THE VISCOSITY OF PECTIN EXTRACTS
OF LOGANBERRIES AND PEACHES AS
RELATED TO THE QUALITY OF THE FROZEN PRODUCT

by

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CHAPTER I

INTRODUCTION

The freezing preservation of fruits has lately become one of the major food industries in the United States. The Pacific Northwest has always taken a great part in the rise of this industry, and has been producing over 30 per cent of the total United States frozen fruit pack in the last few years.

Although fruit freezing has been thoroughly investigated from different angles, the quality of the finished product as served to the consumer has not undergone the adequate research it deserves. An important principle of the industry is that freezing does not improve the quality and, consequently, the quality of the finished product depends entirely on the quality of the raw material, providing the right preparation and freezing techniques have been followed.

The quality of the raw material has always been determined by standards which depend on individual judgment that varies considerably from person to person.

This work has been undertaken to find out whether a certain constituent of the raw fruit, which is evidently
responsible for some degree of quality, does have any relation with the quality of the finished product. The latter can be determined by some objective method which would make possible the comparison between different samples with different contents of the constituent under question.

Pectin has been chosen as the constituent with the most possibilities for this study for several reasons.

No work has ever been done to find out whether the natural pectin content of any fruit can be related to the quality of the frozen product (1, 43), although addition of pectin and pectinates to some fruits before freezing proved to have some effect on the quality of the frozen product (6, 9).

Pectin has also been related to the quality of dehydrated fruits and vegetables (7), and it was not difficult to postulate that such a relation could be found in the case of frozen fruits where the treatment of the raw material cannot change its natural pectin content as much as the comparatively drastic treatment during dehydration.

The rigidity of the plant cell wall depends to a certain extent upon its content of pectic substances and, thus, pectin is directly responsible for the structural framework of many plant tissues. This framework is largely affected by freezing and it was decided that pectin could have something to do with that effect.
If pectin is present in the cell sap, as indicated by some investigators (37), it could have an effect on the quality of the frozen product as measured by the drip; the drip being the amount of juice that exudes from the frozen fruit upon thawing. Bearing in mind the relation between bound water and biocolloids, and the relation between bound water and drip (28), it was postulated that a certain relation might exist between natural pectin content of fruits and the drip. This drip has been used as an index of quality by some investigators (28, 46) and is used for the same purpose in this study. It must also be mentioned that the bound water content of frozen foods is believed to be related to keeping quality in freezing storage and to changes in texture on defrosting (17).

Loganberries have been chosen for this work to represent the berries which compose a large part of the Pacific Northwest frozen pack. Although they are not the most important berries grown in that area, they have been chosen because of their relatively high pectin content.

Peaches are an important frozen product in the Pacific Northwest; they differ greatly from berries in character and receive a different treatment to prepare them for freezing, and, thus, have been chosen as the second fruit for this work.
CHAPTER II
REVIEW OF LITERATURE

I. Pectin in Loganberries and Peaches

Colby (15), of the California Experiment Station, was probably the first to study the composition of the loganberry in this country, and his work has been followed by several investigators. Tartar, as reported by Lewis and Brown (31), Daughters (16), and others have reported a somewhat complete chemical analysis of loganberries where pectin was either not determined or reported as the alcohol precipitate. The more recent data is summarized in Table I and shows loganberries to contain more pectin than strawberries, blackberries or raspberries.

TABLE I
PECTIN CONTENT OF SOME BERRIES

<table>
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<th>Berry</th>
<th>Per Cent Pectin</th>
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<td>Loganberry</td>
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Peaches contain about 0.777 per cent of pectin (40)
which is considered of poor quality as jellies are not usually made from them without the addition of pectin. Appleman and Conrad (3) reported the pectin content of some peaches as 0.78 per cent and state that calcium pectate was not found at any stage of ripening of the fruit.

II. Pectin in Plant Tissue

Although many studies concerning the pectin substances in plant tissues have been carried out, there is still some disagreement in regard to terminology as over the aggregation of pectic compounds, so that it is difficult to summarize the literature on the pectic substances with some degree of satisfaction.

For the object of this work, however, the presence and nature of the pectic compounds in the fruit were all the information needed. According to Carré (11), Norman (36), Carré and Horne (14), Miller (33), and others, it can be concluded that pectin is definitely present in the cell walls and middle lamella of fruit tissues. Norman (36), considers the pectic substances as the most important of the constituents of the cell wall of fruits, roots, and young cells.

Miller (33), reports that the pectic materials in the middle lamella of the cell wall are for the most part calcium pectate.
The presence and nature of the pectic substances in the cell sap have not undergone such extensive study, and Norman (37) seems to be the only one who considers pectin substances to be present in the cell sap. The nature of these substances have not been reported in the literature and no data is available to support or repudiate Norman's opinion.

III. Pectin and Maturity of Fruit

Although some contradiction may still be found as to the nature, nomenclature or methods of determination of pectic substances in plant tissues, there is a unanimous opinion between plant physiologists as to the relation between pectic substances and maturity of fruits. Carré (12), states that the physical modifications in mature apples are due to pectic changes and that the process of ripening and physiological breakdown are intimately associated with the progressive change of an insoluble pectic substance into a soluble modification. The same investigator (11), found out that throughout ripening the middle lamella pectic substance maintains a constant level, but when the soft over-ripe condition of the fruit becomes apparent, the amounts begin to decrease rapidly. Microscopic observations show that this intimate association of the decomposition of the middle lamella constituents with the over-ripe condition of
the fruit is marked by a gradual loosening of the cells, until ultimately in the most advanced condition of over-ripeness the cells are entirely separated from one another, owing to complete disappearance of the middle lamella pectic substance.

Rosa (39), noticed that, during the ripening of cantaloupes, the total pectic material remained the same, while the protopectin was high in amount in the unripe melons but changed into pectin and pectic acid as the fruit ripened.

Appleman and Conrad (3), found that the change in pectin substances in the cell walls parallels closely the degree of softening of peaches during ripening.

Haller (24), also found a definite relation between softening of apples and the pectin content.

It is of great interest to mention here that Appleman and Conrad (4), while studying the pectic constituents of tomatoes and their relation to the canned product, found a striking parallelism between the pectic changes in the tissues of tomatoes and the degree of disintegration of the canned product.

IV. Methods of Pectin Determination

The earliest methods of analysis are based on the precipitation of pectin by addition of two volumes of
alcohol, drying, and weighing as pectin. The results obtained are always high because of the precipitation of other insoluble material such as proteins, gums, dextrins, and other constituents. These methods have also a limitation, as pointed out by Carré (13), who proved that pectin does not precipitate from alcohol solutions when present in concentrations less than 0.5 grams per liter of original solution.

The method of Wichman and Chernoff (44), includes precipitation of pectin by alcohol, saponification with sodium hydroxide, precipitation with hydrochloric acid, drying, and weighing. The product of this process is considered to be di-galacturonic acid which, besides having an uncertain relationship to the original pectin, may decompose as indicated by the formation of furfural during the treatment (18). Pectic acid is also soluble in pure water and precipitates only in the presence of electrolytes. Thus, low results may be obtained by prolonged washings with distilled water which is necessary in this method.

The calcium pectate method of Carré and Haynes (13), depends on mild alkaline hydrolysis of the pectin and subsequent precipitation as calcium pectate by the addition of calcium chloride. One of the objections to this method is that the precipitate is almost colloidal in particle size and is very difficult to filter and wash.
Other methods for the determination of pectin have been devised by several workers. Von Fellenberg (42), proposed a method of analysis based upon his discovery that methoxyl groups occur in all pectins and on the assumption that the number of methoxyl groups in pectin is constant. It has since been found (18), that the percentage of methoxyl varies with the source of pectin, the treatment it receives and many other conditions, so that this method has little value for analysis. Silin and Silina, as reported by Elwell (18), recommend a method based on the colorimetric estimation of furfural from pectin. This method cannot be used when pentoses are present in the test material and is, evidently, not reliable as far as fruits are concerned.

Wilson (45), mentions the method, developed by him and his co-workers, which depends on the precipitation of pectin, the negatively charged lyophilic colloid, with some positively charged compound, aluminum hydroxide in this case.

It is very plain, from the foregoing discussion, that all the methods developed for pectin determination, apart from not being very reliable, are painstaking and require either technical skill or special training for handling and using chemical reagents and apparatus. They are all time consuming and would be of no value where a quick
index of the pectin content of fruits is needed, and several determinations are required per day.

For the object of this work, a quick reliable method was needed. The possible utilization of viscosity measurements as an index of the quantity and quality of pectin content of the fruit was thought of as the most suitable method.

V. Viscometric Methods

Gortner (22), points out Ostwald's findings of the factors that affect viscosity of lyophilic systems. These factors are: 1. concentration, 2. temperature, 3. degree of dispersion, 4. solvation, 5. electrical charge, 6. previous thermal treatment, 7. previous mechanical treatment, 8. the presence or absence of traces of other lyophilic colloids, 9. the age of the lyophilic sol, and 10. the presence of both electrolytes and non-electrolytes.

In comparing different extracts of the same fruit it is most probable that the only factor that will cause difference in the viscosity of the extracts is pectin concentration, providing the same conditions of extraction are carefully repeated in every case.

The presence or absence of other lyophilic colloids must be taken into consideration in fruits that contain them, when different maturities are used. Loganberries and
peaches do not contain any starch, and there is no indication in the literature that they contain any other lyo-
philic colloid besides pectin. It is thus the author's opinion that the viscosity of the extracts from different
lots of these fruits with different maturities mainly differs due to the concentration of pectin, or in other
words, the pectin content of different lots.

It is of interest to mention that Myers and Baker
(35), while working on fruit jellies, found that jelly
strength is a function of the viscosity of the pectic
solution from which the jelly is made, and that the inor-
ganic impurities and salts of the organic acids will not
upset the relationship between viscosity and jelly strength.

Baker (5), states that sugars, proteins and starches
influence the viscosity to a slight extent, and that the
main influence on the viscosity of fruit juice extractions
is pectin. He found a definite relation between the vis-
cosity of these extracts and the quality and quantity of
the pectin present in the fruit. The main object of
Baker's study was to present the fruit jelly manufacturers
with an accurate means by which they can tell just how much
sugar to add to a fruit extraction, of a known viscosity,
to obtain a jelly with good characteristics. He compiled
tables showing the quantity of sugar to be added for each
viscosity found.
VI. **Extraction of Pectin**

The classical methods for pectin extraction have always been extraction with boiling water, with dilute acids or with dilute alkali. Extraction with alkali or with dilute acids is unsatisfactory in determining the viscosity of pectin extracts because of the accompanying hydrolysis of the pectin already in solution. Alkali hydrolysis decomposes pectic substances to products of indefinite composition and thus makes difficult the comparison of different solutions of pectin on the basis of viscosity.

Baker and Woodmansee (8), have used sodium polyphosphates in pectin extraction and obtained very satisfactory results. They state that the largest portion of the pectic substances in high-quality source materials should be present as protopectin or fully methylated pectic substances which readily reacts with potassium and sodium to form soluble pectinates and with calcium to form insoluble pectinates. The polyphosphates act as a calcium sequestering agent and the soluble sodium pectinates are easily thrown out into solution. Considerably more pectin is made soluble by the addition of polyphosphates at pH values above 3.

The polyphosphates extraction has been established as a very satisfactory method for pectin extraction and is
reported by Mottern and Hills (34), to have the advantages of increased yield and prohibition of degradation. Baker and Woodmansee (6), found sodium hexametaphosphate, \( (\text{Na}_6\text{P}_6\text{O}_{18}) \), to be the most effective among the sodium polyphosphates used. This reagent was used by Baker and Murray (7) in their work on the relation between pectinic acids of dehydrated fruits and the texture and quality. It is commercially marketed under the trade name of "Galgon" and is extremely low in price.

VII. Drip as an Index for Quality of Frozen Fruits

Kaloyereas (28), is probably the first worker to develop a reliable method for determination of drip in frozen fruits. Drip had never been used before in quality determination due to the fact that no adequate measure existed. Very few values for drip appeared in the literature and only two of these were concerned with fruits and vegetables. Joslyn and Marsh (27), reported drip as the loss of weight of the fruit after thawing and their results did not actually correlate between quality and drip. Fieger, Dubois and Koloyereas (21), reported the amount of drip in milliliters but were mostly concerned with the chemical and physical properties rather than the amount. Their data gave great variation between similar samples and they concluded that these variations were due to the
inadequate method used in determining the drip. Deterioration and fermentation were responsible for the unstable results and large changes in pH and refractive index of the drip during the time of thawing indicated the abnormal condition of the product.

To prevent deterioration and fermentation, Kaloyereas (28) thawed the frozen product under petroleum ether or Skelly Solve B previously saturated with water. After 2½ hours the sample was drained over a screen and the aqueous phase read directly in a graduated cylinder. The method always gave comparable results and a state was reached when practically no more drip was obtained from the product. This method has been used in the current work with some modifications.

Woodroof and Shelor (46), also used drip as an index for quality of frozen fruits but their method differs from Kaloyereas' method in that they used low temperature instead of petroleum ether to prevent deterioration and fermentation of the product. Their results did not have the same agreement that Kaloyereas reported and the time of thawing was much longer due to the temperature at which they thawed the frozen samples, 35°F.
VIII. Firmness as an Index for Quality of Frozen Fruits

Firmness is one of the characteristics desirable in the frozen fruit after it has been thawed. The changes that occur in the fruits during freezing and thawing are much reflected on the condition of the fruit texture and are mainly responsible for the objectionable flabbiness of the fruit.

Determination of firmness has not, however, been thoroughly investigated and the few workers who were concerned with firmness used a special method each for their determinations.

Woodroof and Shelor (46), measured firmness of strawberries and peaches with a tenderometer by which the number of grams required to crush especially selected berries or peach slices was determined. These workers did not mention what they meant by crushing or the extent to which the fruit is to be crushed, and the especial selection of the berries or peach slices instead of random selection throws some doubt on the dependability of this method.

Kaloyereas (29) devised an apparatus by which the firmness of strawberries could be determined. The principle of the apparatus is to determine the volume of a suitable size frozen sample, to thaw the sample under petroleum ether for 24 hours and then determine the volume of the
thawed sample after it is compressed with mercury. The quotient of the volume of the compressed sample to the volume of the frozen sample is then taken as an index of the firmness of the berries. In experimenting with Kalyerreas' apparatus, it was found that the amount of mercury used for pressing the sample depends on the amount of drip, and as this latter differs with different samples the compression applied is not the same for every sample. Furthermore, the apparatus, though simple to install, causes some air to be entrapped and interferes with the results.

Esselen, Hart and Fellers (19), in their work on the firmness of canned and frozen apples, used the penetrometer type jelly-strength tester developed by Fellers and Clague (20). The main feature of that tester is a scale which registers the tension of a spring to which a plunger is attached. The scale is stamped on a metal cylinder and the maximum pressure necessary to break the jelly layer may be read directly. In order for this tester to work properly there must be a layer or surface to be broken so that the plunger will spring back to its original position after indicating maximum tension reached. Such a layer was probably present in apple slices in the work of Esselen, Hart and Fellers (19), due to the calcium chloride-dip with which they treated the apples. It is very doubtful whether this tester can be used to test firmness of ordinary frozen
fruits where no layer or surface develops on the outside of the fruit.

Personius and Sharp (38), determined the ease of separation of the potato-tuber cells by measuring the tensile strength which they defined as "the minimum longitudinal stress required to pull a section of potato-tuber asunder." Griffin and Kortesz (23) used the same method to determine firmness of apples. The method requires the use of uniform slices, approximately 8 mm thick, to obtain dumbbell-shaped sections upon which the test could be carried out using a special device. These sections can be easily obtained from the relatively firm potato-tuber or apple tissues, but not from loganberries or peaches where the fruit is much softer and any such cutting will result in mushy and non-uniform masses.
CHAPTER III

PROCEDURES

I. Raw Materials

A. Loganberries: The loganberries were obtained from a private farm where five bushes had been assigned for the whole work. This was mainly done to eliminate as much as possible any variation that is due to differences in soil, growing conditions or other factors that may affect the composition of the fruit.

B. Peaches: The peaches were from the college orchard, and the fruits of only one tree of the Elberta variety were used in this work. It must be mentioned, however, that at the beginning a J. H. Hale tree was chosen for the work, but had to be abandoned after one lot had been frozen, when the rest of the fruits were lost due to weather conditions.

The fruits were picked at several times according to maturity as determined by appearance. Either ripe or overripe fruits were used as they do not differ very much in sugar content or appearance and can be easily mistaken for each other unless the individual fruits are very carefully inspected. Green or under-ripe fruits were not used in this work as they are easily detected and cannot be mistaken for ripe fruits by the processor of frozen food.
The term "ripe" as used in this work means the stage of maturity where the fruit is tree- or vine-ripened, and is known to yield a better frozen product (141).

The term "over-ripe" is used to denote the fruit that has passed the ripe stage and yet has not become obviously mushy as to be easily detected, and rejected by the processor.

II. Preparation for Freezing

A. Loganberries: The berries were usually picked in the morning and transferred to the laboratory as fast as possible, taking every care not to subject them to unnecessary heat or handling. They were immediately put into cellophane bags, without any treatment, the bags heat-sealed and put into one-pound cartons and frozen. Every sample weighed 12 ounces and the maximum time between picking and freezing never exceeded one hour.

B. Peaches: The peaches were always picked at the cooler times of the day and transferred to the laboratory as quickly as possible. They were washed in cold water, cut into halves, and put into boiling water for one minute. After cooling, the peel was removed and each half sliced to two or three slices according to size. The sliced peaches were then drained from any water adhering to them, packed in cellophane bags, the bags heat-sealed and put into one-
pound cartons. Each sample weighed 12 ounces, and not more than two hours were allowed to pass between the time of picking and the time of freezing.

One lot of peaches was prepared as above, then dipped into a 2 per cent low-methoxyl pectin solution for 30 minutes, drained for 3 minutes, and then packed and frozen.

Another lot was prepared as above, then dipped in a 2 per cent low-methoxyl pectin solution for 15 minutes under vacuum, drained for 3 minutes, packed and frozen.

III. Freezing and Storage

All samples were quick-frozen at -10°C in about three hours and then stored at 0°C for between 10 and 11 months. The storage time was decided upon as the reasonable maximum time for storage of frozen foods in commercial practices.

IV. Pectin Extraction and Viscosity Measurement

A representative sample of loganberries or peaches is finely disintegrated in a Waring blender until it becomes a sort of a homogeneous liquid. A 50-gram sample is then weighed into a 250 ml beaker, 20 ml of distilled water added and the optimum amount of Calgon (sodium hexameta-
phosphate) mixed thoroughly into the contents of the beaker. The mixture is then heated on a water bath until it reaches 160°F and is left at a temperature between 160°F-180°F for an optimum time. The mixture is stirred thoroughly during heating and the time is measured on an electric timer.

After heating, 30 ml of distilled water are immediately stirred into the mixture to cool it down. The mixture is then filtered through some filtration medium, that will result in a clear liquid, like cheese cloth or other material. The filtrate is received in a 100-ml volumetric flask which is kept at room temperature by immersing in water. The mass on the filter medium is washed with 20 ml of distilled water, in the case of berries, and 30 ml in the case of peaches, and as soon as the volumetric flask is filled up to the mark, it is transferred to a constant temperature bath, kept at 68°F, where the relative viscosity is determined.

A. Determination of the Optimum Concentration of Calgon for Extraction. The extraction method mentioned is followed with different duplicate samples using different concentrations of Calgon. In this work 0, 1, 2, 3, 4, and 5 per cent, of the weight of the fruit, of Calgon were used and the mixtures heated for 20 minutes. After filtration the relative viscosity was determined and it was
found that the highest viscosity is obtained by using 3 per cent of Calgon in the case of loganberries and 2 per cent in the case of peaches. Two representative results for loganberries are reported in Graph I, page 23, and two representative results for peaches are reported in Graph II, page 24.

B. Determination of Optimum Heating-Time for Extraction. After finding out the optimum concentration of Calgon, several samples were used to determine the optimum heating-time. By using the same concentration of Calgon, identical duplicate samples were heated for 5, 10, 15, 20, 25 and 30 minutes and their viscosities compared. From several results, of which two are represented in Graph III, page 25, for loganberries and Graph IV, page 26, for peaches; it was concluded that the optimum heating-time for extraction is 20 minutes in the case of berries and 10 minutes in the case of peaches.

C. Viscosity Measurements. Viscosity in this work means the relative viscosity which is the ratio between the time of flow of the extract under question and the time of flow of pure distilled water through a special pipette. The Ostwald pipette has been used through all this work and all the determinations were made at 68°F by the use of a constant temperature water bath.
GRAPH I

OPTIMUM CONCENTRATION OF CALGON FOR PECTIN EXTRACTION FROM LOGANBERRIES.

Relative Viscosity

Calgon
GRAPH III.

OPTIMUM HEATING-TIME FOR PECTIN EXTRACTION

FROM LOGANBERRIES.

Relative Viscosity

Time in Minutes
GRAPH IV.

OPTIMUM HEATING-TIME FOR PECTIN EXTRACTION FROM PEACHES.

Relative Viscosity

Time in Minutes
V. **Sugar Extraction and Determination**

A. **Extraction.** A representative sample of the fruit was thoroughly disintegrated in a Waring blender, after removal of all foreign material. Fifty-gram samples were then weighed out, and redistilled, 95 per cent isopropyl alcohol, to which precipitated calcium carbonate had been added to neutralize the acidity, was added. The amount of alcohol was measured as to make its final concentration, allowing for water content of sample, only 80 per cent. The fruit was mixed thoroughly with the alcohol and the mixture heated close to boiling point on a water bath for 30 minutes with frequent stirring.

The alcoholic solution was then cooled, and poured through an extraction thimble and the filtrate received in a 500-ml volumetric flask. The insoluble material was transferred to a beaker, covered with 80 per cent isopropyl alcohol, and warmed on a water bath for one hour. It was then cooled and the alcoholic solution again poured through the same extraction thimble into the same volumetric flask.

The residue was allowed to drain, dried and then ground to very fine particles and extracted 12 hours in a Soxhlet apparatus with 80 per cent isopropyl alcohol.

The alcoholic extracts were then combined and 10 per cent of their total volume hydrochloric acid of specific
gravity 1.125 added, and the extracts heated for 2 hours. The heated solution was then neutralized with sodium hydroxide and made up to volume.

A 50-ml aliquot from the final solution was then put into a beaker and the alcohol driven off on a water bath, adding little amounts of water, to prevent drying, whenever necessary. When the odor of alcohol disappeared from the sample, about 100 ml of distilled water were added and the sample heated to 80°C to soften the gummy precipitates and break up the insoluble masses.

The solution was then cooled to room temperature, transferred to a 250-ml volumetric flask and the beaker rinsed thoroughly with distilled water, adding the rinsings to the contents of the flask.

Saturated, neutral lead acetate was then added until a flocculent precipitate was produced and the mixture shaken thoroughly and left to stand for 15 minutes. The supernatent liquid was tested with a few drops of saturated lead acetate for further precipitation and the solution diluted to the mark with distilled water, mixed thoroughly and filtered through a dry filter paper. Sufficient solid sodium oxalate was then added to the filtrate to precipitate all the lead and the solution refiltered through a dry filter paper. The new filtrate was then tested for the presence of lead with a little solid sodium
oxalate, and 50-ml aliquots taken for the determination of sugars.

B. Determination. The standard Shaffer-Hartman method for sugar determination (25) was used and all determinations run in duplicate. As the extraction itself was run in duplicate, four final determinations were used to obtain each of the total-sugar values reported.

VI. pH Determination

The fruit was finely and homogeneously disintegrated to a thick liquid in a Waring blender and samples taken for direct pH readings on a standard Beckman pH-meter. Minimum time was allowed to elapse between disintegrating the fruit and determining pH.

Baker and Woodmansee (8) reported that they found an optimum pH for the extraction of pectin. This has been tried in the present work and it was found that no appreciable difference existed while adjusting the pH between 1 and 5, using calcium carbonate and diluted hydrochloric acid.

VII. Total Solids Determination

The A.O.A.C. official method for total solids determination in fruits has been used (2), and all
determinations were made in duplicate and the average reported.

VIII. Drip Determination

Kaloyereas (28) suggests the use of drip as a constant for quality control of frozen fruits. He states that the drip practically corresponds to the result of the changes occurring during freezing. Drip, as mentioned previously, denotes the liquid which exudes from the frozen product during thawing.

The method of Kaloyereas depends on using petroleum ether or Skelly Solve B previously saturated with water under which the frozen sample is thawed. After thawing for 24 hours, the sample is drained over a screen and the volume of the aqueous phase is read directly in a graduated cylinder.

In experimenting with this method, it was observed that some of the drip sticks to the walls of the cylinder, thus causing an error that cannot be neglected. Reading of the aqueous phase in a cylinder does not give an accurate measure and transferring the liquid to a burette for accurate readings caused loss of some more of the liquid.

A simple apparatus has been installed for direct readings of the drip without any loss of the exuding juice. In installing this apparatus it has been carefully noticed
not to include any expensive parts or irregular glassware. The apparatus consists of an ordinary 50-ml burette, a regular ⅛-inch funnel and some rubber tubing. The burette is filled to the 50 mark with mercury and mounted on a holder. It is then filled with petroleum ether saturated with water, the funnel is fitted tightly to its top with a piece of rubber tubing around its leg, and a piece of wire screen across its neck. The frozen sample is weighed (about 50 grams), put into the funnel and covered with petroleum ether. The funnel is covered to prevent evaporation, and the sample is left to thaw. See Plate I, page 32.

In due time the drip collects at the bottom of the burette and can be read directly by subtracting the mark at the liquids interface from 50. The use of this apparatus allows accurate drip readings whenever necessary and proved to be of great value in determining the time at which maximum drip is reached.

To determine the time at which maximum drip is reached, readings were taken every two hours for 32 hours. The change at the last stage of thawing was very little and it was decided to set 24 hours as a standard time for drip determinations of both loganberries and peaches. Representative results are shown in Graph V, page 33, where the average of two determinations is reported in every case.

In determining the drip of loganberries, it was
GRAPH V.
TIME FOR MAXIMUM DRIP.

Per cent of drip
50
45
40
35
30
25
20
15
10
5
0

Time in hours

Loganberries

Peaches
found that some juice was frozen at the bottom of the pack. This probably exuded from the fruit before freezing and was thus separated from the berries. In the preliminary determinations this juice was not included in drip determinations and only individual berries were used. There was a great difference in the results for duplicate samples and it was decided to include that juice in drip measurements. The solid pack was cut evenly by a sharp knife from the middle of the shorter side lengthwise and cut twice from the other side. This resulted in six blocks, and the two middle blocks were used for the drip determinations. Representative results are shown in Graph VI, page 35, where duplicate determinations were made on a sample with the juice and another without.

IX. **Firmness Index**

Only very few methods for determining firmness of fruits have been reported in the literature and even those are either undependable due to some apparent discrepancies, or unsuitable for use with loganberries or peaches.

It was necessary, however, to have some numerical values of firmness so that a dependable comparison can be obtained without the doubt that is always connected to results based on the human factor where concepts vary
GRAPH VI.

METHOD OF DRIIP-DETERMINATION FOR LOGARITHMS.

Per cent of drip

Duplicates with juice

Without juice

Time in hours
widely between individuals unless the difference is obviously apparent.

A satisfactory firmness index was obtained by the use of the modified A.S.T.M. (American Society for Testing Materials) type consistency-penetrrometer shown in Plate II, page 37. This consists of a plunger that can be released or held at will by means of a special handle, and a distance-magnifying gauge that reads ten divisions for every millimeter the plunger travels downward.

The methods followed in firmness index measurements differed according to the fruit used and are thus separately discussed below.

A. **Method of Determining Firmness-Index for Loganberries.** In the case of loganberries, the firmness index was measured as the reciprocal of the distance by which the whole individual fruit, laid horizontally, can be pressed down by a known force for a known period of time. The force used was the weight of the plunger plus the weight of a plastic disk, that is much larger than the berry (4 cm in diameter), used to distribute the force, as much as possible, over the surface of the berry. The plunger was also fitted with a small cork stopper, turned upside down, to obtain an even contact between the plastic disk and the plunger's tip; the weight of this cork was also included, the total weight being 203.5 grams.
To determine the firmness index, the plunger is held in a starting position high enough to place the berry without touching. The berry is placed in the middle of the platform of the penetrometer and the plastic disk placed on it. The latter has notches showing its center and thus can easily be placed on the berry and kept in a horizontal position. The plunger is then released and glided easily by hand until it is barely touching the plastic disk. A reading is taken while the plunger is at this position. It is then released to glide freely for exactly five seconds, held at the position it reaches after that period, and a second reading is taken. The difference between the two readings is divided by ten and its reciprocal taken as the firmness index. This is a measure of the compressibility of the drupelets of the fruit.

In determining the firmness index for loganberries, readings were taken on 45 berries from each carton examined. Although there was a great variation in the individual readings for berries of the same carton, the average of the 45 readings differ but very little from carton to carton of the same lot. This is shown in Table II where the readings obtained from two cartons of the same lot are reported.

It was also plainly noticed that this firmness index definitely agrees with the firmness of the berries as determined by organoleptic tests.
TABLE II

REPRESENTATIVE READINGS OF FIRMNESS INDEX
MEASUREMENTS FOR LOGANBERRIES

<table>
<thead>
<tr>
<th>Carton I</th>
<th>Carton II</th>
</tr>
</thead>
<tbody>
<tr>
<td>32.1</td>
<td>32.5</td>
</tr>
<tr>
<td>16</td>
<td>27</td>
</tr>
<tr>
<td>35</td>
<td>35</td>
</tr>
<tr>
<td>51.5</td>
<td>41.5</td>
</tr>
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<td>40</td>
<td>28</td>
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<td>28</td>
<td>39</td>
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<tr>
<td>46</td>
<td>36</td>
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<tr>
<td>41</td>
<td>28</td>
</tr>
<tr>
<td>25</td>
<td>37</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Carton I</th>
<th>Carton II</th>
</tr>
</thead>
<tbody>
<tr>
<td>20.5</td>
<td>36</td>
</tr>
<tr>
<td>32.5</td>
<td>34.5</td>
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<tr>
<td>25</td>
<td>45</td>
</tr>
<tr>
<td>28.5</td>
<td>61</td>
</tr>
<tr>
<td>45.5</td>
<td>48.5</td>
</tr>
<tr>
<td>62</td>
<td>51</td>
</tr>
<tr>
<td>53.5</td>
<td>37</td>
</tr>
<tr>
<td>28.5</td>
<td>35</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Carton I</th>
<th>Carton II</th>
</tr>
</thead>
<tbody>
<tr>
<td>30.5</td>
<td>32.5</td>
</tr>
<tr>
<td>27</td>
<td>29</td>
</tr>
<tr>
<td>47.5</td>
<td>29.5</td>
</tr>
<tr>
<td>28</td>
<td>42</td>
</tr>
<tr>
<td>30.5</td>
<td>46.5</td>
</tr>
</tbody>
</table>

Average......... 37.3  Average......... 37.8
Firmness Index.... 0.27  Firmness Index.... 0.26

B. Method of Determining Firmness Index for Peaches.

In the case of peaches, the firmness index was measured as the reciprocal of the distance which the plunger penetrates into the peach slice for five seconds. The plunger's cone was removed and the rod itself used to directly determine the firmness index. The same method used for running the test on berries is used on peach slices except for the use of the plastic disk. The slice is placed under the plunger, with its center-side up, and the plunger lowered until it
barely touches the surface. The initial reading is taken and the plunger released for exactly five seconds and the final reading taken. The difference between the two readings is divided by ten and its reciprocal used as the firmness index. The plunger weighs 46.5 grams and its diameter is 7 mm.

A whole package of peaches was used to determine the firmness index. Three readings were taken on each slice and the packages contain about 15 to 18 slices each. The averages of the readings on the individual slices were used to get the average whose reciprocal was considered the firmness index for the package contents. There was a great variation between the averages taken from the individual slices, but the reciprocal of the over-all average of the package was constant for different packages of the same lot. Representative readings are presented in Table III, where the readings obtained from two cartons of the same lot are reported.

It was also observed that the firmness index of the peach slices agreed with the firmness as determined by organoleptic tests.
### TABLE III

**REPRESENTATIVE READINGS OF FIRMNESS INDEX MEASUREMENTS OF PEACHES**

<table>
<thead>
<tr>
<th>Carton I</th>
<th>Carton II</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Readings on Slices</strong></td>
<td><strong>Average</strong></td>
</tr>
<tr>
<td>117</td>
<td>122</td>
</tr>
<tr>
<td>62</td>
<td>71</td>
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<tr>
<td>61</td>
<td>68</td>
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<tr>
<td>123</td>
<td>73</td>
</tr>
<tr>
<td>98.5</td>
<td>80</td>
</tr>
<tr>
<td>145</td>
<td>77</td>
</tr>
<tr>
<td>122</td>
<td>99</td>
</tr>
<tr>
<td>127</td>
<td>119</td>
</tr>
<tr>
<td>34</td>
<td>30</td>
</tr>
<tr>
<td>101</td>
<td>92</td>
</tr>
<tr>
<td>133</td>
<td>85</td>
</tr>
<tr>
<td>95</td>
<td>79</td>
</tr>
<tr>
<td>70</td>
<td>78</td>
</tr>
<tr>
<td>93</td>
<td>83</td>
</tr>
<tr>
<td>68.5</td>
<td>108</td>
</tr>
<tr>
<td>137</td>
<td>60</td>
</tr>
<tr>
<td>61</td>
<td>75</td>
</tr>
<tr>
<td>97.5</td>
<td>79</td>
</tr>
</tbody>
</table>

**Over-all average**... 91.5

**Firmness Index**... 0.109

**Over-all average**... 88.2

**Firmness Index**... 0.113
CHAPTER IV
RESULTS

I. Samples

The pectic substances of plant materials still need extensive studies to understand how they behave toward different growing and soil conditions. This was the main reason for using somewhat small size samples. As it was practically impossible to know exactly the different operations or treatments that could have been applied to the whole orchard, and as it is not even definitely known whether those treatments have any effect on the behavior of the pectic substances of the fruit, it was decided that the use of one tree or only a few adjacent bushes would eliminate as much as possible the unknown factors that might interfere with the results especially since a statistically designed set-up was not possible.

II. Results

The results presented in this chapter are all the average of a number of determinations run in duplicate in every case. The number of the determinations is always reported with the tables. Duplicates and averages of duplicates for different determinations on samples of the same lot differed only by what is usually considered
III. Pectin Extracts

The results shown in Tables IV and VII indicate that the viscosity of the extracts is mainly due to the pectin content. Especially in the case of loganberries, Table IV, this fact is strikingly apparent. The sample with the highest viscosity value has the lowest total solids and total sugars values. There is even an inverse relationship between the viscosity of the extracts and the total solids and total sugars content of the corresponding samples; the higher the viscosity, the lower the total solids and sugars. These results agree with what is known about the changes that occur in fruits during ripening. Ripe fruits contain more pectin and less solids and sugars than over-ripe fruits.

Another indication that the viscosity of the extracts is mainly due to the pectin concentration is the effectiveness of Calgon in giving a more viscous extract, which can only be accounted for by the transformation of the insoluble calcium pectate to the soluble sodium pectate.

IV. Loganberries

From Table IV, page 44, it can easily be seen that...
### TABLE IV

**ANALYSIS OF FRESH LOGANBERRIES**

<table>
<thead>
<tr>
<th>Sample No</th>
<th>Viscosity of the Pectin Extract</th>
<th>pH</th>
<th>Total Solids Per Cent</th>
<th>Total Sugars Per Cent</th>
</tr>
</thead>
<tbody>
<tr>
<td>I</td>
<td>3.2</td>
<td>2.5</td>
<td>18.7</td>
<td>7.2</td>
</tr>
<tr>
<td>II</td>
<td>2.9</td>
<td>2.6</td>
<td>19.2</td>
<td>7.4</td>
</tr>
<tr>
<td>III</td>
<td>3.9</td>
<td>2.1</td>
<td>17.3</td>
<td>6.0</td>
</tr>
<tr>
<td>IV</td>
<td>4.1</td>
<td>2.1</td>
<td>16.4</td>
<td>5.8</td>
</tr>
</tbody>
</table>

I and III—Each 5-12 oz cartons

II --- 2-12 oz cartons

IV --- 4-12 oz cartons

The loganberries can be separated into two different lots. Samples I and II are over-ripe fruits, lower in pectin content and higher in total solids and total sugars than Samples III and IV which are ripe fruits.

From Table V, page 45, it can be seen that after freezing and storage for 10 months, Samples I and II are still lower in pectin content than Samples III and IV. The very little increase in the viscosity of the extracts after freezing and storage of the berries might have been due to a slight enzymatic hydrolysis of an insoluble pectin fraction to a soluble one during the storage period, and is very insignificant.

The viscosity of the pectin extracts from the fresh berries is compared to the amount of drip and the firmness
TABLE V
DETERMINATIONS ON FROZEN LOGANBERRIES*

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Viscosity of the Pectin Extract</th>
<th>pH</th>
<th>Amount of Drip in ml/100 g</th>
<th>Firmness Index</th>
</tr>
</thead>
<tbody>
<tr>
<td>I</td>
<td>3.31</td>
<td>3.03</td>
<td>42.71</td>
<td>0.1545</td>
</tr>
<tr>
<td>II</td>
<td>3.11</td>
<td>3.01</td>
<td>42.91</td>
<td>0.1544</td>
</tr>
<tr>
<td>III</td>
<td>4.03</td>
<td>3.03</td>
<td>36.23</td>
<td>0.2276</td>
</tr>
<tr>
<td>IV</td>
<td>4.11</td>
<td>2.92</td>
<td>36.52</td>
<td>0.2635</td>
</tr>
</tbody>
</table>

*--Frozen at -10°F and stored at 0°F for ten months.
1--Average of two duplicate determinations.
2--Average of four duplicate determinations.
3--Average of five duplicate determinations.
4--Average of replicates of two cartons.
5--Average of replicates of four cartons.
6--Average of replicates of five cartons.

It can be seen that the two samples low in pectin content have exuded more juice than the two samples high in pectin content. The decrease of pectin content that caused the decrease in the viscosity of the extracts from an average of 4.0 to an average of 3.05 has caused the drip of the over-ripe fruits to increase more than 17 per cent over the amount of the drip of the ripe fruits.

It is to be noticed that this is not a very great difference, but it must be remembered that the samples were chosen close in maturity and their pectin contents were not really much different as indicated by the
TABLE VI

VISCOSITY OF PECTIN EXTRACTS OF FRESH LOGANBERRIES AS COMPARED TO DRIP AND FIRMNESS OF THE FROZEN-THAWED PRODUCT

<table>
<thead>
<tr>
<th>Sample No</th>
<th>Viscosity of the Pectin Extract</th>
<th>Amount of Drip in ml/100 g</th>
<th>Firmness Index</th>
</tr>
</thead>
<tbody>
<tr>
<td>I</td>
<td>3.2</td>
<td>42.7</td>
<td>0.154</td>
</tr>
<tr>
<td>II</td>
<td>2.9</td>
<td>42.9</td>
<td>0.154</td>
</tr>
<tr>
<td>III</td>
<td>3.9</td>
<td>36.2</td>
<td>0.227</td>
</tr>
<tr>
<td>IV</td>
<td>4.1</td>
<td>36.5</td>
<td>0.263</td>
</tr>
</tbody>
</table>

Viscosity determinations.

The results with the firmness index are more significant, and from Table VI it can be seen that the decrease of pectin content which caused the viscosity of the extracts to decrease from an average of 4.0 to an average of 3.05 has caused a decrease of more than 30 per cent in the firmness index which means an appreciable decrease in the firmness of the fruit.

V. Peaches

The results in the case of peaches are contradictory and no definite conclusion can be reached from them. In Table VII, page 47, the viscosity of the pectin extracts shows that there was not much difference in the pectin content of the samples of the Elberta variety. The J. H. Hale variety was apparently higher in pectin content as
TABLE VII
ANALYSIS OF FRESH PEACHES

<table>
<thead>
<tr>
<th>Sample No</th>
<th>Viscosity of the Pectin Extract</th>
<th>pH</th>
<th>Total Solids Per Cent</th>
<th>Total Sugars Per Cent</th>
</tr>
</thead>
<tbody>
<tr>
<td>I</td>
<td>5.6</td>
<td>3.4</td>
<td>11</td>
<td>7.4</td>
</tr>
<tr>
<td>II</td>
<td>2.0</td>
<td>3.7</td>
<td>11</td>
<td>7.2</td>
</tr>
<tr>
<td>III</td>
<td>2.0&lt;sup&gt;1&lt;/sup&gt;-2.1&lt;sup&gt;1&lt;/sup&gt;</td>
<td>3.9</td>
<td>11</td>
<td>7.2</td>
</tr>
<tr>
<td>IV</td>
<td>2.0&lt;sup&gt;2&lt;/sup&gt;-2.7&lt;sup&gt;2&lt;/sup&gt;</td>
<td>3.9</td>
<td>11.2</td>
<td>7.3</td>
</tr>
<tr>
<td>V</td>
<td>1.7</td>
<td>3.9</td>
<td>11.4</td>
<td>7.2</td>
</tr>
</tbody>
</table>

I—J. H. Hale; all others Elberta.
<sup>1</sup>—Viscosity of the pectin extracts without treatment.
<sup>2</sup>—Viscosity of the pectin extract after dipping fruit in 2 per cent low-methoxyl pectin solution for 30 minutes.
<sup>3</sup>—Viscosity of the pectin extract after dipping fruit in 2 per cent low-methoxyl pectin solution for 15 minutes, under vacuum.

shown by viscosity measurements. Total sugars and total solids and pH of the fresh fruits did not differ much.

From Table VIII, page 48, it can be seen that the pectin content of all the samples decreased on freezing and thawing. This was probably due to the hydrolysis of the pectin fraction present to more simple compounds.

Table IX, page 49, represents the viscosity of the pectin extracts of the fresh peaches compared to the drip and firmness index of the frozen and thawed product.

Except for the great difference in the viscosity of the pectin extract between Sample I and the other samples and
TABLE VIII
DETERMINATIONS ON FROZEN PEACHES*

<table>
<thead>
<tr>
<th>Sample No</th>
<th>Viscosity of the Pectin Extract</th>
<th>pH</th>
<th>Amount of Drip in ml/100 g</th>
<th>Firmness Index</th>
</tr>
</thead>
<tbody>
<tr>
<td>I</td>
<td>3.70&lt;sup&gt;1&lt;/sup&gt;</td>
<td>3.75&lt;sup&gt;1&lt;/sup&gt;</td>
<td>23&lt;sup&gt;1&lt;/sup&gt;</td>
<td>0.313&lt;sup&gt;4&lt;/sup&gt;</td>
</tr>
<tr>
<td>II</td>
<td>1.74&lt;sup&gt;2&lt;/sup&gt;</td>
<td>3.75&lt;sup&gt;2&lt;/sup&gt;</td>
<td>34&lt;sup&gt;2&lt;/sup&gt;</td>
<td>0.111&lt;sup&gt;3&lt;/sup&gt;</td>
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<tr>
<td>III</td>
<td>1.83&lt;sup&gt;2&lt;/sup&gt;</td>
<td>3.80&lt;sup&gt;2&lt;/sup&gt;</td>
<td>32.4&lt;sup&gt;2&lt;/sup&gt;</td>
<td>0.074&lt;sup&gt;3&lt;/sup&gt;</td>
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<td>2.10&lt;sup&gt;1&lt;/sup&gt;</td>
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<td>V</td>
<td>1.58&lt;sup&gt;1&lt;/sup&gt;</td>
<td>3.95&lt;sup&gt;1&lt;/sup&gt;</td>
<td>41&lt;sup&gt;1&lt;/sup&gt;</td>
<td>0.086&lt;sup&gt;3&lt;/sup&gt;</td>
</tr>
</tbody>
</table>

*--Frozen at -10°F and stored at 0°F for eleven months.
I.--J. H. Hale; all others Elberta.
1--Average of two duplicate determinations.
2--Average of three duplicate determinations.
3--Average of replicates of two cartons.
4--Average of replicates of three cartons.

the corresponding difference in the amounts of drip and in the firmness indices, the other results contradict each other to a great extent. Even the difference just mentioned cannot be interpreted properly as Sample I is not the same variety as the other samples.

Comparing Samples II and V, it can be seen that the higher the viscosity of the pectin extract, the less is the drip and the higher the firmness index. Sample III, however, has the same pectin content as Sample II and gave almost the same drip, yet there is a very great difference in their firmness indices.

It can be said that addition of pectin reduced a
### TABLE IX

**VISCOSITY OF THE PECTIN EXTRACTS OF FRESH PEACHES AS COMPARED TO DRIP AND FIRMNESS OF THE FROZEN-THAWED PRODUCT**

<table>
<thead>
<tr>
<th>Sample No</th>
<th>Viscosity of the Pectin Extract</th>
<th>Amount of Drip in ml/100 g</th>
<th>Firmness Index</th>
</tr>
</thead>
<tbody>
<tr>
<td>I</td>
<td>5.6</td>
<td>23</td>
<td>0.313</td>
</tr>
<tr>
<td>II</td>
<td>2.0</td>
<td>34</td>
<td>0.111</td>
</tr>
<tr>
<td>III</td>
<td>2.0*–2.1</td>
<td>32.4</td>
<td>0.874</td>
</tr>
<tr>
<td>IV</td>
<td>2.0*–2.7</td>
<td>39.4</td>
<td>0.102</td>
</tr>
<tr>
<td>V</td>
<td>1.7</td>
<td>41</td>
<td>0.086</td>
</tr>
</tbody>
</table>

I—J. H. Hale; all others Elberta.

*—Viscosity of the natural pectin extract.

1—Viscosity of the pectin extract after dipping fruit in 2 per cent low-methoxyl pectin solution for 30 minutes.

2—Viscosity of the pectin extract after dipping fruit in 2 per cent low-methoxyl pectin solution for 15 minutes, under vacuum.

little the drip of Sample III, but this is certainly repudiated by the results obtained with Sample IV where more pectin has been added and its natural pectin content was the same as III, as indicated by the initial viscosities; yet there is an appreciable increase in its drip over that of Sample III. This could have been attributed to the effect of the vacuum treatment on the cell walls, but the firmness index of that sample does not show that such effect could have taken place.
CHAPTER V
SUMMARY AND CONCLUSIONS

There are several indications that the natural pectin content of the fruit may have an important role in maintaining the quality of the frozen fruit during freezing and storage. Loganberries and peaches are used to find out if there is a definite relation between the pectin content of the raw fruit and the quality of the frozen product.

Pectin is not actually determined as there is no accurate method available for a quick determination. The viscosity of Calgon (sodium hexametaphosphate) extracts is used as an adequate index of the pectin content of the fruit. Method of extraction and optimum conditions are reported in detail.

The quality of the frozen product is determined by measuring the drip of the fruit upon thawing, and the firmness of the thawed fruit. A new and simple apparatus for accurate measurements of the drip has been developed and proved to be of great value in drip determinations. The firmness is measured by the use of a penetrometer in the case of peaches and compression-meter in the case of loganberries, and a numerical firmness index obtained to give comparable and dependable results.

From the results obtained, the following can be concluded:
1. Ripe loganberries contain more pectin than over-ripe loganberries and the viscosity of the polyphosphate extracts corresponds to the pectin content of the fruit.

2. The textural quality of the frozen loganberries depends to a great extent upon their pectin contents as indicated by the relation between the viscosity of the extracts and the amount of drip and the firmness indices. The higher pectin content results in a better quality product, i.e., less drip and more firmness.

This evidence is restricted to the case when the difference in maturity does not affect the sugar content of the fruit very much.

3. Peaches gave contradictory results and no dependable conclusion can be given in this work.

4. Much work is still needed to establish enough data so that a frozen food processor might be able to judge the quality of the fresh fruit by running some viscosity tests that do not take more than 30 minutes, and thus be able to judge the different lots of the same fruit as to their suitability for freezing.

5. The results of drip measurements show plainly the dependability of the method and apparatus used and that the method can be adopted for use as a standard method.

6. The firmness index determinations show great variations between individual berries or peach slices, but the
averages of package replicates in both cases agree closely as far as different samples of the same lot are concerned.


13. and Haynes, D. The estimation of pectin as calcium pectate and the application of this method to the determination of soluble pectin in apples. Ibid., 16, (1), p. 60, 1922.


42. von Fellenberg, Th. Uber den nachweis und die bestimmmung des methylalkohols sein vokommen in den verschiedenen nahrungsmitteln und das verhalten der methylalkoholhaltigen nahrungsmitel im organismus. Biochem. Z., 85, p. 54, 1918.


