the cell in a container of water with a flat side for viewing the mantle.

A problem in other cells, temperature gradients caused by the "density phenomenon" (the coldest water in the cell tends to float to the top, pushing warmer water to the bottom, causing ice to deteriorate) are virtually eliminated by the loose network of ice crystals formed earlier in the mantle process. These crystals act as a heat buffer and gradually melt as the cell's temperature stabilizes. The melting ice crystals distributed throughout the volume of the cell reduce temperature gradients and improve mantle growth uniformity.

2.3 Considerations When using Liquid Baths

Although the previous method has proven the simplest and most effective, there are many more ways to form a mantle in Triple Point of Water Cells. The alternate methods and variations covered in this manual include the use of a liquid bath which requires forming mantles in non-subcooled cells. In this case, the Immersion Cooler will function in the same basic fashion, however it is possible to subcool the water in the cell and remove heat from the bath quickly enough to form an ice layer on the outside of the cell body. This is due to the relative high contaminant concentrations in the bath water allowing nucleation of ice crystals on the cell outer wall before nucleation can occur in the high purity water inside the cell. If this condition is observed it will be necessary to make sure that the ice crystals are initiated inside the cell before attempting to form a mantle. If this is not done, the Immersion Cooler may be unable to form a mantle and may simply form ice on the outer wall of the cell.

The recommended procedure in this situation is to operate the Immersion Cooler normally for approximately 5 minutes in a chilled cell. This should cool the glass thermowell (some water immediately adjacent to the glass) below the Triple Point. The cell should then be agitated as described above to initiate crystal formation and the mantle then formed normally as outlined above.

2.4 Special Notes on usage with a cell in an Ice Pack

The Immersion Cooler will function in the same basic fashion with an ice pack, however since there are ice crystals (crushed ice in the ice pack) in contact with the outer cell wall it is not possible to significantly subcool the water in the cell. For this reason, the user should take care that the ice crystals are initiated inside the cell before attempting to form a mantle. If this is not done, the Immersion Cooler may be unable to form a mantle and may simply form ice on the outer wall of the cell.

The recommended procedure in this situation is to operate the Immersion Cooler normally for approximately 5 minutes in a chilled cell. This should cool the glass thermowell (some water immediately adjacent to the glass) below the Triple Point. The cell should then be agitated as described above to initiate crystal formation and the mantle then formed normally as outlined above.

3 Rebuilding a mantle

The K29I Immersion Cooler can also be used to rebuild a mantle. As has been indicated in previous sections of this manual, ice formation can be directed to some extent by the amount of alcohol used to improve thermal contact between the probe and the well. A few drops in the bottom of the well results in ice formation primarily around the end of the well. Uniform ice formation along the entire length of the Cooler shaft can be accomplished by filling the well with alcohol to just below the water level in the cell. Ensuring that the alcohol level is below the water level minimizes the risk of ice bridging at the top of the cell.

Rebuilding a mantle is virtually the same as building one in a completely melted cell except that it requires some examination of the existing mantle to determine the best rebuilding method. Due to the many different techniques for maintaining a Water Triple Point Cell, cells tend to melt in different fashions. If the mantle appears to be melting from the bottom, for example, it will be necessary to build ice on the bottom first. This can be accomplished by using the immersion cooler in the cell with only a few drops of methyl alcohol at the bottom of the thermowell. The additional thickness of ice at the bottom of the mantle should reform fairly rapidly since the thermal contact is greatest where the alcohol is present. Once the desired thickness has been formed, the remainder of the mantle can be rebuilt as usual. If the cell appears to be melting from the top, the mantle should be melted back slightly (as it is difficult to form ice only at the top of a cell) in order to improve the uniformity of the new mantle. The mantle can then be formed as usual, however care must be taken to ensure that the ice thickness does not increase to the point that the ice completely fills the cell envelope. This condition could trap a volume of liquid water under the forming ice mantle and cause breakage of the cell envelope as ice continues to form.

NOTE
If the mantle becomes large enough to touch the outer walls of the cell envelope, or if a solid ice bridge forms from the thermowell to the outer walls of the cell, water can be trapped in a closed volume within the cell. Hydrostatic pressure within the closed volume can be developed by further ice formation may break the cell envelope. Bridges are most likely to occur at the top of the cell, but are possible at any position along the length of the thermowell. It is important to melt back bridges immediately before continuing with mantle formation. The cylindrical shape of the cell’s exterior wall creates an optical distortion, making the mantle appear considerably thicker than it actually is. The mantle size and shape can be viewed with more accuracy by submerging the cell into a container of water with a flat side (or considerably larger diameter than the cell, perhaps 150mm diameter) for viewing the mantle thickness.

4 Guidelines for use of Cell

Due to the inherent physical properties of ice, mechanical strain in induced in the ice as the water is frozen. In general, the faster the ice formation, the greater the mechanical strain. The presence of mechanical strain has been shown to depress the triple point temperature on the order of 0.2 mK. Allowing the cell to anneal in a maintenance system for a period of approximately 24 hours is recommended before using the prepared cell for critical measurements. For additional information on mechanical strain issues and cell annealing, the user is referred to NIST Technical Note 1265 pg. 37.

Before calibration readings are taken, it is recommended that an additional layer of liquid (or inner melt) be established between the mantle and the thermowell. This inner melt can be easily achieved by inserting a room temperature glass rod into the well momentarily. To ensure that this inner melt has been formed, simply rotate the cell body or tilt the cell slightly off vertical (on the order of 30 degrees). The mantle should rotate freely inside the cell, pivoting around the thermowell. With this inner melt verified, the precooled SPRT can be inserted into the cell and, when it reaches thermal equilibrium, measurements taken for calibration purposes.

5 System Hardware

5.1 System Maintenance

The expander is designed to operate with standard refillable carbon dioxide cylinders, however care must be taken to ensure that the cylinders remain clean and are filled with clean carbon dioxide. Specific caution is made against filling the cylinders from larger bulk containers made of steel as these are prone to contaminating the carbon dioxide with particles. Indications of contaminated product are valves that do not seal and continue to leak after the filling cycle. It is likely that the expander will become plugged if the system is operated with contaminated carbon dioxide.

5.2 Component Descriptions

A brief description of system hardware is provided in this section as a reference to aid the user in periodic maintenance of the system. In the event of significant maintenance or repair is required, it is recommended that Pond Engineering be contacted prior to replacing or modifying major system components.

Ref#	NAME	MAKE/MODEL	
1	Probe Shaft	Pond Engineering	K29I0001A
2	Standard Brass Tip	Pond Engineering	K29IM001
3	Extended Brass Tip	Pond Engineering	K29IM201
4	Engraved Laminate Front Panel	Pond Engineering	K29IH001