

# Investigation of Phase- Change Materials for Vaccine Cold Chain Equipment

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## Acronyms

CCE	cold chain equipment
DSC	differential scanning calorimetry
FWHM	full width at half maximum
GHS	Globally Harmonized System
HDPE	high-density polyethylene
HIPS	high-impact polystyrene
PCM	phase-change material
PEG	polyethylene glycol
PP	polypropylene
PVC	polyvinyl chloride
SDS	safety data sheet
T-history	temperature history
$T_{\text{melt}}$	melting-point temperature
$T_{\text{onset}}$	melting onset temperature
$T_{\text{peak}}$	temperature at peak heat flow during phase change

# 1. Introduction

This report presents the results of research and testing of phase-change materials (PCMs) as related to their potential use in vaccine cold chain equipment (CCE). Many vaccines are best stored between 2°C and 8°C and can tolerate limited excursions outside this range as long as they do not freeze or experience excessively high temperatures for too long a period of time. Consequently, we have limited our investigation to PCMs that undergo phase changes in the 0°C to 10°C range. The report is divided into the following sections:

- **Section 2.** Background information on PCMs.
- **Section 3.** Landscape of PCMs potentially used in CCE.
- **Section 4.** Compatibility testing of PCMs with CCE materials.
- **Section 5.** Thermodynamic characterization of PCMs.
- **Section 6.** Discussion and conclusions.

## 2. Background

Many vaccines need to be kept between 0°C and 10°C from manufacture through use to maintain their effectiveness. In low-resource settings where electricity and transportation infrastructure is underdeveloped, keeping vaccines in the desired temperature range is challenging. For stationary CCE such as vaccine refrigerators, an unreliable electricity supply requires that refrigerators maintain cool temperatures in warm environments during frequent intervals without power and thus rely on their ability to slow the absorption of heat into the cold space where the vaccines are kept through a combination of insulation and cold thermal mass. Passive vaccine carriers, without active compressor/thermoelectric cooling, rely exclusively on insulation and cold thermal mass to maintain the desired temperature range.

PCMs are materials that go through a change in phase as they are warmed or cooled. The phase change may be a physical transition (solid-solid, solid-liquid, liquid-gas, solid-gas) or a reversible chemical reaction such as a hydration-dehydration reaction. During the change in phase, the PCM will absorb or release heat while maintaining a nearly constant temperature until all material has reached the new phase. The amount of heat absorbed or released per unit mass during a phase change (latent heat) is typically much larger than the amount of heat absorbed or released per unit mass as a material changes temperature without undergoing a phase change (specific heat). Inclusion of a PCM in the design of CCE can therefore greatly increase the amount of heat that can be absorbed or released while maintaining the desired temperature.

### 2.1 Characteristics

A large number of criteria must be considered in the selection of a PCM for a particular application. The following list of criteria have been adapted from Sharma et al. and Abhat et al.<sup>1,2</sup>

1. Thermal
  - a. Suitable phase-change temperature
  - b. High-latent heat
  - c. Heat-transfer rate
2. Physical
  - a. High density
  - b. Small-volume change during phase change
  - c. Low vapor pressure
3. Kinetic
  - a. No supercooling
  - b. Sufficient crystallization rate
4. Chemical
  - a. Long-term stability
  - b. Material compatibility
  - c. Nontoxic
  - d. Biodegradable
  - e. Low flammability
5. Economic
  - a. Abundant
  - b. Available
  - c. Low price

For CCE applications, we can make some general statements about the above criteria. The initial considerations relate to thermal characteristics of the PCM. To be useful for CCE, the phase change should occur between 0°C to 10°C. Ideally, the nominal phase-change temperature should be near the center of this range to prevent over-cooling or over-heating at the respective extremes of the range. A higher latent heat is better because that means the PCM can absorb more energy without large temperature changes. For CCE, where insulation is important, a low rate of heat transfer is desirable, though lower heat transfer rates require higher temperature gradients and more time to cool the CCE to its target temperature for use.

Regarding physical characteristics, ideally both phases of the PCM should have the same relative chemical concentrations (e.g., 90% chemical A and 10% chemical B) in both solid and liquid phases. PCMs that do not behave this way can have phase separation due to the different densities of the two phases, leading to a reduction of the PCM performance over time. Since latent heat is measured on a per unit mass basis, a higher density PCM can absorb more heat than a lower density PCM with the same latent heat for a given volume of PCM. To reduce equipment complexity and cost, the PCM should undergo minimal volume change during the phase change. So, even though solid-gas and liquid-gas phase changes typically have much larger latent heats than solid-solid or liquid-solid phase changes, a PCM that goes through a gaseous phase change is impractical due to the large change in volume of the PCM. A low vapor pressure reduces the chance of rupturing the PCM primary container, simplifying the design of that component.

Kinetic characteristics include supercooling and the rate of phase change. When a PCM undergoes supercooling, it cools below the phase-change temperature and then rapidly undergoes the phase change.

For example, under some conditions, if water is cooled slowly and left undisturbed, it may cool several degrees below 0°C before beginning to freeze. Supercooling is therefore an undesirable characteristic in a PCM to be used in a freeze-prevention liner. The tendency for supercooling can be reduced by the addition of nucleation particles to a PCM or by careful PCM container design. A high crystallization rate will improve the rate at which the PCM can release heat.

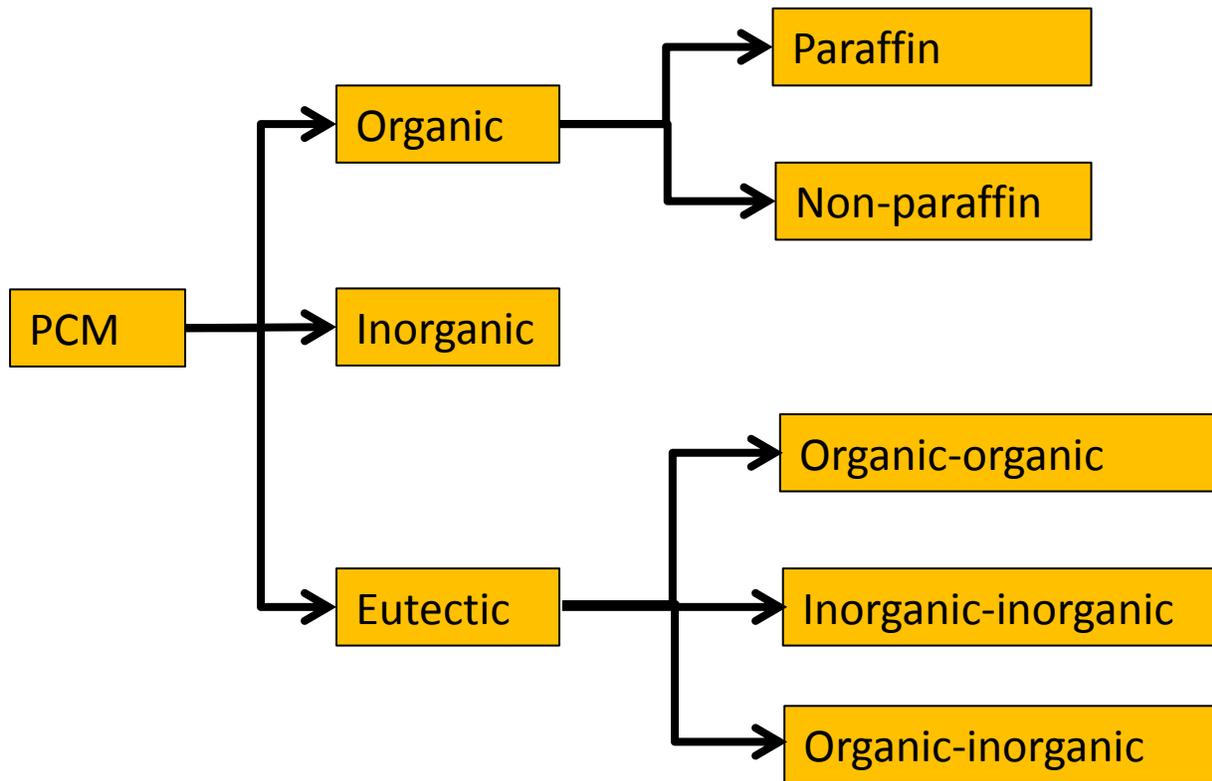
The remaining chemical and economic characteristics will be considered further during the discussion of the PCM landscape.

## 2.2 Classification

Due to the thermal and physical considerations cited above, the most useful PCMs for CCE are those that go through solid-liquid phase transformations. Solid-solid PCMs could also be used since they go through small volume changes during the phase change and are, in some ways, easier to contain. However, the latent heats of the solid-solid PCMs are generally lower than the latent heats of the solid-liquid PCMs,<sup>1</sup> so larger volumes of solid-solid PCM would be required. The PCMs that are currently available in the temperature range suitable for CCE are all of the solid-liquid type, so we will confine our discussion to solid-liquid PCMs.

We have adopted the PCM classification scheme used by Sharma et al. (2009) in our work because it is simple to understand and mirrors classifications used by commercial PCM manufacturers. Figure 1 presents the classification scheme. The scheme breaks down the PCMs by composition; organic, inorganic, and eutectics. The organic PCMs are made of carbon-based compounds. One type of organic PCM is the paraffin consisting of single chains of hydrocarbons. The melting-point temperature of a paraffin can be tailored by adjusting the length of the hydrocarbon chain: the longer the chain, the higher the melting-point temperature. With the exception of melting points, different paraffins are very similar to each other in physical and chemical characteristics, which leads to a single classification. The remaining organic PCMs are grouped together in the non-paraffin category and are very different from each other chemically and physically. The non-paraffin PCM category includes fatty acids, glycols, alcohols, and esters.

Figure 1. Phase-change material classification scheme.



Adapted from Sharma et al. 2009.

The inorganic PCMs consist of materials that are not carbon-based and comprise a wide range of materials including water, hydrated salts, and low-melting-point metals. Of these, water and the hydrated salts are the most useful for CCE. The hydrated salts do not undergo a change in state from solid to liquid, but rather release water molecules from their solid crystal structure with increasing temperature. The release of water from the hydrated salt can lead to the creation of two phases of different composition (i.e., the hydrated salt and a salt-water solution), which can separate due to the higher density of the hydrated salt. The two phases might not easily recombine when the mixture is “refrozen,” leading to decreased PCM performance over repeated cycles unless additional physical or chemical engineering measures are taken to reduce the phase separation.

Water is a great inorganic PCM from many perspectives. It has a very high latent heat; has higher density than many of the organic PCMs; is stable, nontoxic, readily available, and cheap; and expands only moderately during freezing. However, water’s phase change occurs at 0°C, the very low end of the desirable range for CCE, and water is prone to supercooling, making it less than ideal for vaccine freeze-prevention applications.

The eutectics consist of combinations of compounds that will melt/freeze over a very narrow temperature range at a very specific relative concentration of the compounds. The eutectics may be combinations of organic, inorganic, or organic and inorganic compounds. For example, sodium chloride and water form a eutectic solution at a relative concentration of 76.7% water to 23.3% salt by mass. This solution melts/freezes at exactly -21.1°C. Melting/freezing of the two components of the eutectic occurs

simultaneously (congruent melting/freezing). Many of the commercially available eutectics are based on organic and/or inorganic compounds mixed with water and have melting points below 0°C, rendering them unsuitable for CCE.

## 2.3 Packaging

In addition to the PCM itself, consideration should be given to how it is contained. The composition, size, and shape of the container are all important. Certain combinations of PCM and packaging material are incompatible due to the PCM corroding, penetrating, or dissolving the material. Large-volume containers may be thermally inefficient if heat transfer through the PCM is slow. A smaller package with a higher surface area-to-volume ratio or a large-volume package incorporating fins to transfer heat quickly into the volume of the PCM may be preferable for a PCM with slow heat transfer. Packaging can be designed to reduce phase separation for PCMs where this is an issue.

A particular type of PCM packaging allowing novel applications is encapsulation. PCMs may come encapsulated in polymer spheres ranging from macroscopic to microscopic. Encapsulated PCMs may then be incorporated into fibers or compressed together into blocks. PCM encapsulation can improve PCM performance and stability by reducing phase separation during freezing and melting and protecting the PCM from chemical or physical degradation. In particular, eutectic and salt hydrate PCMs depend on precise concentrations of the components. Loss of volatile components from eutectic and salt hydrate PCMs (i.e., the evaporation of water) can adversely affect their performance and useful life. Encapsulation may also decrease risk of PCMs leaking out of their primary containment. However, use of encapsulation can decrease the effective density of a PCM and thus require larger volumes of encapsulated PCMs than the non-encapsulated PCM. The addition of encapsulation material also affects disposal considerations. For example, a biodegradable PCM that is encapsulated in a non-biodegradable plastic becomes a non-biodegradable waste.

## 3. Landscape

We assembled a PCM landscape based on information from two sources: 1) a search of the 2011 Alfa Aesar chemical catalog for chemicals with melting points between 0°C and 10°C; and 2) an Internet search of manufacturers of PCM-specific products with melting points between 0°C and 10°C. These two categories of PCM will be referred to as chemical and engineered PCMs, respectively.

The list of potential chemical and engineered PCMs was further narrowed using additional criteria from the chemical and economic factors given in section 2 above. Strong consideration was given to health and safety hazards associated with the potential PCMs. Given the possibility that PCMs could leak from their primary containment and come into contact with people and the environment, potential PCMs were screened for health and environmental hazards based on the information in each PCM's safety data sheet (SDS) or from the supplier's website. More specifically, hazard screening was based on the Globally Harmonized System (GHS) hazard codes. The GHS codes are a standardized, international coding system for the health and environmental hazards associated with a chemical. SDS and GHS hazard codes were

consulted for all of the chemical PCMs. For some of the engineered PCMs, the SDS was not readily available. Many engineered PCMs have relatively minor health hazards, so we kept PCMs with minor health hazards to avoid narrowing the PCM landscape too much. Specifically, PCMs with the following GHS codes were permitted in the landscape:

- H227 (combustible liquid)
- H302 (harmful if swallowed)
- H304 (may be fatal if swallowed and enters airway)
- H305 (may be harmful if swallowed and enters airway)
- H319 (causes serious eye irritation)

PCMs with a flashpoint below 100°C were removed due to flammability hazards. Because the chemical inventory contained many extremely expensive chemicals with melting points in the correct range, PCMs costing over \$200/kg were removed from the list. The prices used were the lowest prices from either the Alfa Aesar or Sigma-Aldrich online catalogs for the chemical PCMs.

Since CCE has a finite useful life, consideration must also be given to disposal of PCMs. Where available, the SDS or manufacturer information was consulted for disposal recommendations. All consulted sources, however, stated that the PCMs should be disposed of in accordance with local regulations.

Application of the screening criteria to the chemical and engineered PCMs yielded the following results. For the chemical PCMs, 206 PCMs were identified in the Alfa Aesar database as having melting points in the correct range. After screening for health and environmental hazards, 156 chemical PCMs remained. After applying the cost screening criteria, 3 chemical PCMs remained.

The web search identified 44 engineered PCMs in the correct temperature range. Applying the health and environmental hazard criteria, 42 engineered PCMs remained.

Results of the PCM landscape are presented in Table 1.

Table 1. Phase-change material (PCM) landscape.

PCM	Manufacturer	Type	Class	Subclass	Nominal phase change temperature °C	Upper temperature °C	Lower temperature °C	Latent heat J/g	Specific heat, liquid J/(g·°C)	Density, liquid g/cc	Specific heat, solid J/(g·°C)	Density, solid g/cc	Biodegradable	Cost per kg
CrodaTherm 6.5	Crodatherm	Engineered	Organic	Non-paraffin	6.5	7	2.5	183	2.10	—	1.60	—	—	—
Cryogel 0	Cryopak	Engineered	Inorganic	—	0	5	-5	—	—	—	—	—	—	—
Phase 5	Cryopak	Engineered	Organic	Non-paraffin	5	10	0	—	—	—	—	—	—	—
PCM 0	Global Energy Systems	Engineered	Inorganic	Water	0	—	—	—	—	—	—	—	—	—
PCM 8	Global Energy Systems	Engineered	Inorganic	Salt hydrate	8	—	—	—	—	—	—	—	—	—
PC0	Phase Change Products	Engineered	Inorganic	Salt hydrate	0	5	-5	333	4.1	—	2	0.917	—	—
PlusICE® S7	PCMProducts	Engineered	Inorganic	Salt hydrate	7	—	—	150	1.85	1.700	—	—	—	—
PlusICE® S8	PCMProducts	Engineered	Inorganic	Salt hydrate	8	—	—	150	1.90	1.475	—	—	—	Note 2
PlusICE® S10	PCMProducts	Engineered	Inorganic	Salt hydrate	10	—	—	155	1.90	1.470	—	—	—	—
PlusICE® A2	PCMProducts	Engineered	Organic	Paraffin	2	—	—	200	2.2	0.765	—	—	—	—
PlusICE® A3	PCMProducts	Engineered	Organic	Paraffin	3	—	—	200	2.2	0.765	—	—	—	—
PlusICE® A4	PCMProducts	Engineered	Organic	Paraffin	4	—	—	200	2.2	0.766	—	—	—	—
PlusICE® A6	PCMProducts	Engineered	Organic	Non-paraffin	6	—	—	150	2.2	0.770	—	—	—	Note 2
PlusICE® A8	PCMProducts	Engineered	Organic	Paraffin	8	—	—	150	2.16	0.773	—	—	—	—
PlusICE® A9	PCMProducts	Engineered	Organic	Paraffin	9	—	—	140	2.16	0.775	—	—	—	—
PlusICE® E0	PCMProducts	Engineered	Eutectic <sup>3</sup>	—	0	—	—	332	4.186	1.000	—	—	—	Note 2
Frost HS01	Pluss	Engineered	Inorganic	Salt hydrate	0	—	—	290	3.60	1.010	—	0.920	—	—
OM 03	Pluss	Engineered	Organic	Non-paraffin	3.5	—	—	240	0.73	0.835	—	0.935	—	—
OM 05	Pluss	Engineered	Organic	Non-paraffin	5.5	—	—	180	0.73	0.849	—	0.935	—	—
OM 08	Pluss	Engineered	Organic	Non-paraffin	8	—	—	—	—	1.050	—	0.999	—	—
PureTemp 1	PureTemp	Engineered	Organic	Non-paraffin	1	2	-1	301	2.43	1.000	2.32	1.100	—	\$11.11
PureTemp 6	PureTemp	Engineered	Organic	Non-paraffin	6	7	5.5	167	2.23	0.860	2.13	0.950	—	\$11.11
PureTemp 8	PureTemp	Engineered	Organic	Non-paraffin	8	9	7	178	2.15	0.860	2.85	0.950	—	\$9.09
PCM-HS01P	RGEES	Engineered	Inorganic	Water	0	—	—	290	0.99	1.010	—	0.920	—	\$30.00
PCM-OM06P	RGEES	Engineered	Organic	Paraffin	6	6	5	260	—	0.735	—	0.780	—	\$15.00
RT 3 HC_1	Rubitherm	Engineered	Organic	Paraffin	3	3	1	Note 1	—	0.770	—	0.880	—	\$11.00
RT 4	Rubitherm	Engineered	Organic	Paraffin	4	—	—	—	—	—	—	—	—	—
RT 0	Rubitherm	Engineered	Organic	Paraffin	0	2	-1	225	—	0.770	—	0.880	Yes	—
RT 2 HC	Rubitherm	Engineered	Organic	Paraffin	2	3	1	—	—	0.770	—	0.880	Yes	—
RT 5	Rubitherm	Engineered	Organic	Paraffin	5	7.5	4	—	—	0.770	—	0.880	Yes	—
RT 5 HC	Rubitherm	Engineered	Organic	Paraffin	5.5	6	5	—	—	0.760	—	0.880	Yes	—
RT 6	Rubitherm	Engineered	Organic	Paraffin	6	7	2	—	—	0.770	—	0.880	—	—
RT 8	Rubitherm	Engineered	Organic	Paraffin	7.5	9	6	—	—	0.770	—	0.880	Yes	—
RT 8 HC	Rubitherm	Engineered	Organic	Paraffin	8	9	7	—	—	0.770	—	0.880	Yes	—
RT 9	Rubitherm	Engineered	Organic	Paraffin	9	11	8	—	—	0.760	—	0.880	Yes	—
RT 10	Rubitherm	Engineered	Organic	Paraffin	10	11	8	—	—	0.770	—	0.880	—	—
04	Teappcm	Engineered	Inorganic	Salt hydrate	4	—	—	—	—	—	—	—	—	—
07	Teappcm	Engineered	Inorganic	Salt hydrate	7	—	—	135	—	1.400	—	—	—	—
07b	Teappcm	Engineered	Inorganic	Salt hydrate	7	—	—	300	—	1.400	—	—	—	—
10	Teappcm	Engineered	Inorganic	Salt hydrate	10	—	—	—	—	—	—	—	—	—
va-Q-accu +0	Va-Q-Tec	Engineered	Inorganic	—	0	—	—	330	4.2	—	2.1	—	—	—
va-Q-accu +4	Va-Q-Tec	Engineered	Organic	Paraffin	4	8	2	180	2.4	—	1.8	—	—	—
4-(4-Methoxyphenyl)-2-butanone	Sigma Aldrich	Chemical	Organic	Non-paraffin	8	—	—	—	—	1.046	—	—	—	\$92.00
Polyethylene glycol 400	Sigma Aldrich	Chemical	Organic	Non-paraffin	6	8	4	—	—	1.128	—	—	—	\$137.60
Glycerol triacetate	Sigma Aldrich	Chemical	Organic	Non-paraffin	3	—	—	—	—	1.158	—	—	—	\$159.76

Notes:

1. Heat storage capacity between -5°C and 9°C of 200 J/g given instead of latent heat.
2. Minimum order charge applied when ordered for testing, so price is not applicable for calculating per kg price.
3. PCMProducts markets E0 under the eutectic category. According to PCMProducts, E0 consists of water, a biocide, and nucleating agent, and is, therefore, unlikely to be a true eutectic mixture. PCMProducts would not confirm that E0 is a eutectic.

## 4. Material compatibility testing

Durability of CCE equipment is important for vaccination efforts for economic and logistical reasons. Since the use of PCMs in CCE is relatively new, understanding the potential impact of PCMs on the materials that make up the CCE is useful for decision-making regarding how best to use PCMs in CCE. Prior studies have looked at the effect of PCMs on materials such as plastic and metal in the context of thermal energy storage for solar energy and air conditioning,<sup>3,4</sup> but the PCMs in these studies had melting points outside the useful range for CCE and were compositionally different.

### 4.1 Selection of PCMs

To address the lack of information on material compatibility with PCMs relevant to CCE, six engineered and two chemical PCMs from the landscape were selected for material compatibility testing. At least one PCM of each subclass was selected. A variety of melting-point temperatures were selected, and the two nominal 6-degree-melting-point paraffins from different manufacturers were selected for comparison to each other. Table 2 gives the PCMs selected for testing. Distilled water was included in the compatibility testing as a comparison to other PCMs.

Table 2. Phase-change materials (PCMs) selected for testing.

PCM	Manufacturer/supplier	Type	Class	Subclass	Description
A6	PCMProducts	Engineered	Organic	Paraffin	Long-chain hydrocarbon
RT3HC	Rubitherm	Engineered	Organic	Paraffin	Long-chain hydrocarbon
OM06P	RGEES	Engineered	Organic	Paraffin	Long-chain hydrocarbon
PureTemp 8	Entropy Solutions	Engineered	Organic	Non-paraffin	Fatty acid
S8	PCMProducts	Engineered	Inorganic	Salt hydrate	Hydrated sodium sulfate, ammonium chloride
E0	PCMProducts	Engineered	Eutectic	Inorganic-organic	Water with biocide
PEG 400	ChemWorld	Chemical	Organic	Non-paraffin	Fatty acid ester
Triacetin	Sigma-Aldrich	Chemical	Organic	Non-paraffin	Fatty acid ester
Distilled water	Mountain Mist	Chemical	Inorganic		

Abbreviation: PEG, polyethylene glycol.

## 4.2 Material selection and testing methods

PCMs may come in contact with a wide range of container materials and, during unexpected leaks, with CCE structural, insulation, and electrical components. An investigation of World Health Organization Performance, Quality and Safety prequalified refrigerators and vaccine carriers led to a list of three metals, four plastics, two foams, one paint, and polyvinyl chloride (PVC)-coated copper wire for compatibility testing with the nine selected PCMs. To observe changes to mechanical properties that would impact CCE structural integrity, metal and plastic samples were tested in tension, and foam was tested in compression before and after exposure to PCMs. Painted steel samples and electrical hookup wire were visually inspected after exposure; the painted steel was also qualitatively scratch-tested. The exposure was conducted with two batches of samples that were started simultaneously: a short-term batch that soaked in liquid PCMs for approximately 2 to 3 weeks, and a long-term batch that will soak for approximately 3 months. At the time this report was written, the long-term batch was still soaking, so this report presents only the results from the short-term batch. Results from the long-term batch will be presented in an annex to this report.

### 4.2.1 Metals and plastics

Tensile stress tests were performed with an Instron® 5565 materials testing machine for the samples in Table 3 except for high-impact polystyrene (HIPS). The HIPS samples were a late addition to the compatibility testing and were evaluated qualitatively by manual flexing to check for obvious changes in mechanical properties.

Table 3. Materials selected for phase-change material compatibility test.

Material	Application in CCE	Thickness (mm) and shape	Test strain rate (mm/min)
Aluminum (6061)	Refrigerator—structural	0.50 dumbbell	2.54
Copper	Refrigerator—wiring, heat exchange coils	0.65 dumbbell	0.15
Steel (mild, structural)	Refrigerator—structural	0.65 dumbbell	0.50
Polyvinyl chloride	Refrigerator—wire coating	1.45 dumbbell	10.00
Polypropylene	Vaccine carrier—structural	1.45 dumbbell	10.00
High-density polyethylene	Vaccine carrier—structural	1.45 dumbbell	10.00
High-impact polystyrene	Vaccine carrier—lining Refrigerator—non-structural components	1.50 rectangle	NA

Abbreviation: CCE, cold chain equipment.

Metal and plastic samples were removed from the PCM immersion containers and cleaned with dry paper towel and small amounts of isopropyl alcohol. PCMs that were known to be water based, such as S8, were cleaned with water instead of alcohol. Each dumbbell was weighed and measured for thickness (on each end) and width (three equally spaced locations on the thin region) before and after soaking (Figure 2). HIPS samples were not measured for dimensions or mass.

Figure 2. Post-immersion sample measurements. Width (left) was measured at the gauge length positions shown by the white dots and at the center. Thickness (right) was measured on each sample end.



ASTM International material testing documentation was used as a guide for strain rates, dumbbell dimensions, and calculations. From extensive preliminary trials, the ideal strain rates (within ASTM ranges) were chosen to balance accuracy and test duration (Table 3). Samples were positioned in the material-appropriate Instron® grips and pulled to failure while measuring applied force and strain. Engineering stresses were calculated from the pretest cross-sectional area. Figure 3 shows an example of a polypropylene sample during a tensile test.

Figure 3. Polypropylene dumbbell in the Instron® tension grips. After a certain load threshold is surpassed, the sample turns opaque in the most deformed region (thin, center).



#### 4.2.2 Foam

Compression stress tests were performed with an Instron® 5565 materials testing machine for the samples in Table 4. Foam blocks were compressed between platens as shown in Figure 4, and the compression force and the strain were measured. Stress was calculated from the pretest cross-sectional area.

Table 4. Selected materials for the phase-change material (PCM) compatibility test.

Material	Source	Height x length x width (mm) and shape	Test strain rate (mm/min)
Polyurethane foam	Vaccine carrier—insulation	38 x 50 x 50 block	3.81
Polyisocyanurate foam	Refrigerator/freezer—insulation	38 x 50 x 50 block	3.81

Figure 4. Polyurethane block post polyethylene glycol (PEG) 400 immersion being compression tested. Note the PEG 400 releasing from the foam block.



#### 4.2.3 Changes in sample dimensions and mass

Average changes in dimension and mass are reported in Tables 5 to 7. Changes in the dimensions and mass correlate with changes in material properties observed in the Instron® testing data. Large changes in dimension and mass are highlighted in yellow. HIPS samples were not measured for changes in dimension or mass.

Table 5. Changes in dimension and mass for plastic samples (short-term test).

PCM	PVC			PP			HDPE		
	Average change in average width (%)	Average change in average thickness (%)	Average change in mass (%)	Average change in average width (%)	Average change in average thickness (%)	Average change in mass (%)	Average change in average width (%)	Average change in average thickness (%)	Average change in mass (%)
PureTemp 8	0.31	0.11	0.10	0.35	-0.23	0.00	0.80	1.66	1.39
Triacetin	0.27	0.11	0.10	0.07	-0.12	0.17	0.33	0.22	0.47
OM06P	0.42	0.11	0.11	0.31	-0.24	1.91	1.99	0.99	5.19
PEG 400	0.49	0.33	0.11	0.57	0.12	0.00	0.29	0.11	0.31
RT3HC	0.02	-0.33	-0.10	0.44	0.35	1.04	1.78	1.91	4.92
Distilled water	0.24	0.11	0.11	-0.52	-1.70	0.00	0.34	-0.33	-0.16
S8	0.29	0.54	-0.63	0.38	0.53	-1.38	1.06	0.78	-0.62
E0	0.02	0.86	-0.94	0.09	0.47	-1.72	0.35	0.89	-1.71
A6	-0.13	1.41	-0.10	0.27	0.70	0.69	0.79	1.98	4.19

Abbreviations: HDPE, high-density polyethylene; PCM, phase-change material; PEG, polyethylene glycol; PP, polypropylene; PVC, polyvinyl chloride.

Table 6. Changes in dimension and mass for metal samples (short-term test).

PCM	Copper			Aluminum			Steel		
	Average change in average width (%)	Average change in average thickness (%)	Average change in mass (%)	Average change in average width (%)	Average change in average thickness (%)	Average change in mass (%)	Average change in average width (%)	Average change in average thickness (%)	Average change in mass (%)
PureTemp 8	-0.57	1.07	0.34	-0.62	1.60	0.68	-0.80	1.37	-0.11
Triacetin	-0.60	1.06	0.14	-0.74	1.89	-0.45	-0.76	2.07	-0.11
OM06P	-0.48	1.06	0.20	-0.77	1.08	0.46	-0.95	1.02	-0.11
PEG 400	-0.19	1.59	0.00	-0.64	1.61	-0.23	-0.78	1.71	0.43
RT3HC	-0.45	1.78	-0.07	-0.75	1.34	-0.23	-0.66	1.02	0.11
Distilled water	-0.34	1.60	-0.20	-0.81	1.62	-0.23	-0.64	2.41	0.21
S8	-0.77	1.60	-1.42	-0.02	2.68	0.00	-0.19	6.12	-0.32
E0	-0.28	2.93	-0.07	0.64	24.86	2.49	-0.14	4.42	-1.27
A6	-0.69	1.57	0.00	-0.84	1.06	0.23	-0.71	1.70	0.11

Abbreviations: PCM, phase-change material; PEG, polyethylene glycol.

Table 7. Changes in volume and mass for foam samples (short-term test).

PCM	Polyisocyanurate		Polyurethane	
	Average change in volume (%)	Average change in mass (%)	Average change in volume (%)	Average change in mass (%)
PureTemp 8	-0.14	86.1	-1.05	59.9
Triacetin	-0.23	131	-2.83	122
OM06P	-0.24	74.4	-0.60	55.6
PEG 400	-0.46	125	-9.61	341
RT3HC	0.15	82.5	-0.74	61.4
Distilled water	1.23	73.0	-0.37	132
S8	1.27	77.8	-0.82	133
E0	1.42	42.2	-0.55	63.8
A6	0.41	62.8	-0.31	56.7

Abbreviations: PCM, phase-change material; PEG, polyethylene glycol.

The most significant changes in dimensions and mass for the plastic samples were for high-density polyethylene (HDPE). The paraffin PCMs and PureTemp 8 were absorbed by the HDPE samples, resulting in significant increases in mass, thickness, and width. These results are reflected in the changed mechanical properties of paraffin-soaked HDPE in the Instron® testing Table 11 and Figures 11 to 13.

The most significant changes in dimension and mass for the metal samples were for copper soaked in S8, aluminum soaked in E0, and steel soaked in E0 and S8. Copper decreased in mass, aluminum increased in thickness and mass, and steel increased in thickness for E0 and S8 while decreasing in mass for E0.

The largest change in mass and volume of the foams was for polyurethane soaked in polyethylene glycol (PEG) 400. The polyurethane samples gained significant mass, suggesting that the polyurethane absorbed PEG 400. The polyurethane also lost a high percentage of its volume, suggesting that the PEG 400 impacted the material properties of the polyurethane.

#### 4.2.4 Instron® testing data and observations

Sections 4.3 through 4.5 present statistical summaries of the Instron® testing data for each material. The calculations made from the testing data for each material are summarized in Table 8 below. HIPS samples were not tested on the Instron® but were flexed manually to check for obvious changes in mechanical properties.

Table 8. Instron® testing calculations.

Material type	Material	Yield stress (at strain)	Offset yield stress (at strain)	Ultimate tensile strength	Young's modulus	Compressive modulus
Plastic	PP		x (2%)	x	x	
	PVC		x (0.2%)	x	x	
	HDPE		x (1%)	x	x	
Metal	Copper		x (0.2%)	x	x	
	Aluminum		x (0.2%)	x	x	
	Steel			x		
Foam	Polyisocyanurate	x (5%)				x
	Polyurethane	x				x

Abbreviations: HDPE, high-density polyethylene; PP, polypropylene; PVC, polyvinyl chloride.

Error bars given on bar graphs are standard deviations of the graphed quantity. The baseline standard deviations are extended across the graphs to ease comparisons between properties of the samples soaked in PCM and the baseline samples. The “number of samples” data used in the calculation is given in the data tables. Most materials have data from triplicate samples; however, some tests failed and duplicate or single data points were used.

### 4.3 Plastics

#### 4.3.1 Polypropylene

Polypropylene (PP) showed a slight reduction in offset yield stress and ultimate tensile strength for the organic engineered PCMs (A6, RT3HC, OM06P, PureTemp 8). S8 caused a decrease in offset yield stress and an increase in Young’s modulus. Other PCMs caused little change in material characteristics when compared to baseline. Results are given in Table 9 and Figures 5 to 7.

Table 9. Polypropylene testing results.

PCM	Average Young's modulus	Standard deviation Young's modulus	Average offset yield stress	Standard deviation offset yield stress	Average ultimate tensile strength	Standard deviation ultimate tensile strength	Number of samples
	MPa	MPa	MPa	MPa	MPa	MPa	
Baseline	1,400	100	29.0	0.3	34.88	0.04	3
Distilled water	1,400	100	29	1	34.8	0.1	3

PCM	Average Young's modulus	Standard deviation Young's modulus	Average offset yield stress	Standard deviation offset yield stress	Average ultimate tensile strength	Standard deviation ultimate tensile strength	Number of samples
	MPa	MPa	MPa	MPa	MPa	MPa	
A6	1,500	200	24	1	32.5	0.1	3
RT3HC	1,250	20	24.1	0.2	32.26	0.04	3
OM06P	1,110	30	24.4	0.1	32.4	0.2	3
PureTemp 8	1,300	100	27.4	0.4	34.0	0.2	3
S8	2,500	400	27.8	0.2	34.90	0.04	3
E0	1,700	100	29	1	35.1	0.3	3
PEG 400	1,500	100	28.5	0.1	34.6	0.1	3
Triacetin	1,540	70	28.7	0.3	34.8	0.1	3

Abbreviations: PCM, phase-change material; PEG, polyethylene glycol.

Figure 5. Average offset yield stress for polypropylene soaked in phase-change material.

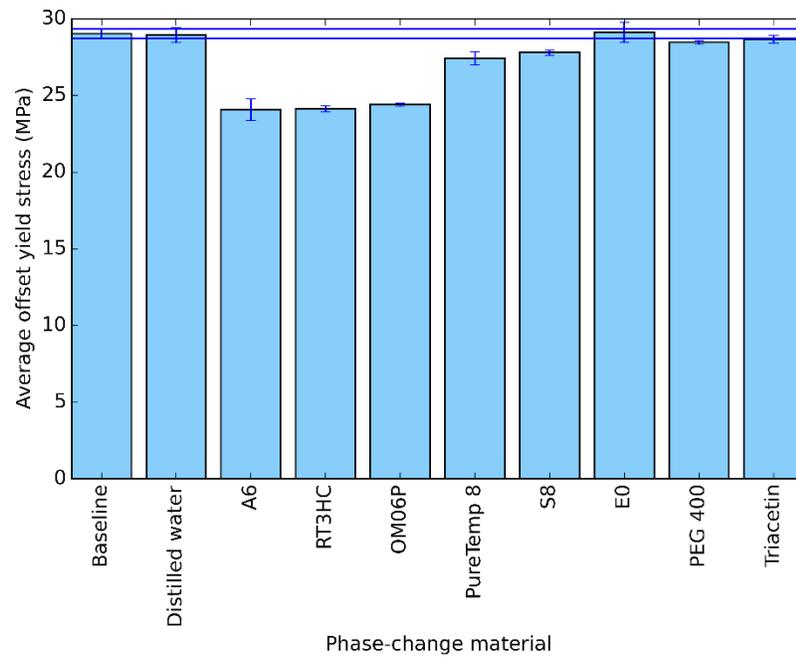


Figure 6. Average ultimate tensile strength for polypropylene soaked in phase-change material.

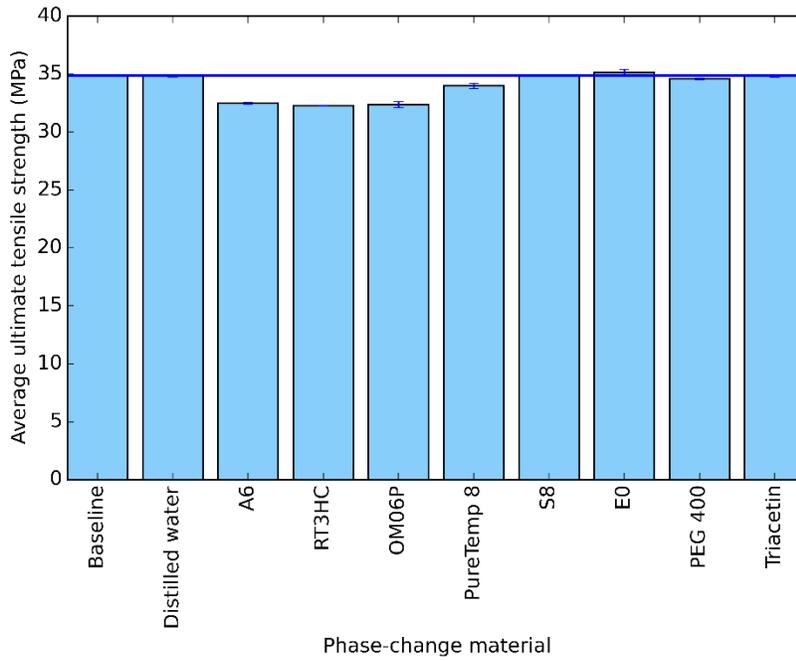
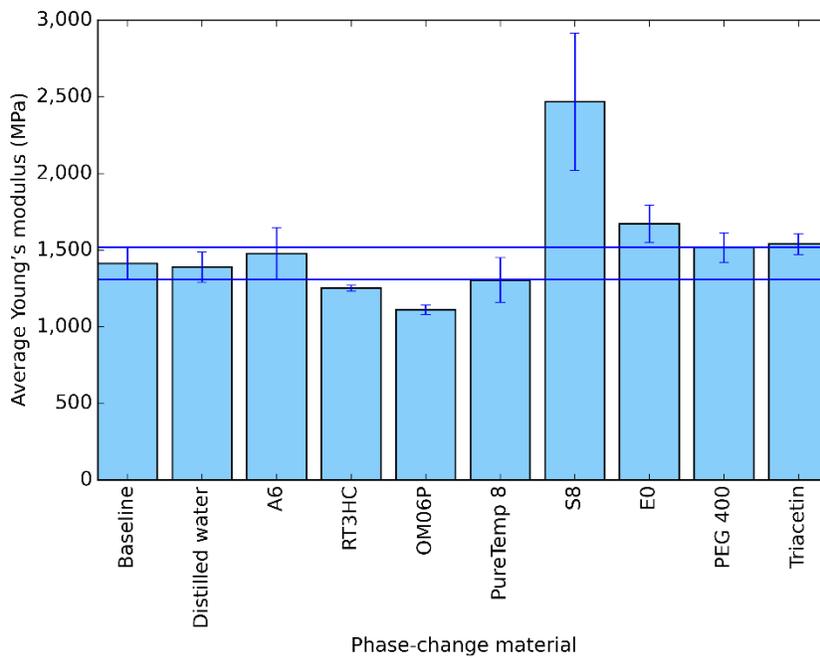


Figure 7. Average Young's modulus for polypropylene soaked in phase-change material.



### 4.3.2 Polyvinyl chloride

Offset yield stress and ultimate tensile strength were not significantly affected by any of the PCMs other than a slight decrease in offset yield stress for E0. Young’s modulus was slightly increased for all PCMs except distilled water, PureTemp 8, and triacetin. Results are given in Table 10 and Figures 8 to 10.

Table 10. Polyvinyl chloride testing results.

PCM	Average Young’s modulus	Standard deviation Young’s modulus	Average offset yield stress	Standard deviation offset yield stress	Average ultimate tensile strength	Standard deviation ultimate tensile strength	Number of samples
	MPa	MPa	MPa	MPa	MPa	MPa	
Baseline	2,310	70	51	1	59	1	3
Distilled water	2,500	100	52	1	59.5	0.5	3
A6	3,100	300	47	5	59.7	0.4	3
RT3HC	2,600	100	52	1	60.5	0.3	3
OM06P	2,700	200	49	4	59.5	0.5	3
PureTemp 8	2,430	60	52	2	59.3	0.2	3
S8	2,730	70	53	2	60	1	2
E0	3,000	100	48	1	59.3	0.4	3
PEG 400	2,690	40	49	1	60	1	3
Triacetin	2,500	200	51	2	60	1	3

Abbreviations: PCM, phase-change material; PEG, polyethylene glycol.

Figure 8. Average offset yield stress for polyvinyl chloride soaked in phase-change material.

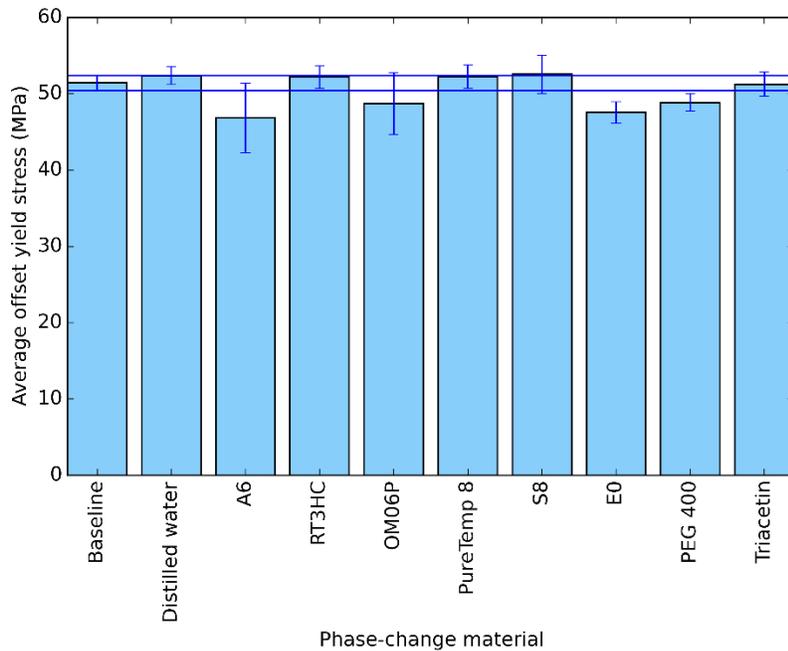


Figure 9. Average ultimate tensile strength for polyvinyl chloride soaked in phase-change material.

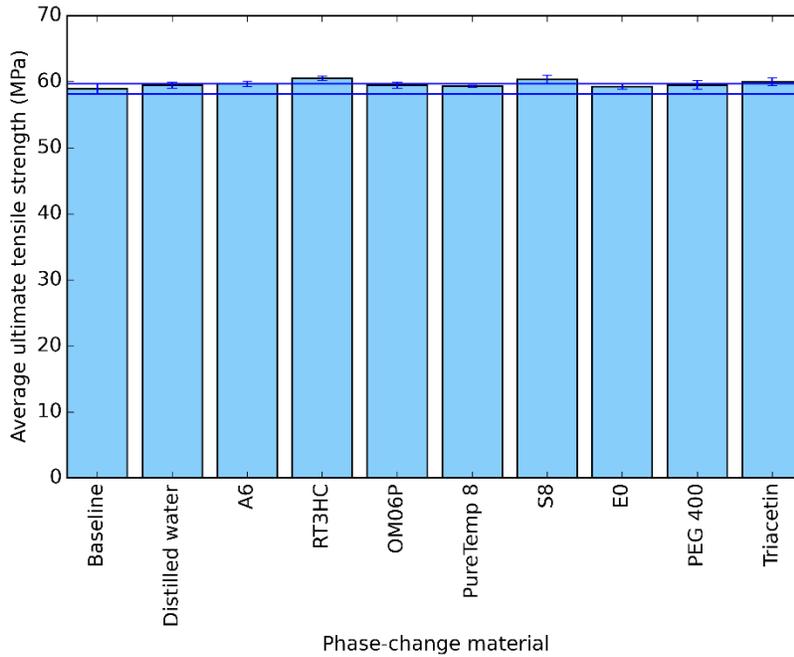
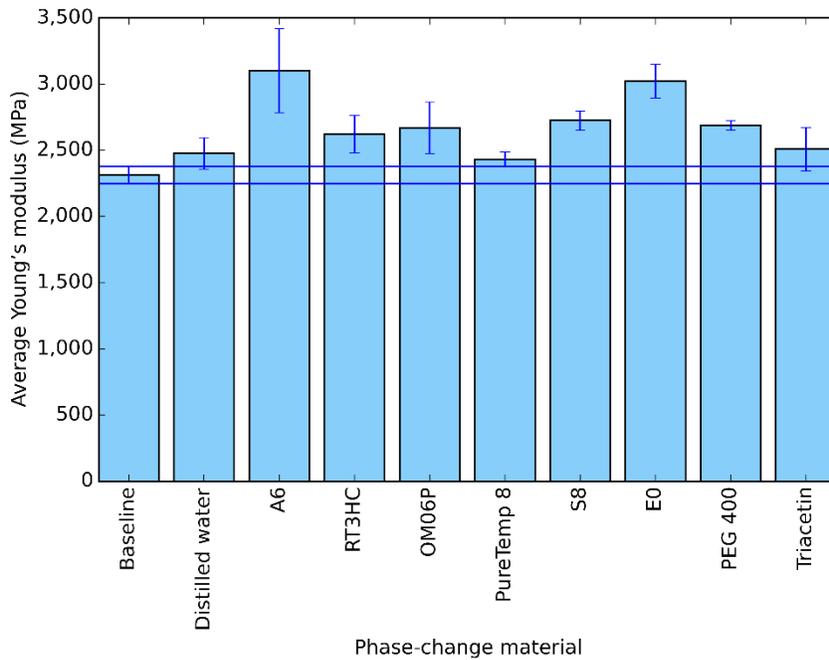


Figure 10. Average Young's modulus for polyvinyl chloride soaked in phase-change material.



### 4.3.3 High-density polyethylene

Offset yield stress, ultimate tensile strength, and Young's modulus were all significantly decreased by the engineered organic PCMs. The other PCMs had little or no effect. Results are given in Table 11 and Figures 11 to 13.

Table 11. High-density polyethylene testing results.

PCM	Average Young's modulus	Standard deviation Young's modulus	Average offset yield stress	Standard deviation offset yield stress	Average ultimate tensile strength	Standard deviation ultimate tensile strength	Number of samples
	MPa	MPa	MPa	MPa	MPa	MPa	
Baseline	800	20	19.1	0.3	25.1	0.3	3
Distilled water	720	20	18	1	24.7	0.2	3
A6	450	20	14.5	0.3	21.4	0.2	3
RT3HC	400	30	14.5	0.5	21.3	0.1	3
OM06P	405	6	13.7	0.1	20.6	0.1	3
PureTemp 8	590	20	16.1	0.1	23.1	0.3	3
S8	870	60	19.2	0.2	25.0	0.1	3
E0	750	70	19.9	0.3	25.1	0.1	3
PEG 400	810	30	19.3	0.1	25.2	0.1	3
Triacetin	822	8	18.9	0.2	25.0	0.1	3

Abbreviations: PCM, phase-change material; PEG, polyethylene glycol.

Figure 11. Average offset yield stress for high-density polyethylene soaked in phase-change material.

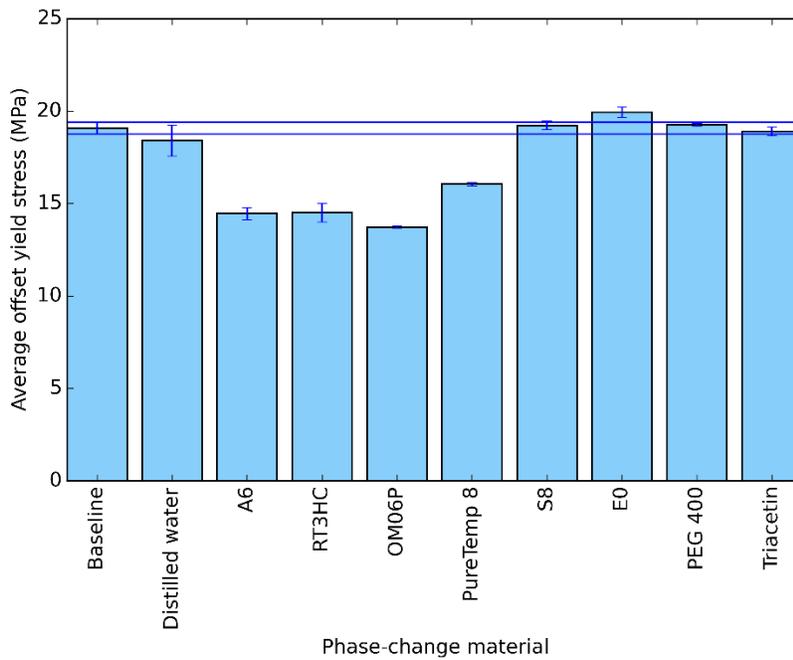


Figure 12. Average ultimate tensile strength for high-density polyethylene soaked in phase-change material.

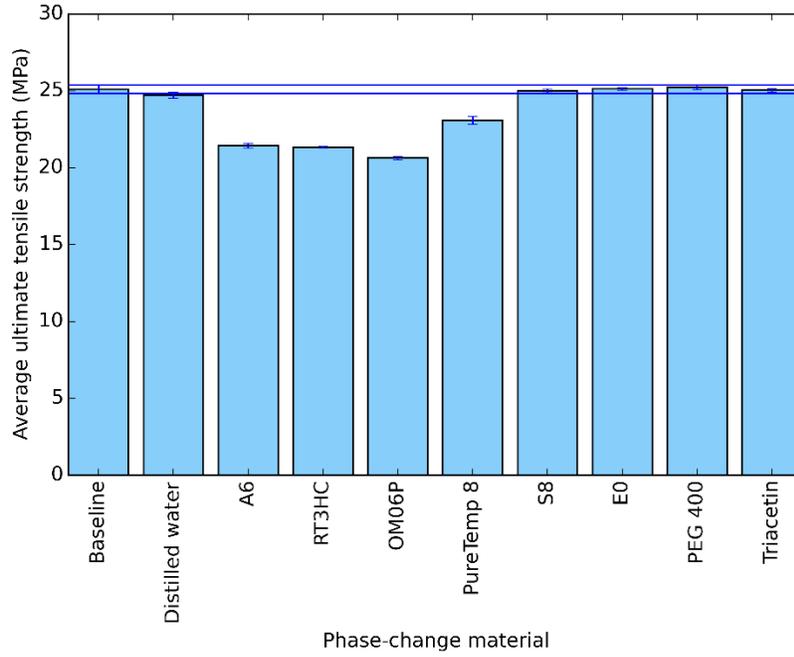
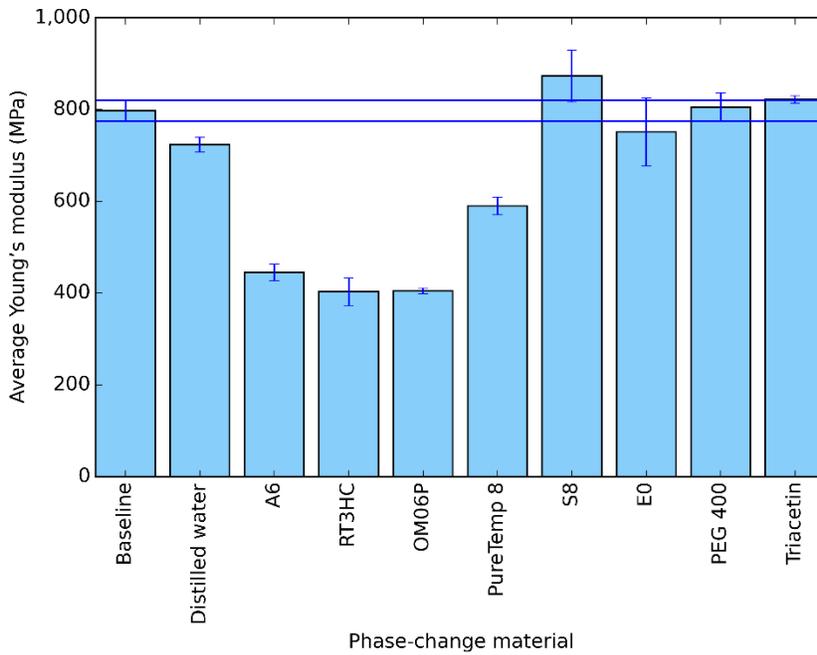


Figure 13. Average Young's modulus for high-density polyethylene soaked in phase-change material.



#### 4.3.4 High-impact polystyrene

Flexing of the HIPS samples by hand did not show obvious changes to the samples for any of the PCMs except for the samples soaked in PureTemp 8. These samples were noticeably weakened and broke easily when flexed.

### 4.4 Metals

As expected, the much higher Young's moduli of the metals compared to the plastics led to low strains during tensile testing that were much more difficult to measure accurately than the strains in the plastic samples. The strain measurements had a lower signal-to-noise ratio, making the determination of a Young's modulus and offset yield stress more difficult. As the Young's modulus increased from aluminum through copper to steel, the noise in the strain data increased. Ultimate tensile strength, which only depends on the measured applied force and the cross-sectional area of the sample, was not affected. As a result, ultimate tensile strength could be calculated accurately for all three metals. Young's modulus and offset yield strength could not be calculated for steel. Offset yield calculations for aluminum and copper appear to be consistent with each other. The Young's modulus calculations for the baseline samples for aluminum and copper agree with published data for those materials, so the Young's modulus calculations for aluminum and copper are considered accurate.

#### 4.4.1 Copper

Offset yield stress and ultimate tensile strength showed a slight, but consistent, decrease for S8. Other PCMs did not affect these two material characteristics. Young's modulus was decreased for all PCMs except PEG 400 and triacetin. Results are given in Table 12 and Figures 14 to 16.

Table 12. Copper testing results.

PCM	Average Young's modulus	Standard deviation Young's modulus	Average offset yield stress	Standard deviation offset yield stress	Average ultimate tensile strength	Standard deviation ultimate tensile strength	Number of samples
	MPa	MPa	MPa	MPa	MPa	MPa	
Baseline	113,000	4,000	264	1	283	4	2
Distilled water	100,000	3,000	266	4	284	3	3
A6	80,000	10,000	265	4	281	4	3
RT3HC	97,000	2,000	265	1	281	1	3
OM06P	91,000	7,000	263	3	280	1	3
PureTemp 8	93,000	7,000	265	2	283	2	3
S8	90,000	10,000	255	3	272	1	3
E0	70,000	10,000	267	9	277	4	3
PEG 400	124,000	7,000	263	2	281.0	0.4	2
Triacetin	100,000	20,000	262.2	0.1	280	2	3

Abbreviations: PCM, phase-change material; PEG, polyethylene glycol.

Figure 14. Average offset yield stress for copper soaked in phase-change material.

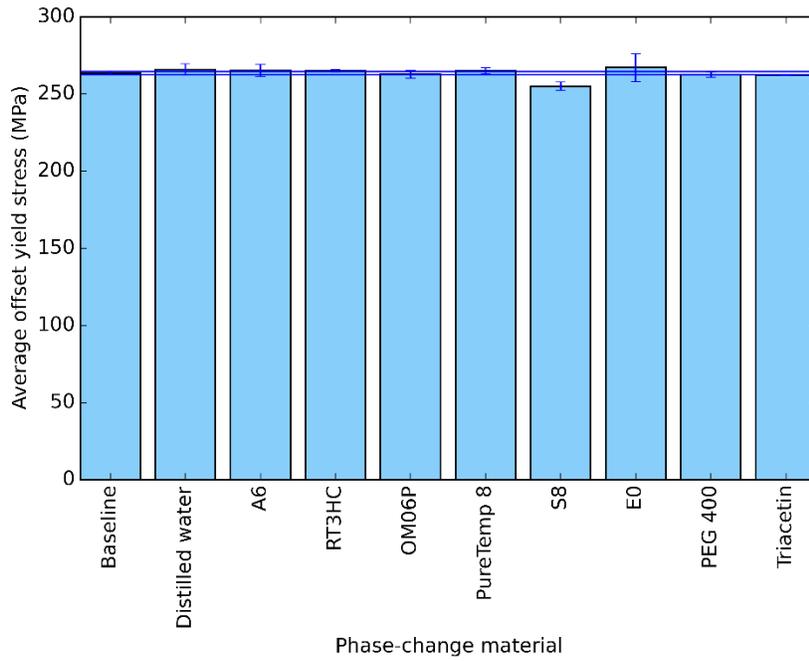


Figure 15. Average ultimate tensile strength for copper soaked in phase-change material.

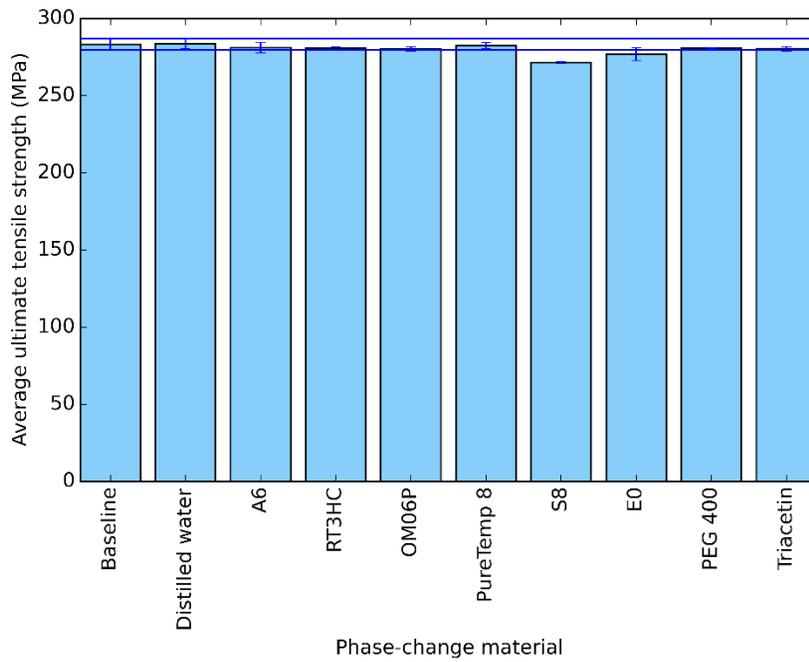
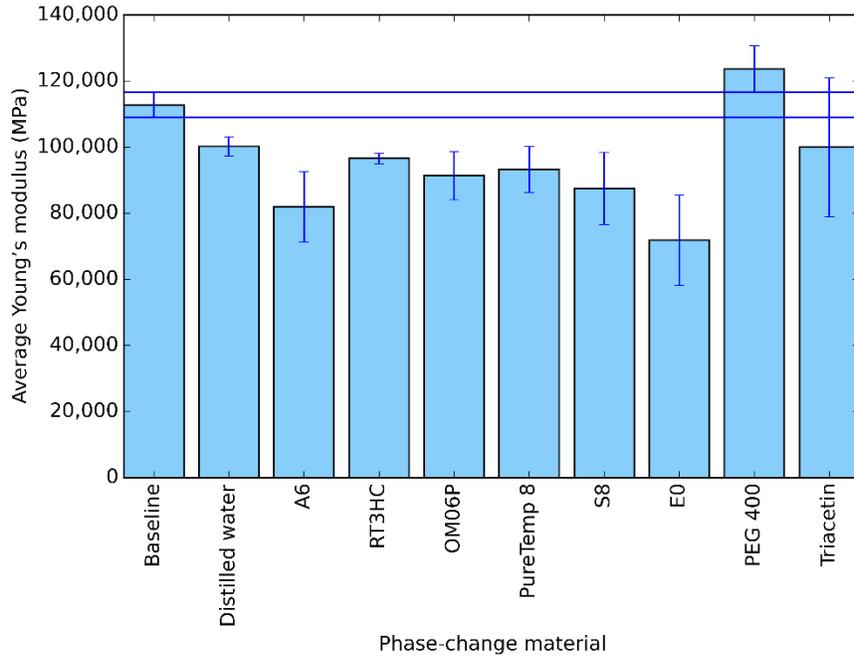


Figure 16. Average Young's modulus for copper soaked in phase-change material.



#### 4.4.2 Aluminum

Aluminum was relatively unaffected by all of the PCMs except for E0. The aluminum samples immersed in E0 exhibited decreased values for all mechanical properties tested. Visual observation of the E0 container found evidence of a reaction between the aluminum sample and the E0 including gas production and deposition of a material on the aluminum. A6 and PEG 400 also decreased Young's modulus for the aluminum samples. Results are given in Table 13 and Figures 17 to 19.

Table 13. Aluminum testing results.

PCM	Average Young's modulus	Standard deviation Young's modulus	Average offset yield stress	Standard deviation offset yield stress	Average ultimate tensile strength	Standard deviation ultimate tensile strength	Number of samples
	MPa	MPa	MPa	MPa	MPa	MPa	
Baseline	75,000	2,000	293	3	334	5	3
Distilled water	72,000	3,000	285	4	328	1	3
A6	66,000	5,000	288	2	329	3	2
RT3HC	69,000	5,000	280	10	320	20	3
OM06P	70,000	8,000	283.6	0.2	325	1	3
PureTemp 8	74,000	5,000	283.4	0.4	324	2	3
S8	72,000	3,000	285	4	328	1	3
E0	52,000	6,000	230	4	257	4	3
PEG 400	63,000	5,000	284	4	320	9	3
Triacetin	80,000	2,000	291	5	330	1	3

Abbreviations: PCM, phase-change material; PEG, polyethylene glycol.

Figure 17. Average offset yield stress for aluminum soaked in phase-change material.

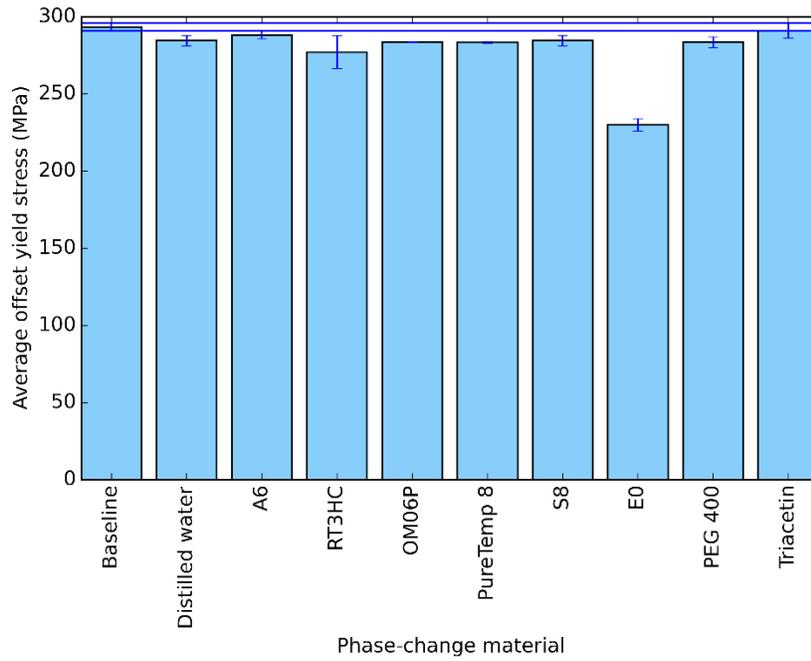


Figure 18. Average ultimate tensile strength for aluminum soaked in phase-change material.

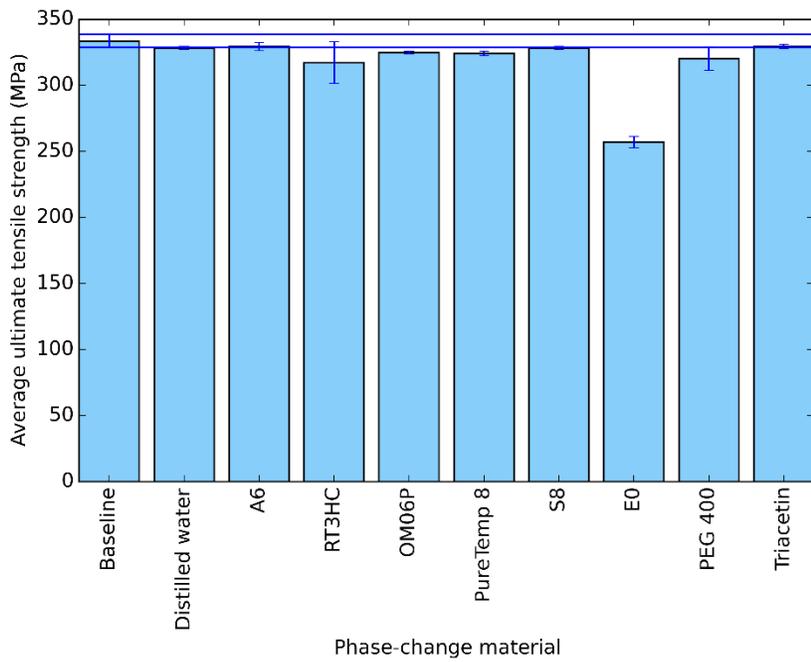
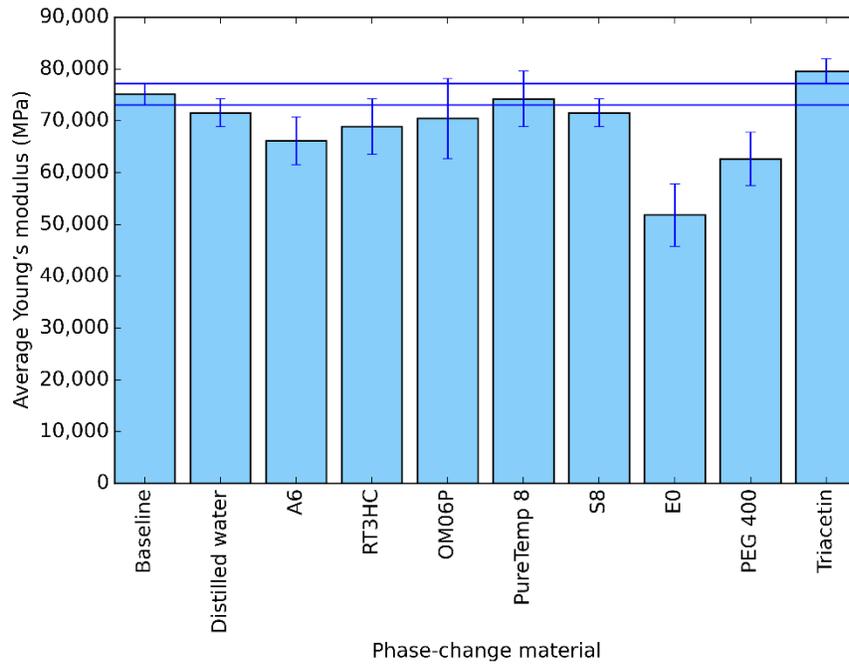


Figure 19. Average Young's modulus for aluminum soaked in phase-change material.



#### 4.4.3 Steel

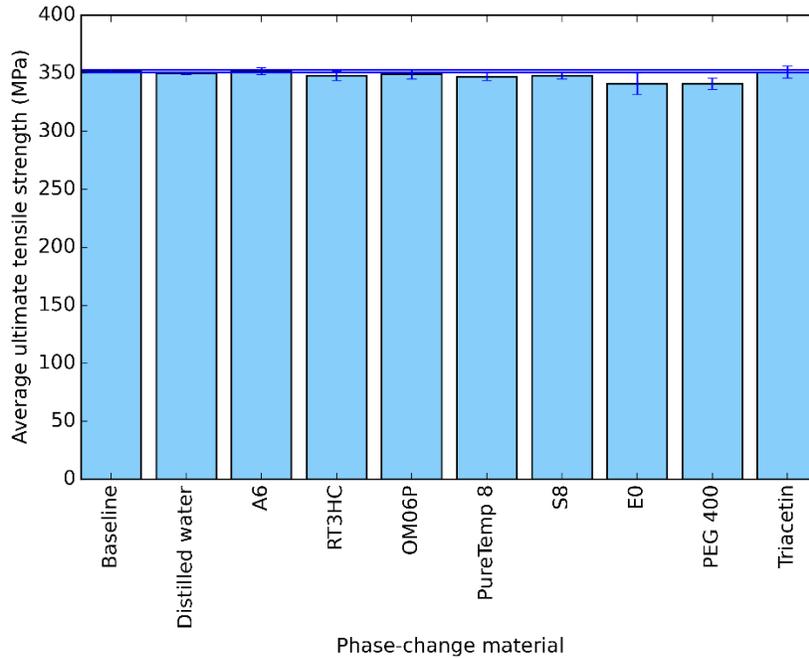
Only ultimate tensile strength data were of sufficient quality to be analyzed. Ultimate tensile strength showed very little effect for any of the PCMs. There may be a slight decrease for steel in PEG 400. Results are given in Table 14 and Figure 20.

Table 14. Steel testing results.

PCM	Average ultimate tensile strength	Standard deviation	Number of samples
	Mpa	Mpa	
PureTemp 8	352	1	3
Triacetin	350	1	3
OM06P	352	3	3
PEG 400	348	4	3
RT3HC	349	4	3
Distilled water	347	3	3
S8	348	3	3
E0	341	9	3
A6	341	5	3
Baseline	351	5	2

Abbreviation: PCM, phase-change material; PEG, polyethylene glycol.

Figure 20. Average ultimate tensile strength for steel soaked in phase-change material.



## 4.5 Foams

### 4.5.1 Polyisocyanurate foam

The compressive modulus and yield strength at 5% strain were increased by A6. Triacetin decreased the compressive modulus. Other PCMs had little to no significant effect. Results are given in Table 15 and Figures 21 to 22.

Table 15. Polyisocyanurate foam testing results.

PCM	Average compressive modulus	Standard deviation compressive modulus	Average 5% yield stress	Standard deviation 5% yield stress	Number of samples
	MPa	MPa	MPa	MPa	
Baseline	4.1	0.5	0.131	0.004	3
Distilled water	3.6	0.7	0.12	0.02	3
A6	5.3	0.3	0.162	0.004	3
RT3HC	4.4	0.5	0.138	0.007	3
OM06P	4.0	0.1	0.124	0.004	3
PureTemp 8	4.2	0.2	0.140	0.009	3
S8	3.7	0.2	0.133	0.007	3
E0	3.57	0.05	0.126	0.001	3
PEG 400	4.4	0.4	0.13	0.01	3
Triacetin	3.1	0.1	0.11	0.01	3

Abbreviations: PCM, phase-change material; PEG, polyethylene glycol.

Figure 21. Average compressive modulus for polyisocyanurate soaked in phase-change material.

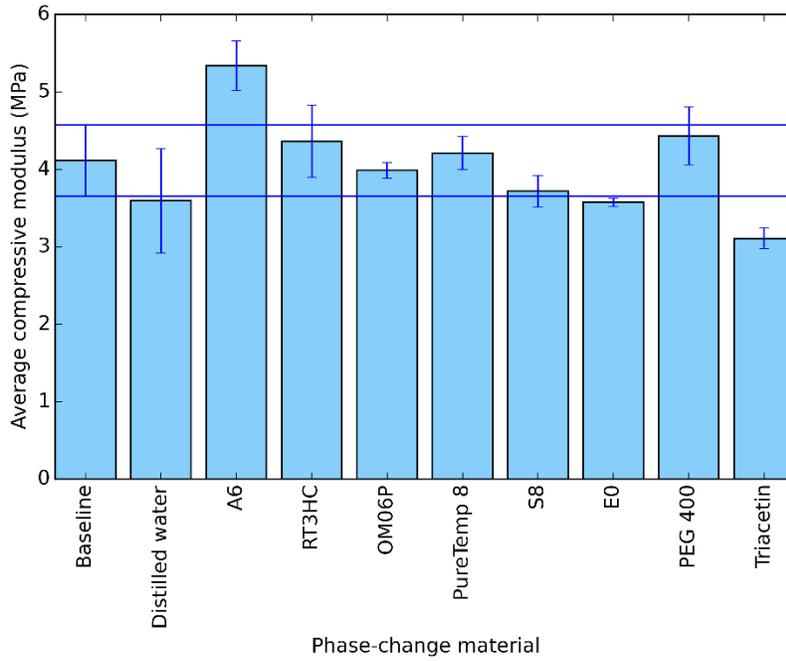
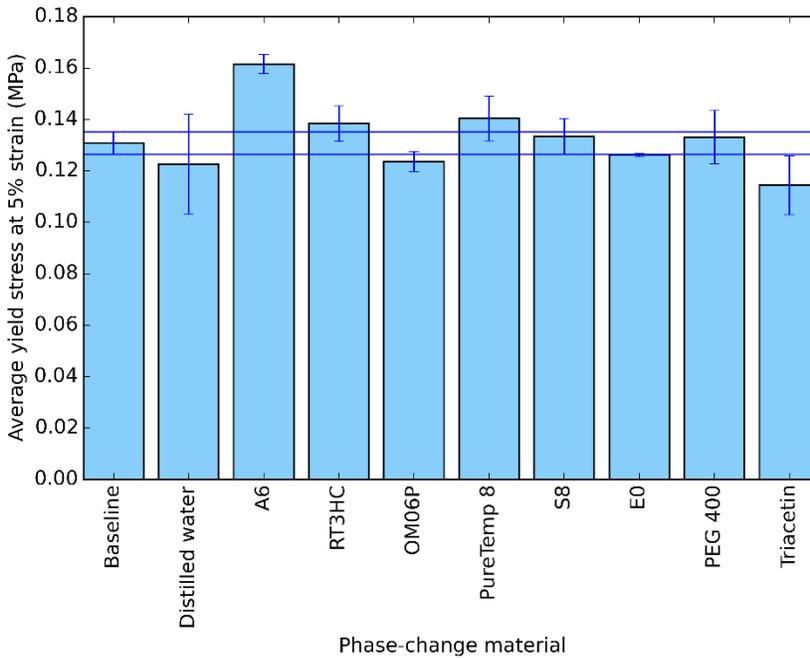


Figure 22. Average yield stress at 5% strain for polyisocyanurate soaked in phase-change material.



#### 4.5.2 Polyurethane foam

The compressive modulus was increased by distilled water, A6, and S8. The compressive modulus was strongly decreased by PEG 400. The yield stress was increased by A6, RT3HC, OM06P, S8, and E0. Note that apparent effects caused by A6 and RT3HC are difficult to verify since only one sample's data

could be used for each analysis and a standard deviation could not be calculated. Results are presented in Table 16 and Figures 23 to 24.

Table 16. Polyurethane foam testing results.

PCM	Average compressive modulus	Standard deviation Young's modulus	Average yield stress	Standard deviation yield stress	Number of samples
	MPa	MPa	MPa	MPa	
Baseline	3.8	0.3	0.13	0.01	4
Distilled water	4.7	0.3	0.125	0.002	3
A6	5.6	—	0.2	—	1
RT3HC	3.9	—	0.2	—	1
OM06P	4.3	0.4	0.150	0.005	3
PureTemp 8	4.3	0.2	0.137	0.005	3
S8	4.8	0.5	0.16	0.01	3
E0	5	1	0.154	0.005	2
PEG 400	2.7	0.1	0.118	0.003	3
Triacetin	4.2	0.2	0.138	0.003	3

Abbreviations: PCM, phase-change material; PEG, polyethylene glycol.

Figure 23. Average compressive modulus for polyurethane soaked in phase-change material.

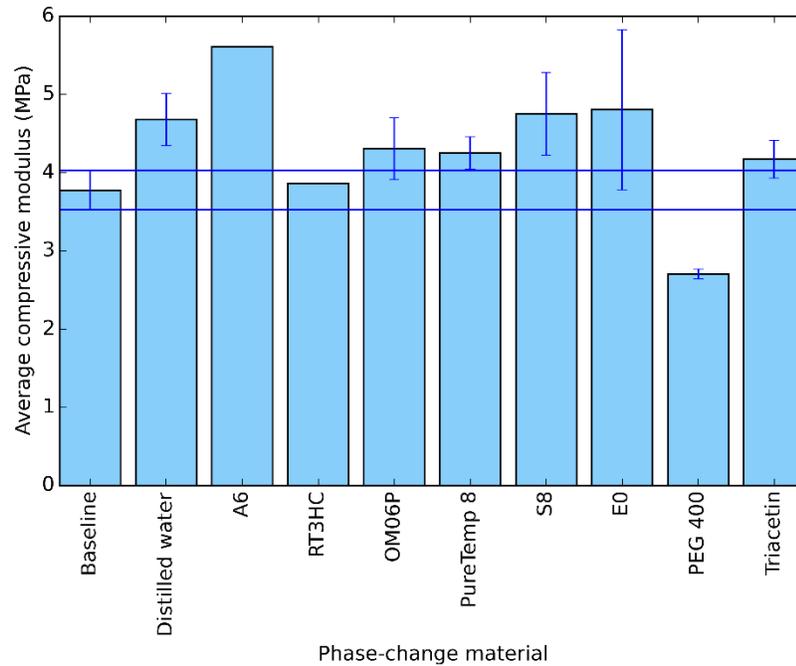
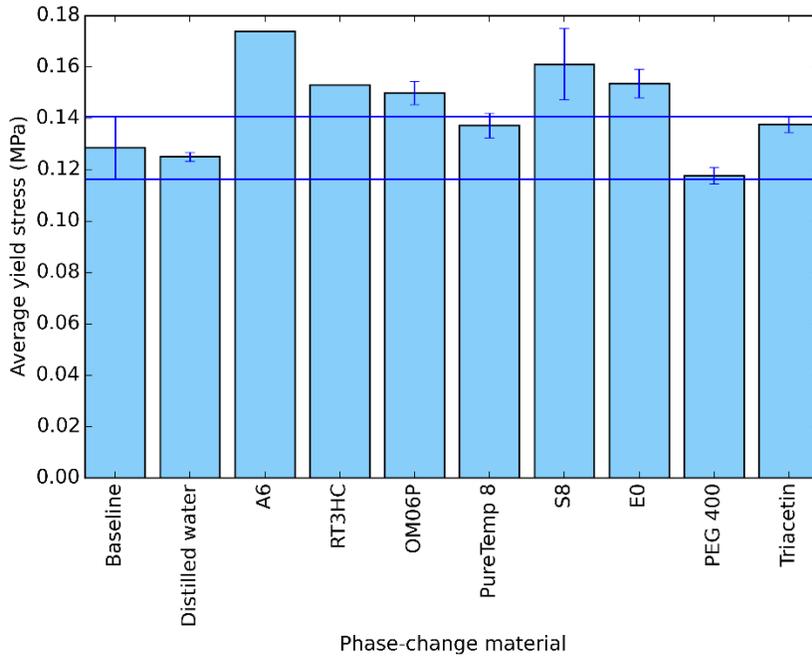


Figure 24. Average yield stress for polyurethane soaked in phase-change material.



## 4.6 Painted steel observations

The enamel on the painted steel samples was generally softer, according to the scratch test, after being immersed in PCMs. The two exceptions were for RT3HC, which seemed to make the paint harder, and A6, which did not cause any change. The PCM with the greatest effect was distilled water. Distilled water and S8 caused a rusty color change to the white paint; otherwise, the steel substrate was not visually affected. E0 caused bubbling in the paint surface. Samples were returned to their immersion containers immediately to continue with a three-month immersion test. Observations for the painted steel are summarized in Table 17. Figure 25 presents photographs of the baseline and soaked painted steel.

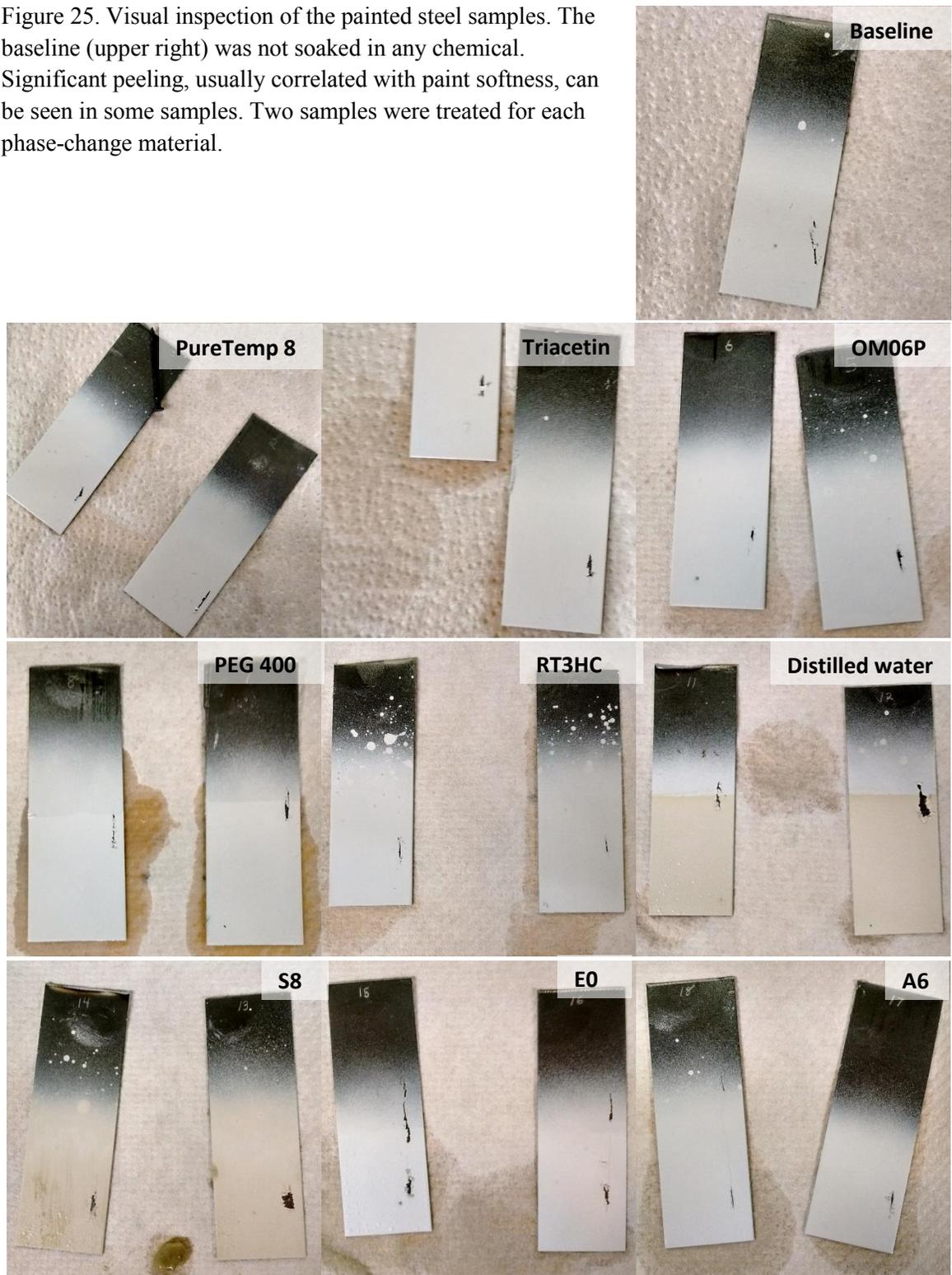
Table 17. Painted steel visual inspection notes.

Material	Sample number	PCM	Immersion duration (days)	Inspection notes	Scratch test (steel nail)
Painted steel	1	PureTemp 8	23	Paint peels slightly upon etch; no visible change to steel	Softer
Painted steel	2	PureTemp 8			Softer
Painted steel	3	Triacetin	23	Paint peels slightly upon etch; no visible change to steel	Softer
Painted steel	4	Triacetin			Softer
Painted steel	5	OM06P	23	Samples differed in paint hardness, but it was thicker on sample 5	No change
Painted steel	6	OM06P			Softer

Material	Sample number	PCM	Immersion duration (days)	Inspection notes	Scratch test (steel nail)
Painted steel	7	PEG 400	23	Interface above/below PCM was sharp; gummier below	Softer
Painted steel	8	PEG 400			Softer
Painted steel	9	RT3HC	23	Paint may have been harder than baseline, if any change	Harder
Painted steel	10	RT3HC			Harder
Painted steel	11	Distilled water	23	Rusty color below PCM line; paint very soft on entire sample	Much softer
Painted steel	12	Distilled water			Much softer
Painted steel	13	S8	20	Paint was flakier; rust coloration on paint	Softer
Painted steel	14	S8			Softer
Painted steel	15	E0	20	Paint peels slightly upon etch; some bubbling under paint	Softer
Painted steel	16	E0			Softer
Painted steel	17	A6	20	No change to paint or steel	No change
Painted steel	18	A6			No change

Abbreviations: PCM, phase-change material; PEG, polyethylene glycol.

Figure 25. Visual inspection of the painted steel samples. The baseline (upper right) was not soaked in any chemical. Significant peeling, usually correlated with paint softness, can be seen in some samples. Two samples were treated for each phase-change material.



## 4.7 Hookup wire observations

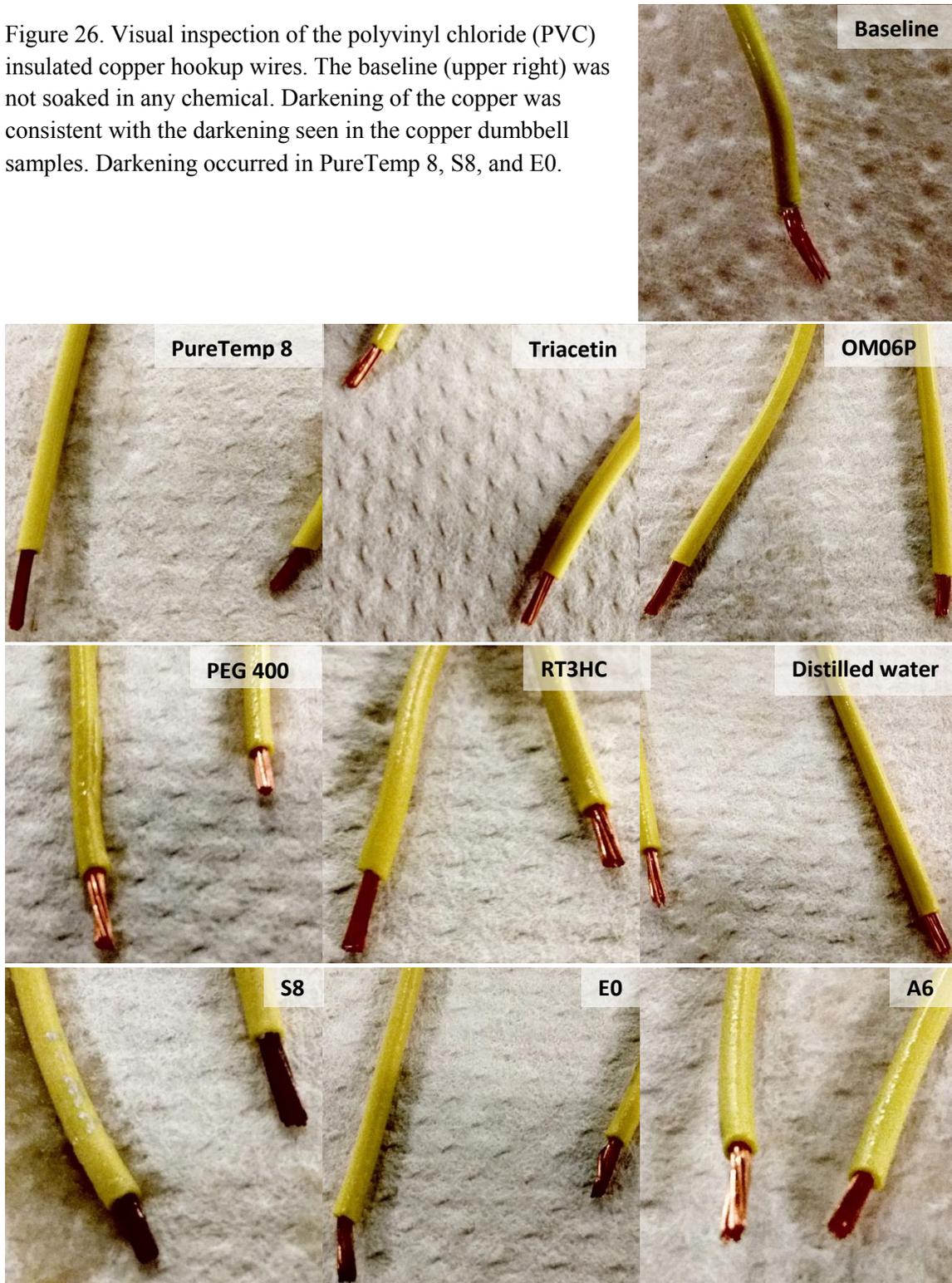
The hookup wires (copper covered in PVC sheaths) gave the same visual results as seen in the coloration on the separate PVC and copper dumbbell samples. The PVC had “no visible change” for all samples. The copper color was darker for immersions in PureTemp 8, S8, and E0. Samples were returned to their immersion containers immediately after inspection to continue with a three-month test. Observations for the hookup wires are summarized in Table 18. Figure 26 presents photographs of the baseline and soaked hookup wires.

Table 18. Hookup wire visual inspection notes.

Sample number	PCM	Immersion duration (days)	Inspection notes
1	PureTemp 8	23	Copper appears darker
2	PureTemp 8		
3	Triacetin	23	No visible change
4	Triacetin		
5	OM06P	23	No visible change
6	OM06P		
7	PEG 400	23	No visible change
8	PEG 400		
9	RT3HC	23	No visible change
10	RT3HC		
11	Distilled water	23	No visible change
12	Distilled water		
13	S8	20	Copper appears darker
14	S8		
15	E0	20	Copper appears darker
16	E0		
17	A6	20	No visible change
18	A6		

Abbreviations: PCM, phase-change material; PEG, polyethylene glycol.

Figure 26. Visual inspection of the polyvinyl chloride (PVC) insulated copper hookup wires. The baseline (upper right) was not soaked in any chemical. Darkening of the copper was consistent with the darkening seen in the copper dumbbell samples. Darkening occurred in PureTemp 8, S8, and E0.



## 5. Thermodynamic characterization of PCMs

To design CCE using PCM, the thermodynamic properties of the PCM must be measured. Important properties include the latent heat absorbed or released during the phase change, the melting-point temperature, and the temperature range over which the phase change occurs. A variety of methods can be used to measure these properties of a PCM including differential scanning calorimetry (DSC), temperature history (T-history),<sup>5</sup> Calvet calorimetry, and three-layer calorimetry. Each method has advantages and disadvantages, and all give comparable results for many PCMs.<sup>6</sup> The PCMs in this study were characterized using DSC due to the small sample size required for measurement and the rapidity of the method. One disadvantage of DSC is that, due to the small sample size (on the order of 10  $\mu\text{l}$ ), heterogeneous PCMs requiring a large sample size to obtain a representative sample cannot be measured. In this study, only the salt hydrate PCM S8 could not be tested using DSC due to the small sample size constraint. Triacetin also could not be characterized using DSC because upon cooling, liquid triacetin will supercool to  $-78^\circ\text{C}$  before it freezes to a glass state. Upon warming, solid triacetin has a nominal melting-point temperature of  $3^\circ\text{C}$ . The DSC used can only cool to  $-45^\circ\text{C}$ , so we could not cool triacetin enough to reach the solid state.

To measure the thermodynamic properties of the PCMs in this study, approximately 10  $\mu\text{l}$  of each PCM were scanned in a Setaram®  $\mu\text{DSC VII}$  micro-differential scanning calorimeter. Each sample was cycled through melting and freezing twice. DSC measures the difference in heat flow into the sample cell and an identical reference cell as a function of time as the temperature of the calorimeter is changed at a constant rate. The reference cell is either empty or contains a material with well-characterized thermodynamic properties. In this study, the reference cell was empty.

Once the heat flow over the temperature scan is measured, graphs of heat flow versus time and heat flow versus temperature are used to determine the melting-point temperature ( $T_{\text{melt}}$ ), the latent heat of the phase change, and the temperature range over which the phase change occurs. Example heat flow versus time and heat flow versus temperature curves for distilled water going through melting are given in Figures 27 and 28, respectively. For pure substances, the melting point is given as the onset temperature ( $T_{\text{onset}}$ ), which is found from the intersection of a line tangent to the inflection point on the melting curve and the integration baseline connecting the two flatter sections of the curve. For substances that are mixtures, the melting point is reported as the temperature at peak heat flow during the phase change ( $T_{\text{peak}}$ ). The only pure substance PCM characterized using DSC in this study was distilled water. All other substances were mixtures since even nominally pure substances such as triacetin, PEG 400, and the paraffin are actually mixtures of polymer or carbon chains of different lengths. The latent heat is the area between the baseline and the curve in the heat flow versus time graph (the yellow integration area). The temperature range of the phase change is the full width at half maximum (FWHM) of the heat flow versus temperature curve. Similar graphs can be constructed for the freezing curves.

Figure 27. Heat flow versus time for distilled water.

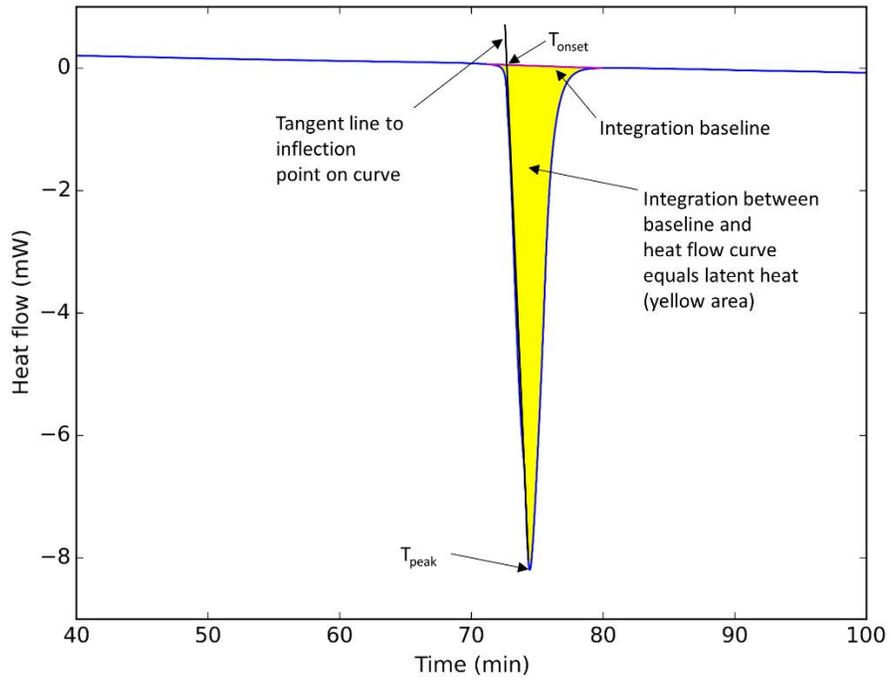
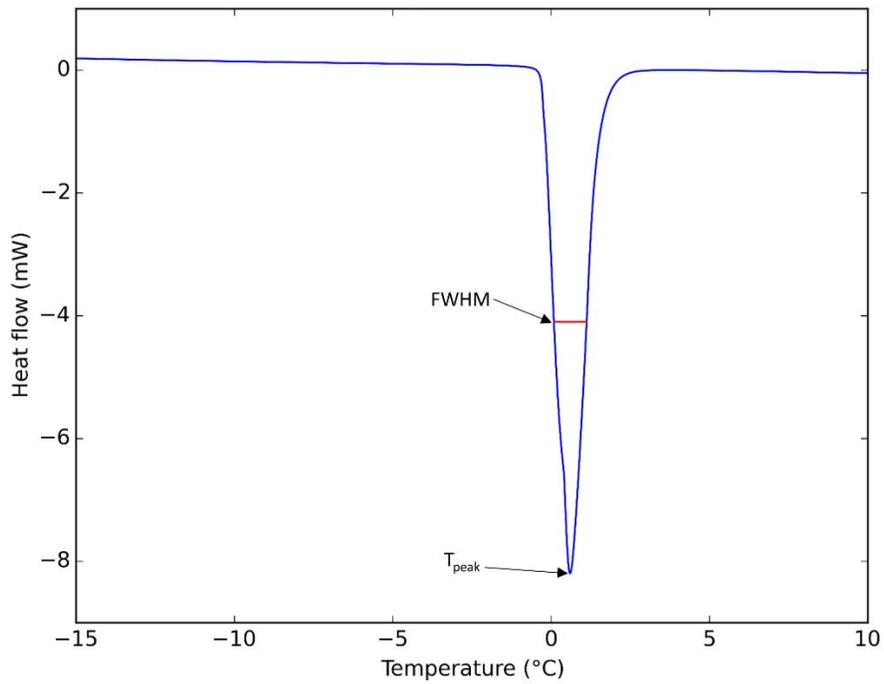


Figure 28. Heat flow versus temperature for distilled water.



Abbreviation: FWHM, full width at half maximum.

DSC measurements of the thermodynamic properties of the PCMs and the corresponding values from the manufacturers are given in Table 19 below. As a general note, only the heating data through the melting phase change gives accurate measurements of the thermodynamic properties because the cooling data through the freezing phase change is affected by supercooling of the PCM. Supercooling leads to very rapid freezing and resultant large errors in the thermodynamic properties measurements. The cooling data is included in the table to illustrate how differently PCMs behave during freezing versus melting.

Table 19. Thermodynamic properties of phase-change materials.

PCM	Heating			Cooling			Manufacturer data	
	T <sub>peak</sub> (°C)	Full width at half maximum (°C)	Latent heat (J/g)	T <sub>peak</sub> (°C)	Full width at half maximum (°C)	Latent heat (J/g)	T <sub>nominal</sub> (°C)	Latent heat (J/g)
Distilled water	-0.107 <sup>1</sup>	1.1	343	-22.3 <sup>1</sup>	0.22	283 <sup>2</sup>	0	334
	-0.265 <sup>1</sup>	1.0	342	-22.0 <sup>1</sup>	0.22	282 <sup>2</sup>		
PureTemp 8	8.3	1.57	176	6.0	1.12	172	8	178
	8.3	1.55	176	5.9	1.17	174		
Triacetin	Note 3						3	118
PEG 400	6.09	7.10	116	-0.242	3.09	103	4 to 8	151
	6.05	7.06	116	-0.108	3.14	103		
OM06P	5.9	0.83	237	2.8	0.41	241	6	260
	5.9	0.83	238	2.5	0.41	241		
RT3HC	2.7	1.28	145	2.8	1.85	152	3	172 <sup>5</sup>
	2.7	1.27	149	2.8	1.86	152		
S8	Note 4						8	150
A6	5.6	1.52	151	5.6	1.87	152	6	150
	5.6	1.52	150	5.6	1.90	151		
EO	0.64	1.47	344	-13.9	0.79	264 <sup>2</sup>	0	332
	0.66	1.48	344	-14.7	0.79	263 <sup>2</sup>		

Abbreviations: DSC, differential scanning calorimetry; PCM, phase-change material; PEG, polyethylene glycol.

Notes

1. For pure substances, the onset temperature (intersection of tangent line to heat-flow versus time curve inflection point and the integration baseline) is reported instead of the T<sub>peak</sub>.
2. Supercooling led to rapid freezing, resulting in oversaturation of heat flow sensor and clipping of heat flow signal. As a result, the latent heat of fusion was underestimated.
3. Pure triacetin will supercool to -78°C before solidifying to a glass even though its melting point is 3°C. The minimum temperature the DSC can reach is -45°C, so DSC data through the melting point could not be collected.
4. DSC could not be performed on S8 due to heterogeneity of S8 and the small sample size used by DSC.
5. Manufacturer reported heat storage capacity (latent heat and sensible heat absorbed/released between -5°C and 9°C) instead of latent heat. Value in table is heat capacity (200 J/g) minus the sensible heat absorbed over -5°C to 9°C at a specific heat of 2 J/(°C·g).

## 6. Discussion and conclusions

### 6.1 Mechanical testing

PP and HDPE were most affected by organic engineered PCMs (A6, RT3HC, OM06P, and PureTemp 8), which generally decreased the values of the measured mechanical properties of the samples. PCMs generally increased the Young's modulus of PVC, which corresponds to a stiffening of the plastic. The HDPE was the most affected by immersion in the PCMs, and the PVC was the least affected, with PP falling between HDPE and PVC in terms of impact on mechanical properties. In qualitative flexural testing by hand, HIPS was strongly affected by PureTemp 8, while other PCMs did not cause any obvious changes in flexural mechanical properties. HIPS soaked in PureTemp 8 became brittle and broke easily when flexed.

PCMs generally had minimal effects on the mechanical properties of the metals. The Young's modulus for copper decreased for all PCMs except PEG 400 and triacetin. The reason for the general decrease is unclear, since one would not expect strong interaction between organic or water-based PCMs and copper. The slight but consistent decrease in all properties for copper soaked in S8 is likely due to observed corrosion of copper samples in S8. The copper samples in S8 were discolored, and the S8 liquid gained a dark-blue color, suggesting the formation of a copper sulfate solution. These results suggest that a larger effect on the mechanical properties of copper immersed in S8 might be observed after longer immersion times.

Visual observation of the aluminum sample immersed in E0 indicated that a reaction between the aluminum and E0 likely took place, as evidenced by the formation of a deposit on the aluminum and the presence of gas bubbles. The apparent decrease in the values of measured mechanical properties for aluminum immersed in E0 is likely due to the increase in thickness of the samples from the deposits that formed on the samples. When the original thickness of the sample was used to calculate the ultimate tensile strength instead of the post-immersion thickness, much of the change in ultimate tensile strength disappeared. Given that a reaction did appear to take place between the E0 and the aluminum, some of the change in mechanical properties could be real and not just an effect of the change in sample thickness. It is also possible that the formation of the deposit on the aluminum affected the thermodynamic properties of the E0. Any change in thermodynamic properties could be measured by performing calorimetry on the E0 used to soak the aluminum.

No significant change was observed in the ultimate tensile strength of the steel samples despite discoloration of the samples immersed in E0, S8, and distilled water. Even the significant rusting observed on the sample immersed in the distilled water had no effect on ultimate tensile strength. This suggests that any corrosion of the steel by the PCMs only affected the surface of the steel samples and not the bulk properties of the samples. Testing after longer immersion times may show effects on steel's mechanical properties. There was a very slight decrease for the tensile strength of steel in PEG 400. PEG 400-water mixtures can be corrosive to steel,<sup>7</sup> and it is possible that the PEG 400 absorbed some atmospheric moisture that caused a small amount of corrosion.

The polyisocyanurate foam saw little effect from any of the PCMs except for A6 and triacetin. Absorption of PCM by polyisocyanurate is minimal, as shown by the small increase in mass of the samples. Longer immersion times may show increased effects.

The polyurethane foam was much more strongly affected by the PCMs. The engineered organic, S8, and E0 PCMs all increased the yield stress, and the compressive modulus was increased by distilled water, A6, and S8. The PEG 400 was strongly absorbed by the polyurethane, as shown by the much larger increase in post-immersion mass compared to the other PCMs. The absorption resulted in a large decrease in the compressive modulus. Given that the polyurethane is a closed-cell foam, the strong absorption of PEG 400 and the large decrease in compressive modulus suggest a substantial impact on the structure of the polyurethane foam.

## 6.2 Painted steel visual inspection

With the exception of RT3HC and A6, the PCMs caused softening of the enamel paint. The RT3HC-soaked enamel seemed slightly harder than the baseline, and perhaps RT3HC created an environment to promote curing of the enamel. However, the hardness of the enamel was difficult to differentiate with a qualitative test when there was only a slight change. The A6 also had little effect on the enamel.

Distilled water, and the water-containing S8 (a salt-hydrate PCM), caused expected rusting of the steel. It could be important to keep water (from a PCM or condensation) off of the CCE enamel surfaces. It is presently unclear whether the bubbling seen from E0 will degrade the paint more severely than other PCMs in a longer test, but it does indicate a problem in addition to the paint softening.

The 3-month painted steel inspection will give more information regarding any correlation between rust and paint integrity. Enamel damage may be accelerated by rust. If that is true, it still could be possible to use problematic PCMs with paint on non-steel surfaces. For example, distilled water might be acceptable to use with painted aluminum or copper.

## 6.3 Hookup wire visual inspection

Minimal changes were observed in the hookup wire. The PVC insulation showed no visible change, and the copper wire exhibited some darkening after soaking in PureTemp 8, S8, and E0. The copper was not necessarily mechanically weaker or less electrically conductive due to the color change. The darkness could be depositions of PCM chemicals, rather than the result of corrosion. Given the decrease in mass and mechanical properties of the S8 soaked dumbbell copper sample, the copper wire likely corroded to some extent. A future test with “live” (voltage applied) wires during the duration of PCM immersion might have important/different results.

## 6.4 Differential scanning calorimetry measurements

$T_{\text{peak}}$  data determined during the heating phase measurements agreed well with manufacturer and published data. Supercooling depressed the  $T_{\text{peak}}$  measurements for all PCMs except for the A6 and RT3HC paraffin PCMs. As discussed above, triacetin supercools to  $-78^{\circ}\text{C}$  before solidifying, rendering pure triacetin unsuitable as a PCM for CCE. Additives such as freezing nuclei or other chemical additions could ameliorate the supercooling problem with triacetin. The FWHM measurements for the melting temperature range were generally less than  $2^{\circ}\text{C}$  except for PEG 400. The wide melting temperature range for PEG 400 may limit its usefulness for CCE. Latent heats were very close to the manufacturer and published data except for PEG 400, OM06P, and RT3HC, which deviated from specification by 23%, 9%, and 15%, respectively. Manufacturer thermodynamic properties data for the studied PCMs is generally accurate.

## 7. Additional resources

Alfa Aesar website. Available at: <http://www.alfa.com>. Accessed May 2, 2016.

Alfa Aesar melting point data now openly available page. Useful chemistry site. Available at: <http://usefulchem.blogspot.com/2011/02/alfa-aesar-melting-point-data-now.html>. Accessed May 2, 2016.

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## 8. References

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