Use of DSC and DMA to Study Rubber Crystallization as a Possible Cause for a Tear in a Neoprene Glove Used in a Space Shuttle Pressurized Astronaut Suit

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ABSTRACT

The Advanced Crew Escape Suit (ACES) is a pressurized suit normally worn by astronauts during launch and landing phases of Space Shuttle operations. In 2008, a large tear (0.5-1 in. long, between the pinky and ring finger) in the ACES left-hand glove made of neoprene latex rubber was found during training for Shuttle flight STS-124. An investigation to help determine the cause(s) of the glove tear was headed by the NASA Johnson Space Center (JSC) in Houston, Texas. Efforts at JSC to reproduce the actual glove tear pattern by cutting/tearing or rupturing were unsuccessful. Chemical and material property data from JSC such as GC-MS, FTIR, DSC and TGA mostly showed little differences between samples from the torn and control gloves. One possible cause for the glove tear could be a wedding ring/band worn by a male astronaut. Even with a smooth edge, such a ring could scratch the material and initiate the tear observed in the left-hand glove. A decision was later made by JSC to not allow the wearing of such a ring during training or actual flight.

Another possible cause for the ACES glove tear is crystallinity induced by strain in the neoprene rubber over a long period of time and use. Neoprene is one several elastomeric materials known to be susceptible to crystallization, and such a process is accelerated with exposure of the material to cold temperatures plus strain. When the temperature is lowered below room temperature, researchers have shown that neoprene crystallization may be maintained at temperatures as high as 45-50°F, with a maximum crystallization rate near 20-25°F (1). A convenient conditioning temperature for inducing neoprene crystallization is a typical freezer that is held near 0°F. For work at the NASA Marshall Space Flight Center (MSFC), samples were cut from several areas/locations (pinky/ring finger crotch, index finger and palm) on each of two pairs of unstrained ACES gloves for DSC and DMA thermal analysis testing. The samples were conditioned in a freezer for various times up to about 14 days. Some rectangular conditioned samples were unstrained, while most were subjected to strains up to 250% with the aid of two slotted aluminum blocks and two aluminum clamps per sample. Trends were observed to correlate DSC data (heat of fusion) and DMA data (linear CTE and stress for iso-strain testing) with: (a) sample location on each glove; and (b) level of strain during conditioning. Control samples cut “as is” from each glove location were also tested by DSC and DMA.

INTRODUCTION

Historical Background Involving NASA/MSFC

MSFC became involved in this investigation largely because of a previous incident with a torn neoprene latex rubber glove. Non-pressurized neoprene gloves are used in science glove box experiments inside the International Space Station (ISS). In 1999, it was found that some of these gloves stored in a metal cabinet drawer for four years showed significant signs of corrosion in the drawer. Analysis of the corrosion products by energy dispersive x-ray spectroscopy (EDS) revealed the presence of free chlorine. It was discovered that the ISS neoprene (polychloroprene) latex gloves released small quantities of hydrochloric acid (HCl) with material aging, a process enhanced by the presence of air and light. The HCl not only degraded the neoprene glove material properties (much
lower tensile strength and elongation, much higher modulus), but also induced crystallinity in the material. Values of percent crystallinity up to 9.70 were determined by DSC for HCl-affected material, compared to only 0.38 for unaffected material (2).

**Literature on Crystallization of Neoprene (Polychloroprene) and Other Elastomers**

*Crystallization Rate of Neoprene*

Dilatometric studies were made to determine crystallization and glass transition data for several unvulcanized and vulcanized polychloroprenes and polychloroprene/styrene-butadiene blends. For five temperatures ranging from -14° to 5°C, the volume decrease due to crystallization was measured as a function of time. The maximum rate of crystallization of both unvulcanized polychloroprene and the blends occurred near -5°C. For a crystallization-resistant polychloroprene (Neoprene WRT), the following approximate times were required to reach an equilibrium degree of crystallization: 6.5 days at -5°C, 11 days at -14.3°C (lowest temperature measured). Melting temperatures of crystallized samples of several neoprenes ranged from 36-43°C by an optical method (1). In another paper, a dilatometric application of the Avrami equation was used to study the crystallization kinetics of several grades of polychloroprene (3). In this study, a curve of crystallization half-period (t1/2) vs. crystallization temperature for Neoprene W passed through a minimum near -5°C, indicating the maximum crystallization rate. For crystallization-resistant Neoprene WRT, t1/2 was determined to be 175 hr (7.3 days), presumably measured at 0°C.

*Tear Strength of Neoprene*

Tear strength of several elastomers was measured over a range of rates, temperatures and crosslinking. For neoprene WRT, tear strength was measured for several temperatures from 45-150°C. Even when the amount of curing agent was doubled, tear strength remained high even at a high temperature (150°C), which could be attributed to strain-induced crystallization along the rough fracture surface upon tearing. The effect of the rough fracture/tear surface was nullified as follows: Two layers of partially crosslinked neoprene were interlinked in contact for a time to achieve the final degree of crosslinking, creating a type of peel specimen. Even with this special specimen to create a smooth fracture surface, data for detachment energy vs. reduced rate of crack propagation did not fit on a continuous master curve for temperatures ranging from 0-150°C. The discontinuity in the master curve was attributed to strain-induced crystallization at the high rates and low temperatures (4).

*Mechanical Property Data to Study the Crystallization of Polyisoprene (Natural Rubber)*

In a 1983 aerospace study, Morton Thiokol found that torques on the natural rubber nozzle flex bearing of the Peacekeeper static test motor were higher than expected for the motor conditioned and tested at 45°F. For unstrained samples of natural rubber conditioned at 25°F (-4°C), there was a sharp increase in Shore A hardness after 7 days. For strains of 50 and 150%, the rate of increase in hardness increased with increasing strain for 2-7 days of conditioning (5).

As part of the test motor study, the shear modulus of natural rubber was also determined as a function of time at temperature, strain rate, cure temperature and test temperature (6). This data showed that for several combinations of storage temperature/test temperature, the greatest increase in shear modulus (at 395% strain) was for the 40°F storage/40°F test conditions. For these conditions, the rate of the shear modulus increase slowed between 6 and 13 days. Additional shear modulus data at several temperatures (50-65°F) was compared to previous data at 40 and 45°F. From this comparison testing, it was determined that strain-induced crystallization in the natural rubber was formed and retained at temperatures below 50°F. For several cure temperatures of 280-320°F, shear modulus data also showed that crystallization rate decreased as cure temperature (and crosslink density) increased.
EXPERIMENTAL

Materials and Fixtures Used

Figure 1 shows one of the ACES gloves used for the testing described in this work. The glove material near the palm area and below is Gore-Tex—a fabric containing expanded PTFE. All of the glove material above the Gore-Tex is neoprene. The Gore-Tex was selected to improve glove comfort and breathability. To further improve comfort, the neoprene glove section is “flocked” on the inside with a very thin layer of cotton material. Figure 1 also shows the three primary areas that samples were cut from in the neoprene section for DSC and DMA testing: palm, index finger and pinky/ring finger crotch.

Figure 2 shows a sketch of a slotted aluminum block fixture used for conditioning rectangular neoprene samples cut from an ACES glove. The block is ~3.5 in. x 2.75 in., and contains a series of 0.25 in. wide recessed slots. Each rectangular sample was held at each end with an aluminum clamp tightened with Allen screws. The effective sample length held between the two clamps is 0.625 in. Each rectangular sample was cut from the pinky/ring finger crotch to allow the sample to span that crease. Two aluminum blocks were machined—with accompanying clamps—to allow for freezer conditioning of six glove samples at a time. Each fixture allows for samples to be stretched and held at 100, 150 or 250% strain during conditioning. At least two of the set of six rectangular samples were unstrained (but clamped) during conditioning.

Two pairs of ACES gloves were sent from JSC to MSFC for DSC and DMA testing. The gloves were not previously strained, and were within the recommended 78-month (6.5-year) usage life. Both DSC and DMA samples were cut to include the thin layer of cotton flocking on the bottom. Three types/groups of samples were tested in this work, with average weights and dimensions summarized in Table 1. For DSC samples only, each disk sample was weighed, crimped in a standard aluminum pan and lightly wrapped in aluminum foil. The wrapped samples were placed in a Ziploc bag before freezer conditioning, to minimize the effects of condensation from moisture. For DMA samples only, each rectangular sample was also wrapped and stored like the DSC samples. For DSC/DMA samples that were cut from the same rectangular specimen, these specimens were mounted in one of two aluminum blocks shown in Figure 2. Each block plus mounted specimens was also wrapped and stored as described previously, although it was more difficult to keep condensation out during freezer storage.

Instruments Used

The TA Instruments 2920 Differential Scanning Calorimeter (DSC) was used in the standard heating mode. Each disk-shaped neoprene glove sample (“as is” or freezer-conditioned) was equilibrated and held at -20°C for 5 min., followed by heating at 10°C/min. to 250°C. Each DSC sample was crimped in a standard aluminum pan, and a steady flow of argon gas was used to purge each sample during heating.

The TA Instruments 2980 Dynamic Mechanical Analyzer (DMA) was also used in this work. Each rectangular sample was equilibrated and held at -22°C for 5 min., followed by heating at 3°C/min. to 6.5°C. Missile grade air at 60-65 psi pressure was used to operate the drive shaft as well as purge each sample during heating. Each rectangular sample was vertically clamped at each end of the DMA film tension clamp with ~4-5 in. lbs. of torque. Each clamped sample was given some pre-test tension, followed by testing at 22.5% strain in the iso-strain testing mode. For each sample, test data was reported as: (a) linear coefficient of thermal expansion (CTE) in μm/m°C from -18 to 0°C; (b) slope of stress (psi/°C) from -18 to 0°C; and (c) stress at -18°C.
RESULTS AND DISCUSSION

DSC Data

Figure 3 is a DSC plot of heat flow vs. temperature for "as is" and strained glove samples cut from the palm area of a right-handed glove (RH2). The strained sample was stretched 250% and freezer-conditioned for 4.9 days. The strained sample shows a clear increase in crystallinity compared to the "as is" sample.

Figure 4 is a plot of heat of fusion vs. time in freezer (days) for samples cut from the pinky/ring, index and palm areas of a left-handed glove (designated LH1). Each of these samples was weighed and crimped in a standard aluminum DSC pan before being conditioned in the freezer. Since each sample was sealed in a DSC pan during conditioning, no sample was subjected to strain. Figure 4 indicates that the rate of crystallization goes through a maximum at ~6-7 days in conditioning. Samples from the index finger appeared to crystallize less than samples from the pinky/ring crotch or palm. Samples in Figure 4 ranged in estimated crystallinity from 0.88 to 1.74%. Percent crystallinity by DSC was estimated based on the heat of fusion of 95 Joules/gram for the crystalline phase of unvulcanized polychloroprene (7, 8).

Figure 5 is a plot of heat of fusion vs. time in freezer (days) for samples cut from the three previously designated areas of a right-handed glove (designated RH2). Each of these samples was cut as a rectangular strip, clamped at each end and mounted in the slotted aluminum block in Figure 2. For each set of samples conditioned from a specific glove area, most samples were strained 250%, while a few samples were unstrained. After a DSC sample was cut from each rectangular strip, the remainder of the same strip was used as a DMA sample. Perhaps the most significant data from Figure 5 is that heat of fusion (and % crystallinity) is considerably higher for the pinky/ring crotch samples (0% strain) than for samples from the same area at 250% strain. At 0% strain, heat of fusion (and % crystallinity) was considerably higher for pinky/ring samples than for samples from the index finger or palm. At 250% strain, heat of fusion for samples from the palm area was considerably higher than at 0% strain. In fact, palm samples showed the highest crystallinity in Figure 5 (4.33%) compared to as low as 1.07% for "as is" material. Inflections in most of the curves in Figure 5 again indicate that the maximum crystallization rate occurs at ~6-7 days of freezer conditioning. One study yielded percent crystallinity as low as 5 percent by DSC for Neoprene W and WHV (9).

The temperature onset of heat of fusion ranged from ~41-48°C as a function of freezer conditioning time (days) and % strain, for all three glove areas tested. This temperature onset averaged 46.5 ± 0.82°C for "as is" material (no freezer conditioning, no strain), taken from all three glove areas of two different gloves. These onset temperatures are at the high end of the range of optical melting temperatures reported in the literature for grades of neoprene—36-43°C and 39-44°C (1, 9).

DMA Data

Figure 6 is a DMA plot of dimension change and stress vs. temperature for a strained glove sample cut from the index finger of a right-handed glove (RH2). The strained sample was stretched 250% and freezer-conditioned for 6.1 days. Figure 6 shows the calculations of: (a) linear CTE from -18 to 0°C (slightly negative for this sample); (b) slope of stress from -18 to 0°C; and (c) stress at -18°C.

Figure 7 is a plot of linear coefficient of thermal expansion (CTE) vs. time in freezer (days) for samples cut from the three previously designated areas of a right-handed glove (RH2). Of the three glove areas tested, the palm shows the steepest increase in CTE for about 3-6 days in the freezer. With the exception of one data point for the index finger at 250% strain, the index and pinky/ring areas show a slightly negative CTE (shrinkage) for about 3-14 days in the freezer.

Figure 8 is a plot of stress, psi (at -18°C) vs. time in freezer (days) for samples cut from the pinky/ring finger crotch area for two different right-handed gloves (RH1 and RH2). Samples were conditioned in the freezer at 0, 150 and 250% strain. The stress was reduced by as much as ~25-30% in
going from unstrained to strained samples. The stress was reduced by ~25% in going from 150% strain to 250% strain. Stress is proportional to modulus—an indicator of material toughness/stiffness. Therefore, this data indicates that the glove material (at least in the pinky/ring crotch area) loses toughness when strain is applied, as well as when the level of strain is increased. This reduction in material toughness may indicate a greater tendency for the glove to tear in an area such as the pinky/ring finger crotch.

Figure 9 is a plot of stress (at -18°C) vs. time in freezer (days) for samples cut from the three previously designated areas of one right-handed glove (RH2). Samples were conditioned in the freezer at 0 and 250%. Unstrained index finger samples showed a much higher stress (for ~7-15 days in the freezer) than any other samples tested. Samples from the pinky/ring finger crotch and index finger with 250% strain showed the lowest values of stress for ~4-15 days in the freezer—data curves for both sample areas were very similar. However, for the palm area with 250% strain, the stress was almost 25% higher than for the pinky/ring and index finger areas for ~7-15 days in the freezer. This data shows that for 250% strain, glove material in the pinky/ring and index finger areas would be more likely to tear than in the palm area.

Table 1 Types of Tests Performed on Neoprene Glove Samples, with Average Sample Weights and Dimensions.

<table>
<thead>
<tr>
<th>Sample Type Tested</th>
<th>Average DSC Sample Weight, mg</th>
<th>Average DMA Sample Dimensions, mm</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Length</td>
</tr>
<tr>
<td>DSC only</td>
<td>10.1</td>
<td>-----</td>
</tr>
<tr>
<td>DMA only</td>
<td>-----</td>
<td>11.43</td>
</tr>
<tr>
<td>DSC + DMA</td>
<td>6.1</td>
<td>8.62</td>
</tr>
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</table>

Figure 1 Typical ACES glove showing samples cut from the index finger, pinky/ring finger crotch, and palm for the neoprene upper area. The lower glove area below the palm is made of Gore-Tex fabric.
Figure 2 Aluminum block machined with recessed slots for straining neoprene glove samples. Aluminum clamps (upper right) allow each sample to be stretched 100, 150 or 250% strain on sample.
Figure 3 DSC plot of heat flow vs. temperature for two neoprene samples cut from a right-handed glove (RH2): "as is" sample, and a sample strained at 250% for 4.9 days in a freezer.

Figure 4 DSC heat of fusion vs. time in freezer (days) for samples cut from three areas (pinky/ring finger crotch, index finger and palm) of a left-handed glove (LH1). All samples were unstrained.
**Figure 5** DSC heat of fusion vs. time in freezer (days) for samples cut from three areas (pinky/ring finger crotch, index finger and palm) of a right-handed glove (RH2). Samples were strained 0 and 250%.

**Figure 6** DMA dimension change and stress vs. temperature for a rectangular neoprene sample tested in the iso-strain mode. Calculations show: linear CTE (-18 to 0°C), slope of stress (-18 to 0°C) and stress at -18°C.
Figure 7 Linear coefficient of thermal expansion (CTE) vs. time in freezer (days) for rectangular DMA samples cut from three areas (pinky/ring finger crotch, index finger and palm) of a right-handed glove (RH2). Samples were strained at 0 and 250% in the freezer, then 22.5% in the DMA.

Figure 8 Stress (at -18°C) vs. time in freezer (days) for rectangular DMA samples cut from the pinky/ring finger crotch of two different gloves (RH1 and RH2). Samples were strained at 0-150% (glove RH1) and 0-250% (glove RH2) in the freezer, then 22.5% in the DMA.
Figure 9 Stress (at -18°C) vs. time in freezer (days) for rectangular DMA samples cut from three areas (pinky/ring finger crotch, index finger and palm) of a right-handed glove (RH2). Samples were strained at 0 and 250% in the freezer, followed by 22.5% strain in the DMA.

REFERENCES
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Introduction

• 0.5-1 in. long tear in the pinky/ring finger crotch (left hand) of a pressurized neoprene latex rubber glove used with Advanced Crew Escape Suit (ACES) during Space Shuttle flight training in 2008.

• Actual glove tear pattern could not be reproduced by cutting/tearing or rupturing at Johnson Space Center (JSC) in Houston, Texas.
  - Chemical and material property data from JSC such as GC-MS, FTIR, DSC and TGA mostly showed minimal differences between torn and control gloves.

• Possible causes for the ACES glove tear:
  - Wedding ring/band worn by male astronaut on left hand could scratch glove material and initiate a tear.

  - Strain-induced crystallinity in glove material over a long period of time. The tendency to crystallize could be a function of certain areas on the glove.
Use of DSC and DMA to Study Rubber Crystallization in a Neoprene Glove

- **Historical Background at NASA/MSFC**
  - In 1999, neoprene latex rubber gloves used in a science glove box experiment on International Space Station released small quantities of HCl during storage.
  - Resulted in much lower tensile strength/elongation, much higher modulus.
  - Induced unusually high crystallinity (9.70% vs. 0.38% for unaffected material).

The small heat of fusion (onset ≈33°C) is used to estimate DSC % crystallinity.

Presented at NATAS 2001 for work done at NASA/MSFC.
Use of DSC and DMA to Study Rubber Crystallization in a Neoprene Glove

• **Literature on Crystallization of Neoprene (Polychloroprene) and Other Elastomers**
  - For several types of polychloroprenes, the maximum rate of crystallization occurred near -5°C, based on a range of five temperatures from -14 to 5°C. For a crystallization-resistant polychloroprene (Neoprene WRT), the half-time of crystallization was about 5.7 days at -14°C [Kell, et al., *J. Appl. Polym. Sci.*, 2(4), 8 (1959)].
  - Tear strength data for Neoprene WRT was influenced by strain-induced crystallization. The author implied that such crystallization could contribute to roughness of the fracture surface specimen upon tearing [Bhowmick, *J. Matl. Sci.*, 21, 3927 (1986)].
  - In a 1983 study, Morton Thiokol found that torques on the natural rubber nozzle flex bearing of a static test motor were higher than expected for the motor conditioned and tested at 45°F, which was likely influenced by strain-induced crystallization.
    - *There was a sharp increase in Shore A hardness of unstrained natural rubber after 7 days at 25°F (~-4°C), with a greater rate of increase at strains of 50 and 150%.*
    - *The rate of shear modulus (at 395% strain) increase slowed between 6-13 days for a storage temperature/test temperature of 40°F/40°F.*
    - *Strain-induced crystallization was formed and retained at temperatures below 50°F.*
Use of DSC and DMA to Study Rubber Crystallization in a Neoprene Glove

- ACES glove has Gore-Tex fabric below palm area, neoprene latex rubber above. A thin layer of flocked cotton material is on the inside of the neoprene section for comfort.

- The figure shows that samples were cut from the palm, index finger and pinky/ring finger crotch for DSC and DMA testing.

  - Two pairs of unstrained ACES gloves were sent from JSC (Houston, TX) to MSFC for such testing. The gloves were within the recommended 78-mo. (6.5-yr.) usage life.
Use of DSC and DMA to Study Rubber Crystallization in a Neoprene Glove

Clamped rectangular glove samples were unstrained or strained 150-250% during conditioning in a freezer at ≈ 0°F (-18°C).

Two aluminum fixtures and clamps were machined to allow for conditioning of six samples at a time.
Use of DSC and DMA to Study Rubber Crystallization in a Neoprene Glove

DSC data for neoprene samples cut from palm area of a right-handed glove:

- “as is” material.
- Freezer conditioned at 250% strain for 4.9 days.

All DSC samples were equilibrated at -20°C for 5 min., and heated at 10°C/min. to 250°C with a TA Instruments 2920 DSC.
Neoprene samples were cut from three areas of a left-handed glove (unstrained: “as is” and freezer-conditioned).

- Each sample was weighed and crimped in a standard Al DSC pan before conditioning.

- Approximate maximum in crystallization rate (6-7 days).

- Estimated % crystallinity range was 0.88-1.74% (heat of fusion of crystalline phase of polychloroprene = 95 J/g).
Neoprene samples were cut from three areas of a right-handed glove:
- “as is” material.
- Freezer-conditioned (0, 250% strain).
- DSC, DMA samples cut from same rectangular specimen.
- Applied strain appears to lower crystallinity in pinky/ring crotch.
- Palm, index finger at 250% strain had similar high crystallinity as pinky/ring crotch at 0% strain.
- Estimated % crystallinity range was 1.07-4.33%.
Use of DSC and DMA to Study Rubber Crystallization in a Neoprene Glove

DMA data for neoprene samples cut from index finger of a right-handed glove:

- Freezer-conditioned at 250% for 6.1 days.

All DMA samples were equilibrated at -22°C for 5 min., and heated at 3°C/min. to 6.5°C in air with a TA Instruments 2980 DMA.

- DMA film tension clamp
- 22.5% strain (isostrain)
- Linear CTE (-18 to 0°C)
- Stress slope (-18 to 0°C)
- Stress at -18°C
Use of DSC and DMA to Study Rubber Crystallization in a Neoprene Glove

Neoprene samples were cut from three areas of a right-handed glove:
- “as is” material.
- Freezer-conditioned (0, 250% strain).
- DSC, DMA samples cut from the same rectangular specimen.
- Palm samples showed the steepest increase in CTE for ≈ 3-6 days.
- Samples from most glove areas showed slightly negative linear CTE (shrinkage) for -18 to 0°C.
Neoprene samples were cut from three areas of a right-handed glove:
- “as is” material.
- Freezer-conditioned (0, 250% strain).
- DSC, DMA samples cut from the same rectangular specimen.

- Trends in stress at -18°C:
  - 0% index (high)
  - 250% palm (middle)
  - 250% pinky/ring, index (low)

- Low stress is proportional to low modulus.
- Modulus is an indicator of material toughness/stiffness, and possibly a tendency to tear.
Use of DSC and DMA to Study Rubber Crystallization in a Neoprene Glove

Neoprene samples cut from the pinky/ring crotch of two different right-handed gloves:
- “as is” material.
- Freezer-conditioned (0, 150 & 250% strain).
- Up to 25-30% decrease in stress in going from unstrained to strained material.
- Up to 25% decrease in stress in going from 150 to 250% strain.
Use of DSC and DMA to Study Rubber Crystallization in a Neoprene Glove

Summary and Conclusions

- 0.5-1 in. long tear was found in the pinky/ring finger crotch of a left-hand neoprene latex rubber glove used as part of an ACES pressurized astronaut suit. The fairly smooth tear pattern could not be reproduced, and could have been caused by strain-induced crystallinity.

- NASA/MSFC was involved in testing in 1999 when crystallinity was induced in neoprene latex rubber gloves (used on Space Station) because of slow HCl release.

- Two pairs of ACES gloves were tested at MSFC by DSC and DMA techniques. Samples were cut from three areas: palm, index finger and pinky/ring finger crotch. Samples were tested “as is,” as well as freezer-conditioned at 0, 150 and/or 250% strain for up to ≈ two weeks.

  - Highest crystallinity is induced with 0% strain in pinky/ring, 250% in palm, index finger.
  - Lowest stress (at -18°C) induced with 250% strain in pinky/ring, index finger.
  - Highest linear CTE (-18 to 0°C) of palm might make it least likely of three areas to tear.
  - Most data indicated a maximum crystallization rate for 6-7 days of freezer conditioning.