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Characterization and Correlation Analysis of Pharmaceutical Gelatin

Pascal Georges Felix
University of South Florida

Characterization and Correlation Analysis of Pharmaceutical Gelatin

by

Pascal Georges Felix

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Department of Chemical Engineering
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University of South Florida

Major Professor: William E. Lee III, Ph.D.
Leo E. Ondrovic, Ph.D.
Rickey S. Shelley, Ph.D.

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ABSTRACT

The properties of the aged gel and subsequent softgels were examined using mechanical and chemical testing methods. Our hypothesis was that a negligible variation will exist between the aged gel of the same type. The greater difference is expected to be seen between the types of gels described as 150 Bloom (alkaline treated collagen) and 195 Bloom (acid treated collagen).

The types of gelatin used were the acid processed (195 Acid Bone) and alkaline processed (150 Lime Bone). Because of the differences expressed as the result of their manufacture sequence (namely their molecular weights), it follows that physical attributes will further contribute to their distinction. In addition to observing different characteristics between the types of gels, we aged the gelatin and produced softgel capsules to qualify and quantify the changes that occur as a function of time. Two production lots of over 1 million softgel capsules were executed to produce a population that lends itself to statistical analysis. Softgel capsules were manufactured with gelatin which was aged at intervals of 0-8 hrs, 32-40 hrs, 66-72 hrs and 88-96 hrs. The manufacturing process made use of this strategy for the acid and alkaline treated gelatin where a total of eight lots were made (4 acid and 4 alkaline). One hundred thousand softgels were manufactured for the acid processed gelatin, per lot. Additionally, one

hundred and fifty thousand softgels were manufactured for the alkaline processed gelatin per lot.

The results of the different tests provided trends that were not solely a function of time. Gel extensibility for both gel types showed a decrease in the amount of force needed to rupture the gelatin ribbon, as a function of time. The resilience of the tested ribbon remained constant throughout the aging process. The burst strength was the only test showing an inverse relationship between the two gel types. The amount of force needed to rupture the 150 Bloom softgels decrease in time whereas the amount of force needed to rupture the 195 Bloom softgels increase with time. The rheological testing was described in the literature as being associated with the molecular weight distribution. Such association was seen in our research and both the results of the rheological and the molecular weight tests decreased with the aging process.

CHAPTER 1.

INTRODUCTION

Background

The understanding of gelatin is important in the formation of capsules because gelatin-related factors are directly related to the absorption and bioavailability of a drug. Gelatin is defined as a mixture of water soluble proteins derived from collagen by hydrolysis. Gelatin is a protein, therefore it will behave like materials that are hydrolyzed by the majority of the proteolytic pathways to ultimately be degraded to amino acid levels (Singh et al., 2002). The protein fractions consist of all the known amino acids. These amino acids are joined by an amide linkage to form a linear polymer varying in molecular weight from 15K to 250K.

Type A and Type B gelatin are the two most common varieties of gelatin commercially available. Type A, as it relates to the gel used in this study, is manufactured by acidic hydrolysis of bovine bone and pork skin collagen (in this study we only used bone gelatin); the final product (softgels) displays relatively high plasticity and elasticity. Type B gelatin is manufactured by the hydrolysis of bones and bovine hide; the final product displays high gel strength. The qualitative attributes of the gelatin are not their only differentiating modalities. Type A gelatin has a pH between 3.8-6.0 and an isoelectric point between 6-8 whereas, Type B gelatin has a pH between 5.0-7.4 and an isoelectric point between 4.7-5.3. Table 1 summarizes the main differences between the two types of gelatin used in this study. One must note that the usage of these types of gelatin are not mutually exclusive, for gelatin used in the pharmaceutical manufacture of hardshell capsule blends are sometimes used, but not in the manufacture of softgels. However there are a few exceptions but for the most part, blends tend to be restricted to hardhsell capsules.

Table 1. Differences Between the Major Types of Gelatin.

Type A Gelatin (195 Bloom)	Type B Gelatin (150 Bloom)
Extracted by acidic hydrolysis of bovine bone, bovine hide and pork skin	Extracted by alkaline hydrolysis of bovine bones and hide
High gel strength and can resist cross-linking better than type B	High plasticity and elasticity (compared to acid pork skin)
pH ~ 3.8-6.0	pH ~ 5.0-7.4
Isoelectric point ~ 6-8	Isoelectric point ~ 4.7-5.3

Gelatin is normally characterized by its jelly strength or bloom strength. Bloom strength is defined as the weight in grams that, when applied with a 12.7 mm diameter plunger, will produce a depression exactly 4mm deep in a matured jelly containing 6.66% of gelatin in water at 10°C for 16-18 hours.

Gelatin is insoluble in acetone, chloroform, 95% ethanol, ether and methanol. It is soluble in glycerin, acids and bases. It is susceptible to swelling and softening in water, absorbing 5-10 times its own weight in water. Reactivity is experienced with acids, bases aldehydes, aldehydic sugars, anionic and cationic polymers, electrolytes, metal ions, plasticizers, preservatives and surfactants (Singh et al., 2002).

Cross-linking of gelatin before or after, the drying aspect of manufacturing of capsules, permits the gradual transmission of pharmaceutical agents throughout the gastrointestinal tract. No major pharmaceutical products use cross linking of gelatin in the United States due to the various difficulties associated with it. One of the difficulties with cross-linking as a formulation tool for softgels is the lack of reproducibility in the application of such technology. Gradual or time release of pharmaceutical agents allow for a more localized delivery along the gastrointestinal tract. The medical usage of gelatin is not limited to the encapsulation of drugs, but also includes applications in tissue engineering, bioadhesives, and plasma substitutes. It is rare that gelatin is not biocompatible. Biocompatibility can be defined as the ability of a material to function within an organism for a specific purpose. This ability will prevent the initiation of a cascade that may manifest a possible immune, toxic and necrotic responses. Under certain conditions, gelatin can be toxic and/or an irritant.

The increase in dissolution time (Q value) as a result of aging is one of the biggest problems associated with gelatin-based formulations. This lack of performance is due to the cross-linking of chemically altered gelatin. The cross-linking results in the appearance of a swollen, very thin, though, rubbery, water-insoluble membrane known as a pellicle. The pellicle is an important contributor in the reduction of softgels' dissolution. Once the pellicle is formed, it is not easily disrupted by gentle agitation and the dissolution values (Q value) have a tendency to drop to the point of rejection. The Q value is a United States Pharmacopeia sanctioned test that ascertains the dissolution and degradation limits of pharmaceuticals. The dissolution is a Quality Control test, which is used to show reproducibility of the softgels. Dissolution is used to show lot-to-lot reproducibility. Studies can be done to show in-vivo/in-vitro correlation but the results would not be indicative of the normal dissolution test. A softgel may have an acceptance criterion of not less than 80% dissolution after 60 minutes. The ranges for the Q value are from 0-100%, where the closer to 100% the test result is, the better the softgel ability to act as a drug delivery system. If the softgel should not meet the requirements of such test, it can be said that the values have dropped to the point of rejection.

During the manufacture of softgels, the shell has high water content. This water is removed from the shell by drying the softgels at low humidity conditions. After the manufacture of the softgels for hydrophilic fills, water can migrate from the shell to the fill. During drying, the water is removed from the shell to the atmosphere which reduces the amount of water in the shell and results in water migrating from the fill to the shell where the water continues to be lost to the atmosphere. If there are cross-linking agents in the fill these agents will migrate from the fill into the shell. When the softgel is stored at accelerated conditions during stability or stored at room temperature over a long period of time, the cross-linking agents will cause the softgel shell to become insoluble. During dissolution, the shell will not dissolve and a pellicle will be formed. This will reduce the percentage of drug released and can result in dissolution failure.

Moisture can move from the gelatin shell into the fill material during storage when hydrophilic ingredients have hygroscopic properties. If the environment where the capsules are stored is high in humidity, it is possible that the ambient water concentration increases the water concentration within the softgel. Figure 1 shows a softgel. The entire structure is the capsules and the casing (capsule without the fill material) is the shell.

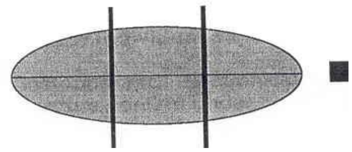


Figure 1. Diagram of a Softgel.

Even though dissolution testing can be used as a predictor of the bioavailibility of the drug, failing such test as a result of cross-linking of the gelatin and formation of pellicle does not necessarily mean that the drug will not be absorbed by the body. Because it has been shown that cross-linked gelatin with pellicle formation can be degraded by the gastrointestinal tract enzymes.

To the question of what causes the cross-linking of the gelatin, it has been proposed that it is gelatin's interaction with chemicals, humidity, temperature and light. An increase of these factors above what has been determined as acceptable will create unpredictable products. To prevent cross-linking of gelatin, one could use one or more of the following strategies: 1) usage of Type A gelatin; 2) reducing or preventing aldehydes formation and potential subsequent release; 3) use of inhibitors; 4) humidity control; and 5) photostabilization. The appearance and/or rate of formation of aldehydes are prevented by lysine, phenylamine, glutamine and other compounds. However, manipulation of pH will prevent aldehyde associated cross-linking. When inhibitors are used, it is to prevent changes in the dissolution rate of the softgels. Compounds that are used as inhibitors are semicarbazide hydrochloride, hydroxylamine hydrochloride, piperazide hydrate, pyridine, and others. An increase in ambient humidity of the softgel in some cases can increase crosslinking. Humidity control is achieved by using waterproof packaging. Photostabilization can be accomplished by adding color to the gelatin shell and selecting packaging to shield the softgel from destructive lighting. The incorporation of titanium oxide, iron oxide and color pigments may offer good protection against cross-linking resulting from light exposure (Singh et al., 2002). It is noteworthy to recognize that some of the strategies previously mentioned by Singh et al are not feasible for the formulation and manufacture of softgels.

In conclusion, the incorporation of gelatin in a manufacturing process should take into consideration the selection of the raw material. The introduction of inferior raw material will have a serious effect in the manufacturing operations downhill of such selection. Depending on the need, type A and/or type B gelatin will be the raw material used in the formulation of the softgel. These two types of gelatin not only have different sources but also have different innate characteristics as a result of their origins. It is these innate characteristics that give softgel manufacturing choices in their formulation. The gelatin portion of the softgel can interact with its environment but also with the active ingredient and exipients. The challenge for the manufacturer of softgels is to balance the aforementioned elements to produce a stable product with the ability to deliver a drug and/or vitamin to a desired GI tract location.

The manufacture of gelatin is not restricted to the delivery of pharmaceuticals. Gelatin is also involved in less critical time-release applications such as of cosmetics (bath oils) and recreational products (paintballs). Oils packaged in gelatin will be released once placed in water. This release resembles the behavior of gelatin within the human body whose degradation is also related to an increase in ambient temperature and solution. In the case of paintballs, the gelatin provides a good barrier against the degradation of the paint while yielding to a force that does not injure the player.

Research Objectives

When dealing with natural materials and their derivatives, one is forced to deal with their intricacies. Gelatin and its constituent collagen are prone to degradation which may be a function of time. From the moment collagen is synthesized, it undergoes cross-linking. The cross-linking which occurs as a result of aging adds a variable in the manufacturing of gelatin and subsequent products. In spite of the variability attributes of raw material and various sources of impurity, the manufacturing process oftentimes yields commodities with lesser variability than its constituents (Eastoe and Leach, 1977).

The overall objective of this research were: 1) to explore gel stability during the first 96 hours post-production; and 2) to demonstrate that the novel application of specific analytical methods provide useful information which can be used as a baseline for future research. Ultimately everything is prone to decay. However for the times delineated in this work, it is argued that the visible decay does not affect the finished product where it would be compromised. A secondary objective of this work is to determine if minimal or no changes occur in the finished product. Statistical analysis will be performed on the result of all test results to prove that there is no correlation between time and test results. The manufacturing of the softgels will be made using gel made between the times of 0-96 hours. It is hypothesized that there will be no appreciable difference between softgels produced between time 0 and those produced at 96 hours.

Research Questions

To demonstrate that equivalence exists between the types and different aged gelatin, answers to the following questions will be sought:

1) Are any differences seen in the test results statistically significant? Differences are expected in the different aged samples and between the types of gel used. However, it is proposed that these differences are negligible.

2) Are any observed differences satisfactorily discounted by statistical analysis? Again, differences are expected; however, mathematical justification will serve as a tool of the defense that the product made with the aged gelatin are similar.

3) Can the results of this research be generalized? By analyzing at the results of the characterization, a possible correlation between the types of gel and age at manufacture will be explored.

Importance of the Study

Many things are best understood when characterized at their basic level. This research will not only merge some of the knowledge of gelatin from the academic and corporate settings but also serve as a baseline for future research.

As means to analyze and clarify gelatin arise, it would be a great exercise to use these options to predict and explain the behavior of this material. This research made use of relatively new testing devices such as a rheometer and texture analyzer. These tests not only allowed the characterization of the different types of gelatin but also provided the trending of the results.

Limitations of the Study

As with most enterprises, constraints were placed upon this study. It was proposed that the investigation would include the use of electron microscopy. The lack of funding and time constraints did not allow for such ventures. The usage of an electron microscope would allow one to visualize changes at a level which could potentially explain or at least strengthen the theories associated with this research. In the event that this test was a possibility, it would have been impossible to test the samples due to the lack of people to prepare and execute microscopic determinations.

These visualization and other investigative tools could have become a reality if more funding was available. However, the practicality of such investment is challenged by the most important determinant in the business world, profit. It would not be good business to invest too much of one’s resources if there is no appreciable return on that investment. The accumulation of knowledge is not a tangible commodity and will always be placed as a secondary goal to many.

Corporate knowledge is not always communicated due to the laws associated with intellectual properties. The companies must protect themselves by recuperating their Research and Development investments. As a result of corporate policies, some information was excluded from this document. One noticeable exclusion is the compositional information of gel formulations used in this research.

CHAPTER 2.

LITERATURE REVIEW

An Overview of Collagen

Collagen is the predominant body protein found in of the skin, tendon, cartilage, bone and connective tissue. Its appearance within the organism is typically white, opaque, nonbranching fibrils integrated in mucopolysaccharide and other proteins. Collagen content will differ from organism to organism and consequently between species' populations. It is noteworthy to point out that the type of tissue and the age of the animal will also be a contributing factor in the collagen content.

The amino acid composition of collagen is surprisingly constant in the mammalian tissues. Glycine, the simplest amino acid accounts for approximately two-thirds of the collagen composition, proline and hydroxyproline for about one-fifth and alanine for roughly one ninth. Figure 2 gives a visual representation of common amino acids in collagen. The combined occurrence of the aforementioned amino acids are about two-thirds of the collagen composition. The common knowledge of the structure of collagen is mainly a product of X-ray diffraction and electron microscopy investigation (Balian and Bowes, 1977).

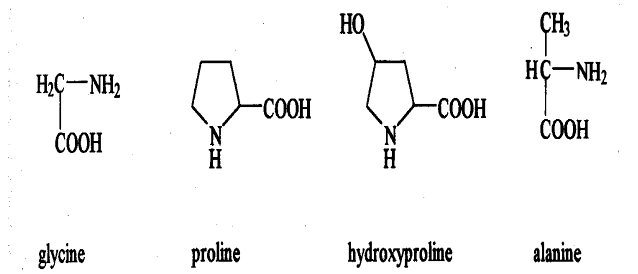


Figure 2. Chemical Structures of Main Amino Acids of Collagen.

The visualization of gelatin suggests that it is a rod-like molecule with the following dimensions; length of 2800 Å, diameter of 15 Å with a molecular weight of approximately 300,000 Daltons. The constituents of the helix are roughly the same length with a molecular weight of approximately 95,000 Daltons each (hence 3 chains make up the helix) (Balian and Bowes, 1977). Figure 3 gives a representation of the collagen helix.

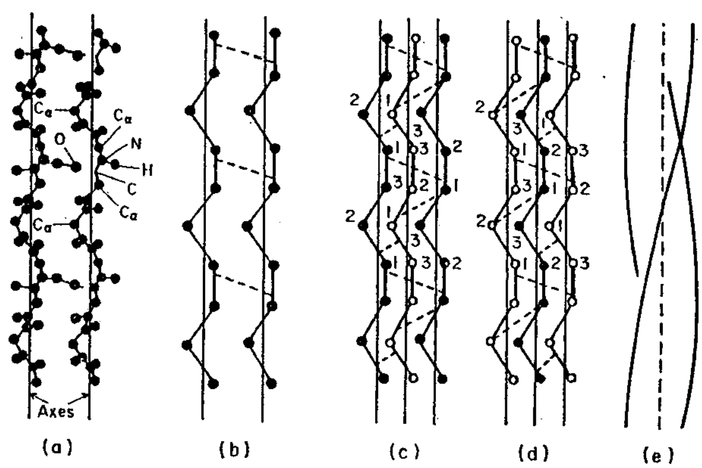


Figure 3. Triple Helix Visualizations of Collagen Constituents. a) Two polypeptide chains each helically wound with a left-handed three-fold screw axis. b) Simplified version. c) Third chain added behind. d) Third chain added in front. e) Twisting of the minor axis into the collagen super helix. (Balian and Bowes, 1977)

The stability seen in collagen is directly related to the native collagen's ability to resist the degradation of enzymes and chemicals. The attempt to degrade collagen at low pH values are negligible as long as the temperature is below 20 °C. Furthermore, the swelling and dispersing action of alkali is mitigated by the addition of a salt to stabilize the helix, by protecting the peptide bonds. A decrease in the viscous properties of collagen suggests denaturation which is the collapse of the rod like structure. The loss of physical properties is only one of the manifestation of the denaturation of collagen. The temperature where collagen losses its properties or is denatured varies between 37 °C and 40 °C.

There exists an inverse relationship between the viscous and elastic properties of collagen. Viscosity decreases as collagen approximated denaturation temperature (~40°C) and elasticity increases as collagen approaches the denaturation temperature. Elasticity is defined as the ability of a material to regain its shape after undergoing deformation due to a strain (Balian and Bowes, 1977).

An Overview of Gelatin

The gelatin manufacturing goal is to facilitate the conversion of collagen to gelatin and the removal of "impurities" (Johns, 1977). These impurities may be organic or inorganic in nature. Examples of organic impurities are proteins from blood, keratins, glycoproteins, mucopolysaccharides such as hyaluronic acid, keratosulphate and chondroitin sulphate, lipids, nucleic acids and other cell components which more than likely contribute to the soluble degradation of the gelatin. Inorganic contribution to degradation is made possible by the following ions; sodium, potassium, calcium, magnesium, iron, chloride and phosphate. Even though the mentioned impurities are associated with the organism before the extraction process, one should note that contamination may be an addition of the extraction process. Furthermore, one should pay heed to known changes in composition during the collagen-gelatin conversion (Eastoe and Leach, 1977). The majority of collagen is located in the corium layer. The corium layer is the preferred source of pure skin collagen therefore mechanical extraction concentrates on that area. The corium is freed of soluble components by mincing and prolonged interaction with various solutions followed by organic compound extraction to isolate fat and dehydrate the residue. The result of gelatin extraction is highly reproducible and the variance would be a function of the crosslinking associated with the age of the animal. When dealing with the gelatin process, the term tropocollagen describes acid soluble collagen (Johns, 1977).

Factors such as species, age of the animal and extraction techniques affect the quantities of soluble collagen during collagen manufacturing (Johns, 1977). Collagen is insoluble as the result of calcification which explains why mature bones contain little or no extractable collagen. The triple helix conformation is the result of hydrogen bonding. When collagen is insoluble as in the case of bones, it is often associated with the presence of covalent bonds (Johns, 1977).

The aforementioned complications in gelatin manufacturing may create variability in the end result (in our case, gelatin used to manufacture softgels). However, the intrinsic properties of gelatin make it a good candidate for the encapsulation of pharmaceutical, cosmetic and recreational products. The preferential usage of gelatin in certain applications is due to its “drug delivery vehicle” properties.

Gelatin Manufacturing

Two main manufacturing processes are used in the manufacturing of gelatin: alkaline and acid processes. Figure 4 outlines the gelatin manufacture process. The resulting products can be very different in terms of composition and physical properties. Generally, the alkali-processed gelatins possess higher hydroxyproline and lower tyrosine concentrations than acid-processed gelatins or the raw materials. The reasons for such discrepancies are: a) gelatins do not have the same purity ; and b) acid-processed materials are prone to losing peptides richer in hydroxyproline and maintaining peptides richer in tyrosine (and vise versa for the alkaline treated raw materials). In addition to these two processes, one could also extract gelatin by using the dual soak process (using alkaline and acid processes) and the autoclaving process.

Acid processed collagen is soaked in dilute acid and then extracted at about pH 4 for gelatin manufacturing. Non-collagenous proteins and mucoproteins of the tissue are isoelectric at this pH and are therefore less soluble and more readily coagulated under the extraction conditions. Contaminants which are removed in this way depend on the quality and origin of the raw material and the reproducibility of the manufacturers process (Eastoe and Leach, 1977).

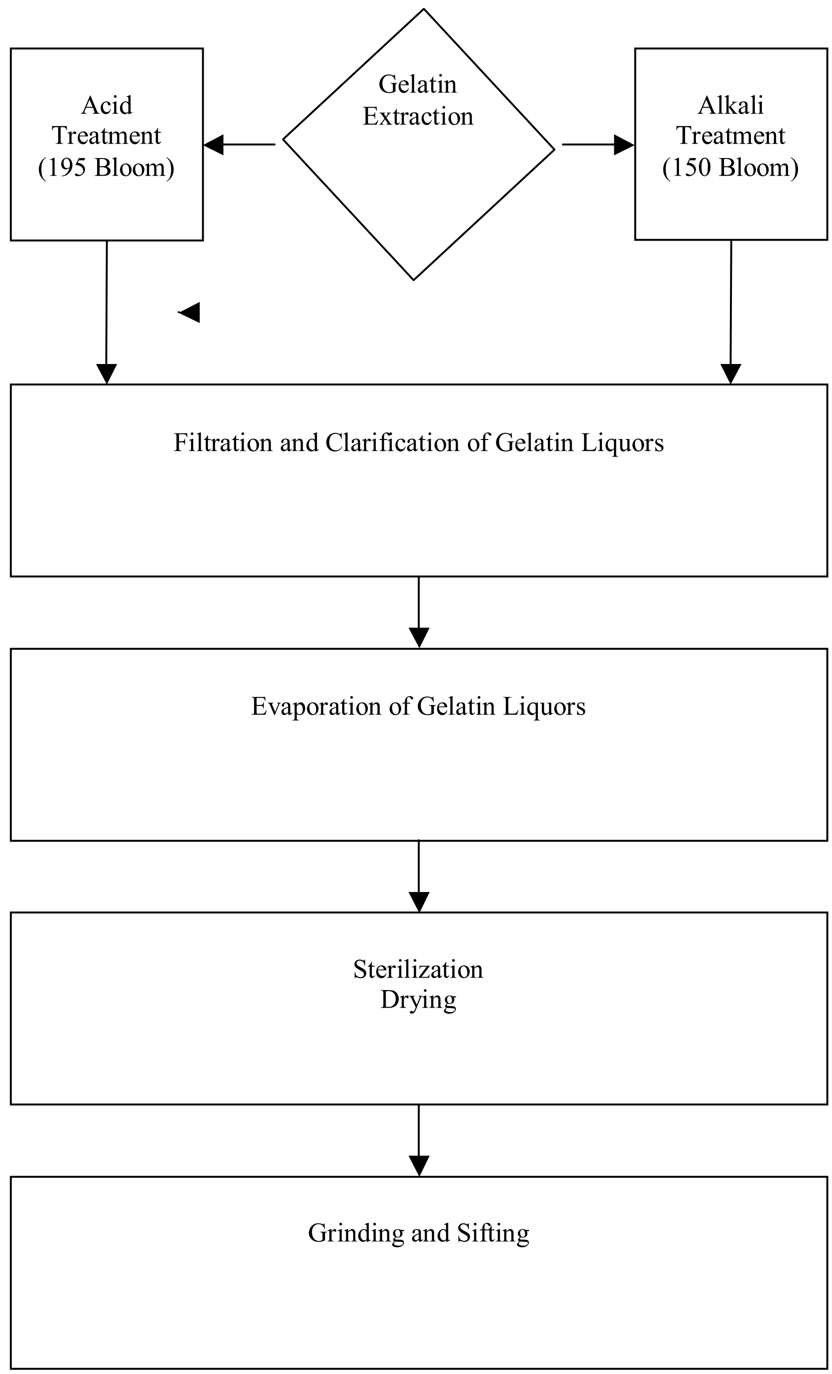


Figure 4. Alkali and Acid Treatment Processes for Gelatin.

When dealing with the alkaline process, the pretreatment of the collagen requires a prolonged soaking in the alkaline solutions (generally, saturated lime-water). A good amount of the impurities (proteins and mucosubstances) are soluble at the subjected pH and are extracted. Gelatins from alkali process tend to be purer than acid produced gelatin, but this variation may be due to the manufacturing process (Eastoe and Leach, 1977).

Additionally to the difficulties mentioned previously, the manufacture of gelatin is subject to more complications. For example the total number of carboxyl groups available for ionization depends upon the extraction method. Different gelatins can have different ratios of acidic and basic group therefore different isoelectric points. Charged groups influence the interactions between adjacent gelatin molecules, between each molecule and the solvent and between different parts of the same molecule, as the protein chains are flexible. The extent of these variables vary with pH, and are also dependent on the total ionic composition of the system therefore a detailed description of the solvent as well as that of the gelatin is a necessity (Stainsby, 1977).

Comparison Obtaining Gelatin from Collagen

Literature review offers no conclusive evidence of the correlation between viscosity and molecular weight for gelatins of different origins even if they have the same isoelectric point. Acid processed gels have lower viscosity than lime processed gels. Highly branched molecules in gelatin contribute to the difficulties in determining the correlation between physical properties to molecular arrangement. The branching also makes it hard to explain solubility (Stainsby, 1977). However, the relationship between viscosity and molecular weight exists over a wide range (Stainsby, 1977).

Variables exist in the industrial gelatin processes which impact the uniformity of the end product. A source for the variations is the impossibility to extract gelatin from collagen without incorporating “impurities”. The impurities may contribute to the accelerated decay of the gelatin. However, one should note that many advances and improvements have been made in gelatin manufacture, making impurities less of an issue than during the early days of gelatin manufacturing. Amidst the contributions to the variances, one should note that the way to manufacture gelatin is to denature soluble collagen. The process destroys the triple helix to produce one, two or three random chain gelatin molecules. The molecular change is catalyzed by mild conditions either by heating at neutral pH to about 40˚C or by adding hydrolysis promoters at room temperature or lower. The process has the objective of breaking the hydrogen bonds and hydrophobic bonds that stabilize the collagen helix. These process conditions prevent destruction of covalent bonds before their time, within the collagen chain (Johns and Courts, 1977).

The formation of gelatin is related to the denaturation of soluble collagen. The transition can not simply be explained in terms of the uncoiling of the helixes, and consideration must be given to the molecular association. When the soluble collagen is exposed to denaturation conditions , such as elevated temperatures or in an hydrogen bond breaker environment, the molecular substituents are dissociated. This dissociation is represented by random chain molecules. Upon the reversal of the denaturation conditions, the molecular chains aggregate to achieve collagen’s native state of a triple-helix (Johns and Courts, 1977). Figure 5 provides a description of the associated and dissociated aspects of collagen.

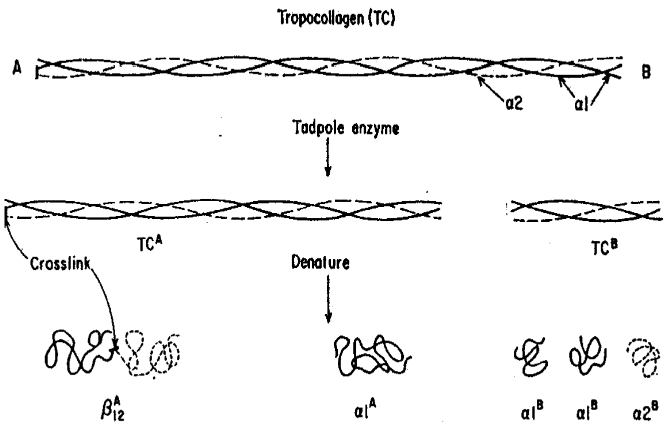


Figure 5. Products of the Dissociation of the Triple Helix (Balian and Bowes, 1977).

Gelatin may be considered as a blend of fine and coarse networks whose proportions depend largely on the thermal history of the gel. To achieve the formation of gel there must be two bonding sites per gelatin molecule and this necessity can be assessed by molecular weight. The approximation of molecules by their sites of attachment increase molecular weight. It has been suggested that roughly 1/6 of the α-chain is needed for such bonding. If values greater than the mentioned numbers are achieved, the quality of the gelatin will increase and plateau around the molecular weight of around 90 K. However, one should note that the correlation between molecular weight and gelatin quality is the determinant for the quality of the product.

The inherent characteristics of gelatin and its constituent collagen make them choice materials in pharmaceutical preparations. In our application of gelatin, this biomaterial is used to contain pharmaceutical ingredients and serve as a drug delivery vehicle. However, gelatin can also be used as a binding agent in tablets where its properties are also exploited.

CHAPTER 3.

RESEARCH DESIGN AND METHODS

Four analytical methods were used in this investigation to measure mechanical properties. Two of the methods measured compression forces, the third method measured tension force, and the fourth recorded viscoelastic properties. Two instruments were principally used in the testing of the gelatin during this exercise. The first instrument used to test mechanical attributes was a texture analyzer whose function was to apply compressive and tension forces on the samples of interest. The second instrument was a rheometer which principal use was the determination of the viscoelastic properties of the samples.

Gel Extensibility

Gel Extensibility is a destructive test that punches a hole into a gelatin ribbon. Sample of the material of interest is cut in a rectangular shape with an area of approximately 2.5 X 5 inches with a typical thickness approximating 0.010 inches. The gelatin ribbon was extracted from an encapsulation machine similar to that illustrated in Figure 6. The ribbon was consistently gathered from the "pre-wedge" area, meaning that the ribbon was collected before it was to be shaped and made to contain fill material.

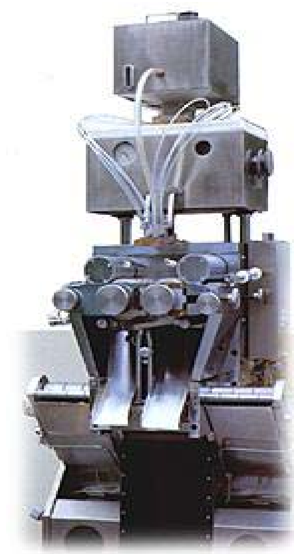


Figure 6. Encapsulation Machine.

The texture analyzer was manufactured by Stable Micro Systems in Surrey, England. The model used was the TA.XT2 (HR) and was attached to a computer terminal to allow real time data collection (see Figure 7). A robotic arm is activated and a plunger proceeds towards the sample. When testing the gelatin ribbon, an attachment resembling a softgel was used for the extensibility test, with a length and width of 2 cm and 1 cm, respectively. During the extensibility test, the softgel attachment will pierce the ribbon and the compressive force necessary to achieve the destruction was recorded. The results of the Gel Extensibility are culminated in a printout as seen in Figure 6. The printout is a combination of the analysis of ten (10) trials for the same sample, which quantifies the amount of force (in grams) needed to punch a hole through the ribbon. The other types of data that can be extracted from the printout are the time needed to punch the hole and the distance traveled before the puncture (in millimeter). The variability seen in the results can be due to the raw material variability, the intricacies associated with preparing a highly viscous liquid, and the variability of the different samples subjected to destructive testing.

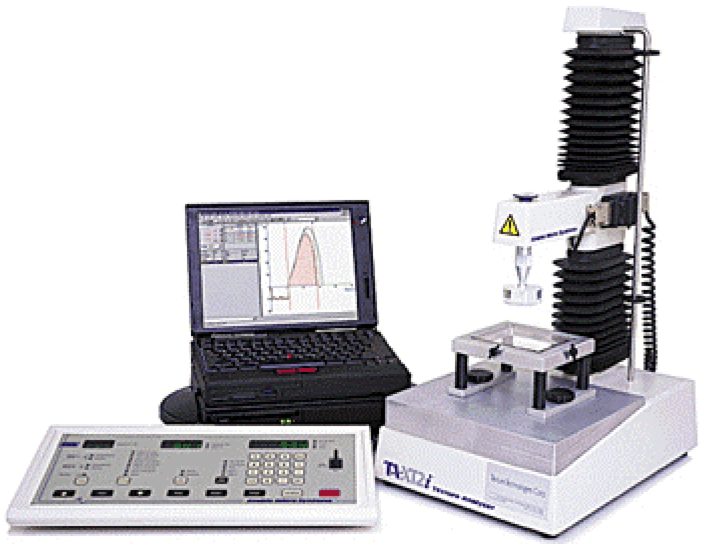
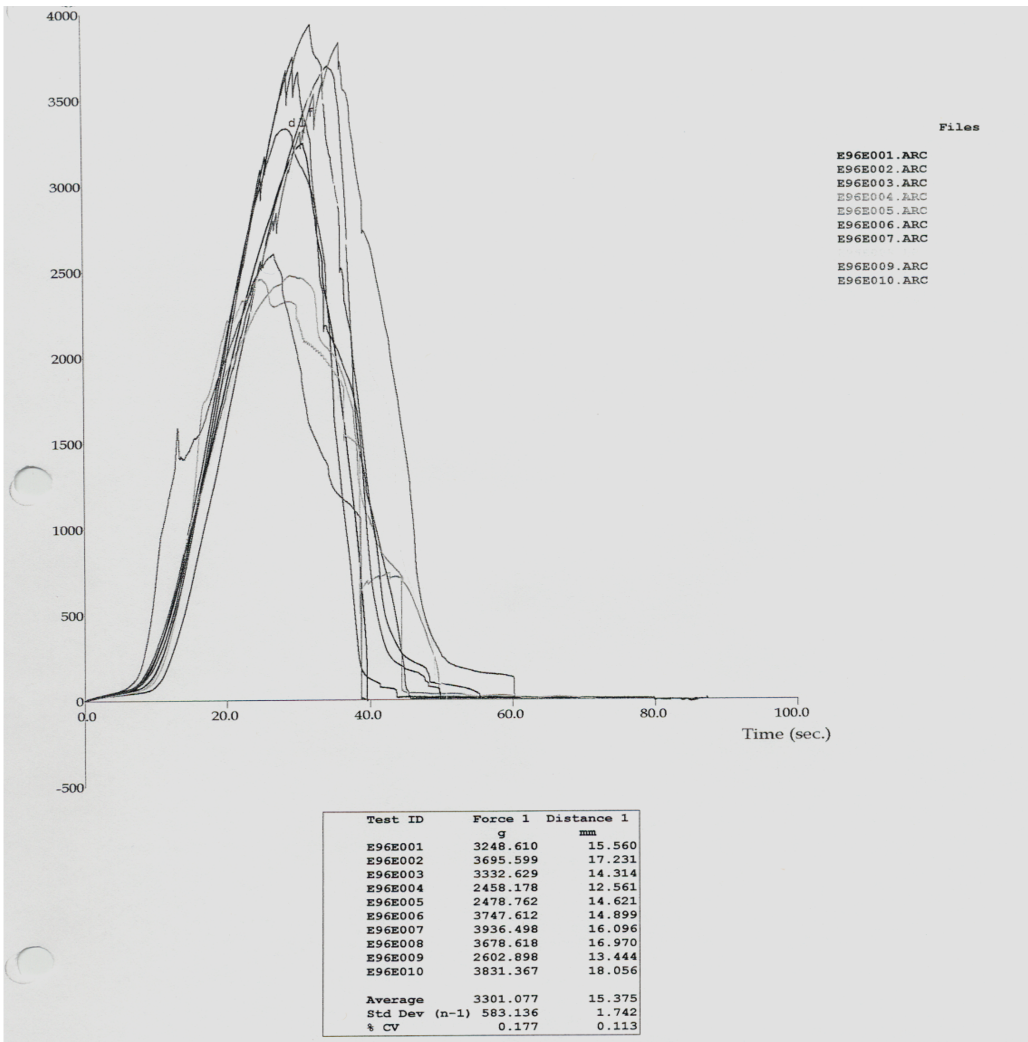


Figure 7. TA.XTR Texture Analyzer.

When dealing with gelatin of a particular molecular weight, the ascribed value is an average and as a result of variability in the main component of gelatin, variability will also be seen in the final product. Even though great care was taken to avoid degradation of the gelatin ribbon between testing, it is possible that the ribbon lost water and in turn plasticity since water is a natural plasticizer. Another precautionary step taken to prevent unnecessary variability in the testing methods was to calibrate the texture analyzer using a ten pound weight as per manufacturer's recommended calibration procedure. The weight was placed where the triangle points on the mechanical arm of the Texture Analyzer in Figure 7. Once the weight was in place, the calibration mode of the software was selected. The calibration was done for everyday of testing and was applicable for the other tests using the texture analyzer (i.e. resilience and wet burst strength).

Resilience

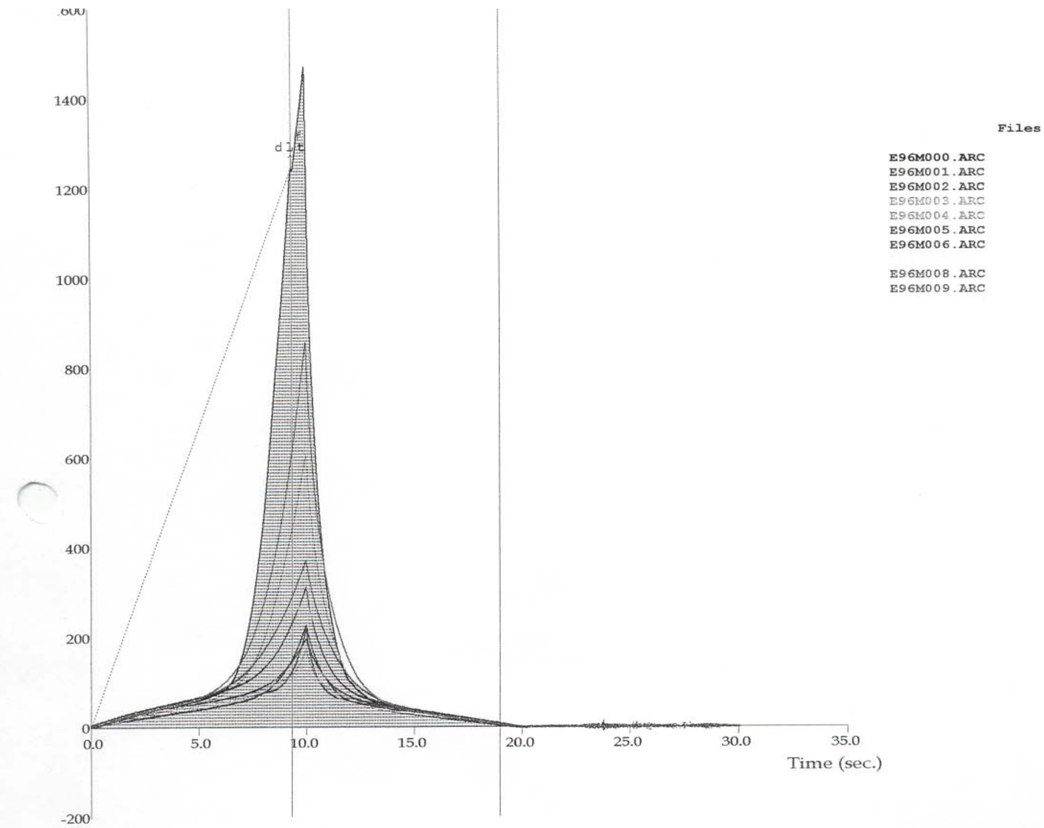
The same sample setup for Gel Extensibility was used for the resilience test but the softgel attachment was not allowed to destroy the sample and the test used tension forces. The goal of the resilience test was to ascertain the absorbed energy required to displace the ribbon at a predetermined distance of 5 mm. Both tests pressed through a ribbon, which is harnessed to a stage to insure stability and uniformity.



Test ID	Force 1 g	Distance 1 mm
E96E001	3248.610	15.560
E96E002	3695.599	17.231
E96E003	3332.629	14.314
E96E004	2458.178	12.561
E96E005	2478.762	14.621
E96E006	3747.612	14.899
E96E007	3936.498	16.096
E96E008	3678.618	16.970
E96E009	2602.898	13.444
E96E010	3831.367	18.056
Average	3301.077	15.375
Std Dev (n-1)	583.136	1.742
% CV	0.177	0.113

Figure 8. An Example of a Gel Extensibility Test Printout.

Resilience testing also makes use of ten (10) trials per sample. The similarity of these two tests does not stop with the resemblance of the printouts, but also include the fact that they use the exact setup but different applications. The Gel Extensibility test is made using compression forces whereas the Resilience test is made using tension forces. Figure 8 shows the printout for the Gel Extensibility test and Figure 9 shows the printout for the Resilience test. The resilience printout provides more information than what was needed for this study. We were interested in the resilience aspect of the printout as the title of our test suggested. Similar to the printout of the gel extensibility test, the force is calculated in grams. Also, the force used can be correlated to time and distance traveled in mm. The calibration was done for everyday of testing following the method mentioned in the Gel Extensibility section (page 22).



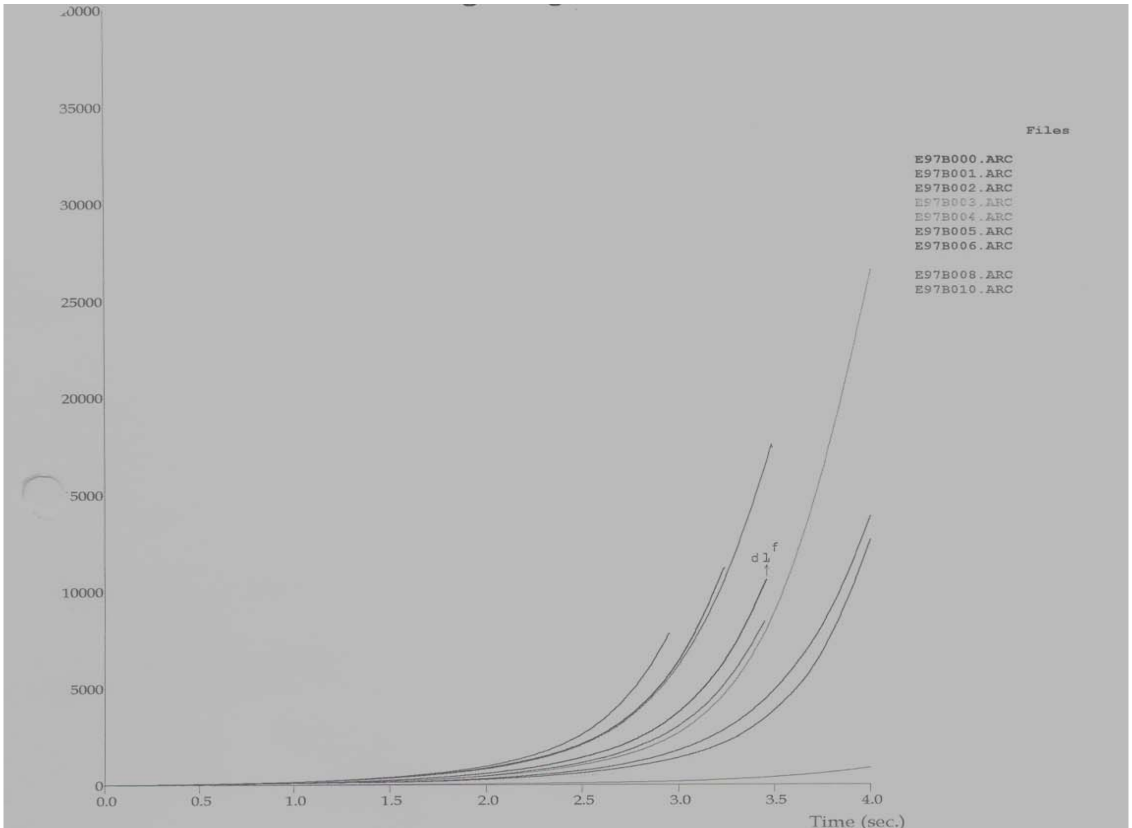
Test ID	Firmness	Area	Stifness	Area	Resilience	Distance1	Distance2	Springiness	Time 1
	g	g s	g/s	g s	%	mm	mm	%	s
E96M000	1244.828	1726.003	133.141	1929.757	111.805	4.677	4.813	102.908	9.345
E96M001	227.107	534.946	22.652	421.600	78.812	5.000	4.716	94.320	10.000
E96M002	219.923	564.512	21.894	411.697	72.930	5.000	4.603	92.060	10.000
E96M003	33.353	41.460	11.879	942.792	2273.980	1.373	8.242	600.296	2.745
E96M004	609.245	1189.377	60.794	836.874	70.362	5.000	4.830	96.600	9.995
E96M005	196.902	615.380	19.483	484.543	78.739	4.988	4.797	96.171	9.970
E96M006	312.432	869.501	30.977	585.171	67.300	5.000	4.736	94.720	9.995
E96M007	35.682	37.439	15.528	1115.959	2980.739	1.050	8.530	812.388	2.100
E96M008	371.637	992.221	37.053	695.623	70.108	5.000	4.517	90.340	10.005
E96M009	858.106	1605.089	85.658	1039.597	64.769	5.000	4.629	92.580	9.995
Average	410.922	817.593	43.906	846.361	586.954	4.209	5.441	217.238	8.415
Std Dev (n-1)	386.636	578.564	38.829	457.574	1088.294	1.585	1.557	262.605	3.168
% CV	0.941	0.708	0.884	0.541	1.854	0.377	0.286	1.209	0.377

Figure 9. An Example of a Resilience Test Printout.

Wet Burst Strength

Unlike the previously mentioned tests, the Wet Burst Strength test measures a parameter of the final product. The wet burst strength test involved taking a newly formed softgel and applying a compressive test until rupture of that softgel. Unlike the previously cited texture analyzer test procedures, the attachment was a disk with a 25 mm diameter which allowed for a uniform distribution of force. The softgel is placed with its seam parallel to the floor, to assure uniformity in testing. Figure 10 shows a printout of such test.

The wet burst strength printout gives the distance traveled before the softgel ruptured and the amount of force needed to achieve such failure. As with the gel extensibility and resilience tests, ten trials for the same sample location (beginning, middle and end of the encapsulation runs) were used to provide statistically significant results. The calibration was done for everyday of testing following the method mentioned in the Gel Extensibility section (page 22).



Test ID	Force 1	Distance 1
	g	mm
E97B000	10569.848	6.914
E97B001	12633.872	7.997
E97B002	11170.496	6.473
E97B003	874.082	7.995
E97B004	26574.520	7.998
E97B005	7820.502	5.906
E97B006	13854.972	7.999
E97B007	7086.323	7.839
E97B008	8392.510	6.896
E97B010	17535.484	6.970
Average	11651.261	7.299
Std Dev (n-1)	6886.626	0.766
% CV	0.591	0.105

Figure 10. An Example of a Wet Burst Strength Test Printout.

Rheometer

The Haake Rheostress rheometer (model RS150) was a German made instrument whose function was to melt the gel mass samples and during that process determine the viscous and elastic regions of the melting gelatin (see figure 11). The sample was situated between sensors and a heat transfer system will bring the temperature to 60° C and then drop the temperature to 40° C. The temperature range was established in consideration of the manufacturing parameters and gelatin tolerance. As with the texture analyzer, the probe was attached to a computer to allow the capture of real time data.

Unlike the previous tests, we used one sample from the different points of interest and looked at the viscoelastic behavior as a function of temperature. The sample was tested once. Figure 12 shows the printout of the rheometer test where four of the columns were of interest which were: time in seconds; G' which represents the elastic aspect of the material; G'' which represent the viscous aspect of the material; and the complex viscosity of the material which is measured in Pascal were its G' and G'' constituents. An outside firm calibrates the instrument annually. However, before the beginning of the experiment the instrument was verified using the Brookfield Viscosity standard 5040 cps (lot# 051401). The verification sequence involves placing the standard on the instrument's plates and taking the reading. In the event the reading does not correlate to the standard (5040 cps), the instrument would have been recalibrated.

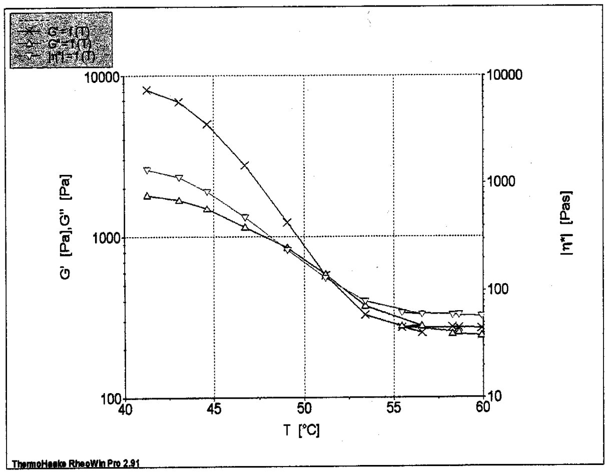
Figure 11. The Haake Rheostress Research Grade Rheometer. All tests in this research made use of the plate to plate combination.

RheoWin Pro 2.91	Test 1	Page 1

Company	RP Scherer NA	**Measure device**	RS150		
Operator	RP Scherer NA	**Temperature device**	TC81 <---> RS150		
Date/Time	19.09.2002 / 17:22:57 PM	**Sensor**	PP35 Ti	**Gap**	3.629 mm
Substance	E21995 Mid	**A-factor**	128800.000 Pa/Nm		
Sample no		**M-factor**	4.822 (1/s)/(rad/s)		
Description					

Comment

Moduli for gel free IS 430



Element definition / Notes

CD lin, 40.00 °C - 60.00 °C, t 300.00 s, 0.05 -, f 1.000 H

Filename: -----

Job: C:\Program Files\RheoWin\Jobs\Modulus Test.rwj

	t [s]	t_seg [s]	T [°C]	G' [Pa]	G" [Pa]	|η*| [Pas]
1-1	18.67	11.36	41.29	8186	1804	1334
1-2	39.95	32.63	43.05	6846	1674	1122
1-3	66.39	59.08	44.64	4999	1483	830.0
1-4	97.25	89.94	46.72	2774	1142	477.4
1-5	132.6	125.3	49.07	1221	851.0	236.9
1-6	165.8	158.4	51.20	572.9	583.6	130.2
1-7	197.0	189.7	53.40	325.4	370.7	78.50
1-8	223.9	216.6	56.58	251.1	278.2	59.65
1-9	245.2	237.9	55.45	272.2	276.5	61.75
1-10	268.2	260.9	58.64	271.2	253.8	59.12
1-11	291.2	283.9	58.27	274.3	247.6	58.80
1-12	312.5	305.1	59.91	269.7	242.1	57.69

Evaluation

Crossover : G' = G" = 605.1 Pa at ω = 6.283 rad/s f =

Figure 12. An Example of Rheometer Testing Printout.

HPLC (Size Exclusion Chromatography)

The HPLC method was also employed to provide additional information. This method is based on size exclusion chromatography where a column was filled with a material having fixed filtration capacity. The sample is allowed to go through the column and is ultimately segregated by molecular size. Large molecules go through the column relatively fast and the smaller molecules adhere to the material in the column and are dissociated later. Such technique is also known as gel filtration or gel permeation chromatography. Three samples were taken from the points of interest (beginning, middle and end of encapsulation run) and subjected to the HPLC method.

This testing portion of the study was performed by the gel supplier who also calibrated the instrument. The HPLC method used in this experiment was qualitative. The goal of this method was to obtain a fingerprint of the gel. The molecular weight data are relative and should only be compared if samples are run in sequence. It is not possible to use such method quantitatively due to the heterogeneity of the gelatin, variables within the method itself such as column-to-column variability, and column variability due to age. Due to the qualitative nature of the HPLC test, accuracy values can not be given.

Experimental Design

The goal of this research was to test two types of gelatin (150 Bloom Lime Processed and 195 Bloom Acid Processed) using the five methods previously mentioned. Table 1 summarizes the experimental design.

Table 2. Summary of the Experimental Design.

Gel Types / Test Methods	150 Bloom	195 Bloom
Gel Extensibility	Samples were tested at four time intervals[1]. Ten times per sample points.	Samples were tested at four time intervals[1]. Ten times per sample points.
Resilience	Samples were tested at four time intervals[1]. Ten times per sample points.	Samples were tested at four time intervals[1]. Ten times per sample points..
Wet Burst Strength	Samples were tested at four time intervals[1]. Ten times per sample points.	Samples were tested at four time intervals[1]. Ten times per sample points.
Rheology	Samples were tested at four time intervals[1]. One time per sample points.	Samples were tested at four time intervals[1]. One time per sample points.
HPLC	Samples were tested at four time intervals[1]. Three times per sample points.	Samples were tested at four time intervals[1]. Three times per sample points.

[1] Time intervals: 0,4,8; 32,36,40; 64,68,72; 88, 92, 96 hours.

CHAPTER 4.

RESULTS AND DISCUSSION

Results

The results of the different tests are summarized in Table 3, where the standard deviation and sample size are also given. The reason for the variability in sample size was due to the omission of data. This omission was due to occasional technical difficulties with the testing apparatus. On many occasions, the piston refused to progress and simply came back to its initial position when it sensed the sample.

The raw data relating to the test methods is presented in Appendices 1 through 5. Appendix 1 contains Microsoft Excel graphical representation of test results. Appendix 2 contains printouts of the Wet Burst Strength test results. Appendix 3 has the printouts of the Rheology test results. Appendix 4 is the location of the Gel Extensibility test results. Appendix 5 contains the Resilience test results and Appendix 6 contains the Molecular Weight analysis .

When looking at the data for all test parameters, significant differences are observed between the 150 gelatin and 195 gelatin mean values for a given time interval. When regressed as a function of time, significant differences between gel types are also observed.

The 195 Acid Processed gelatin ribbon had stronger mechanical properties that the 150 Lime Processed gelatin ribbon based on the Gel Extensibility test. The deformation seen in the Resilience testing of the 150 Lime Processed gelatin type hovered around the value of 54%, meaning that the gelatin was deformed on an average value of 54% from time 0 to time 96 hrs. The deformation of the 195 Acid Processed gelatin was statistically greater than its counterpart, displaying an average value of about 75%.

The Burst Strength results resembled the Gel Extensibility trends, where the 150 Lime Processed gelatin had smaller values than the 195 Acid Processed gelatin. The viscoelastic aspect of the rheological testing shows that the 150 Lime Processed gelatin has lower values than the 195 Acid Processed gelatin. However, the 150 gelatin type had a greater molecular weight distribution than the 195 gelatin.

When looking at the test results as a function of time, the following were observed, 1) There was a decline in the amount of force needed to break the 150 and 195 gelatin ribbons in the Gel Extensibility test; 2) The Resilience for the two gelatin types were constant as a function of time; 3) The trend seen in the Gel Extensibility were also seen in the Burst Strength results, in that it took less force to rupture the softgels as a function of time; 4) The viscoelastic test shows decrease in values as a function of time; 5) The molecular weight distribution showed a steady decrease for the two types of gelatin.

Table 4 shows a general decrease as a function of time for Gel Extensibility, however some increases can be seen. The first two values (times 0 and 4) for the 150 Gelatin type were omitted due to instrument malfunction. Table 5 shows uniformity of the test results for Resilience. The first two results were omitted for the two gelatin types and the third result was omitted for the 195 gelatin types. Again, instrument malfunction was the cause of such absence.

Table 3. Summary of Experimental Results for the Various Methods.[1]

Type of Test And Corresponding Times of Execution	150 Gelatin	195 Gelatin	Are the means significantly different ?[2]
Gel Extensibility (mm) 0-8 hours	2266 ± 18 (n=10)	3310 ± 131 (n=30)	Yes
32-40 hours	1829 ± 273 (n=30)	2741 ± 485 (n=30)	Yes
66-72 hours	1277 ± 103 (n=30)	2759 ± 123 (n=30)	Yes
88-96 hours	1202 ± 161 (n=30)	2582 ± 177 (n=30)	Yes
Resilience (mm) 0-8 hours	54 ± 3 (n=8)	N/A	Yes
32-40 hours	54 ± 2 (n=29)	75 ± 2 (n=22)	Yes
66-72 hours	55 ± 1 (n=23)	75 ± 0.18 (n=24)	Yes
88-96 hours	54 ± 1 (n=29)	76 ± 1 (n=20)	Yes
Burst Strength (mm) 0-8 hours	12,312 ± 2715 (n=30)	13,672 ± 8 (n=12)	Yes
32-40 hours	10,330 ± 1687 (n=30)	11,450 ± 7 (n=30)	Yes
66-72 hours	10,245 ± 432 (n=30)	12,049 ± 562 (n=20)	Yes
88-96 hours	7195 ± 1323 (n=30)	14,022 ± 2239 (n=30)	Yes
Rheological Test (Pa) 0-8 hours	41 ± 32 (n=14)	186 ± 148 (n=20)	Yes
32-40 hours	108 ± 80 (n=28)	362 ± 417 (n=19)	Yes
66-72 hours	17 ± 12 (n=22)	256 ± 135 (n=30)	Yes
88-96 hours	53 ± 9 (n=30)	241 ± 101 (n=29)	Yes
Molecular Weight (Da) 0-8 hours	128,602 ± 2397 (n=9)	98,566 ± 2499 (n=9)	No
32-40 hours	119,822 ± 2260 (n=9)	90,943 ± 702 (n=9)	No
66-72 hours	112,595 ± 3394 (n=9)	82,046 ± 797 (n=9)	No
88-96 hours	106,535 ± 3564 (n=9)	81,262 ± 865 (n=9)	No

[1] Values shown are mean ± standard deviation , followed by number of observations in parenthesis.

[2] t-test using $p \leq 0.05$

Table 4. Summary of Experimental Results for the Gel Extensibility Method.[1]

Gel Extensibility And Corresponding Times of Execution	150 Gelatin (mm)	195 Gelatin (mm)
0-8 hrs Beginning of lot (Beg) Middle of lot (Mid) End of lot (End)	N/A N/A 2265 ± 959 (n=10)	3440 ± 1697 (n=10) 3311 ± 615 (n=10) 3179 ± 726 (n=10)
32-40 hrs Beg Mid End	2143 ± 520 (n=10) 1691 ± 294 (n=10) 1654 ± 517 (n=10)	2457 ± 628 (n=10) 2463 ± 719 (n=10) 3301 ± 583 (n=10)
66-72 hrs Beg Mid End	1180 ± 565 (n=10) 1267 ± 359 (n=10) 1384 ± 585 (n=10)	2826 ± 528 (n=10) 2617 ± 380 (n=10) 2834 ± 516 (n=10)
88-96 hrs Beg Mid End	1069 ± 301 (n=10) 1156 ± 216 (n=10) 1381 ± 544 (n=10)	2377 ± 674 (n=10) 2683 ± 354 (n=10) 2685 ± 320 (n=10)

[1] Values shown are mean ± standard deviation, followed by number of observations in parenthesis.

Table 6 shows a general decrease in the Wet Burst Strength results for both gelatin types. Unlike the previous tables, the technical difficulties were experienced during the testing as opposed to the beginning. Time 4, 8, 36, 40 and 72 were omitted as the results of the aforementioned difficulties. A divergence can be seen at the end of the test time (96th hour), where the 195 Gelatin value increases and the 150 Gelatin value decreases.

Table 5. Summary of Experimental Results for the Resilience Method.[1]

Resilience And Corresponding Times of Execution	150 Gelatin (%)	195 Gelatin (%)
0-8 hrs Beginning of lot (Beg)	N/A	N/A
Middle of lot (Mid)	N/A	N/A
End of lot (End)	54 ± 3 (n=8)	N/A
32-40 hrs Beg	56 ± 2 (n=10)	73 ± 5 (n=8)
Mid	52 ± 4 (n=10)	77 ± 15 (n=8)
End	54 ± 2 (n=9)	77 ± 3 (n=6)
66-72 hrs Beg	53 ± 3 (n=4)	75 ± 3 (n=9)
Mid	56 ± 5 (n=9)	75 ± 4 (n=8)
End	54 ± 4 (n=10)	75 ± 2 (n=7)
88-96 hrs Beg	55 ± 4 (n=10)	75 ± 3 (n=7)
Mid	52 ± 4 (n=9)	77 ± 1 (n=6)
End	53 ± 3 (n=10)	76 ± 3 (n=7)

[1] Values shown are mean ± standard deviation , followed by number of observations in parenthesis. Test results are a percentage of the sustained deformation.

Table 6. Summary of Experimental Results for the Wet Burst Strength Method.[1]

Wet Burst Strength And Corresponding Times of Execution	150 Gelatin (mm)	195 Gelatin (mm)
0-8 hrs Beginning of lot (Beg)	11,368 ± 2760 (n=10)	13,672 ± 9551 (n=12)
Middle of lot (Mid)	15,374 ± 3554 (n=10)	N/A
End of lot (End)	10,195 ± 4269 (n=10)	N/A
32-40 hrs Beg	8460 ± 2992 (n=10)	11,450 ± 7763 (n=30)
Mid	11,739 ± 3600 (n=10)	N/A
End	10,790 ± 1892 (n=10)	N/A
66-72 hrs Beg	10,684 ± 3208 (n=10)	11,651 ± 6887 (n=10)
Mid	9820 ± 4010 (n=10)	12,446 ± 9543 (n=10)
End	10,230 ± 3939 (n=10)	N/A
88-96 hrs Beg	8677 ± 2458 (n=10)	16,552 ± 6990 (n=10)
Mid	12999 ± 9726 (n=10)	13,220 ± 5159 (n=10)
End	6780 ± 2730 (n=10)	12,294 ± 3439 (n=10)

[1] Values shown are mean ± standard deviation , followed by number of observations in parenthesis.

Table 7 does not show a particular trend in the viscoelastic properties of the two gelatin types. The first result for the 150 Gelatin type was omitted due to technical difficulties. Table 8 shows a strong relationship between the molecular weight distribution and time.

Table 7. Summary of Experimental Results for the Rheology Method.[1]

Rheological Testing And Corresponding Times of Execution	150 Gelatin (Pa)	195 Gelatin (Pa)
0-8 hrs Beginning of lot (Beg)	N/A	345 ± 46 (n=9)
Middle of lot (Mid)	76 ± 6 (n=6)	63 ± 8 (n=6)
End of lot (End)	14 ± 2 (n=8)	48 ± 3 (n=5)
32-40 hrs Beg	209 ± 19 (n=10)	65 ± 10 (n=7)
Mid	77 ± 9 (n=11)	1041 ± 73 (n=5)
End	14 ± 2 (n=7)	138 ± 27 (n=7)
66-72 hrs Beg	7 ± 1 (n=5)	57 ± 12 (n=9)
Mid	9 ± 1 (n=9)	317 ± 59 (n=11)
End	33 ± 5 (n=8)	369 ± 41 (n=10)
88-96 hrs Beg	62 ± 13 (n=11)	414 ± 15 (n=6)
Mid	42 ± 7 (n=11)	245 ± 40 (n=12)
End	57 ± 8 (n=8)	142 ± 34 (n=11)

[1] Values shown are mean ± standard deviation , followed by number of observations in parenthesis. Test results are in Pascals.

Table 8. Summary of Experimental Results for the Molecular Weight Analysis Method.[1]

Molecular Weight Analysis And Corresponding Times of Execution	150 Gelatin (Da)	195 Gelatin (Da)
0-8 hrs Beginning of lot (Beg)	130,886 ± 2919 (n=3)	100,682 ± 797 (n=3)
Middle of lot (Mid)	128,814 ± 1123 (n=3)	99,207 ± 562 (n=3)
End of lot (End)	126,107 ± 2144 (n=3)	95,809 ± 412 (n=3)
32-40 hrs Beg	121,999 ± 3328 (n=3)	90,682 ± 1584 (n=3)
Mid	117,487 ± 3178 (n=3)	91,738 ± 768 (n=3)
End	119,980 ± 998 (n=3)	90,408 ± 960 (n=3)
66-72 hrs Beg	108,762 ± 371 (n=3)	82,966 ± 777 (n=3)
Mid	113,806 ± 1609 (n=3)	81,576 ± 398 (n=3)
End	115,217 ± 883 (n=3)	81,595 ± 533 (n=3)
88-96 hrs Beg	104,670 ± 766 (n=3)	81,909 ± 1159 (n=3)
Mid	104,291 ± 1948 (n=3)	80,279 ± 945 (n=3)
End	110,645 ± 810 (n=3)	81,598 ± 277 (n=3)

[1] Values shown are mean ± standard deviation , followed by number of observations in parenthesis. Test results are in Daltons.

Figure 13-15 provide graphical representation of the tabulated data of Table 3 where strong associations could be recognized visually. Figure 13 represents the Gel Extensibility data of Table 4. A gradual decrease in the amount of force necessary to destroy the sample can be observed. Figure 14 represents the Wet Burst Strength data of Table 6. Figure 15 represents the Wet Burst Strength data of Table 7. As previously mentioned a divergence can be seen at the end point of the data set hence also in the graph.

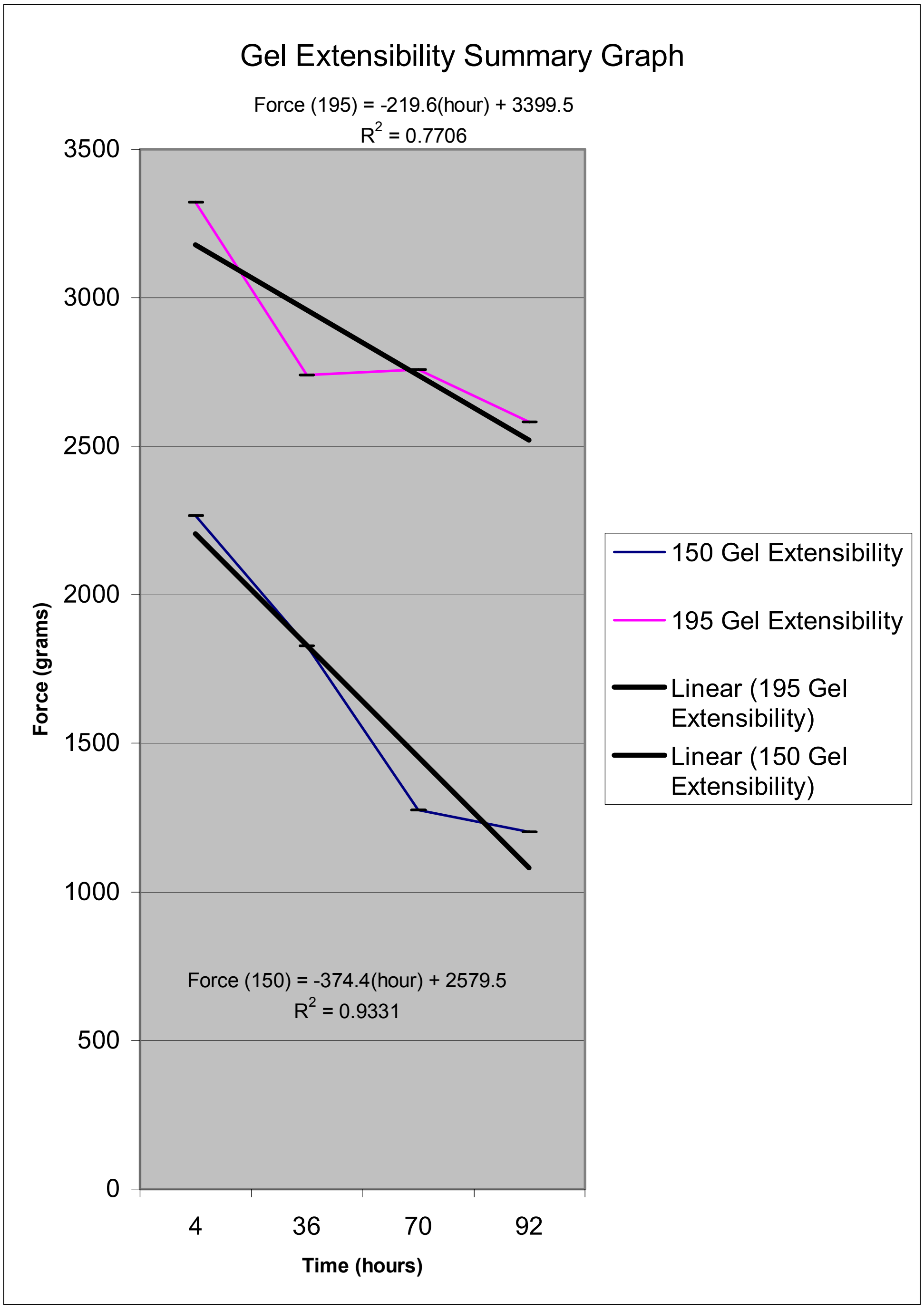
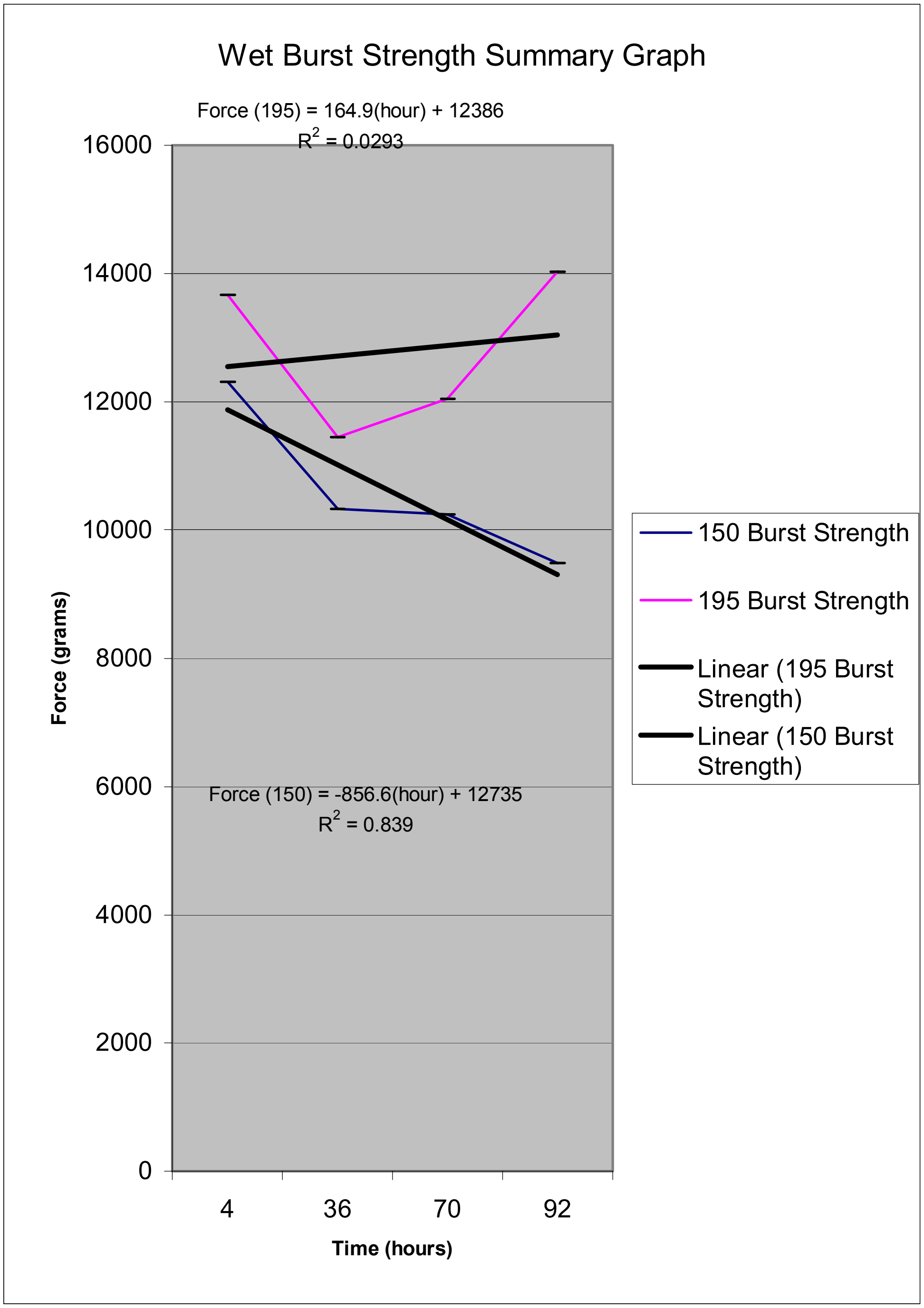


Figure 13. Summary Graph of Averaged Gelatin Extensibility. Error bars represent ± standard deviation.



Note: For clarity, only half of the standard deviation appears on this graph.

Figure 14. Summary Graph of Averaged Gelatin Wet Burst Strength. Error bars represent ± standard deviation.

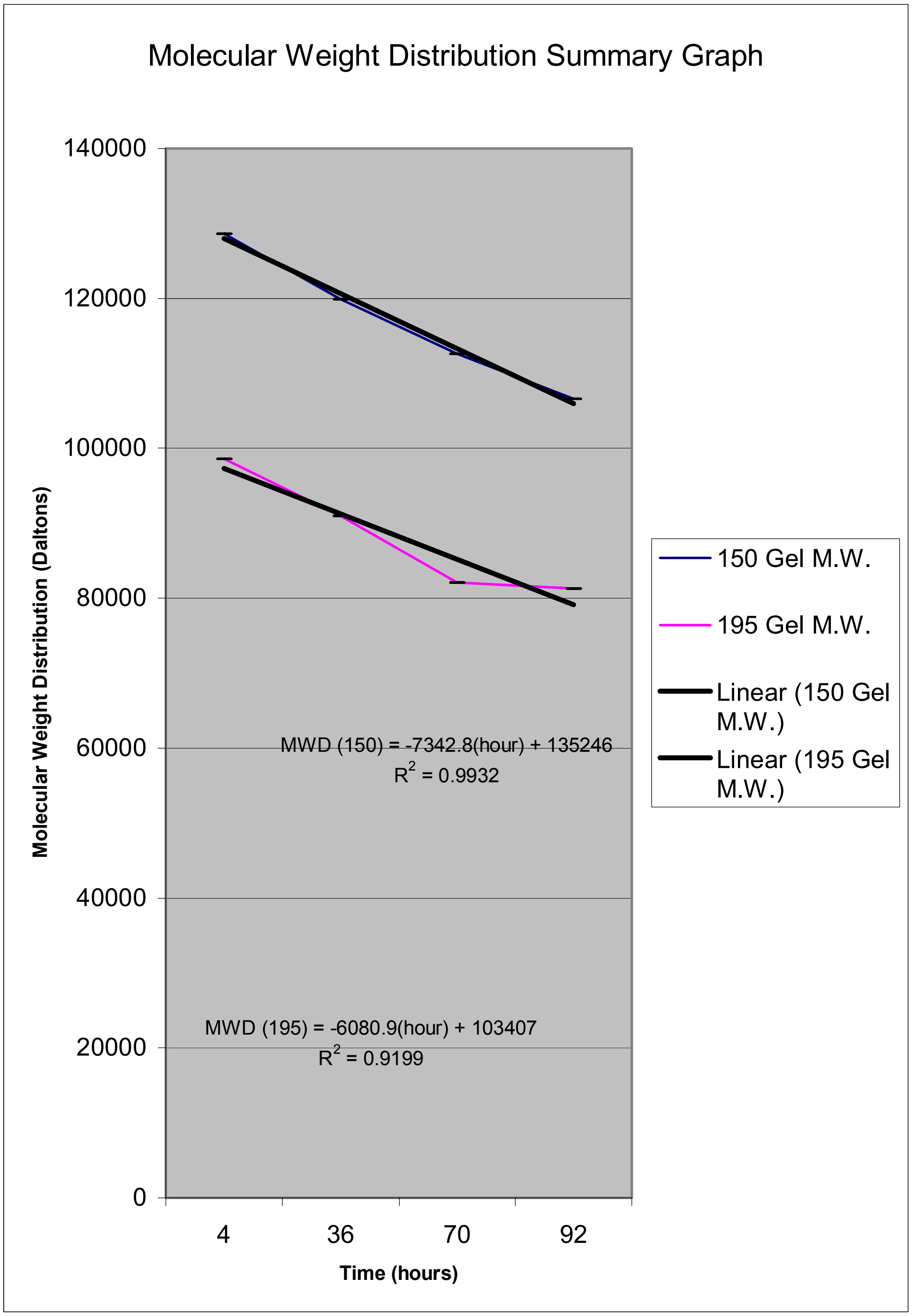


Figure 15. Summary Graph of Averaged Gelatin Molecular Weight. Error bars represent ± standard deviation.

Figure 12 visualizes the Molecular Weight data of Table 8. The association seen in this graph is quasi-linear and is the behavior being sought between the different tests in the interest of interchangeability.

Tables 9 and 10 are the matrices relating to the 1st order data analysis. As can be seen, no strong correlation exists in Table 9.

Discussion

Certain trends have been identified within the Results section. For example, the Gel Extensibility values decrease as the gel ages. It seems that such decline suggests a correlation to time. When looking at the 150 gelatin type, a gradual decrease can be seen for the Gel Extensibility, Burst Strength and Molecular Weight Analysis tests. Since mechanical properties are related to atomic/molecular aggregations, molecular weight degradation suggests mechanical strength decline. The behavior of the 195 Acid Processed gelatin shows a decline in Gel Extensibility and Molecular Weight Analysis test results. However, the Burst Strength test does not show a correlation with the Gel Extensibility and Molecular Weight Analysis as with the 150 Lime Processed gelatin. The difference between the 195 Acid Processed and 150 Lime Processed behavior suggest that the extraction processes impart different characteristics to the raw material and ultimately the end product. This is summarized in Table 9.

Table 9. Summary of Statistical Analysis of Table 3 Data.

Test Type	Is 150 Gel Type Different from the 195 Gel Type for a given time interval?	Are the results a function of time (0-96 hrs)?	Are the overall means different for the 150 and 195 Gel Types?
Gel Extensibility	Yes	150: Yes 195: Yes	Yes
Resilience	Yes	150: No 195: No	Yes
Burst Strength	Yes	150: Yes 195: No	Yes
Viscoelasticity	Yes	150: No 195: No	Yes
Molecular Weight	Yes	150: Yes 195: Yes	Yes

The differences between the two different gel types can also be seen not only in the different test results, lack of correlation between some of the tests, but also in behaviors as a function of time. When looking at the 195 gelatin type, the burst strength results decreased to ultimately achieve a greater value then the one seen at the start of the test. Such behavior suggest that either: 1) the gelatin becomes stronger as a function of time due to molecular rearrangement or; 2) the softgel becomes brittle as a function of time. The latter of the two possibilities can be disregarded due the resilience test results whose value remains constant as a function of time. When dealing with the resilience of both the 195 Acid Processed and 150 Lime Processed gelatin, it is relatively constant throughout this research.

Most of the literature suggest that molecular weight analysis and rheological determination are related. However, the characterization of this research does not show a correlation between these two tests at first glance therefore, the data collected from the various tests were subjected to statistical analysis using the SAS statistical software. The correlation used in the comparison of the tests is the Pearson Correlation Coefficients. It is understood that any values that come close to one (1) has a correlation to whatever characteristic it is being compared to.

Table 10. Pearson Correlation Coefficient for 150 Bloom Gel.

Pearson Correlation Coefficient	Time in minutes	Wet Burst Strength in mm	Gel Extensibility in minutes	% Resilience	Complex Viscosity	HPLC results
Time in minutes	1	-0.915 0.085	-0.985 0.0015	0.336 0.663	-0.20 0.8	0.998 0.001
Wet Burst Strength in mm		1	0.835 0.164	-0.071 0.93	0.076 0.92	0.93 0.07
Gel Extensibility in minutes			1	-0.488 0.512	0.275 0.724	0.978 0.4667
% Resilience				1	-0.653 0.34	0.3 0.699
Complex Viscosity					1	0.158 0.84
HPLC results						1

Table 11. Pearson Correlation Coefficient for 195 Bloom Gel.

Pearson Correla-tion Coeffi-cient	Time in minutes	Wet Burst Strength in mm	Gel Extensibility in minutes	% Resilience	Complex Viscosity	HPLC results
Time in minutes	1	0.1 0.9	-0.877 0.113	-0.797 0.412	-0.13 0.88	0.98 0.02
Wet Burst Strength in mm		1	0.24 0.76	-0.97 0.14	-0.81 0.19	-0.045 0.95
Gel Extensibi-lity in minutes			1	-0.995 0.06	0.565 0.434	-0.88 0.11
% Resilien-ce				1	-0.595 0.594	0.56 0.62
Complex Viscosity					1	-0.17 0.82
HPLC results						1

Table 10 and 11 provide a correlation analysis of the data provided by the various test methods used in this research. The tests in the Table 10 and 11 for the 195 Bloom gel relate to the following tests; time in minutes, Wet Bust Strength in mm, the distance traveled in mm for the Gel Extensibility test. The following were also part of the table, the % Resilience aspect of the Resilience test, the complex viscosity factor of the rheometer test and the results of a HPLC molecular weight distribution analysis.

The 150 Bloom gel does not have the high frequency of correlation when compared to the 195 Bloom gel. When looking at the 195 Gel, a correlation (0.99) can be seen between % Resilience and HPLC results, a negative correlation (-0.96) can be seen between complex viscosity and HPLC results, a negative correlation can be seen between % Resilience and complex viscosity (-0.97). These strong correlations suggest that either of these test can be a predictor for the other correlated test. However, the tests that do not show strong correlations provide unique independent data and their execution can not be substituted.

It can be inferred that the tests represented by % Resilience, complex viscosity, and HPLC results can be used interchangeably to predict an outcome measured by one of them. Something not seen in the first order analysis is a correlation between the individual tests and time. It has been established that molecular weight distribution and rheological properties have a correlation. The correlation seen in the matrix reconciles general knowledge to the data being analyze.

As a whole, weak correlations are seen and such behavior may be corrected by transformation of x and y variables. Such transformation is further warranted by the residual plots that show uneven distributions around the residual baseline. Also, the nonlinear appearance of the regression line suggests that a transformation of the data may provide a better model of the treatments over time.

The data of the test results were subjected to the following transformations so that a direct relationship with time, could be found for most of the tests results; 1) taking the square root of the data sets and plotting them, 2) natural logarithm transformation, 3) logarithm transformation to the base 2 and , 4) common logarithm transformation. However, when the data was subjected to the four transformations previously mentioned, an inverse correlation was been between molecular weight distribution and time. That correlation coefficient was -0.881 with an error margin of 0.0017. It was deduced that molecular weight distribution decreases with time. The transformation of Table 10 did not provide stronger correlations. However, when the data was subjected to the four transformations previously mentioned, an inverse correlation was been between molecular weight distribution and time. That correlation coefficient was -0.881 with a error margin of 0.0017. It was deduced that molecular weight distribution decreases with time. The transformation of Table 10 did not provide stronger correlations.

CHAPTER 5.

CONCLUSIONS AND RECOMMENDATIONS

Conclusions

When looking at the MS Excel representation of the test data, it is clear that the profiles of the 150 and 195 Bloom gels differ substantially. This divergence may be related to the obvious (molecular weight distribution) and may be compounded by process related additions. Because of the intrinsic behavior of the two types of gelatin, it is expected that their end products also differ.

The change of behavior was a function of time and that determination was made possible by multiple regression analysis. It can be said that differences were seen as a function of aging the gel. Eventhough there were differences in the gel due to aging, the intrinsic properties of the gelatin allowed for the production of good products (softgels). Thus the variability betweeen the two gelatins used and their properties over time was negligible.

Mathematical justification was the validation tool for the study. It can be said with a great amount of certainty that the products made with the aged gelatin are similar to the ones not being aged. There were no appreciable changes in the mechanical properties of the gelatin. The mechanical properties were mainly ascertained by using the Wet Burst and Gel Extensibility tests. However, changes were observed on the molecular level and it is these changes that are the contributors in the decay of gelatin. The determinants of decay are the rheological, resilience and molecular weight tests. It was observed that the 195 Bloom Gelatin data has strong correlation between test methods and that the 150 Bloom Gelatin has an inverse correlation between time and molecular weight. The dynamic between the two gelatins should be the same and the observed divergence may be due to human error or a skewness due to sample size. Therefore if the gels should show the same dynamics, the correlations seen in the two types should apply to each other. As a result of these assumptions, it can be said that rheological, resilience, and moleculaer weight analysis have a correlation with time. When production unit operation uses gelatin between time 0-96 hours of its manufacture, one can extrapolate the results of this study.

Softgels undergo certain dynamics which should be mentioned before addressing the findings of this study. Once the manufacturing process is completed, the softgels undergo a drying period where they stabilize and are readily manipulated. In this period of stabilization, the softgel hardens and its content loose moisture to the environment. By manipulating the parameters that were the subject of this document, one could possibly affect the final product and possibly increase its yield.

When looking at the dynamics of the results provided by the Extensibility, Resilience, Wet Burst Strength, Rheological and Molecular Weight analysis testing, one can generally observe that the properties of gelatin are subject to change as a function of time. The literature review presented many theories for such behavior and these prove themselves to point to the unavoidable degradation which affects whatever has an organic base.

In this study, we were able to quantify the changes experienced by the gelatin as a function of time and provide visual representations of such changes. Our assumption that degredation is the primary force in this work has been explained as the chemical reaction known as hydrolysis. The presence of hydrolysis can be ascertained by the molecular weight analysis of the gelatin which shows that the number decreases as a result of time.

Recommendations

The incorporation of more tests during the manufacturing process could have provided more information about the molecular dynamics of gelatin. In future research, it would be advisable to perform molecular weight analysis and rheology on samples as they are gathered from the encapsulation machine. In this study it was necessary to keep the gel mass samples days before the pertinent tests were performed. This waiting period was due to the lack of resources, mainly manpower.

By performing the tests as the samples come out of the machine, the results are more representative of the sample at the time of collection. The gel mass samples were stored in glass jars that provided a good barrier to degradation due to air. The sampling technique introduced air and the ramifications however minute, could be enough to affect the results. When preparing the samples for the various tests, molds would prove themselves beneficial in reducing human error. Great care was taken to minimize human error but variations were unavoidable oftentimes.

As the sample size increases, the parameters of interest resemble that of the population. It can be said that great care was taken to provide data with minimal interference however, few ventures follow the charted course.

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APPENDICES

Appendix 1. Microsoft (MS) Excel Graphical Representation of Test Results

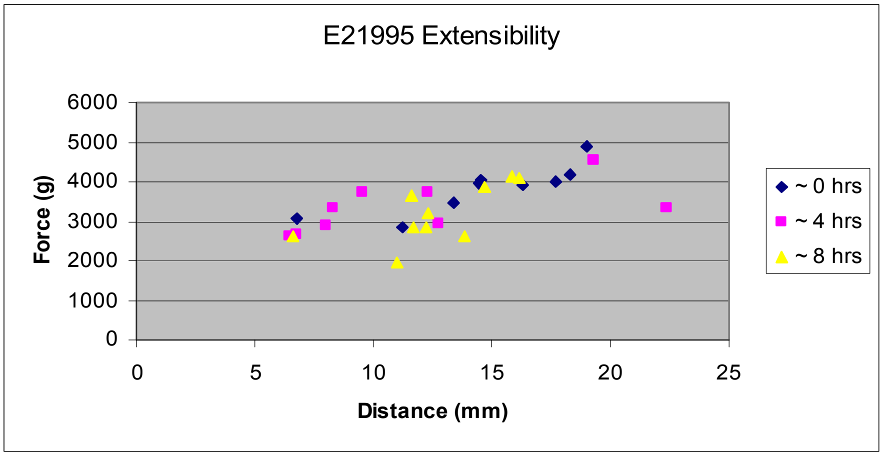

Figure 16. MS Excel Representation of Gel Extensibility Test Results for 195 Gelatin (0-8 hrs).

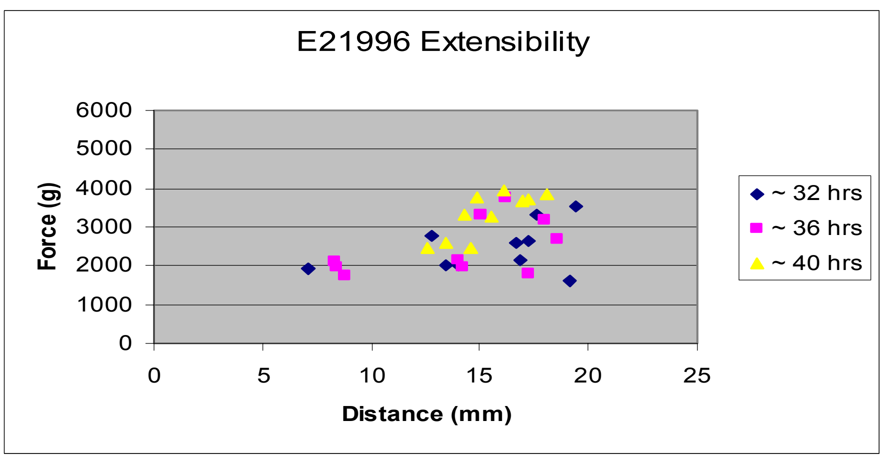

Figure 17. MS Excel Representation of Gel Extensibility Test Results for 195 Gelatin (32-40 hrs).

APPENDIX 1. Microsoft (MS) Excel Graphical Representation of Test Results (Continued)

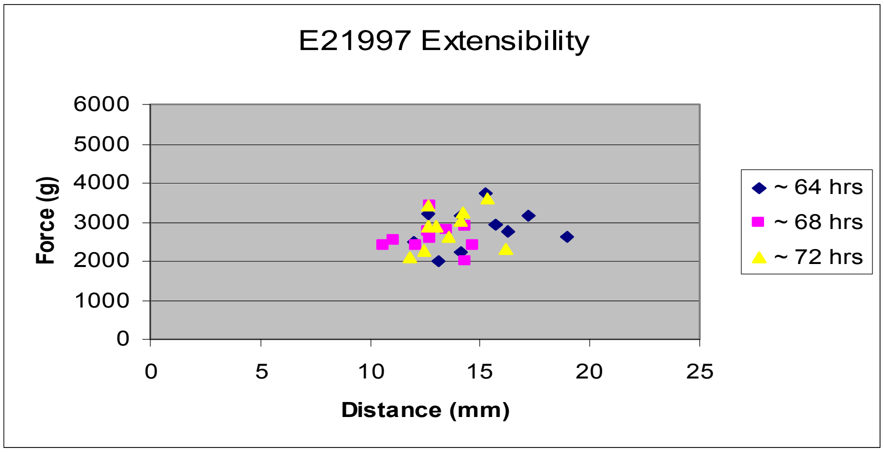

Figure 18. MS Excel Representation of Gel Extensibility Test Results for 195 Gelatin (64-72 hrs).

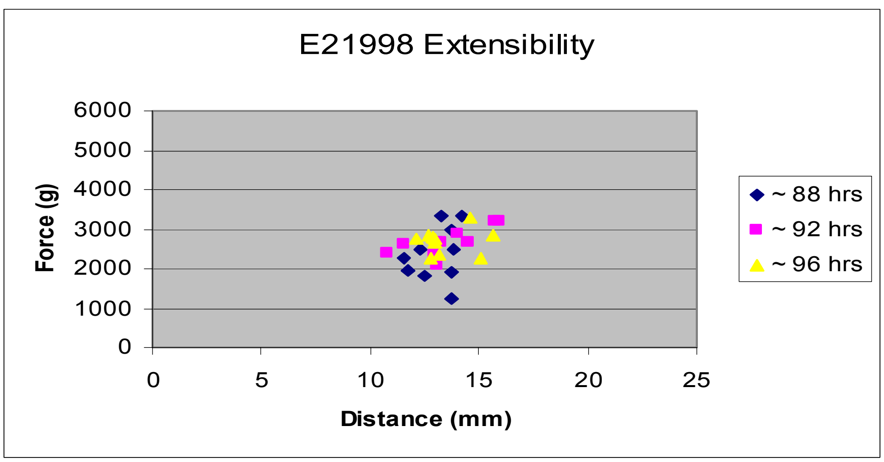

Figure 19. MS Excel Representation of Gel Extensibility Test Results for 195 Gelatin (88-96 hrs).

APPENDIX 1. Microsoft (MS) Excel Graphical Representation of Test Results (Continued)

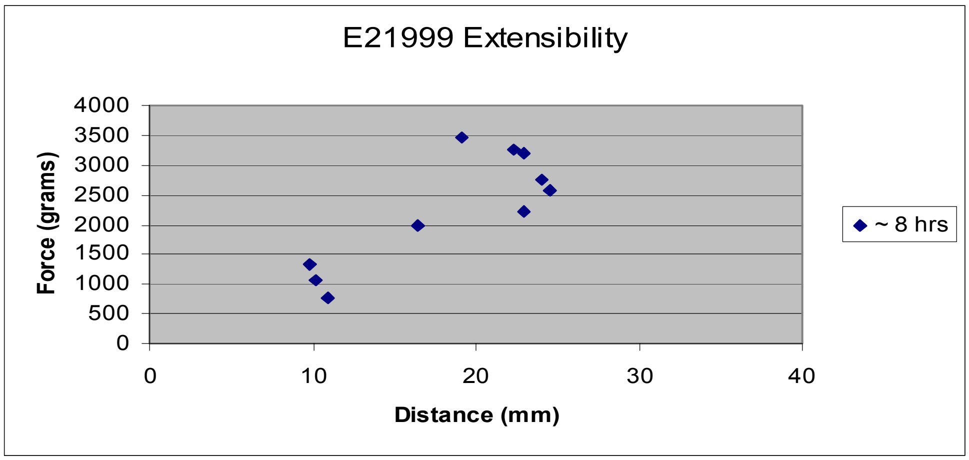

Figure 20. MS Excel Representation of Gel Extensibility Test Results for 150 Gelatin (0-8 hrs).

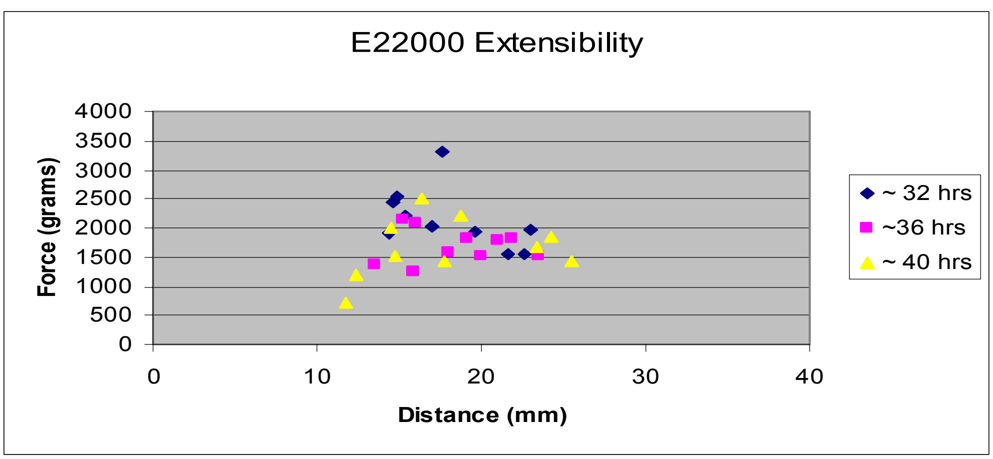

Figure 21. MS Excel Representation of Gel Extensibility Test Results for 150 Gelatin (32-40 hrs).

APPENDIX 1. Microsoft (MS) Excel Graphical Representation of Test Results (Continued)

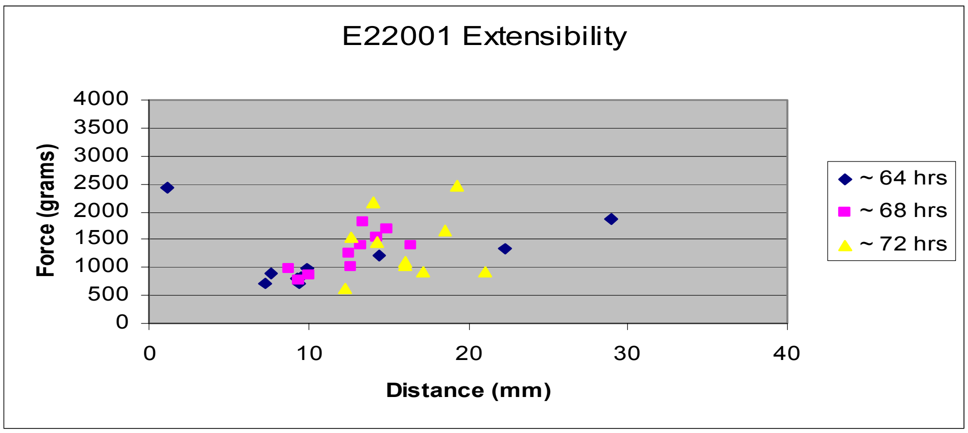

Figure 22. MS Excel Representation of Gel Extensibility Test Results for 150 Gelatin (64-72 hrs).

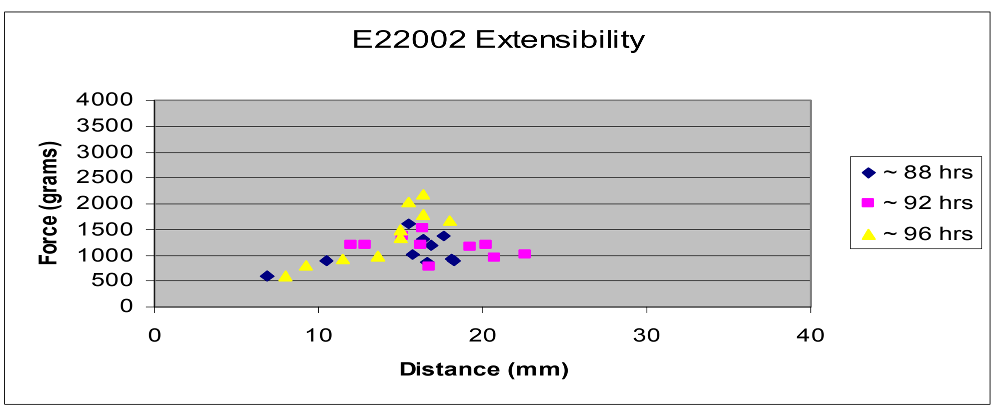

Figure 23. MS Excel Representation of Gel Extensibility Test Results for 150 Gelatin (88-96 hrs).

APPENDIX 1. Microsoft (MS) Excel Graphical Representation of Test Results (Continued)

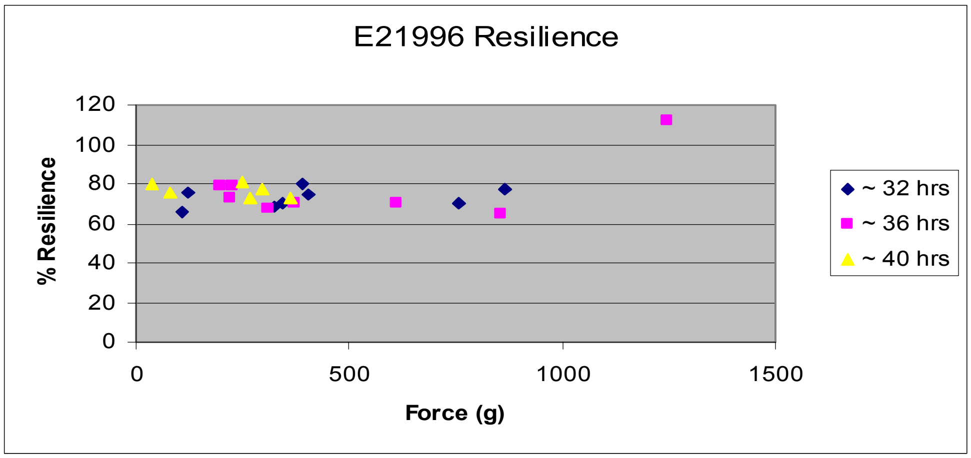

Figure 24. MS Excel Representation of Resilience Test Results for 195 Gelatin (32-40 hrs).

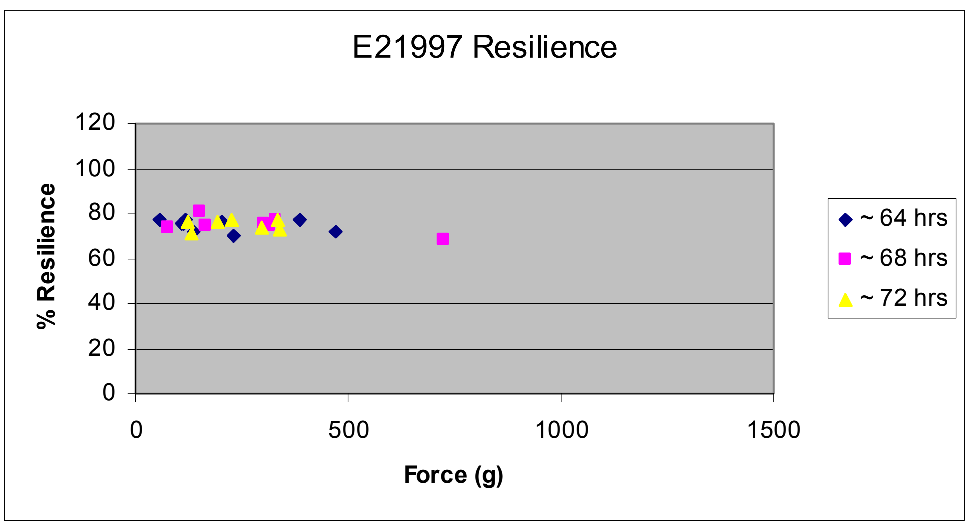

Figure 25. MS Excel Representation of Resilience Test Results for 195 Gelatin (64-72 hrs).

APPENDIX 1. Microsoft (MS) Excel Graphical Representation of Test Results (Continued)

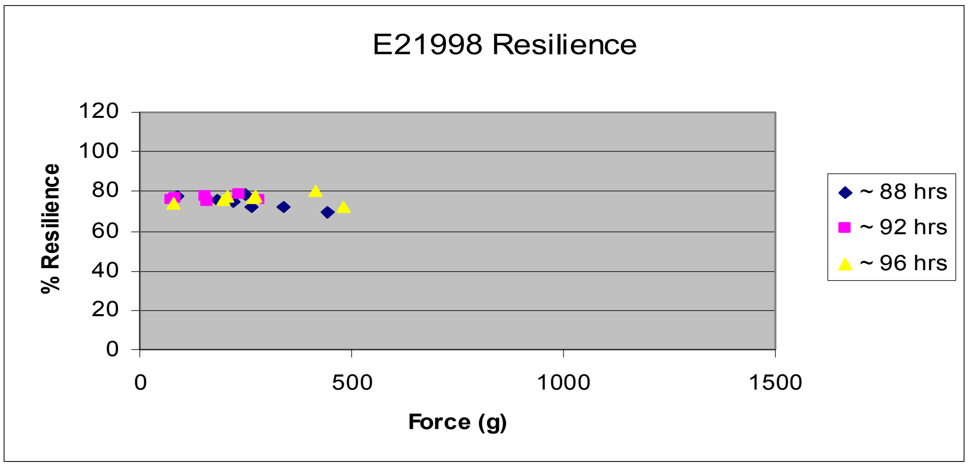

Figure 26. MS Excel Representation of Resilience Test Results for 195 Gelatin (88-96 hrs).

APPENDIX 1. Microsoft (MS) Excel Graphical Representation of Test Results (Continued)

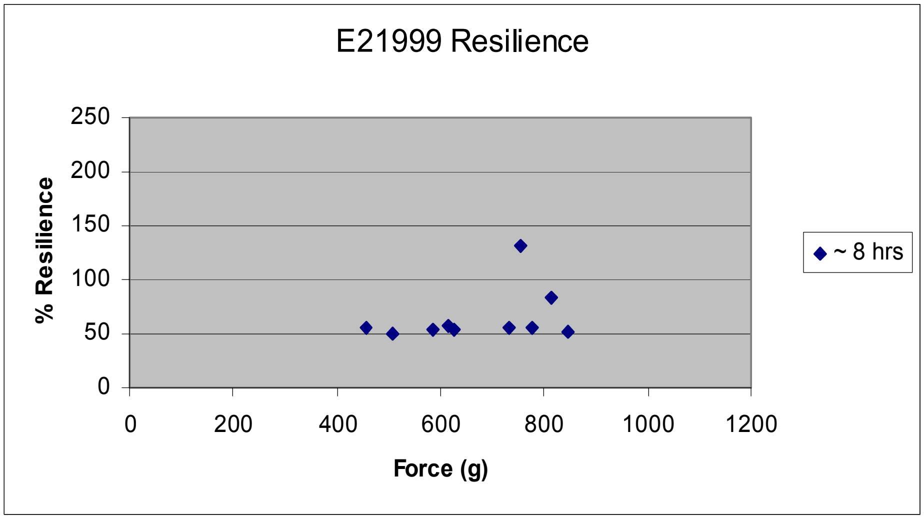

Figure 27. MS Excel Representation of Resilience Test Results for 150 Gelatin (0-8 hrs).

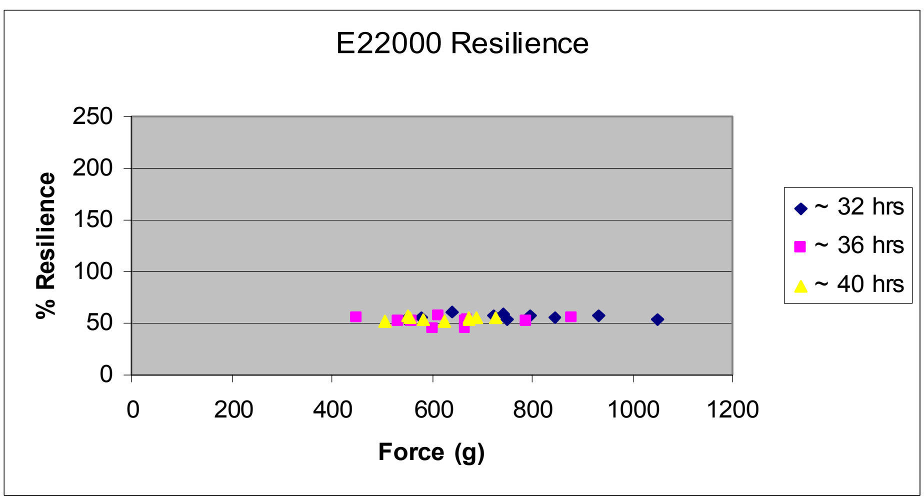

Figure 28. MS Excel Representation of Resilience Test Results for 150 Gelatin (32-40 hrs).

APPENDIX 1. Microsoft (MS) Excel Graphical Representation of Test Results (Continued)

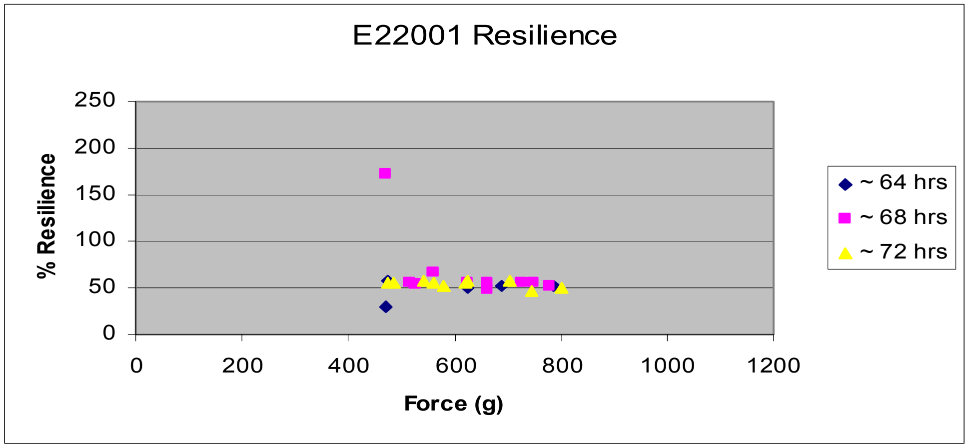

Figure 29. MS Excel Representation of Resilience Test Results for 150 Gelatin (64-72 hrs).

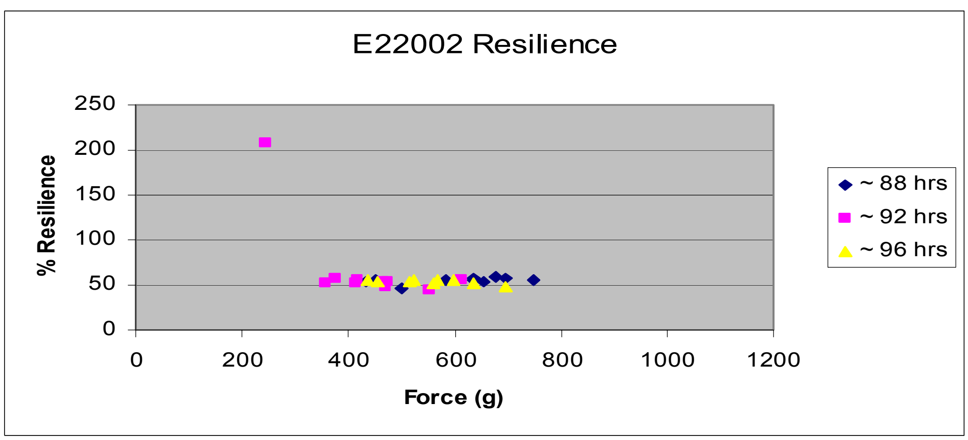

Figure 30. MS Excel Representation of Resilience Test Results for 150 Gelatin (88-96 hrs).

APPENDIX 1. Microsoft (MS) Excel Graphical Representation of Test Results (Continued)

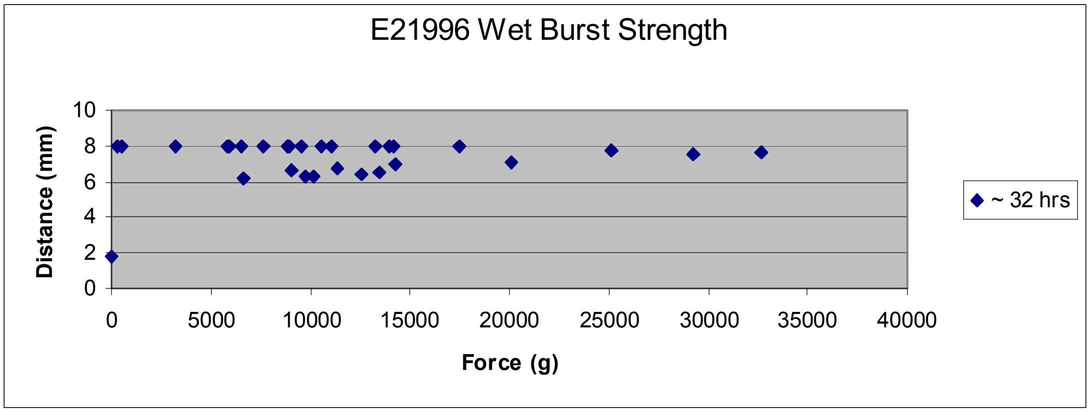

Figure 31. MS Excel Representation of Wet Burst Strength Test Results for 195 Gelatin (32-40 hrs).

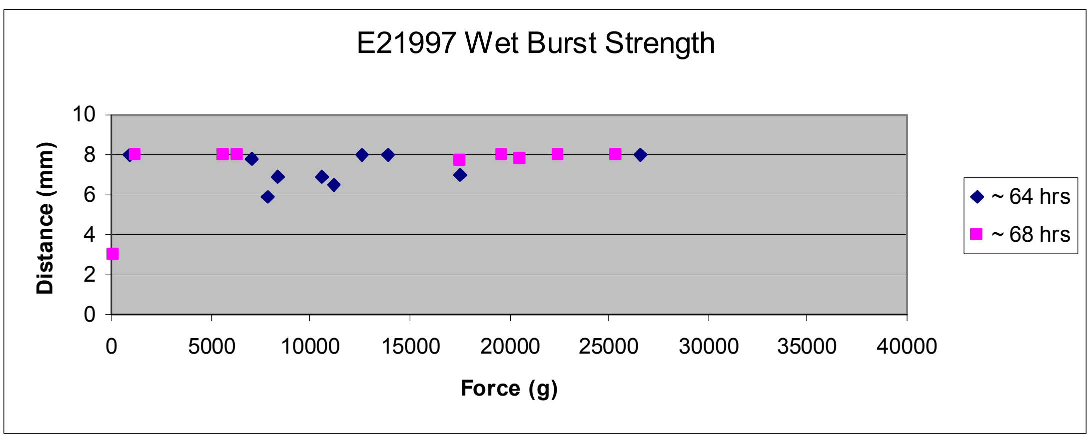

Figure 32. MS Excel Representation of Wet Burst Strength Test Results for 195 Gelatin (64-72 hrs).

APPENDIX 1. Microsoft (MS) Excel Graphical Representation of Test Results (Continued)

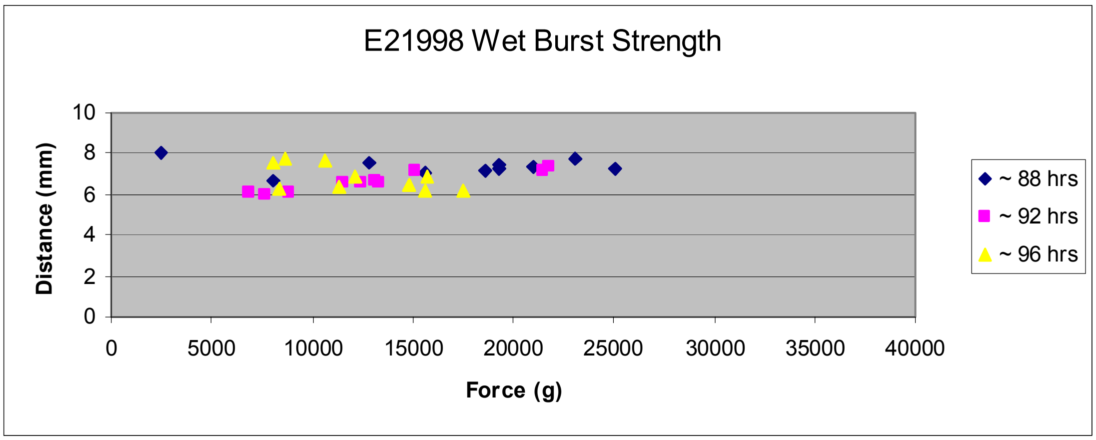

Figure 33. MS Excel Representation of Wet Burst Strength Test Results for 195 Gelatin (88-96 hrs).

APPENDIX 1. Microsoft (MS) Excel Graphical Representation of Test Results (Continued)

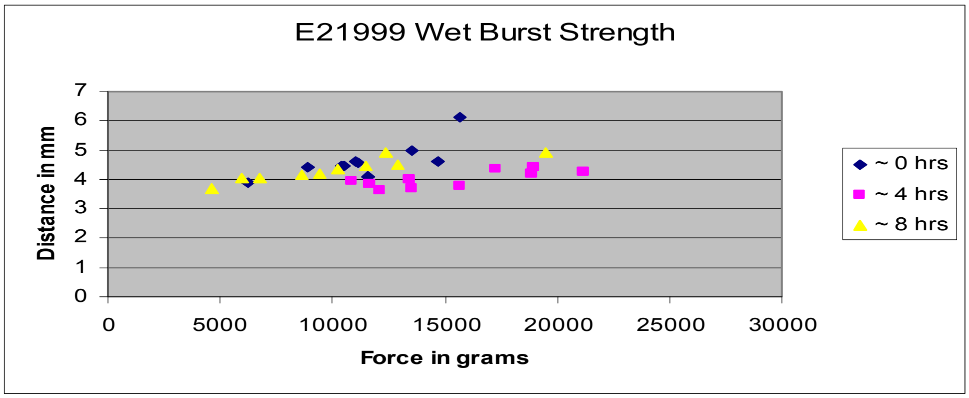

Figure 34. MS Excel Representation of Wet Burst Strength Test Results for 150 Gelatin (0-8 hrs).

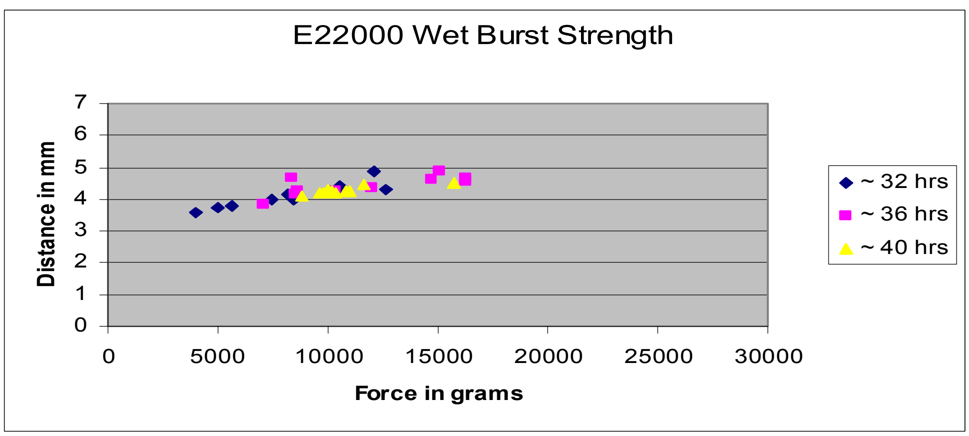

Figure 35. MS Excel Representation of Wet Burst Strength Test Results for 150 Gelatin (32-40 hrs).

APPENDIX 1. Microsoft (MS) Excel Graphical Representation of Test Results (Continued)

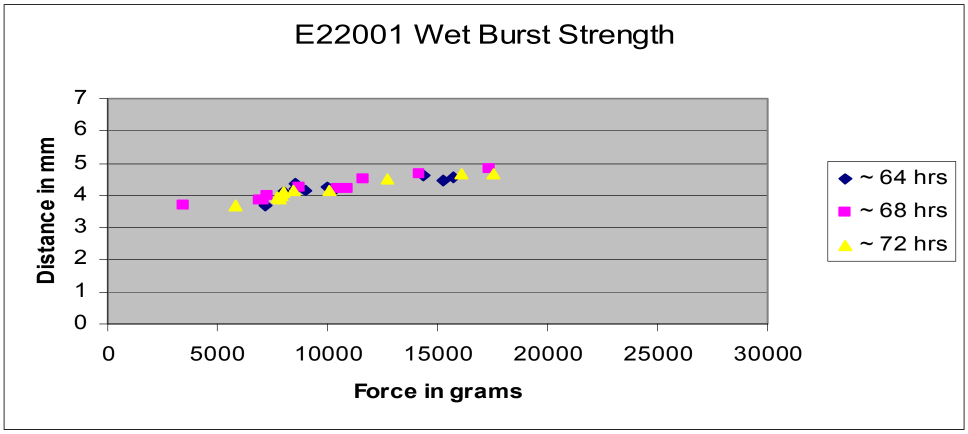

Figure 36. MS Excel Representation of Wet Burst Strength Test Results for 150 Gelatin (64-72 hrs).

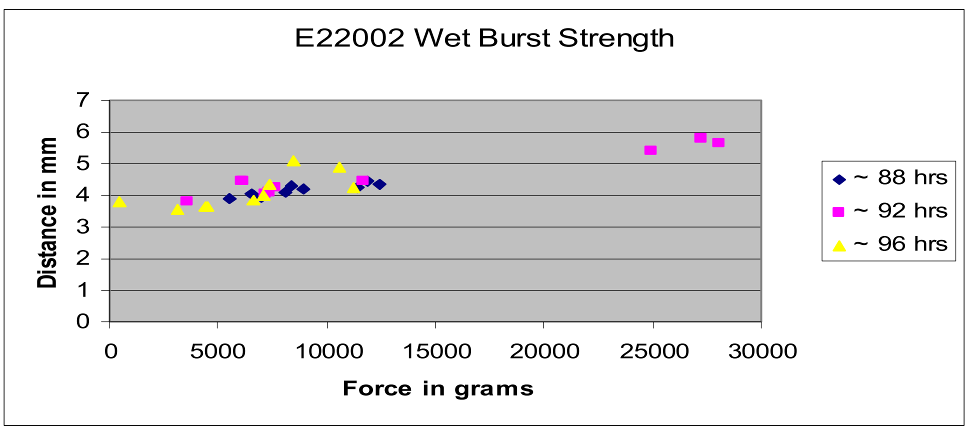

Figure 37. MS Excel Representation of Wet Burst Strength Test Results for 150 Gelatin (88-96 hrs).

Appendix 2. Wet Burst Strength Test Results

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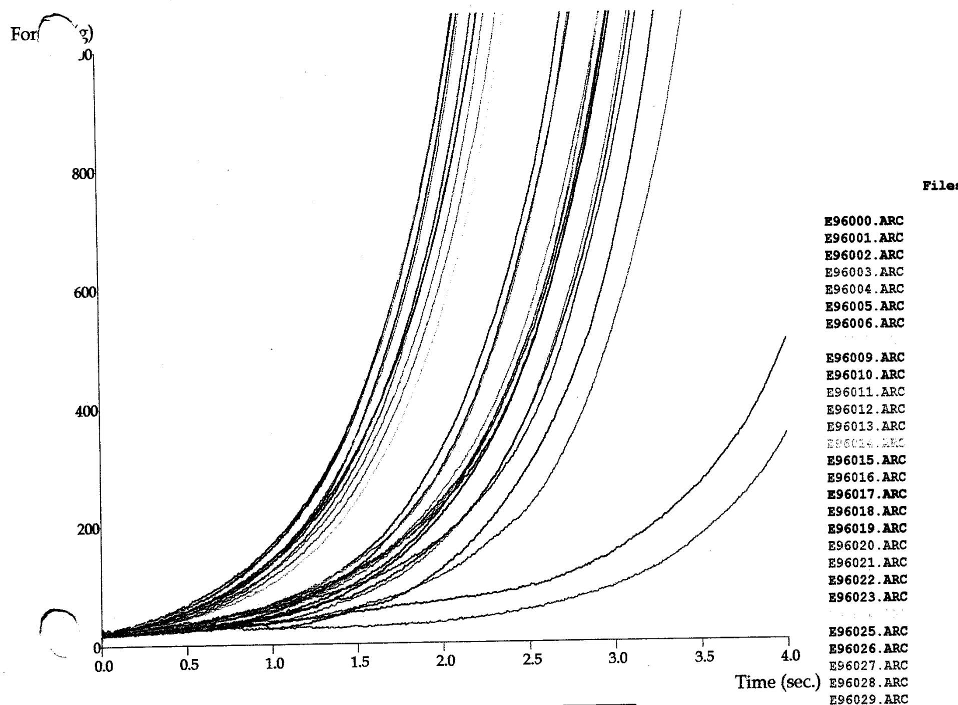
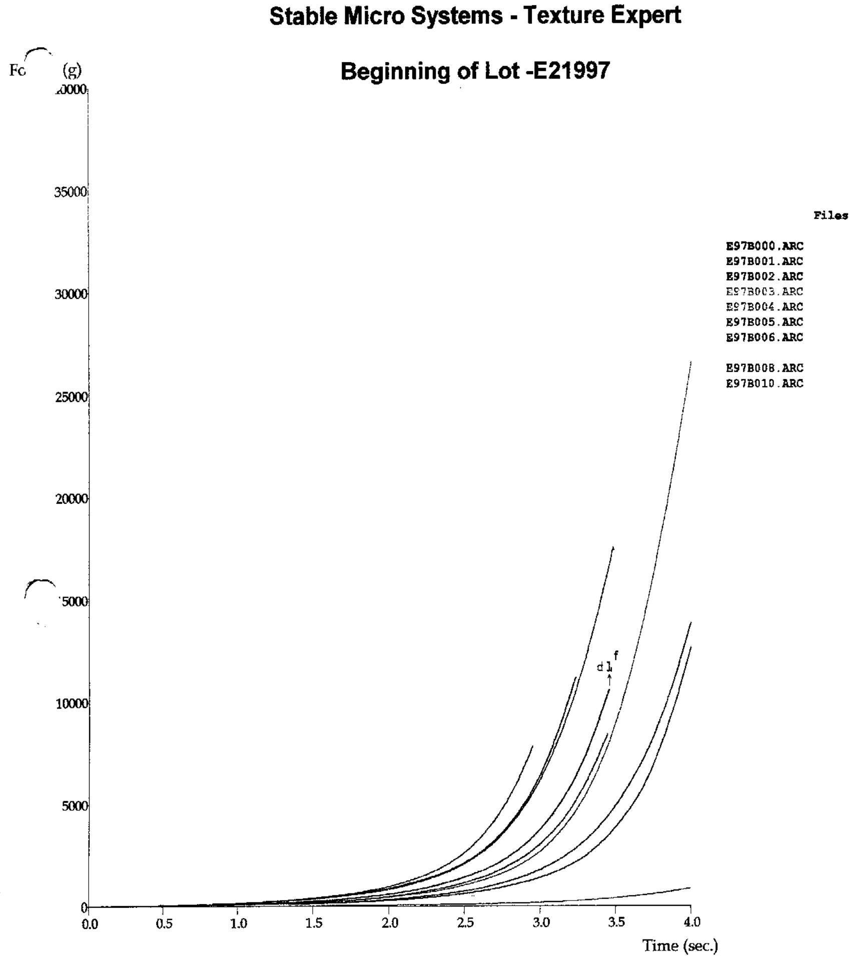


Test ID	Force 1	Distance 1
	g	mm
E96000	13431.239	6.555
E96001	9579.999	7.983
E96002	17500.282	7.992
E96003	3238.210	8.000
E96004	11378.416	6.785
E96005	14258.220	6.996
E96006	29274.282	7.517
E96007	13232.001	7.993
E96009	8931.721	7.992
E96010	20060.822	7.048
E96011	6570.182	7.999
E96012	347.086	7.991
E96013	6624.261	6.202
E96014	25149.658	7.771
E96015	9746.785	6.289
E96016	5856.559	7.994
E96017	7565.506	8.000
E96018	36.244	1.763
E96019	11086.397	7.995
E96020	10531.366	7.992
E96021	10136.039	6.248
E96022	13961.469	7.993
E96023	5859.030	8.001
E96024	12530.559	6.385
E96025	32622.795	7.616
E96026	504.805	7.993
E96027	9027.074	6.585
E96028	14151.403	7.999
E96029	8859.280	7.993
Average	11450.059	7.299
Std Dev (n-1)	7763.764	1.259
% CV	0.678	0.173

Figure 38. 195 Gelatin Wet Burst Strength Printout at 32 hrs.

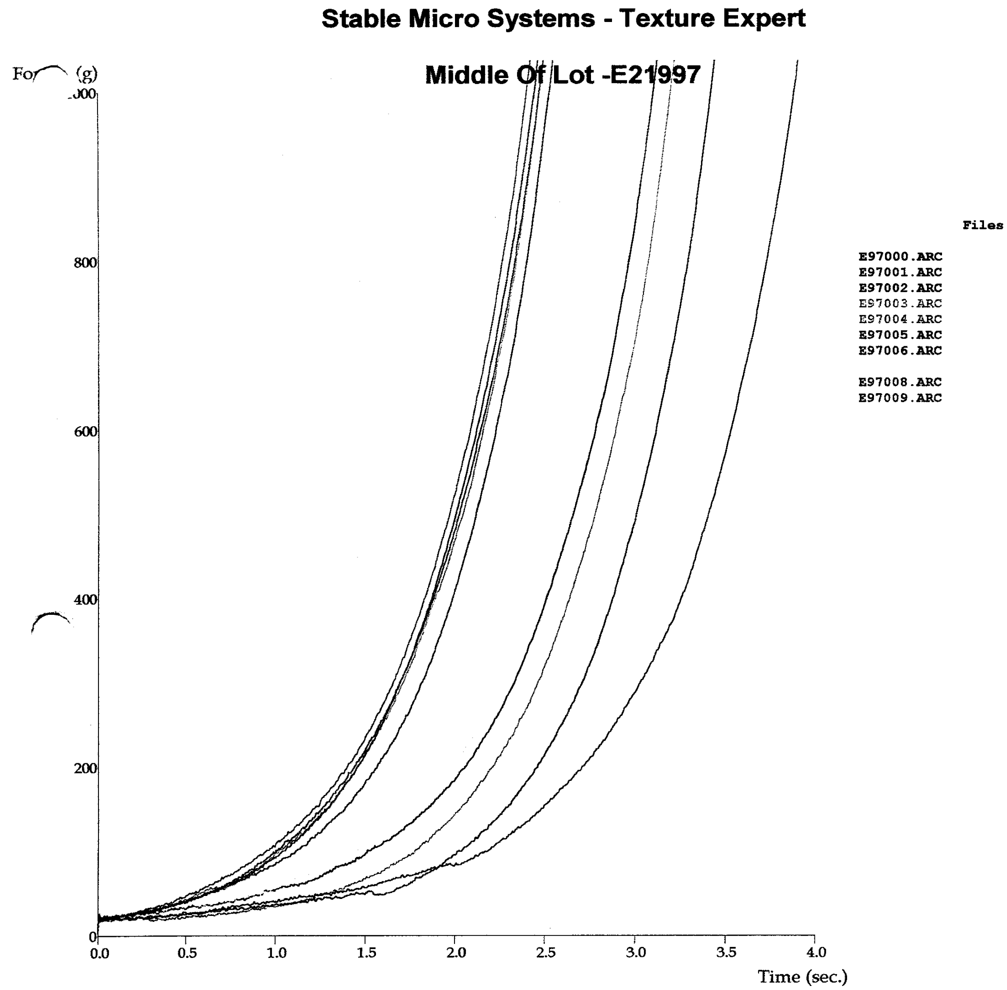
APPENDIX 2. Wet Burst Strength Test Results (Continued)



Test ID	Force 1	Distance 1
	g	mm
E97B000	10569.848	6.914
E97B001	12633.872	7.997
E97B002	11170.496	6.473
E97B003	874.082	7.995
E97B004	26574.520	7.998
E97B005	7820.502	5.906
E97B006	13854.972	7.999
E97B007	7086.323	7.839
E97B008	8392.510	6.896
E97B010	17535.484	6.970
Average	11651.261	7.299
Std Dev (n-1)	6886.626	0.766
% CV	0.591	0.105

Figure 39. 195 Gelatin Wet Burst Strength Printout at 64 hrs.

APPENDIX 2. Wet Burst Strength Test Results (Continued)



Test ID	Force 1 g	Distance 1 mm
E97000	5682.719	7.997
E97001	22478.464	7.992
E97002	53.238	3.047
E97003	5618.424	7.993
E97004	20567.268	7.841
E97005	17544.821	7.668
E97006	1234.811	7.998
E97007	6317.850	7.993
E97008	19619.055	7.999
E97009	25347.881	7.994
Average	12446.453	7.452
Std Dev (n-1)	9543.455	1.552
% CV	0.767	0.208

Figure 40. 195 Gelatin Wet Burst Strength Printout at 68 hrs.

APPENDIX 2. Wet Burst Strength Test Results (Continued)

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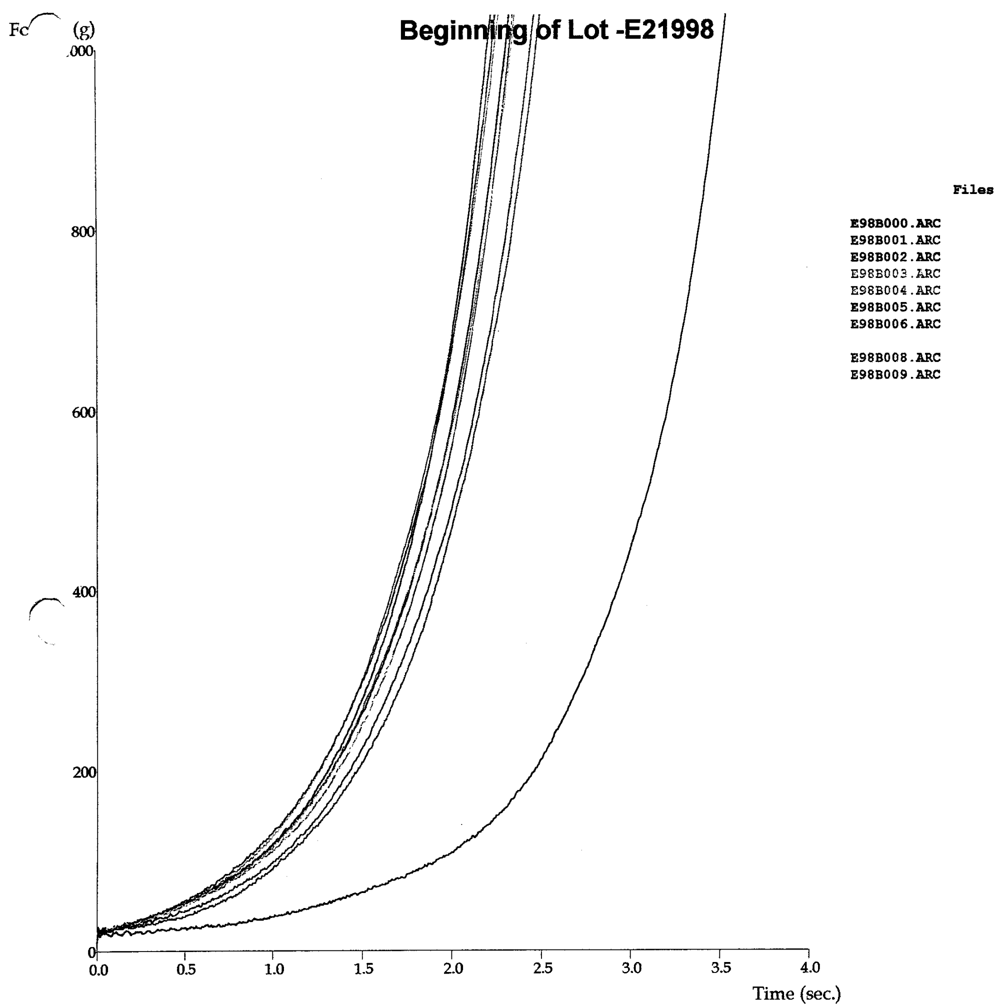
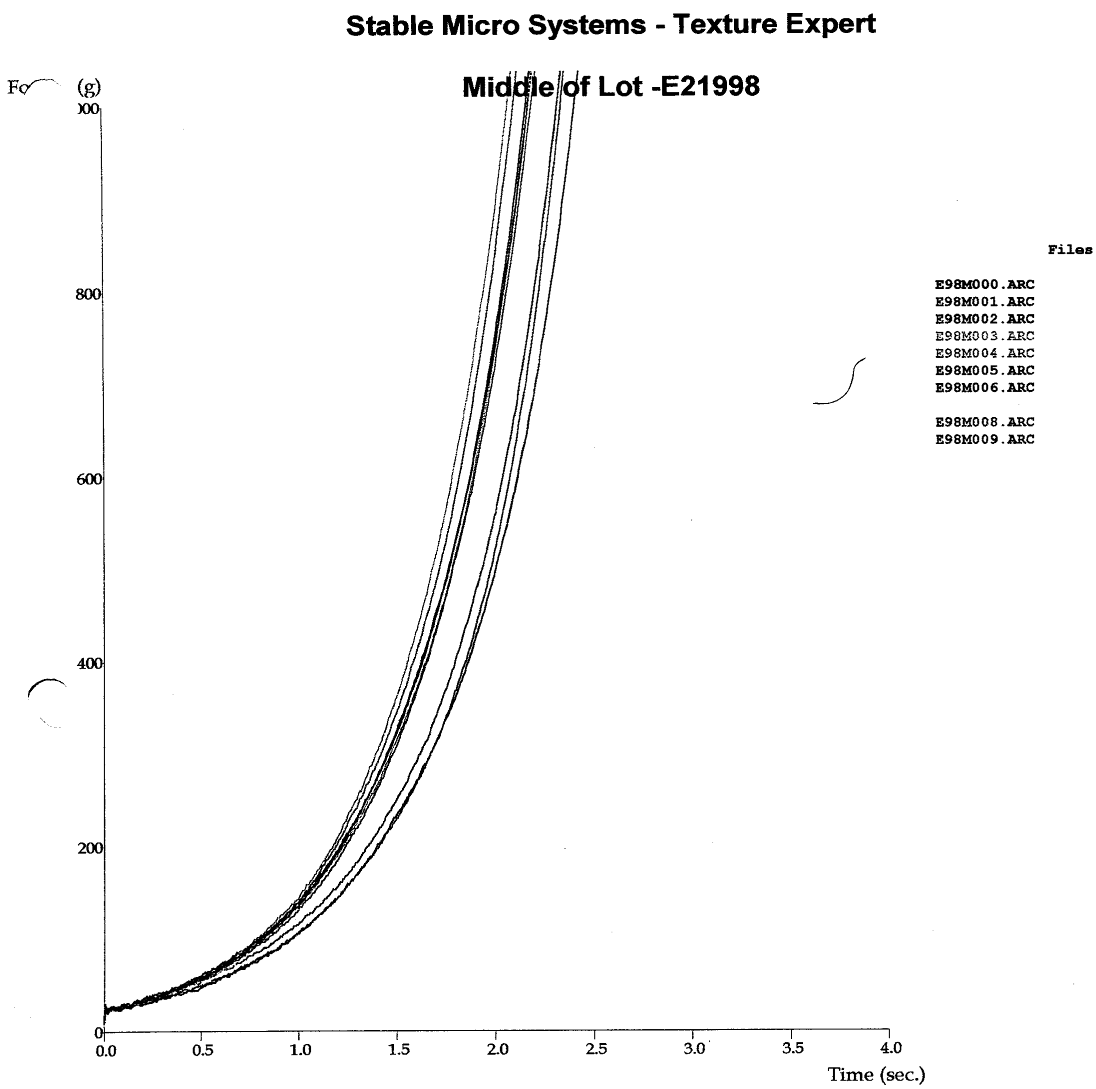


Test ID	Force 1 g	Distance 1 mm
E98B000	2517.631	7.996
E98B001	25064.299	7.260
E98B002	19338.009	7.245
E98B003	19346.676	7.443
E98B004	8026.451	6.636
E98B005	23119.791	7.763
E98B006	15641.668	7.075
E98B007	21028.927	7.326
E98B008	12832.657	7.533
E98B009	18602.825	7.115
Average	16551.893	7.339
Std Dev (n-1)	6989.887	0.378
% CV	0.422	0.052

Figure 41. 195 Gelatin Wet Burst Strength Printout at 88 hrs.

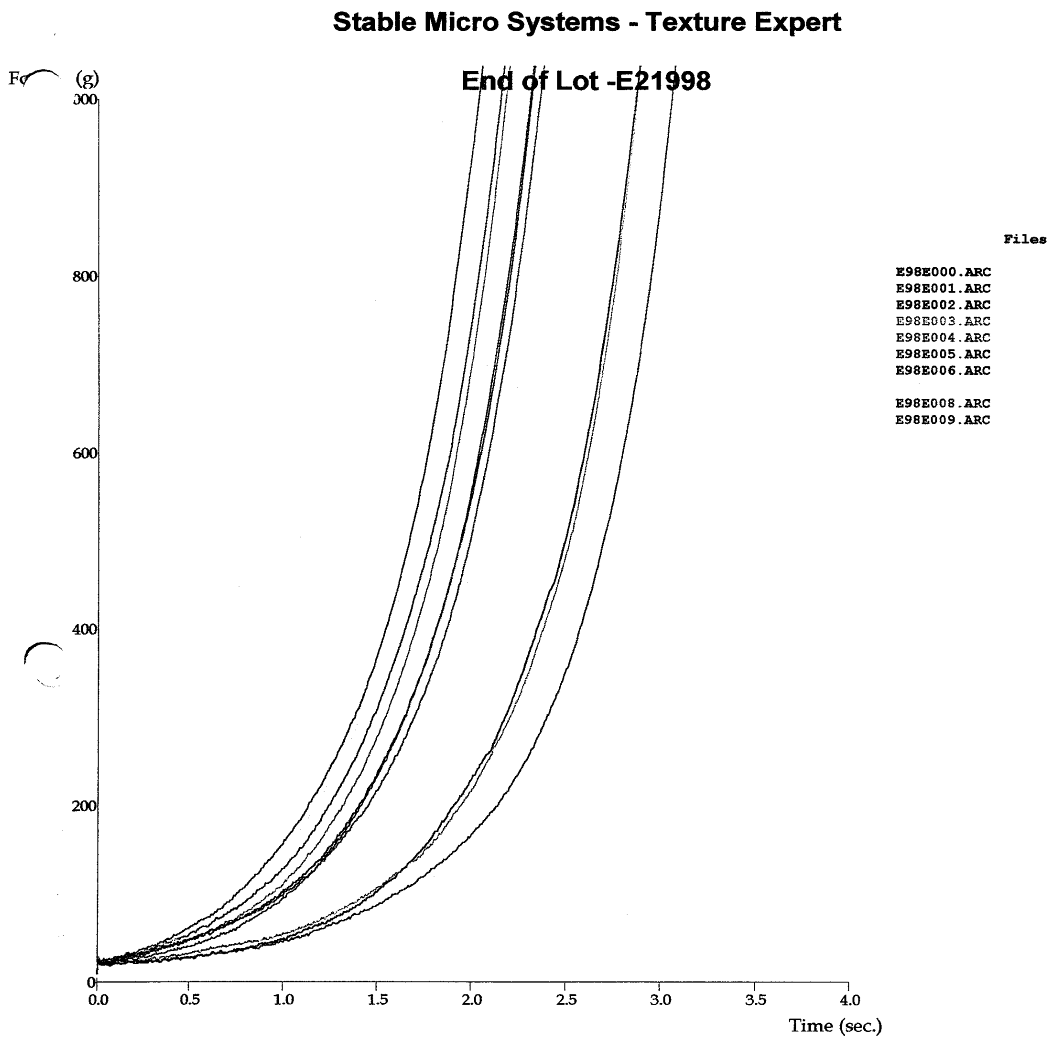
APPENDIX 2. Wet Burst Strength Test Results (Continued)



Test ID	Force 1	Distance 1
	g	mm
E98M000	15086.603	7.122
E98M001	11535.096	6.576
E98M002	21796.892	7.368
E98M003	13352.504	6.539
E98M004	8840.866	6.095
E98M005	6872.551	6.110
E98M006	13128.763	6.667
E98M007	12467.415	6.578
E98M008	7631.171	5.980
E98M009	21489.456	7.148
Average	13220.132	6.618
Std Dev (n-1)	5159.288	0.477
% CV	0.390	0.072

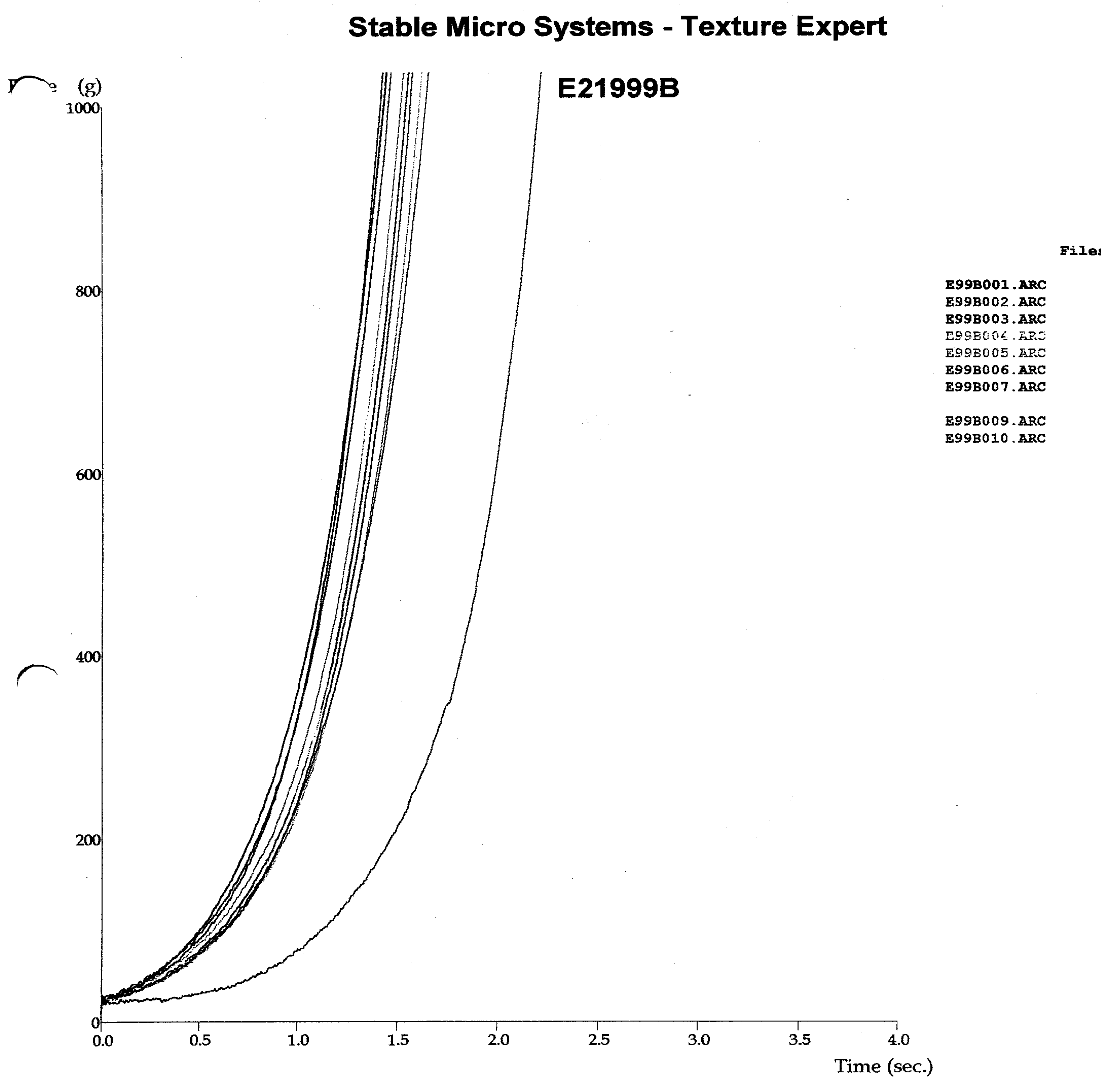
Figure 42. 195 Gelatin Wet Burst Strength Printout at 92 hrs.

APPENDIX 2. Wet Burst Strength Test Results (Continued)



Test ID	Force 1	Distance 1
	g	mm
E98E000	8104.531	7.524
E98E001	8619.293	7.746
E98E002	15699.936	6.852
E98E003	10688.218	7.623
E98E004	14827.753	6.449
E98E005	17510.424	6.175
E98E006	11367.415	6.331
E98E007	15632.066	6.155
E98E008	12137.377	6.822
E98E009	8352.378	6.312
Average	12293.939	6.799
Std Dev (n-1)	3438.785	0.623
% CV	0.280	0.092

Figure 43. 195 Gelatin Wet Burst Strength Printout at 96 hrs.



Test ID	Force 1	Distance 1	Force 2
	g	mm	g
E99B010	13511.782	4.974	13511.782
E99B001	14699.432	4.608	
E99B002	10471.109	4.449	
E99B003	10414.124	4.435	
E99B004	11014.778	4.593	
E99B005	11127.035	4.580	
E99B006	6259.450	3.912	
E99B007	8889.431	4.422	
E99B008	11604.144	4.119	
E99B009	15689.916	6.138	
Average	11368.120	4.623	13511.782
Std Dev (n-1)	2760.167	0.604	
% CV			

Figure 44. 150 Gelatin Wet Burst Strength Printout at 0 hrs.

APPENDIX 2. Wet Burst Strength Test Results (Continued)

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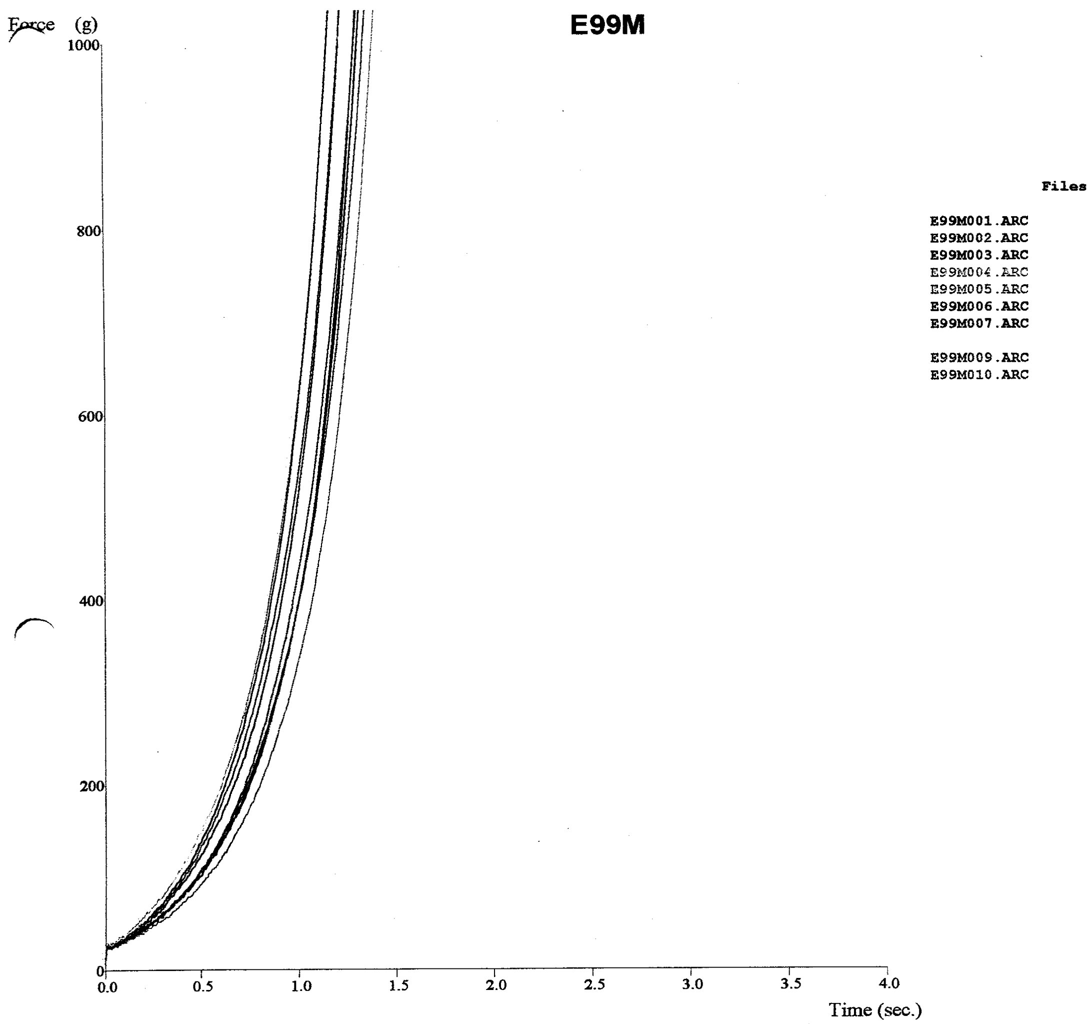
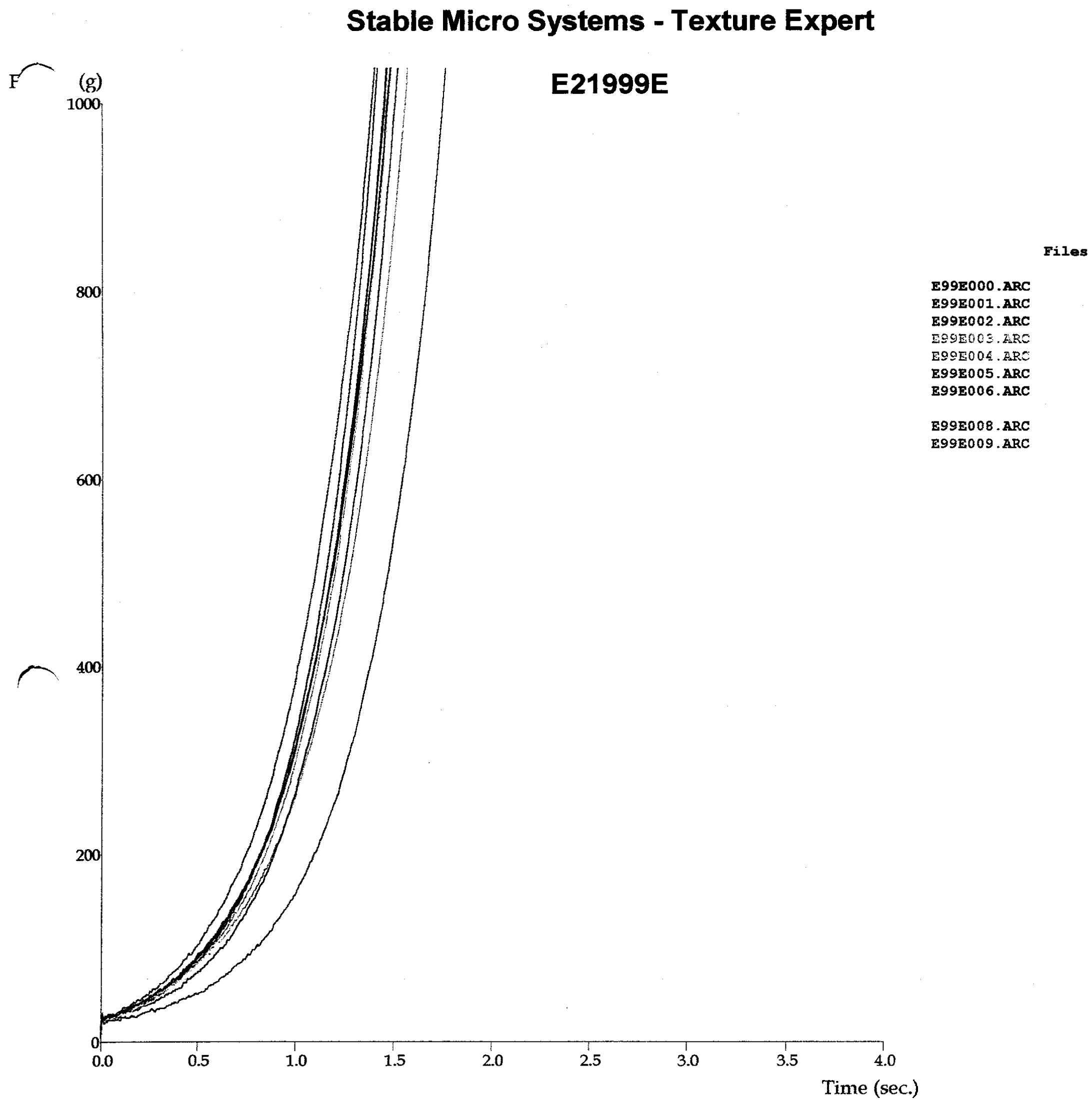


Test ID	Date and Time	Force 1 g	Distance 1 mm
E99M001	Mon Oct 07 13:04:24 2002	18875.963	4.211
E99M002	Mon Oct 07 13:05:00 2002	17297.297	4.360
E99M003	Mon Oct 07 13:05:16 2002	13479.504	4.010
E99M004	Mon Oct 07 13:05:42 2002	15646.684	3.762
E99M005	Mon Oct 07 13:05:56 2002	18999.928	4.433
E99M006	Mon Oct 07 13:06:12 2002	12129.233	3.642
E99M007	Mon Oct 07 13:06:26 2002	21158.421	4.262
E99M008	Mon Oct 07 13:06:42 2002	13563.030	3.659
E99M009	Mon Oct 07 13:06:56 2002	10896.555	3.962
E99M010	Mon Oct 07 13:07:12 2002	11688.299	3.838
Average	AVG("COL")	15373.491	4.014
Std Dev n-1	SD1("COL")	3553.921	0.290
% cv	SD1("COL")/AVG("COL")	0.231	0.072

Figure 45. 150 Gelatin Wet Burst Strength Printout at 4 hrs.

APPENDIX 2. Wet Burst Strength Test Results (Continued)



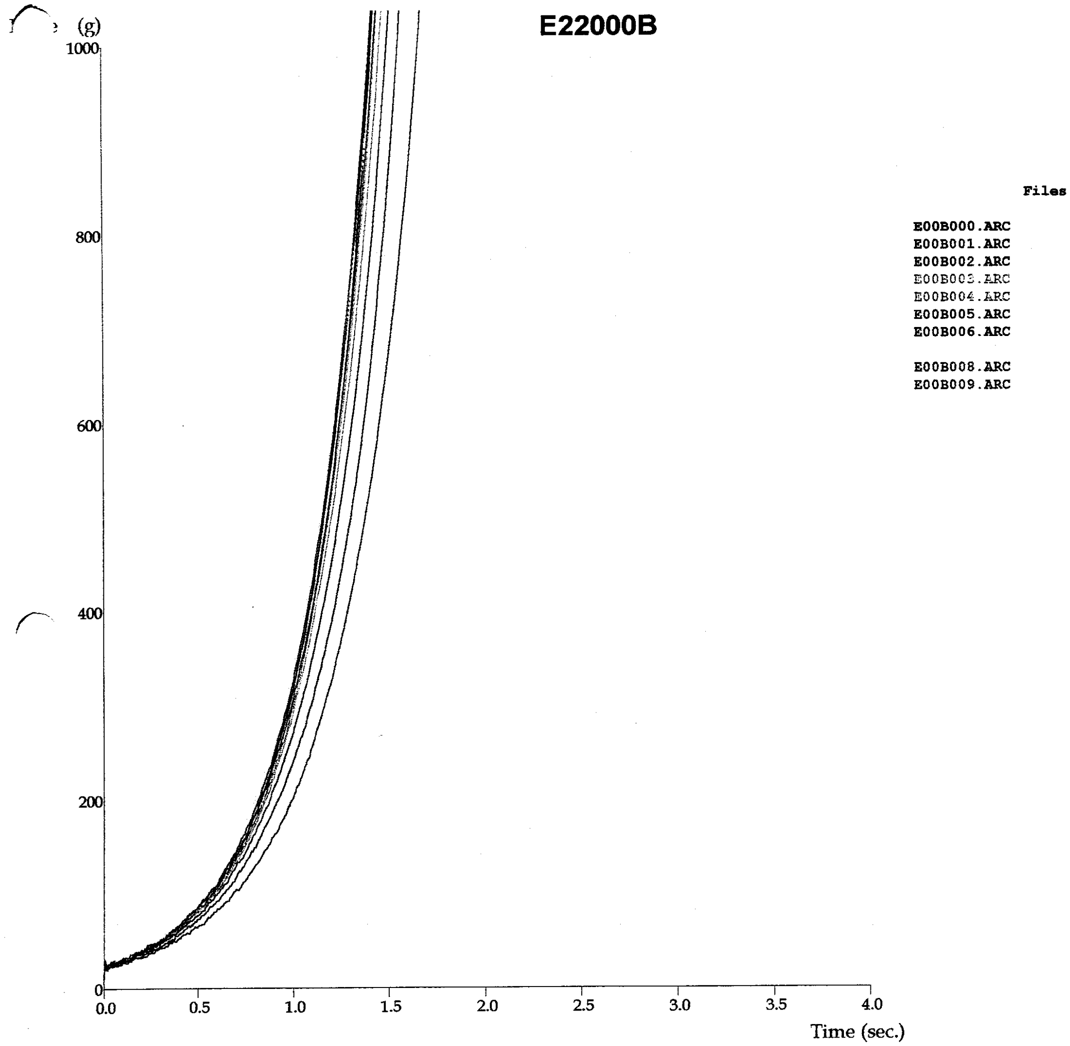
Test ID	Force 1	Distance 1
	g	mm
E99E000	11447.781	4.465
E99E001	8673.583	4.160
E99E002	4641.229	3.656
E99E003	19507.282	4.943
E99E004	6765.607	4.051
E99E005	5923.579	4.021
E99E006	9466.339	4.194
E99E007	10252.295	4.373
E99E008	12886.087	4.486
E99E009	12386.270	4.933
Average	10195.005	4.328
Std Dev (n-1)	4269.149	0.403
% CV	0.419	0.093

Figure 46. 150 Gelatin Wet Burst Strength Printout at 8 hrs.

APPENDIX 2. Wet Burst Strength Test Results (Continued)

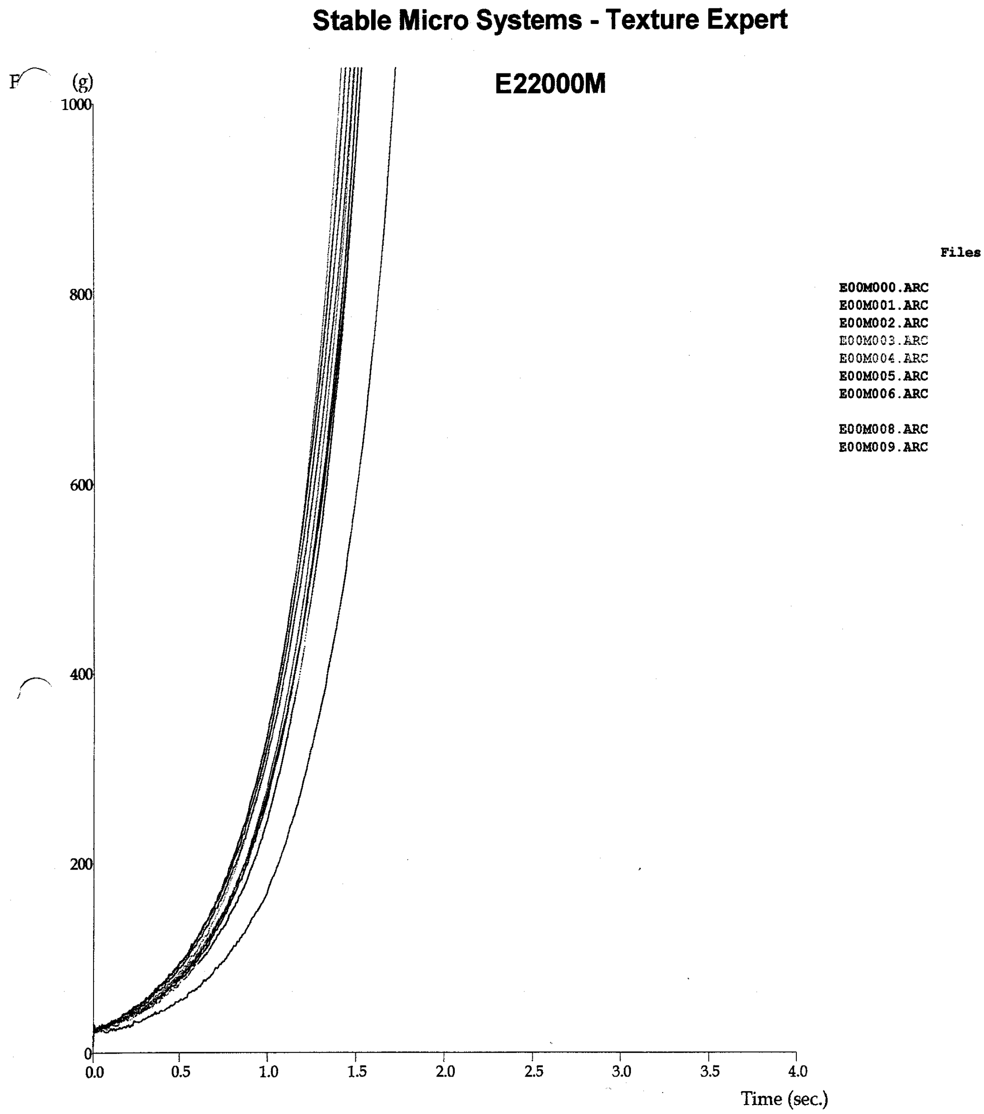
Stable Micro Systems - Texture Expert

E22000B



Test ID	Force 1 g	Distance 1 mm
E00B000	5648.971	3.785
E00B001	10519.650	4.389
E00B002	4039.642	3.565
E00B003	8410.121	3.997
E00B004	12601.480	4.289
E00B005	10696.218	4.237
E00B006	12117.449	4.876
E00B007	8138.259	4.154
E00B008	7473.368	3.975
E00B009	4958.716	3.756
Average	8460.388	4.102
Std Dev (n-1)	2991.683	0.376
% CV	0.354	0.092

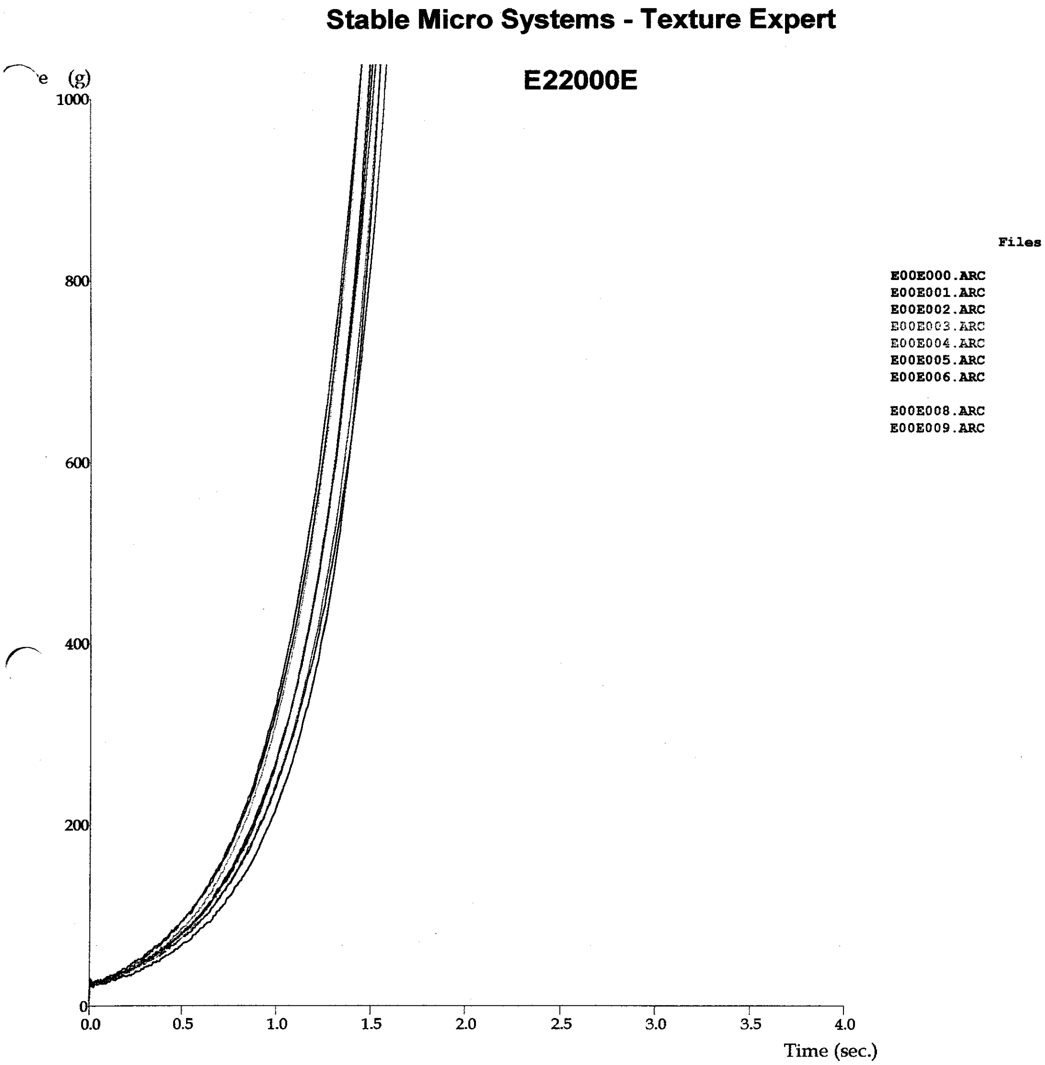
Figure 47. 150 Gelatin Wet Burst Strength Printout at 32 hrs.



Test ID	Force 1 g	Distance 1 mm
E00M000	16311.258	4.558
E00M001	10362.388	4.242
E00M002	11970.011	4.350
E00M003	7114.681	3.839
E00M004	8572.409	4.171
E00M005	8327.135	4.663
E00M006	8605.186	4.237
E00M007	15108.352	4.878
E00M008	14747.099	4.614
E00M009	16267.105	4.675
Average	11738.563	4.423
Std Dev (n-1)	3599.879	0.309
% CV	0.307	0.070

Figure 48. 150 Gelatin Wet Burst Strength Printout at 36 hrs.

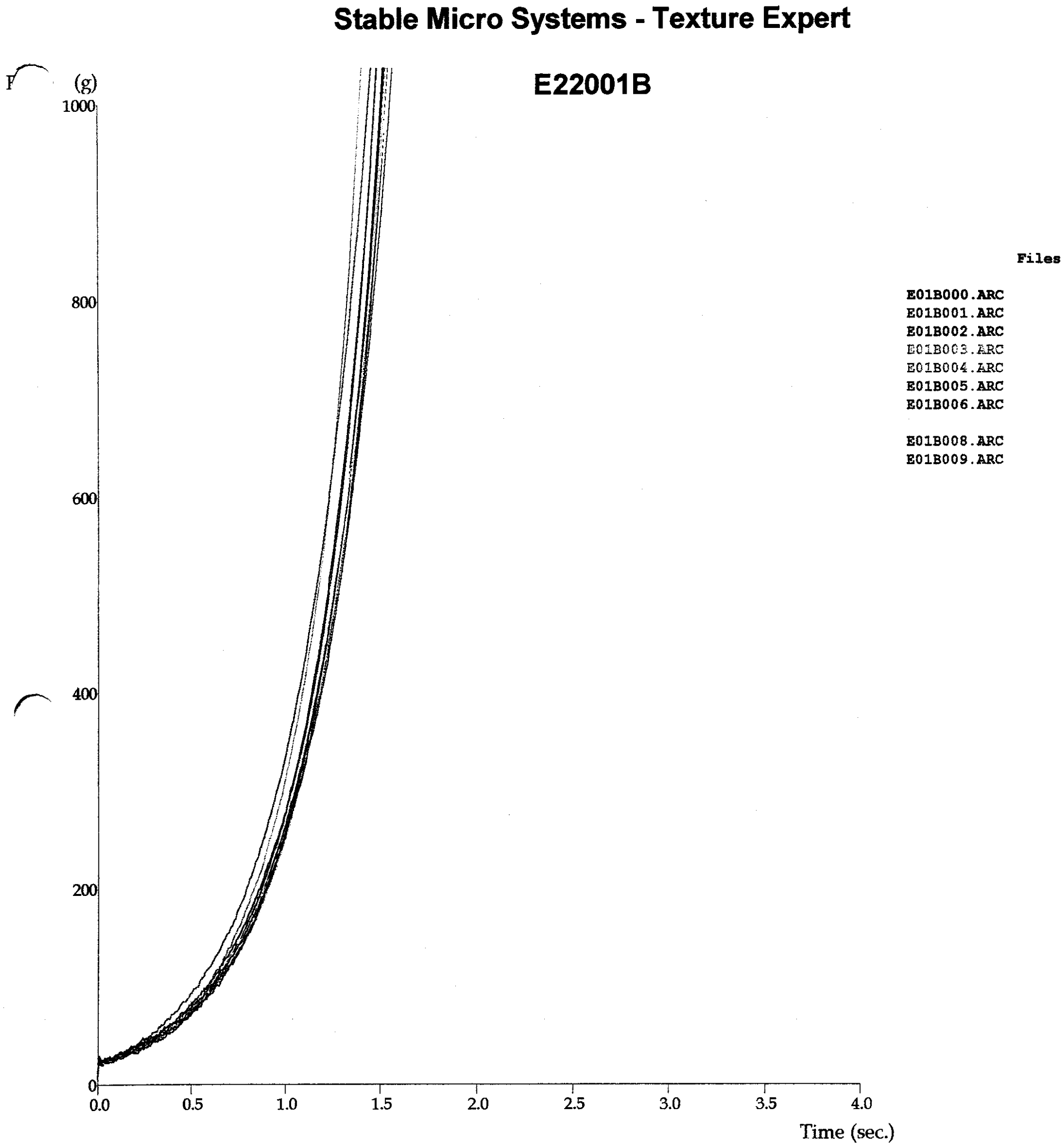
APPENDIX 2. Wet Burst Strength Test Results (Continued)



Test ID	Force 1	Distance 1
	g	mm
E00E000	9848.632	4.190
E00E001	9624.518	4.184
E00E002	10968.580	4.241
E00E003	8811.715	4.105
E00E004	15714.552	4.510
E00E005	11591.485	4.445
E00E006	10367.289	4.186
E00E007	10157.166	4.264
E00E008	10040.332	4.329
E00E009	10779.453	4.236
Average	10790.372	4.269
Std Dev (n-1)	1892.232	0.126
% CV	0.175	0.029

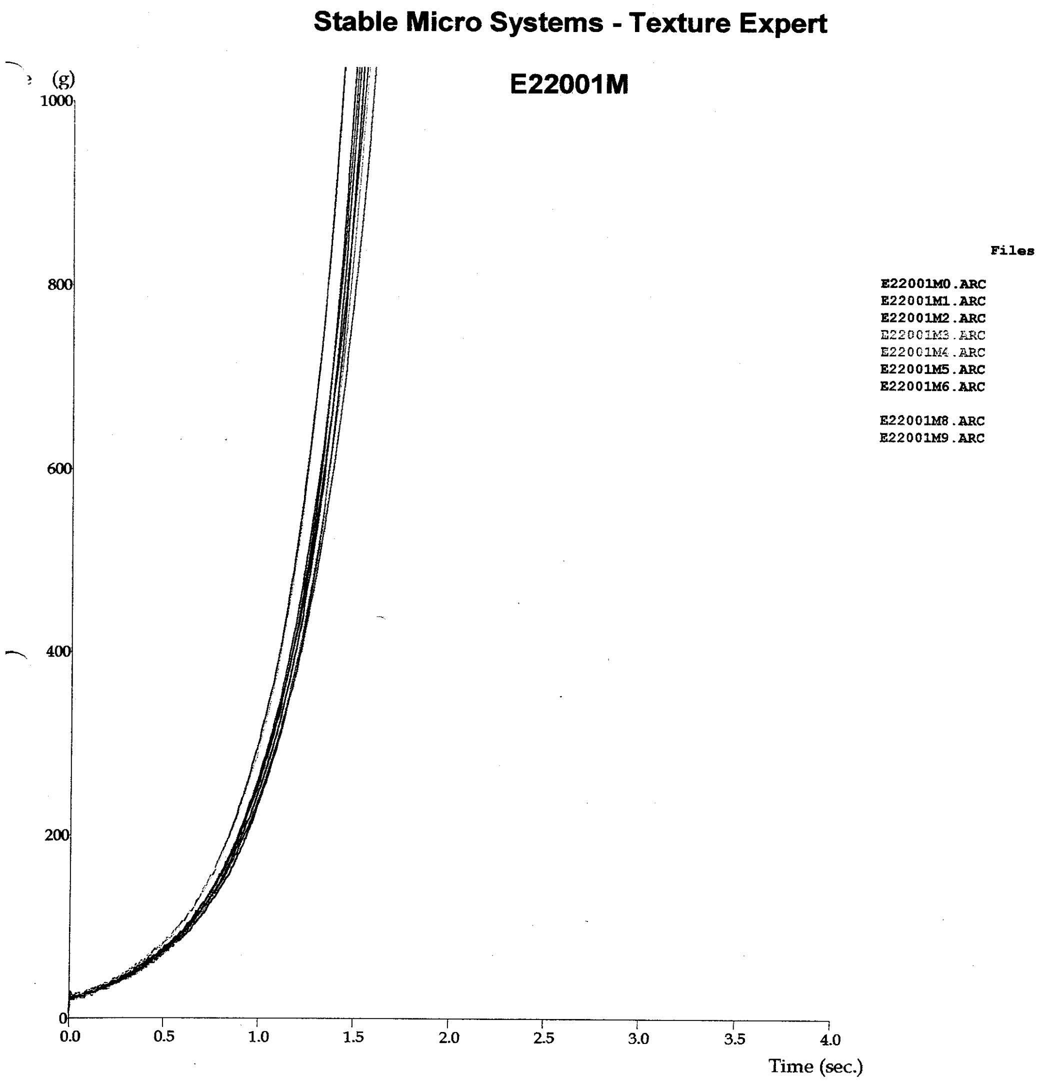
Figure 49. 150 Gelatin Wet Burst Strength Printout at 40 hrs.

APPENDIX 2. Wet Burst Strength Test Results (Continued)



Test ID	Force 1	Distance 1
	g	mm
E01B000	15232.950	4.460
E01B001	9004.426	4.145
E01B002	15755.528	4.587
E01B003	9994.821	4.246
E01B004	7162.510	3.686
E01B005	10297.632	4.138
E01B006	7966.997	4.093
E01B007	14379.959	4.597
E01B008	8507.357	4.202
E01B009	8538.629	4.360
Average	10684.081	4.251
Std Dev (n-1)	3208.358	0.271
% CV	0.300	0.064

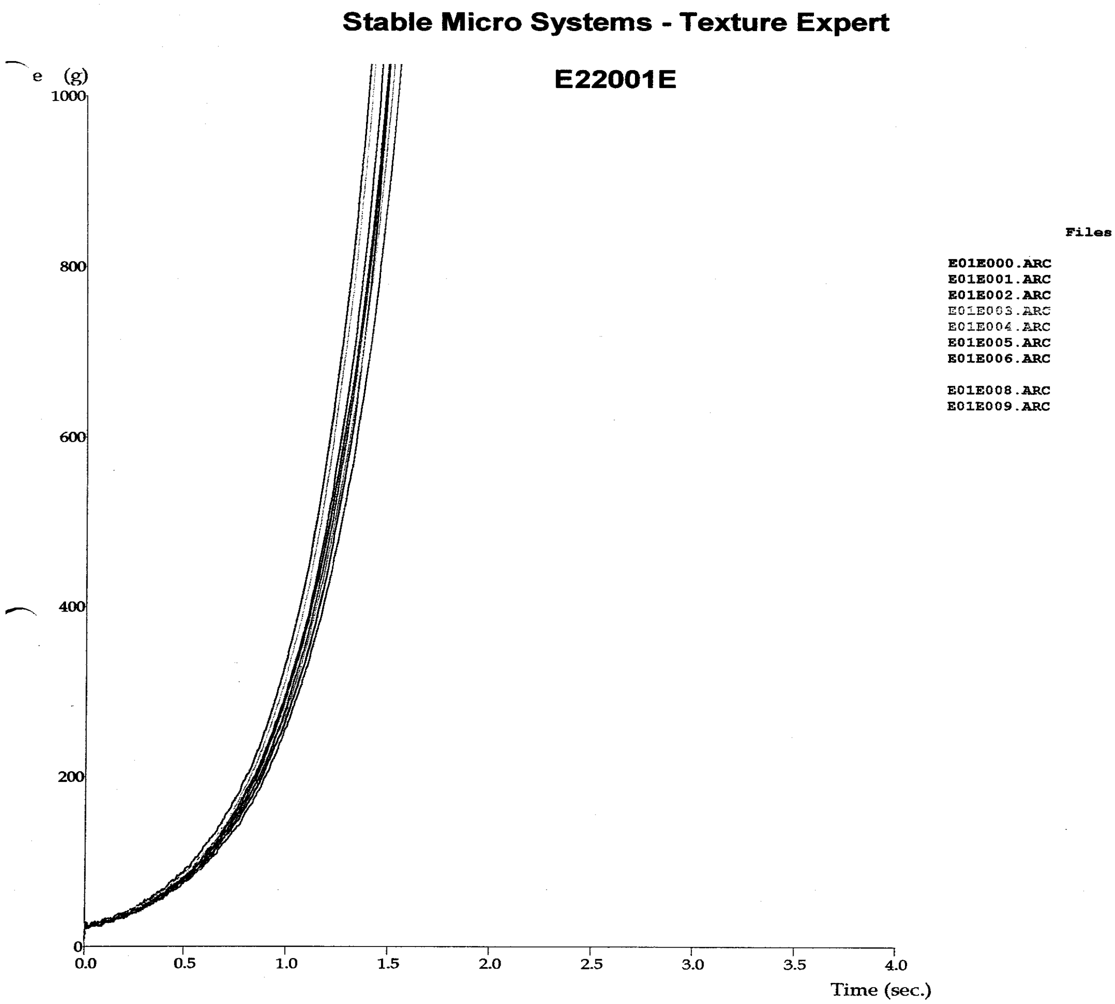
Figure 50. 150 Gelatin Wet Burst Strength Printout at 64 hrs.



Test ID	Force 1 g	Distance 1 mm
E22001M0	8760.940	4.242
E22001M1	10466.259	4.221
E22001M2	10938.151	4.212
E22001M3	7128.463	3.813
E22001M4	11624.182	4.499
E22001M5	6936.326	3.854
E22001M6	17389.271	4.812
E22001M7	7312.922	4.002
E22001M8	14186.122	4.641
E22001M9	3457.633	3.664
Average	9820.027	4.196
Std Dev (n-1)	4010.079	0.374
% CV	0.408	0.089

Figure 51. 150 Gelatin Wet Burst Strength Printout for at 68 hrs.

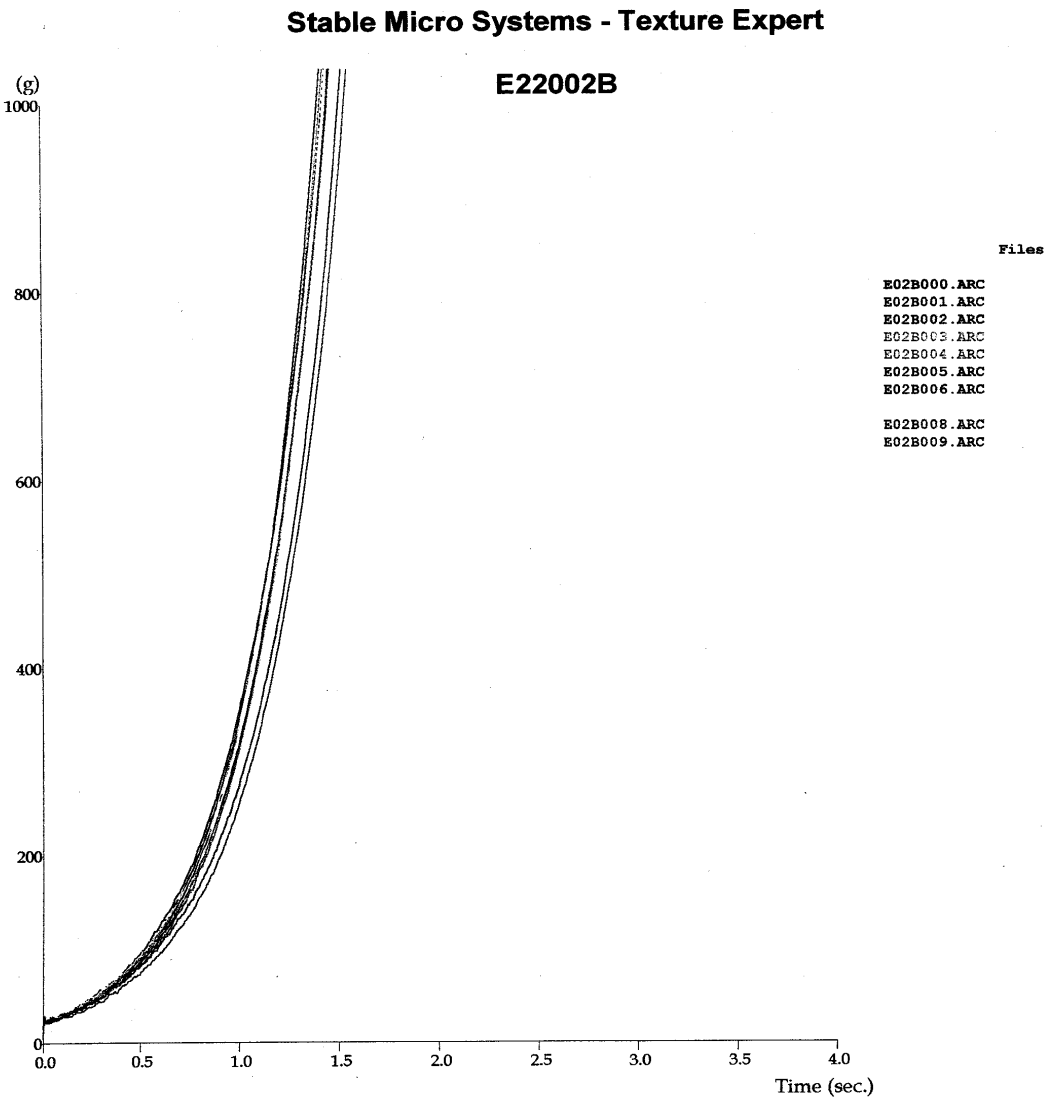
APPENDIX 2. Wet Burst Strength Test Results (Continued)



Test ID	Force 1 g	Distance 1 mm
E01E000	7878.404	4.012
E01E001	7766.084	3.950
E01E002	5833.401	3.669
E01E003	7793.678	3.898
E01E004	16118.120	4.688
E01E005	12752.175	4.523
E01E006	17559.565	4.681
E01E007	10108.719	4.170
E01E008	8001.160	4.105
E01E009	8484.740	4.166
Average	10229.604	4.186
Std Dev (n-1)	3939.104	0.342
% CV	0.385	0.082

Figure 52. 150 Gelatin Wet Burst Strength Printout at 72 hrs.

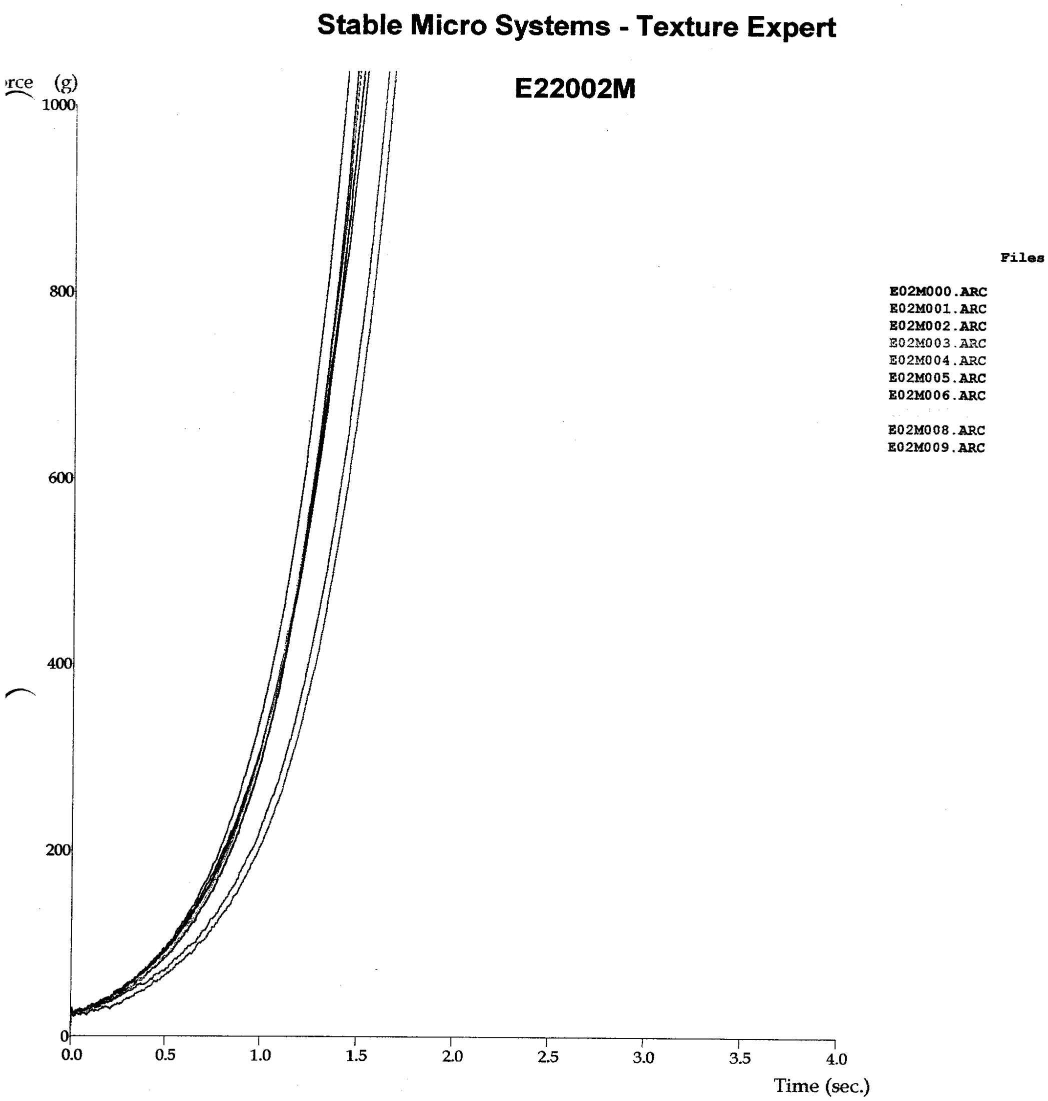
APPENDIX 2. Wet Burst Strength Test Results (Continued)



Test ID	Force 1	Distance 1
	g	mm
E02B000	8058.039	4.078
E02B001	6958.662	3.941
E02B002	8970.541	4.191
E02B003	12464.149	4.356
E02B004	6631.002	4.005
E02B005	11832.781	4.448
E02B006	6491.824	4.028
E02B007	11439.352	4.274
E02B008	5513.613	3.916
E02B009	8406.155	4.298
Average	8676.611	4.154
Std Dev (n-1)	2458.443	0.185
% CV	0.283	0.045

Figure 53. 150 Gelatin Wet Burst Strength Printout at 88 hrs.

APPENDIX 2. Wet Burst Strength Test Results (Continued)

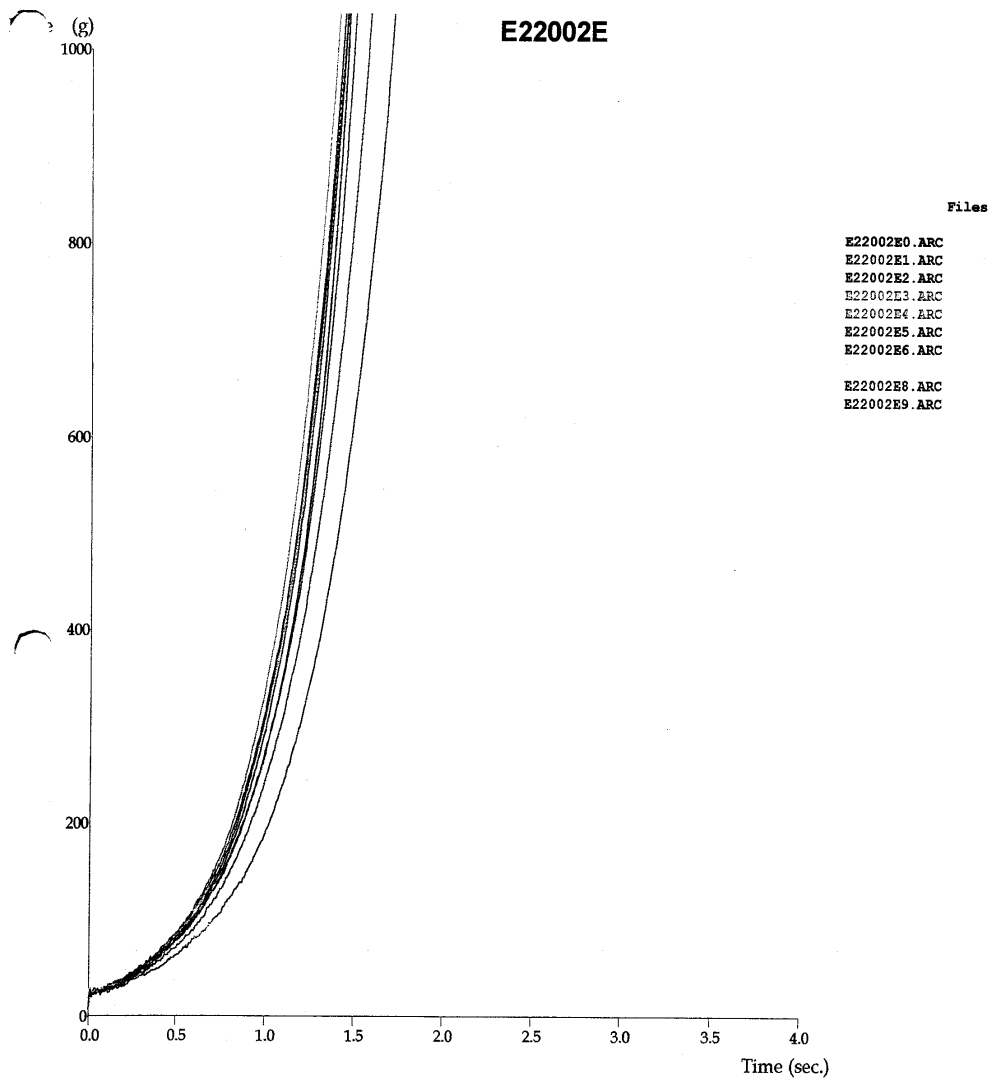


Test ID	Force 1 g	Distance 1 mm
E02M000	24915.581	5.404
E02M001	28066.254	5.655
E02M002	11693.697	4.469
E02M003	3585.448	3.823
E02M004	7353.730	4.108
E02M005	7680.807	4.254
E02M006	7198.103	4.063
E02M007	27230.837	5.804
E02M008	6035.874	4.449
E02M009	6129.265	4.427
Average	12988.960	4.646
Std Dev (n-1)	9726.088	0.708
% CV	0.749	0.152

Figure 54. 150 Gelatin Wet Burst Strength Printout at 92 hrs.

APPENDIX 2. Wet Burst Strength Test Results (Continued)

Stable Micro Systems - Texture Expert



Test ID	Force 1 g	Distance 1 mm
E22002E0	4547.211	3.645
E22002E1	7070.741	3.984
E22002E2	4382.133	3.820
E22002E3	6619.471	3.874
E22002E4	4384.509	3.651
E22002E5	11236.813	4.248
E22002E6	10561.681	4.881
E22002E7	8481.791	5.100
E22002E8	7354.041	4.332
E22002E9	3161.683	3.539
Average	6780.007	4.107
Std Dev (n-1)	2729.793	0.532
% CV	0.403	0.130

Figure 55. 150 Gelatin Wet Burst Strength Printout at 96 hrs.

Appendix 3. Rheology Test Results

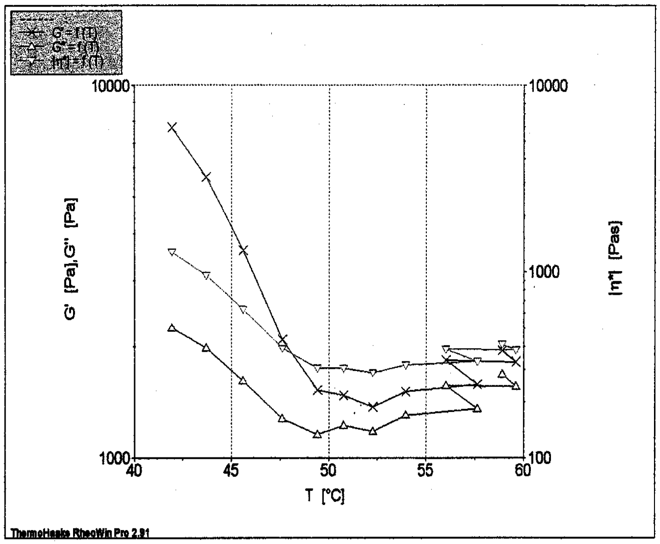
RheoWin Pro 2.91 | Test 1 | Page 1

Company	RP Scherer NA	**Measure device**	RS150
Operator	RP Scherer NA	**Temperature device**	TC81 <---> RS150
Date/Time	19.09.2002 / 16:49:50 PM	**Sensor**	PP35 Ti
Substance	E21995 Beg	**A-factor**	128800.000 Pa/Nm
Sample no		**M-factor**	8.772 (1/s)/(rad/s)
Description			

Gap 1.995 mm

Comment

Moduli for gel free IS 430



Element definition / Notes

CD lin, 40.00 °C - 60.00 °C, t 300.00 s, 0.05 -, f 1.000 H

Filename: ----

Job: C:\Program Files\RheoWin\Jobs\Modulus Test.rwj

	t [s]	t_seg [s]	T [°C]	G' [Pa]	G" [Pa]	|η*| [Pas]
1-1	24.87	17.54	41.95	7691	2235	1275
1-2	51.19	43.87	43.70	5658	1973	953.7
1-3	80.53	73.21	45.60	3607	1609	628.6
1-4	110.5	103.2	47.62	2083	1275	388.7
1-5	137.9	130.6	49.44	1520	1156	304.0
1-6	159.2	151.9	50.77	1470	1221	304.1
1-7	181.9	174.6	52.29	1367	1176	287.1
1-8	205.4	198.1	53.97	1505	1301	316.7
1-9	228.7	221.4	57.63	1574	1351	330.2
1-10	253.0	245.7	56.02	1833	1564	383.5
1-11	276.1	268.7	59.59	1814	1559	380.6
1-12	297.4	290.1	58.89	1947	1681	409.3

Evaluation

CrossOver: no result found

Figure 56. 195 Gelatin Rheological Testing at 0 hrs.

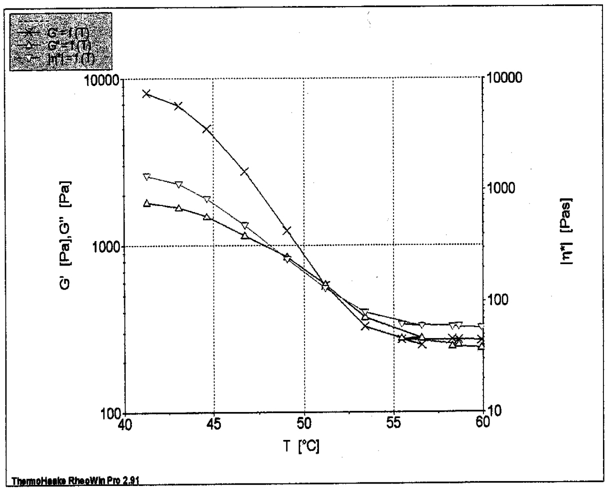
APPENDIX 3. Rheology Test Results (Continued).

RheoWin Pro 2.91 | Test 1 | Page 1

Company	RP Scherer NA	**Measure device**	RS150		
Operator	RP Scherer NA	**Temperature device**	TC81 <---> RS150		
Date/Time	19.09.2002 / 17:22:57 PM	**Sensor**	PP35 Ti	**Gap**	3.629 mm
Substance	E21995 Mid	**A-factor**	128800.000 Pa/Nm		
Sample no		**M-factor**	4.822 (1/s)/(rad/s)		
Description					

Comment

Moduli for gel free IS 430



Element definition / Notes

CD lin, 40.00 °C - 60.00 °C, t 300.00 s, 0.05 -, f 1.000 H

Filename: -----

Job: C:\Program Files\RheoWin\Jobs\Modulus Test.rwj

	t [s]	t_seg [s]	T [°C]	G' [Pa]	G" [Pa]	|η*| [Pas]
1-1	18.67	11.36	41.29	8186	1804	1334
1-2	39.95	32.63	43.05	6846	1674	1122
1-3	66.39	59.08	44.64	4999	1483	830.0
1-4	97.25	89.94	46.72	2774	1142	477.4
1-5	132.6	125.3	49.07	1221	851.0	236.9
1-6	165.8	158.4	51.20	572.9	583.6	130.2
1-7	197.0	189.7	53.40	325.4	370.7	78.50
1-8	223.9	216.6	56.58	251.1	278.2	59.65
1-9	245.2	237.9	55.45	272.2	276.5	61.75
1-10	268.2	260.9	58.64	271.2	253.8	59.12
1-11	291.2	283.9	58.27	274.3	247.6	58.80
1-12	312.5	305.1	59.91	269.7	242.1	57.69

Evaluation

Crossover : G' = G" = 605.1 Pa at ω = 6.283 rad/s f =

Figure 57. 195 Gelatin Rheological Testing at 4 hrs.

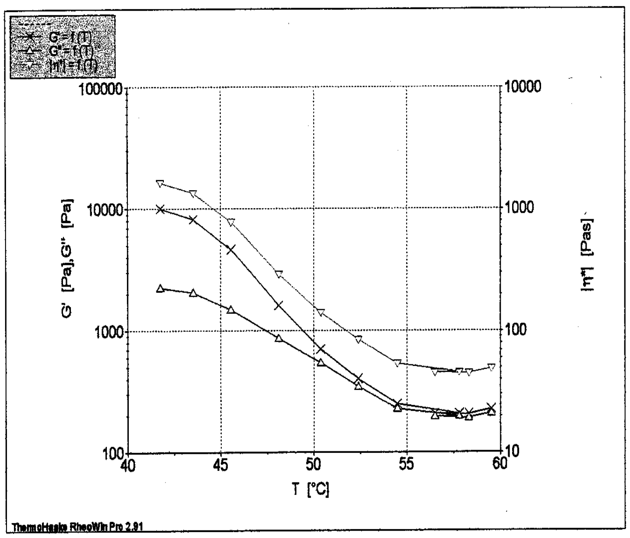
APPENDIX 3. Rheology Test Results (Continued).

RheoWin Pro 2.91 | Test 1 | Page 1

Company	RP Scherer NA	**Measure device**	RS150		
Operator	RP Scherer NA	**Temperature device**	TC81 <---> RS150		
Date/Time	19.09.2002 / 17:37:33 PM	**Sensor**	PP35 Ti	**Gap**	3.183 mm
Substance	E21995 End	**A-factor**	128800.000 Pa/Nm		
Sample no		**M-factor**	5.498 (1/s)/(rad/s)		
Description					

Comment

Moduli for gel free IS 430



Element definition / Notes

CD lin, 40.00 °C - 60.00 °C, t 300.00 s, 0.05 -, f 1.000 H

Filename: -----

Job: C:\Program Files\RheoWin\Jobs\Modulus Test.rwj

	t [s]	t_seg [s]	T [°C]	G' [Pa]	G" [Pa]	|η*| [Pas]
1-1	24.03	16.63	41.76	10060	2243	1640
1-2	48.23	40.83	43.53	8176	2044	1341
1-3	79.64	72.24	45.55	4650	1486	776.9
1-4	118.4	111.0	48.11	1607	864.8	290.5
1-5	152.6	145.2	50.38	703.9	542.7	141.5
1-6	183.6	176.2	52.39	401.1	346.0	84.30
1-7	213.7	206.3	54.49	247.9	226.0	53.39
1-8	237.6	230.2	57.79	206.3	197.9	45.50
1-9	260.0	252.6	56.52	205.9	195.1	45.14
1-10	283.8	276.4	58.31	205.3	191.7	44.71
1-11	305.0	297.6	59.51	226.9	208.6	49.04

Evaluation

CrossOver: no result found

Figure 58. 195 Gelatin Rheological Testing at 8 hrs.

APPENDIX 3. Rheology Test Results (Continued).

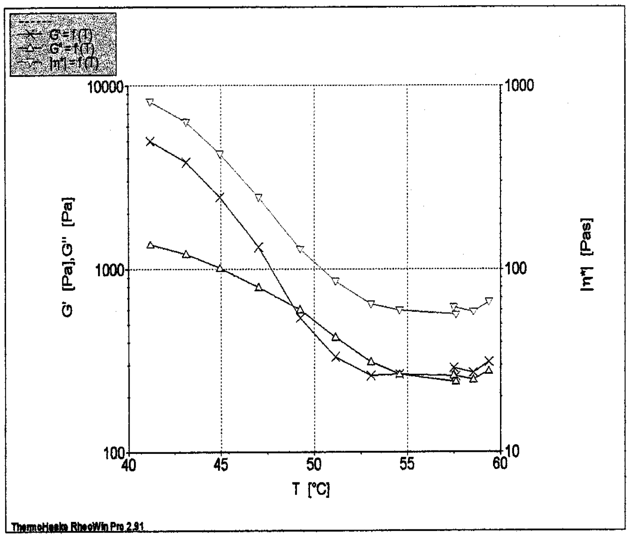
RheoWin Pro 2.91 Test 1 Page 1

Company	RP Scherer NA	**Measure device**	RS150
Operator	RP Scherer NA	**Temperature device**	TC81 <---> RS150
Date/Time	19.09.2002 / 17:54:14 PM	**Sensor**	PP35 Ti
Substance	E21996 Beg	**A-factor**	128800.000 Pa/Nm
Sample no		**M-factor**	4.584 (1/s)/(rad/s)
Description			

Gap 3.818 mm

Comment

Moduli for gel free IS 430



Element definition / Notes

CD lin, 40.00 °C - 60.00 °C, t 300.00 s, 0.05 -, f 1.000 H

Filename: ----

Job: C:\Program Files\RheoWin\Jobs\Modulus Test.rwj

	t [s]	t_seg [s]	T [°C]	G' [Pa]	G" [Pa]	|η*| [Pas]
1-1	18.40	11.00	41.18	4960	1355	818.4
1-2	41.61	34.21	43.13	3787	1208	632.7
1-3	70.60	63.20	44.94	2457	1008	422.6
1-4	102.3	94.88	47.05	1315	795.3	244.6
1-5	135.5	128.1	49.26	538.9	600.0	128.3
1-6	164.7	157.3	51.15	331.5	423.8	85.63
1-7	192.4	185.0	53.05	260.5	310.3	64.47
1-8	214.8	207.4	54.57	265.4	265.3	59.73
1-9	239.2	231.8	57.59	259.0	241.5	56.35
1-10	260.5	253.1	57.48	289.0	261.2	61.99
1-11	281.8	274.4	58.53	271.5	247.7	58.49
1-12	303.0	295.6	59.38	310.8	276.8	66.24

Evaluation

Crossover : G' = G" = 679.9 Pa at ω = 6.283 rad/s f=

Figure 59. 195 Gelatin Rheological Testing at 32 hrs.

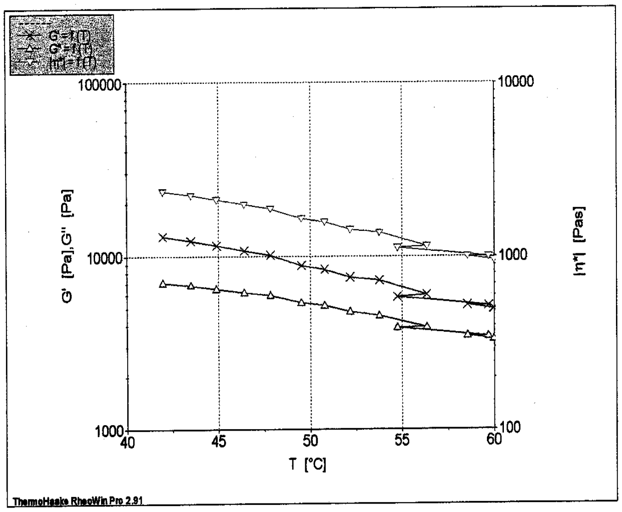
APPENDIX 3. Rheology Test Results (Continued).

RheoWin Pro 2.91 Test 1 Page 1

Company	RP Scherer NA	**Measure device**	RS150		
Operator	RP Scherer NA	**Temperature device**	TC81 <---> RS150		
Date/Time	19.09.2002 / 18:12:18 PM	**Sensor**	PP35 Ti	**Gap**	3.334 mm
Substance	E21996 Mid	**A-factor**	128800.000 Pa/Nm		
Sample no		**M-factor**	5.249 (1/s)/(rad/s)		
Description					

Comment

Moduli for gel free IS 430



Element definition / Notes

CD lin, 40.00 °C - 60.00 °C, t 300.00 s, 0.05 -, f 1.000 H

Filename: ----

Job: C:\Program Files\RheoWin\Jobs\Modulus Test.rwj

	t [s]	t_seg [s]	T [°C]	G' [Pa]	G" [Pa]	|η*| [Pas]
1-1	26.26	19.01	41.96	12970	7025	2348
1-2	47.94	40.69	43.49	12250	6804	2231
1-3	69.89	62.64	44.90	11540	6469	2105
1-4	92.52	85.27	46.41	10770	6183	1977
1-5	114.2	106.9	47.85	10150	5960	1874
1-6	139.3	132.0	49.54	8836	5392	1647
1-7	159.8	152.6	50.79	8407	5207	1574
1-8	180.4	173.1	52.17	7561	4784	1424
1-9	203.2	195.9	53.78	7274	4543	1365
1-10	226.3	219.1	56.34	6019	3890	1141
1-11	249.4	242.2	54.75	5828	3870	1113
1-12	271.5	264.2	59.70	5211	3452	994.8
1-13	294.3	287.1	58.55	5226	3495	1001
1-14	315.0	307.7	59.96	4991	3334	955.2

Evaluation

CrossOver: no result found

Figure 60. 195 Gelatin Rheological Testing at 36 hrs.

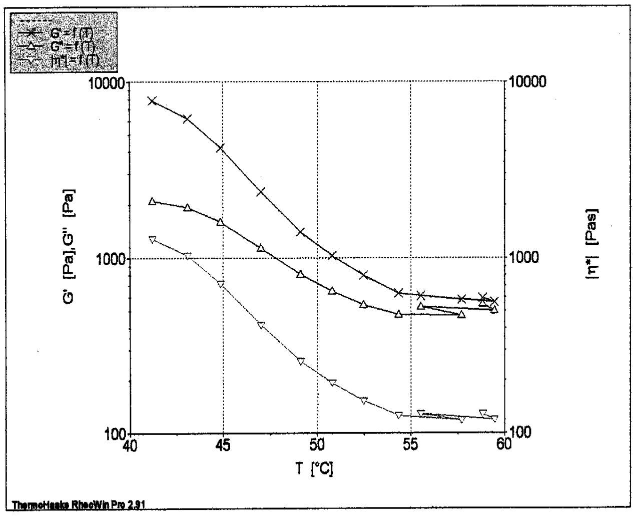
APPENDIX 3. Rheology Test Results (Continued).

RheoWin Pro 2.91 | Test 1 | Page 1

Company	RP Scherer NA	**Measure device**	RS150		
Operator	RP Scherer NA	**Temperature device**	TC81 <---> RS150		
Date/Time	19.09.2002 / 18:49:57 PM	**Sensor**	PP35 Ti	**Gap**	2.738 mm
Substance	E21996 Bcl	**A-factor**	128800.000 Pa/Nm		
Sample no		**M-factor**	6.392 (1/s)/(rad/s)		
Description					

Comment

Moduli for gel free IS 430



Element definition / Notes

CD lin, 40.00 °C - 60.00 °C, t 300.00 s, 0.05 -, f 1.000 H

Filename: -----

Job: C:\Program Files\RheoWin\Jobs\Modulus Test.rwj

	t [s]	t_seg [s]	T [°C]	G' [Pa]	G" [Pa]	|η*| [Pas]
1-1	18.72	11.22	41.22	7784	2098	1283
1-2	41.52	34.02	43.11	6185	1926	1031
1-3	69.38	61.88	44.87	4207	1592	715.9
1-4	101.6	94.09	47.01	2355	1136	416.1
1-5	132.9	125.4	49.09	1400	807.8	257.2
1-6	159.4	151.9	50.79	1029	650.0	193.7
1-7	185.1	177.6	52.48	792.8	541.7	152.8
1-8	211.4	203.9	54.34	627.1	476.0	125.3
1-9	233.5	226.0	57.70	577.2	470.5	118.5
1-10	254.8	247.3	55.54	608.0	527.4	128.1
1-11	276.1	268.6	59.46	559.3	500.4	119.4
1-12	297.4	289.9	58.83	590.7	546.2	128.0

Evaluation

CrossOver: no result found

Figure 61. 195 Gelatin Rheological Testing at 40 hrs.

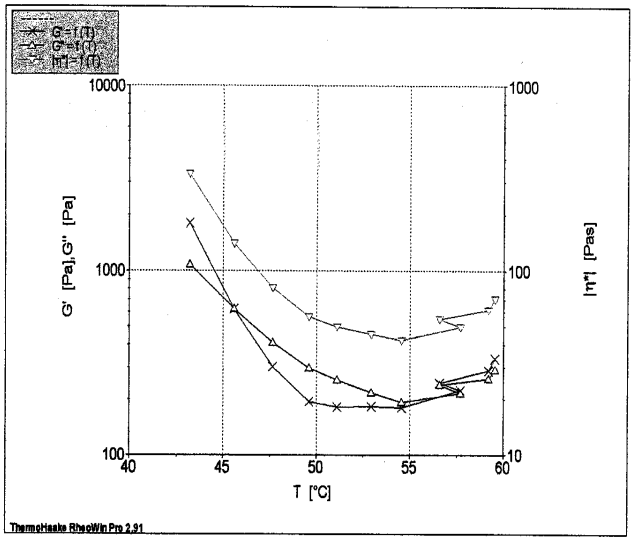
APPENDIX 3. Rheology Test Results (Continued).

RheoWin Pro 2.91 | Test 1 | Page 1

Company	RP Scherer NA	Measure device	RS150		
Operator	RP Scherer NA	Temperature device	TC81 <---> RS150		
Date/Time	19.09.2002 / 15:08:48 PM	Sensor	PP35 Ti	Gap	0.601 mm
Substance	E21997 Beg	A-factor	128800.000 Pa/Nm		
Sample no		M-factor	29.118 (1/s)/(rad/s)		
Description					

Comment

Moduli for gel free IS 430



Element definition / Notes

CD lin, 40.00 °C - 60.00 °C, t 300.00 s, 0.05 -, f 1.000 H

Filename: -----

Job: C:\Program Files\RheoWin\Jobs\Modulus Test.rwj

	t [s]	t_seg [s]	T [°C]	G' [Pa]	G" [Pa]	|η*| [Pas]
1-1	42.74	35.48	43.20	1792	1077	332.7
1-2	80.05	72.79	45.59	621.2	623.3	140.1
1-3	111.4	104.1	47.65	302.6	408.3	80.89
1-4	140.2	132.9	49.61	194.4	296.9	56.48
1-5	165.7	158.4	51.12	181.7	255.4	49.88
1-6	191.0	183.8	52.94	182.9	218.1	45.30
1-7	214.1	206.8	54.55	179.9	193.2	42.02
1-8	236.5	229.2	57.68	224.8	215.6	49.57
1-9	260.4	253.1	56.58	246.5	240.4	54.80
1-10	283.2	276.0	59.19	287.5	259.5	61.64
1-11	306.8	299.5	59.53	334.4	289.9	70.44

Evaluation

Crossover : G' = G" = 629.5 Pa at ω = 6.283 rad/s f =

Figure 62. 195 Gelatin Rheological Testing at 64 hrs.

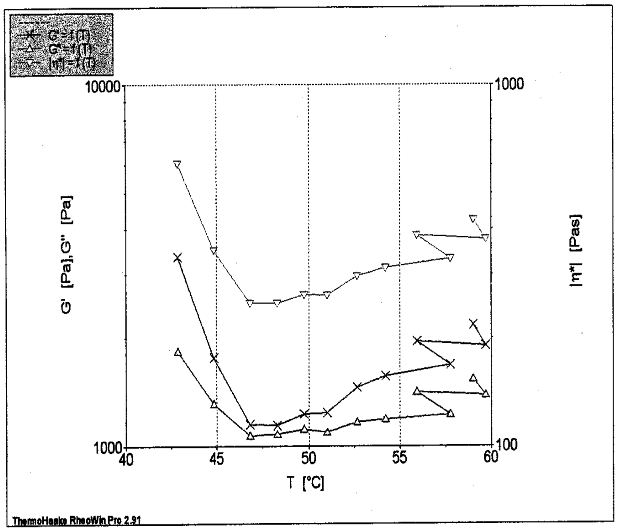
APPENDIX 3. Rheology Test Results (Continued).

RheoWin Pro 2.91 | Test 1 | Page 1

Company	RP Scherer NA	**Measure device**	RS150		
Operator	RP Scherer NA	**Temperature device**	TC81 <---> RS150		
Date/Time	19.09.2002 / 15:23:48 PM	**Sensor**	PP35 Ti	**Gap**	1.336 mm
Substance	E21997 Mid	**A-factor**	128800.000 Pa/Nm		
Sample no		**M-factor**	13.099 (1/s)/(rad/s)		
Description					

Comment

Moduli for gel free IS 430



Element definition / Notes

CD lin, 40.00 °C - 60.00 °C, t 300.00 s, 0.05 -, f 1.000 H

Filename: ----

Job: C:\Program Files\RheoWin\Jobs\Modulus Test.rwj

	t [s]	t_seg [s]	T [°C]	G' [Pa]	G" [Pa]	|η*| [Pas]
1-1	37.86	30.48	42.87	3338	1829	605.8
1-2	69.22	61.84	44.85	1760	1308	349.0
1-3	98.93	91.55	46.83	1148	1068	249.5
1-4	121.3	113.9	48.32	1140	1077	249.6
1-5	142.6	135.2	49.75	1225	1111	263.3
1-6	163.8	156.4	51.02	1235	1089	262.0
1-7	187.4	180.0	52.64	1452	1165	296.3
1-8	209.7	202.3	54.22	1564	1184	312.2
1-9	230.9	223.6	57.75	1680	1223	330.8
1-10	254.7	247.3	55.94	1951	1411	383.2
1-11	277.0	269.6	59.69	1899	1382	373.9
1-12	298.3	290.9	59.01	2168	1532	422.4

Evaluation

CrossOver: no result found

Figure 63. 195 Gelatin Rheological Testing at 68 hrs.

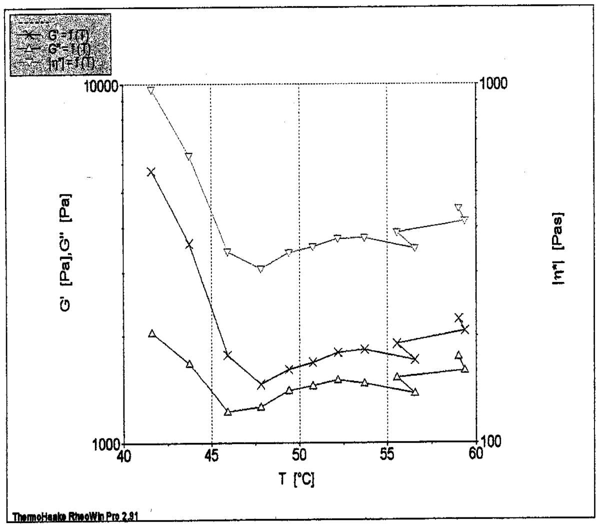
APPENDIX 3. Rheology Test Results (Continued).

RheoWin Pro 2.91	Test 1	Page 1

Company	RP Scherer NA	Measure device	RS150		
Operator	RP Scherer NA	Temperature device	TC81 <---> RS150		
Date/Time	19.09.2002 / 15:40:09 PM	Sensor	PP35 Ti	Gap	1.577 mm
Substance	E21997 End	A-factor	128800.000 Pa/Nm		
Sample no		M-factor	11.097 (1/s)/(rad/s)		
Description					

Comment

Moduli for gel free IS 430



Element definition / Notes

CD lin, 40.00 °C - 60.00 °C, t 300.00 s, 0.05 -, f 1.000 H

Filename: -----

Job: C:\Program Files\RheoWin\Jobs\Modulus Test.rwj

	t [s]	t_seg [s]	T [°C]	G' [Pa]	G" [Pa]	|η*| [Pas]
1-1	22.38	15.03	41.61	5717	2033	965.8
1-2	52.13	44.78	43.75	3598	1667	631.0
1-3	85.13	77.78	45.92	1762	1224	341.4
1-4	113.2	105.9	47.81	1457	1261	306.7
1-5	137.4	130.1	49.40	1603	1401	338.9
1-6	159.5	152.1	50.77	1682	1444	352.8
1-7	180.8	173.4	52.18	1789	1498	371.4
1-8	202.1	194.7	53.70	1823	1468	372.4
1-9	223.4	216.1	56.56	1708	1373	348.7
1-10	245.7	238.3	55.55	1892	1522	386.5
1-11	272.6	265.3	59.37	2058	1593	414.2
1-12	298.4	291.0	59.02	2223	1743	449.6

Evaluation

CrossOver: no result found

Figure 64. 195 Gelatin Rheological Testing at 72 hrs.

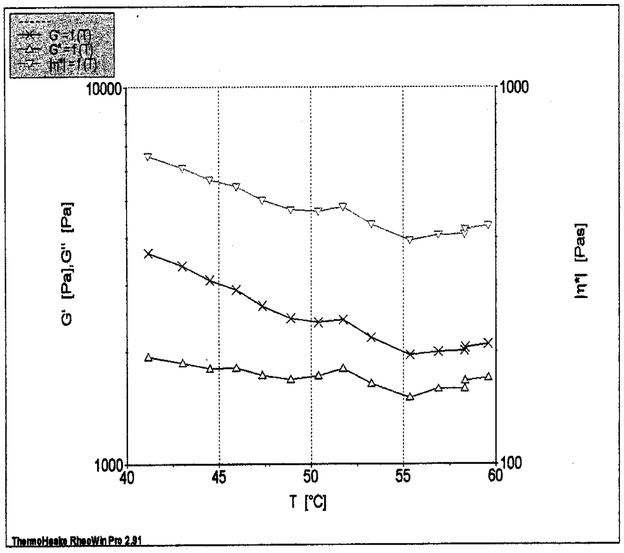
APPENDIX 3. Rheology Test Results (Continued).

RheoWin Pro 2.91 | Test 1 | Page 1

Company	RP Scherer NA	Measure device	RS150		
Operator	RP Scherer NA	Temperature device	TC81 <--> RS150		
Date/Time	19.09.2002 / 15:54:53 PM	Sensor	PP35 Ti	Gap	2.058 mm
Substance	E21998 Beg	A-factor	128800.000 Pa/Nm		
Sample no		M-factor	8.503 (1/s)/(rad/s)		
Description					

Comment

Moduli for gel free IS 430



Element definition / Notes

CD lin, 40.00 °C - 60.00 °C, t 300.00 s, 0.05 -, f 1.000 H

Filename: ----

Job: C:\Program Files\RheoWin\Jobs\Modulus Test.rwj

	t [s]	t_seg [s]	T [°C]	G' [Pa]	G" [Pa]	|η*| [Pas]
1-1	18.17	10.90	41.16	3629	1931	654.2
1-2	39.43	32.16	43.01	3350	1858	609.7
1-3	64.01	56.74	44.50	3079	1796	567.4
1-4	85.61	78.34	45.94	2906	1806	544.6
1-5	106.9	99.62	47.36	2637	1722	501.2
1-6	129.9	122.7	48.90	2446	1681	472.4
1-7	152.9	145.7	50.40	2383	1718	467.6
1-8	174.2	167.0	51.73	2424	1799	480.5
1-9	195.5	188.2	53.25	2169	1634	432.2
1-10	219.2	211.9	55.36	1956	1502	392.4
1-11	241.5	234.2	56.89	1992	1584	405.0
1-12	262.7	255.5	58.30	2005	1587	407.0
1-13	284.0	276.7	58.34	2047	1665	420.0
1-14	305.3	298.0	59.59	2088	1698	428.4

Evaluation

CrossOver: no result found

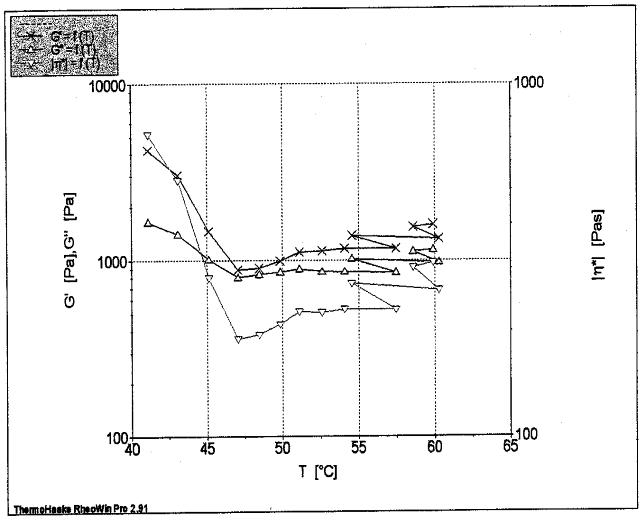
Figure 65. 195 Gelatin Rheological Testing at 88 hrs.

RheoWin Pro 2.91 Test 1 Page 1

Company	RP Scherer NA	**Measure device**	RS150		
Operator	RP Scherer NA	**Temperature device**	TC81 <---> RS150		
Date/Time	19.09.2002 / 16:19:29 PM	**Sensor**	PP35 Ti	**Gap**	0.885 mm
Substance	E21998 Mid	**A-factor**	128800.000 Pa/Nm		
Sample no		**M-factor**	19.774 (1/s)/(rad/s)		
Description					

Comment

Moduli for gel free IS 430



Element definition / Notes

CD lin, 40.00 °C - 60.00 °C, t 300.00 s, 0.05 -, f 1.000 H

Filename: ----

Job: C:\Program Files\RheoWin\Jobs\Modulus Test.rwj

	t [s]	t_seg [s]	T [°C]	G' [Pa]	G" [Pa]	|η*| [Pas]
1-1	18.20	10.70	41.09	4195	1641	716.9
1-2	41.01	33.51	43.09	3031	1405	531.7
1-3	73.38	65.88	45.13	1457	1008	282.1
1-4	102.4	94.93	47.06	885.2	797.7	189.7
1-5	123.4	115.9	48.48	900.1	831.3	195.0
1-6	144.4	136.9	49.85	991.2	858.5	208.7
1-7	165.4	157.9	51.12	1111	890.3	226.6
1-8	186.4	178.9	52.61	1126	860.4	225.6
1-9	207.5	200.0	54.08	1159	860.0	229.7
1-10	228.5	221.0	57.46	1163	851.8	229.4
1-11	251.9	244.4	54.57	1374	1016	271.9
1-12	273.9	266.4	60.29	1318	970.6	260.5
1-13	294.9	287.4	58.60	1533	1114	301.6
1-14	315.9	308.4	59.90	1587	1141	311.1

Evaluation

CrossOver: no result found

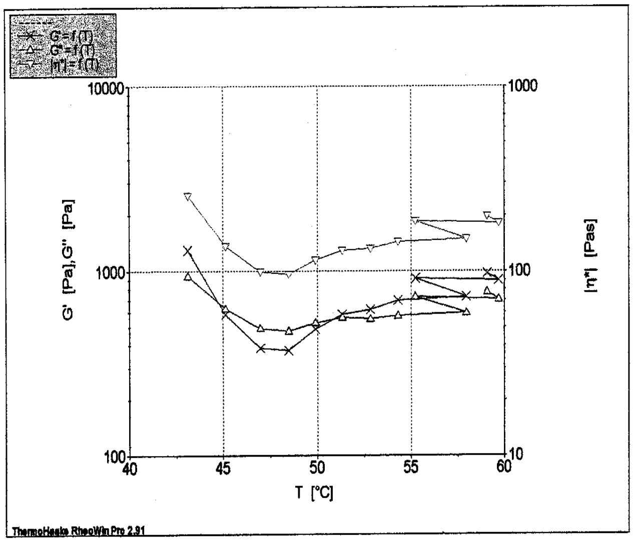
Figure 66. 195 Gelatin Rheological Testing at 92 hrs.

RheoWin Pro 2.91 | Test 1 | Page 1

Company	RP Scherer NA	**Measure device**	RS150		
Operator	RP Scherer NA	**Temperature device**	TC81 <---> RS150		
Date/Time	19.09.2002 / 16:37:07 PM	**Sensor**	PP35 Ti	**Gap**	0.600 mm
Substance	E21998 End	**A-factor**	128800.000 Pa/Nm		
Sample no		**M-factor**	29.167 (1/s)/(rad/s)		
Description					

Comment

Moduli for gel free IS 430



Element definition / Notes

CD lin, 40.00 °C - 60.00 °C, t 300.00 s, 0.05 -, f 1.000 H

Filename: -----

Job: C:\Program Files\RheoWin\Jobs\Modulus Test.rwj

	t [s]	t_seg [s]	T [°C]	G' [Pa]	G" [Pa]	|η*| [Pas]
1-1	41.30	33.96	43.12	1294	936.0	254.2
1-2	73.52	66.18	45.15	581.9	626.4	136.1
1-3	101.2	93.88	46.99	383.4	491.4	99.19
1-4	123.6	116.3	48.49	372.7	474.1	95.98
1-5	144.5	137.2	49.90	487.3	525.9	114.1
1-6	168.4	161.1	51.34	583.5	560.2	128.7
1-7	189.6	182.2	52.83	619.5	550.7	131.9
1-8	210.4	203.1	54.29	693.0	574.0	143.2
1-9	231.6	224.3	57.95	726.0	593.7	149.3
1-10	255.9	248.6	55.24	907.8	724.9	184.9
1-11	276.9	269.6	59.69	891.0	702.8	180.6
1-12	300.7	293.4	59.08	969.4	771.5	197.2

Evaluation

Crossover : G' = G" = 715.7 Pa at ω = 6.283 rad/s f =

Figure 67. 195 Gelatin Rheological Testing at 96 hrs.

Page: 1 ThermoHaake RheoWin 12/20/02 / 3:51 PM

C:\Program Files\RheoWin\Data\E21999B.rwd
Company / Operator: RP Scherer NA / RP Scherer NA
Date / Time / Version: 20.12.2002 / 10:39:30 AM / RheoWin Pro 291
Substance / Sample no: E21999B / 1

	t [s]	t seg [s]	τ [Pa]	$\dot{\gamma}$ [1/s]	T [°C]	η [Pas]	G' [Pa]	G" [Pa]
1 - 1	57.25	49.59	357.9		44.0		992.8	993.4
1 - 2	96.87	89.21	0.390		46.7		4.573	0.150
1 - 3	118.3	110.6	0.390		48.1		4.566	0.151
1 - 4	139.8	132.2	0.390		49.5		4.635	0.128
1 - 5	161.1	153.4	0.390		50.9		4.570	0.151
1 - 6	182.5	174.8	0.390		52.3		4.481	0.130
1 - 7	203.8	196.2	0.390		53.8		4.554	0.152
1 - 8	225.1	217.5	0.390		56.6		4.570	0.150
1 - 9	246.5	238.9	0.390		55.4		4.472	0.125
1 - 10	267.8	260.1	0.390		59.0		4.661	0.109
1 - 11	289.1	281.4	0.390		58.6		4.517	0.145
1 - 12	310.3	302.7	0.390		59.7		4.543	0.152

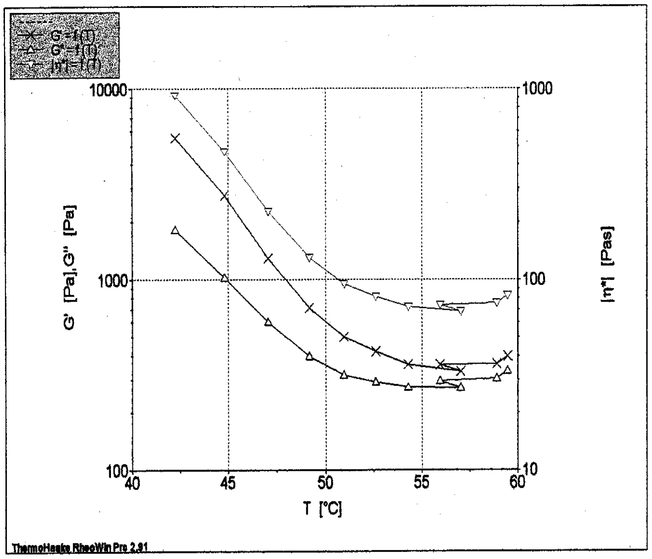
Figure 68. 150 Gelatin Rheological Testing at 0 hrs.

Company	RP Scherer NA	**Measure device**	RS150
Operator	RP Scherer NA	**Temperature device**	TC81 <--> RS150
Date/Time	20.12.2002 / 11:33:49 AM	**Sensor**	PP35 Ti
Substance	E21999M	**A-factor**	128800.000 Pa/Nm
Sample no	1	**M-factor**	8.616 (1/s)/(rad/s)
Description			

Gap 2.031 mm

Comment

Moduli for gel free IS 430



Element definition / Notes

CD lin, 40.00 °C - 60.00 °C, t 300.00 s, 0.05 -, f 1.000 H

Filename: ----

Job: C:\Program Files\RheoWin\Jobs\Modulus Test.rwj

	t [s]	t_seg [s]	T [°C]	G' [Pa]	G" [Pa]	|η*| [Pas]
1-1	34.97	27.52	42.25	5515	1812	923.8
1-2	68.55	61.10	44.81	2756	1017	467.6
1-3	102.2	94.80	47.07	1291	601.9	226.7
1-4	134.0	126.6	49.15	711.1	396.0	129.5
1-5	162.3	154.9	50.95	502.5	315.0	94.39
1-6	186.4	179.0	52.62	419.6	291.3	81.30
1-7	210.8	203.3	54.29	360.0	272.8	71.88
1-8	234.7	227.3	57.04	330.9	271.8	68.16
1-9	257.1	249.7	55.96	357.8	294.7	73.78
1-10	278.4	270.9	58.91	361.8	303.4	75.14
1-11	303.2	295.7	59.48	397.5	333.6	82.59

Evaluation

CrossOver: no result found

Figure 69. 150 Gelatin Rheological Testing at 4 hrs.

APPENDIX 3. Rheology Test Results (Continued).

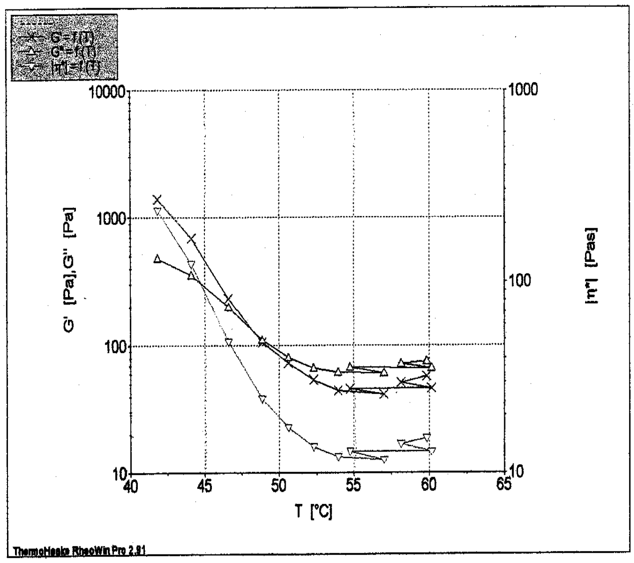
RheoWinPro 2.91 | Test 1 | Page 1

Company	RP Scherer NA	**Measure device**	RS150
Operator	RP Scherer NA	**Temperature device**	TC81 <--> RS150
Date/Time	20.12.2002 / 11:49:12 AM	**Sensor**	PP35 Ti
Substance	E21999E	**A-factor**	128800.000 Pa/Nm
Sample no	1	**M-factor**	7.219 (1/s)/(rad/s)
Description			

Gap 2.424 mm

Comment

Moduli for gel free IS 430



Element definition / Notes

CD lin, 40.00 °C - 60.00 °C, t 300.00 s, 0.05 -, f 1.000 H

Filename: ----

Job: C:\Program Files\RheoWin\Jobs\Modulus Test.rwj

	t [s]	t_seg [s]	T [°C]	G' [Pa]	G" [Pa]	|η*| [Pas]
1-1	25.43	18.11	41.81	1392	479.5	234.3
1-2	57.54	50.22	44.10	690.9	351.4	123.4
1-3	95.40	88.08	46.61	232.4	198.9	48.68
1-4	129.1	121.8	48.86	106.3	110.2	24.37
1-5	156.6	149.3	50.61	72.87	80.33	17.26
1-6	182.5	175.2	52.31	53.87	66.59	13.63
1-7	205.7	198.4	53.96	44.38	61.32	12.05
1-8	226.4	219.1	57.01	41.31	60.63	11.68
1-9	247.1	239.8	54.77	45.48	67.29	12.93
1-10	267.8	260.4	60.16	46.30	66.46	12.89
1-11	288.4	281.0	58.15	51.45	72.25	14.12
1-12	309.1	301.8	59.84	57.43	75.69	15.12

Evaluation

Crossover : G' = G" = 130.3 Pa at ω = 6.283 rad/s f =

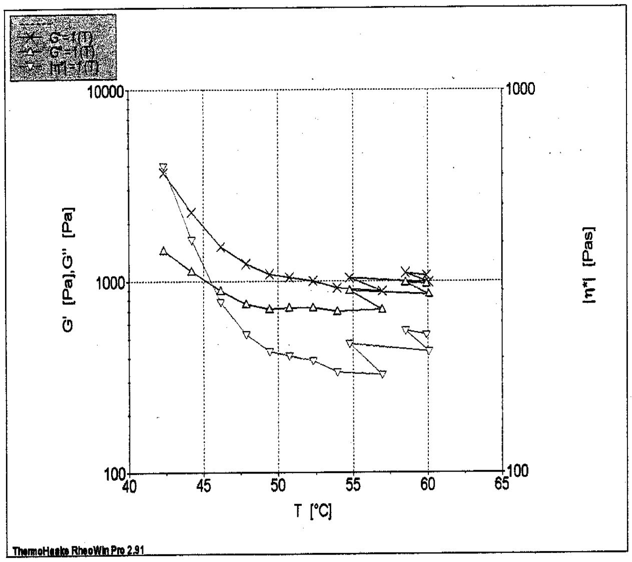
Figure 70. 150 Gelatin Rheological Testing at 8 hrs.

RheoWin Pro 2.91 | Test 1 | Page 1

Company	RP Scherer NA	**Measure device**	RS150		
Operator	RP Scherer NA	**Temperature device**	TC81 <---> RS150		
Date/Time	20.12.2002 / 13:19:01 PM	**Sensor**	PP35 Ti	**Gap**	1.303 mm
Substance	E22000B	**A-factor**	128800.000 Pa/Nm		
Sample no	1	**M-factor**	13.431 (1/s)/(rad/s)		
Description					

Comment

Moduli for gel free IS 430



Element definition / Notes

CD lin, 40.00 °C - 60.00 °C, t 300.00 s, 0.05 -, f 1.000 H

Filename: ----

Job: C:\Program Files\RheoWin\Jobs\Modulus Test.rwj

	t [s]	t_seg [s]	T [°C]	G' [Pa]	G" [Pa]	|η*| [Pas]
1-1	30.55	23.19	42.33	3698	1444	631.9
1-2	59.41	52.05	44.21	2286	1122	405.3
1-3	88.93	81.57	46.18	1511	890.9	279.2
1-4	114.6	107.3	47.87	1227	759.2	229.7
1-5	137.5	130.2	49.42	1087	716.3	207.2
1-6	158.9	151.5	50.76	1040	727.9	202.1
1-7	182.4	175.1	52.32	996.5	726.4	196.3
1-8	205.2	197.9	53.95	916.8	693.1	182.9
1-9	226.5	219.1	57.00	881.6	710.3	180.2
1-10	247.8	240.4	54.77	1036	888.2	217.1
1-11	269.0	261.7	60.09	985.4	853.3	207.5
1-12	291.9	284.6	58.50	1104	983.7	235.3
1-13	314.9	307.5	59.92	1070	966.1	229.4

Evaluation

CrossOver: no result found

Figure 71. 150 Gelatin Rheological Testing at 32 hrs.

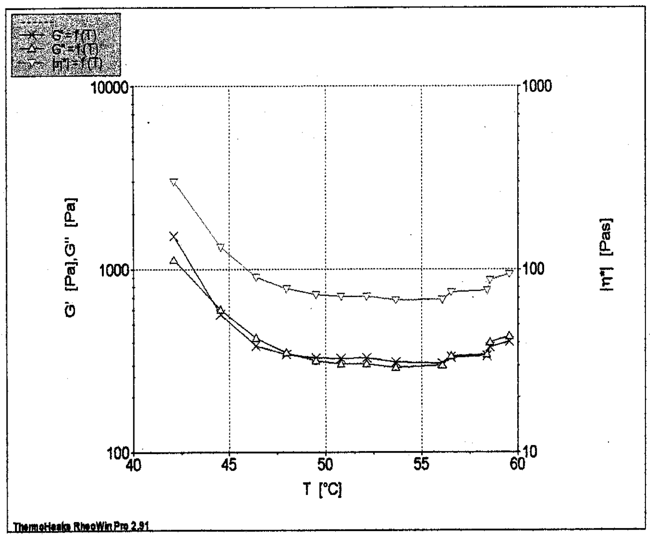
APPENDIX 3. Rheology Test Results (Continued).

RheoWin Pro 2.91 | Test 1 | Page 1

Company	RP Scherer NA	**Measure device**	RS150		
Operator	RP Scherer NA	**Temperature device**	TC81 <---> RS150		
Date/Time	20.12.2002 / 13:42:48 PM	**Sensor**	PP35 Ti	**Gap**	0.894 mm
Substance	E22000M	**A-factor**	128800.000 Pa/Nm		
Sample no	1	**M-factor**	19.575 (1/s)/(rad/s)		
Description					

Comment

Moduli for gel free IS 430



Element definition / Notes

CD lin, 40.00 °C - 60.00 °C, t 300.00 s, 0.05 -, f 1.000 H

Filename: ----

Job: C:\Program Files\RheoWin\Jobs\Modulus Test.rwj

	t [s]	t_seg [s]	T [°C]	G' [Pa]	G" [Pa]	|η*| [Pas]
1-1	28.54	21.11	42.09	1515	1122	300.0
1-2	64.53	57.10	44.55	567.0	601.8	131.6
1-3	92.63	85.20	46.40	383.0	420.2	90.48
1-4	116.1	108.7	47.98	342.3	351.3	78.06
1-5	139.2	131.8	49.51	328.0	315.5	72.43
1-6	159.9	152.5	50.80	326.4	303.3	70.91
1-7	180.7	173.3	52.16	327.7	303.3	71.07
1-8	201.5	194.0	53.65	310.4	289.8	67.59
1-9	222.2	214.7	56.05	306.7	298.5	68.11
1-10	242.9	235.5	56.52	331.6	335.4	75.07
1-11	263.7	256.3	58.37	337.0	342.1	76.43
1-12	284.5	277.0	58.52	376.6	398.2	87.23
1-13	307.6	300.2	59.54	402.5	430.8	93.83

Evaluation

Crossover : G' = G" = 725.5 Pa at ω = 6.283 rad/s f =

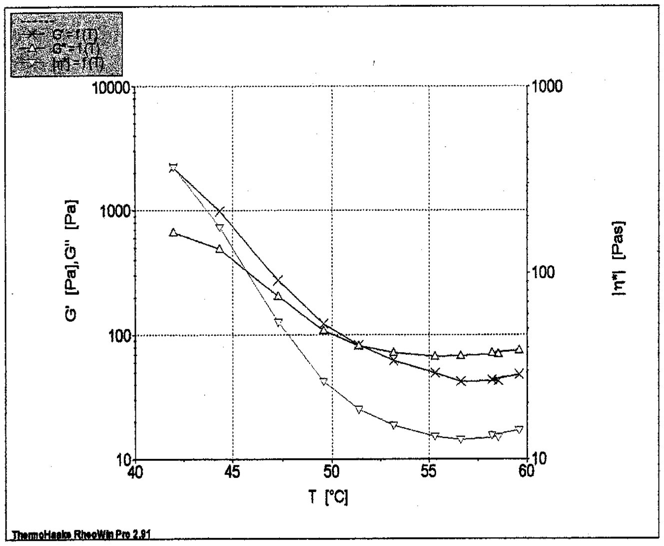
Figure 72. 150 Gelatin Rheological Testing at 36 hrs.

RheoWin Pro 2.91 Test 1 Page 1

Company	RP Scherer NA	**Measure device**	RS150		
Operator	RP Scherer NA	**Temperature device**	TC81 <---> RS150		
Date/Time	20.12.2002 / 13:58:04 PM	**Sensor**	PP35 Ti	**Gap**	2.995 mm
Substance	E22000E	**A-factor**	128800.000 Pa/Nm		
Sample no	1	**M-factor**	5.843 (1/s)/(rad/s)		
Description					

Comment

Moduli for gel free IS 430



Element definition / Notes

CD lin, 40.00 °C - 60.00 °C, t 300.00 s, 0.05 -, f 1.000 H

Filename: ----

Job: C:\Program Files\RheoWin\Jobs\Modulus Test.rwj

	t [s]	t_seg [s]	T [°C]	G' [Pa]	G" [Pa]	|η*| [Pas]
1-1	25.86	18.50	41.96	2213	665.7	367.7
1-2	61.46	54.10	44.34	985.7	489.5	175.2
1-3	105.4	98.00	47.28	273.4	204.4	54.33
1-4	140.4	133.0	49.59	123.4	108.7	26.17
1-5	169.5	162.1	51.39	82.22	81.59	18.43
1-6	194.4	187.0	53.18	62.47	72.13	15.19
1-7	218.2	210.9	55.31	49.41	67.21	13.28
1-8	241.6	234.3	56.62	42.12	68.03	12.73
1-9	262.9	255.5	58.54	42.80	70.32	13.10
1-10	284.2	276.8	58.22	43.52	72.08	13.40
1-11	305.5	298.2	59.59	48.10	75.44	14.24

Evaluation

Crossover : G' = G" = 81.21 Pa at ω = 6.283 rad/s f =

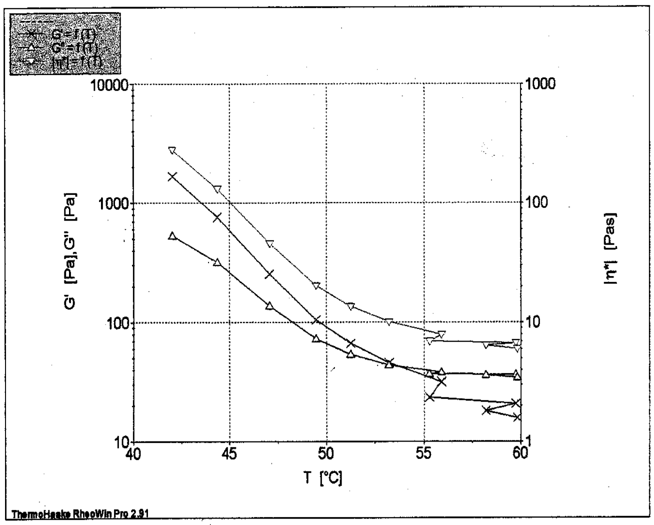
Figure 73. 150 Gelatin Rheological Testing at 40hrs.

RheoWin Pro 2.91 | Test 1 | Page 1

Company	RP Scherer NA	**Measure device**	RS150		
Operator	RP Scherer NA	**Temperature device**	TC81 <---> RS150		
Date/Time	20.12.2002 / 14:12:18 PM	**Sensor**	PP35 Ti	**Gap**	3.588 mm
Substance	E22001B	**A-factor**	128800.000 Pa/Nm		
Sample no	1	**M-factor**	4.877 (1/s)/(rad/s)		
Description					

Comment

Moduli for gel free IS 430



Element definition / Notes

CD lin, 40.00 °C - 60.00 °C, t 300.00 s, 0.05 -, f 1.000 H

Filename: ----

Job: C:\Program Files\RheoWin\Jobs\Modulus Test.rwj

	t [s]	t_seg [s]	T [°C]	G' [Pa]	G" [Pa]	|η*| [Pas]
1-1	26.71	19.34	42.02	1661	531.0	277.6
1-2	61.90	54.53	44.36	758.5	319.5	131.0
1-3	102.3	94.93	47.07	253.2	137.2	45.83
1-4	138.5	131.1	49.48	105.0	72.84	20.33
1-5	167.4	160.0	51.27	66.49	53.71	13.60
1-6	194.8	187.4	53.24	45.69	43.75	10.07
1-7	220.5	213.1	55.92	31.65	38.08	7.881
1-8	246.4	239.0	55.29	23.52	37.00	6.978
1-9	267.7	260.3	59.73	20.73	36.63	6.699
1-10	288.9	281.6	58.20	18.04	36.13	6.428
1-11	311.6	304.3	59.82	15.69	34.32	6.006

Evaluation

Crossover : G' = G" = 43.01 Pa at ω = 6.283 rad/s f =

Figure 74. 150 Gelatin Rheological Testing at 64 hrs.

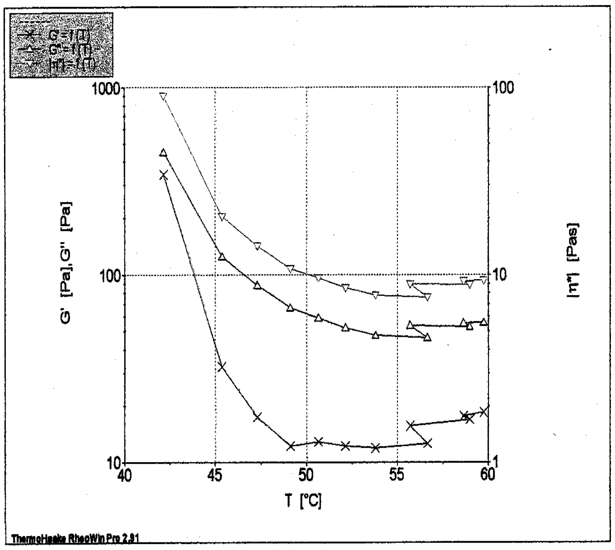
APPENDIX 3. Rheology Test Results (Continued).

RheoWin Pro 2.91 | Test 1 | Page 1

Company	RP Scherer NA	**Measure device**	RS150			
Operator	RP Scherer NA	**Temperature device**	TC81 <--> RS150			
Date/Time	20.12.2002 / 14:26:33 PM	**Sensor**	PP35 Ti		**Gap**	0.602 mm
Substance	E22001M	**A-factor**	128800.000 Pa/Nm			
Sample no	1	**M-factor**	29.070 (1/s)/(rad/s)			
Description						

Comment

Moduli for gel free IS 430



Element definition / Notes

CD lin, 40.00 °C - 60.00 °C, t 300.00 s, 0.05 -, f 1.000 H

Filename: -----

Job: C:\Program Files\RheoWin\Jobs\Modulus Test.rwj

	t [s]	t_seg [s]	T [°C]	G' [Pa]	G" [Pa]	|η*| [Pas]
1-1	32.72	25.34	42.14	342.4	448.9	89.85
1-2	77.13	69.75	45.36	32.42	124.6	20.50
1-3	105.8	98.46	47.30	17.33	87.80	14.24
1-4	133.2	125.8	49.13	12.21	66.56	10.77
1-5	157.3	149.9	50.66	12.86	58.86	9.590
1-6	180.4	173.0	52.15	12.20	52.07	8.512
1-7	203.5	196.2	53.81	11.86	47.35	7.768
1-8	225.0	217.6	56.65	12.54	46.12	7.607
1-9	246.4	239.0	55.71	15.57	53.68	8.896
1-10	267.8	260.4	58.98	16.84	52.77	8.816
1-11	289.2	281.8	58.63	17.59	55.42	9.253
1-12	310.5	303.1	59.75	18.39	55.50	9.306

Evaluation

CrossOver: no result found

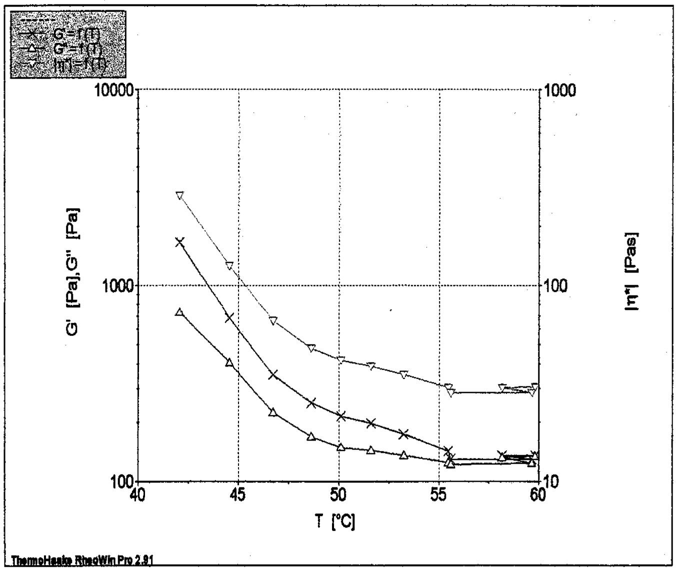
Figure 75. 150 Gelatin Rheological Testing at 68 hrs.

RheoWin Pro 2.91 | Test 1 | Page 1

Company	RP Scherer NA	**Measure device**	RS150		
Operator	RP Scherer NA	**Temperature device**	TC81 <---> RS150		
Date/Time	20.12.2002 / 14:38:36 PM	**Sensor**	PP35 Ti	**Gap**	2.769 mm
Substance	E22001E	**A-factor**	128800.000 Pa/Nm		
Sample no	1	**M-factor**	6.320 (1/s)/(rad/s)		
Description					

Comment

Moduli for gel free IS 430



Element definition / Notes

CD lin, 40.00 °C - 60.00 °C, t 300.00 s, 0.05 -, f 1.000 H

Filename: -----

Job: C:\Program Files\RheoWin\Jobs\Modulus Test.rwj

	t [s]	t_seg [s]	T [°C]	G' [Pa]	G" [Pa]	|η*| [Pas]
1-1	27.31	19.92	42.04	1658	728.9	288.2
1-2	64.34	56.95	44.52	683.6	403.8	126.4
1-3	97.37	89.97	46.73	350.9	224.2	66.27
1-4	125.6	118.2	48.61	250.7	168.1	48.05
1-5	148.9	141.6	50.11	215.0	148.7	41.60
1-6	172.4	165.0	51.60	197.4	143.7	38.86
1-7	195.0	187.6	53.23	173.8	135.1	35.03
1-8	220.2	212.8	55.46	143.1	124.3	30.16
1-9	244.8	237.4	55.58	129.8	122.0	28.34
1-10	266.5	259.1	59.63	129.4	123.8	28.50
1-11	287.8	280.4	58.17	135.6	132.1	30.13
1-12	309.1	301.8	59.81	135.7	134.0	30.35

Evaluation

CrossOver: no result found

Figure 76. 150 Gelatin Rheological Testing at 72 hrs.

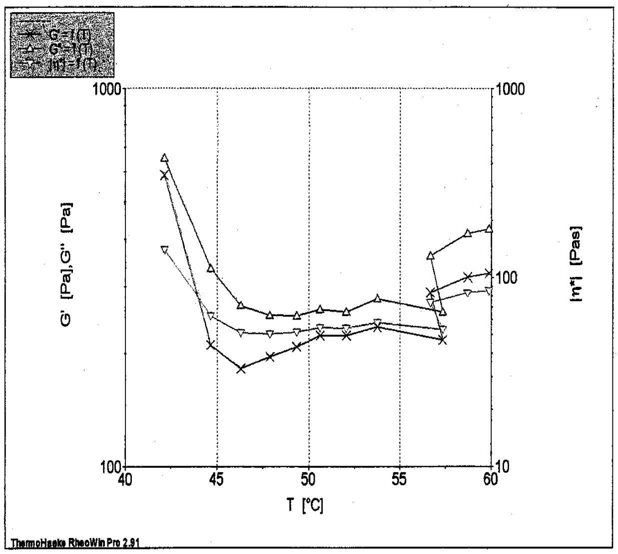
APPENDIX 3. Rheology Test Results (Continued).

RheoWin Pro 2.91 Test 1 Page 1

Company	RP Scherer NA	**Measure device**	RS150		
Operator	RP Scherer NA	**Temperature device**	TC81 <---> RS150		
Date/Time	20.12.2002 / 14:52:32 PM	**Sensor**	PP35 Ti	**Gap**	0.763 mm
Substance	E22002B	**A-factor**	128800.000 Pa/Nm		
Sample no	1	**M-factor**	22.936 (1/s)/(rad/s)		
Description					

Comment

Moduli for gel free IS 430



Element definition / Notes

CD lin, 40.00 °C - 60.00 °C, t 300.00 s, 0.05 -, f 1.000 H

Filename: ----

Job: C:\Program Files\RheoWin\Jobs\Modulus Test.rwj

	t [s]	t_seg [s]	T [°C]	G' [Pa]	G" [Pa]	|η*| [Pas]
1-1	29.37	21.96	42.13	588.3	652.2	139.8
1-2	65.70	58.28	44.63	209.7	333.7	62.74
1-3	91.31	83.90	46.31	181.7	266.8	51.38
1-4	115.1	107.7	47.90	195.1	251.2	50.62
1-5	136.4	129.0	49.35	207.4	250.5	51.76
1-6	157.7	150.3	50.65	222.1	260.6	54.49
1-7	179.0	171.6	52.06	222.4	256.1	53.98
1-8	202.8	195.4	53.77	233.8	277.5	57.76
1-9	228.7	221.3	57.34	216.5	256.5	53.42
1-10	255.4	248.0	56.66	288.2	360.8	73.49
1-11	290.2	282.8	58.72	316.4	413.3	82.84
1-12	313.3	305.9	59.88	323.7	424.0	84.90

Evaluation

CrossOver: no result found

Figure 77. 150 Gelatin Rheological Testing at 88 hrs.

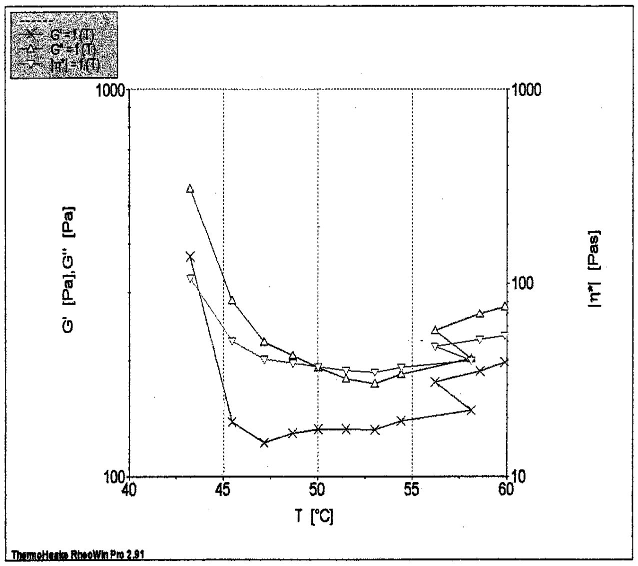
APPENDIX 3. Rheology Test Results (Continued).

RheoWin Pro 2.91 | Test 1 | Page 1

Company	RP Scherer NA	**Measure device**	RS150		
Operator	RP Scherer NA	**Temperature device**	TC81 <---> RS150		
Date/Time	20.12.2002 / 15:06:45 PM	**Sensor**	PP35 Ti	**Gap**	0.689 mm
Substance	E22002M	**A-factor**	128800.000 Pa/Nm		
Sample no	1	**M-factor**	25.399 (1/s)/(rad/s)		
Description					

Comment

Moduli for gel free IS 430



Element definition / Notes

CD lin, 40.00 °C - 60.00 °C, t 300.00 s, 0.05 -, f 1.000 H

Filename: ----

Job: C:\Program Files\RheoWin\Jobs\Modulus Test.rwj

	t [s]	t_seg [s]	T [°C]	G' [Pa]	G" [Pa]	|η*| [Pas]
1-1	44.19	36.94	43.24	369.4	552.7	105.8
1-2	78.36	71.11	45.47	138.5	285.4	50.50
1-3	104.1	96.82	47.18	122.0	222.9	40.44
1-4	126.5	119.3	48.69	129.4	205.6	38.66
1-5	147.1	139.9	50.04	132.5	191.9	37.12
1-6	170.4	163.1	51.50	132.5	179.2	35.47
1-7	192.1	184.9	53.02	131.9	173.6	34.70
1-8	212.7	205.5	54.41	138.9	183.8	36.66
1-9	233.9	226.6	58.13	148.3	201.8	39.85
1-10	261.0	253.7	56.20	175.6	239.0	47.20
1-11	292.1	284.8	58.59	187.4	262.9	51.38
1-12	314.8	307.5	59.89	197.3	274.5	53.81

Evaluation

CrossOver: no result found

Figure 78. 150 Gelatin Rheological Testing at 92 hrs.

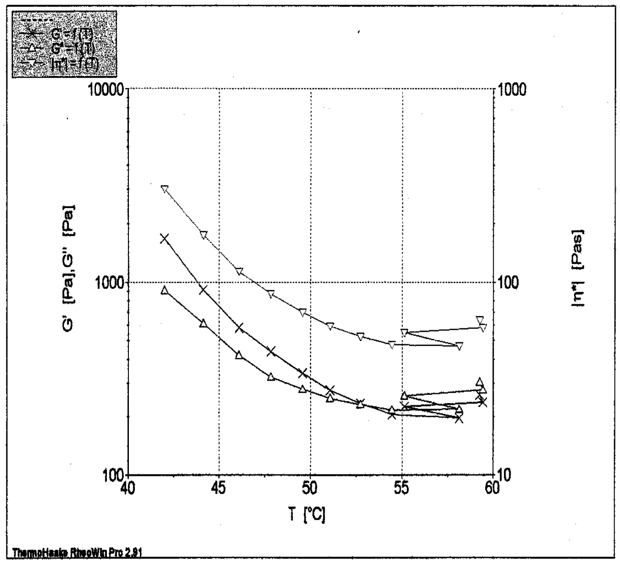
APPENDIX 3. Rheology Test Results (Continued).

RheoWin Pro 2.91 | Test 1 | Page 1

Company	RP Scherer NA	**Measure device**	RS150		
Operator	RP Scherer NA	**Temperature device**	TC81 <---> RS150		
Date/Time	20.12.2002 / 15:24:49 PM	**Sensor**	PP35 Ti	**Gap**	2.086 mm
Substance	E22002E	**A-factor**	128800.000 Pa/Nm		
Sample no	1	**M-factor**	8.389 (1/s)/(rad/s)		
Description					

Comment

Moduli for gel free IS 430



Element definition / Notes

CD lin, 40.00 °C - 60.00 °C, t 300.00 s, 0.05 -, f 1.000 H

Filename: ----

Job: C:\Program Files\RheoWin\Jobs\Modulus Test.rwj

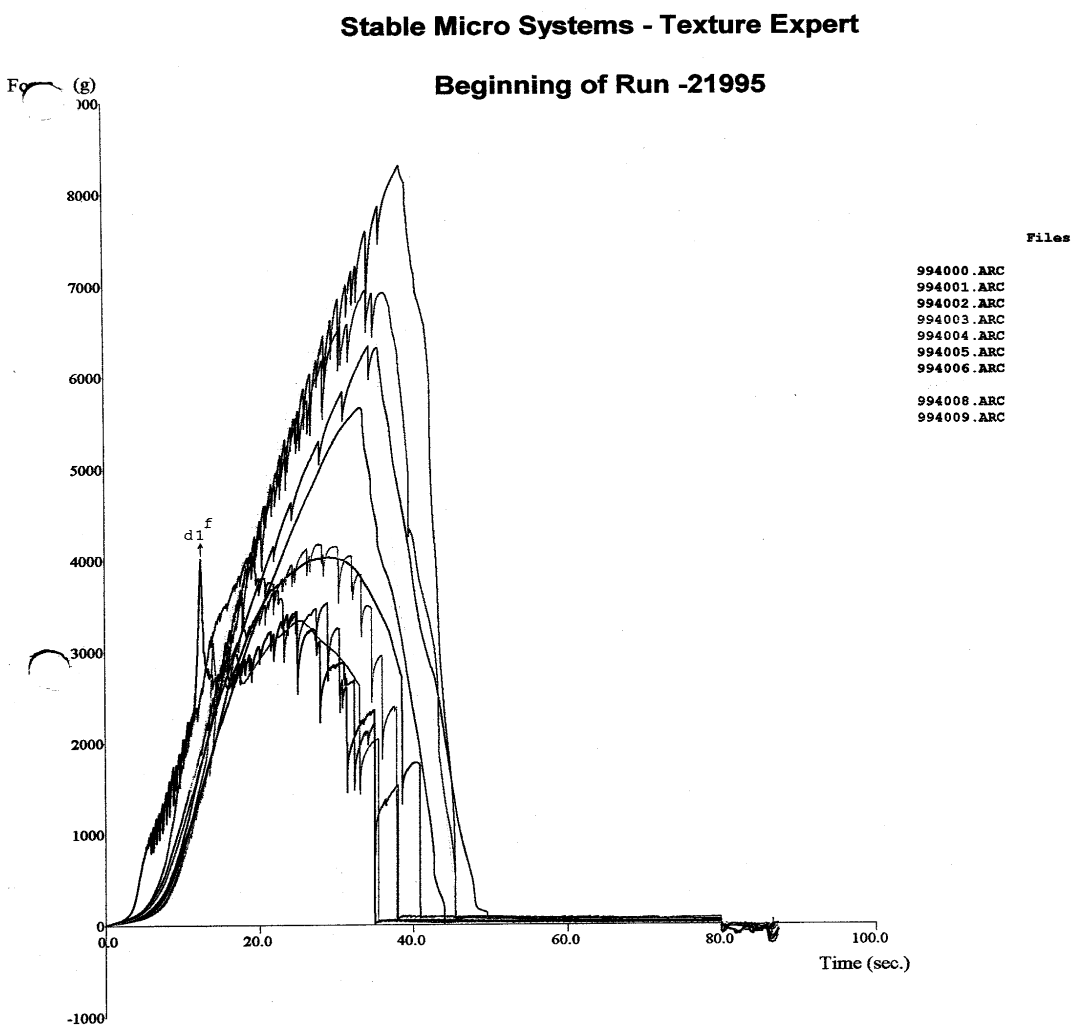
	t [s]	t_seg [s]	T [°C]	G' [Pa]	G" [Pa]	|η*| [Pas]
1-1	26.63	19.33	41.98	1667	902.9	301.8
1-2	57.84	50.54	44.13	908.3	610.2	174.1
1-3	87.52	80.22	46.10	580.6	416.8	113.8
1-4	114.2	106.9	47.85	438.6	323.3	86.72
1-5	139.9	132.6	49.58	338.1	279.1	69.77
1-6	164.4	157.1	51.08	276.4	250.2	59.33
1-7	188.1	180.8	52.73	234.9	231.1	52.44
1-8	212.6	205.3	54.45	205.6	216.9	47.57
1-9	233.9	226.6	58.12	196.8	219.0	46.86
1-10	255.2	247.9	55.12	226.7	258.2	54.69
1-11	277.3	270.0	59.43	238.1	276.8	58.11
1-12	301.6	294.3	59.26	261.6	304.9	63.94

Evaluation

Crossover : G' = G" = 228.1 Pa at ω = 6.283 rad/s f =

Figure 79. 150 Gelatin Rheological Testing at 96 hrs.

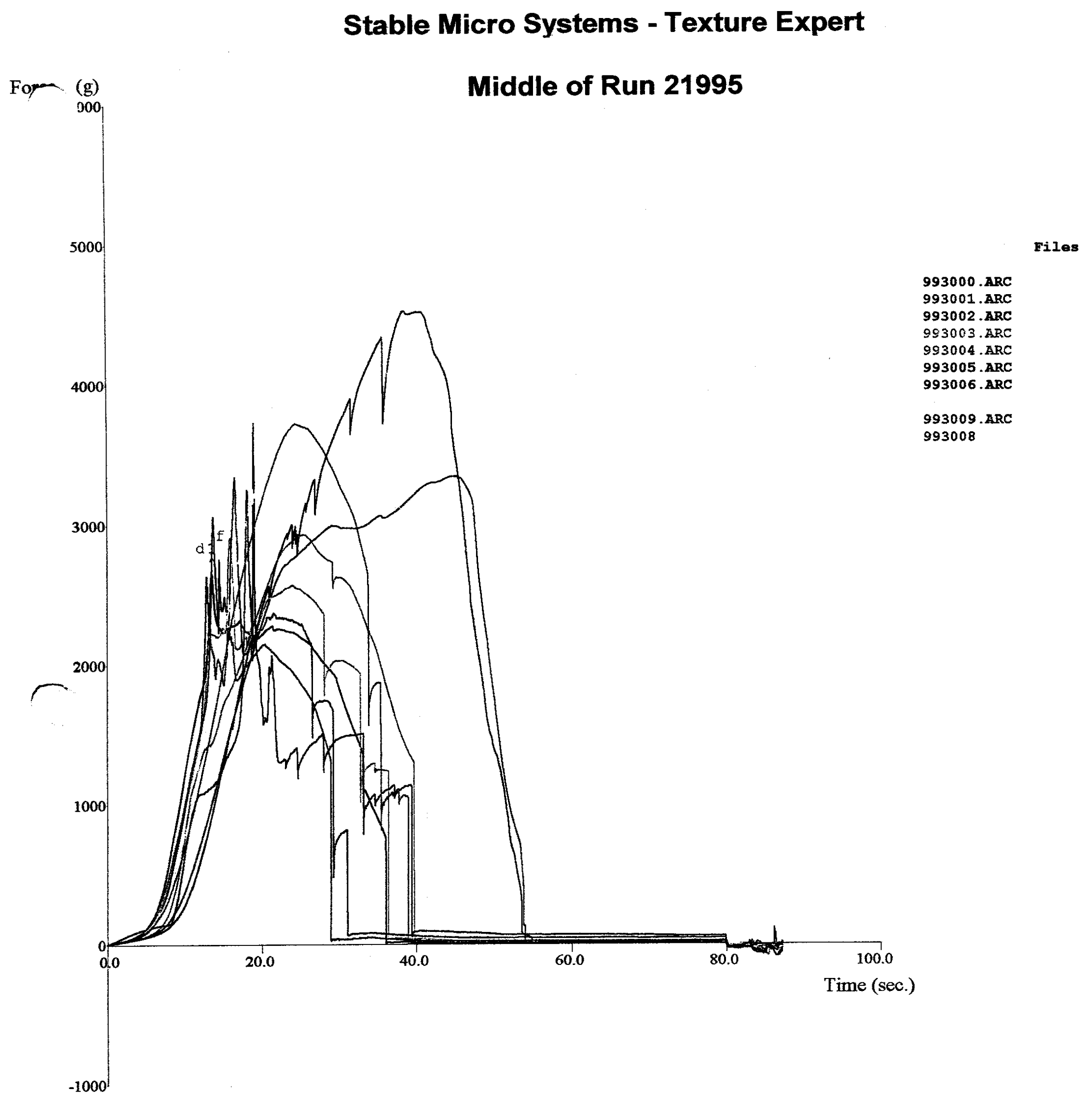
Appendix 4. Gel Extensibility Test Results



Test ID	Date and Time		Force 1 g	Distance 1 mm
994000	Mon Sep 09 22:32:34 2002		4010.666	6.231
994001	Mon Sep 09 22:36:38 2002		8309.070	19.244
994002	Mon Sep 09 22:39:28 2002		4027.752	14.531
994003	Mon Sep 09 22:44:08 2002		4172.754	13.923
994004	Mon Sep 09 22:46:52 2002		3337.269	12.798
994005	Mon Sep 09 22:48:32 2002		6335.285	17.222
994006	Mon Sep 09 22:50:10 2002		5664.568	16.641
994007	Mon Sep 09 22:54:20 2002		7023.562	15.694
994008	Mon Sep 09 22:56:56 2002		4019.930	9.312
994009	Mon Sep 09 22:59:38 2002		6948.304	17.042
Average		AVG("COL")	5384.916	14.264
Std Dev n-1		SD1("COL")	1696.967	3.949
% cv		SD1("COL")/AVG("COL")	0.315	0.277

Figure 80. 195 Gelatin Extensibility Test Printout at 0 hrs.

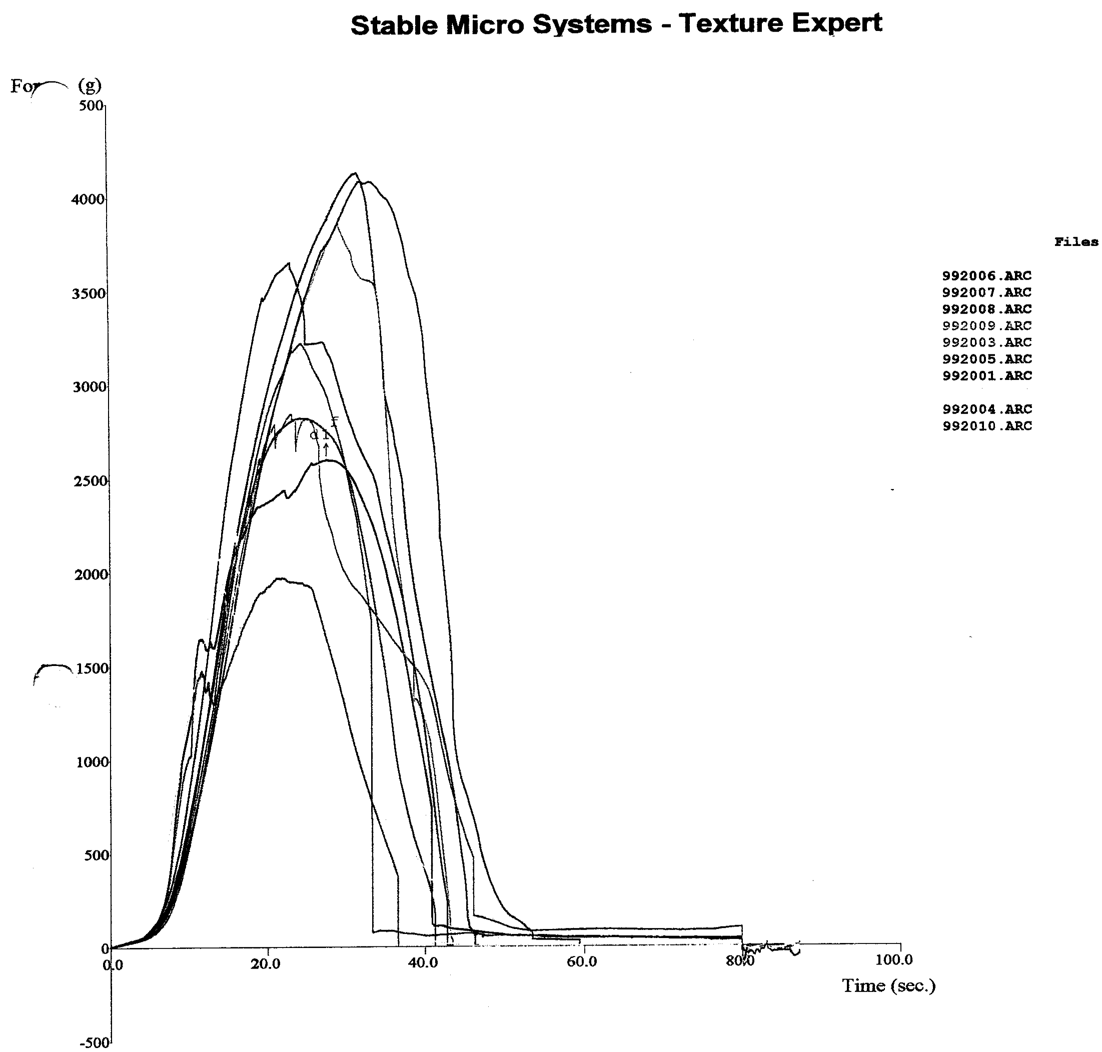
APPENDIX 4. Gel Extensibility Test Results (Continued).



Test ID	Date and Time	Force 1 g	Distance 1 mm
993008	Mon Sep 09 22:16:30 2002	3724.231	12.280
993000	Mon Sep 09 21:41:20 2002	2654.757	6.803
993001	Mon Sep 09 21:44:30 2002	3347.524	22.350
993002	Mon Sep 09 21:47:52 2002	4527.646	19.277
993003	Mon Sep 09 21:54:04 2002	2909.276	7.989
993004	Mon Sep 09 21:59:48 2002	2933.321	12.789
993005	Mon Sep 09 22:03:50 2002	3344.338	8.327
993006	Mon Sep 09 22:06:32 2002	3728.187	9.559
993009	Mon Sep 09 22:13:24 2002	2632.642	6.481
Average	AVG("COL")	3311.325	11.762
Std Dev n-1	SD1("COL")	614.647	5.627
% cv	SD1("COL")/AVG("COL")		

Figure 81. 195 Gelatin Extensibility Test Printout at 4 hrs.

APPENDIX 4. Gel Extensibility Test Results (Continued).



Test ID	Date and Time	Distance 1 mm	Distance 2 mm	Force 1 g
992009	Mon Sep 09 20:59:16 2002	14.690	0.000	3857.467
992006	Mon Sep 09 21:10:24 2002	13.858		2601.961
992007	Mon Sep 09 20:54:36 2002	12.219		2822.898
992008	Mon Sep 09 20:57:24 2002	15.821		4126.644
992003	Mon Sep 09 20:48:14 2002	11.679		2844.247
992005	Mon Sep 09 21:02:56 2002	11.620		3652.323
992001	Mon Sep 09 21:13:36 2002	16.123		4081.483
992002	Mon Sep 09 21:17:50 2002	6.652		2613.036
992004	Mon Sep 09 21:18:50 2002	11.013		1970.578
992010	Mon Sep 09 21:23:50 2002	12.288		3221.627
Average	AVG("COL")	12.596	0.000	3179.226
Std Dev n-1	SD1("COL")	2.763		726.568
% cv	SD1("COL")/AVG("COL")			0.229

Figure 82. 195 Gelatin Extensibility Test Printout at 8 hrs.

APPENDIX 4. Gel Extensibility Test Results (Continued).

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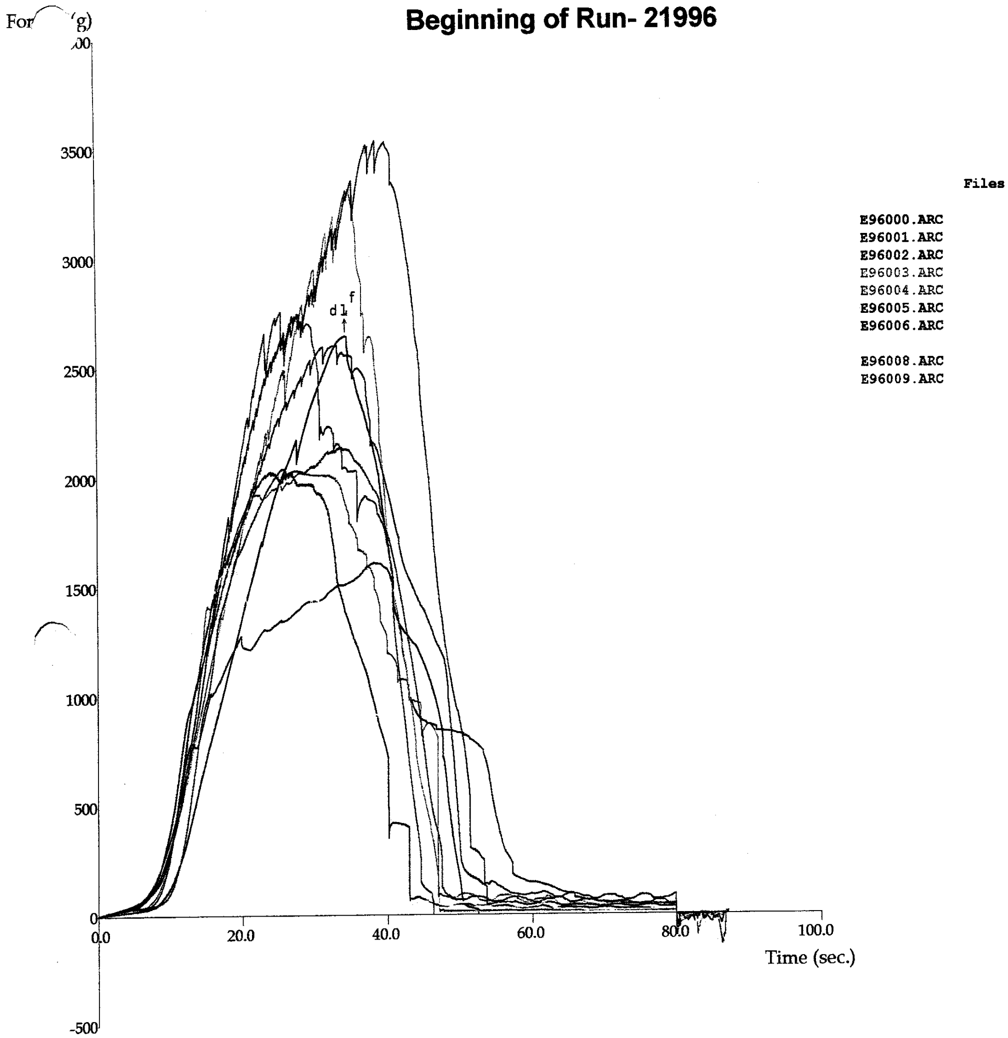
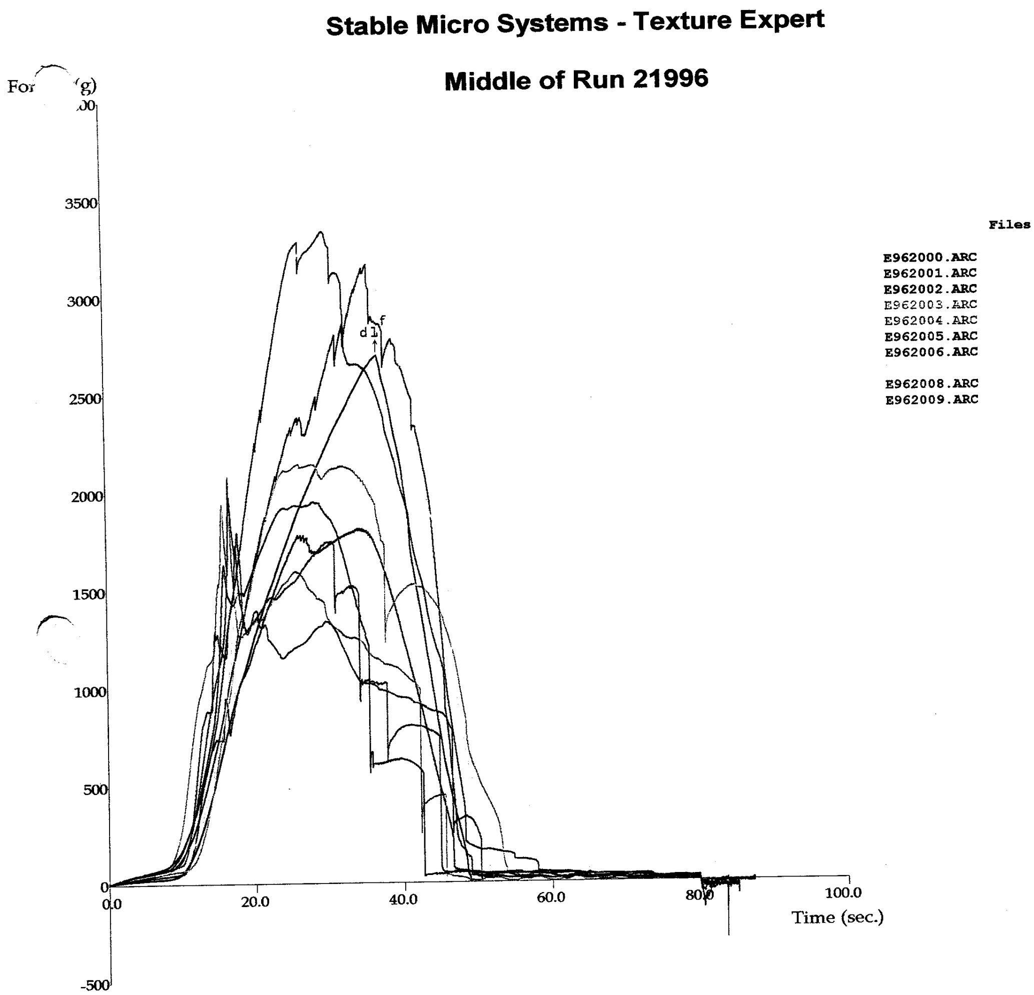
Beginning of Run- 21996



Test ID	Force 1	Distance 1
	g	mm
E96000	2645.290	17.223
E96001	1609.436	19.158
E96002	2026.877	13.462
E96003	3319.710	17.575
E96004	2025.009	14.052
E96005	3536.661	19.368
E96006	2604.394	16.667
E96007	1907.867	7.135
E96008	2140.627	16.900
E96009	2756.778	12.815
Average	2457.265	15.436
Std Dev (n-1)	627.925	3.687
% CV	0.256	0.239

Figure 83. 195 Gelatin Extensibility Test Printout at 32 hrs.

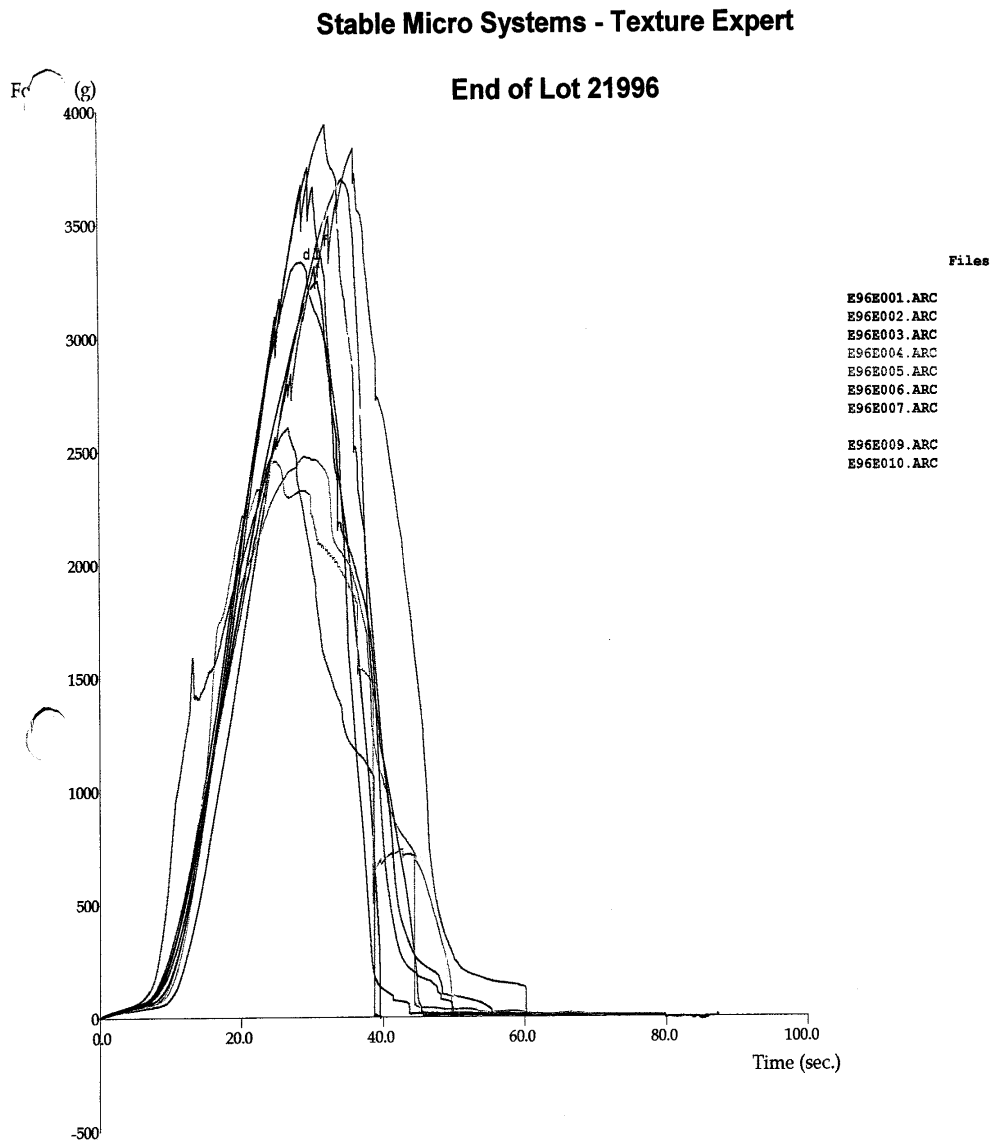
APPENDIX 4. Gel Extensibility Test Results (Continued).



Test ID	Force 1 g	Distance 1 mm
E962000	2695.211	18.516
E962001	3163.738	17.960
E962002	1812.568	17.255
E962003	2144.500	14.059
E962004	2082.256	8.363
E962005	1954.010	14.178
E962006	3332.941	15.020
E962007	3744.877	16.192
E962008	1973.381	8.411
E962009	1730.262	8.820
Average	2463.375	13.877
Std Dev (n-1)	718.806	3.976
% CV	0.292	0.286

Figure 84. 195 Gelatin Extensibility Test Printout at 36 hrs.

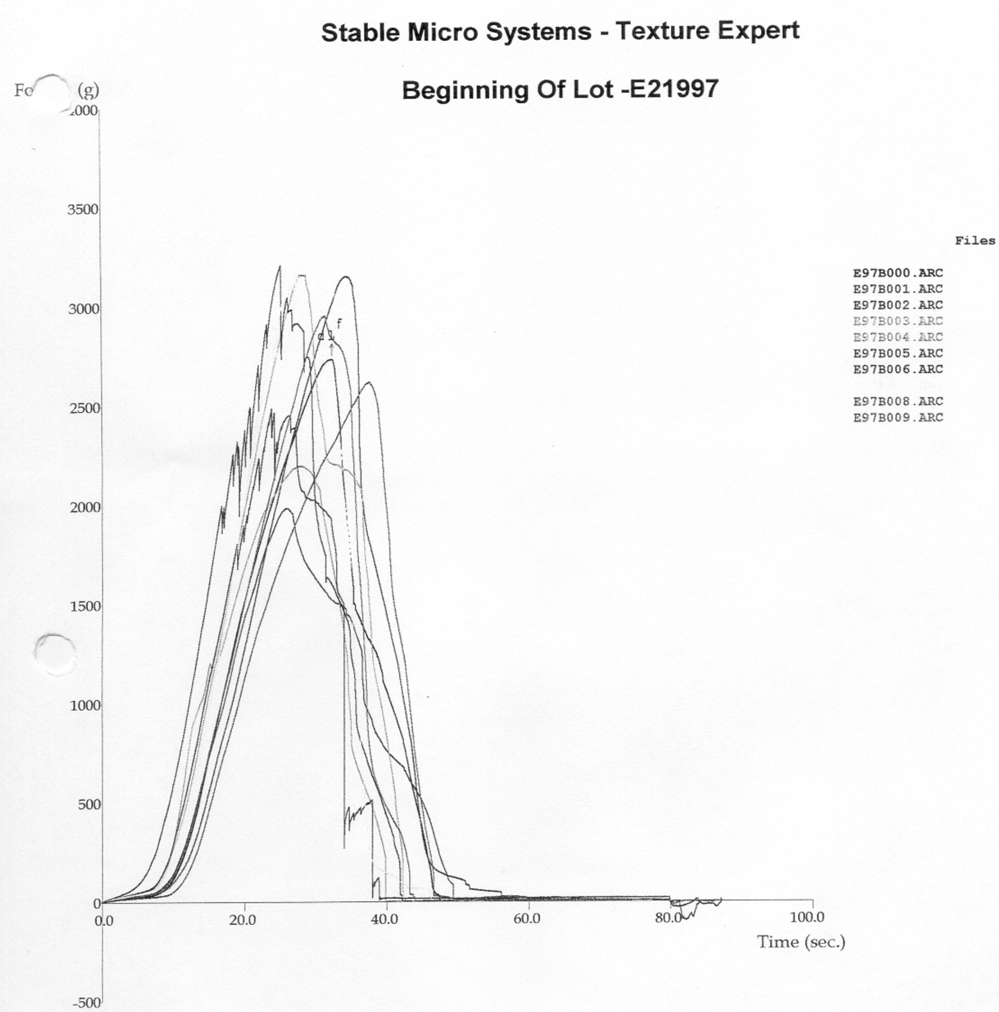
APPENDIX 4. Gel Extensibility Test Results (Continued).



Test ID	Force 1	Distance 1
	g	mm
E96E001	3248.610	15.560
E96E002	3695.599	17.231
E96E003	3332.629	14.314
E96E004	2458.178	12.561
E96E005	2478.762	14.621
E96E006	3747.612	14.899
E96E007	3936.498	16.096
E96E008	3678.618	16.970
E96E009	2602.898	13.444
E96E010	3831.367	18.056
Average	3301.077	15.375
Std Dev (n-1)	583.136	1.742
% CV	0.177	0.113

Figure 85. 195 Gelatin Extensibility Test Printout at 40 hrs.

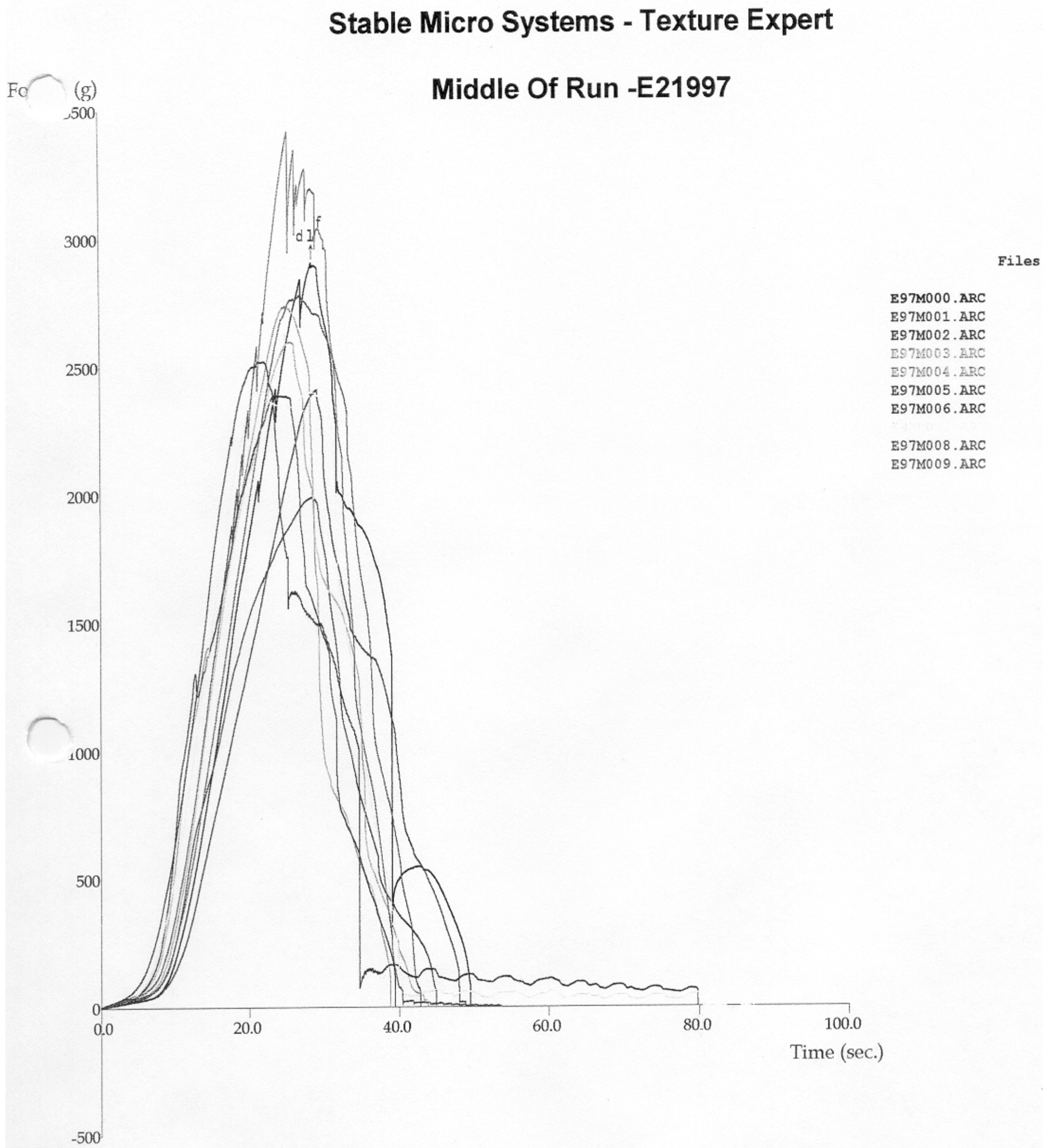
APPENDIX 4. Gel Extensibility Test Results (Continued).



Test ID	Force 1	Distance 1
	g	mm
E97B000	2735.242	16.232
E97B001	1986.662	13.094
E97B002	3152.060	17.181
E97B003	3161.457	14.088
E97B004	2200.230	14.116
E97B005	2486.955	12.018
E97B006	3209.238	12.670
E97B007	3753.689	15.253
E97B008	2619.416	18.924
E97B009	2954.112	15.724
Average	2825.906	14.930
Std Dev (n-1)	527.539	2.155
% CV	0.187	0.144

Figure 86. 195 Gelatin Extensibility Test Printout at 64 hrs.

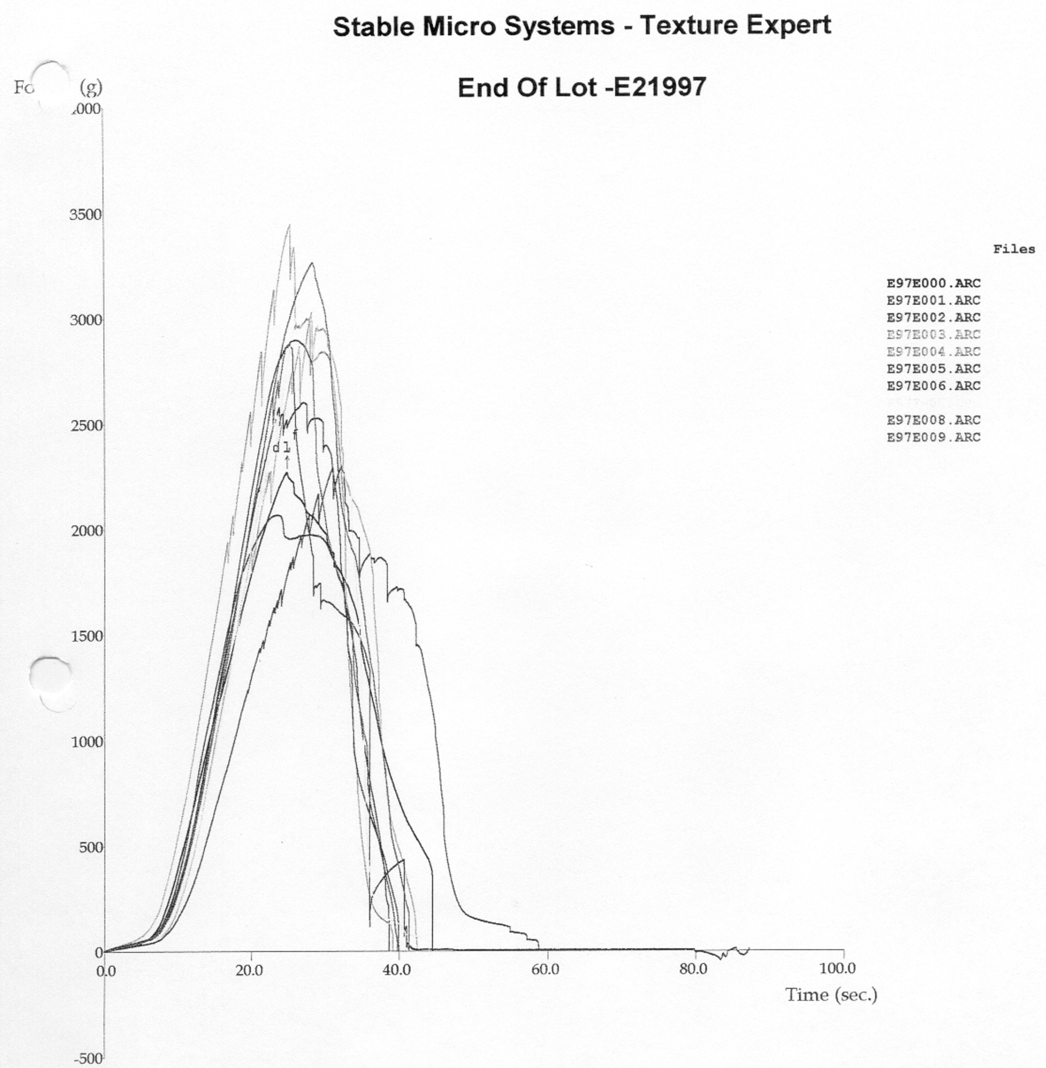
APPENDIX 4. Gel Extensibility Test Results (Continued).



Test ID	Force 1	Distance 1
	g	mm
E97M000	2905.552	14.310
E97M001	1992.512	14.322
E97M002	2411.401	14.648
E97M003	2599.260	12.763
E97M004	2739.034	12.679
E97M005	2390.032	12.069
E97M006	2521.546	11.073
E97M007	2415.300	10.592
E97M008	2780.945	13.480
E97M009	3417.477	12.732
Average	2617.306	12.867
Std Dev (n-1)	380.011	1.368
% CV	0.145	0.106

Figure 87. 195 Gelatin Extensibility Test Printout at 68 hrs.

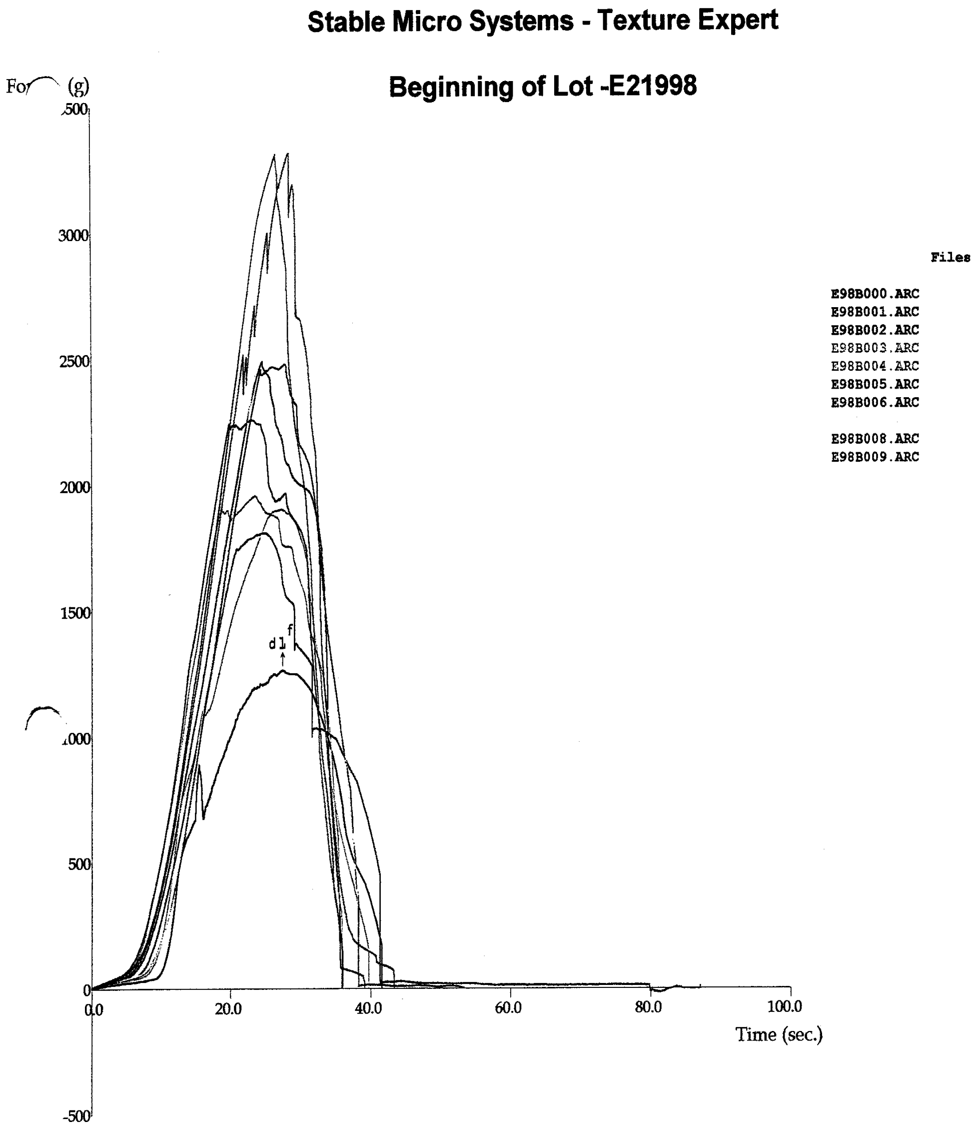
APPENDIX 4. Gel Extensibility Test Results (Continued).



Test ID	Force 1	Distance 1
	g	mm
E97E000	2272.689	12.426
E97E001	2303.381	16.130
E97E002	2069.460	11.833
E97E003	3026.958	14.134
E97E004	3443.862	12.673
E97E005	2874.475	12.611
E97E006	2602.740	13.595
E97E007	3581.809	15.345
E97E008	2897.380	13.045
E97E009	3266.017	14.222
Average	2833.877	13.601
Std Dev (n-1)	515.893	1.365
% CV	0.182	0.100

Figure 88. 195 Gelatin Extensibility Test Printout at 72 hrs.

APPENDIX 4. Gel Extensibility Test Results (Continued).



Test ID	Force 1	Distance 1
	g	mm
E98B000	1265.838	13.715
E98B001	1811.984	12.457
E98B002	2492.787	12.291
E98B003	1906.953	13.717
E98B004	1961.327	11.770
E98B005	2481.255	13.881
E98B006	2261.503	11.527
E98B007	2964.861	13.733
E98B008	3316.432	14.208
E98B009	3311.232	13.259
Average	2377.417	13.056
Std Dev (n-1)	673.809	0.961
% CV	0.283	0.074

Figure 89. 195 Gelatin Extensibility Test Printout at 88 hrs.

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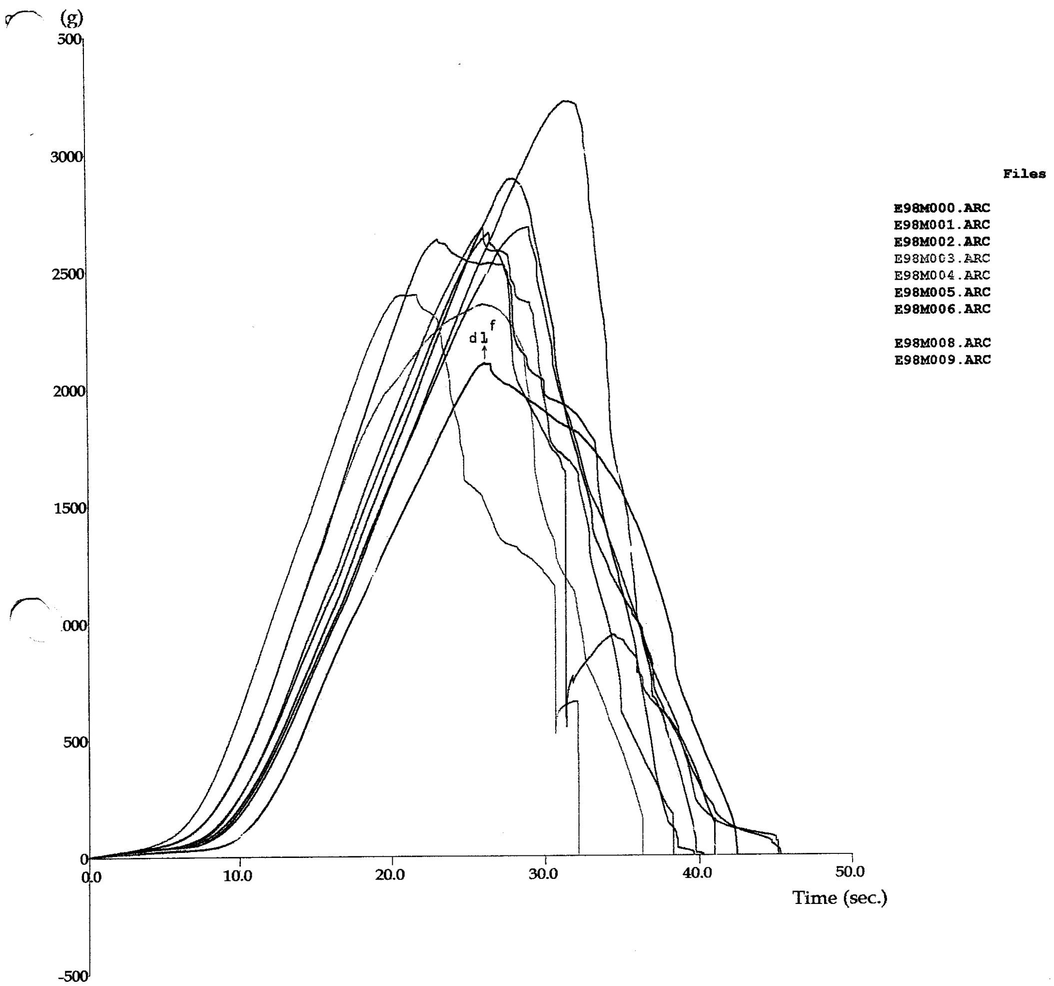

Test ID	Force 1	Distance 1
	g	mm
E98M000	2103.994	13.069
E98M001	3216.236	15.730
E98M002	2890.207	14.021
E98M003	2355.036	13.015
E98M004	2398.326	10.752
E98M005	2682.596	14.499
E98M006	2660.277	13.232
E98M007	3205.808	15.897
E98M008	2679.921	13.036
E98M009	2633.124	11.552
Average	2682.552	13.480
Std Dev (n-1)	353.940	1.634
% CV	0.132	0.121

Figure 90. 195 Gelatin Extensibility Test Printout at 92 hrs.

APPENDIX 4. Gel Extensibility Test Results (Continued).

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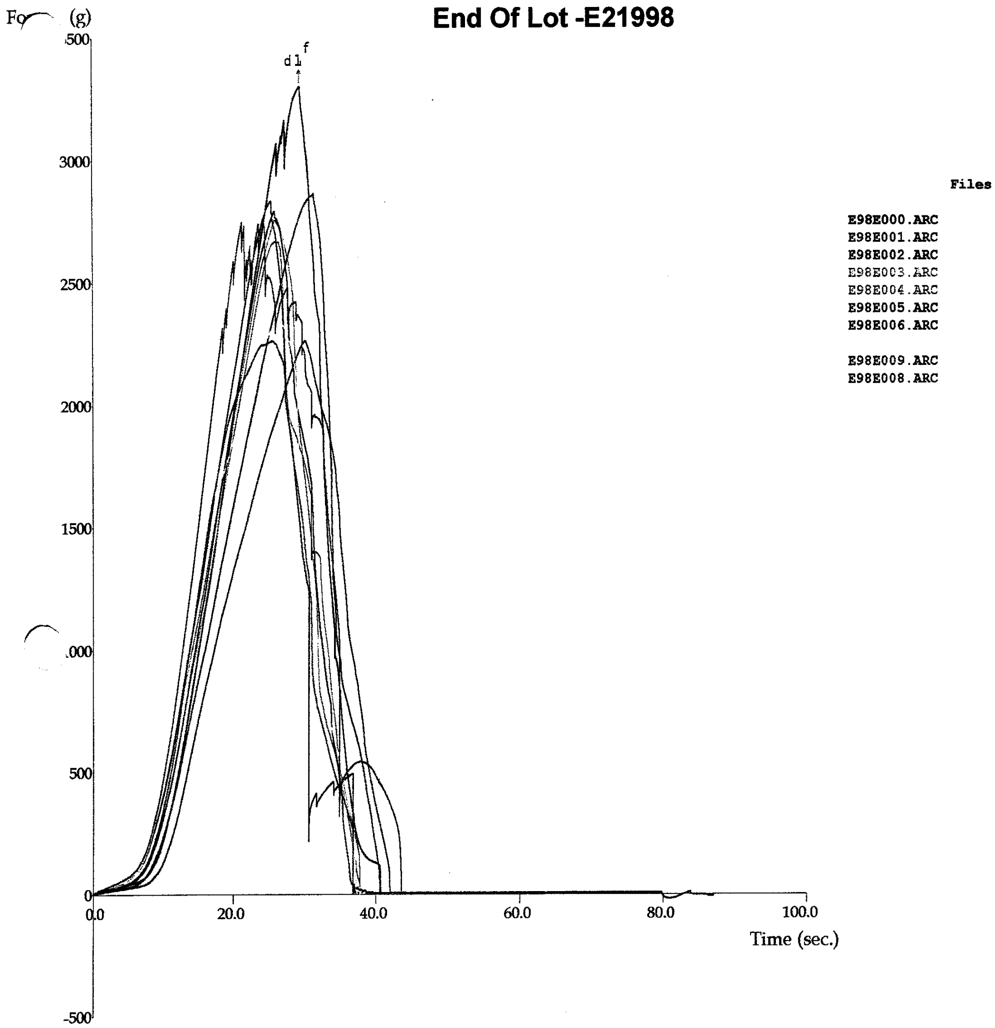
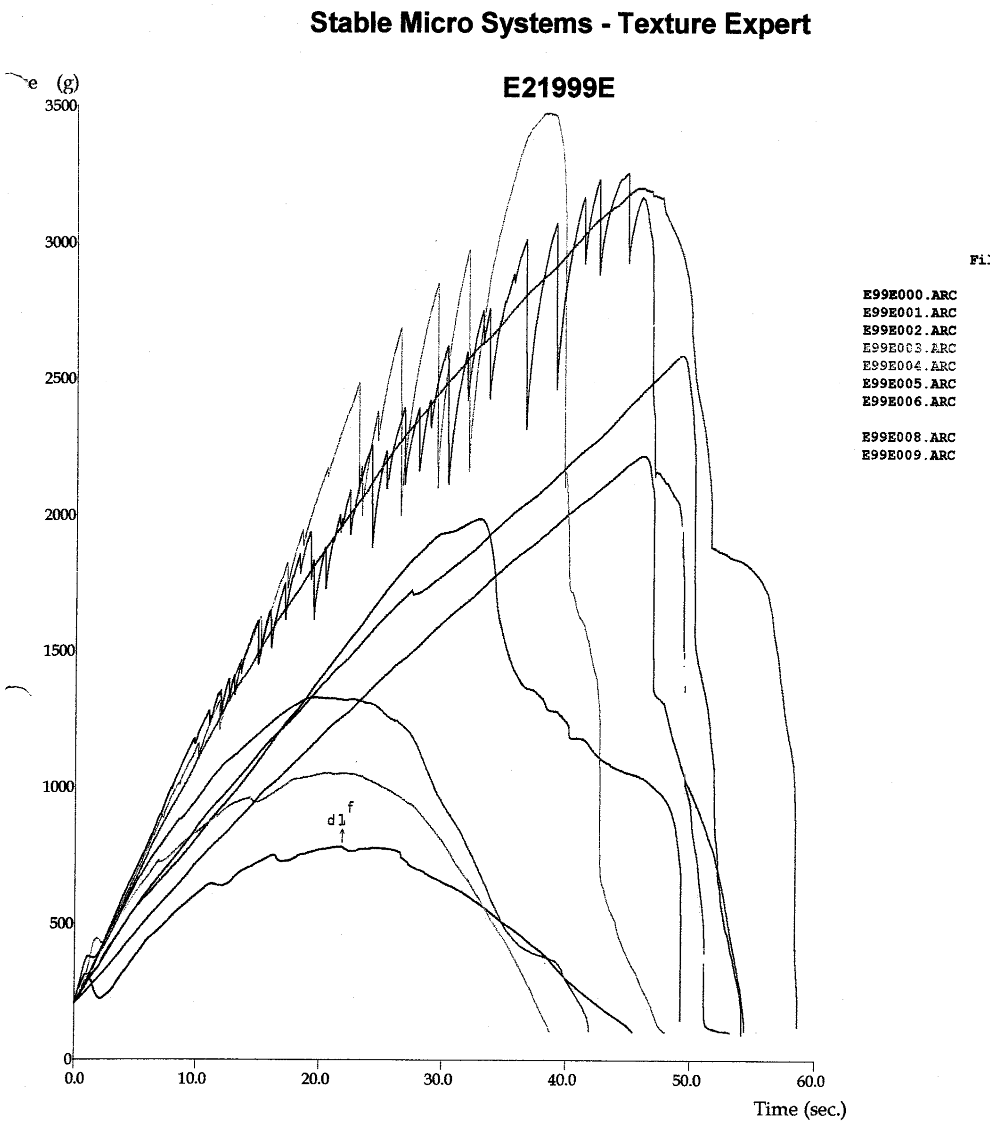
End Of Lot -E21998



Test ID	Force 1	Distance 1
	g	mm
E98E000	3301.230	14.618
E98E001	2264.564	15.051
E98E002	2864.679	15.609
E98E003	2757.751	12.949
E98E004	2668.931	12.944
E98E005	2793.403	12.868
E98E006	2835.142	12.650
E98E007	2347.622	13.153
E98E009	2264.902	12.741
E98E008	2750.276	12.128
Average	2684.850	13.471
Std Dev (n-1)	320.278	1.174
% CV	0.119	0.087

Figure 91. 195 Gelatin Extensibility Test Printout at 96 hrs.



Test ID	Force 1	Distance 1
	g	mm
E99E000	782.863	10.912
E99E001	2220.521	22.993
E99E002	1989.526	16.451
E99E003	1053.401	10.212
E99E004	3476.133	19.139
E99E005	3256.269	22.332
E99E006	2587.783	24.540
E99E007	2756.031	24.099
E99E008	3202.006	22.900
E99E009	1334.278	9.870
Average	2265.881	18.345
Std Dev (n-1)	959.256	6.029
% CV	0.423	0.329

Figure 92. 150 Gelatin Extensibility Test Printout at 8 hrs.

APPENDIX 4. Gel Extensibility Test Results (Continued).

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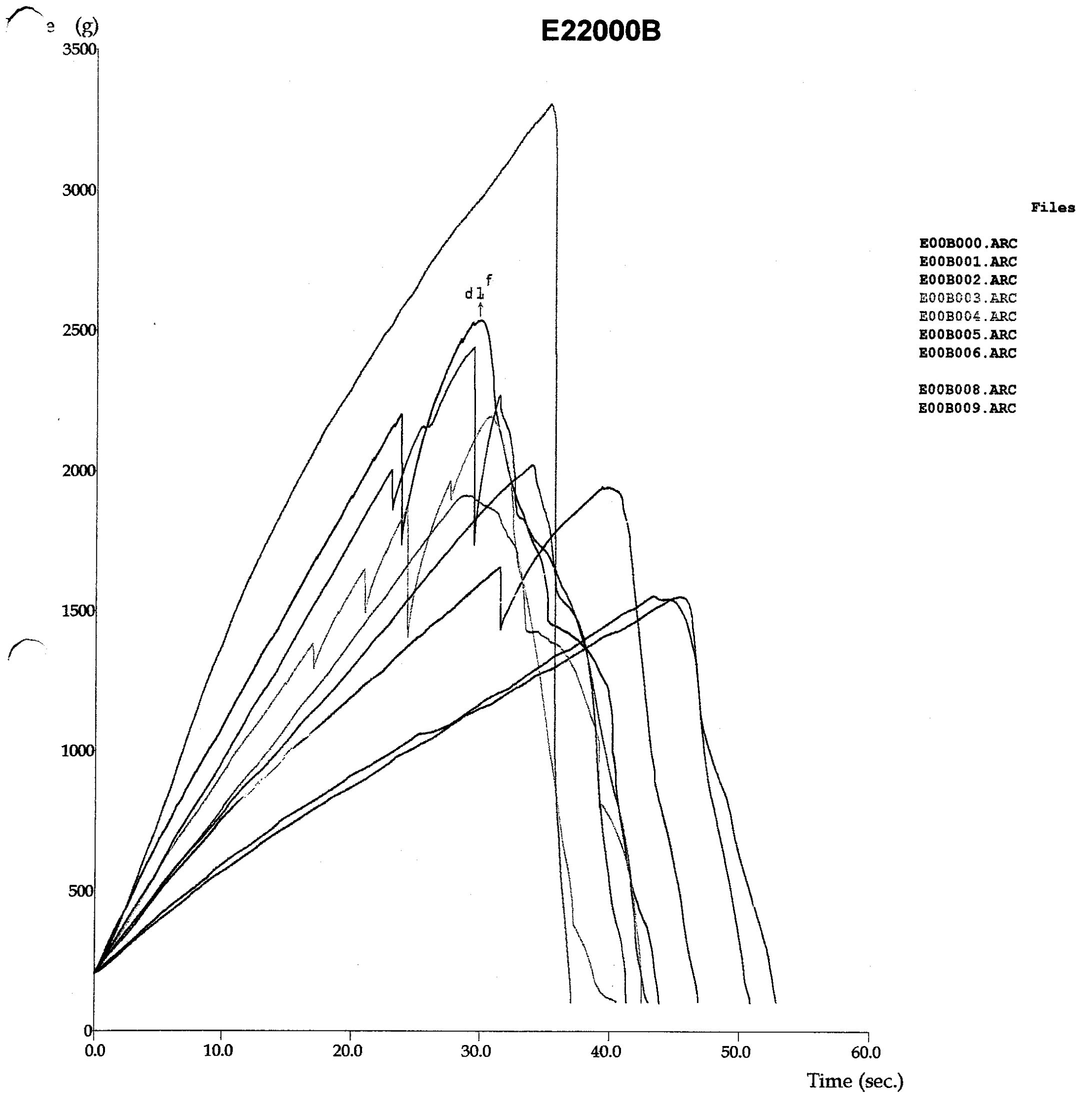
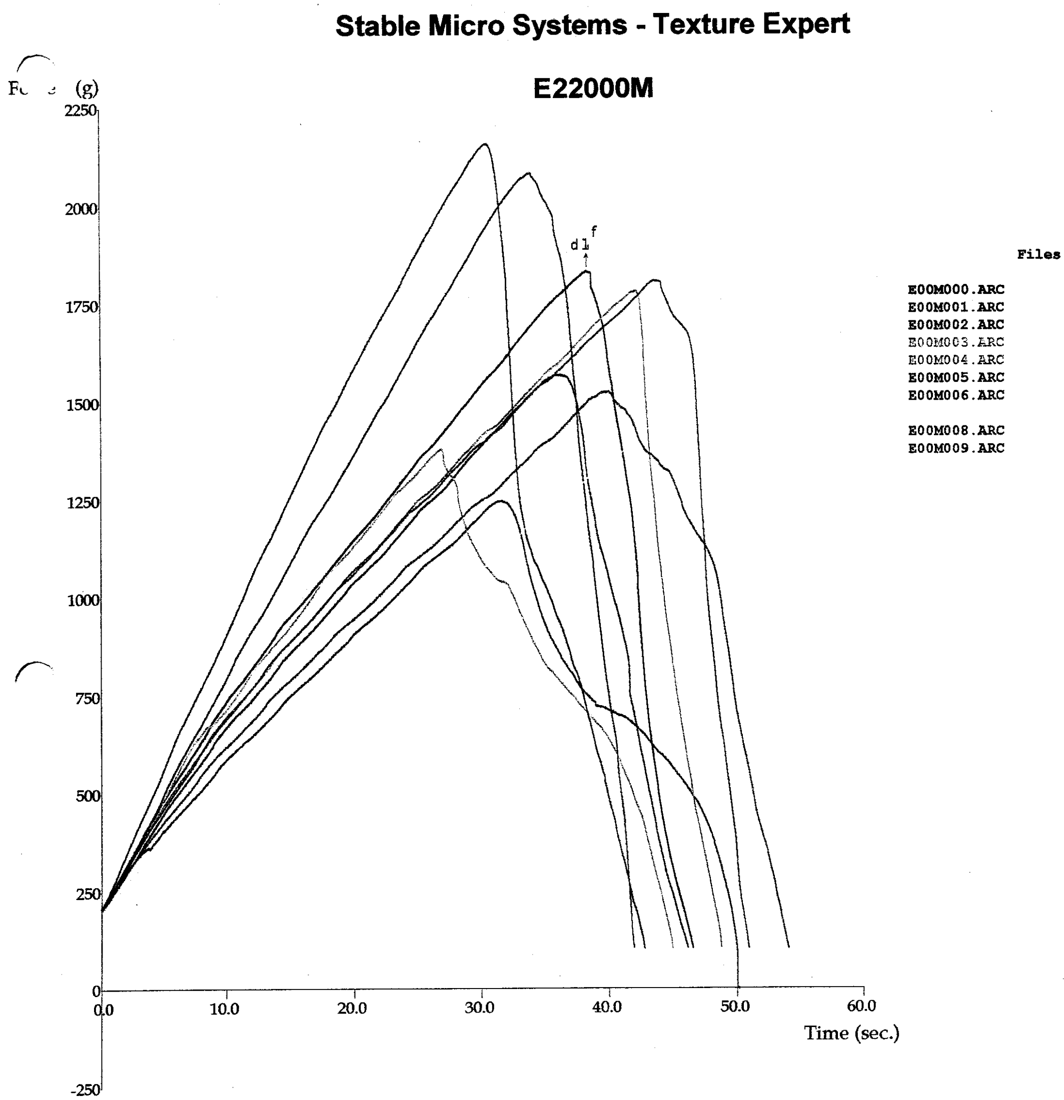


Test ID	Force 1 g	Distance 1 mm
E00B000	2535.953	14.877
E00B001	1552.419	22.610
E00B002	1943.925	19.632
E00B003	2196.652	15.332
E00B004	1913.151	14.406
E00B005	2440.401	14.676
E00B006	1555.006	21.633
E00B007	1967.427	23.046
E00B008	3307.169	17.604
E00B009	2022.367	16.944
Average	2143.447	18.076
Std Dev (n-1)	519.977	3.410
% CV	0.243	0.189

Figure 93. 150 Gelatin Extensibility Test Printout at 32 hrs.

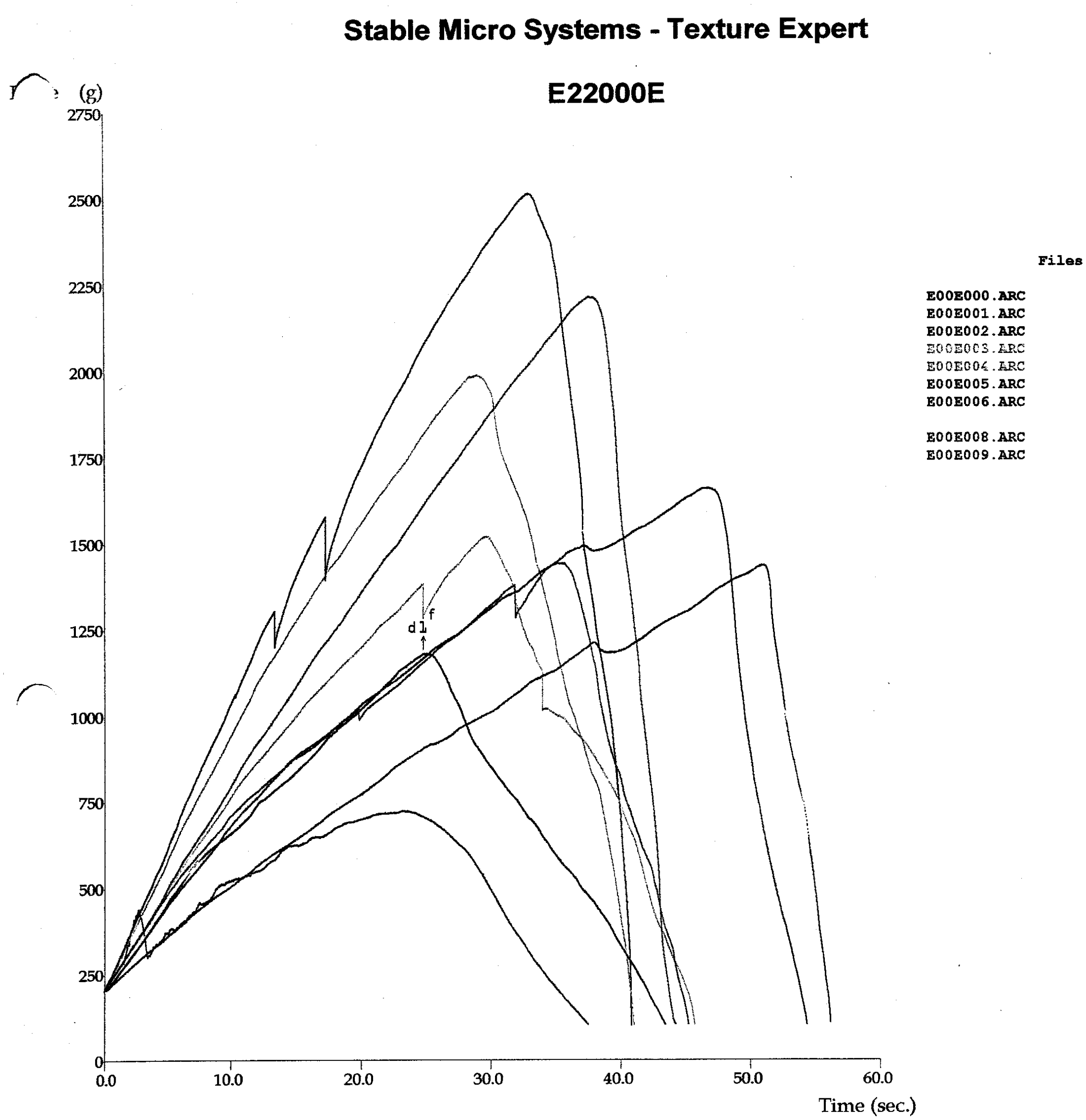
APPENDIX 4. Gel Extensibility Test Results (Continued).



Test ID	Force 1 g	Distance 1 mm
E00M000	1832.962	19.131
E00M001	1248.009	15.832
E00M002	1569.116	17.960
E00M003	1380.863	13.455
E00M004	1782.802	21.056
E00M005	2082.967	16.917
E00M006	2157.800	15.189
E00M007	1518.002	23.481
E00M008	1525.775	19.956
E00M009	1809.642	21.885
Average	1690.794	18.486
Std Dev (n-1)	294.148	3.191
% CV	0.174	0.173

Figure 94. 150 Gelatin Extensibility Test Printout at 36 hrs.

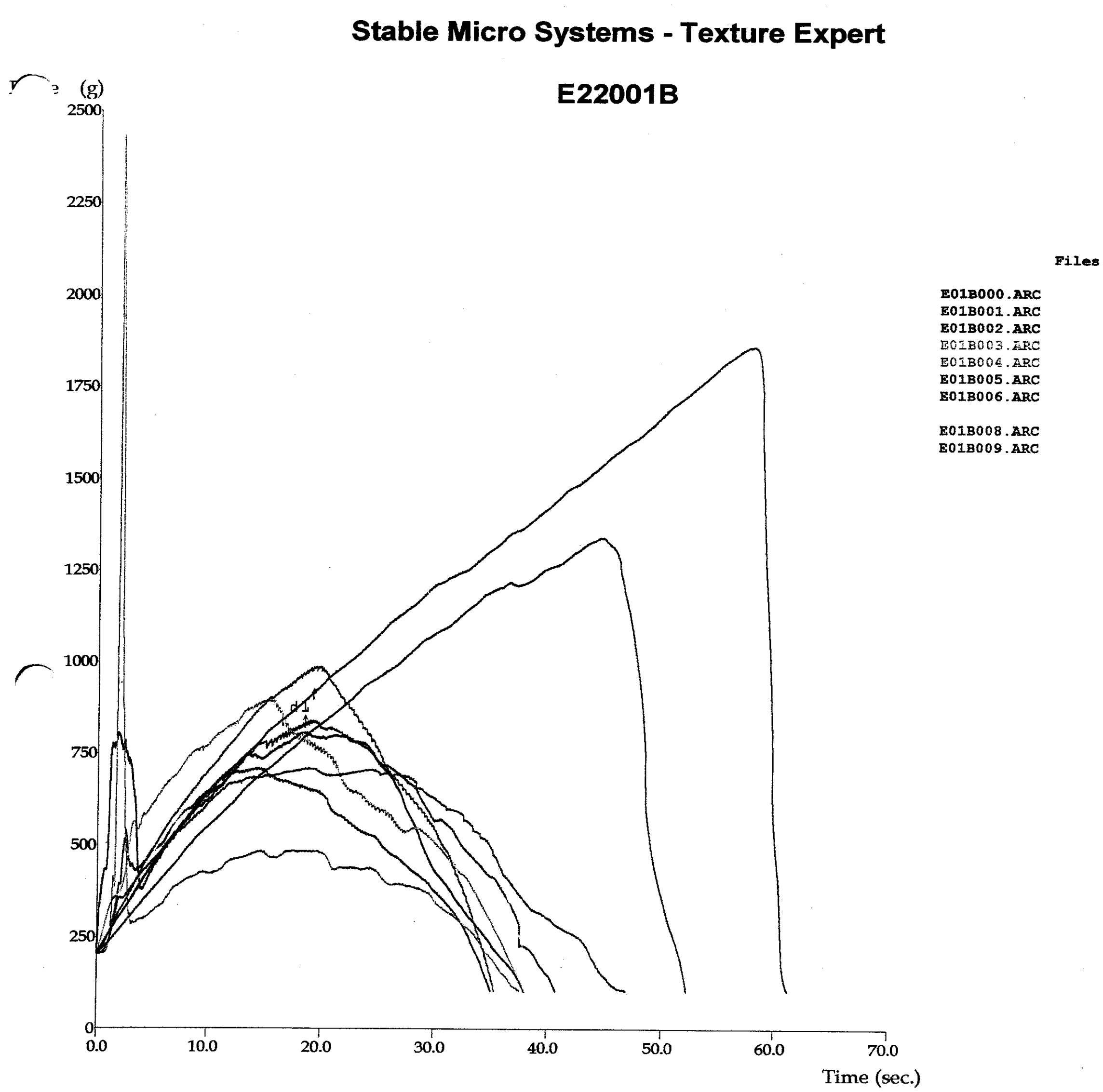
APPENDIX 4. Gel Extensibility Test Results (Continued).



Test ID	Force 1	Distance 1
	g	mm
E00E000	1180.983	12.398
E00E001	724.625	11.729
E00E002	2515.248	16.397
E00E003	1521.219	14.812
E00E004	1989.324	14.450
E00E005	1436.114	25.449
E00E006	1661.994	23.362
E00E007	1850.905	24.199
E00E008	2215.462	18.732
E00E009	1443.941	17.747
Average	1653.981	17.928
Std Dev (n-1)	516.933	4.936
% CV	0.313	0.275

Figure 95. 150 Gelatin Extensibility Test Printout at 40 hrs.

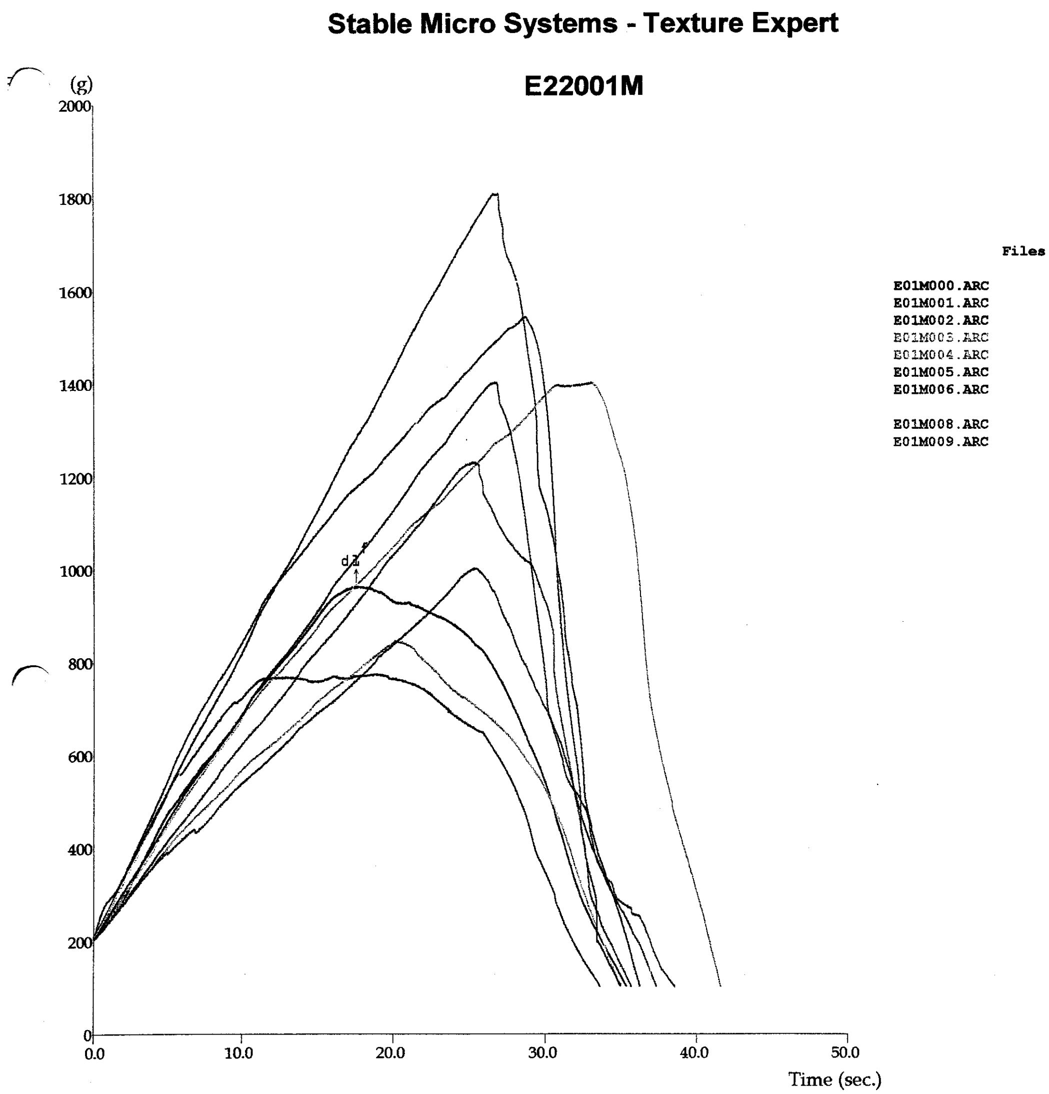
APPENDIX 4. Gel Extensibility Test Results (Continued).



Test ID	Force 1 g	Distance 1 mm
E01B000	808.109	9.239
E01B001	841.039	9.495
E01B002	710.409	7.222
E01B003	896.912	7.693
E01B004	2431.695	1.081
E01B005	1340.208	22.273
E01B006	1861.100	28.918
E01B007	1212.597	14.445
E01B008	986.832	9.938
E01B009	709.725	9.440
Average	1179.863	11.974
Std Dev (n-1)	564.672	8.049
% CV	0.479	0.672

Figure 96. 150 Gelatin Extensibility Test Printout at 64 hrs.

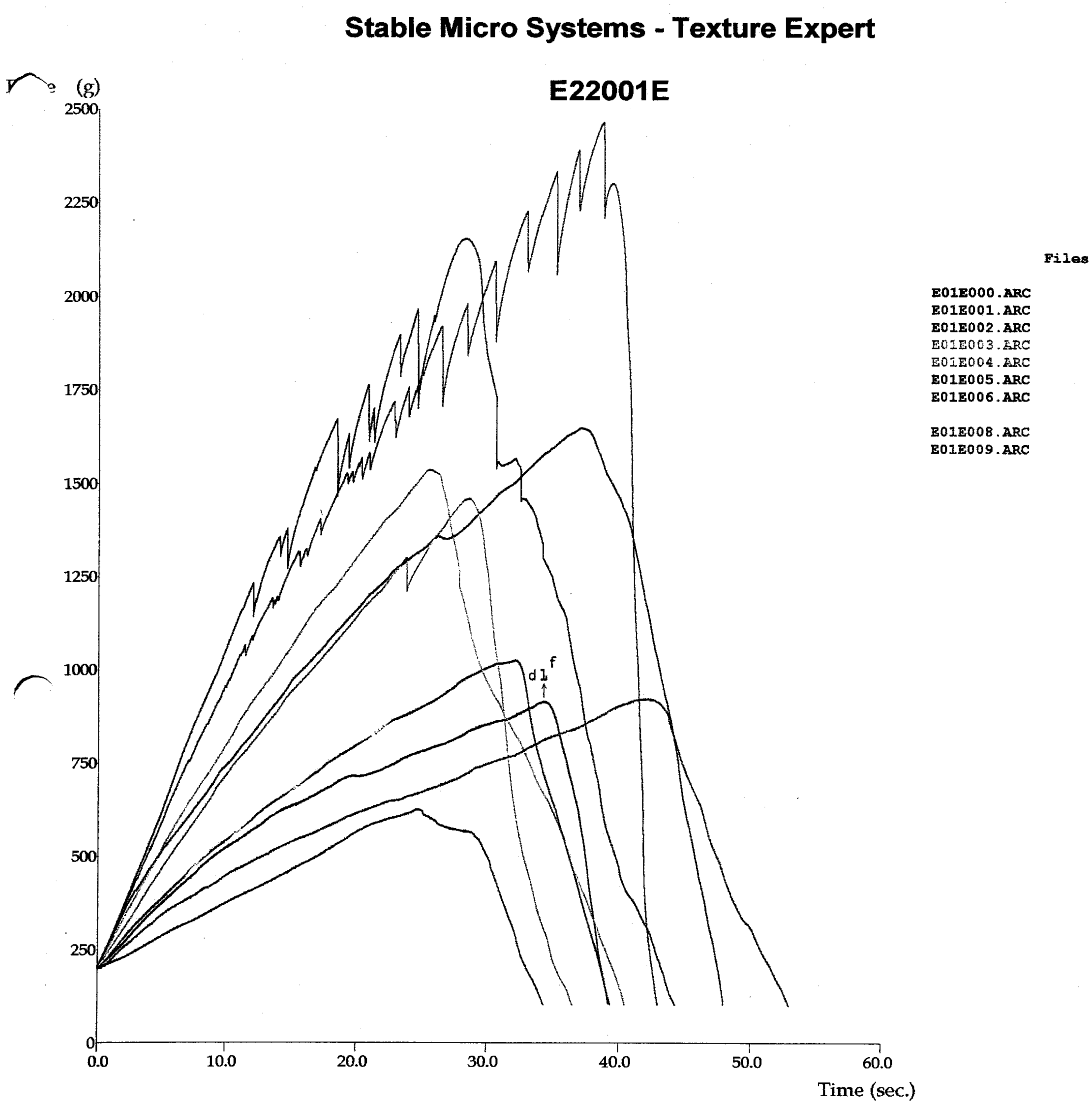
APPENDIX 4. Gel Extensibility Test Results (Continued).



Test ID	Force 1	Distance 1
	g	mm
E01M000	964.330	8.750
E01M001	776.648	9.389
E01M002	1808.539	13.375
E01M003	847.188	10.037
E01M004	1402.994	16.486
E01M005	1403.556	13.293
E01M006	1545.218	14.273
E01M007	1684.930	14.860
E01M008	1232.898	12.566
E01M009	1003.240	12.620
Average	1266.954	12.565
Std Dev (n-1)	359.244	2.490
% CV	0.284	0.198

Figure 97. 150 Gelatin Extensibility Test Printout at 68 hrs.

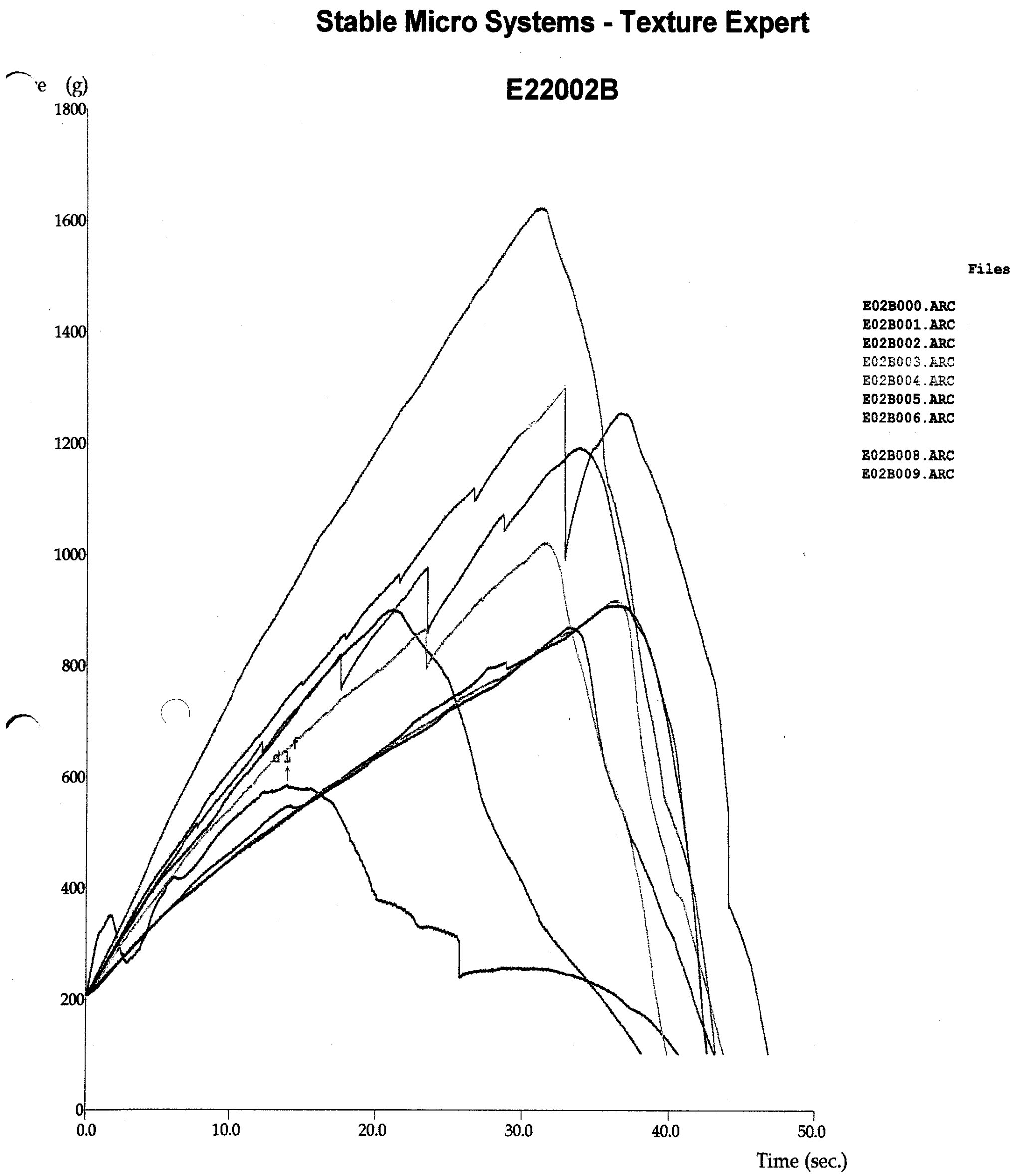
APPENDIX 4. Gel Extensibility Test Results (Continued).



Test ID	Force 1	Distance 1
	g	mm
E01E000	916.922	17.128
E01E001	1648.580	18.517
E01E002	1026.371	16.036
E01E003	1537.553	12.680
E01E004	1460.837	14.257
E01E005	2155.055	14.084
E01E006	626.712	12.320
E01E007	1084.370	16.064
E01E008	923.883	21.049
E01E009	2463.813	19.274
Average	1384.410	16.141
Std Dev (n-1)	585.096	2.885
% CV	0.423	0.179

Figure 98. 150 Gelatin Extensibility Test Printout at 72 hrs.

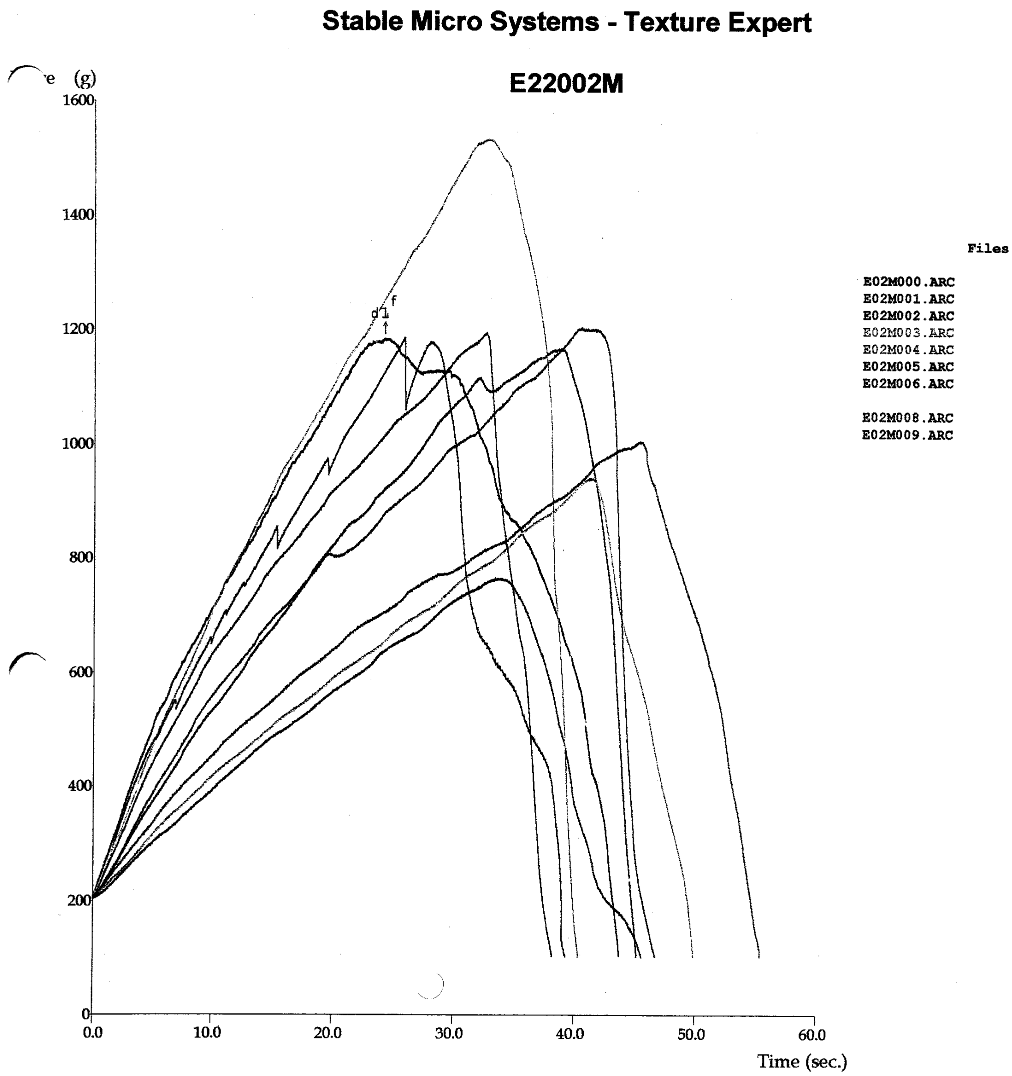
APPENDIX 4. Gel Extensibility Test Results (Continued).



Test ID	Force 1	Distance 1
	g	mm
E02B000	585.621	6.930
E02B001	869.530	16.581
E02B002	900.686	10.553
E02B003	1021.270	15.772
E02B004	917.946	18.115
E02B005	1302.992	16.361
E02B006	909.536	18.199
E02B007	1364.517	17.568
E02B008	1622.932	15.550
E02B009	1191.888	16.896
Average	1068.692	15.253
Std Dev (n-1)	301.168	3.646
% CV	0.282	0.239

Figure 99. 150 Gelatin Extensibility Test Printout at 88 hrs.

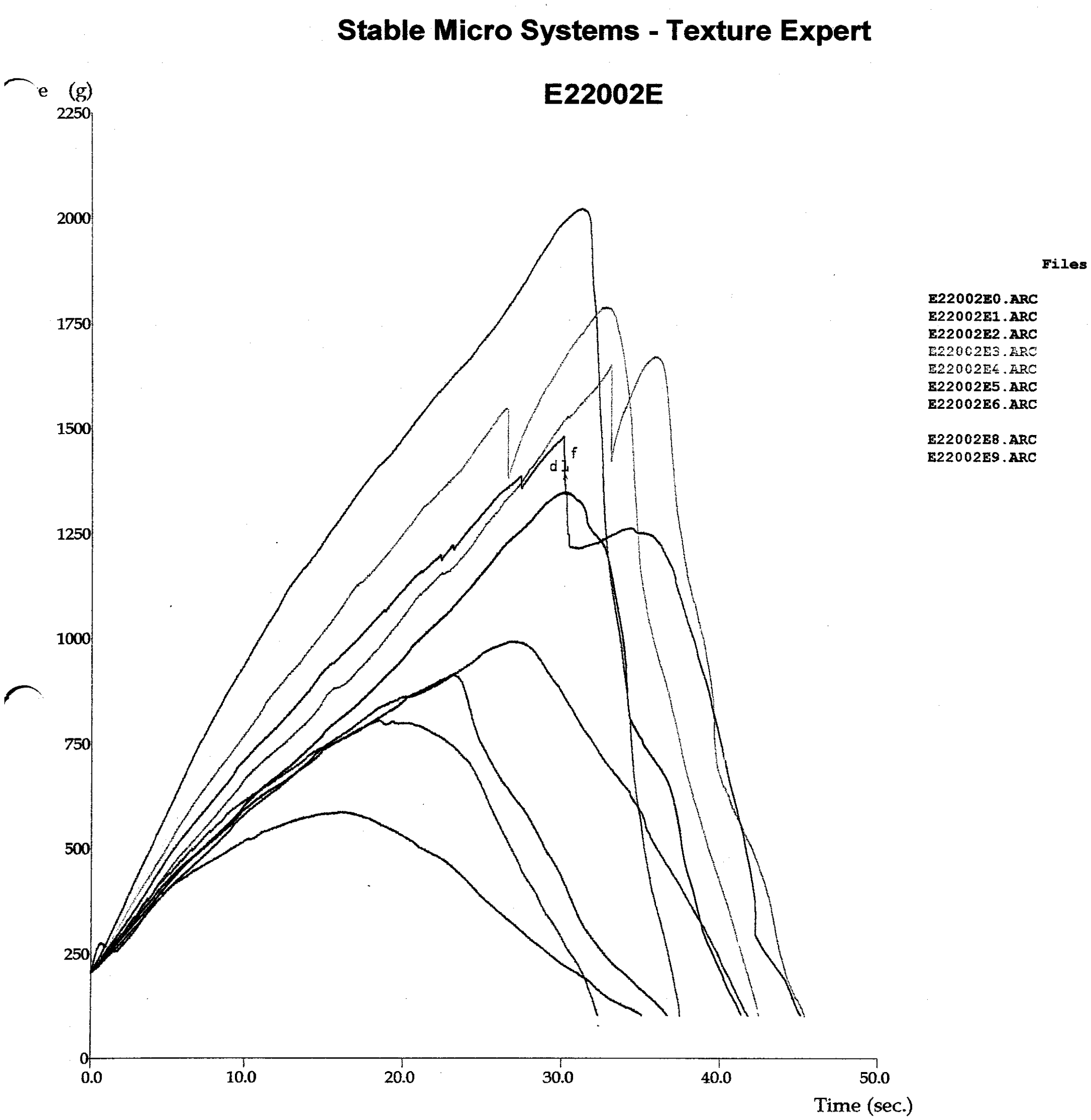
APPENDIX 4. Gel Extensibility Test Results (Continued).



Test ID	Force 1	Distance 1
	g	mm
E02M000	1184.026	12.036
E02M001	765.651	16.690
E02M002	1166.658	19.233
E02M003	940.786	20.695
E02M004	1533.455	16.362
E02M005	1194.373	16.267
E02M006	1186.220	12.879
E02M007	1384.506	15.073
E02M008	1004.714	22.625
E02M009	1204.049	20.194
Average	1156.444	17.205
Std Dev (n-1)	216.501	3.439
% CV	0.187	0.200

Figure 100. 150 Gelatin Extensibility Test Printout at 92 hrs.

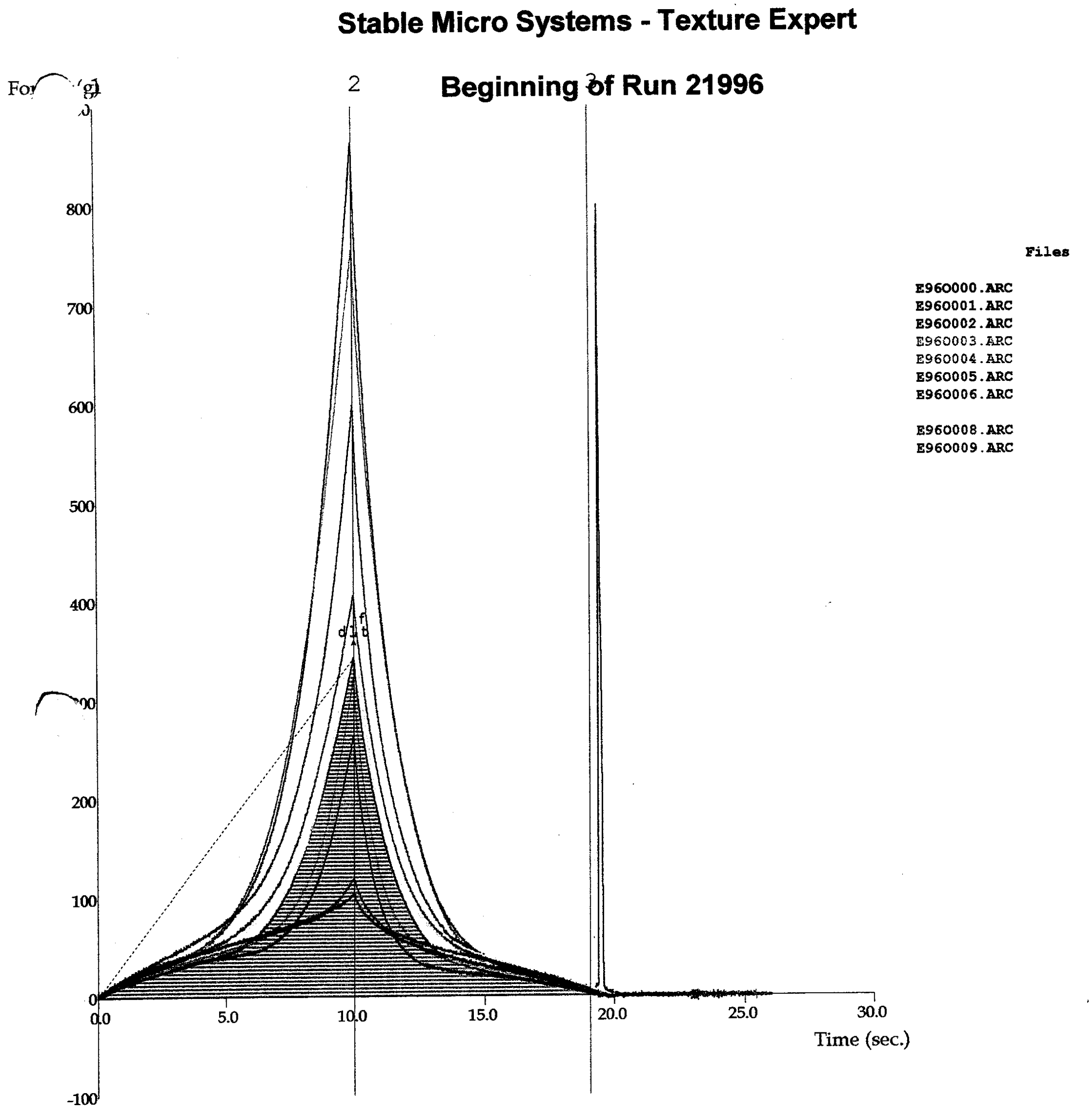
APPENDIX 4. Gel Extensibility Test Results (Continued).



Test ID	Force 1	Distance 1
	g	mm
E22002E0	1348.936	15.044
E22002E1	992.850	13.566
E22002E2	914.580	11.560
E22002E3	1789.291	16.313
E22002E4	1671.185	17.944
E22002E5	1480.944	14.995
E22002E6	587.300	8.023
E22002E7	2192.701	16.318
E22002E8	807.518	9.232
E22002E9	2023.201	15.556
Average	1380.851	13.855
Std Dev (n-1)	543.676	3.252
% CV	0.394	0.235

Figure 101. 150 Gelatin Extensibility Test Printout at 96 hrs.

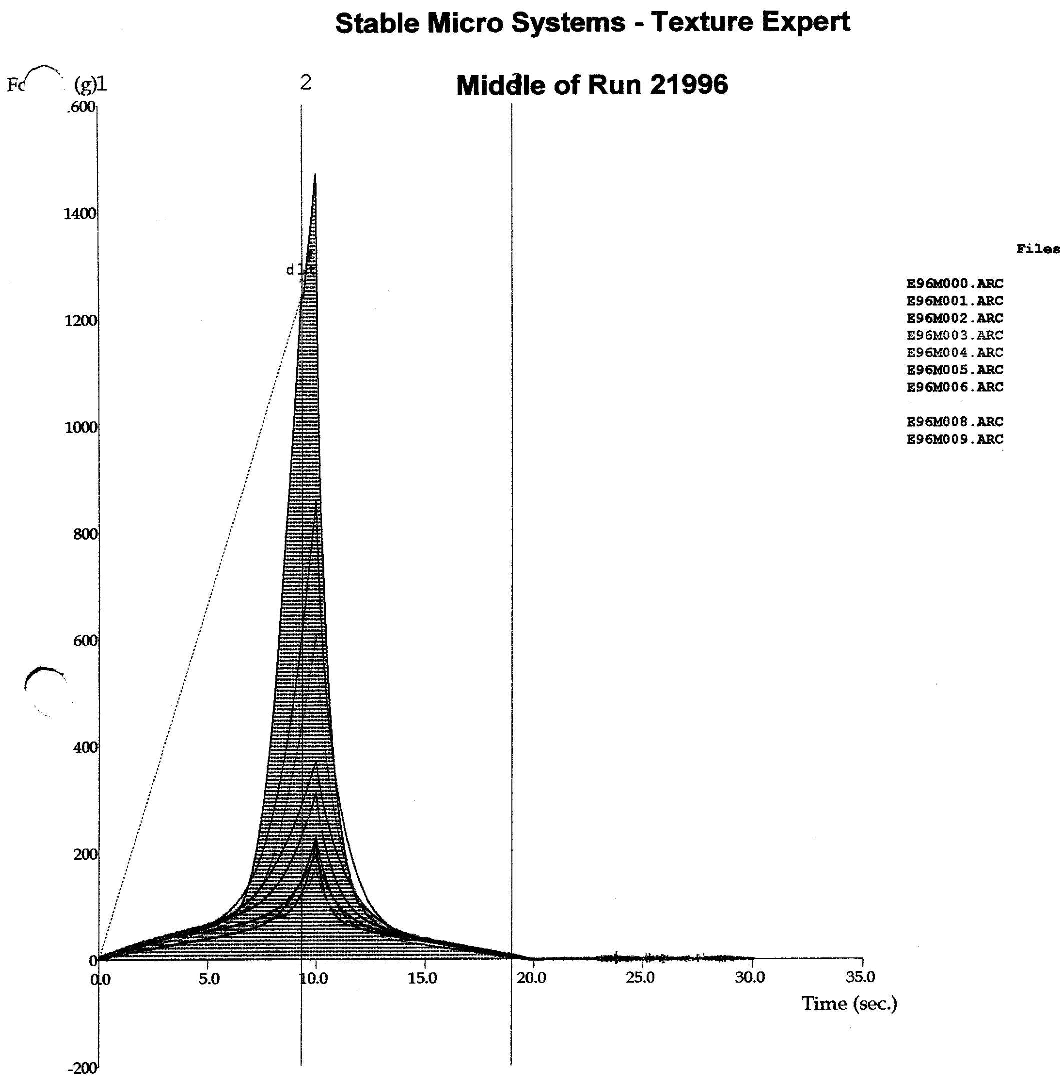
Appendix 5. Resilience Test Results



Test ID	Firmness	Area	Stifness	Area	Resilience	Distance1	Distance2	Springiness	Time 1
	g	g s	g/s	g s	%	mm	mm	%	s
E960000	343.037	814.624	34.255	572.401	70.266	5.000	4.530	90.600	10.000
E960001	35.066	54.457	11.966	924.073	1696.886	1.435	8.068	562.234	2.870
E960002	863.177	1753.954	86.439	1360.446	77.565	4.999	4.948	98.980	9.990
E960003	755.108	1727.311	75.427	1208.034	69.937	5.000	4.751	95.020	9.995
E960004	322.772	710.530	32.213	485.230	68.291	5.000	4.561	91.220	9.995
E960005	49.082	78.185	15.371	2196.026	2808.756	1.540	8.084	524.939	3.080
E960006	106.072	523.764	10.535	346.060	66.072	4.996	4.487	89.812	9.985
E960007	389.843	893.292	39.147	711.720	79.674	4.997	4.837	96.798	9.990
E960008	120.541	522.384	12.059	392.880	75.209	4.999	4.656	93.139	9.990
E960009	406.053	999.364	40.764	748.650	74.913	5.000	4.655	93.100	9.995
Average	339.075	807.786	35.818	894.552	508.757	4.297	5.358	183.584	8.589
Std Dev (n-1)	285.516	582.735	26.610	566.529	955.841	1.481	1.440	189.962	2.959
% CV	0.842	0.721	0.743	0.633	1.879	0.345	0.269	1.035	0.345

Figure 102. 195 Gelatin Resilience Test Printout at 32 hrs.

APPENDIX 5. Resilience Test Results (Continued).



Test ID	Firmness	Area	Stifness	Area	Resilience	Distance1	Distance2	Springiness	Time 1
	g	g s	g/s	g s	%	mm	mm	%	s
E96M000	1244.828	1726.003	133.141	1929.757	111.805	4.677	4.813	102.908	9.345
E96M001	227.107	534.946	22.652	421.600	78.812	5.000	4.716	94.320	10.000
E96M002	219.923	564.512	21.894	411.697	72.930	5.000	4.603	92.060	10.000
E96M003	33.353	41.460	11.879	942.792	2273.980	1.373	8.242	600.296	2.745
E96M004	609.245	1189.377	60.794	836.874	70.362	5.000	4.830	96.600	9.995
E96M005	196.902	615.380	19.483	484.543	78.739	4.988	4.797	96.171	9.970
E96M006	312.432	869.501	30.977	585.171	67.300	5.000	4.736	94.720	9.995
E96M007	35.682	37.439	15.528	1115.959	2980.739	1.050	8.530	812.388	2.100
E96M008	371.637	992.221	37.053	695.623	70.108	5.000	4.517	90.340	10.005
E96M009	858.106	1605.089	85.658	1039.597	64.769	5.000	4.629	92.580	9.995
Average	410.922	817.593	43.906	846.361	586.954	4.209	5.441	217.238	8.415
Std Dev (n-1)	386.636	578.564	38.829	457.574	1088.294	1.585	1.557	262.605	3.168
% CV	0.941	0.708	0.884	0.541	1.854	0.377	0.286	1.209	0.377

Figure 103. 195 Gelatin Resilience Test Printout at 36 hrs.

APPENDIX 5. Resilience Test Results (Continued).

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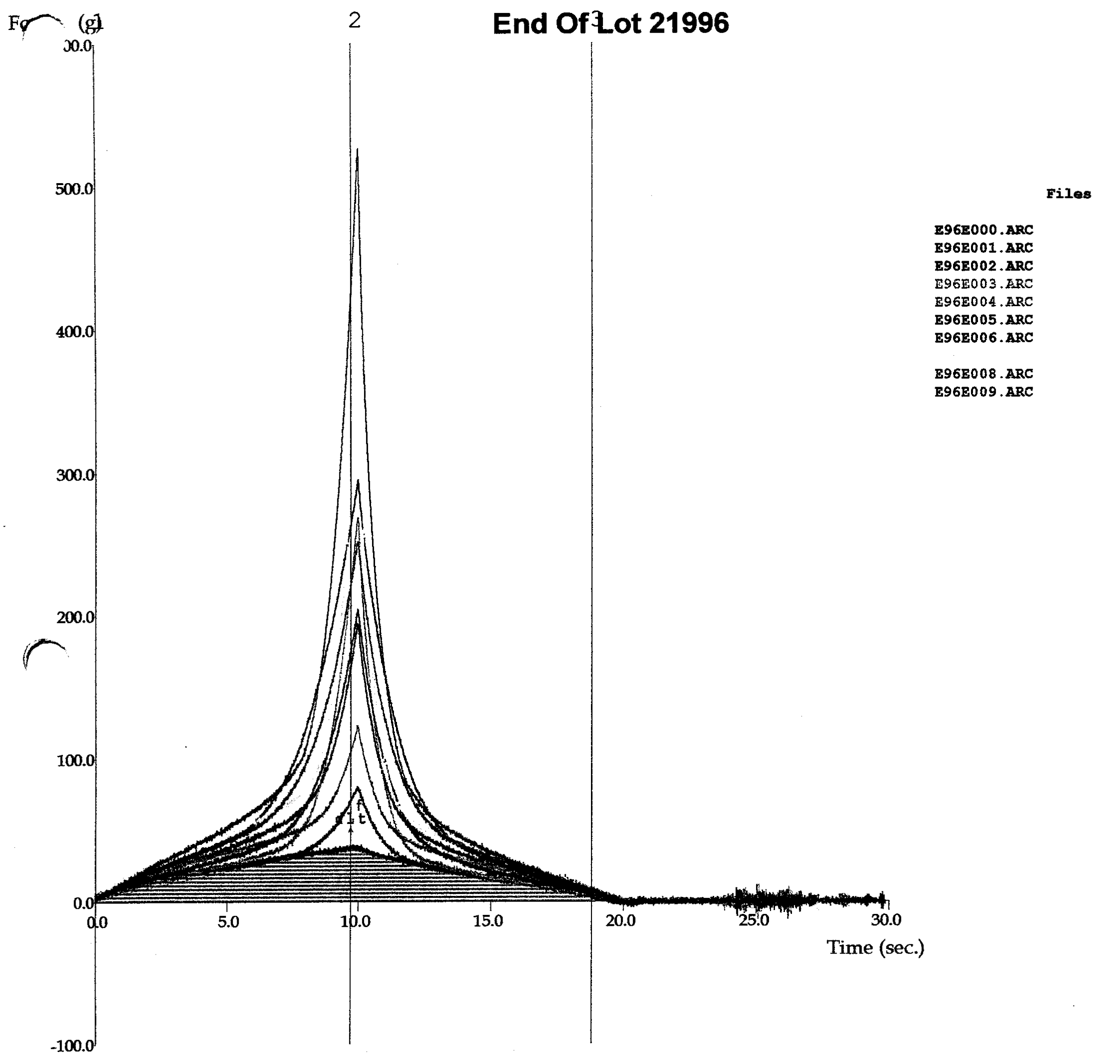
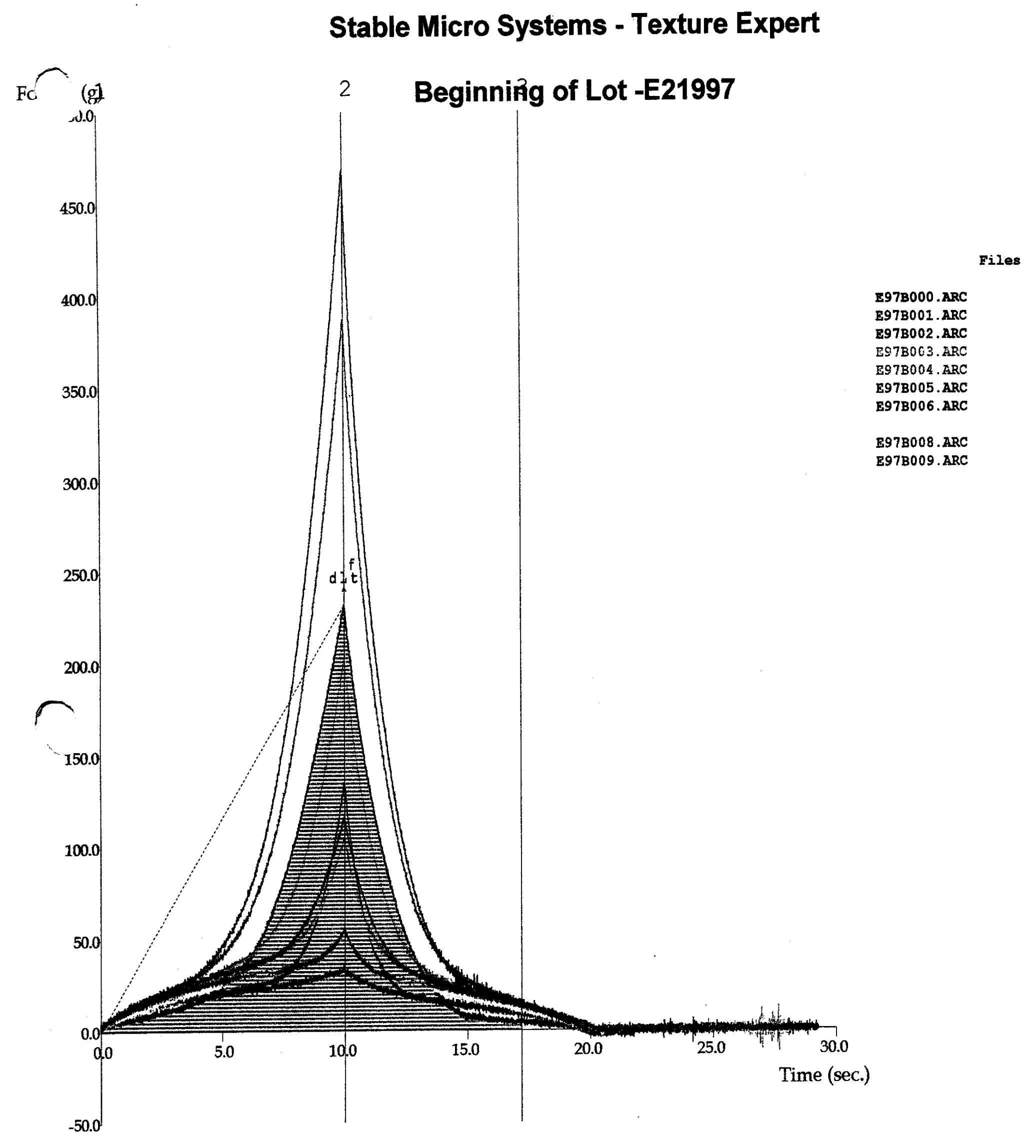


Test ID	Firmness	Area	Stifness	Area	Resilience	Distance1	Distance2	Springiness	Time 1
	g	g s	g/s	g s	%	mm	mm	%	s
E96E000	39.420	225.723	3.834	180.657	80.035	4.863	4.532	93.194	9.720
E96E001	54.776	186.723	7.781	663.831	355.516	3.428	6.277	183.110	6.850
E96E002	80.166	265.639	7.869	200.030	75.301	5.000	4.573	91.460	9.995
E96E003	268.558	444.750	26.674	325.067	73.090	5.000	4.557	91.140	9.995
E96E004	41.600	142.656	6.607	541.004	379.237	3.027	6.775	223.820	6.050
E96E005	25.275	44.400	6.936	780.303	1757.439	1.642	7.951	484.230	3.280
E96E006	295.350	719.409	29.412	555.789	77.256	5.000	4.668	93.360	10.000
E96E007	363.329	650.768	36.032	475.939	73.135	5.000	4.756	95.120	10.000
E96E008	251.468	614.559	25.052	498.484	81.112	4.997	4.753	95.117	9.985
E96E009	51.688	124.086	10.619	1376.001	1108.909	2.403	7.119	296.257	4.805
Average	147.163	341.871	16.082	559.711	406.103	4.036	5.596	174.681	8.068
Std Dev (n-1)	130.831	245.374	11.823	344.008	574.901	1.296	1.302	130.344	2.591
% CV	0.889	0.718	0.735	0.615	1.416	0.321	0.233	0.746	0.321

Figure 104. 195 Gelatin Resilience Test Printout at 40 hrs.



Test ID	Firmness	Area	Stifness	Area	Resilience	Distance1	Distance2	Springiness	Time 1
	g	g s	g/s	g s	%	mm	mm	%	s
E97B000	232.316	544.569	23.258	381.258	70.011	5.000	3.605	72.100	10.005
E97B001	135.556	294.295	13.386	211.198	71.764	5.000	4.424	88.480	9.995
E97B002	15.491	13.020	8.782	323.239	2482.634	0.844	8.563	1014.583	1.690
E97B003	202.220	504.468	20.022	385.969	76.510	4.999	4.736	94.739	10.015
E97B004	105.914	338.298	10.519	256.339	75.773	5.000	4.640	92.800	10.010
E97B005	468.782	1055.616	46.626	756.045	71.621	5.000	4.719	94.380	10.000
E97B006	56.400	262.788	5.577	203.789	77.549	4.995	4.668	93.453	9.985
E97B007	113.314	307.408	11.136	231.296	75.241	5.000	4.631	92.620	10.010
E97B008	387.439	881.992	38.488	683.764	77.525	5.000	4.836	96.720	9.995
E97B009	116.557	372.075	11.498	280.726	75.449	5.000	4.681	93.620	10.015
Average	183.399	457.453	18.929	371.362	315.408	4.584	4.950	183.350	9.172
Std Dev (n-1)	144.460	308.017	13.615	195.221	761.490	1.314	1.317	292.149	2.629
% CV	0.788	0.673	0.719	0.526	2.414	0.287	0.266	1.593	0.287

Figure 105. 195 Gelatin Resilience Test Printout at 64 hrs.

APPENDIX 5. Resilience Test Results (Continued).

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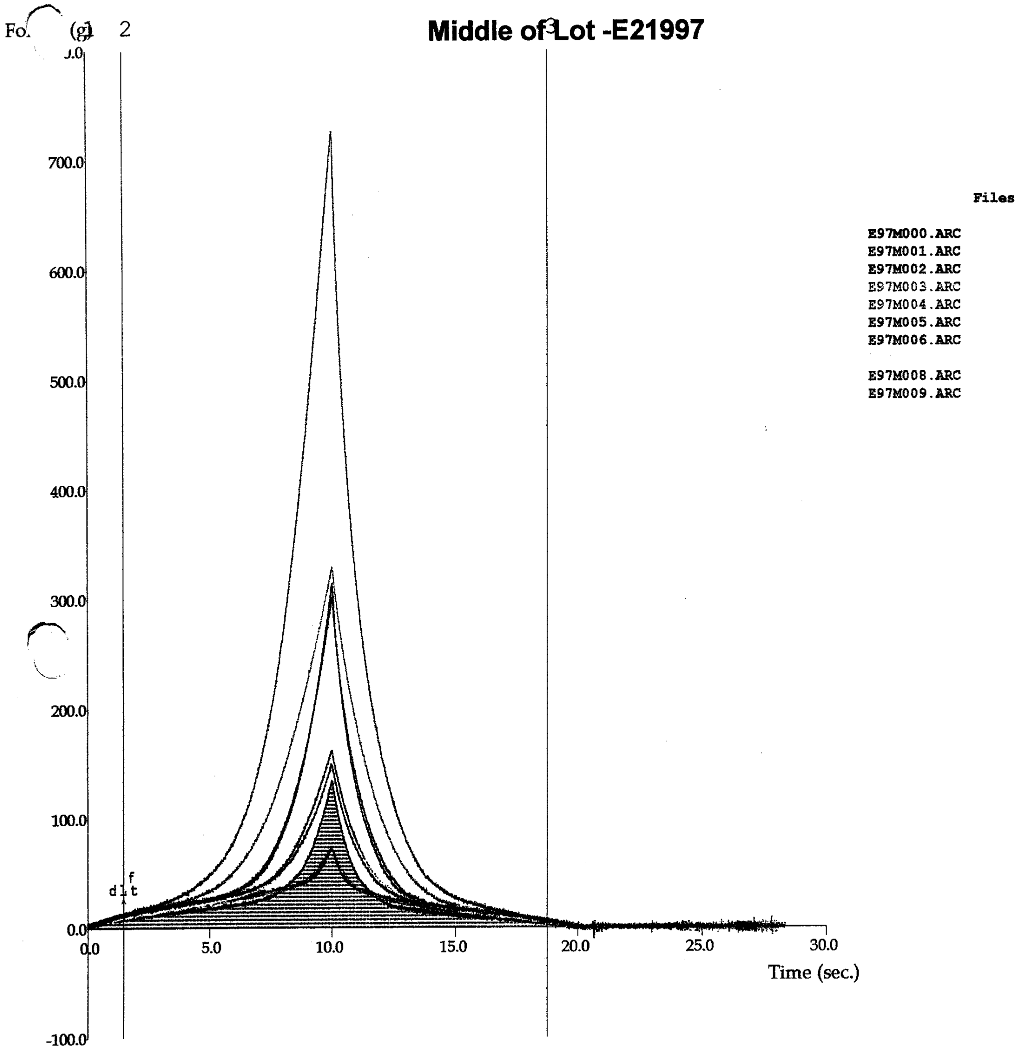
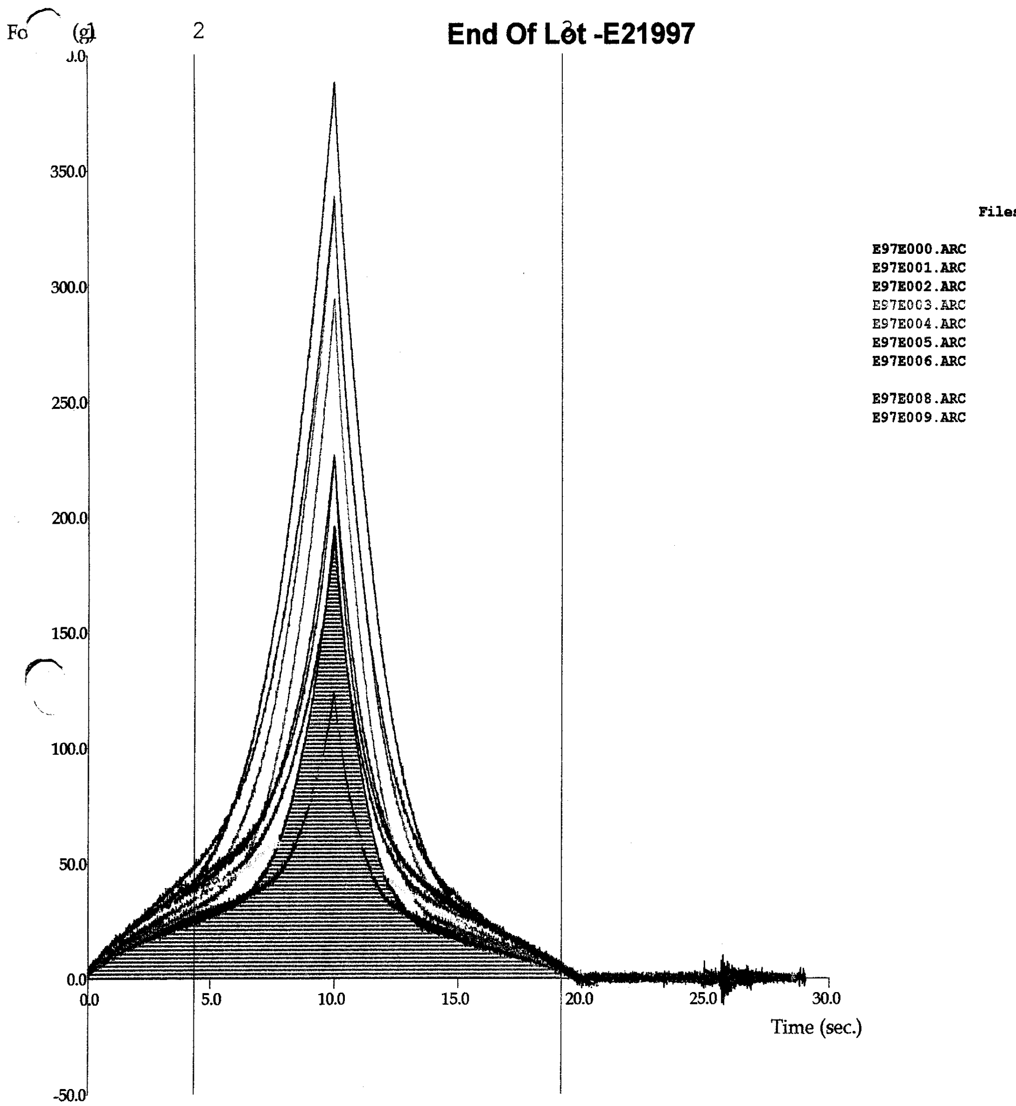


Test ID	Firmness	Area	Stifness	Area	Resilience	Distance1	Distance2	Springiness	Time 1
	g	g s	g/s	g s	%	mm	mm	%	s
E97M000	11.646	8.487	7.052	454.812	5358.925	0.746	8.624	1156.042	1.490
E97M001	302.101	563.787	30.071	426.106	75.579	5.000	4.584	91.680	10.015
E97M002	74.972	262.460	7.464	194.468	74.094	5.000	4.649	92.980	10.015
E97M003	329.238	756.016	32.760	583.496	77.180	4.998	4.569	91.417	9.990
E97M004	75.012	258.762	7.262	191.474	73.996	4.990	4.713	94.449	10.040
E97M005	314.329	561.783	31.324	418.575	74.508	5.000	4.546	90.920	10.005
E97M006	162.327	369.270	16.074	276.035	74.752	5.000	4.415	88.300	10.015
E97M007	149.124	349.828	14.950	283.312	80.986	4.997	4.599	92.035	9.985
E97M008	725.824	1432.522	72.441	975.465	68.094	5.000	4.802	96.040	10.000
E97M009	16.947	18.114	7.889	635.694	3509.407	0.947	8.637	912.047	1.895
Average	216.152	458.103	22.729	443.944	946.752	4.168	5.414	280.591	8.345
Std Dev (n-1)	215.594	415.470	20.393	240.327	1889.021	1.751	1.698	401.253	3.508
% CV	0.997	0.907	0.897	0.541	1.995	0.420	0.314	1.430	0.420

Figure 106. 195 Gelatin Resilience Test Printout at 68 hrs.

APPENDIX 5. Resilience Test Results (Continued).

Stable Micro Systems - Texture Expert

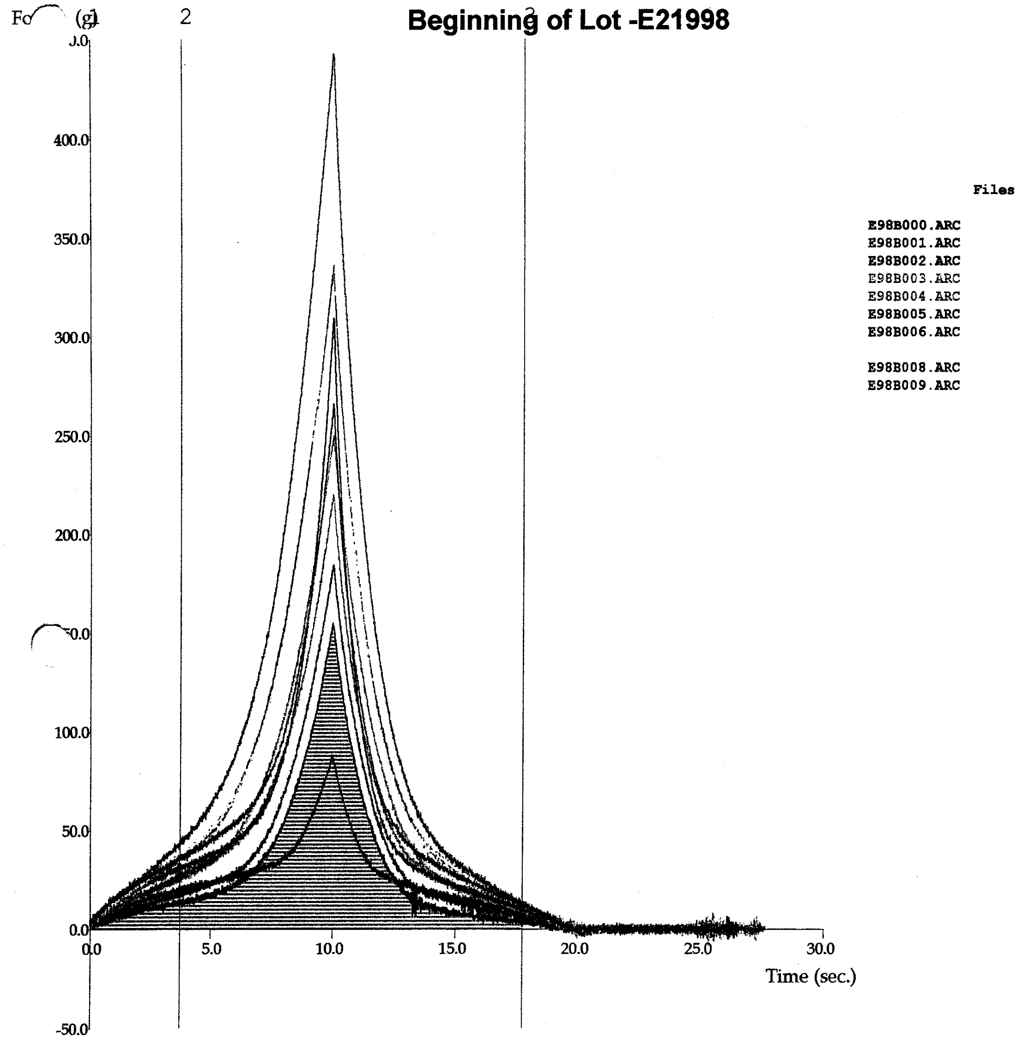


Test ID	Firmness	Area	Stifness	Area	Resilience	Distance1	Distance2	Springiness	Time 1
	g	g s	g/s	g s	%	mm	mm	%	s
E97E000	28.064	68.419	5.936	706.733	1032.948	2.192	7.392	337.229	4.380
E97E001	124.215	355.051	12.396	271.280	76.406	4.993	4.675	93.631	9.980
E97E002	14.442	7.912	12.549	1731.818	21888.498	0.551	9.119	1655.006	1.105
E97E003	294.247	665.314	29.195	491.497	73.874	5.000	4.748	94.960	10.015
E97E004	334.966	805.067	33.350	621.118	77.151	5.000	4.759	95.180	9.995
E97E005	190.893	531.690	19.097	406.455	76.446	5.000	4.643	92.860	9.995
E97E006	226.283	609.460	22.545	469.873	77.097	4.997	4.709	94.237	9.985
E97E007	131.026	434.078	12.991	309.241	71.241	5.000	4.608	92.160	10.015
E97E008	19.019	11.992	13.128	1059.600	8835.891	0.590	9.135	1548.319	1.180
E97E009	338.541	911.211	33.887	664.841	72.962	4.999	4.660	93.219	9.990
Average	170.170	440.019	19.507	673.246	3228.251	3.832	5.845	419.680	7.664
Std Dev (n-1)	127.111	326.330	9.820	436.323	7102.970	1.929	1.925	628.073	3.858
% CV	0.747	0.742	0.503	0.648	2.200	0.503	0.329	1.497	0.503

Figure 107. 195 Gelatin Resilience Test Printout at 72 hrs.

APPENDIX 5. Resilience Test Results (Continued).

Stable Micro Systems - Texture Expert

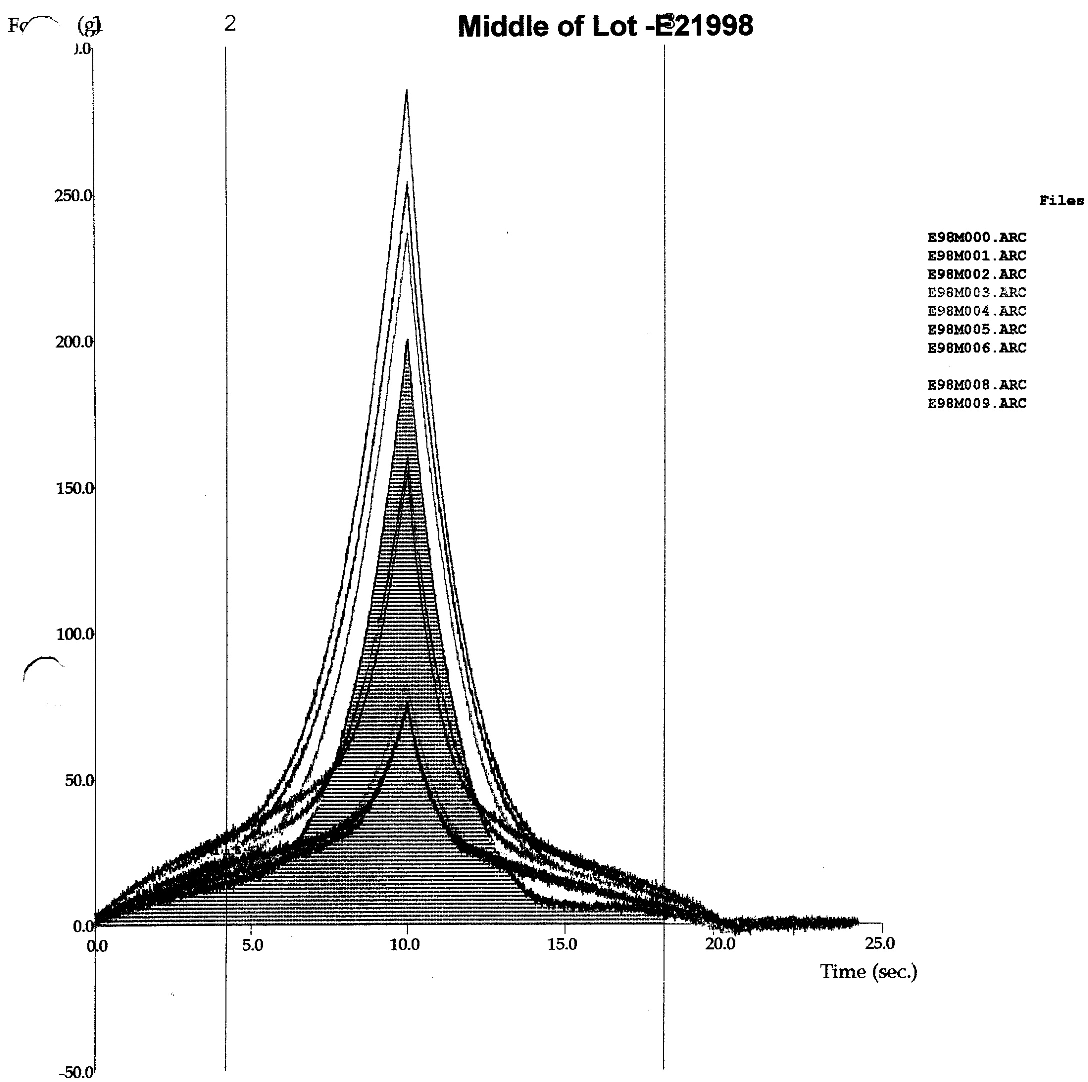


Test ID	Firmness	Area	Stifness	Area	Resilience	Distance1	Distance2	Springiness	Time 1
	g	g s	g/s	g s	%	mm	mm	%	s
E98B000	17.267	31.343	4.662	536.453	1711.556	1.884	6.999	371.500	3.765
E98B001	184.277	414.248	18.160	314.756	75.983	5.000	4.479	89.580	10.005
E98B002	88.157	268.899	8.580	209.373	77.863	4.979	4.547	91.324	9.950
E98B003	250.007	619.463	24.654	483.607	78.069	5.000	4.767	95.340	10.015
E98B004	219.860	547.898	21.853	412.334	75.257	5.000	4.553	91.060	9.995
E98B005	24.045	19.037	13.235	1030.887	5415.176	0.835	8.773	1050.669	1.670
E98B006	22.251	20.545	11.365	1465.088	7131.117	0.868	8.813	1015.332	1.735
E98B007	340.323	861.821	33.889	618.763	71.797	5.000	4.584	91.680	10.000
E98B008	442.605	1120.792	44.075	782.570	69.823	4.997	4.626	92.576	9.985
E98B009	265.946	653.551	26.405	472.347	72.274	5.000	4.622	92.440	9.995
Average	185.474	455.760	20.688	632.618	1477.891	3.856	5.676	308.150	7.712
Std Dev (n-1)	146.205	376.898	12.126	374.186	2609.887	1.857	1.807	391.935	3.715
% CV	0.788	0.827	0.586	0.591	1.766	0.482	0.318	1.272	0.482

Figure 108. 195 Gelatin Resilience Test Printout at 88 hrs.

APPENDIX 5. Resilience Test Results (Continued).

Stable Micro Systems - Texture Expert

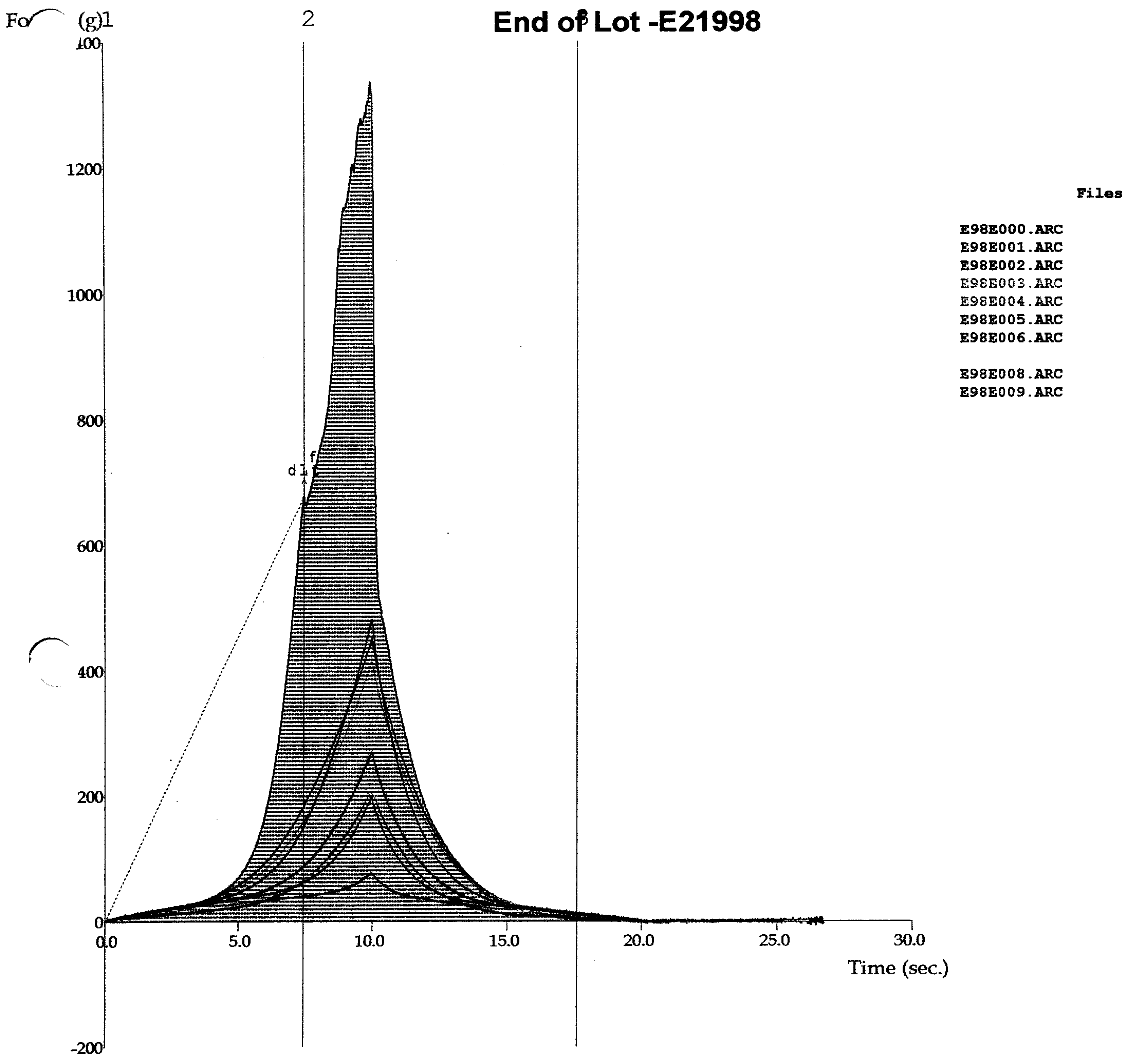


Test ID	Firmness	Area	Stifness	Area	Resilience	Distance1	Distance2	Springiness	Time 1
	g	g s	g/s	g s	%	mm	mm	%	s
E98M000	16.807	36.922	4.124	654.556	1772.808	2.115	6.955	328.845	4.230
E98M001	160.509	406.371	16.197	305.564	75.193	5.000	4.584	91.680	10.020
E98M002	14.701	14.451	6.826	1046.440	7241.298	1.048	8.391	800.675	2.095
E98M003	82.767	252.285	8.141	192.512	76.307	5.000	4.603	92.060	9.995
E98M004	236.403	529.560	23.736	417.719	78.880	5.000	4.460	89.200	10.010
E98M005	285.292	687.517	28.397	521.162	75.804	5.000	4.713	94.260	10.000
E98M006	28.643	96.513	4.680	343.505	355.916	2.977	6.538	219.618	5.950
E98M007	15.960	10.477	10.527	682.429	6513.592	0.706	8.806	1247.320	1.410
E98M008	154.672	425.460	15.256	330.786	77.748	4.996	4.780	95.677	9.985
E98M009	76.117	228.876	7.471	173.230	75.687	4.990	4.448	89.139	10.035
Average	107.187	268.843	12.536	466.790	1634.323	3.683	5.828	314.847	7.373
Std Dev (n-1)	97.918	236.533	8.246	267.542	2817.759	1.799	1.712	396.203	3.605
% CV	0.914	0.880	0.658	0.573	1.724	0.488	0.294	1.258	0.489

Figure 109. 195 Gelatin Resilience Test Printout at 92 hrs.

APPENDIX 5. Resilience Test Results (Continued).

Stable Micro Systems - Texture Expert

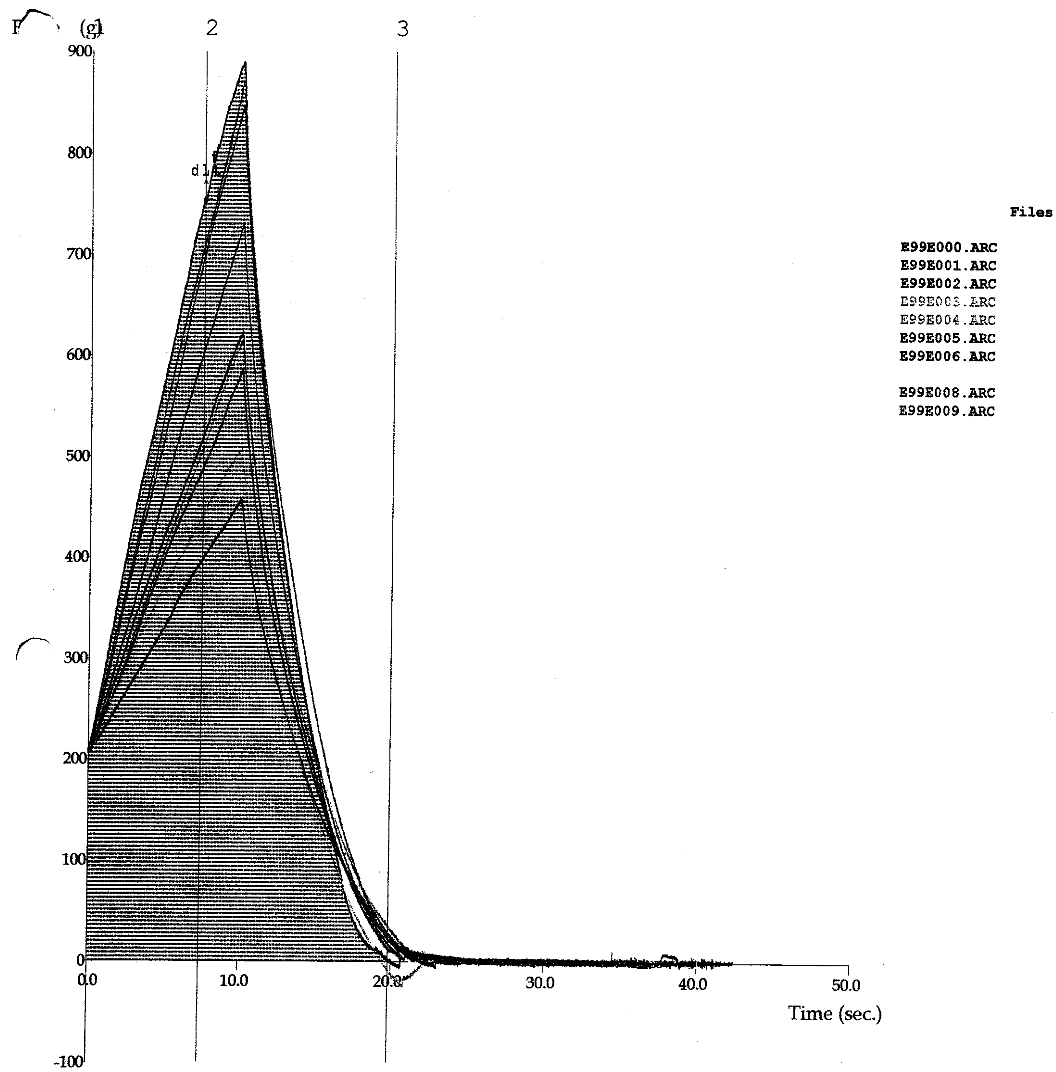


Test ID	Firmness	Area	Stifness	Area	Resilience	Distance1	Distance2	Springiness	Time 1
	g	g s	g/s	g s	%	mm	mm	%	s
E98E000	676.956	771.932	90.888	3611.810	467.892	3.729	5.070	135.962	7.455
E98E001	30.119	45.066	8.479	1981.160	4396.130	1.667	7.906	474.269	3.335
E98E002	12.568	9.637	7.952	835.640	8671.163	0.818	8.606	1052.088	1.635
E98E003	414.308	1039.767	41.232	835.714	80.375	4.995	4.572	91.532	9.985
E98E004	273.144	661.830	27.280	515.956	77.959	5.000	4.689	93.780	10.020
E98E005	78.216	311.405	7.836	231.468	74.330	5.000	4.517	90.340	10.010
E98E006	481.114	1081.686	47.850	778.003	71.925	5.000	4.852	97.040	10.010
E98E007	208.186	507.931	20.921	393.513	77.474	5.000	4.520	90.400	10.000
E98E008	269.061	655.478	26.894	500.788	76.400	5.000	4.564	91.280	10.015
E98E009	197.542	510.318	19.482	385.172	75.477	5.000	4.679	93.580	10.010
Average	264.121	559.505	29.881	1006.922	1406.913	4.121	5.398	231.027	8.248
Std Dev (n-1)	210.908	365.417	25.392	1038.537	2886.380	1.581	1.525	312.087	3.165
% CV	0.799	0.653	0.850	1.031	2.052	0.384	0.283	1.351	0.384

Figure 110. 195 Gelatin Resilience Test Printout at 96 hrs.

APPENDIX 5. Resilience Test Results (Continued).

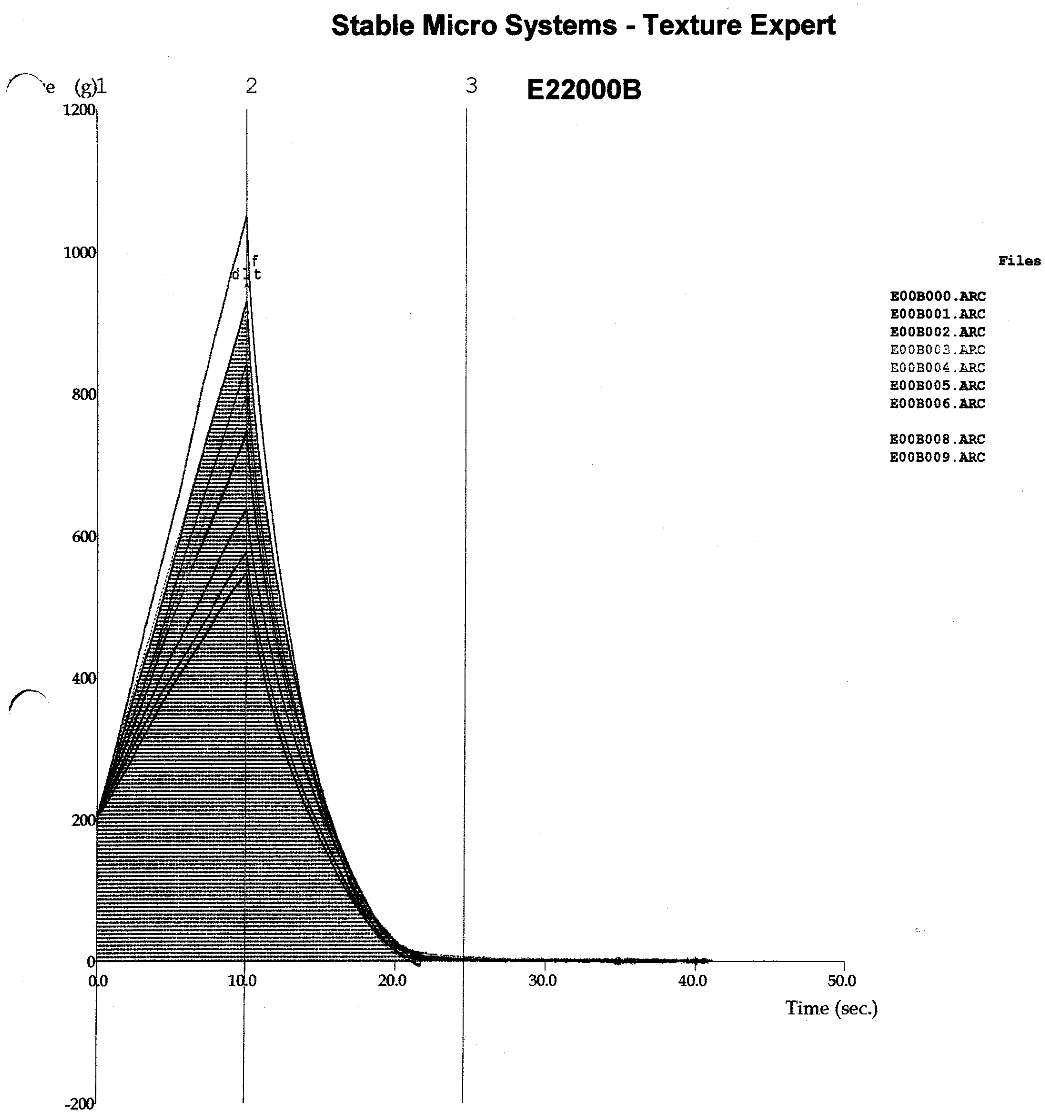
Stable Micro Systems - Texture Expert



Test ID	Firmness	Area	Stifness	Area	Resilience	Distance1	Distance2	Springiness	Time 1
	g	g s	g/s	g s	%	mm	mm	%	s
E99E000	755.982	3636.951	73.678	4774.975	131.291	3.731	6.190	165.907	7.455
E99E001	456.843	3315.677	25.069	1859.866	56.093	5.000	6.106	122.119	10.000
E99E002	586.787	3947.851	38.045	2127.450	53.889	4.999	5.699	114.002	9.995
E99E003	507.437	3615.025	30.385	1813.719	50.172	4.969	4.711	94.808	9.930
E99E004	615.333	4066.192	40.863	2360.279	58.046	5.000	5.937	118.739	9.995
E99E005	624.156	4172.902	41.991	2220.937	53.223	5.000	6.111	122.219	9.995
E99E006	731.525	4663.140	52.607	2622.172	56.232	4.998	5.818	116.405	9.990
E99E007	777.839	4923.071	57.509	2724.592	55.343	4.999	5.510	110.221	9.990
E99E008	846.978	5275.472	64.027	2760.047	52.318	5.000	5.415	108.299	9.995
E99E009	812.676	4609.255	66.762	3836.275	83.230	4.528	7.418	163.824	9.050
Average	671.556	4222.554	49.094	2710.031	64.984	4.822	5.891	123.654	9.640
Std Dev (n-1)	132.639	630.682	16.297	929.154	25.094	0.411	0.693	23.146	0.822
% CV	0.198	0.149	0.332	0.343	0.386	0.085	0.118	0.187	0.085

Figure 111. 150 Gelatin Resilience Test Printout at 8 hrs.

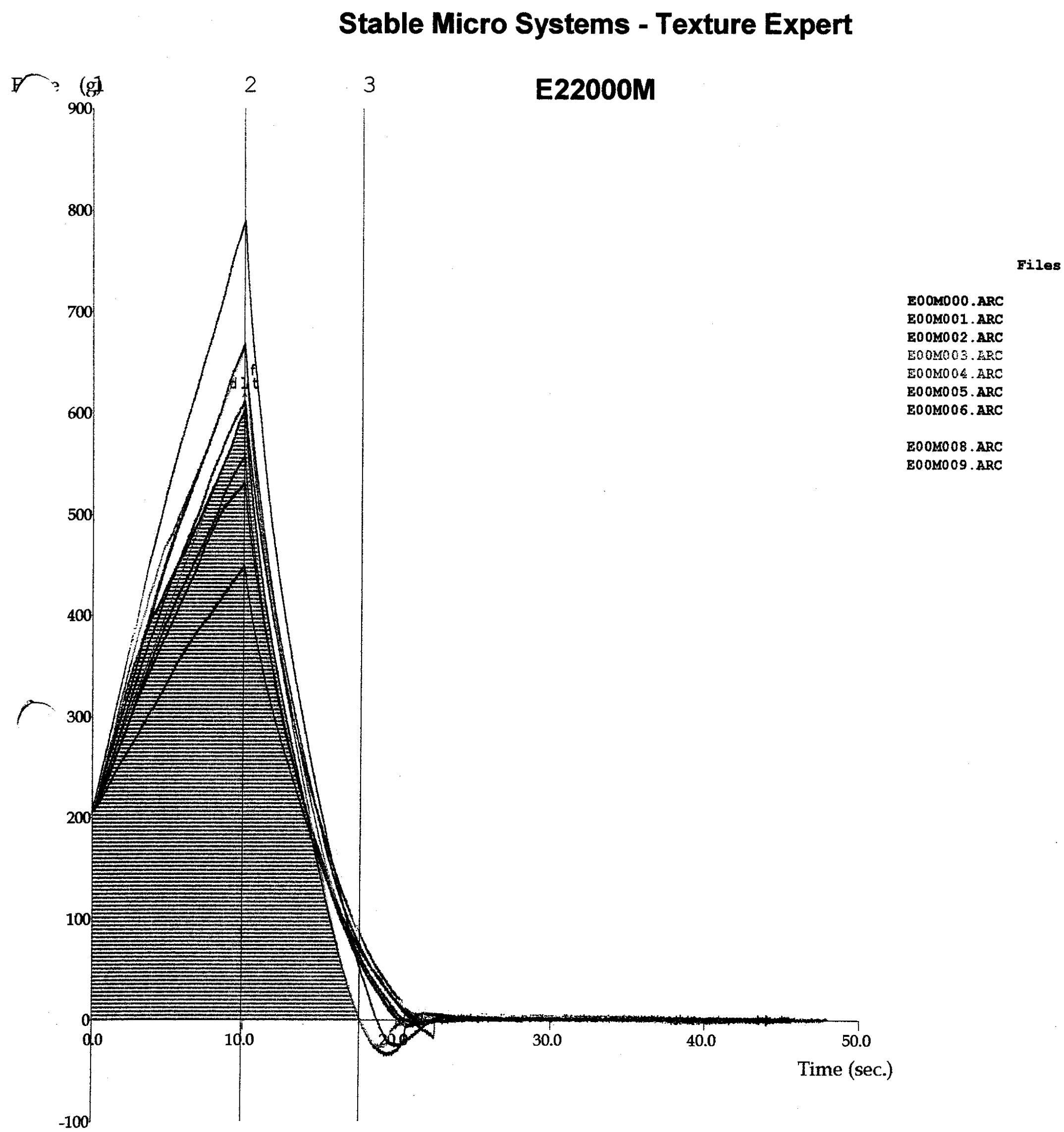
APPENDIX 5. Resilience Test Results (Continued).



Test ID	Firmness g	Area g s	Stifness g/s	Area g s	Resilience %	Distance1 mm	Distance2 mm	Springiness %	Time 1 s
E00B000	931.458	5589.975	72.734	3196.758	57.187	4.999	7.278	145.589	9.990
E00B001	546.812	3797.668	34.002	2031.356	53.490	5.000	5.461	109.219	9.995
E00B002	1050.425	6237.150	84.295	3323.098	53.279	5.000	7.158	143.159	9.995
E00B003	751.316	4757.748	54.401	2587.962	54.395	4.997	5.905	118.170	9.985
E00B004	797.305	4937.984	59.029	2779.712	56.292	5.000	6.509	130.179	9.995
E00B005	744.186	4726.805	53.793	2789.036	59.005	4.990	6.841	137.093	9.975
E00B006	638.261	4238.141	43.237	2548.214	60.126	4.985	7.858	157.633	9.965
E00B007	724.178	4728.844	51.732	2738.248	57.905	4.997	7.238	144.846	9.985
E00B008	577.518	3954.297	37.165	2170.832	54.898	4.990	6.080	121.843	9.975
E00B009	844.336	5147.098	63.788	2881.855	55.990	5.000	7.033	140.659	9.995
Average	760.580	4811.571	55.418	2704.707	56.257	4.996	6.736	134.839	9.986
Std Dev (n-1)	155.429	736.011	15.543	400.875	2.310	0.005	0.736	14.788	0.011
% CV	0.204	0.153	0.280	0.148	0.041	0.001	0.109	0.110	0.001

Figure 112. 150 Gelatin Resilience Test Printout at 32 hrs.

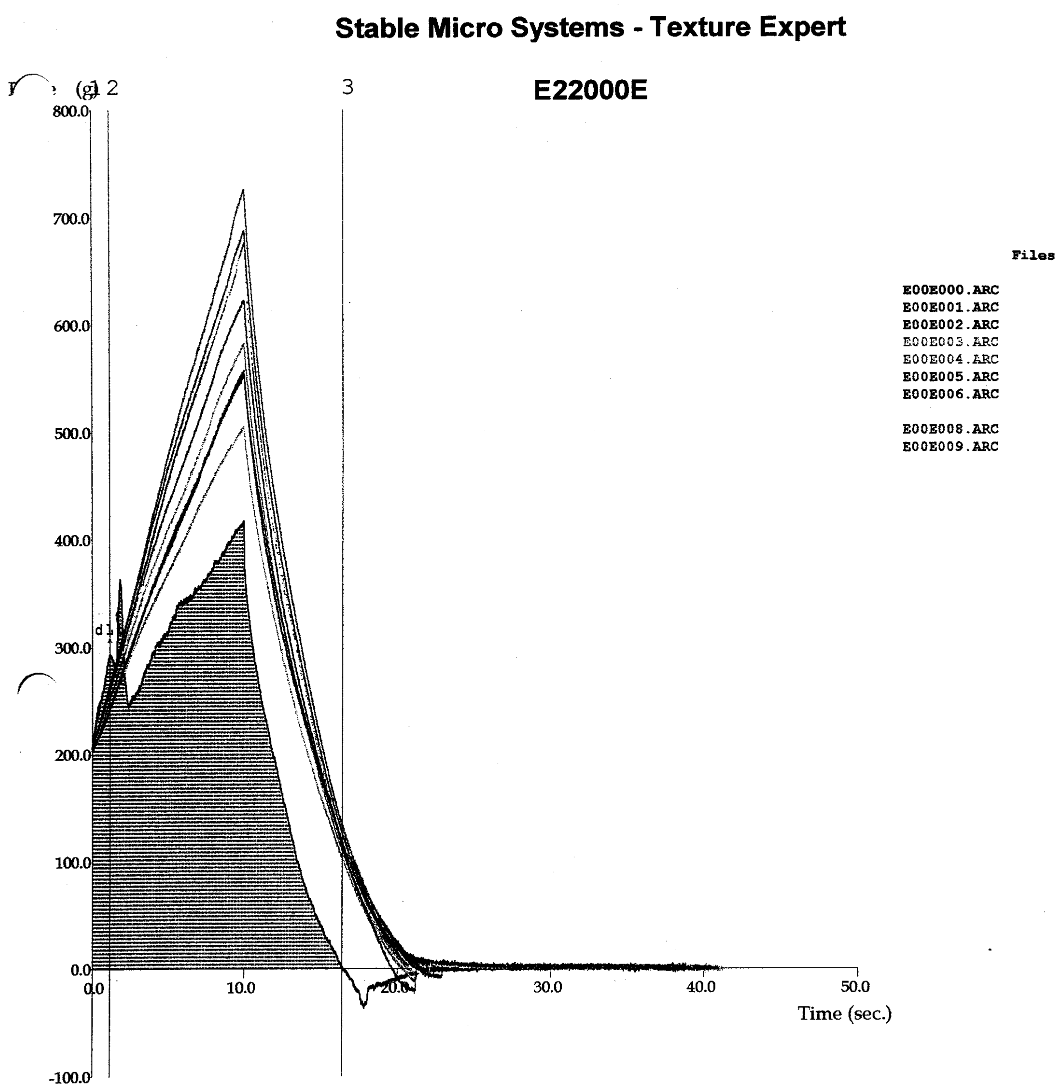
APPENDIX 5. Resilience Test Results (Continued).



Test ID	Firmness g	Area g s	Stifness g/s	Area g s	Resilience %	Distance1 mm	Distance2 mm	Springiness %	Time 1 s
E00M000	602.618	4170.136	39.701	1879.888	45.080	4.983	3.828	76.821	9.960
E00M001	532.193	3779.447	32.723	1978.383	52.346	4.990	5.145	103.106	9.975
E00M002	612.077	4146.733	40.865	2366.271	57.064	4.984	5.644	113.241	9.960
E00M003	667.884	4535.393	46.089	2021.397	44.569	4.998	3.821	76.451	9.990
E00M004	666.943	4454.026	46.229	2279.927	51.188	4.980	5.034	101.084	9.955
E00M005	669.076	4448.101	46.642	2400.947	53.977	4.999	5.556	111.141	9.990
E00M006	789.802	5093.511	58.250	2608.020	51.203	4.992	4.366	87.460	9.980
E00M007	878.103	5422.868	67.296	3050.078	56.245	5.000	8.540	170.800	10.010
E00M008	449.343	3283.386	24.632	1842.395	56.113	4.973	5.471	110.013	9.940
E00M009	559.162	3858.411	35.322	1965.247	50.934	4.986	5.065	101.584	9.965
Average	642.720	4319.201	43.775	2239.255	51.872	4.989	5.247	105.170	9.973
Std Dev (n-1)	123.870	626.073	12.326	382.186	4.346	0.009	1.336	26.682	0.021
% CV	0.193	0.145	0.282	0.171	0.084	0.002	0.255	0.254	0.002

Figure 113. 150 Gelatin Resilience Test Printout at 36 hrs.

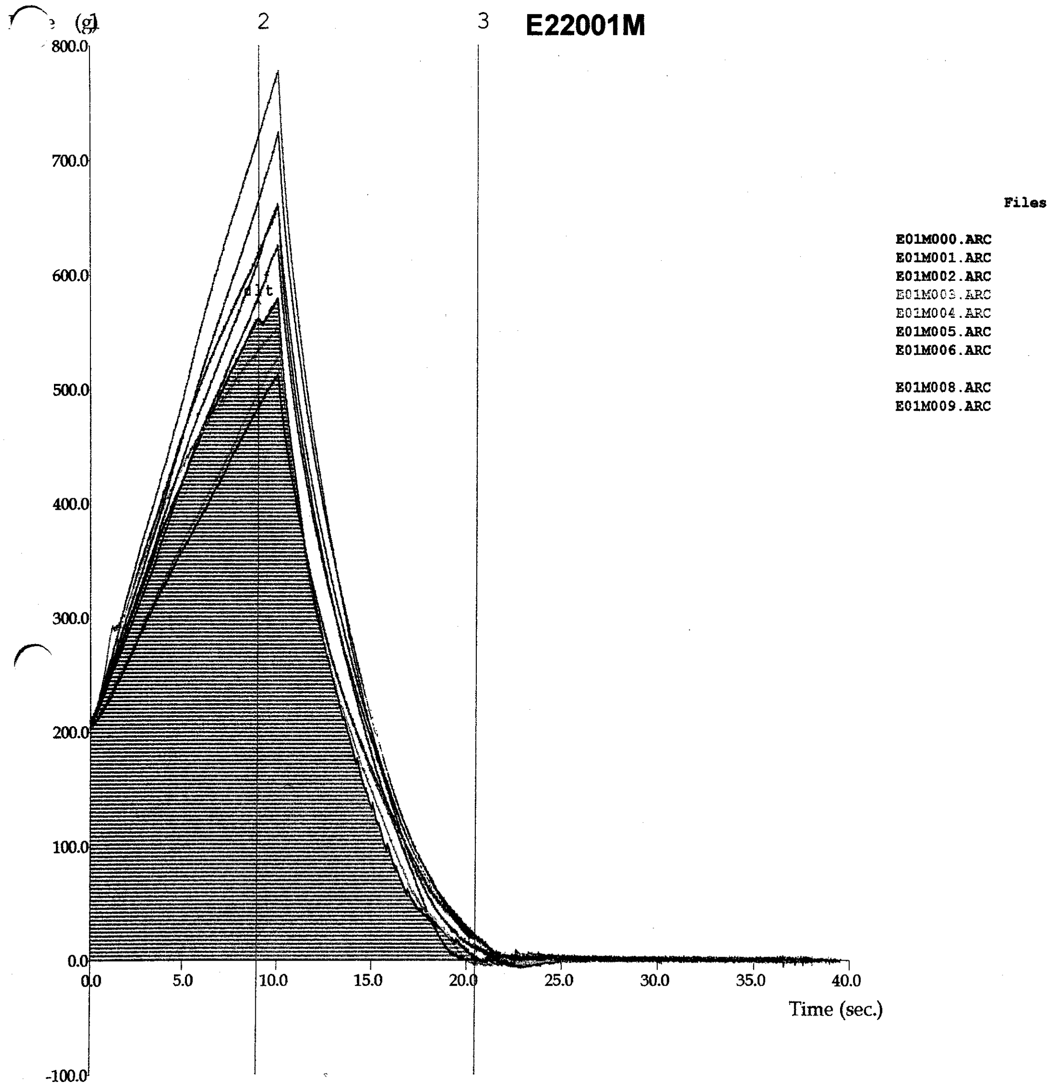
APPENDIX 5. Resilience Test Results (Continued).



Test ID	Firmness	Area	Stifness	Area	Resilience	Distance1	Distance2	Springiness	Time 1
	g	g s	g/s	g s	%	mm	mm	%	s
E00E000	293.326	298.990	71.858	3829.854	1280.930	0.592	7.614	1286.157	1.185
E00E001	557.584	3849.193	35.335	2123.200	55.160	5.000	5.763	115.259	9.995
E00E002	675.975	4430.205	47.101	2401.016	54.196	4.994	6.065	121.445	9.980
E00E003	505.333	3636.374	30.115	1901.366	52.287	4.999	5.124	102.500	9.990
E00E004	582.813	4020.144	37.785	2122.538	52.798	5.000	5.280	105.599	9.995
E00E005	623.141	4230.953	41.774	2208.113	52.189	5.000	4.807	96.140	9.995
E00E006	553.522	3847.112	34.838	2166.756	56.322	5.000	5.660	113.199	10.010
E00E007	672.617	4392.469	46.694	2407.759	54.816	5.000	5.468	109.359	9.995
E00E008	726.053	4670.767	52.099	2624.581	56.192	4.990	5.516	110.540	9.975
E00E009	688.197	4546.694	48.374	2498.242	54.946	4.999	7.020	140.427	9.995
Average	587.856	3792.290	44.597	2428.343	176.984	4.557	5.832	230.063	9.111
Std Dev (n-1)	125.184	1273.056	11.877	536.456	387.890	1.393	0.867	371.268	2.785
% CV	0.213	0.336	0.266	0.221	2.192	0.306	0.149	1.614	0.306

Figure 114. 150 Gelatin Resilience Test Printout at 40 hrs.

Stable Micro Systems - Texture Expert

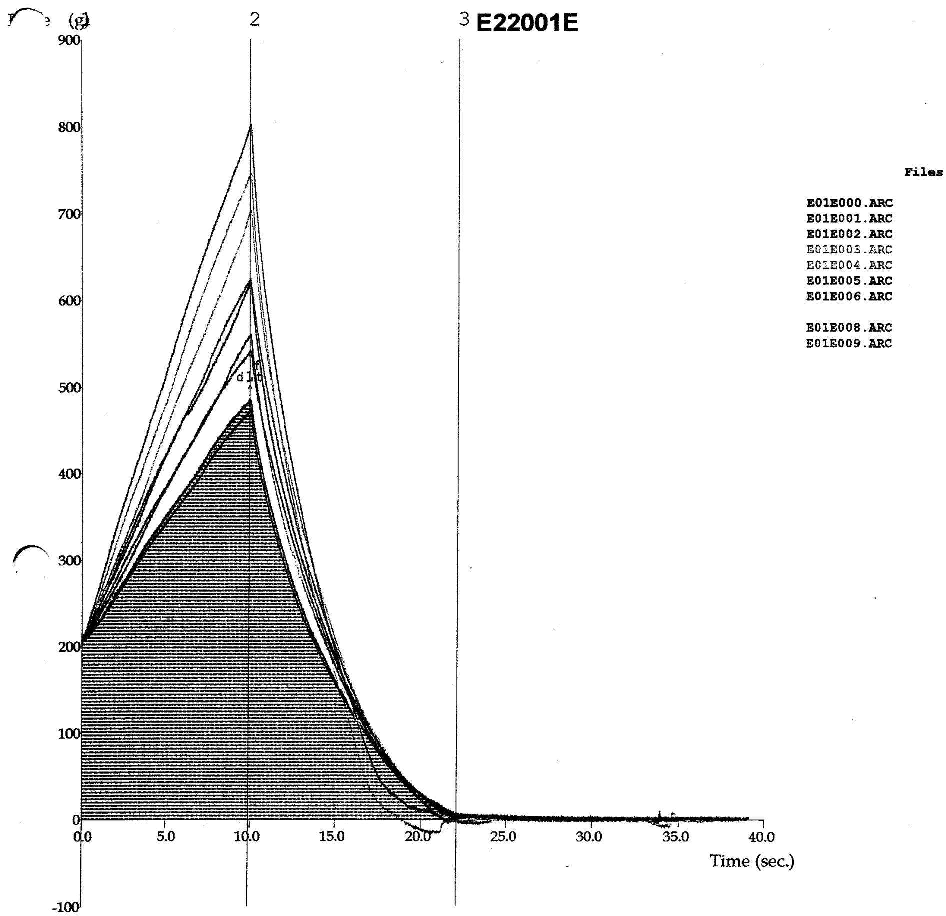


Test ID	Firmness	Area	Stifness	Area	Resilience	Distance1	Distance2	Springiness	Time 1
	g	g s	g/s	g s	%	mm	mm	%	s
E01M000	562.097	3520.853	39.735	2368.296	67.265	4.483	5.755	128.373	8.960
E01M001	626.055	4141.615	42.058	2318.590	55.983	4.996	6.062	121.336	9.985
E01M002	660.989	4483.922	45.571	2177.663	48.566	5.000	4.826	96.520	9.995
E01M003	527.413	3635.697	32.249	1967.327	54.111	5.000	5.920	118.399	9.995
E01M004	471.920	2190.136	43.023	3756.857	171.535	3.098	6.869	221.724	6.190
E01M005	662.086	4321.970	45.664	2419.641	55.985	4.988	5.705	114.373	9.970
E01M006	724.290	4583.196	51.750	2587.537	56.457	5.000	5.718	114.359	9.995
E01M007	750.002	4811.838	54.265	2643.807	54.944	5.000	6.529	130.579	9.995
E01M008	777.420	4931.893	56.983	2547.462	51.653	5.000	5.992	119.839	10.005
E01M009	513.998	3571.358	30.883	1987.612	55.654	4.999	6.069	121.403	9.995
Average	627.627	4019.248	44.218	2477.479	67.215	4.756	5.944	128.691	9.509
Std Dev (n-1)	105.701	819.448	8.640	506.854	36.963	0.605	0.540	33.986	1.210
% CV	0.168	0.204	0.195	0.205	0.550	0.127	0.091	0.264	0.127

Figure 115. 150 Gelatin Resilience Test Printout at 68 hrs.

APPENDIX 5. Resilience Test Results (Continued).

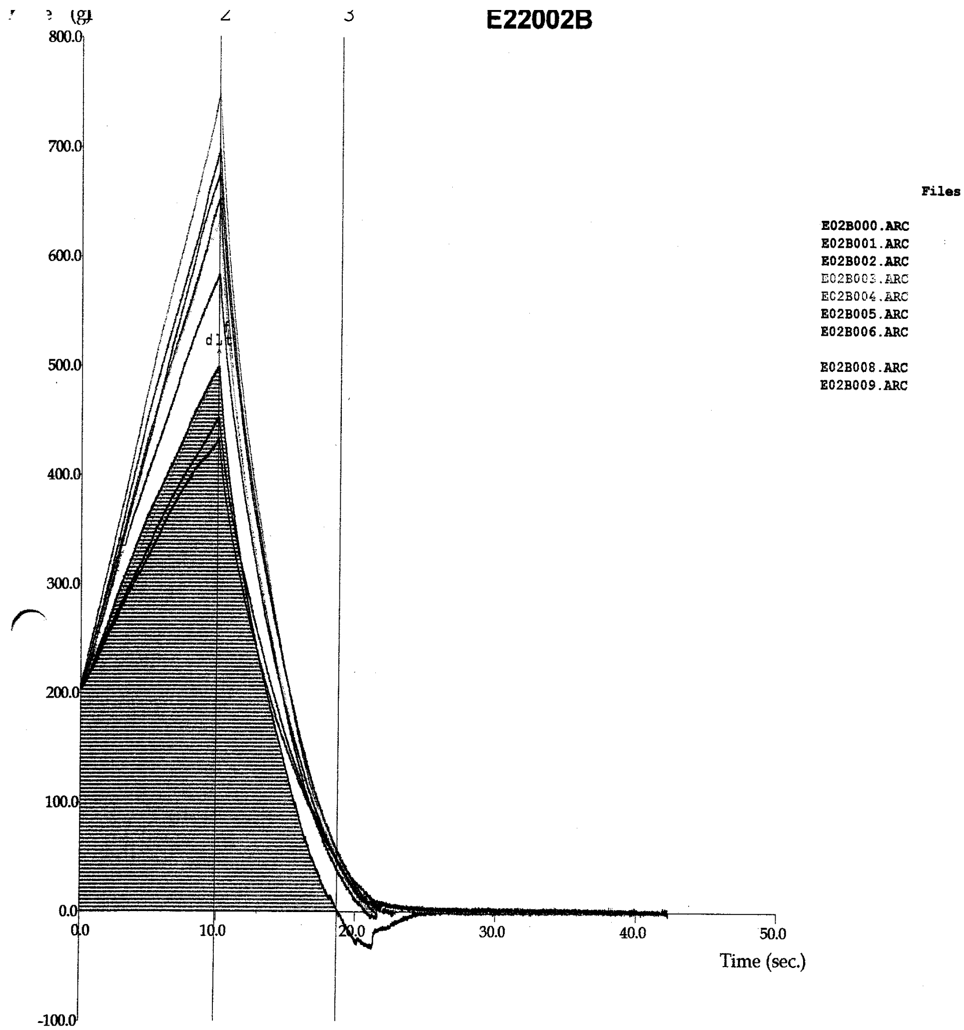
Stable Micro Systems - Texture Expert



Test ID	Firmness	Area	Stifness	Area	Resilience	Distance1	Distance2	Springiness	Time 1
	g	g s	g/s	g s	%	mm	mm	%	s
E01E000	484.941	3443.724	27.980	1930.669	56.063	4.974	6.085	122.335	9.940
E01E001	559.977	3790.318	35.378	2119.997	55.932	4.999	6.076	121.543	9.990
E01E002	619.920	4119.961	41.505	2260.442	54.866	4.998	5.744	114.925	9.990
E01E003	703.887	4490.747	49.594	2538.715	56.532	5.000	5.706	114.119	10.010
E01E004	746.156	4807.069	54.091	2203.137	45.831	5.000	4.400	88.000	9.995
E01E005	802.360	5113.132	59.533	2535.134	49.581	5.000	6.402	128.039	9.995
E01E006	472.407	3397.802	26.745	1868.555	54.993	4.998	6.401	128.070	9.990
E01E007	580.123	3950.381	37.408	2081.715	52.697	4.991	5.963	119.474	9.975
E01E008	624.849	4171.333	42.166	2384.014	57.152	4.993	5.588	111.916	9.980
E01E009	541.762	3814.178	33.619	2183.844	57.256	5.000	6.855	137.099	10.010
Average	613.638	4109.864	40.802	2210.622	54.090	4.995	5.922	118.552	9.988
Std Dev (n-1)	109.120	558.504	10.861	227.666	3.720	0.008	0.657	13.161	0.020
% CV	0.178	0.136	0.266	0.103	0.069	0.002	0.111	0.111	0.002

Figure 116. 150 Gelatin Resilience Test Printout at 72 hrs.

APPENDIX 5. Resilience Test Results (Continued).



Test ID	Firmness	Area	Stifness	Area	Resilience	Distance1	Distance2	Springiness	Time 1
	g	g s	g/s	g s	%	mm	mm	%	s
E02B000	498.941	3548.441	29.527	1633.598	46.037	4.973	4.390	88.277	9.940
E02B001	433.773	3250.970	22.870	1769.024	54.415	5.000	6.005	120.099	10.010
E02B002	653.436	4300.364	44.834	2271.193	52.814	4.997	5.324	106.543	9.985
E02B003	634.910	4257.363	43.152	2326.770	54.653	4.983	5.736	115.110	9.960
E02B004	747.978	4768.974	54.209	2658.516	55.746	4.995	6.677	133.673	9.985
E02B005	583.285	4000.118	37.963	2233.737	55.842	4.992	5.953	119.250	9.980
E02B006	452.868	3313.341	24.984	1870.549	56.455	4.986	5.798	116.285	9.965
E02B007	637.178	4178.264	43.163	2362.480	56.542	5.000	5.654	113.079	9.995
E02B008	697.030	4498.428	49.140	2604.088	57.889	4.987	5.623	112.752	9.970
E02B009	677.644	4351.894	47.404	2571.988	59.100	4.997	7.196	144.006	9.990
Average	601.704	4046.816	39.725	2230.194	54.949	4.991	5.836	116.907	9.978
Std Dev (n-1)	106.739	512.765	10.623	360.023	3.597	0.009	0.748	14.884	0.020
% CV	0.177	0.127	0.267	0.161	0.065	0.002	0.128	0.127	0.002

Figure 117. 150 Gelatin Resilience Test Printout at 88 hrs.

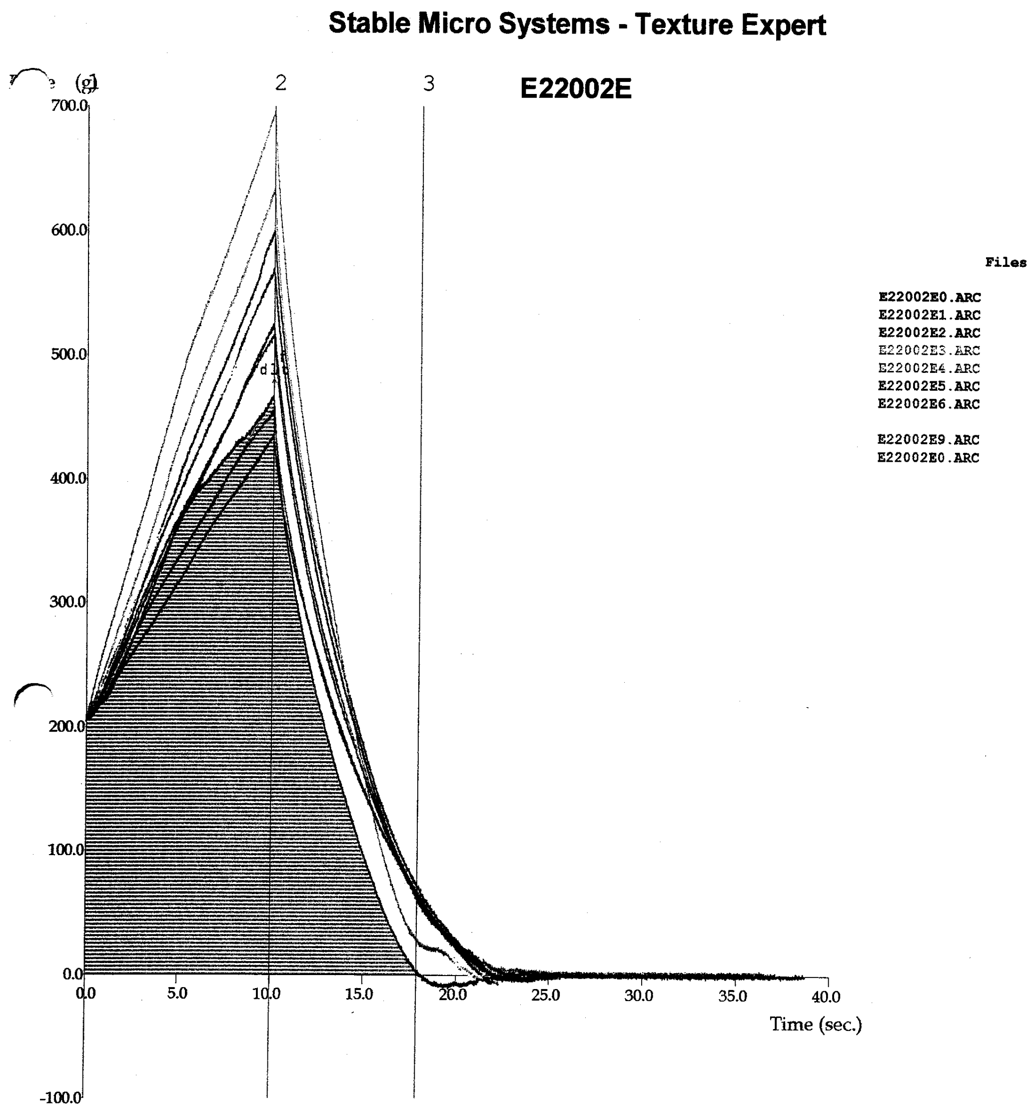
APPENDIX 5. Resilience Test Results (Continued).

E220

	A	K	L	M	N	O	P	Q	R	S
1	Test ID	Firmness	Area	Stifness	Area	Resilience	Distance1	Distance2	Springiness	Time 1
2		g	g s	g/s	g s	%	mm	mm	%	s
3		Force 1	Area-FT 1:2	Grad.-FT 1:2	Area-FT 2:3	N#/L#*100	Distance 1	Travel 2:3	(Q#/P#)*100	Time 1
5	E02M000	418.558	3136.464	21.495	1752.780	55.884	4.993	6.688	133.947	9.980
6	E02M001	244.954	933.410	9.283	1941.352	207.985	2.200	5.890	267.729	4.400
7	E02M002	472.323	3415.836	26.935	1821.091	53.313	4.970	6.075	122.232	9.935
8	E02M003	415.640	3143.759	21.191	1610.649	51.233	4.999	5.445	108.921	9.995
9	E02M004	376.047	2871.222	17.187	1627.881	56.696	4.984	6.330	127.005	9.960
10	E02M005	356.424	2759.953	15.232	1417.941	51.376	4.985	4.899	98.275	9.965
11	E02M006	467.894	3410.285	26.416	1819.538	53.354	5.000	5.789	115.779	9.995
12	E02M007	468.754	3416.342	26.275	1656.825	48.497	5.000	4.720	94.400	9.995
13	E02M008	613.404	4091.805	40.708	2237.760	54.689	4.990	6.453	129.318	9.975
14	E02M009	554.811	3885.596	34.873	1724.930	44.393	4.995	3.676	73.594	9.985
15	End of Test Data									
16	Average	438.881	3106.467	23.959	1761.075	67.742	4.712	5.596	127.120	9.419
17	Std Dev (n-1)	103.311	866.785	9.256	220.379	49.410	0.883	0.930	52.782	1.763
18	% CV	0.235	0.279	0.386	0.125	0.729	0.187	0.166	0.415	0.187

Figure 118. 150 Gelatin Resilience Test Printout at 92 hrs.

APPENDIX 5. Resilience Test Results (Continued).



Test ID	Firmness	Area	Stifness	Area	Resilience	Distance1	Distance2	Springiness	Time 1
	g	g s	g/s	g s	%	mm	mm	%	s
E22002E0	516.025	3623.185	31.237	1939.140	53.520	4.991	5.863	117.470	9.975
E22002E1	524.482	3596.896	31.964	1989.097	55.300	4.987	5.836	117.023	9.965
E22002E2	599.753	3987.792	39.496	2206.222	55.324	5.000	5.910	118.199	10.015
E22002E3	633.176	4217.914	42.946	2223.164	52.708	4.999	6.594	131.906	9.990
E22002E4	695.953	4631.777	48.990	2192.905	47.345	4.995	5.346	107.026	9.985
E22002E5	435.933	3183.196	23.000	1750.786	55.001	5.000	5.920	118.399	10.005
E22002E6	569.742	3838.729	36.516	2120.546	55.241	4.990	5.602	112.264	9.975
E22002E7	561.806	3857.524	35.748	1993.277	51.672	4.986	5.270	105.695	9.965
E22002E9	455.563	3331.873	25.050	1762.422	52.896	5.000	6.217	124.339	9.995
Average	554.715	3807.654	34.994	2019.729	53.223	4.994	5.840	116.925	9.986
Std Dev (n-1)	82.735	444.150	8.279	181.311	2.583	0.006	0.410	8.134	0.017
% CV	0.149	0.117	0.237	0.090	0.049	0.001	0.070	0.070	0.002

Figure 119. 150 Gelatin Resilience Test Printout at 96 hrs.

Appendix 6. Molecular Weight Analysis Results

RPS Gel Mass In Process
SEC EVALUATION
Dec-02

Identification		Mw avg 1	Mw avg 2	Mw avg 3	avg	std	rstd
SF222263	Sample 1	131937	128367	126067	128790	2958	2%
SF222263	Sample 2	115313	111000	113370	113228	2160	2%
SF222263	Sample 3	106020	104882	105197	105366	588	1%
SF222264	Sample 1	128158	124850	121738	124915	3210	3%
SF222264	Sample 2	121215	119429	120225	120290	895	1%
SF222264	Sample 3	110171	109465	110551	110062	551	1%

Gel Study - 195 Lipophilic & Self Emulsifying Fill Material

Identification		Mw avg 1	Mw avg 2	Mw avg 3	avg	std	rstd
Lot E21995	Beginning	101158	101126	99762	100682	797	1%
Lot E21995	Middle	98874	99856	98891	99207	562	1%
Lot E21995	End	95667	96274	95487	95809	412	0%
Lot E21996	Beginning	91878	88886	91283	90682	1584	2%
Lot E21996	Middle	90995	91690	92528	91738	768	1%
Lot E21996	End	90348	91397	89480	90408	960	1%
Lot E21997	Beginning	83849	82660	82388	82966	777	1%
Lot E21997	Middle	81913	81677	81137	81576	398	0%
Lot E21997	End	82089	81667	81030	81595	533	1%
Lot E21998	Beginning	80758	83075	81895	81909	1159	1%
Lot E21998	Middle	80303	79323	81212	80279	945	1%
Lot E21998	End	81724	81280	81790	81598	277	0%

Gel Study - 150 & Oil Fill

Identification		Mw avg 1	Mw avg 2	Mw avg 3	avg	std	rstd
E21999	Beginning	130374	128257	134028	130886	2919	2%
E21999	Middle	130067	128476	127898	128814	1123	1%
E21999	End	127675	126983	123664	126107	2144	2%
E22000	Beginning	118206	123357	124433	121999	3328	3%
E22000	Middle	119517	119119	113825	117487	3178	3%
E22000	End	120975	118979	119987	119980	998	1%
E22001	Beginning	108334	108974	108979	108762	371	0%
E22001	Middle	115163	112029	114227	113806	1609	1%
E22001	End	115850	115593	114208	115217	883	1%
E22002	Beginning	105127	103786	105098	104670	766	1%
E22002	Middle	102131	105916	104826	104291	1948	2%
E22002	End	110479	111525	109930	110645	810	1%

Figure 120. 195 & 150 Gelatin Molecular Weight Analysis Results.