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PHASE CHANGE LIQUIDS

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DISCLAIMER

ABSTRACT

Work is being performed to develop a new shipping system for frozen environmental samples (or other materials) that uses an optimal phase change liquid (PCL) formulation and an insulated shipping container with an on-board digital temperature data logger to provide a history of the temperature profile within the container during shipment. In previous work, several PCL formulations with temperatures of fusion ranging from approximately -14 to -20 ºC were prepared and evaluated. Both temperature of fusion and heat of fusion of the formulations were measured, and an optimal PCL formulation was selected. The PCL was frozen in plastic bags and tested for its temperature profile in a cooler using a digital temperature data logger. This testing showed that the PCL formulation can maintain freezer temperatures (<-7 to -20 ºC) for an extended period, such as the time for shipping samples by overnight courier.

The results of the experiments described in this report provide significant information for use in developing an integrated freezer system that uses a PCL formulation to maintain freezer temperatures in coolers for shipping environmental samples to the laboratory. Experimental results show the importance of the type of cooler used in the system and that use of an insulating material within the cooler improves the performance of the freezer system. A new optimal PCL formulation for use in the system has been determined. The new formulation has been shown to maintain temperatures at <-7 to -20 ºC for 47 hours in an insulated cooler system containing soil samples. These results are very promising for developing the new technology.

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EXECUTIVE SUMMARY

Soil samples for volatile organic compound (VOC) analysis are usually shipped to the laboratory in coolers with ice packs at refrigerator temperatures of approximately 4 ºC. Once they arrive at the laboratory, the samples are either kept in a refrigerator prior to analysis, or they are placed in a freezer for longer storage. Both EPA and ASTM recognize the benefit of freezing samples as a preservation technique, and many discussions have taken place with these entities about the feasibility of freezing samples in the field and shipping them at freezer temperatures, down to approximately -20 ºC, to the laboratory for analysis. Two possible ways of shipping frozen samples are to use, a small, power-operated freezer compartment or dry ice storage. However, in most cases, use of a power-operated freezer in the field is not feasible. Dry ice storage is also not a viable option since air shipment of packages containing dry ice is regulated, because dry ice sublimes to gaseous carbon dioxide, which can displace air in sealed aircraft. Even more important relative to sample storage is that dry ice has a temperature of -78 ºC, which is so cold that it will cause the seals of sample containers to be compromised and VOCs will be lost from samples when they thaw.

Work is being performed to develop a new shipping system for frozen samples (or other materials) that uses an optimal phase change liquid (PCL) formulation and an insulated shipping container with an on-board digital temperature data logger to provide a history of the temperature profile within the container during shipment. In previous work, several PCL formulations with temperatures of fusion ranging from approximately -14 to -20 ºC were prepared and evaluated. Both temperature of fusion and heat of fusion of the formulations were measured by differential scanning calorimetry (DSC), and an optimal PCL formulation was selected. The PCL was frozen in plastic bags and tested for its temperature profile in a cooler using a digital temperature data logger. This testing showed that the PCL formulation can maintain freezer temperatures (<-7 to -20 ºC) for an extended period, such as the time for shipping samples by overnight courier.

The results of the experiments described in this report provide significant information for use in developing an integrated freezer system. A new optimal PCL formulation for use in the system has been determined. The original and new PCL formulations were used in experiments involving storage of refrigerated and frozen soil samples to provide information that can be used in developing a procedure for using the PCL formulation in the field for environmental sample storage. In addition, the performances of the original and new PCL formulations were compared to that of a commercially available material. Experiments were performed using various cooler types packed with soil samples as would be done in the field for sample shipment to a laboratory. Experimental results show the importance of the type of cooler used in the system and that use of an insulating material within the cooler improves the performance

of the freezer system. Using an optimal cooler configuration, the new PCL formulation was shown to maintain temperatures at <-7 to -20 ºC for 47 hours in an insulated cooler system containing soil samples. These results are very promising for developing the new technology.

INTRODUCTION

Soil samples for volatile organic compound (VOC) analysis are usually shipped to the laboratory in coolers with ice packs at refrigerator temperatures near 4 ºC. Once they arrive at the laboratory, the samples are either kept in a refrigerator prior to analysis, or they are placed in a freezer for longer-term storage. Both The U.S. Environmental Protection Agency (EPA) and ASTM International (ASTM) recognize the benefit of freezing samples, and many discussions have taken place with these entities concerning the feasibility of freezing samples in the field and shipping them at freezer temperatures (<-7 to -20 ºC) to the laboratory for analysis.

Two possible ways of shipping frozen samples are to use a small, power-operated freezer compartment or dry ice storage. However, use of a power-operated freezer in the field is not feasible in most cases. Dry ice storage is also not a viable option since air shipment of packages containing dry ice is regulated, because dry ice sublimes to gaseous carbon dioxide, which can displace air in sealed aircraft. Even more important relative to sample storage is that dry ice has a temperature of -78 ºC, which is so cold that it will cause the seals of sample containers to be compromised, and VOCs will be lost from samples when they thaw.

ASTM and EPA are prescribing freezing temperatures of approximately -12 ± 5 ºC for storage of soil samples containing VOCs during shipment to the laboratory for analysis (ASTM 2005a, 2005b, U.S. EPA 2002). Phase change liquids (PCLs) have the property of storing or releasing heat energy at a specific temperature, which is the temperature of fusion. For example, water acts as a phase change liquid (PCL) at 0 ºC. Salt-water solutions melt at lower temperatures than water depending on the salt type and concentration. Sodium chloride (NaCl) solutions are used to achieve temperatures of fusion below 0º C. A water/urea phase change formulation that has a melting range of -11 to -15 ºC has previously been described (Salyer 1997).

In addition to the temperature of fusion, the heat of fusion is an important parameter. The higher the heat of fusion, the greater the capacity for the material to store or release energy at the temperature of fusion. The heat of fusion of water is near 80 cal/g (Bolz and Tuve 1980). A PCL formulation to be used in a cooler for shipping frozen samples should have as high a heat of fusion as possible. Addition of chemicals to water can lower the freezing point, but can also decrease the heat of fusion. Therefore, an optimal PCL formulation has a temperature of fusion in the desired temperature range while having a heat of fusion as close to that of water as possible.

In previous work, a PCL formulation was developed (Schabron and Sorini 2004). This formulation is 17 wt.% NaCl, 3 wt.% KCl, 3 wt.% 7HF, and 77 wt.% deionized, distilled water. This report describes cooler experiments that have been conducted to evaluate the performance of the original PCL formulation and a variation of this

formulation to provide information for use in designing an integrated freezer system for shipping frozen samples.

OBJECTIVES

The overall objective of the work is to develop an integrated freezer system that uses a PCL formulation to maintain freezer temperatures in coolers for shipping environmental samples to the laboratory. A successful outcome will result in a new and improved capability for preserving environmental samples during shipment to the laboratory. The activities that were performed over the last 12 months to help achieve this objective include the following.

- The original PCL formulation was modified and the performance of the new formulation was evaluated.
- The performance of the PCL formulations was evaluated during storage with refrigerated and frozen samples to provide information that can be used in developing a procedure for using a formulation in the field for environmental sample storage.
- The performance of the PCL formulations was compared with the performance of a commercially available material that is advertised as having a temperature of fusion of approximately -23 ºC.
- The performance of the PCL formulations was evaluated in various cooler types.

TECHNICAL APPROACH

Cooler Experiment to Evaluate the Performance of the Original PCL Formulation during Storage with Refrigerated and Frozen Samples

Six plastic bags, each containing 1,160 grams of PCL formulation (17 wt.% NaCl, 3 wt.% KCl, 3 wt.% 7HF, and 77 wt.% deionized, distilled water), were prepared. The bags were placed in a digitally controlled freezer and were kept in the freezer at a mean temperature of -28 ºC for approximately four days to make sure they were completely frozen at the time the cooler experiment was started.

The coolers used in the experiment were 8-quart polyethylene Coleman insulated coolers designed for food and beverage storage. These coolers are insulated; however, their lids are not insulated. These types of coolers are commonly used for sample shipment with ice packs.

The temperature data loggers that were used in the coolers to record the temperature profiles are Testo 174 mini portable battery-powered data loggers from Testo, Inc., Lander, NJ. These have a measuring range of -30 to +70 ºC.

The soil used in this experiment is 75% sand, 13% silt, 12% clay, 4.3% organic material, and ~12% moisture. Ten samples of the soil were collected in ten five-gram En Core® samplers. Five of the samples were placed in a refrigerator for storage at 4 ± 2 ºC for approximately four days to make sure they were at a temperature of 4 ± 2 ºC when the cooler experiment was started. The remaining five samples were placed in a freezer for storage at -17 ± 2 ºC for approximately four days to make sure they were at a temperature of -17 ± 2 ºC when the cooler experiment was started.

Three temperature data loggers were programmed to record temperatures on an hourly basis. A cooler was packed with three frozen bags of PCL containing a total mass of 3,480 grams of PCL, five soil samples in En Core samplers in En Core protective bags at a temperature of 4 ± 2 ºC, and a temperature data logger in an En Core protective bag. Approximately five minutes later, a cooler was packed with three frozen bags of PCL containing a total mass of 3,480 grams of PCL, five soil samples in En Core samplers in En Core protective bags at a temperature of -17 ± 2 ºC, and a temperature data logger in an En Core protective bag. In both coolers, the soil samples and temperature data logger were packed between the frozen bags of PCL. The coolers were at room temperature when they were packed. Both coolers were packed by volume and could hold no additional bags. The two coolers were placed side-by-side for storage for 48 hours. A temperature data logger in a protective bag was placed in the room near the coolers. The three temperature data loggers were programmed for the first temperature recordings to begin one half hour after the coolers were packed. The coolers were opened approximately 48 hours later after the 49th temperature readings were recorded.

The 4 ± 2 ºC samples were used in this experiment to simulate field sampling activities in which samples would be collected in the field, placed in a cooler with ice packs during sample collection, and transferred to a cooler containing the frozen PCL bags for freezing during shipment to the laboratory. The frozen samples were used in this experiment to simulate field sampling activities in which samples would be collected in the field, placed in a cooler with frozen PCL bags for freezing during sample collection, and transferred to a second cooler containing frozen PCL bags for shipment to the laboratory.

DSC Characterization of the Original PCL Formulation and Commercial Freezer Bag Material

The original PCL formulation, which as previously mentioned is 17 wt.% NaCl, 3 wt.% KCl, 3 wt.% 7HF, and 77 wt.% deionized, distilled water, and the material from a commercial freezer bag, which is advertised as having a temperature of fusion of approximately -23 ºC and contains approximately 13 wt.% NaCl in 83 wt.% water, were analyzed by differential scanning calorimetry (DSC). The DSC analysis was performed using a TA Instruments 2920 DSC having a refrigerated cooling system (RCS). Samples

(7-12 mg) were sealed in aluminum-hermetic pans for the tests. Samples were allowed to equilibrate in the DSC at 20 ºC for five minutes and were cooled to -70 ºC at 5 ºC per minute. After a five-minute hold at -70 ºC, the samples were heated at 5 ºC per minute to 20 ºC. Data were stored only during the temperature ramps.

Cooler Experiments to Compare Performance of the Original PCL Formulation and Commercial Freezer Bag Material

Comparison of Performance of the Original PCL Formulation and Commercial Freezer Bag Material Using Coolers Packed by Cooler Volume

Five plastic bags, each containing 1,160 grams of PCL were prepared. The bags were placed in the digitally controlled freezer, along with five of the commercially available freezer bags. The bags were kept in the freezer at a mean temperature of -28 ºC for approximately five days to make sure they were completely frozen at the time the cooler experiment was started.

The previously described 8-quart polyethylene Coleman coolers and Testo temperature data loggers were used in this experiment. Three temperature data loggers were programmed to record temperatures on an hourly basis. A cooler was packed with five of the frozen commercial freezer bags containing a total mass of 3,172 grams of commercial freezer bag material. A temperature data logger in a protective bag was packed between the frozen bags in the cooler. Approximately five minutes later, a cooler was packed with four frozen bags of PCL containing a total mass of 4,640 grams of PCL. A temperature data logger in a protective bag was packed between the frozen bags of PCL in the cooler. The coolers were at room temperature when they were packed. Both coolers were packed by volume and could hold no additional bags. The two coolers were placed side-by-side for storage for 48 hours. A temperature data logger in a protective bag was placed in the room near the coolers. The three temperature data loggers were programmed for the first temperature recordings to begin one half hour after the coolers were packed. The coolers were opened approximately 48 hours later after the 49th temperature readings were recorded.

Listed below is additional information on the PCL bags and the commercial freezer bags used in the cooler experiment.

- The dimensions of the PCL bags prior to freezing were 8½" x 6" x 1½". The dimensions of the commercial freezer bags prior to freezing were 8" x 6" x 1".
- The mass of PCL in each bag was 1,160 grams, giving a total mass of PCL used in the experiment of 4,640 grams. The volume of the PCL in each bag was about 1,050 mL, based on a PCL density of approximately 1.1 g/mL. The total volume of PCL in the cooler was approximately 4,200 mL.

- The mass of the commercial freezer bag material in each bag varies. The five bags used in the experiment contained 635 grams, 629 grams, 633 grams, 636 grams, and 639 grams of material. This gives a total mass of the commercial freezer bag material used in the experiment of 3,172 grams. The density of the commercial freezer bag material is approximately 1.1 g/mL, giving a total volume of the commercial freezer bag material used in the experiment of about 2,880 mL.

Comparison of Performance of the Original PCL Formulation and Commercial Freezer Bag Material Using Coolers Packed by Mass

Three plastic bags, each containing 1,160 grams of PCL were prepared. The bags were placed in the digitally controlled freezer, along with five of the commercially available freezer bags. The bags were kept in the freezer at a mean temperature of -28 ºC for approximately five days to make sure they were completely frozen at the time the cooler experiment was started.

The previously described 8-quart polyethylene Coleman coolers and Testo temperature data loggers were used in the experiment. Once again, three temperature data loggers were programmed to record temperatures on an hourly basis. A cooler was packed with five of the frozen commercial freezer bags containing a total mass of 3,147 grams of commercial freezer bag material. No additional bags could be packed in the cooler. A temperature data logger in a protective bag was packed between the frozen bags in the cooler. Approximately five minutes later, a cooler was packed with three frozen bags of PCL containing a total mass of 3,480 grams of PCL. A temperature data logger in a protective bag was packed between the frozen bags of PCL in the cooler. There was room in the PCL cooler for one more bag; however, since this experiment was based on having approximately equal masses of the materials in the coolers, three frozen PCL bags were used. The coolers were at room temperature when they were packed. The two coolers were placed side-by-side for storage for 48 hours. A temperature data logger in a protective bag was placed in the room near the coolers. The three temperature data loggers were programmed for the first temperature recordings to begin one half hour after the coolers were packed. The coolers were opened approximately 48 hours later after the 49th temperature readings were recorded.

Listed below is additional information on the PCL bags and the commercial freezer bags used in the cooler experiment.

- The dimensions of the PCL bags prior to freezing were 8½" x 6" x 1½". The dimensions of the commercial freezer bags prior to freezing were 8" x 6" x 1".

- The mass of PCL in each bag was 1,160 grams, giving a total mass of PCL used in the experiment of 3,480 grams. The volume of the PCL in each bag was about

1,050 mL, based on a PCL density of approximately 1.1 g/mL. The total volume of PCL in the cooler was approximately 3,150 mL.

- The mass of the commercial freezer bag material in each bag varies. The five bags used in the experiment contained 629 grams, 623 grams, 632 grams, 631 grams, and 632 grams of material. This gives a total mass of the commercial freezer bag material used in the experiment of 3,147 grams. The density of the commercial freezer bag material is approximately 1.1 g/mL, giving a total volume of the commercial freezer bag material used in the experiment of about 2,860 mL.

Cooler Experiment to Compare Performance of the Original PCL Formulation and Two Variations of the PCL Formulation

The results from the cooler experiment conducted to evaluate the performance of the PCL formulation and commercial freezer bag material using coolers packed by mass show that approximately equal masses of the PCL formulation and the commercial freezer bag material performed very similarly in the coolers over the 48-hour period. Based on these results, variations of the original PCL formulation were tested to determine if a formulation showing better performance than the original one could be determined.

The 7HF component of the original PCL formulation is added to the formulation to serve as a thickening agent to make the PCL bags easy to handle. The addition of 7HF lowers the heat of melting of the formulation by seven cal/g (Schabron and Sorini 2004). As shown in Table 1, the heat of melting of the commercial freezer bag material is 6 cal/g higher than that of the PCL material. As a result, a decision was made to prepare a new PCL formulation that does not contain 7HF. This formulation is 17 wt.% NaCl, 3 wt.% KCl, and 80 wt.% deionized, distilled water and is designated as PCL-1.

The KCl component of the original PCL formulation is added to the formulation because it was shown to lower the melting point maxima by 2 to 3 ºC (Schabron and Sorini 2004). Originally, this contribution to the performance of the PCL formulation was believed to be of a greater significance than the fact that the addition of the 3 wt.% KCl to the formulation lowers its heat of melting by three cal/g as shown in Table 1 (Schabron and Sorini 2004). However, this may not be the case, and it was decided to prepare a second new PCL formulation that does not contain 7HF or KCl. This formulation is 17 wt.% NaCl and 83 wt.% deionized, distilled water and is designated as PCL-2. The temperatures and heats of fusion of this formulation are given in Table 1. A cooler experiment to compare performances of the original PCL formulation, the PCL-1 formulation, and the PCL-2 formulation was performed.

Four bags of each of the new formulations, PCL-1 and PCL-2, were prepared. Each bag contained 1,160 grams of formulation. After the bags were prepared, they were

placed in the digitally controlled freezer, along with four bags of the original PCL formulation, each also containing 1,160 grams of the PCL formulation. The bags were kept in the freezer at a mean temperature of -28 ºC for approximately six days to make sure they were completely frozen at the time the cooler experiment was started. The bags containing the PCL, PCL-1, and PCL-2 formulations had dimensions of 8½" x 6" x 1½" prior to being frozen.

The 8-quart polyethylene Coleman coolers and Testo temperature data loggers were used in the experiment. Four temperature data loggers were programmed to record temperatures on an hourly basis. A cooler was packed with three bags of the frozen PCL formulation containing a total mass of 3,480 grams of PCL. When the bags were frozen, they expanded in such a way that only three bags could be packed in the cooler. A temperature data logger in a protective bag was packed between the frozen bags in the cooler. Approximately five minutes later, a cooler was packed with three bags of the frozen PCL-1 formulation containing a total mass of 3,480 grams of the PCL-1. As with the cooler containing the PCL material, no additional bags could be packed in the cooler. A temperature data logger in a protective bag was packed between the frozen bags in the cooler. Approximately five minutes later, a cooler was packed with three bags of the frozen PCL-2 formulation containing a total mass of 3,480 grams of PCL-2. When these bags froze, they also expanded in such a way that only three bags could be packed in the cooler. A temperature data logger in a protective bag was packed between the frozen bags in the cooler. The coolers were at room temperature when they were packed. The three coolers were placed side-by-side for storage for 48 hours. A temperature data logger in a protective bag was placed in the room near the coolers. The four temperature data loggers were programmed for the first temperature recordings to begin one half hour after the coolers were packed. The coolers were opened approximately 48 hours later after the 49th temperature readings were recorded.

Cooler Experiment to Compare Performance of a New PCL Formulation and Commercial Freezer Bag Material

Three plastic bags, each containing 1,160 grams of 17 wt.% NaCl and 83 wt.% deionized, distilled water (the new PCL formulation selected for evaluation) were prepared. The bags were placed in the digitally controlled freezer, along with five of the commercially available freezer bags. The bags were kept in the freezer at a mean temperature of -28 ºC for approximately four days to make sure they were completely frozen at the time the cooler experiment was started.

The 8-quart polyethylene Coleman coolers and Testo temperature data loggers were used in the experiment. Three temperature data loggers were programmed to record temperatures on an hourly basis. A cooler was packed with five of the frozen commercial freezer bags containing a total mass of 3,139 grams of commercial freezer bag material.

No additional bags could be packed in the cooler. A temperature data logger in a protective bag was packed between the frozen bags in the cooler. Approximately five minutes later, a cooler was packed with three frozen bags of 17 wt.% NaCl and 83 wt.% deionized, distilled water containing a total mass of 3,480 grams of the formulation. A temperature data logger in a protective bag was packed between the frozen bags of PCL in the cooler. Because of the way the solution in the bags expanded when the bags were frozen, three bags filled most of the space in the cooler. The coolers were at room temperature when they were packed. The two coolers were placed side-by-side for storage for 48 hours. A temperature data logger in a protective bag was placed in the room near the coolers. The three temperature data loggers were programmed for the first temperature recordings to begin one half hour after the coolers were packed. The coolers were opened approximately 48 hours later after the 49th temperature readings were recorded.

Listed below is additional information on the PCL bags and the commercial freezer bags used in the cooler experiment.

- The dimensions of the 17 wt.% NaCl and 83 wt.% deionized, distilled water bags prior to freezing were 8½" x 6" x 1½". The dimensions of the commercial freezer bags prior to freezing were 8" x 6" x 1".

- The mass of the 17 wt.% NaCl and 83 wt.% deionized, distilled water in each bag was 1,160 grams, giving a total mass of PCL used in the experiment of 3,480 grams.

- The mass of the commercial freezer bag material in each bag varies. The five bags used in the experiment contained 622 grams, 630 grams, 628 grams, 628 grams, and 631 grams of material. This gives a total mass of the commercial freezer bag material used in the experiment of 3,139 grams.

Cooler Experiment to Compare Performance of the New PCL Formulation and Commercial Freezer Bag Material Using Igloo MaxCold™ Coolers

Three plastic bags, each containing 1,160 grams of the new PCL formulation were prepared. The bags were placed in a digitally controlled freezer, along with six of the commercially available freezer bags. The bags were kept in the freezer at a mean temperature of -28 ºC for approximately six days to make sure they were completely frozen at the time the cooler experiment was started.

The coolers used in the experiment were 18-quart polyethylene Igloo MaxCold™ insulated coolers designed for food and beverage storage. These coolers are insulated and have insulated lids. The Testo temperature data loggers were also used. Each cooler was packed to simulate a cooler prepared for actual sample shipment from the field to the

laboratory. The cooler containing the PCL bags was packed with 12 five-gram soil samples in volatile organic analysis (VOA) vials at a temperature of 4 ± 2 ºC that were packed in polyethylene bubble wrap. Additional bubble wrap was used to fill space in the cooler. Bubble wrap is used in packing coolers for sample shipment to prevent breakage during shipment. The mass of bubble wrap packed in the cooler was 202.5 grams. The bubble wrap used in the experiment was at room temperature. Three bags of the PCL formulation containing a total mass of 3,480 grams of the formulation were packed in the cooler. The cooler was packed so there were alternating layers of PCL bags and rows of samples. A temperature data logger was placed along the inside wall of the cooler.

The cooler containing the commercial freezer bags was packed with 12 five-gram soil samples in VOA vials at a temperature of 4 ± 2 ºC that were packed in bubble wrap. Additional bubble wrap was used to fill space in the cooler. The mass of bubble wrap packed in the cooler was 202.5 grams. Six commercial freezer bags containing a total mass of 3,722 grams of the commercial material were packed in the cooler. The cooler was packed so there were alternating layers of commercial freezer bags and rows of samples. A temperature data logger was placed along the inside wall of the cooler.

The coolers were at room temperature when they were packed. After being packed, the two coolers were placed side-by-side for storage for 48 hours. A temperature data logger in a protective bag was placed in the room near the coolers. The three temperature data loggers were programmed for the first temperature recordings to begin one hour after the coolers were packed. The coolers were opened after 48 hours of storage.

Listed below is additional information on the PCL bags, the commercial freezer bags, and bubble wrap used in the cooler experiment.

- The dimensions of the PCL bags prior to freezing were 8½" x 6" x 1½". The dimensions of the commercial freezer bags prior to freezing were 8" x 6" x 1".
- The mass of the PCL in each bag was 1,160 grams, giving a total mass of PCL of 3,480 grams.
- The mass of the commercial freezer bag material in each bag varies. The six bags used in the experiment contained 621 grams, 619 grams, 619 grams, 617 grams, 623 grams, and 623 grams of material. This gives a total mass of the commercial freezer bag material of 3,722 grams.
- Each strip of bubble wrap had a mass of 13.5 grams. Each cooler contained 15 strips of bubble wrap, giving a total mass of 202.5 grams.

Cooler Experiment to Evaluate the Performance of the New PCL Formulation Using an Igloo MaxCold™ Cooler Packed with Refrigerated Soil Samples

The next experiment involved storage of four of the PCL freezer bags with 12 five-gram soil samples that were at 4 ± 2 ºC and packed in bubble wrap to simulate an actual cooler packed with samples for shipment to a laboratory. The Igloo 18-quart MaxCold cooler used in the previous experiment to store the commercial freezer bags was used in this experiment.

Four plastic bags, each containing 1,160 grams of the new PCL formulation were prepared. The bags were placed in the digitally controlled freezer at a mean temperature of -28 ºC for approximately five days to make sure they were completely frozen at the time the cooler experiment was started.

The cooler was packed with 12 five-gram soil samples in VOA vials at a temperature of 4 ± 2 ºC that were packed in bubble wrap. Additional bubble wrap was used to fill space in the cooler. The mass of bubble wrap packed in the cooler was 216.0 grams. The bubble wrap used in the experiment was at room temperature. Four bags of the PCL formulation containing a total mass of 4,640 grams of the formulation were packed in the cooler. The cooler was packed so there were alternating layers of PCL bags and rows of samples. A Testo temperature data logger in a protective bag was placed along the inside wall of the cooler.

The cooler was at room temperature when it was packed. A temperature data logger in a protective bag was placed in the room near the cooler. The two temperature data loggers were programmed to record temperatures on an hourly basis with the first temperature recordings to begin one hour after the cooler were packed. The cooler was opened after 48 hours of storage.

Listed below is additional information on the PCL bags and bubble wrap used in the cooler experiment.

- The dimensions of the PCL bags prior to freezing were 8½" x 6" x 1½".
- The mass of the PCL in each bag was 1,160 grams, giving a total mass of PCL used in the experiment of 4,640 grams.
- Each strip of bubble wrap had a mass of 13.5 grams. The cooler contained 16 strips of bubble wrap, giving a total mass of 216.0 grams.

Cooler Experiment to Evaluate the Performance of the New PCL Formulation Using an Igloo MaxCold™ Cooler Packed with Frozen Soil Samples

The next experiment involved storage of four of the PCL freezer bags with 12 five-gram soil samples that were frozen at approximately -17 ± 2 ºC and packed in bubble wrap to simulate an actual cooler packed with samples for shipment to a laboratory. The Igloo 18-quart MaxCold cooler used in the previous experiment was used in this experiment.

Four plastic bags, each containing 1,160 grams the new PCL formulations were prepared. The bags were placed in the digitally controlled freezer at a mean temperature of -28 ºC for approximately six days to make sure they were completely frozen at the time the cooler experiment was started.

The cooler was packed with 12 five-gram soil samples in VOA vials at a temperature of -17 ± 2 ºC that were packed in bubble wrap. Additional bubble wrap was used to fill space in the cooler. The mass of bubble wrap packed in the cooler was 216.0 grams. The bubble wrap used in the experiment was at room temperature. Four bags of the PCL formulation containing a total mass of 4,640 grams of the formulation were packed in the cooler. The cooler was packed so there were alternating layers of PCL bags and rows of samples. A temperature data logger in a protective bag was placed along the inside wall of the cooler.

The cooler was at room temperature when it was packed. A temperature data logger in a protective bag was placed in the room near the cooler. The two temperature data loggers were programmed to record temperatures on an hourly basis with the first temperature recordings to begin one hour after the cooler were packed. The cooler was opened after 48 hours of storage.

Listed below is additional information on the PCL bags and bubble wrap used in the cooler experiment.

- The dimensions of the PCL bags prior to freezing were 8½" x 6" x 1½".
- The mass of the PCL in each bag was 1,160 grams, giving a total mass of PCL of 4,640 grams.
- Each strip of bubble wrap had a mass of 13.5 grams. The cooler contained 16 strips of bubble wrap, giving a total mass of 216.0 grams.

Cooler Experiment to Evaluate the Temperature Distribution within an Igloo MaxCold™ Cooler Packed with Refrigerated Soil Samples and Bags of the New PCL Formulation

The next experiment involved storage of four of the PCL freezer bags with 12 five-gram soil samples that were at 4 ± 2 ºC and packed in bubble wrap. The Igloo 18-quart MaxCold cooler was used. Two temperature data loggers were placed in the cooler. One temperature data logger was placed along the inside wall of the cooler as was done previously in the experiments involving the Igloo cooler packed with soil samples, and one temperature data logger was packed in bubble wrap with one of the center-most positioned samples.

Four plastic bags, each containing 1,160 grams of the new PCL were prepared. The bags were placed in a digitally controlled freezer at a mean temperature of -28 ºC for approximately 10 days.

The cooler was packed with 12 five-gram soil samples in VOA vials at a temperature of 4 ± 2 ºC that were packed in bubble wrap. Additional bubble wrap was used to fill space in the cooler. The mass of bubble wrap packed in the cooler was 216.0 grams. The bubble wrap used in the experiment was at room temperature. Four bags of the PCL containing a total mass of 4,640 grams of the formulation were packed in the cooler. The cooler was packed so there were alternating layers of PCL bags and rows of samples. As described above, one temperature data logger was placed along the inside wall of the cooler and the second temperature data logger was packed in bubble wrap with one of the center-most positioned samples.

The cooler was at room temperature when it was packed. A temperature data logger in a protective bag was placed in the room near the cooler. The three temperature data loggers were programmed to record temperatures on an hourly basis with the first temperature recordings to begin one hour after the cooler was packed. The cooler was opened after 48 hours of storage.

Cooler Experiment to Evaluate the Temperature Distribution within a Polystyrene Cooler Packed with Refrigerated Soil Samples and Bags of the New PCL Formulation

The last experiment that was performed involved a design similar to that of the previous experiment. However, to evaluate the performance of a different cooler material, a 20-quart polystyrene cooler was used instead of the 18-quart Igloo MaxCold polyethylene cooler.

In this experiment, the polystyrene cooler was packed with 12 five-gram soil samples in VOA vials at a temperature of 4 ± 2 ºC that were packed in bubble wrap.

Additional bubble wrap was used to fill space in the cooler. The mass of bubble wrap packed in the cooler was 229.5 grams. The bubble wrap used in the experiment was at room temperature. Four bags of the PCL formulation that had been frozen at a mean temperature of -28 ºC for approximately five days were packed in the cooler. Each bag contained 1,160 grams of the new PCL formulation. One temperature data logger was placed along the inside wall of the cooler and the second temperature data logger was packed in bubble wrap with one of the center-most positioned samples. The cooler was packed so there were alternating layers of PCL bags and rows of samples, but because the polystyrene cooler is wider and shorter than the Igloo cooler, the PCL bags and samples were packed using a different configuration than in the Igloo cooler and one additional strip of bubble wrap was packed in the cooler.

The cooler was at room temperature when it was packed. A temperature data logger in a protective bag was placed in the room near the cooler. The three temperature data loggers were programmed to record temperatures on an hourly basis with the first temperature recordings to begin one hour after the cooler was packed. The cooler was opened after 48 hours of storage.

RESULTS AND DISCUSSION

Cooler Experiment to Evaluate the Performance of the Original PCL Formulation during Storage with Refrigerated and Frozen Samples

The temperature in the room where the coolers were stored ranged from 25.4 to 27.1 ºC over the 48-hour period. The temperature profiles for the coolers containing the 4 ± 2 ºC and -17 ± 2 ºC samples with the PCL bags are shown in Figure 1. Figure 1 shows that after initial cooling, the temperature in the cooler containing the refrigerated samples gradually increased by about 0.2 to 0.7 ºC per hour for up to 14 hours, at which time the temperature in the cooler was -7.0 ºC, which is above the EPA recommended temperature for frozen storage (<-7 to -20 ºC).

Figure 1 shows that after initial cooling, the temperature in the cooler containing the frozen samples ranged from approximately -15 ºC to approximately -13 ºC for 18 hours. The temperature in this cooler did not go above the EPA recommended temperature for frozen storage until after 23 hours. After 24 hours of storage, the temperature in the cooler was -6.9 ºC. Results of this experiment are summarized in Table 2 under Experiment 1.

DSC Characterization of the Original PCL Formulation and Commercial Freezer Bag Material

Data from DSC characterization of the original PCL formulation and the commercial freezer bag material are listed in Table 1. These data show that the

commercial freezer bag material, which as mentioned contains approximately 13 wt.% NaCl, has a slightly higher heat of melting than the original PCL, which is one of the reasons the new PCL formulation was determined. The commercial freezer bag material exhibits a bimodal melting point profile, with melting point maxima at -19 and -13 ºC. This is shown by the DSC profile of the commercial freezer bag material in Figure 2. The original PCL, which is a more concentrated NaCl solution (17 wt.% NaCl), has a single melting peak maximum at -21 ºC as shown in Figure 2. This trend has been observed in previous testing. In analyzing various weight percent solutions of NaCl, Western Research Institute (WRI) has found that the two melting point maxima converge to a single peak at higher concentrations, starting at about 17 wt.% NaCl. This is shown in Figure 3. The new PCL formulation, which is also 17 wt.% NaCl, also has a single melting point maximum at -21 ºC. The DSC profiles shown in Figures 2 and 3 are for comparing the shapes of the peaks only.

Cooler Experiments to Compare Performance of the Original PCL Formulation and Commercial Freezer Bag Material

Comparison of Performance of the Original PCL Formulation and Commercial Freezer Bag Material Using Coolers Packed by Cooler Volume

The temperature in the room where the coolers were stored ranged from 23.0 to 25.0 ºC over the 48-hour period. The temperature profiles for the coolers containing the PCL bags and the commercial freezer bags are shown in Figure 4. Figure 4 shows a continuous gradual temperature increase in the cooler containing the commercial freezer bags. The temperature profile shown in Figure 4 for the PCL cooler does not show the same continuous gradual temperature increase. Instead, in the PCL cooler, the temperature remained at approximately -15 ºC for 22 hours before gradually increasing with time.

The range of frozen temperature storage recommended by EPA is <-7 to -20 ºC (US EPA 2002). The range of frozen temperature storage recommended by ASTM is -7 to -17 ºC (ASTM 2005a). After storage for 25 hours, the cooler containing the commercial freezer bags had a temperature of -7.4 ºC. After storage for 26 hours, the temperature in this cooler had increased to -6.8 ºC, which is above the EPA recommended temperature. After storage for 32 hours, the cooler containing the PCL bags had a temperature of -8.0 ºC. After storage for 33 hours, the temperature in this cooler had increased to -6.5 ºC, above the EPA recommended temperature. These data show that the PCL bags maintained the freezer temperatures recommended by EPA in the cooler for seven hours longer than the commercial freezer bags. Results of this experiment are summarized in Table 2 under Experiment 2.

It was suspected that the lower temperatures in the PCL cooler for a longer period of time were due to the greater mass of the PCL that could be packed into the cooler.

However, based on the DSC data for the two materials and the temperature profiles for the two coolers, it appeared that the difference in the composition of the commercial freezer bag material and PCL could also be partly responsible for the difference in the performance of the bags in the coolers. As previously mentioned, the material from the commercial freezer bags has a bimodal melting point profile, with melting point maxima at -19 and -13 ºC. This bimodal melting point could be the reason for the continuous gradual temperature increase shown in Figure 4 for the cooler containing the commercial freezer bags.

Because of the uncertainty concerning what caused the difference in performance of the PCL material and the commercial freezer bag material, a second experiment to compare the materials was conducted. In this experiment, approximately equal masses of the materials were used to eliminate the uncertainty concerning the effect of using different masses of the materials in the cooler experiment.

Comparison of Performance of the Original PCL Formulation and Commercial Freezer Bag Material Using Coolers Packed by Mass

The temperature in the room where the coolers were stored ranged from 23.5 to 26.4 ºC over the 48-hour period. Hourly temperature readings in the two coolers varied by approximately 1 ºC or less during storage for up to 24 hours, at which time the temperature in both coolers was near -7 ºC. After 24 hours of storage, the temperatures in the coolers continued to increase to approximately 20 ºC at the end of the 48-hour storage time. Results of this experiment are summarized in Table 2 under Experiment 3.

The results from this cooler experiment show that approximately equal masses of the PCL formulation and the commercial freezer bag material performed very similarly in the coolers over the 48-hour period, and indicated that the PCL formulation should be modified to give better performance.

The PCL cooler had air space of approximately 1,000 cm^3, which was not present in the cooler containing the commercial freezer bags. This air space may have had an affect on the performance of the PCL formulation during the experiment; however, based on the results of this experiment, variations of the original PCL formulation were tested to determine if a formulation showing better performance than the original one could be determined.

Cooler Experiment to Compare Performance of the Original PCL Formulation and Two Variations of the PCL Formulation

The temperature in the room where the coolers were stored ranged from 18.9 to 23.2 ºC over the 48-hour period. The temperature profiles for the coolers containing the 17 wt.% NaCl and 83 wt.% deionized, distilled water (PCL-2) bags, the 17 wt.% NaCl, 3

wt.% KCl, and 80 wt.% deionized, distilled water (PCL-1) bags, and the original PCL bags are shown in Figure 5. The temperature profiles for the coolers containing the PCL-1 and PCL-2 bags are similar, with the cooler containing the PCL-2 formulation showing slightly lower temperatures than the cooler containing the PCL-1 formulation. Both formulations show better performance than the original PCL formulation to maintain cooler temperatures at <-7 ºC.

After storage for 29 hours, the cooler containing the 17 wt.% NaCl and 83 wt.% deionized, distilled water (PCL-2) bags had a temperature of -7.4 ºC. After 30 hours of storage, the temperature in this cooler increased to -6.5 ºC, which is above the EPA recommended temperature of <-7 ºC. After storage for 28 hours, the cooler containing the 17 wt. % NaCl, 3 wt.% KCL, and 80 wt.% deionized, distilled water (PCL-1) bags had a temperature of -7.5 ºC. After 29 hours of storage, the temperature in this cooler increased to -6.0 ºC. After storage for 22 hours, the cooler containing the PCL bags had a temperature of -7.4 ºC. After 23 hours of storage, the temperature in this cooler increased to -6.4 ºC. Results from this experiment are summarized in Table 2 under Experiment 4.

These results show that the addition of the 7HF to the PCL formulation does affect the performance of the formulation to maintain freezer temperatures in the coolers for a longer period of time. The results also show that the addition of 3 wt.% KCl does not improve the performance of the 17 wt.% NaCl solution to maintain freezer temperatures at <-7 ºC for a longer period of time and may hinder the performance of the formulation to maintain freezer conditions. Based on the results of this experiment, it appears that a more optimal formulation to use in frozen bags for maintaining cooler temperatures at <-7 to -20 ºC is 17 wt.% NaCl and 83 wt.% deionized, distilled water.

Cooler Experiment to Compare Performance of a New PCL Formulation and Commercial Freezer Bag Material

The temperature in the room where the coolers were stored ranged from 22.2 to 23.9 ºC over the 48-hour period. The temperature profiles for the cooler containing the bags of new PCL formulation (17 wt.% NaCl and 83 wt.% deionized, distilled water) and the cooler containing the commercial freezer bags are shown in Figure 6. The temperature profile for the PCL cooler shows slightly lower temperatures than the profile for the commercial freezer bag cooler for most of the 48-hour storage period. After storage for 26 hours, the cooler containing the bags of new PCL formulation had a temperature of -7.7 ºC. After 27 hours of storage, the temperature in this cooler increased to -6.9 ºC, which is above the EPA recommended temperature of <-7 ºC. After storage for 23 hours, the cooler containing the commercial freezer bags had a temperature of -7.4 ºC. After storage for 24 hours, the temperature in this cooler increased to -6.9 ºC. Results from this experiment are summarized in Table 2 under Experiment 5.

The temperature profile for the cooler containing the new PCL formulation determined in this experiment differs from the temperature profile for the cooler containing the same mass of the new PCL formulation in the previous experiment. This is shown in Figures 5 and 6. In the previous cooler experiment, the new PCL formulation held the temperature in the cooler at <-7 ºC for 29 hours, three hours longer than in this experiment. Two different coolers were used to store the bags in the experiments. It is believed that variations in the coolers are responsible for the different temperature profiles determined in the two experiments.

In the previous cooler experiment that involved storing 3,147 grams of the commercial freezer bag material for 48 hours (Experiment 3 in Table 2), the material held the temperature in the cooler at <-7 ºC for 25 hours when stored in a room at 23.5 to 26.4 ºC. Results of the experiment being described in this section show that 3,139 grams of the commercial freezer bag material held the temperature in the cooler at <-7 ºC for 23 hours when stored in a room at 22.2 to 23.9 ºC. Once again, it is believed that variations in the coolers used to store the commercial freezer bags in the two experiments are responsible for the different results.

Despite the effects of cooler variations on the results of the experiments discussed above, it can be concluded that the new PCL formulation of 17 wt.% NaCl in 83 wt.% deionized, distilled water is an optimal formulation for use in an integrated freezer system for shipping environmental samples to the laboratory. These experimental results are promising for use of the new formulation in an integrated freezer system.

Cooler Experiment to Compare Performance of the New PCL Formulation and Commercial Freezer Bag Material Using Igloo MaxCold™ Coolers

The temperature in the room where the coolers were stored ranged from 21.8 to 23.7 ºC over the 48-hour period. This temperature range is very similar to temperatures recorded in the room where previous experiments had been performed. Results from this experiment showed very poor performance by the PCL formulation. The results varied so much from the results of the previous experiments in which performance of the two materials was compared that it was concluded that the cooler used to store the PCL bags was damaged and/or faulty and should not be used in any further experiments. Results of this experiment are summarized in Table 2 under Experiment 6.

Cooler Experiment to Evaluate the Performance of the New PCL Formulation Using an Igloo MaxCold™ Cooler Packed with Refrigerated Soil Samples

The temperature in the room where the cooler was stored ranged from 22.9 to 25.5 ºC over the 48-hour period. After storage for 25 hours, the cooler had a temperature of -7.3 ºC. After 26 hours of storage, the temperature in this cooler increased to -6.9 ºC,

which is above the EPA recommended temperature of <-7 ºC. Results of this experiment are summarized in Table 2 under Experiment 7.

Cooler Experiment to Evaluate the Performance of the New PCL Formulation Using an Igloo MaxCold™ Cooler Packed with Frozen Soil Samples

The temperature in the room where the cooler was stored ranged from 23.4 to 25.5 ºC over the 48-hour period. The data from the temperature data logger stored in the cooler showed that the temperature in the cooler ranged from -9.9 to 7.0 °C and went above -7 °C after 17 hours (Table 2, Experiment 8). These results are not what would be expected for this cooler packed with frozen samples since the cooler containing refrigerated samples had a temperature of <-7 °C for 24 hours (Table 2, Experiment 7). It was noted that one of the PCL bags in the center of the cooler had frozen particles when the cooler was opened after 48 hours. This indicates that there was not a uniform temperature distribution within the cooler and that the side of the cooler where the temperature data logger was located was at a higher temperature. As a result, a cooler experiment to evaluate the temperature distribution within the Igloo MaxCold cooler packed with refrigerated soil samples and new PCL formulation was performed.

Cooler Experiment to Evaluate the Temperature Distribution within an Igloo MaxCold™ Cooler Packed with Refrigerated Soil Samples and Bags of the New PCL Formulation

The temperature in the room where the cooler was stored ranged from 23.2 to 26.5 ºC over the 48-hour period. The temperature profile from the data logger that was packed in bubble wrap with one of the center-most positioned samples in the cooler, Figure 7, shows that the temperature at that location ranged from -21.7 ºC one hour after the cooler was packed to -6.5 ºC after 48 hours of storage. The temperature at that location in the cooler was -7.4 ºC at 47 hours. This temperature profile is very promising for developing the new freezer technology.

The temperature data recorded by the data logger that was placed along the inside wall of the cooler varies significantly from the temperature data recorded in the center of the cooler. The temperature along the inside wall of the cooler ranged from -2.0 ºC one hour after the cooler was packed to 6.2 ºC at 48 hours of storage. The temperature at that location dropped to -8.2 ºC after 3 hours and was <-7 ºC for 10 hours. The results from this experiment show that there was not a uniform temperature distribution within the cooler during the 48 hours of sample storage and that the temperature in the center of the cooler where the samples were stored within the bubble wrap was much colder than along the side of the cooler. Results of this experiment are summarized in Table 2 under Experiment 9.

Cooler Experiment to Evaluate the Temperature Distribution within a Polystyrene Cooler Packed with Refrigerated Soil Samples and Bags of the New PCL Formulation

The temperature in the room where the cooler was stored ranged from 22.7 to 24.9 ºC over the 48-hour period. Results of this experiment, show a significant difference in the temperature in the location where the samples were stored versus the temperature along the side of the cooler. The data from the temperature data logger that was wrapped with one of the center-most samples show that the temperature in that location ranged from -18.7 to 2.2 °C and went above -7 °C after 37 hours. The data from the temperature data logger, that was placed along the inside wall of the cooler show that the temperature in that location ranged from -5.9 to 9.7 °C. The temperature along the inside wall of the cooler never went below -5.9°C. The results of this experiment show that there was not a uniform temperature distribution within the cooler and that the temperature was significantly colder in the center of the cooler than along the side of the cooler. Based on this experiment, the performance of the polystyrene cooler does not seem to be as good as the performance of the Igloo MaxCold cooler for use in the integrated freezer system. The results of this experiment are summarized in Table 2 under Experiment 10.

CONCLUSIONS

The results of the experiments described in this report provide significant information for use in developing an integrated freezer system. A new optimal PCL formulation for use in the system has been determined. The new formulation has been shown to maintain temperatures at <-7 to -20 ºC for 47 hours in an insulated cooler system containing soil samples. These results are very promising for developing the new technology. Information for use in designing an integrated freezer system determined by the work described in this report is summarized below.

- The heat of melting of the PCL formulation is critical to the performance of the formulation in maintaining cooler temperatures at <-7 ºC for an extended period of time.
- Addition of a polymer to the PCL limits the performance of the formulation to maintain freezer temperatures in coolers for a longer period of time.
- The cooler used in the integrated freezer system can be at room temperature. Pre-cooling the cooler for use in the system is not required.
- Samples can be placed in a cooler with ice packs to bring their temperature to 4 ± 2 ºC during sample collection in the field. When sample collection is completed, the samples can be transferred to the integrated freezer system for freezing during shipment to the laboratory.

- The cooler used in the system must be fully insulated (body and lid). A fully insulated polyethylene cooler has been shown to perform well in the system.
- There should be minimum air space within the cooler system.
- An insulating material, such as bubble wrap, should be used to maintain freezing temperatures within the system.
- The temperature data logger should be located with the samples in the center of the cooler system. Temperature distribution within the cooler is not uniform. The temperature outside of the insulating material, along the sides, bottom, and top of the cooler, will be higher than the temperature inside the insulating material where the samples are located with the PCL bags.

Future work for developing the integrated freezer system for shipping environmental samples to the laboratory should include determining an optimum bag size for use in the system and an optimum bag/sample arrangement for the system. By optimizing these variables, storage time at <-7 to -20 ºC within the system may be increased to 48 hours. This is the EPA's specified holding time for soil samples collected and stored in airtight coring devices that are preserved by freezing. Evaluation of use of the system in the field will also be required, with samples collected in the field, transferred to the integrated freezer system, and shipped to a location, so temperature profiles within the system can be evaluated

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Table 1. Temperatures and Heats of Fusion for the Original PCL, PCL-1, and PCL-2 Formulations and the Commercial Freezer Bag Material (7-12 mg Samples)

Substance	Melting Onset ºC	Melting Peak Maxima ºC	Heat of Melting cal/g
Distilled de-ionized water	0.4	2	80 (reference)
Original PCL	-25	-21	52
PCL-1	-23	-21	59
PCL-2	-21	-18	62
Commercial Freezer Bag Material	-22	-19 and -13	58

Table 2. Summary of Cooler Experiments

Formulation	Cooler Type	Samples	Bubble Wrap	Cooler Temperature <-7 to -20°C
Experiment 1 (3,480 g PCL in each cooler)				
Original PCL	Coleman	Refrigerated	No	13 hours
Original PCL	Coleman	Frozen	No	23 hours
Experiment 2 (Coolers packed by cooler volume: 4,640 g PCL; 3,172 g Commercial)				
Original PCL	Coleman	No	No	32 hours
Commercial	Coleman	No	No	25 hours
Experiment 3 (Coolers packed by mass: 3,480 g PCL; 3,147 g Commercial)				
Original PCL	Coleman	No	No	23 hours
Commercial	Coleman	No	No	25 hours
Experiment 4 (3,480 g PCL in each cooler)				
Original PCL	Coleman	No	No	22 hours
PCL-1	Coleman	No	No	28 hours
PCL-2	Coleman	No	No	29 hours
Experiment 5 (Coolers packed by mass; 3,480 g PCL; 3,139 g Commercial)				
New PCL (PCL-2)	Coleman	No	No	26 hours
Commercial	Coleman	No	No	23 hours
Experiment 6 (Coolers packed by mass: 3,480 g PCL; 3,722 g Commercial)				
New PCL	Igloo MaxCold™	Refrigerated	Yes	No Results Faulty Cooler
Commercial	Igloo MaxCold™	Refrigerated	Yes	20 hours Data logger at side of cooler
Experiment 7 (4,640 g PCL in cooler)				
New PCL	Igloo MaxCold™	Refrigerated	Yes	25 hours Data logger at side of cooler
Experiment 8 (4,640 g PCL in cooler)				
New PCL	Igloo MaxCold™	Frozen	Yes	17 hours Data logger at side of cooler
Experiment 9 (4,640 g PCL in cooler)				
New PCL	Igloo MaxCold™	Refrigerated	Yes	47 hours Data logger in center of cooler 10 hours Data logger at side of cooler
Experiment 10 (4,640 g PCL in cooler)				
New PCL	Polystyrene	Refrigerated	Yes	37 hours Data logger in center of cooler 0 hours Data logger at side of cooler

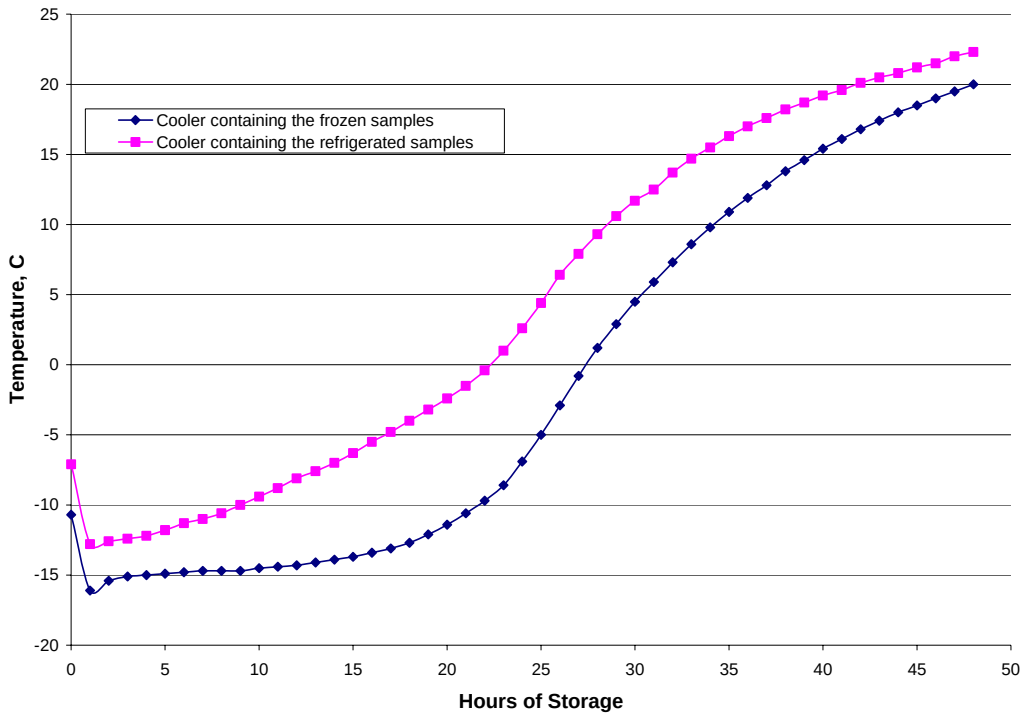

Figure 1. Temperature Profiles for the Coolers Containing the Original PCL Bags with Frozen Samples and with Refrigerated Samples (Cooler Storage at 25-27 ºC)

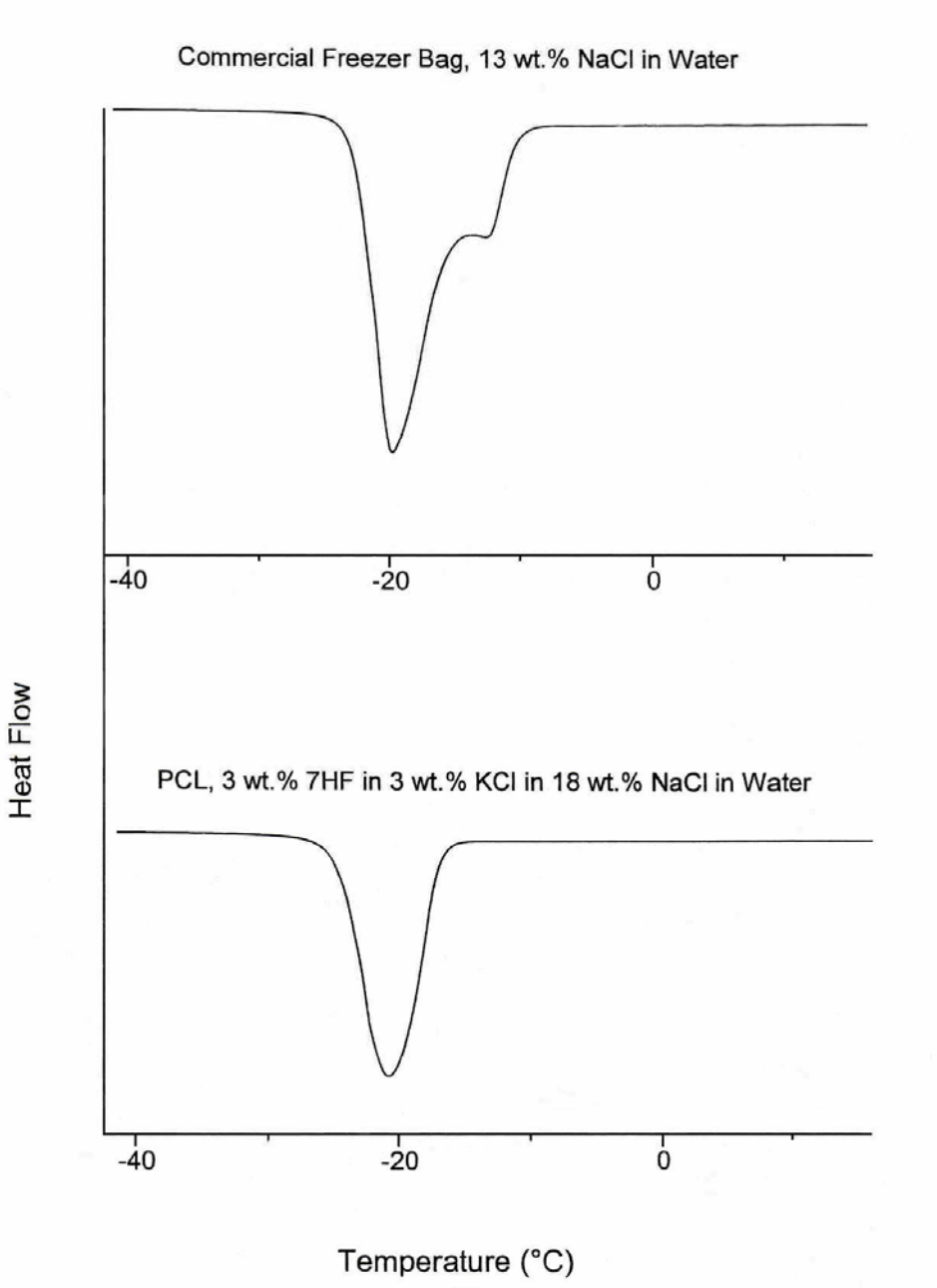

Figure 2. DSC Profiles of the Commercial Freezer Bag Material and the Original PCL Formulation

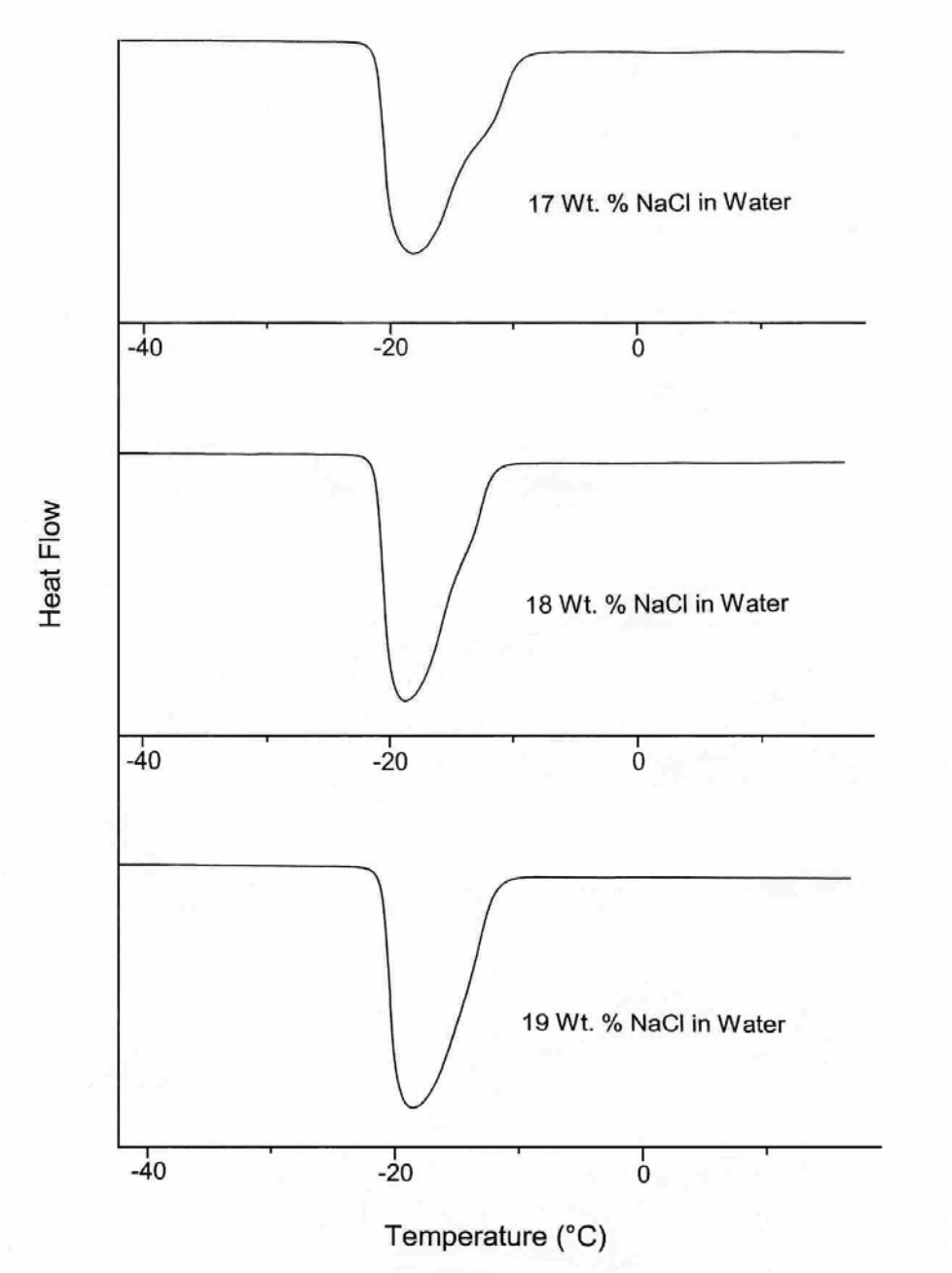

Figure 3. DSC Profiles of 17, 18, and 19 Wt.% NaCl in Water

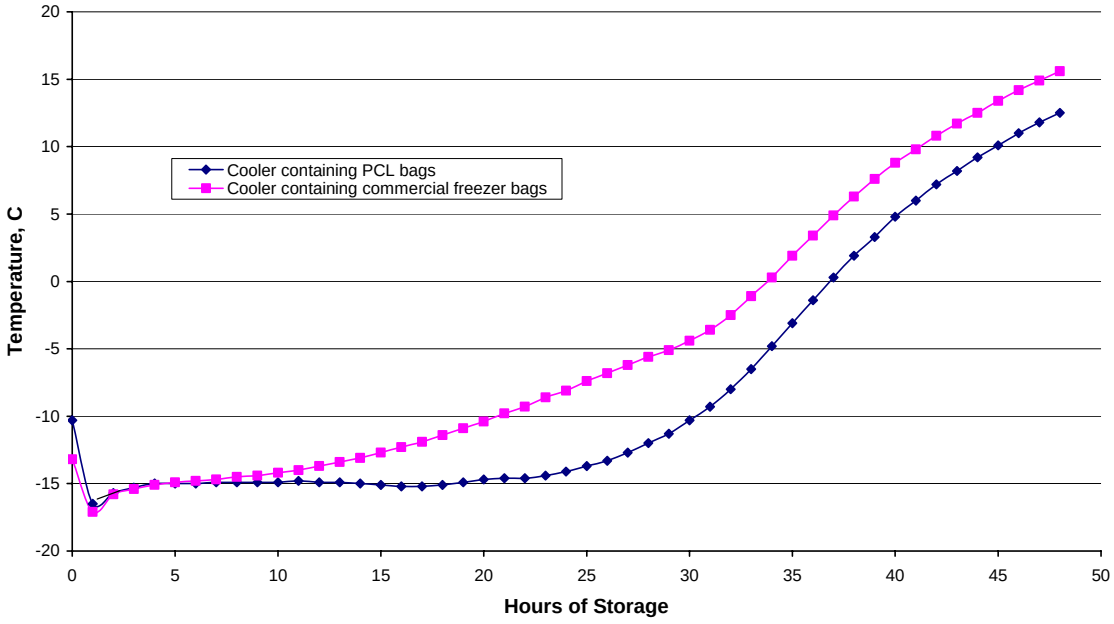

Figure 4. Temperature Profiles for the Cooler Containing the Original PCL Bags and the Cooler Containing the Commercial Freezer Bags (Cooler Storage at 23-25 ºC, Coolers packed by Cooler Volume)

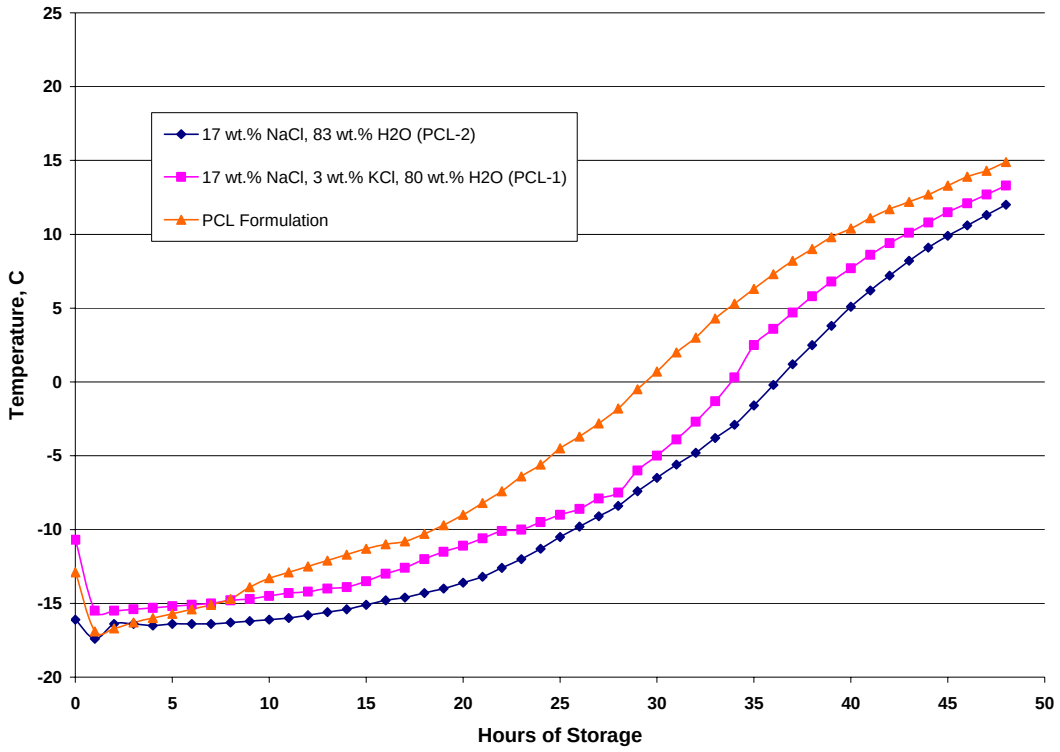

Figure 5. Temperature Profiles for the Cooler Containing the 17 wt.% NaCl and 83 wt.% H_2O (PCL-2) Bags, the Cooler Containing 17 wt.% NaCl, 3 wt.% KCl, 80 wt.% H_2O (PCL-1) Bags, and the Cooler Containing the original PCL Bags (Cooler Storage at 19-23 ºC, Coolers packed by Mass)

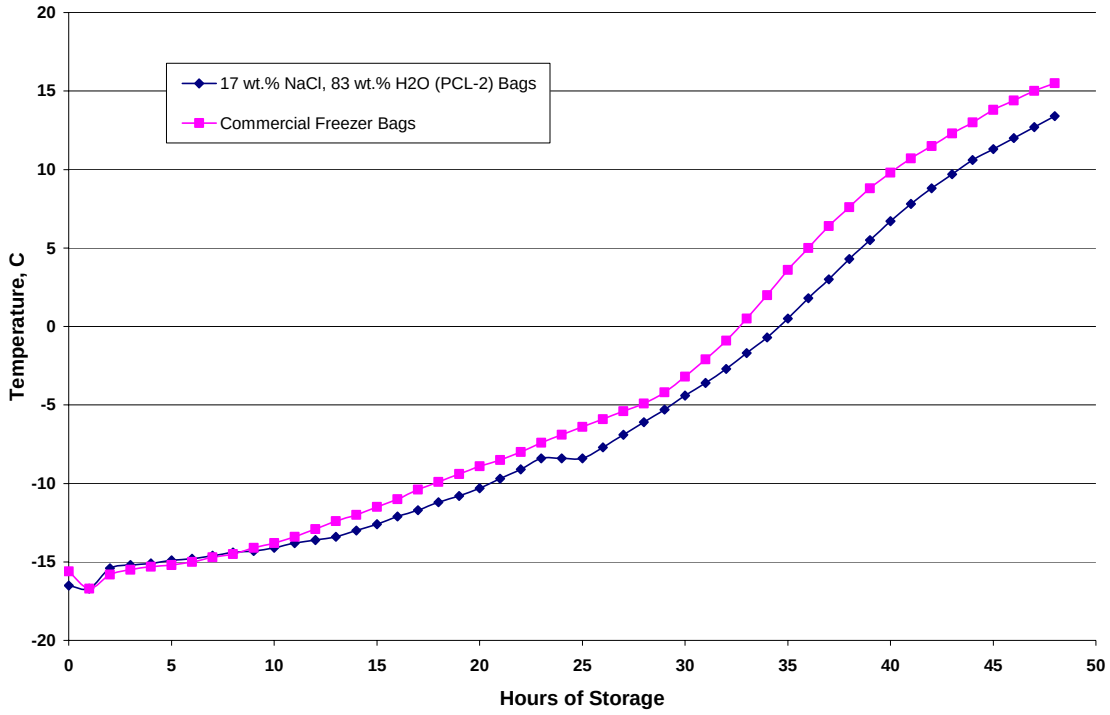

Figure 6. Temperature Profiles for the Cooler Containing Bags of the New PCL Formulation (17 wt.% NaCl and 83 wt.% H_2O) and the Cooler Containing the Commercial Freezer Bags (Cooler Storage at 22-24 ºC, Coolers packed by Mass)

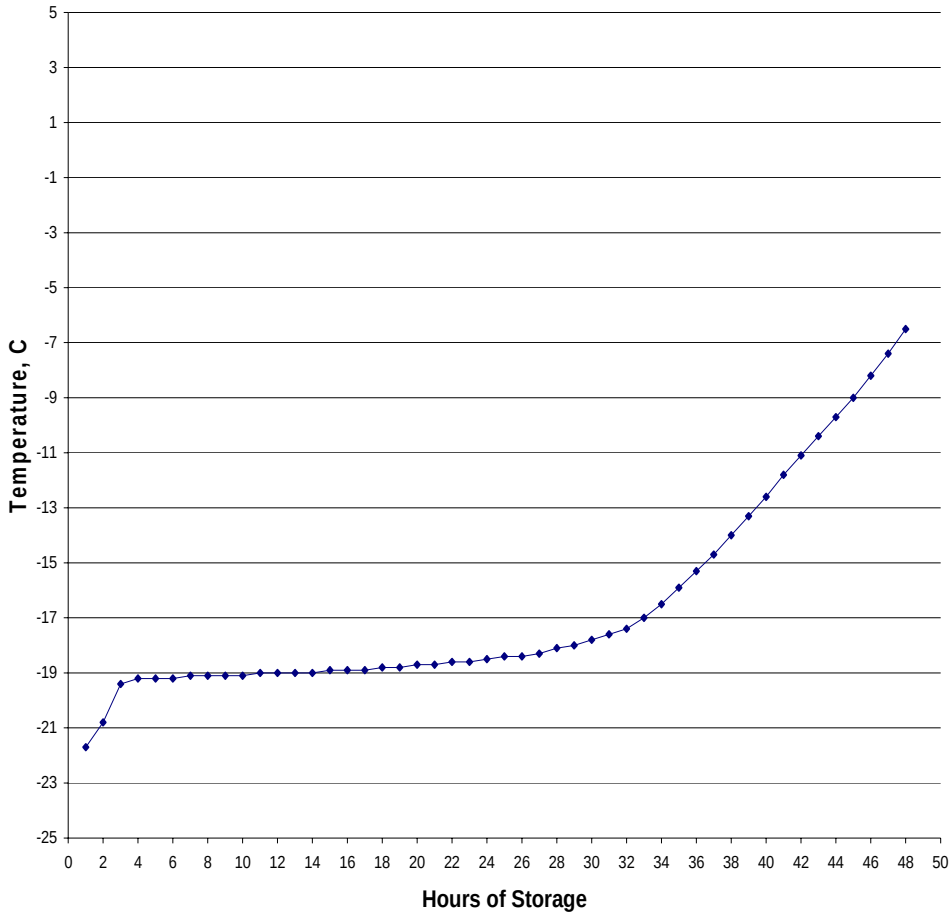

Figure 7. Temperature Profile for the Center of the Igloo MaxCold™ Cooler Containing Bags of the New PCL Formulation, Refrigerated Soil Samples, and Bubble Wrap (Cooler Storage at 23-26 ºC, Cooler packed by Mass)