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DETERMINING PERMISSIBLE OXYGEN AND WATER VAPOR TRANSMISSION RATE FOR NON-RETORT MILITARY RATION PACKAGING

by
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U.S. Army Natick Soldier Research, Development and Engineering Center
Natick, Massachusetts 01760-5018

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14. ABSTRACT
This report documents a study that was conducted by the Department of Defense (DoD) Combat Feeding Directorate at the Natick Soldier Research, Development and Engineering Center (NSRDEC) to determine baseline barrier requirements, such as oxygen transmission rate (OTR) and water vapor transmission rate (WVTR), for the non-retort pouch found in the Meal, Ready to Eat™ (MRE™) individual ration. During this study, cracker samples were packed in pouches with known permeation to oxygen and water vapor. Cracker pouches were then stored under controlled environmental conditions. Crackers were pulled from storage at predetermined intervals and tested for various attributes, such as sensory quality, moisture content, water activity, texture, and headspace. From the sensory analysis it was found that the required WVTR for the non-retort pouch to meet a 3-year shelf life is at least 0.13 $g/m^2/d$, using the current units for WVTR that are found in military specification PCR-C-037. However, when taking into account pouch size, which is a more accurate and realistic way to determine and specify required WVTR, the allowable water vapor ingress is 0.004 g/pouch/d. Cracker samples used to determine permissible OTR did not fall below the overall quality requirement for sensory attributes during the 32-week study. Thus, an allowable OTR for the non-retort pouch cannot be calculated from the results obtained.

15. SUBJECT TERMS

RETORT	STORAGE	SHELF LIFE	RETORT POUCHES	SENSORY ANALYSIS
OXYGEN	CRACKERS	PACKAGING	SENSORY TESTING	STORAGE STABILITY
POUCHES	HEADSPACE	SHELF STABLE	MOISTURE CONTENT	
RATIONS	PERMEANT	PERMEABILITY	FOOD PRESERVATION	
BARRIER	POLYMERS	WATER VAPOR	MRE(MEAL, READY-TO-EAT)	

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Preface

This report documents a study that was conducted to determine baseline barrier requirements for the non-retort pouch found in the Meal, Ready to Eat^{TM} (MRE^{TM}) individual ration. These baseline requirements, for oxygen transmission rate (OTR) and water vapor transmission rate (WVTR), will be used in a permeability prediction model that allows for calculation of barrier performance of developmental packaging structures in a wide range of environments. The Natick Soldier Research, Development and Engineering Center (NSRDEC) conducted this work between March 2009 and January 2010 under the project entitled "Transport Properties for the Prediction of Barrier Requirements." TB 07-01. That project was funded by the Combat Feeding Research and Engineering Program from FY07 through FY09. The work described in this report was also leveraged by funding for the "Modeling and Processing of Smart Blended Polymer Nanocomposites and Oxygen Scavenger Formulations" project, which was funded through the Environmental Quality Basic Research and Development Program, from FY06 through FY08.

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DETERMINING PERMISSIBLE OXYGEN AND WATER VAPOR TRANSMISSION RATE FOR NON-RETORT MILITARY RATION PACKAGING

1. Introduction

This report documents a study that was conducted from March 2009 through January 2010 to determine baseline barrier requirements for the non-retort pouch, found in the Meal, Ready to Eat^{TM} (MRE^{TM}) individual ration. These baseline requirements will be used as a benchmark in calculation of barrier performance of non-foil developmental packaging structures to ensure they meet the military's shelf-life requirements in a wide range of environments.

The pouch used for non-retort food items does not have any specified barrier requirements. Without a clear baseline for oxygen transmission rate (OTR) and water vapor transmission rate (WVTR), it is difficult to develop new packaging materials, as there is no target to meet or exceed in permeability modeling studies. Use of a permeability prediction model is the most resourceful technique for calculating the total barrier performance of developmental packaging structures and technologies.

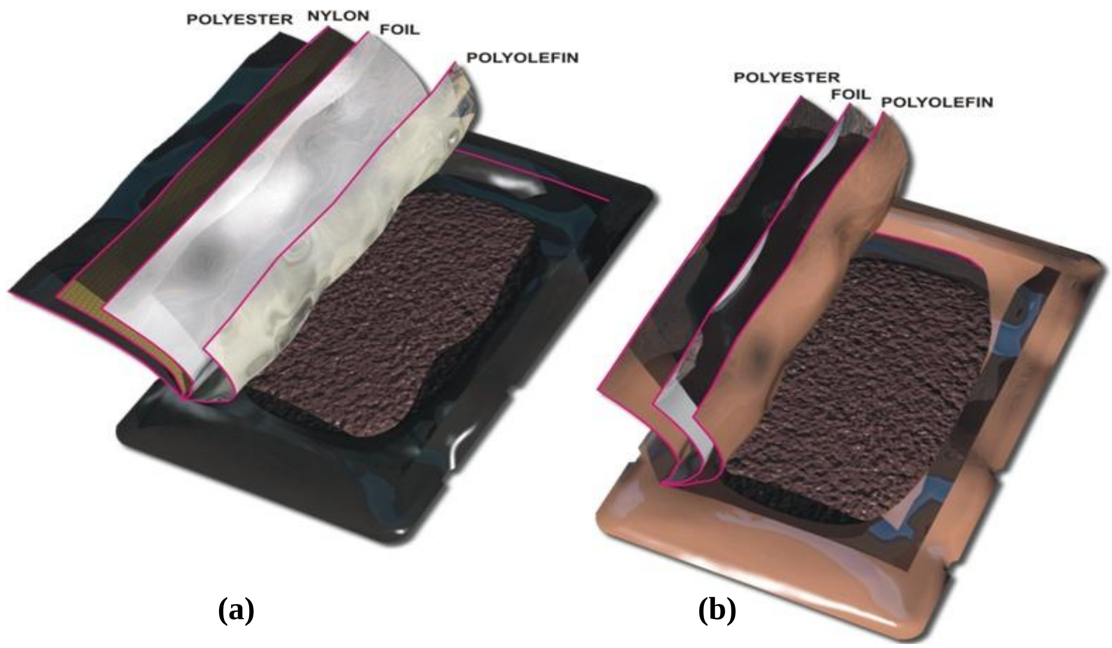
The Natick Soldier Research, Development and Engineering Center (NSRDEC) conducted the storage study, testing, and analyses that are outlined in this report in an attempt to determine the required OTR and WVTR. The work was part of the project entitled, "Transport Properties for the Prediction of Barrier Requirements." That project was funded by the Combat Feeding Research and Engineering Program from FY07 through FY09. The work described in this report was also leveraged by funding for the "Modeling and Processing of Smart Blended Polymer Nanocomposites and Oxygen Scavenger Formulations" project, which was funded through the Environmental Quality Basic Research and Development Program from FY06 through FY08.

The continued need for low-cost, non-foil, high performance packaging materials for existing and future combat ration packaging systems has been addressed with a significant amount of research and development in the area of high barrier polymers. There are several reasons why non-foil packaging is being pursued by the military. First, foil does not perform well. Due to its inherent nature, foil tends to fail in the form on pinholes and stress cracks, which allow the ingress of oxygen and moisture, thereby shortening the overall shelf life of the ration. Furthermore, elimination of the foil layer in ration packaging allows packaging waste to be recycled, if the appropriate facilities are in place. Lastly, it is expected that non-foil packaging will be required in order to be compatible with food sterilization techniques, such as microwave and high pressure pasteurization.

Cutting edge technologies such as nanocomposites, active packaging (i.e., oxygen scavengers), barrier coatings, smart blending, and layer multiplying are currently being investigated by commercial resin manufacturers, material converters, food packaging companies, and the military. It is expected that a combination of these technologies will be required to meet the stringent shelf-life requirements of military rations such as the MRE^{TM} and the Unitized

Group Ration™ (UGR™). Each of these technologies improves barrier performance in a unique way, whether it be active, passive, or process induced.

The two main packaging structures used for the military's individual ration, the MRE™, are shown in Figure 1: the quad laminate pouch for retort food items and the tri-laminate pouch for non-retort items. The retort pouch has four layers. From the inside-out, they are: polyolefin, aluminum foil, nylon, and polyester. The non-retort pouch does not have the nylon layer; it has the other three layers in the same sequence. The retort pouch has specified barrier requirements to ensure it meets the military's shelf life requirements of 3 years at 27^oC or 6 months at 38^oC. The barrier requirements include an OTR of at least 0.06 $cc/m^2/day$ at 50% RH and 23^oC and a WVTR of at least 0.01 $g/m^2/day$ at 90% RH and 37.8^oC.[1] The study described here is the first step in specifying them for the non-retort pouch.



(a) **(b)**

Figure 1: Primary Packaging for MRE. (a) MRE Retort Pouch Quad-Laminate Structure; (b) MRE Non-Retort Pouch Tri-Laminate Structure

The major tasks for this study included:

- Develop test plan, and determine packaging, food component, and storage study criteria.
- Prepare test samples for storage study.
- Conduct sensory analysis on all cracker samples at predetermined intervals throughout storage study.
- Conduct water activity and moisture content analysis on crackers stored at elevated relative humidity.
- Conduct texture analysis on crackers stored at elevated relative humidity.
- Conduct headspace analysis for samples stored at low relative humidity.
- Analyze the results.

[1] Military Performance Specification: Retort Pouch Specification MIL-C-44073F, February 12, 2003.

2. Experimental Approach

2.1 Packaging Materials, Food Components, and Storage Conditions

At the start of this study suitable food items, packaging materials, and storage conditions were determined that would ensure the most valuable and informative data would be collected over an accelerated period of time. The MRE™ plain cracker, shown in Figure 2, was the food component chosen because it is sensitive to moisture and is susceptible to oxidation.

Figure 2: Meal Ready to Eat™ Plain Cracker

To accurately separate and measure the effect of each permeant (water vapor and oxygen) on the properties and sensory attributes of the cracker, it was necessary to pack the crackers in materials with different permeation rates and store them under different controlled environmental conditions. The polymer substrates chosen for the pouch materials were a polyethylene (PE)/nylon blend to determine permissible WVTR and a pure PE to determine permissible OTR. These materials were chosen because they are known to be highly permeable to water vapor and oxygen, respectively. Samples used to determine permissible WVTR were packed in the pouch made from a PE/nylon blend, which has better oxygen barrier than the pure PE pouch and therefore minimizes oxygen ingress into the package, while allowing water vapor to permeate through. To further accelerate water vapor permeability, the samples packed in the PE/nylon blend pouches were stored at an elevated RH of 90% at 100^{o}F. Samples used to determine permissible OTR were packed in the PE pouch, which is more permeable to oxygen. To minimize the ingress of water vapor into the package, samples were stored in dry conditions of approximately 10% RH at 100^{o}F.

This study included five sets of samples. They are shown in Table 1, along with pouch material/configurations, environmental storage conditions, and the barrier requirement the sample is being used to determine. Test Pouch samples (#1 and #2) utilized either a PE or PE/nylon pouch, as specified in Table 1. For Control #1 and #2, crackers were first placed in either a PE or PE/nylon pouch (unsealed), as specified in Table 1, and then sealed in a tri-laminate foil pouch. This packing configuration was utilized to ensure that Control #1 and #2 samples were stored directly next to the polymeric pouch, in case the pouch imparted an off taste to the cracker as a result of migration of compounds from the polymeric film to the cracker. A third control (Control #3), which utilized identical packaging materials (tri-laminate pouch) and process as the MRE™ cracker, was used as a comparison to Control #1, to determine if the polymeric pouch affected sensory attributes of the cracker. All cracker samples were supplied by Interbake Foods. All except the Control #3 pouches were packed at NSRDEC from Bulk Lot 9125. The Control #3 samples were packed at Ameriqual, from Bulk Lot 9148, using the same filling and sealing line that is used for the MRE™ cracker.

Table 1: Sample ID, Pouch Material/Configuration, Storage Conditions, and Barrier Requirement

Sample ID	Pouch Material/Configuration	Storage Conditions	Barrier Requirement
Test Pouch #1	PE pouch (packed at NSRDEC)	100°F, 10% RH	OTR
Test Pouch #2	PE/nylon pouch (packed at NSRDEC)	100°F, 90% RH	WVTR
Control #1	PE pouch with tri-laminate overwrap (packed at NSRDEC)	100°F, 10% RH	OTR
Control #2	PE/nylon pouch with tri-laminate overwrap (packed at NSRDEC)	100°F, 90% RH	WVTR
Control #3	MRE™ tri-laminate pouch (packed at Ameriqual)	100°F, 10% RH	OTR

2.2 Packing Technique

The crackers were vacuum packed and sealed using a Multivac AG800, which allowed for five pouches to be sealed simultaneously. Each pouch contained two crackers, which were placed directly on top of one another in the center of the pouch. The PE pouches were sealed using heat/seal time set at 3 and vacuum set at 6, which equates to approximately 28 in Hg. The PE/nylon pouches were sealed using a heat/seal time of 4.5 and vacuum set at 6. The foil control pouches were sealed with a heat/seal time setting of 4.5 and vacuum of 6. Figure 3 shows the storage arrangement of the crackers in their pouches.

Figure 3: Arrangement of Crackers during Storage Study

3. Testing and Analysis Methodology

Figure 4 shows the test plan that was followed during this study. All samples underwent sensory analysis. All samples tested for WVTR (those stored at 100°F and 90% RH) were also tested for water activity, moisture content, and texture. All samples tested for OTR (those stored at 100°F and 10% RH) were also tested for headspace to determine hexanal levels and, in turn, occurrence of oxidation or rancidity. Samples were pulled and analyzed weekly for the first 6 weeks, and then again after weeks 8, 12, 16, 24, and 32. Additionally, at the start of the study, OTR and WVTR were measured for each pouch material (described in Section 2.1) to record actual permeation rates of the films at the specified storage conditions. These data will be used in conjunction with sensory results and physical properties to determine required oxygen and water vapor requirements.

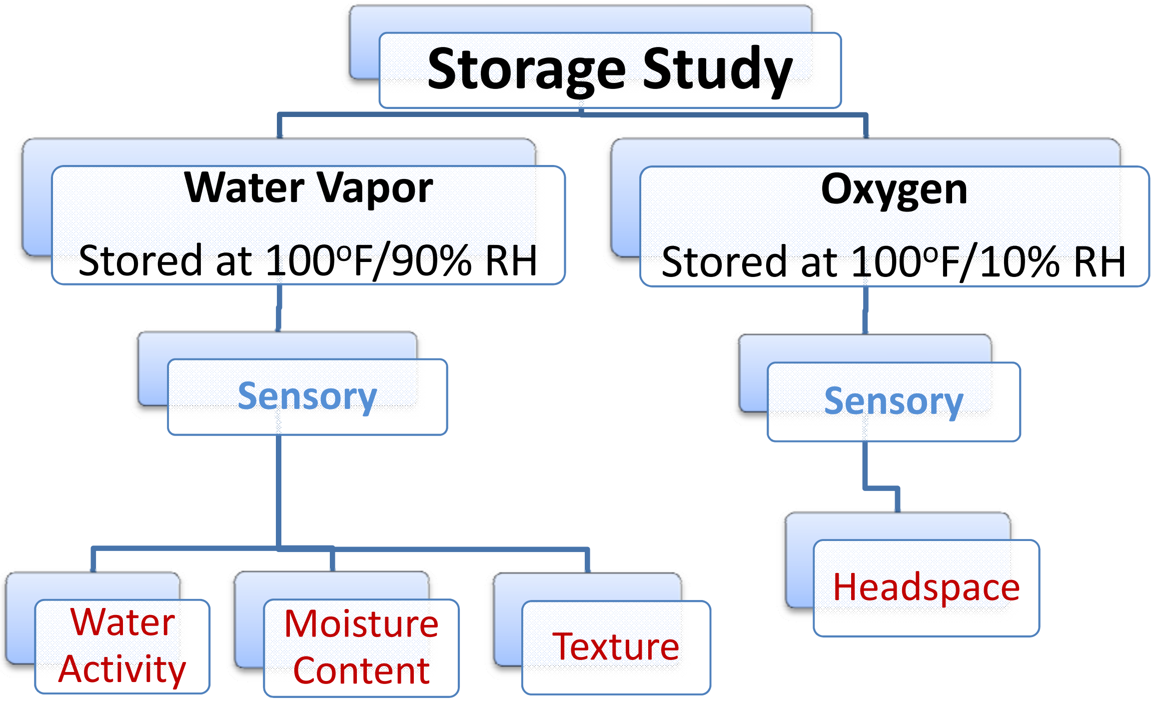
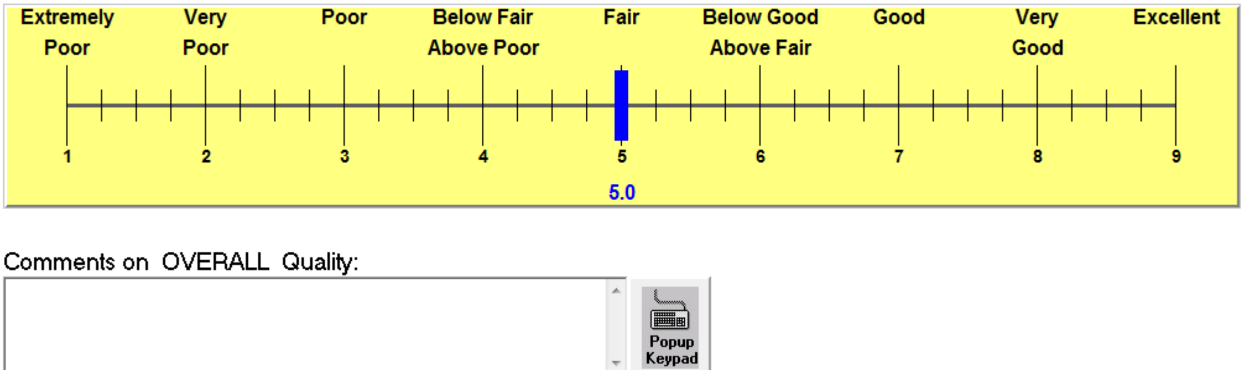


Figure 4: Test Plan

3.1 Sensory Analysis

Sensory analysis allows for evaluation of food items with respect to appearance and palatability. Acceptability of the crackers was determined by a trained sensory evaluation panel, which assessed and rated the cracker for flavor, taste, odor, and texture. Ratings were based on a 9-point qualitative scale for each cracker attribute, along with an overall quality score for the cracker. According to military specification PCR-C-037[2], "Crackers, Fortified, Packaged in a Flexible Pouch, Shelf Stable", the cracker must receive an overall score of 5.0 or higher based on the 9-point scale to be considered acceptable. The crackers were analyzed after each pull to determine at what point the crackers fell below an overall score of 5.0. A screen shot of the scale used in this study is shown in Figure 5.

[2] PCR-C-037, Crackers, Fortified, Packaged in a Flexible Pouch, Shelf Stable, 23 December 2004 (w/change 01 05, May 2009).



Blue marker is the "cut off point" of 5.0.

Figure 5: Scale and Comment Box for Rating and Describing Quality Attributes

3.2 Moisture Content Analysis

Moisture content analysis allows for a quantitative measure of the total amount of water present in a food item which can influence the texture, taste, and appearance of food products. According to military specification PCR-C-037, “Crackers, Fortified, Packaged in a Flexible Pouch, Shelf Stable”, moisture content shall be not less than 1.5 percent and not greater than 4.0 percent. Crackers were analyzed after each pull to determine at what point the crackers fell out of this moisture content range. Samples from Test Pouch #2 and Control #2 were tested for moisture content, as they were the samples stored at elevated humidity for determining baseline WVTR.

The contents of individual pouches were ground into a powder, using an Osterizer 12-speed blender for approximately 7 s on the pulse setting. Three pouches were tested for each sample, with one pouch of crackers being an individual test specimen. Sample boat weights were taken. Ground samples were then loaded into the boats and re-weighed to obtain a total weight of the boat plus sample. Filled sample boats were loaded into the Hotpack 207380 vacuum oven in the Combat Feeding Directorate pilot plant for 27 h at 60^{o}C. Upon removal from the oven, the samples were re-weighed to determine overall weight loss.

3.3 Water Activity Analysis

Water activity is a measure of the energy status of the water in a system, and is an indicator of perishability. Although there is no military specification for water activity analysis, it was conducted to see how it compared with the sensory results and the moisture content results. The water activity was measured by equilibrating the liquid phase water in the sample with the vapor phase water in the headspace and measuring the RH of the headspace. Samples from Test Pouch #2 and Control #2 were tested for water activity, as these samples were stored at elevated humidity for determining baseline WVTR. The samples were taken from the same samples that were ground up for moisture content. Again, three pouches were tested for each sample, with one pouch of crackers being an individual test specimen. The samples were stored in a sealed container before they were tested for water activity using an Aqua Lab 4TE. Before testing, a verification standard of LiCl 13.41 molal in H_2O (aw = 0.250 +/-0.003) was run on the water activity meter to ensure the equipment was running properly.

3.4 Texture Analysis

There is also no military specification for texture analysis. It was conducted because the presence of moisture is known to affect the hardness of food items like the crackers, as they absorb moisture from the environment around them. A TA-XT2 Texture Analyzer was used to run the texture tests, and Texture Exponent software was used to analyze the data. The testing profile "Biscuit Bending – BIS4_3PB" was used as the test set-up. This test method utilizes a compression test mode, a test speed of 3 mm/s, and a target mode distance of 5.00 mm. This test set-up utilized a three-point bend fixture with a curved blade. The distance between the supports of the fixture was set to 40 mm. Figure 6 is a photograph of the test set-up.

Figure 6: Test Set-Up for Texture Analysis

Samples from Test Pouch #2 and Control #2 were tested for texture, as these samples were stored at elevated humidity for determining baseline WVTR. Three pouches were tested for each sample. Prior to testing, the cracker dimensions (width and height/thickness) were recorded. In addition, all samples were tested with the lines on the back of the cracker parallel with the length of the probe, and the crackers were placed flush with the front of the adjustable supports. A macro in the software was used to calculate hardness.

3.5 Headspace Analysis

Although there is also no military specification for headspace analysis, it was conducted to assess the extent to which oxidation or rancidity occurred and to correlate the results with the sensory data. It involves measuring levels of hexanal, which is a volatile compound that is a secondary oxidation product of fats and is often used as a marker to track lipid oxidation in food analysis.

Hexanal levels were measured via gas chromatography with mass spectroscopy (GCMS), using an Agilent 6890 gas chromatographer coupled to an Agilent 5975 mass spectrometer. The data were analyzed using Agilent ChemStation software, which is the standard software for the analysis of gas and liquid chromatography. All samples were pulverized prior to testing and tested in triplicate. Samples from Test Pouch #1, Control #1, and Control #3 underwent hexanal analysis, as these samples were stored at low humidity for determining baseline OTR. The samples were placed in 20 mL headspace vials and sampled with solid phase microextraction (SPME). A standard of 1 ppm hexanal was run prior to each sequence to determine retention time and reproducibility of the instrument.

4. Results

Table 2 shows the results of the OTR and WVTR measurements for each pouch material taken at the start of the study to record actual permeation rates of the films at the specified storage conditions. The OTR of the pouch material was measured at 0% RH and 100°F using air, which has an average oxygen concentration of 21%, as the test gas. The WVTR was measured at 90% RH and 100°F. These data will be used in conjunction with sensory results and physical properties to determine required oxygen and water vapor requirements.

Table 2: Oxygen and Water Vapor Permeability of Selected Pouches

Pouch	OTR cc/(m²-d)	WVTR gm/(m²-d)
Polyethylene / Nylon	94	5
Polyethylene	521	6

4.1 Sensory Analysis

Sensory results for all samples are shown in Figure 7, and the raw data are provided in Appendix A. Due to the limited number of samples that could undergo sensory analysis, Control #2 (stored at 90% RH) was not included in this testing. Moisture content and water activity are stronger indicators of when samples for WVTR failed, and sensory analysis and hexanal are stronger indicators for OTR, which is why this rationale was chosen.

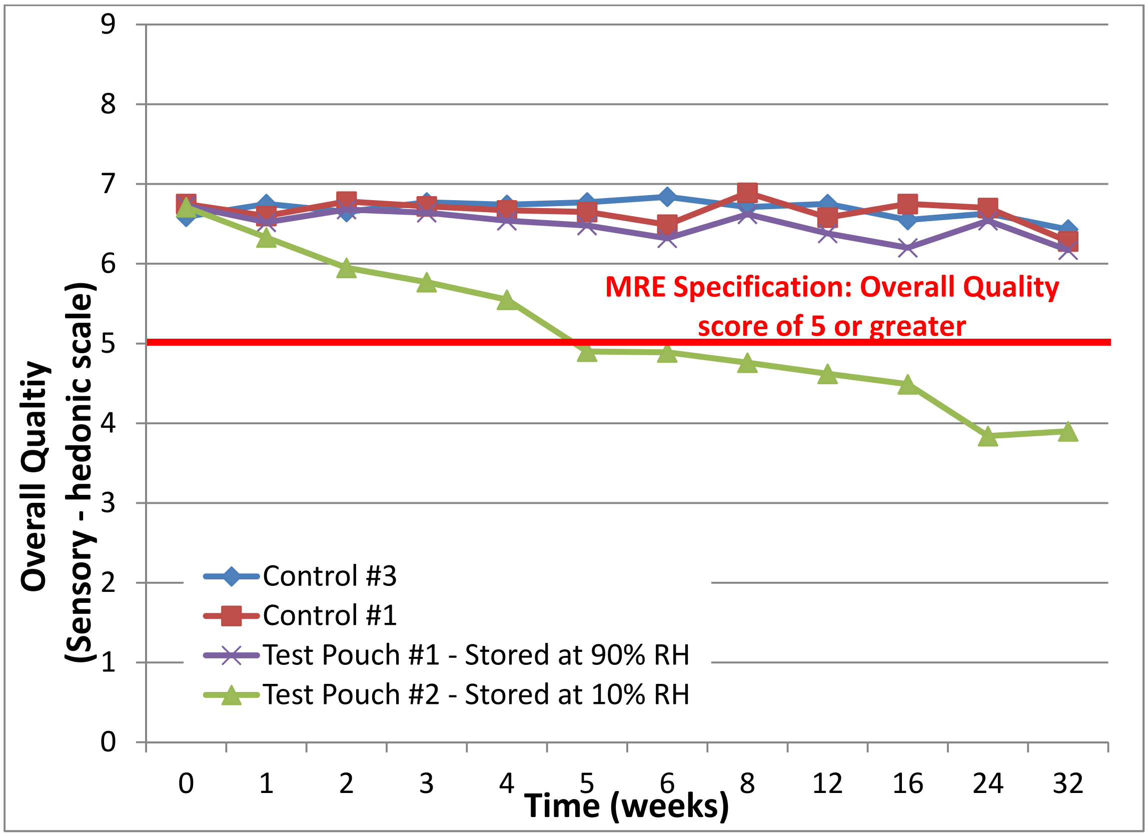


Figure 7: Overall Quality Results from Sensory Evaluation

4.1.1 WVTR Samples

The green curve in Figure 7 indicates that the samples tested for WVTR (stored in Test Pouch #2, the PE/nylon pouch, at 90% RH) were first rated below the requirement of $\geq$5 on a 9-point hedonic scale for overall quality at week 5. This signifies that the ingress of water vapor caused the package to fail between weeks 4 (28 d) and 5 (35 d). As specified in the PCR-C-037 military specification, allowable WVTR is calculated as g/m^2/d. The WVTR of the film tested (5 g/ m^2/d) and the day (28) of the last acceptable rating were used to calculate the maximum WVTR that will meet the required 3-year (1095-d) shelf life, as follows:

$$\underbrace{5\,\frac{\text{g}}{\text{m}^2\text{ x d}}}_{\substack{\text{WVTR}\\\text{of film}}} \times \underbrace{28\text{ d}}_{\substack{\text{Time}\\\text{before failure}}} \times \underbrace{\frac{1}{1095\text{ d}}}_{\text{Shelf life}} = \underbrace{0.13\,\frac{\text{g}}{\text{m}^2\text{x d}}}_{\substack{\text{Allowable}\\\text{WVTR}}}$$

Based on this calculation, the required WVTR is $\leq$ 0.13 g/m^2/d. However, expressing WVTR in g/m^2/d does not take into account the size of the pouch or package. The size of the pouch should be considered, as many of the non-retort food items have different size pouches. For example, the maximum inside dimensions of the cheese pouch are 2-7/8 in wide by 5-3/8 in long, whereas the maximum outside dimensions of the cracker pouch is 6 in wide by 6 in long. Pouch size impacts water vapor permeation, and permeation in general, because the larger the surface area the higher the ingress of water vapor into the package.

Expressing WVTR in terms of package size would be ideal to ensure that all pouch configurations meet the requirement. In the case of the cracker used for this study, the data collected during the sensory study and the size of the cracker pouch can be used to determine what quantity or ingress of water vapor caused the cracker to become unacceptable. The calculation is shown below:

$$\underbrace{5\,\frac{\text{g}}{\text{m}^2\text{ x d}}}_{\substack{\text{WVTR}\\\text{of film}}} \times \underbrace{28\text{ d}}_{\substack{\text{Time before}\\\text{failure}}} \times \underbrace{0.030\text{ m}^2}_{\substack{\text{Area of}\\\text{pouch}}} = \underbrace{4.2\text{ g}}_{\substack{\text{Allowable}\\\text{ingress}}}$$

Next, the allowable water vapor of 4.2 g was divided by the required shelf life of 3 years, to determine the required WVTR for the cracker pouch itself, 0.004 g/pouch-day, as follows.

$$4.2\,\frac{\text{g}}{\text{pouch}} \times \frac{1}{1095\text{ d}} = .004\,\frac{\text{g}}{\text{pouch} \times \text{d}}$$

4.1.2 OTR Samples

In contrast to the WVTR performance, the purple curve in Figure 7 indicates no failure of the samples tested for OTR (stored in Test Pouch #1, the PE pouch, at 10% RH) during the study. Thus, it is not possible to determine a permissible OTR, as the crackers did not become unacceptable with respect to sensory attributes.

No detectable changes in the sensory attributes, as a result of oxidation or rancidity of the cracker, were observed even after 32 weeks, although the PE pouches were permeable to oxygen. In addition, the overall quality ratings of the samples in Test Pouch #1 were similar to those for the OTR control samples (Control #1 and Control #3, the red and blue curves, respectively), which were also stored at 10% RH,.

4.2 Moisture Content Analysis

As mentioned earlier, military specification PCR-C-037 also requires that the moisture content of the MRE™ cracker be no greater that 4% and no less than 1.5%. The red curve in Figure 8, indicates that the control samples tested for moisture content (Control #2) remained within the required range throughout the entire 8 months, whereas the crackers stored in Test Pouch #2 (PE/nylon pouch, the blue curve) exceeded the 4% moisture threshold between week 1 (7 d) and week 2 (14 d), which is a relatively short period of time. The data do not correlate with sensory data, where the overall sensory quality requirement was exceeded between weeks 4 and 5 (28 d and 35 d). This set of data provides an alternative method to sensory analysis for calculating the WVTR required for 3-year shelf life. As shown below, the required WVTR based on moisture content analysis would be $\leq$ 0.032 g/m^2/d. These data could also be used to calculate a pouch transmission rate.

$$\underbrace{5\,\frac{\mathrm{g}}{\mathrm{m^2\ x\ d}}}_{\text{WVTR of film}} \times \underbrace{7\ \mathrm{d}}_{\text{Time before failure}} \times \underbrace{\frac{1}{1095\ \mathrm{d}}}_{\text{Shelf life}} = \underbrace{0.032\,\frac{\mathrm{g}}{\mathrm{m^2 x\ d}}}_{\text{Allowable WVTR}}$$

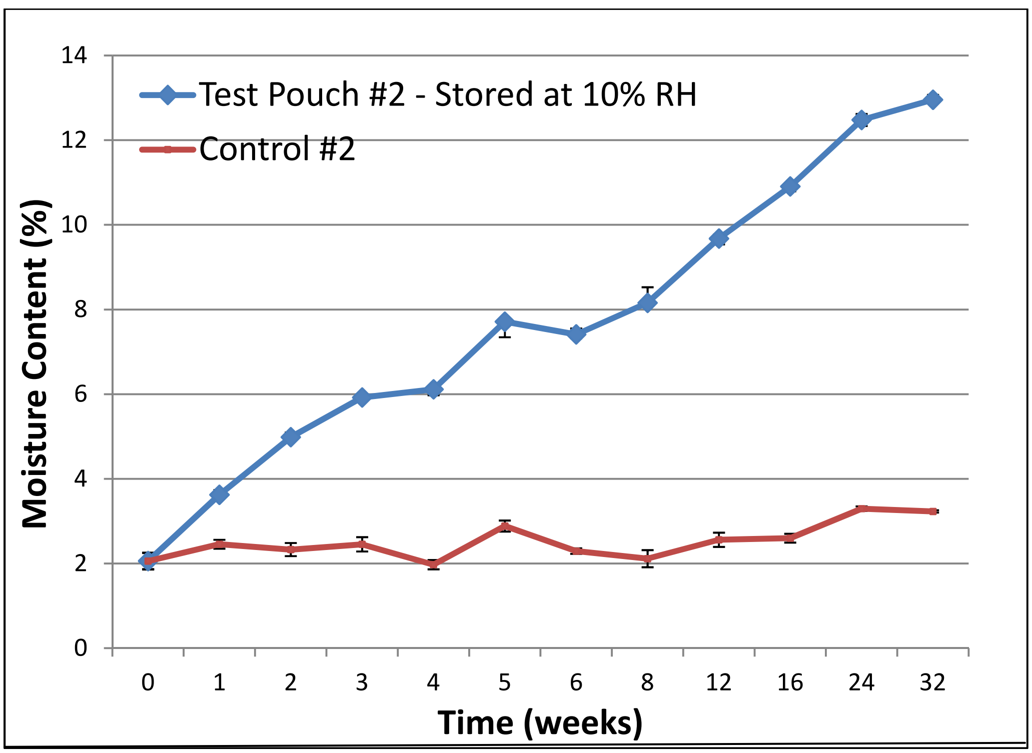


Figure 8: Moisture Content Results

4.3 Water Activity Analysis

The water activity results are shown in Figure 9. Test Pouch #2 (PE/nylon pouch, the blue curve) showed a steady increase in water activity, with the greatest rate of increase in water activity from time = 0 to week 5. The water activity continued to increase until week 24, but at a lower rate. The rate was level between weeks 24 and 32. The trends of increasing moisture content and water activity in Test Pouch #2 were similar to each other over the course of the study. There was very little water activity change in the Control #2 samples (the red curve) over the course of 32 weeks, also similar to the trend for moisture content.

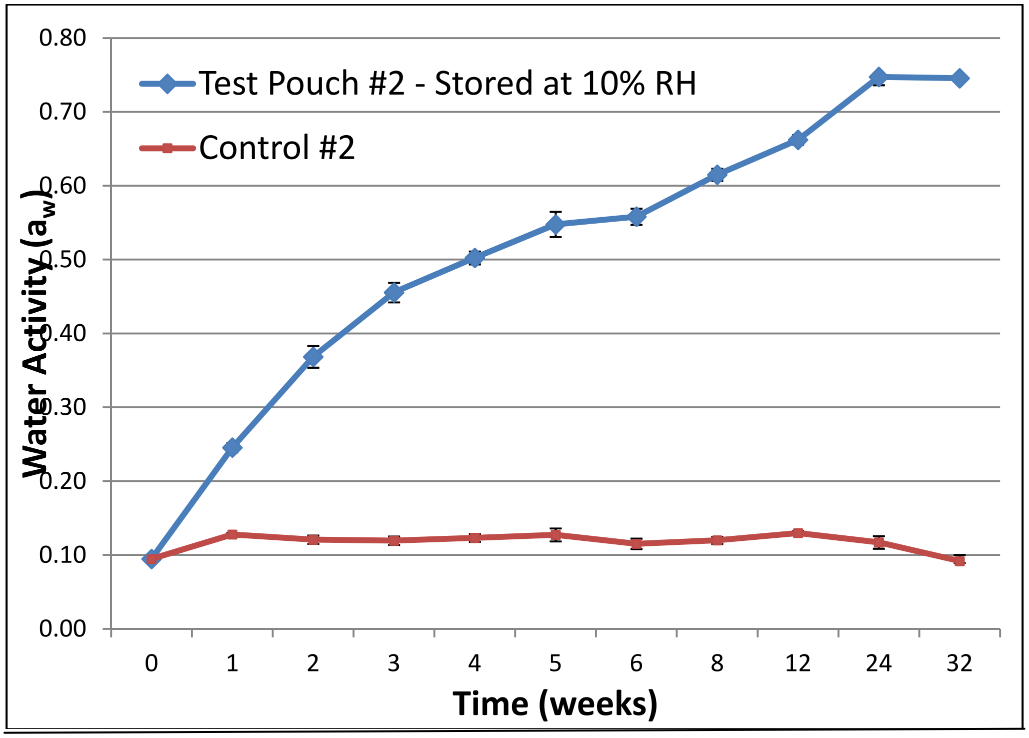


Figure 9: Water Activity Results

4.4 Texture Analysis

Figure 10 shows the results of the texture analysis over the course of the 32-week study. The texture of the crackers stored in Test Pouch #2 (PE/nylon pouch, the blue curve) varied a slightly from week to week between weeks 0 through 5, but remained steady overall during that period. After week 5 those samples showed a steady decline in hardness that continued through week 32. These data somewhat correlate with the sensory results, where the overall quality fell below the military specification at week 5. The samples stored in the Control #2 pouch (the red curve), however, showed relatively no change in hardness over the course of 32 weeks, indicating that moisture ingress into the foil control pouch was minimal.

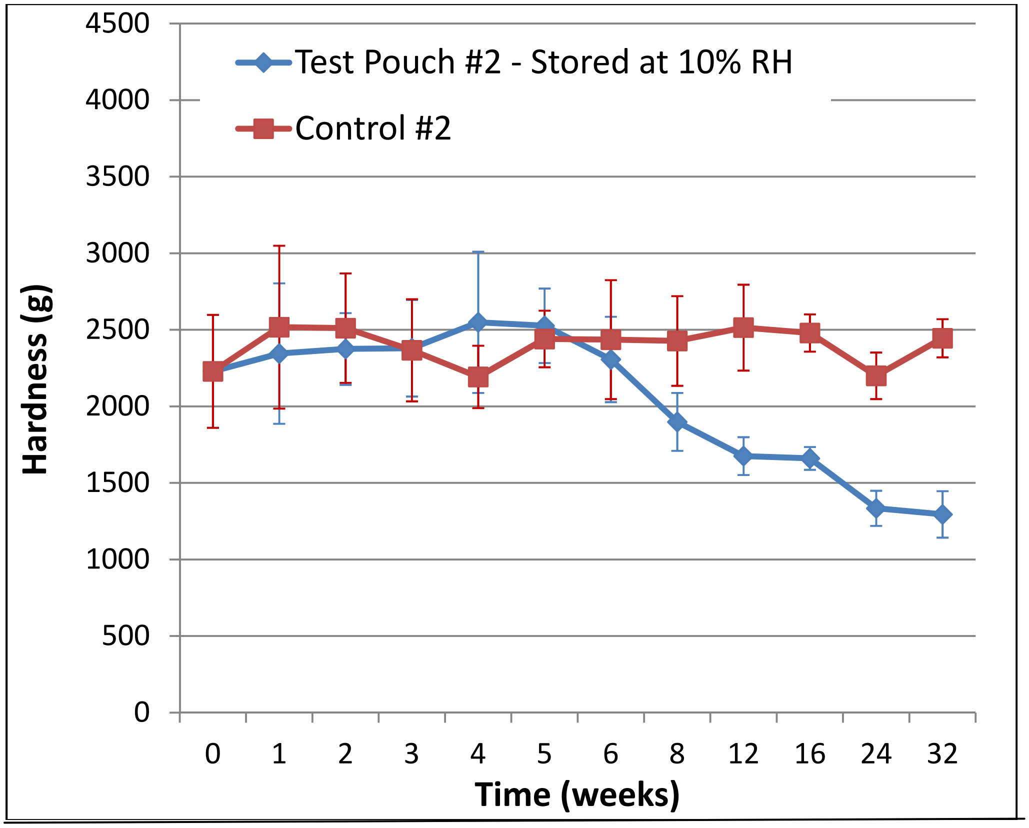


Figure 10. Texture Analysis Results

4.5 Headspace Analysis

Hexanal production was measured to assess the extent to which oxidation or rancidity occurred throughout the 32-week study and to provide results to correlate with the sensory data.

As shown in Figure 11, the crackers packed in the Test Pouch #1 (PE pouch), which was permeable to oxygen, had no significant change in hexanal production from week 0 (black curve) to week 16 (red and blue curves, duplicate tests performed). Samples showed a hexanal abundance of approximately 54,000 at 0 weeks and a hexanal abundance between 45,000 and 55,000 at 16 weeks, which is below the sensory threshold of approximately 750,000. However, data for the crackers packed in the two control pouches, Control #1 and Control #3, show higher abundances of hexanal after 16 weeks in comparison to crackers packed in the PE pouch. As shown in Figure 12, the hexanal abundance in the crackers packed in the Control #1 pouch doubled over the course of 16 weeks, increasing from 40,000 at 0 weeks (black curve) to over 80,000 at 16 weeks (red and blue curves). Figure 13 shows hexanal abundance for crackers packed in the Control #3 pouch; Figure 13 also shows an increase in hexanal from week 0 to week 16. What is also interesting is that Control #3, the crackers packed at Ameriqual, had a hexanal content of 80,000 at week 0, which is roughly twice the week 0 hexanal abundance for Control #1 (packed at NSRDEC) and the PE Pouch. It is not clear why the crackers packed in foil (Control #1 and Control #3) show higher concentrations of hexanal after 16 weeks than those packed in the polymer based PE pouch (Test Pouch #1). One possible cause could be that the PE pouch is permeable to hexanal, and therefore the hexanal permeated through the pouch instead of building up as much as in the foil pouches.

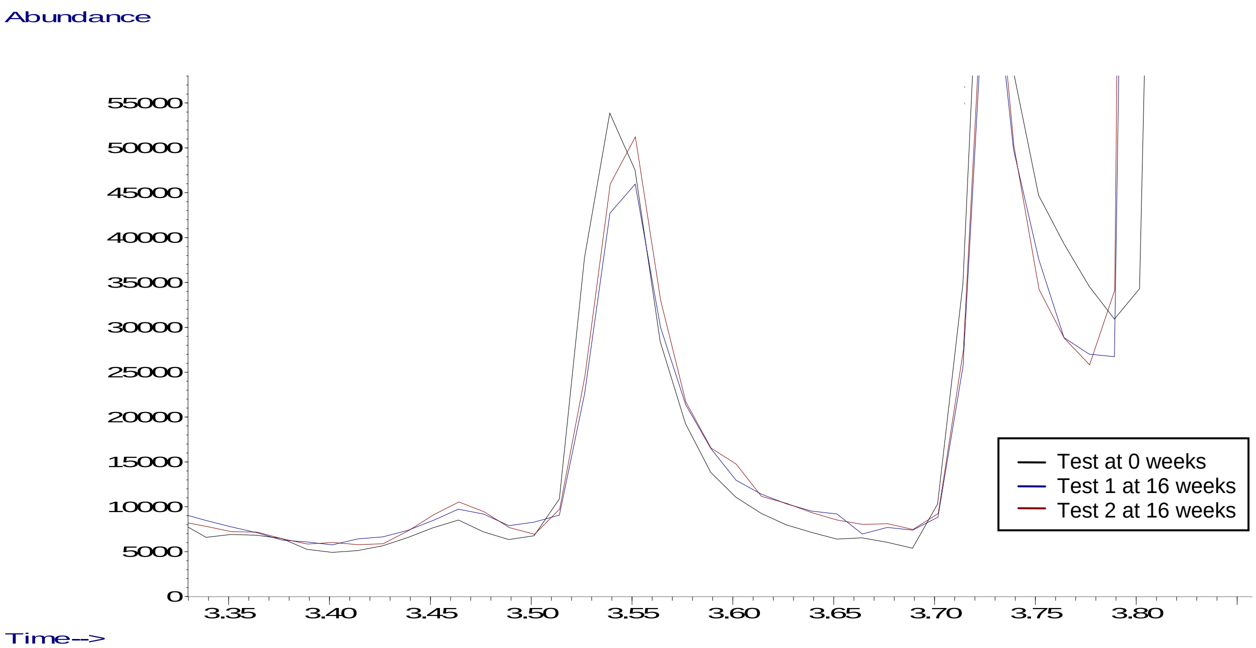


Figure 11. Hexanal Abundance at 0 and 16 Weeks for Test Pouch #1

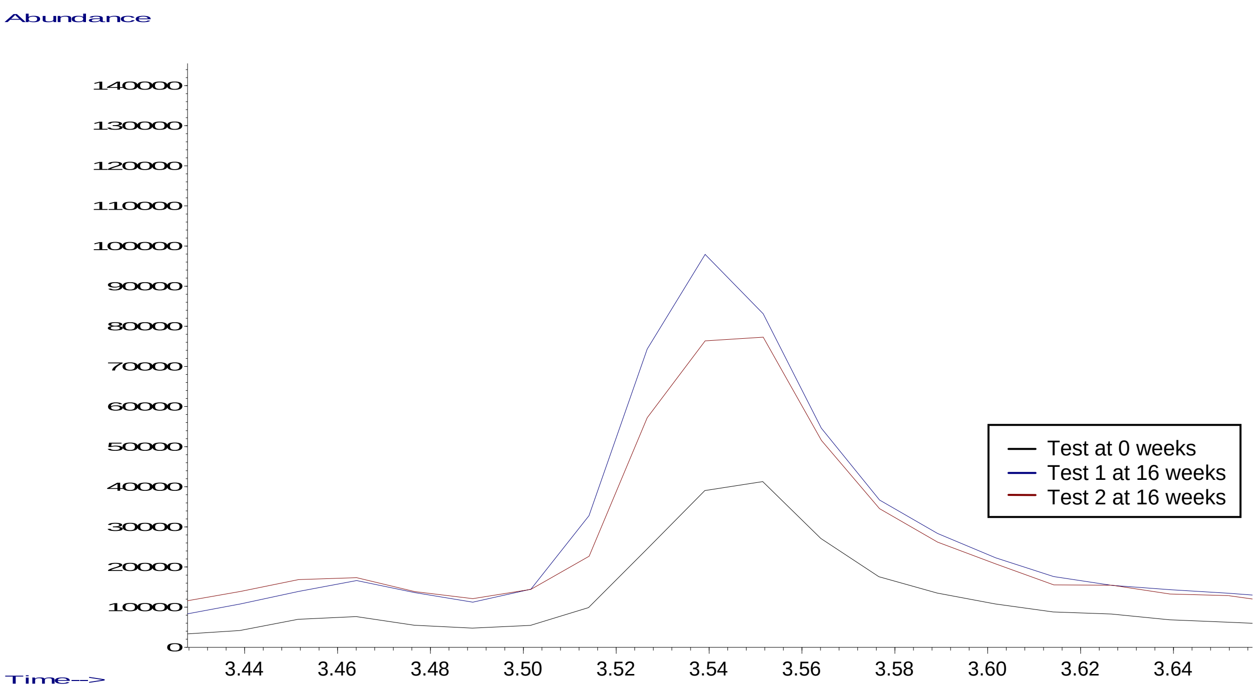


Figure 12: Hexanal Abundance at 0 and 16 Weeks for Control #1

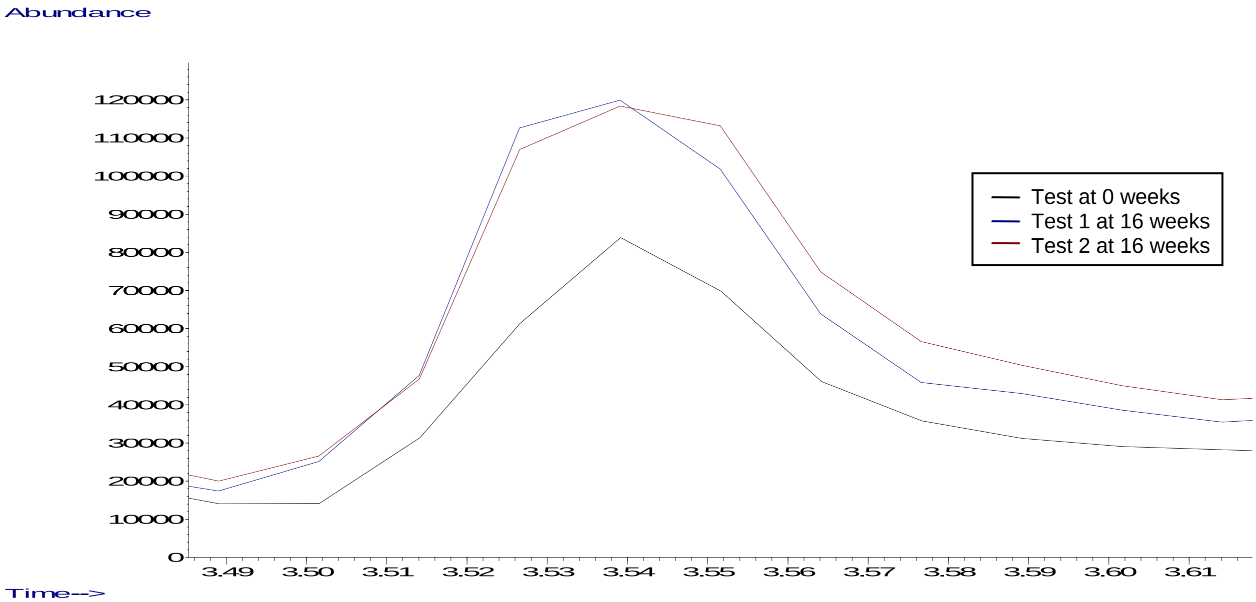


Figure 13: Hexanal Abundance at 0 and 16 Weeks for Control #3

Figure 14 shows hexanal abundance at 32 weeks for Test Pouch #1, the PE pouch, indicated by the peak at 7.379. As shown, there is a significant increase from 16 weeks, with an abundance of approximately 600,000, which is still below the sensory threshold. Figure 15 shows hexanal abundance at 32 weeks for Control #1, indicated by the peak at 7.38. Again, there was an increase in abundance from 16 weeks, with an abundance of approximately 300,000. Finally, Figure 16 shows hexanal abundance of approximately 340,000 at 32 weeks for Control #3, indicated by the peak at 7.435.

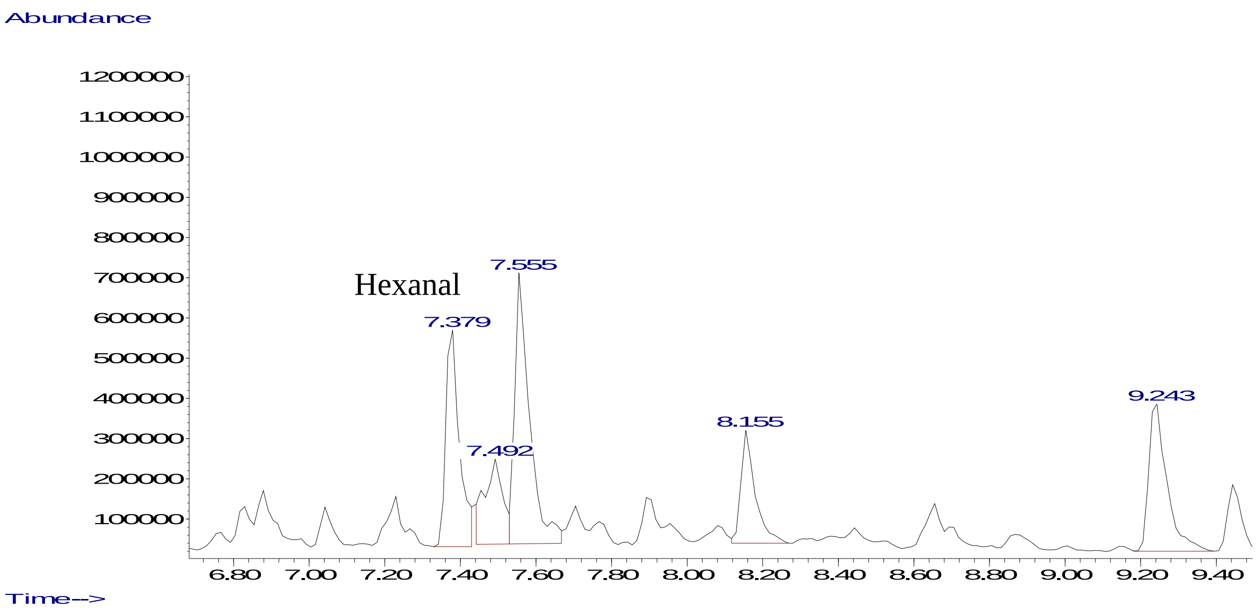


Figure 14: Hexanal Abundance at 32 Weeks for Test Pouch #1

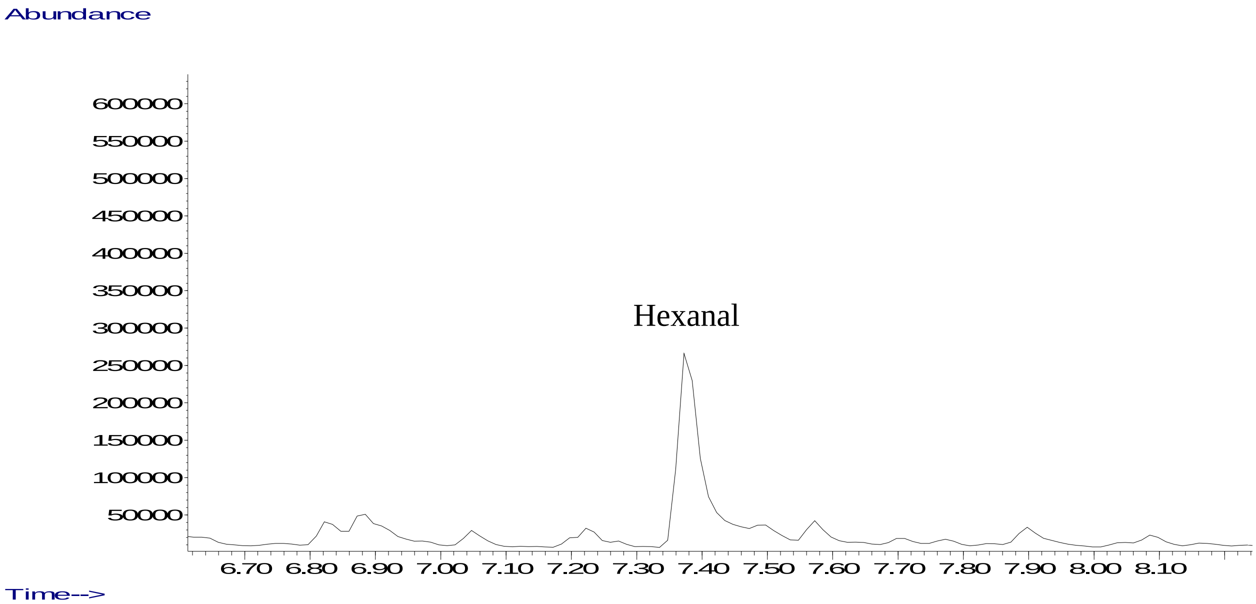


Figure 15: Hexanal Abundance at 32 Weeks for Control #1

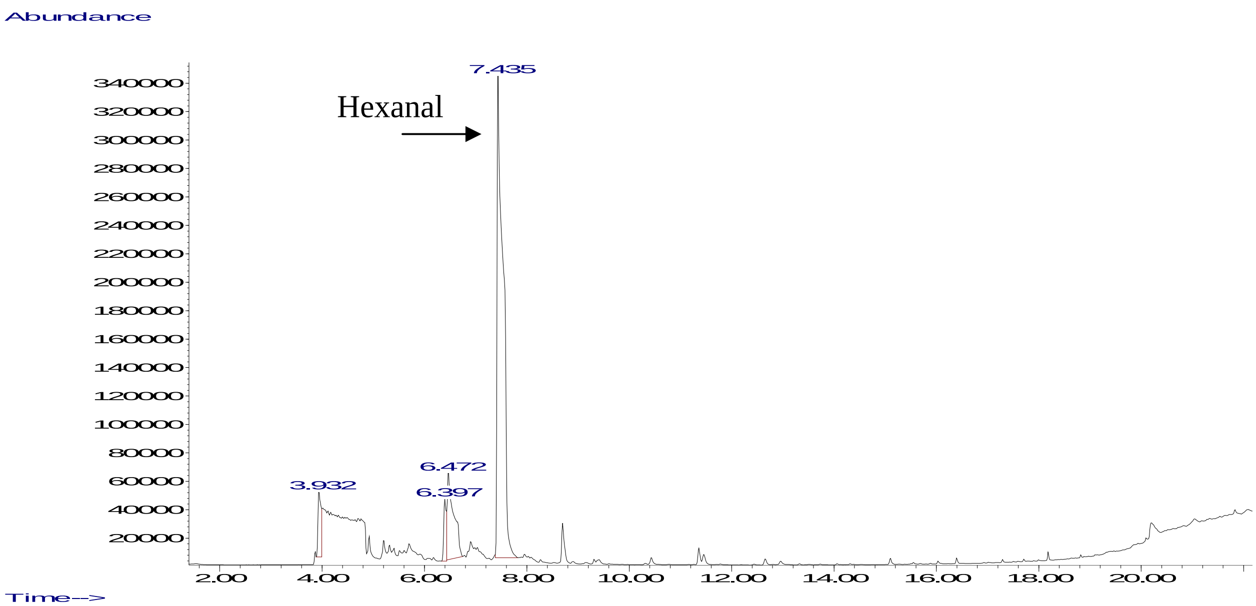


Figure 16: Hexanal Abundance at 32 Weeks for Control #3

Table 3 summarizes the hexanal abundance data for the PE pouch (Test Pouch #1) and both control pouches (#1 and #3) at weeks 0, 16, and 32. At the end of the study (32 weeks), the PE pouch samples had twice as much hexanal abundance as the control pouches. This was expected, as the PE pouch is permeable to oxygen and the foil controls are not. However, this difference in hexanal from control to PE pouch samples is not reflected in the sensory data, most likely because all the hexanal abundance values were below the sensory threshold.

Table 3: Hexanal Abundance

Sample ID	Hexanal Abundance		
	0 Weeks	16 Weeks	32 Weeks
Test Pouch #1	54,000	50,000	600,000
Control #1	40,000	80,000	300,000
Control #3	80,000	120,000	340,000

5. Conclusions

This study was successful in determining an allowable WVTR for the non-retort pouch based on both the sensory analysis and the moisture content analysis. However, it is not possible to calculate an allowable OTR for the non-retort pouch using the data from either the sensory analysis or the headspace analysis.

From the sensory analysis it was found that the required WVTR for the non-retort pouch to meet a 3-year shelf life is at least 0.13 $g/m^2/d$, using the current units for WVTR that are found in military specification PCR-C-037. However, when taking into account pouch size, which is a more accurate and realistic way to determine and specify required WVTR, the allowable water vapor ingress is 0.004 g/pouch/d. Moisture content data were also used to determine required WVTR for the non-retort pouch. Using that method, the maximum allowable WVTR was found to be 0.032 $g/m^2/day$. However, the sensory and moisture data did not correlate with one another. Moisture content and water activity data for the crackers stored in the PE/nylon pouch at 90% RH had similar trends to one another, as expected, over the course of the 32-week study. Also as expected, the control samples stored at 90% RH showed no significant change in moisture content or water activity over the course of the study. Texture analysis showed a decrease in hardness after week 5 for the samples in the PE/nylon pouch, with a continued decline through week 32. The texture data did correlate with the sensory data, as overall quality for the crackers packed in the PE/nylon pouch fell below the military specification at week 5. The control samples showed no significant changes in hardness over the course of the study.

The samples used to determine permissible OTR, which were stored in the PE pouch at 10% RH, did not fall below the overall quality requirement for sensory testing during the 32-week study. The results from headspace analysis for those samples correlate with the sensory data, as hexanal abundance did not exceed the sensory threshold over the course of the 32-week study. Thus, no baseline for OTR can be obtained from the data from either analysis.

This document reports research undertaken at the U.S. Army Natick Soldier Research, Development and Engineering Center, Natick, MA, and has been assigned No. NATICK/TR- 12/008 in a series of reports approved for publication.

Appendix A
Sensory Data

Table A-1: Summary of Mean-Scores, P-Values, and Significance for T=0 Weeks

Summary of Mean-Scores, P-Values, and Significance (Duncan's) Test Result Code - T09+26A						
This test was evaluated by 15 panelists.						
Attribute	Manufacturer pkg (MRE), T=0	NSRDEC pkg >~2wks (MRE overlay), T=0	NSRDEC pkg >~2wks (PE/Nylon-VT), T=0	NSRDEC pkg >~2wks (PE-OT), T=0	P-Value	Sig
APPEARANCE Quality	6.95	6.97	7.03	7.01	0.7212	NS
ODOR Quality	6.78	6.83	6.88	6.83	0.7598	NS
FLAVOR Quality	6.65	6.71	6.79	6.73	0.397	NS
TEXTURE Quality	6.68	6.83	6.65	6.76	0.5855	NS
OVERALL Quality	6.59	6.75	6.71	6.73	0.5906	NS

Table A-2: Summary of Mean-Scores, P-Values, and Significance for T=1 Week

Summary of Mean-Scores, P-Values, and Significance (Duncan's) Test Result Code - T09+26B						
This test was evaluated by 16 panelists.						
Attribute	T=1wk, PE Pouch - 1A OT, 100°F, 10% RH	T=1wk, Control#3 - 1C Pkg MRE, 100°F, 10% RH	T=1wk, Control#1 -1B OT/MRE Overlay, 100°F, 10% RH	T=1wk, PE/Nylon Pouch - 2A VT, 100°F, 90% RH	P-Value	Sig
APPEARANCE Quality	6.89	6.86	6.88	6.88	0.9775	NS
ODOR Quality	6.59	6.68	6.61	6.39	0.0744	NS
FLAVOR Quality	6.52	6.76	6.64	6.37	0.0888	NS
TEXTURE Quality	6.49	6.8	6.6	6.47	0.1342	NS
OVERALL Quality	6.52	6.75	6.6	6.33	0.0705	NS

Table A-3: Summary of Mean-Scores, P-Values, and Significance for T=2 Weeks

Summary of Mean-Scores, P-Values, and Significance (Duncan's) Test Result Code - T09+26C						
This test was evaluated by 13 panelists.						
Attribute	Manufacturer pkg (MRE), T=2wks@100°F	NSRDEC pkg >~2wks (MRE overlay), T=2wks@100°F	NSRDEC pkg >~2wks (PE/Nylon-VT), T=2wks@100°F	NSRDEC pkg >~2wks (PE-OT), T=2wks@100°F	P-Value	Sig
APPEARANCE Quality	6.92	6.92	6.98	7	0.1163	NS
ODOR Quality	a 6.76	a 6.88	b 6.45	a 6.85	0.0056	0.01
FLAVOR Quality	a 6.72	a 6.81	b 6.14	a 6.74	0.0002	0.001
TEXTURE Quality	ab 6.6	a 6.75	b 6.2	a 6.62	0.0489	0.05
OVERALL Quality	a 6.65	a 6.78	b 5.95	a 6.68	0.0001	0.001

Table A-4: Summary of Mean-Scores, P-Values, and Significance for T=3 Weeks

Summary of Mean-Scores, P-Values, and Significance, Tukey's HSD (1-9 Sliding Quality Scale) Test Result Code - T09+26D						
This test was evaluated by 15 panelists.						
Attribute	Manufacturer pkg (MRE), T=3wks@100°F	NSRDEC pkg >~2wks (MRE overlay), T=3wks@100°F	NSRDEC pkg >~2wks (PE/Nylon-VT), T=3wks@100°F	NSRDEC pkg >~2wks (PE-OT), T=3wks@100°F	P-Value	Sig
APPEARANCE Quality	6.96	6.9	6.96	7.01	0.1866	NS
ODOR Quality	6.71	6.74	6.57	6.65	0.6131	NS
FLAVOR Quality	a 6.76	a 6.69	b 6.03	a 6.64	0.0001	0.001
TEXTURE Quality	a 6.79	a 6.76	b 5.76	a 6.75	0.0001	0.001
OVERALL Quality	a 6.77	a 6.72	b 5.77	a 6.64	0.0001	0.001

Table A-5: Summary of Mean-Scores, P-Values, and Significance for T=4 Weeks

Summary of Mean-Scores, P-Values, and Significance (Duncan's) Test Result Code - T09+26E						
This test was evaluated by 11 panelists.						
Attribute	**Manufacturer pkg (MRE), T=4wks@100°F**	**NSRDEC pkg >~2wks (MRE overlay), T=4wks@100°F**	**NSRDEC pkg >~2wks (PE/Nylon-VT), T=4wks@100°F**	**NSRDEC pkg >~2wks (PE-OT), T=4wks@100°F**	**P-Value**	**Sig**
APPEARANCE Quality	6.9	6.98	6.89	6.96		
ODOR Quality	abcde 6.62	abcdef 6.49	efg 6.21	abcd 6.7		
FLAVOR Quality	abc 6.72	abc 6.66	fg 5.44	abcd 6.54		
TEXTURE Quality	a 6.8	a 6.75	d 5.27	ab 6.55		
OVERALL Quality	ab 6.74	ab 6.67	d 5.55	ab 6.54		

Table A-6: Summary of Mean-Scores, P-Values, and Significance for T=5 Weeks

Summary of Mean-Scores, P-Values, and Significance (Duncan's) Test Result Code - T09+26F						
This test was evaluated by 12 panelists.						
Attribute	**Manufacturer pkg (MRE), T=5wks@100°F**	**NSRDEC pkg >~2wks (MRE overlay), T=5wks@100°F**	**NSRDEC pkg >~2wks (PE/Nylon-VT), T=5wks@100°F**	**NSRDEC pkg >~2wks (PE-OT), T=5wks@100°F**	**P-Value**	**Sig**
APPEARANCE Quality	6.87	6.78	6.85	6.78		
ODOR Quality	abcde 6.63	abcde 6.58	def 6.35	bcdef 6.43		
FLAVOR Quality	abc 6.73	abc 6.65	f 5.57	bcde 6.37		
TEXTURE Quality	a 6.83	ab 6.73	ef 4.78	ab 6.56		
OVERALL Quality	ab 6.77	ab 6.65	e 4.9	ab 6.48		

Table A-7: Summary of Mean-Scores, P-Values, and Significance for T=6 Weeks

Summary of Mean-Scores, P-Values, and Significance (Duncan's) Test Result Code - T09+26G						
This test was evaluated by 11 panelists.						
Attribute	**Manufacturer pkg (MRE), T=6wks@100^{o}F**	**NSRDEC pkg >~2wks (MRE overlay), T=6wks@100^{o}F**	**NSRDEC pkg >~2wks (PE/Nylon-VT), T=6wks@100^{o}F**	**NSRDEC pkg >~2wks (PE-OT), T=6wks@100^{o}F**	**P-Value**	**Sig**
APPEARANCE Quality	6.96	6.87	6.89	6.95		
ODOR Quality	abcd 6.71	abcde 6.61	efg 6.2	abcde 6.58		
FLAVOR Quality	a 6.85	abc 6.61	gh 5.11	cde 6.33		
TEXTURE Quality	a 6.84	ab 6.5	de 4.95	ab 6.38		
OVERALL Quality	ab 6.84	ab 6.49	e 4.89	bc 6.32		

Table A-8: Summary of Mean-Scores, P-Values, and Significance for T=8 Weeks

Summary of Mean-Scores, P-Values, and Significance (Duncan's) Test Result Code - T09+26H						
This test was evaluated by 15 panelists.						
Attribute	**Manufacturer pkg (MRE), T=8wks@100^{o}F**	**NSRDEC pkg >~2wks (MRE overlay), T=8wks@100^{o}F**	**NSRDEC pkg >~2wks (PE/Nylon-VT), T=8wks@100^{o}F**	**NSRDEC pkg >~2wks (PE-OT), T=8wks@100^{o}F**	**P-Value**	**Sig**
APPEARANCE Quality	6.94	7.01	6.59	7.01		
ODOR Quality	abcd 6.79	abcd 6.78	fg 6.13	abcd 6.7		
FLAVOR Quality	abc 6.73	a 6.89	fg 5.43	abc 6.61		
TEXTURE Quality	ab 6.7	a 6.86	ef 4.57	ab 6.67		
OVERALL Quality	ab 6.71	a 6.89	e 4.76	ab 6.62		

Table A-9: Summary of Mean-Scores, P-Values, and Significance for T=12 Weeks

Summary of Mean-Scores, P-Values, and Significance (Duncan's) Test Result Code - T09+26I						
This test was evaluated by 13 panelists.						
Attribute	Manufacturer pkg (MRE), T=12wks @100°F	NSRDEC pkg >~2wks (MRE overlay), T=12wks @100°F	NSRDEC pkg >~2wks (PE/Nylon-VT), T=12wks @100°F	NSRDEC pkg >~2wks (PE-OT), T=12wks @100°F	P-Value	Sig
APPEARANCE Quality	6.9	6.84	6.63	6.75		
ODOR Quality	abcd 6.68	abcde 6.6	g 5.83	abcde 6.55		
FLAVOR Quality	abc 6.72	abc 6.6	h 4.95	bcde 6.35		
TEXTURE Quality	a 6.76	ab 6.62	f 4.35	ab 6.45		
OVERALL Quality	ab 6.75	ab 6.58	e 4.62	abc 6.38		

Table A-10: Summary of Mean-Scores, P-Values, and Significance for T=16 Weeks

Summary of Mean-Scores, P-Values, and Significance (Duncan's) Test Result Code - T09+26J						
This test was evaluated by 12 panelists.						
Attribute	Manufacturer pkg (MRE), T=16wks @100°F	NSRDEC pkg >~2wks (MRE overlay), T=16wks @100°F	NSRDEC pkg >~2wks (PE/Nylon-VT), T=16wks @100°F	NSRDEC pkg >~2wks (PE-OT), T=16wks @100°F	P-Value	Sig
APPEARANCE Quality	6.91	6.83	6.7	6.9	0.0649	NS
ODOR Quality	a 6.5	a 6.64	b 5.35	a 6.63	0.0001	0.001
FLAVOR Quality	ab 6.48	a 6.77	c 4.84	b 6.08	0.0001	0.001
TEXTURE Quality	a 6.62	a 6.78	b 4.38	a 6.58	0.0001	0.001
OVERALL Quality	ab 6.55	a 6.75	c 4.49	b 6.2	0.0001	0.001

Table A-11: Summary of Mean-Scores, P-Values ,and Significance for T=24 Weeks

Summary of Mean-Scores, P-Values, and Significance (Duncan's)
Test Result Code - T09+26K

This test was evaluated by 13 panelists.

Attribute	Manufacturer pkg (MRE), T=24wks @100°F	NSRDEC pkg >~2wks (MRE overlay), T=24wks @100°F	NSRDEC pkg >~2wks (PE/Nylon-VT), T=24wks @100°F	NSRDEC pkg >~2wks (PE-OT), T=24wks @100°F	P-Value	Sig
APPEARANCE Quality	a 6.95	a 6.84	b 6.31	a 6.86	0.0042	0.01
ODOR Quality	a 6.52	a 6.67	b 5.35	a 6.65	0.0001	0.001
FLAVOR Quality	a 6.61	a 6.71	b 4.25	a 6.46	0.0001	0.001
TEXTURE Quality	a 6.75	a 6.63	b 3.65	a 6.67	0.0001	0.001
OVERALL Quality	a 6.63	a 6.7	b 3.84	a 6.54	0.0001	0.001

Table A-12: Summary of Mean-Scores, P-Values, and Significance for T=32 Weeks

Summary of Mean-Scores, P-Values, and Significance (Duncan's)
Test Result Code - T09+26L

This test was evaluated by 10 panelists.

Attribute	Manufacturer pkg (MRE), T=32wks @ 100°F	NSRDEC pkg >~2wks (MRE overlay), T=32wks @ 100°F	NSRDEC pkg >~2wks (PE/Nylon-VT), T=32wks @ 100°F	NSRDEC pkg >~2wks (PE-OT), T=32wks @100°F	P-Value	Sig
APPEARANCE Quality	a 6.82	a 6.78	b 5.6	a 6.63	0.0007	0.001
ODOR Quality	a 6.4	a 6.04	b 4.7	a 6.19	0.0001	0.001
FLAVOR Quality	a 6.48	a 6.38	b 4.41	a 5.92	0.0001	0.001
TEXTURE Quality	a 6.66	a 6.57	b 3.5	a 6.46	0.0001	0.001
OVERALL Quality	a 6.43	a 6.28	b 3.9	a 6.17	0.0001	0.001