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CONTENTS

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J. H. IRISH, M. A. JOSLYN, AND J. W. PARCELL

HEAT PENETRATION IN THE PASTEURIZING OF SYRUPS AND CONCENTRATES IN GLASS CONTAINERS

J. H. IRISH,[1] M. A. JOSLYN,[2] AND J. W. PARCELL[3]

INTRODUCTION

The enforcement of the food and drug laws during the past decade has had a marked influence on the methods used for the preservation of fruit juices and syrups. Chemical preservatives, at one time very generally used, have been to a large extent replaced by pasteurization. The only chemical preservatives permitted by law are sodium-benzoate and sulfur-dioxide. Used in moderate concentrations these preservatives are considered not injurious to health; but the consuming public has reacted unfavorably toward them in recent years, largely because they affect the flavor of the product adversely.

Since pasteurization is the method best suited to the preservation of these beverages, it is very desirable that accurate information regarding the factors affecting this operation should be available.

Pasteurization is that process of food preservation in which the food is heated to a temperature sufficient to destroy or inhibit the growth of any microorganisms that would affect the food injuriously, but not necessarily to destroy all the living microorganisms with which the food may be infected.

In the pasteurization of fruit juices, beverages, and syrups, the time and temperature of heating must be such as to destroy the yeasts and molds the activity of which would cause deterioration of the product. Although pasteurized fruit-juice products are not

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[1] Junior Chemist in the Experiment Station.
[2] Research Assistant in Fruit Products.
[3] Research Assistant in Fruit Products.

sterile, their acidity and sugar content act as mild preservatives and prevent the activity and multiplication of the bacteria present. They have for bacteria an antiseptic rather than a germicidal action.

No uniform system of pasteurizing fruit juices and syrups is at present in use. Various temperatures and various periods of time are used by different manufacturers. In some cases these are excessive and injure the products by overheating, while in others they are not sufficient and permit considerable spoiling. Standardization of the methods of pasteurization is desirable. The time of heating should be as short and the temperature as low as is compatible with the destruction of all microorganisms that might spoil the product. The time necessary for pasteurization depends on certain factors, the most important of which are:

1. Lethal rate[4] of the microorganisms which initially infect the product: This depends upon the nature of the product, especially its pH value, and on the extent and kind of infection.

2. The rate of heat penetration into the product, that is, the rate of rise of temperature at the center of the product when heated in a container.

Extensive experiments have been conducted by the United States Department of Agriculture, various can companies, and by the National Canners' Association, on heat penetration in canned foods, but little has been done on the rate of heat penetration in fruit juices and syrups in glass containers.

Purpose of the Investigation.—The purpose of these experiments was to determine the rate of heat penetration into various fruit juices, syrups, and concentrates under the conditions of pasteurization to be described, and to correlate this with the time required for the temperature in the center of different-sized bottles containing various liquids to reach the pasteurizing point. These investigations were undertaken in 1924 and were completed in May, 1927.
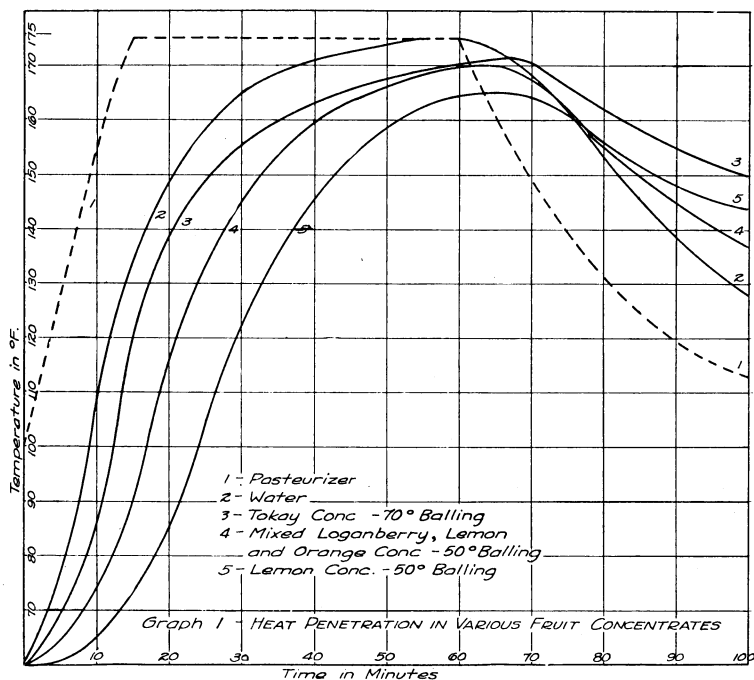
Factors Affecting Heat Penetration.—Magoon and Culpepper[5] conclude from their preliminary experiments "that the factors affecting the rate of change of temperature at the center of the can are the diameter of the container, the conductivity, thickness, and radia-

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[4] The term lethal rate was introduced by Bigelow, who defined it as the fraction of the microorganisms killed per minute, or the reciprocal of the number of minutes required to destroy all spores of the organism at a given temperature and under the conditions obtaining within the food. (See: Bigelow, W. D., *et al.* Heat penetration in processing canned foods. National Canners' Assoc. Research Bul. **16 L:** 3–128, 89 figs. 1920.

[5] Magoon, C. A., and C. W. Culpepper. A study of the factors affecting temperature changes in the container during the canning of fruits and vegetables. U. S. Dept. Agr. Dept. Bul. **956:** 1–54. 57 figs. 1921.

tive power of its walls, the temperature, conductivity, and mobility of its contents, and the temperature, conductivity, and movement of the medium surrounding it.''

Bigelow[6] points out that heat penetration is influenced by the size of the container, the initial temperature of the food, the retort temperature, the nature and consistentcy of the food, and by the presence of forced convection currents due to the rotation of the container during processing.

1 – Pasteurizer
2 – Water
3 – Tokay Conc – 70° Balling
4 – Mixed Loganberry, Lemon and Orange Conc – 50° Balling
5 – Lemon Conc. – 50° Balling

Graph 1 – HEAT PENETRATION IN VARIOUS FRUIT CONCENTRATES

That the nature of the product is an important factor affecting heat penetration is evident from a consideration of graph 1 showing heat penetration in various fruit concentrates in gallon glass bottles.

An examination of graph 1 will show that heat penetration is most rapid in 70°-Balling Tokay-grape concentrate and slowest in the 50°-Balling lemon concentrate. A 72°-Balling orange concentrate, not shown in the graph, approached the lemon closely.

Evidently the rate of heating is not in the order of decreasing concentration but depends on other factors. Although the concentration of the product is not the only factor which determines the rate

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6 Bigelow, W. D., *et al.* Heat penetration in processing canned foods. National Canners' Assoc. Research Bul. **16 L:** 3–128. figs. 1–89. 1920.

of heat penetration into the product, it has a considerable effect, as may be seen by comparing graphs 1 and 2. A comparison of the two will show that heat penetration in general decreases with increasing concentration.

Graph 2 - HEAT PENETRATION IN FRUIT JUICE

1 - Pasteurizer
2 - Water
3 - Apple Juice - 15° Balling
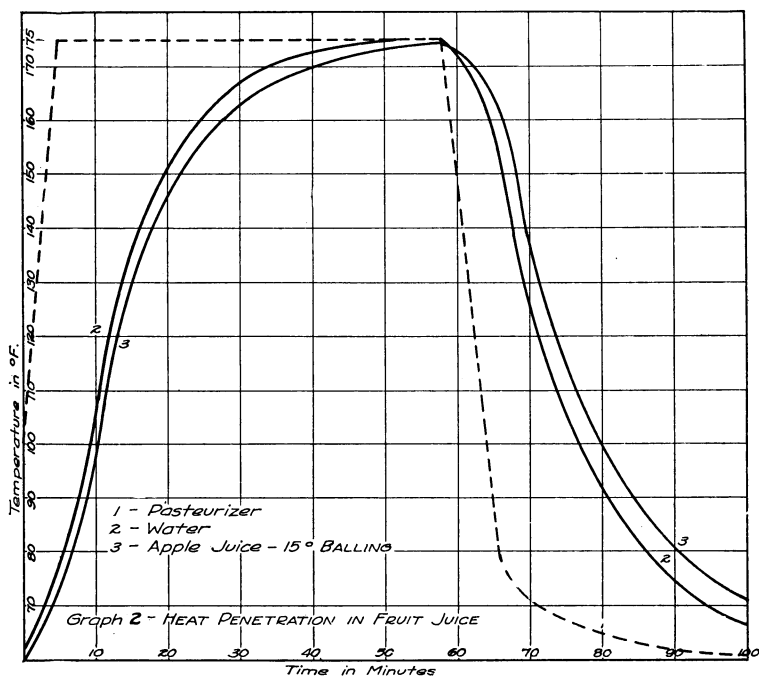
Graph 2 shows the typical heat penetration in fruit juices in gallon glass bottles. In this test heat penetration was determined in the following fruit juices:

20° Balling-muscat grape juice	15° Balling-apple juice
20° Balling-strawberry juice	11° Balling-lemon juice
18° Balling-pomegranate juice	

It was found that although the heat penetration in these juices did not differ very greatly from that in water, it was appreciably slower. Heat penetration was least rapid in the apple juice and most rapid in the grape juice, but the differences in rates in the various juices were so slight that they could not be shown in the graph. Apple juice was therefore taken for the purpose of illustration.

It is known that oranges and lemons are rich in pectin. Furthermore the concentrates of these fruits, at the time these experiments were made, appeared somewhat gelatinous. Concentrated Tokay-grape juice, on the other hand, is low in pectin and gum content. It

showed the most rapid heating, although its concentration was 20° Balling greater than that of the lemon concentrate.

This difference in the rate of heat penetration in the fruit juice concentrates is probably due to difference in viscosity occasioned by differences in the content of pectin and gums.

In the case of the juices it is known that apple juice is richer in pectin content than grape juice. This difference probably caused the difference observed in the rate of heat penetration.

Apparently, then, the rate of heat penetration will depend greatly on the viscosity of the product. This viscosity will be affected by the presence of gums and pectins and the concentration and acidity of the product.

In general then the rate of heat penetration depends upon:

1. The size and shape of the container and the thickness and conductivity of its walls.

2. The viscosity and the rate of change of viscosity with change in temperature of the product.

3. The method of heating the product.

In these investigations we confined ourselves to the study of factors 1 and 2.

EXPERIMENTAL PROCEDURE

Preparation of Materials.—

1. Simple Syrups.—Simple syrups of distilled water and cane sugar were made with Balling degrees (percentages of sugar by weight) of 10, 20, 30, 40, 50, 60 and 70.

2. Fruit Juice Concentrates and Syrups.—The following concentrates were made by concentrating the respective juices in vacuo:

> Orange concentrate—72° Balling
> Tokay-grape concentrate—70° Balling
> Mixed loganberry, lemon, and orange concentrate—50° Balling
> Lemon concentrate—50° Balling.

3. Fruit Juices.—Lemon juice of 11° Balling, strawberry juice of 20° Balling, apple cider of 15° Balling, Muscat grape juice of 20° Ball, and pomegranate juice of 18° Balling were prepared as described in Bulletin 359.[7] These were not filtered clear, although they were free from sediment.

4. Pectin Syrups.—Powdered citric acid, of a fair degree of purity, and pectin[8] were added to simple syrups of 40° and 70°

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[7] Cruess, W. V., and J. H. Irish. Fruit juice investigations. California Agr. Exp. Sta. Bul. 359: 525–568. 1923.

Balling. The citric acid dissolved readily but the pectin with diffi-
culty. The pectin was incorporated with the sugar and acid by
rapid, mechanical stirring and heat. This series included the following
solutions:

> 40° Balling simple syrup and 0.5 per cent citric acid.
> 40° Balling simple syrup and 1.5 per cent citric acid.
> 40° Balling simple syrup, 0.5 per cent citric acid and
>   1 per cent pectin.
> 40° Balling simple syrup, 1.5 per cent citric acid and
>   1 per cent pectin.
> 70° Balling simple syrup and 0.5 per cent citric acid.
> 70° Balling simple syrup and 1.5 per cent citric acid.
> 70° Balling simple syrup, 0.5 per cent citric acid and
>   1 per cent pectin.
> 70° Balling simple syrup, 1.5 per cent citric acid and
>   1 per cent pectin.

5. Simple Pectin Sols.—Solutions of 0.5, 1.0 and 1.5 per cent
pectin were prepared by allowing the required amount of pectin to
soak in a limited amount of water until dissolved and then diluting
to the proper strength. Any lumps of pectin which formed were
broken up with a spoon occasionally during soaking. No heat or
mechanical stirring was used to aid solution. In this manner sols of
the maximum viscosity resulted.

6. Acidified Pectin Sols.—To pectin sols made as in (5), enough
powdered citric acid was added to bring the acidity to 1 per cent as
citric.

7. Pectin Sugar Syrups.—To pectin sols made as in (5), enough
sugar was added to give solutions of 40 per cent and 55 per cent,
sugar respectively.

8. Acidified Pectin-Sugar Syrups.—To syrups prepared as in (7),
1 per cent of citric acid was added.

Containers.—Gallon, half-gallon, quart, pint, eight-ounce, and
four-ounce glass bottles were used (fig 1) to determine the effect of
size. These are the bottles commonly used in commercial practice.
Care was taken that bottles of the same size had the same distribution
of glass, i.e., bottles of the same height and weight.

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[8] In these experiments powdered citrus pectin was used. It was free from
sodium acetate and sugar but was slightly acid in reaction. Its analysis, fur-
nished by the Exchange Lemon Products Company, was as follows:

Grade—165 jelly units (forms jelly with 165 parts of sugar).
Moisture—10 per cent.
Ash—3.5 per cent.
Acidity towards methyl red—0.9 per cent as hydrochloric.
Acidity towards phenolphthalein—1.7 per cent as hydrochloric.

To determine the effect of form, various types of pint glass containers were used (fig. 2): slender-necked and short-necked grape-juice bottles, milk bottle, Mason fruit jar, and tall and flat syrup jugs.

Gallon glass bottles were used in all other tests because their greater volume made more accurate measurements possible.
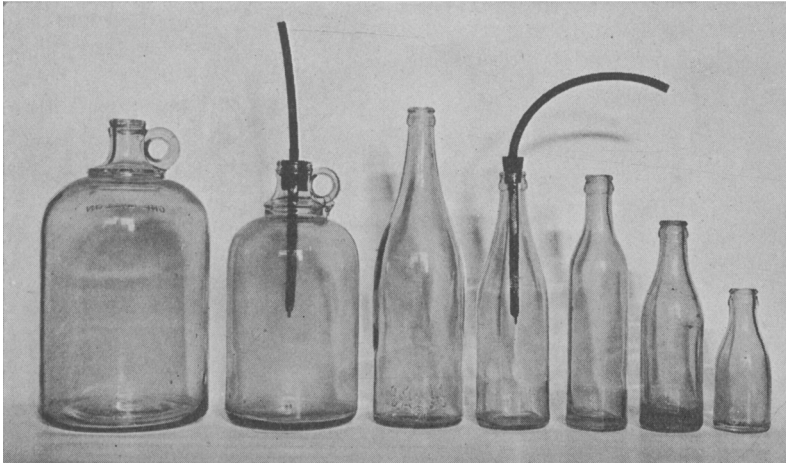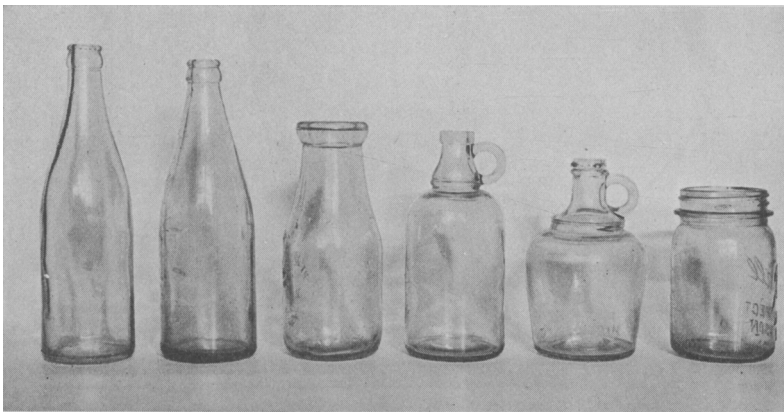

Fig. 1. Bottles used for simple syrup.

Fig. 2. Pint glass containers of various shapes.

Methods of Measuring Temperature.—In the preliminary tests in the Fruit Products Laboratory at Berkeley, glass thermometers were used to indicate the temperature at the center of the bottles. These were soon abandoned for thermocouples constructed of copper and constantan. The first hot junctions used were incased in metal sheaths,

but Bakelite sheaths were used later.[9]   The hot junction was soldered in a copper tip which screwed into a piece of Bakelite tubing.   By means of a one-hole rubber stopper, this was so placed in the bottle that the tip was at approximately the center of the contents (fig. 3).
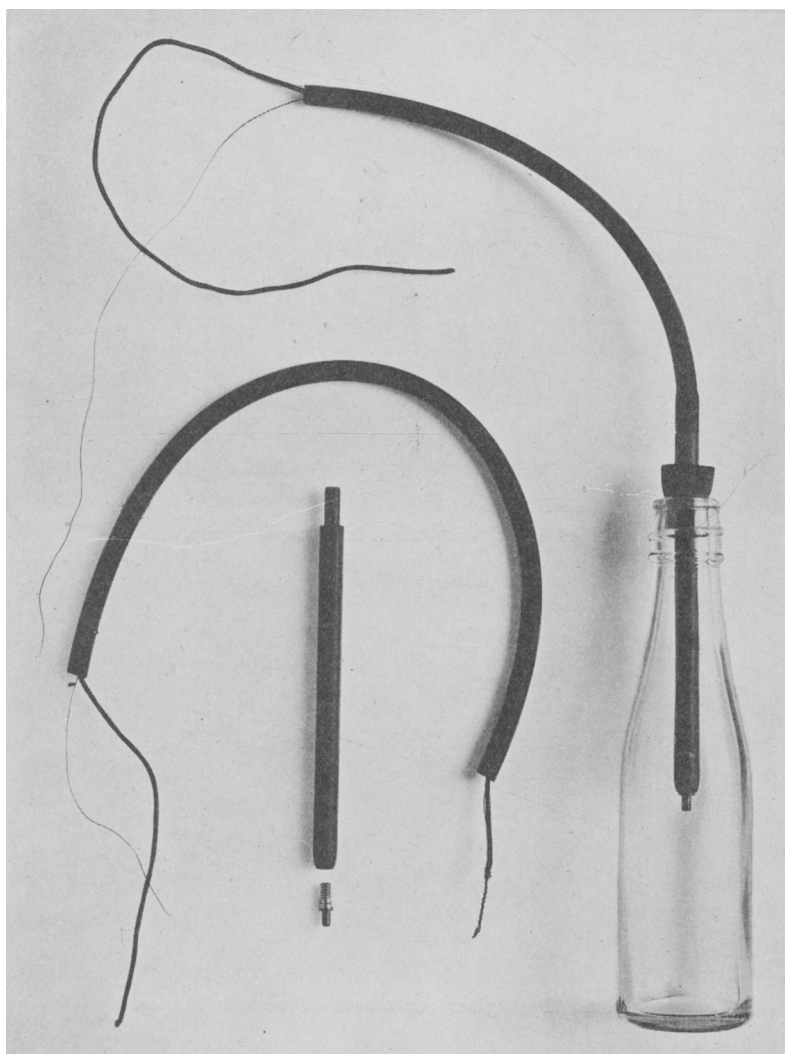

Fig. 3.   Showing position of hot junction in the bottle and the parts of the thermocouple sheathing.

The rubber stopper allowed no leaks and was wired to the neck of the bottle.

The leads from the hot junction were protected by being incased in rubber tubing and the cold junction was placed in the potentiometer box (fig 4). A Leeds and Northrup potentiometer indicator reading directly in degrees Fahrenheit and equipped with a cold-junction compensator was used. The potentiometer was in all cases calibrated for the conditions under which measurements were made, and the thermocouples were frequently checked against standard mercury thermometers.
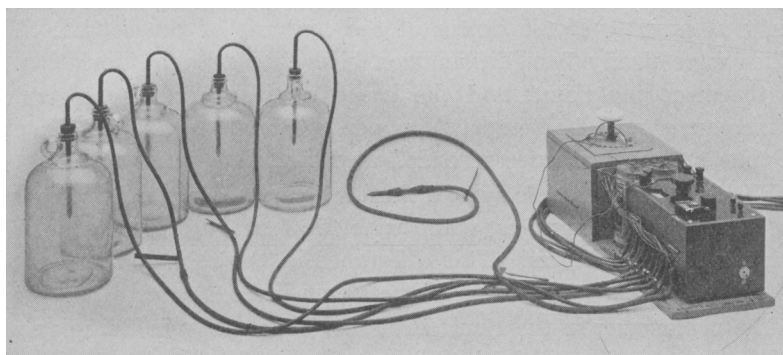

Fig. 4.   Showing method of arrangement of potentiometer, junction box and thermocouples.

In order that temperature measurements could be made with the one potentiometer indicator and with more than one hot junction, a junction box[10] was used. In the final arrangement in the Fruit Products Laboratory ten thermocouples were used. This arrangement was essentially that used in the Fruit Products retort laboratory in San Francisco, where temperature measurements were made with twenty thermocouples in some of our experiments.

Method of Pasteurizing.—In preliminary tests a small pasteurizer 20 by 20 by 30 inches heated by steam jets was used for heating the bottles. It was difficult to maintain a uniform temperature because the pasteurizer was not equipped with an automatic temperature control. The water in the pasteurizer was agitated by intermittent stirring with a paddle. However, in spite of lack of automatic control, fairly consistent results were obtained. These data have not been used in this paper.

[10] For a detailed description see: Foote, P. D., C. O. Fairchild, and T. R. Harrison. Pyrometric practise. U.S. Bur. of Standards Technologic Paper **170**: 83. 1921.

The equipment used in the first set of experiments, the data from which were used in this paper, consisted of a vertical canning retort arranged for use as a discontinuous pasteurizer with automatic temperature control and an air agitator to insure uniform heating of the water bath.[11]

It has been established that ordinary yeasts are destroyed at or below 150° F, and most molds at a temperature of 175° F. In these investigations, 175° F was taken as the pasteurizing temperature. In all cases the temperature of the water was raised to 100° F, before submerging the bottles, which were at room temperature.

The thermocouples were placed in the bottles with the points as nearly as possible at the center of the contents of the bottles. The bottles were firmly fixed in an iron basket to prevent deviation of the thermocouple point, and the basket was suspended midway in the pasteurizer. The temperature control was set at 175° F, and the time and temperature recorded at five-minute intervals.

With all of the simple syrups the water in the pasteurizer reached 175° F within ten minutes, while with the fruit-juice concentrates it required fifteen minutes. This difference in the rate of heating was due to fluctuation of pressure in the steam line.

The pasteurizer in the Fruit Products Laboratory in Berkeley was used in the second set of final experiments. During these experiments, the water in the bath was continuously agitated by compressed air. The heating was controlled very carefully by hand. The maximum variation in the temperature of the bath was 2° F.

As in experiments carried out in the retort laboratory in San Francisco, the bath was raised to 100° F before submerging the bottles, which were at room temperature when placed in the bath. Then the temperature was rapidly raised to 175° F. In all experiments this temperature was attained within five minutes, as high-pressure steam was used. By means of hand control, the bath temperature in the first few experiments was maintained to within 2° F of 175° F. In later runs the range was within 1° F.

In filling the bottles, care was taken to allow for expansion, as otherwise the bottles would burst during heating. The bottles were placed in a specially devised wire rack which prevented motion of the bottles during the heating and subsequent cooling and helped to keep the hot junction at the exact center of the bottle.

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[11] A description of the retort will be found in the catalog of the Sprague Sells Corp., of Chicago; of the potentiometer temperature-measuring equipment in the catalog of the Leeds & Northrup Company; and of the temperature controller in the catalog of the Foxboro Instrument Company.

Methods of Cooling.—For the cooling period, the steam was turned off, and cold tap water was allowed to enter at the bottom of the retort and displace the hot water, which escaped through an overflow valve at the top.   The water in the bath was agitated by means of compressed air during this cooling as well as during heating.   The cooling was fairly uniform in all experiments.

During the heating and cooling periods, temperature readings were taken frequently, usually at intervals of 2 minutes.

Accuracy of the Experimental Work.—The data obtained in these investigations were determined with a degree of accuracy sufficient for the purpose in hand, which was to introduce certain improvements into the practice of pasteurization.   From the graphs, the time for pasteurization may be determined to within five minutes.   The potentiometer was accurate to within 1° F.

THE THEORY OF HEAT PENETRATION

Before considering the results of the experiments, it may be well to consider briefly the more important aspects of the theory of heat penetration.

Heat may flow from a warmer body to a colder body or from one portion to another of the same body by any one or all of three ways, conduction, convection, and radiation.   As the conductivity of most liquids is low, the rate at which heat is transferred in such media by simple conduction is slow.   The flow of heat through any medium or through vacuum by radiation is proportional to the fourth power of the temperature drop.   In the process of pasteurization, the heat transferred by radiation is usually negligible.   The rate at which heat is transferred by convection is proportional to the temperature difference, in accordance with Newton's law.   Where the viscosity of the medium is such as not to interfere with or impede convection currents, the rate at which heat is transferred by this process in liquids is rapid.

When the bottle is placed in the pasteurizer, heat enters from all sides and the coldest portion is, theoretically at least, always at the center.   The surface of the contents will rapidly reach the pasteurizing temperature, but the time required will vary according to the conductivity and the thickness of the glass walls of the container.   If the viscosity of the fluid is such as to impede convection currents, the rate of heat penetration into the interior will be slow.   Where the viscosity is low enough to freely permit convection currents, the

temperature in the center will rapidly reach the pasteurizing temperature, although the time required is also dependent upon the size and shape of the containers.

The influence of viscosity will be further discussed in considering the results of the experiments.

If all other conditions are kept constant, the maximum rate of heat penetration will occur as pointed out by Bigelow[12] in those products which contain a minimum amount of substances in solution and in which the liquid is of such a nature that it affords the maximum freedom for movement of convection currents. In such a product heat penetration is due chiefly to convection, but conduction also exerts its full force. In a product where the viscosity or consistency is such as to inhibit or eliminate all convection currents, we get the minimum rate of heat penetration.

The rate of heat penetration in fruit juices which do not contain gums and pectins in solution will approach the maximum. Heat penetration in jellies and in concentrated syrups rich in gums and pectins will approach the minimum.

RESULTS OF EXPERIMENTS

The temperature readings taken during heating and cooling of the containers in the various experiments have been plotted against time in graphs 1 to 10. The data tables have been omitted in order to conserve space; the graphs illustrate the results much better than do tables.
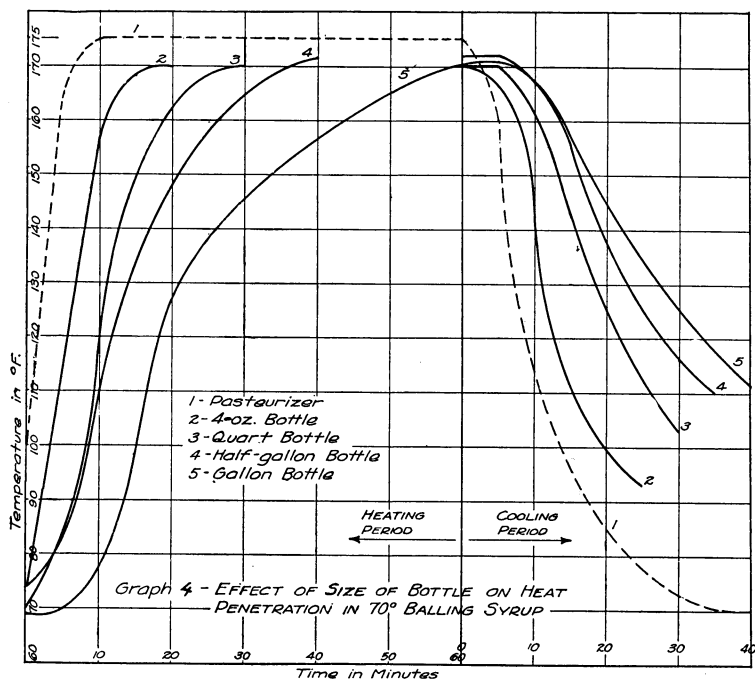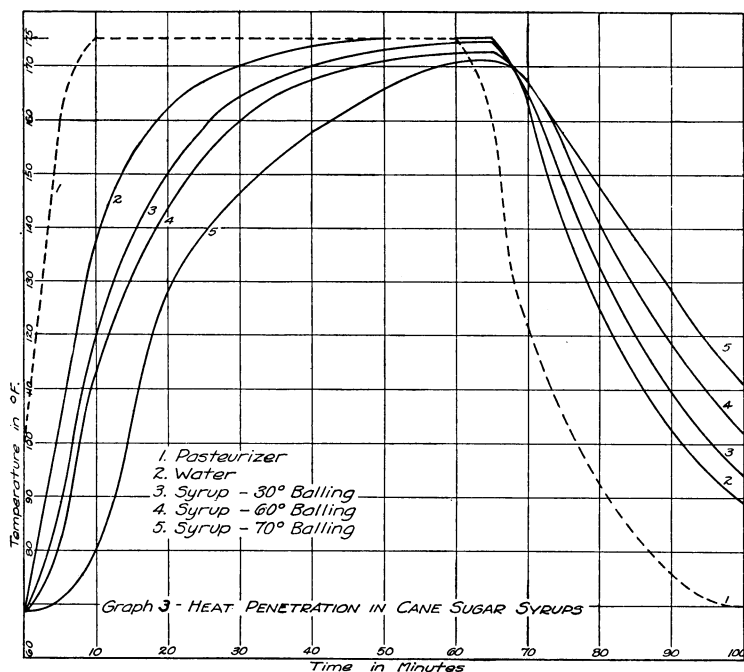
The graphs show only the general form of the heat-penetration curves under certain typical conditions. They do not represent all the data taken. Deviations from these graphs are discussed in the text where the specific experiments are considered.

INFLUENCE OF SUGAR AND OF SIZE AND SHAPE OF CONTAINER

In view of the fact that syrups are being marketed to a very large extent in glass bottles and the production of fruit syrups for beverage purposes is increasing, it was deemed advisable to obtain data on the rate of heat penetration in simple cane-sugar syrups of various densities. Information of this sort for glass containers has been lacking, and sterilizing practices among bottlers vary widely.

Cane-sugar syrups of 10, 20, 30, 40, 50, 60, and 70 per cent were placed in gallon, half-gallon, quart, pint, eight-ounce (soda pop) and

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[12] Bigelow, W. D., *et al. Loc. cit.*

Graph 3 - Heat Penetration in Cane Sugar Syrups

1. Pasteurizer
2. Water
3. Syrup – 30° Balling
4. Syrup – 60° Balling
5. Syrup – 70° Balling

Time in Minutes

Graph 4 – Effect of Size of Bottle on Heat Penetration in 70° Balling Syrup

1 - Pasteurizer
2 - 4-oz. Bottle
3 - Quart Bottle
4 - Half-gallon Bottle
5 - Gallon Bottle

Heating Period   Cooling Period

Time in Minutes

four-ounce bottles, fitted with thermocouples and heated in the pasteurizer previously described.

It was found that in the containers of each size used the rate of heat penetration decreased as the concentration of syrup increased. Graph 3, showing the heat penetration in cane-sugar syrups in gallon bottles, is typical of the results obtained, with the exception to be noted later.

It is also obvious from graph 4 that the rate of heat penetration increased with decrease in size of container.

The rates of heat penetration closely approach one another in syrups from 10° Balling to 60° Balling, but were nevertheless in the inverse order of the concentrations of the syrups. Between 60° Balling and 70° Balling, however, there was a very pronounced increase in the lag and this lag was more marked during cooling. In fact in all cases during cooling, there is more separation of the various curves than during heating. This lag between the 70° Balling and the other syrups was not so great in the eight-ounce and four-ounce bottles as in the larger containers (see graph 5), owing to the fact that heat transfer is much more rapid in the smaller containers than in the larger. The heat transfer by conduction is more rapid, because in the smaller containers there is a greater heating surface per unit volume, and the heat wave has a shorter distance to travel to reach the center. Thus the temperature of the contents increases, and hence the viscosity decreases, thus allowing rapid convection even in the case of the more concentrated syrups.
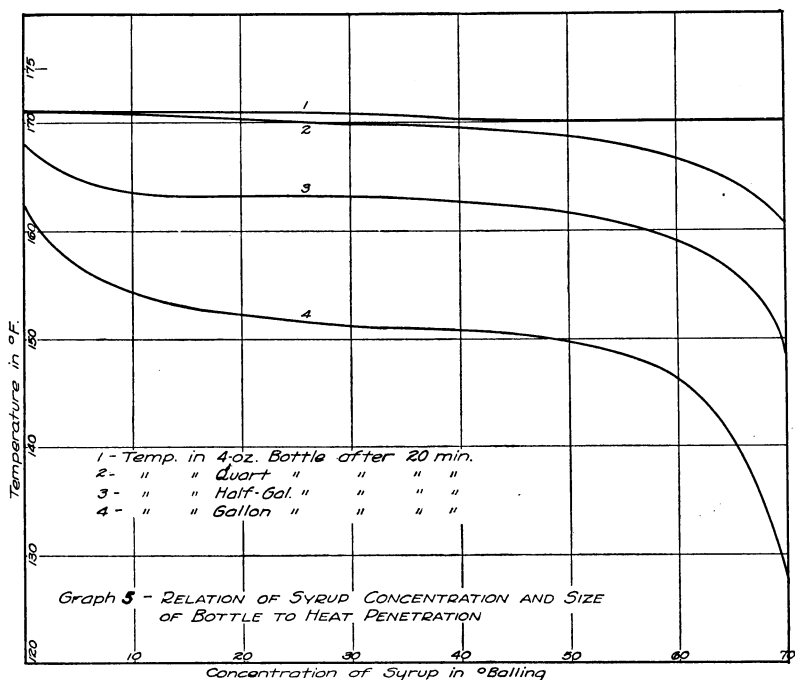
If we keep the size of the container constant and change its shape, the heating surface per unit volume will change. The more nearly a container approaches a sphere in shape, the less is its heating surface per unit volume, and the less is the rate of heat penetration in that container, assuming all other factors to remain constant.

There is on the market a great variety of glass containers. Each *fruit-products manufacturer desires an individual bottle for his* product. Moreover, the types of containers vary with the product. Nevertheless, there is a certain degree of standardization. Familiar forms of glass containers are those used for milk, the Mason jar for home preserves, syrup jugs, and bottles used for cider and grape juice.

To determine whether the effect of shape was marked, investigation was made of heat penetration in water and in 68°-Balling simple syrup in the following pint-size glass containers: slender-necked and short-necked bottles, milk bottle, Mason fruit jar, and tall and flat syrup jugs (see fig. 2). In each of these was placed exactly 400 cc of the fluid investigated. The copper tip of the hot junction was

fixed at approximately the center of the contents.   They were then pasteurized as previously described.

The experiments showed that in the various containers studied there was no very marked difference in heat penetration with either water or syrup, probably because there was no large variation in the heating surface per unit volume in these containers. Moreover the glass in the bottles which had the greatest heating surface was much thicker than in the Mason jar, which had the smallest.

1 - Temp. in 4-oz. Bottle after 20 min.
2 -   "   "   Quart   "   "   "   "
3 -   "   "   Half-Gal. "   "   "   "
4 -   "   "   Gallon   "   "   "   "

Graph 5 - RELATION OF SYRUP CONCENTRATION AND SIZE OF BOTTLE TO HEAT PENETRATION

The thickness of the glass walls of the container is an important factor and the effect of increasing this thickness is to decrease the rate of heat penetration.

From graphs 3, 4, and 5, we may conclude that sugar in dilute water solutions has very little retarding effect on the heat penetration. However, when the concentration is high, the effect on heat penetration is very appreciable, and the rate of heat penetration decreases as the concentration of the solution increases. This decrease in the rate of heat penetration is caused by an increase of viscosity, due to the dissolved sugar, which has an impeding effect on convection currents. The sugar in dilute solutions does not greatly increase the viscosity, but in concentrated solutions, 60–70° Balling, it does so greatly, and thereby proportionately decreases heat penetration.

These results are in accordance with theory and in agreement with the work of other investigators. The effect of viscosity of sugar solutions is discussed fully below.

Bigelow[13] has found that sugar syrups of 10, 20, 30 and 50 per cent in tin cans diminish the rate of heat penetration in the order named.

Magoon and Culpepper[14] have found that sugar solutions show no marked effect upon the rate of changes of temperature at the center of tin cans where the concentration was 10 per cent or less. Even in a 60-per-cent solution the effect was not very marked. Their very interesting and important discussion of the effect of sugar on heat penetration follows:

"The effect of the sugar solution upon the rate of change of temperature at the center of the can is due to the greater viscosity, which decreases the rate of convection in the sugar solutions. The value of the force which tends to produce convection currents in the solution depends upon the steepness of the gradient between the temperature at the center of the can and the temperature at the margin of the solution, so that the force tending to produce convection currents becomes less and less as the temperature at the center of the can approaches that of the bath. It is known that the viscosity of sugar solutions decreases as the temperature increases. It is this characteristic of sugar solutions that makes the temperature shown by the upward curves follow so closely that of distilled water.

In the cooling off, however, there is an increase in the viscosity. Also as the temperature at the center of the can falls, the temperature gradient between the center and the margin becomes flatter; hence, the force tending to cause convection becomes smaller, until finally the resistance due to viscosity is great enough to stop all convection, and the process becomes one of pure conduction, which is very much slower than convection. The difference in viscosity at high and low temperatures makes the cooling curve much different from the upper curve."

The cause of the abrupt lag of the curve at 70° Balling must then be the high viscosity of syrup of this concentration. This viscosity is evidently high enough to markedly impede convection. To test this assumption, the viscosity of the syrups was determined, with the results given in table 1.

It is evident that there is a marked difference between the 70-percent syrup and the syrups of lower concentration.

13 Bigelow, W. D., *et al. Loc. cit.*
14 Magoon, C. A., and C. W. Culpepper. *Loc. cit.*

TABLE 1

RELATIVE VISCOSITY OF SIMPLE SYRUPS AT ROOM TEMPERATURE

Concentration in percentage of sugar	Specific gravity	Relative viscosity*
0 (water)	1.00	1.00
10	1.04	1.05
20	1.08	1.15
30	1.13	1.43
40	1.18	2.00
50	1.23	3.10
60	1.29	6.33
70	1.35	49.00

*All viscosity measurements were made by means of a Stormer viscosimeter. They are intended to show the relative trend of the viscosity and not of its absolute value.

The relative viscosity of sugar solutions increases with increase in concentration. This increase is very slight at first, but accelerates until between 60° and 70° it is very marked. The heat penetration that could be predicted from a consideration of these results is in accord with that actually found. Evidently viscosity is an extremely important factor in heat penetration.

INFLUENCE OF PECTIN

From what has been said concerning the simple syrups, it is seen that in solutions of crystalloids such as sugar, the viscosity does not markedly differ from that of water until high concentrations are reached. On the other hand, it is known that the presence of reversible colloids, such as starch and gum arabic, in water markedly affect the viscosity even in dilute solutions.

Thus Magoon and Culpepper[15] and Bigelow[16] have shown that, in starch solutions, the formation of convection currents was greatly impeded and heat penetration delayed. This was noticeable in the case of 1-per-cent and 2-per-cent solutions but became more marked as the amount of starch increased. Bigelow proved that in a starch solution of 6 per cent, heat penetration was due almost entirely to conduction.

Hence it would be expected that the presence of pectin, another reversible colloid, would markedly affect the rate of heat penetration.

15 Magoon, C. A., and C. W. Culpepper. *Loc. cit.*
16 Bigelow, W. D., *et al. Loc. cit.*

To show conclusively that this is the case, the following series of experiments was made.

Simple Pectin Sols.—Pectin sols with concentrations of 0.5, 1.0, and 1.5 per cent were prepared as previously described. These were pasteurized in gallon bottles.

The results of the experiments showed that pectin in solution retarded heat penetration. Curve 4 in graph 9 is typical of the behavior of pectin sols. The retardation was greater in the more concentrated solutions.

Acidified Simple Pectin Sols.—Pectin sols of the above concentrations with 1 per cent of citric acid added were pasteurized in gallon bottles. Although here too the retardation of heat penetration was appreciable (see curve 3 in graph 9), it was noticeably less than in the former experiment.

The addition of acid probably favored finer dispersion or aided the hydrolysis of pectin to insoluble pectic acid, which would be precipitated from solution. That it had an effect on the relative viscosity may be seen from table 2.

TABLE 2

RELATIVE VISCOSITY OF PECTIN SOLS AT ROOM TEMPERATURE

Per cent of citric acid added	0.0 per cent pectin	0.5 per cent pectin	1.0 per cent pectin	1.5 per cent pectin
	Relative viscosity			
0	1.00	1.13	1.48	2.08
1	....	1.00	1.04	1.93

Cane-Sugar Syrups Containing Pectin with and without Added Acid.

Four lots each of simple syrup of 40° Balling and 70° Balling were made. The first contained 0.5 per cent and the second 1.5 per cent citric acid. The third contained 0.5 per cent citric acid and 1.0 per cent pectin; the fourth contained 1.5 per cent citric acid and 1.0 per cent pectin. These were pasteurized in gallon and pint bottles.

It was found that the presence of pectin had a decided retarding effect on the rate of heat penetration. Graph 6 is typical of the results obtained.

However, the presence of acid in simple syrups had a decided tendency to hasten the rate of heat penetration, especially as the concentration of acid was increased. This was perhaps due to inversion of the cane sugar by high concentrations of acid. The presence of acid in pectin sugar sols also had some tendency to hasten the rate

of heat penetration, probably owing to reduction in viscosity of pectin due to increased acidity.

The syrups of the above series to which pectin was added contained also acid; and in preparing them relatively high temperature and vigorous mechanical stirring were used.   This probably accounted for the fact that they did not jell, a result contrary to the findings of Lal Singh[17] and others.   Wendelmuth[18] has found that pectin solu-

Graph 6 – EFFECT OF PECTIN ON HEAT PENETRATION IN ACIDIFIED 70° BALLING SYRUP

1 - Pasteurizer
2 - Water
3 - 70% Sugar, 1½% Citric Acid
4 - 70% Sugar, 1½% Citric Acid 1% Pectin (not jelled)
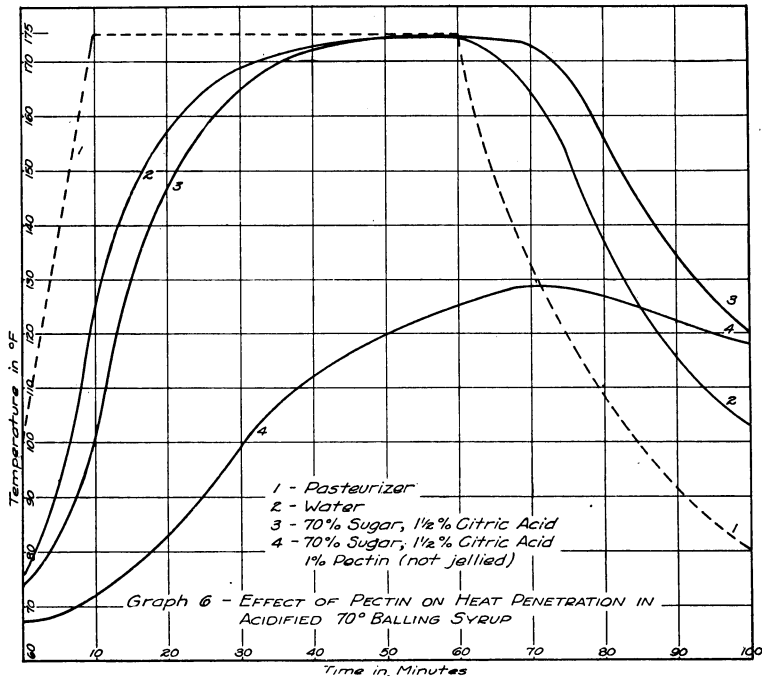
tions which were excessively stirred or heated did not jell, even though the proper amount of acid and sugar was present.

In pectin sols, the results obtained depend upon the manner of preparation.   Heating and rapid mechanical stirring, especially in the presence of acid, tend to reduce the viscosity of the pectin sols by hydrolysis of the pectin to a less highly methylated state and by the coagulation of particles in suspension.   Experiments have shown

17 Singh, Lal.   Important discoveries in jelly making.   Canning Age 3:5–8. 1922.

Also, Practical experiments in jelly making.   Canning Age 3:11–14.   1922.

18 Wendelmuth, Gerta.   Über die Gelierfähigkeit von Obstsäften und Pektinlösung.   [On the jellying ability of fruit juices and pectin solutions.]   Kolloidchemische Beihafte 19: 115–137.   1924.

that the difference noted in graph 7 is due rather to the use of heat in preparing the products than to a difference in acid content, although the difference in acidity also contributes to the effect noted.
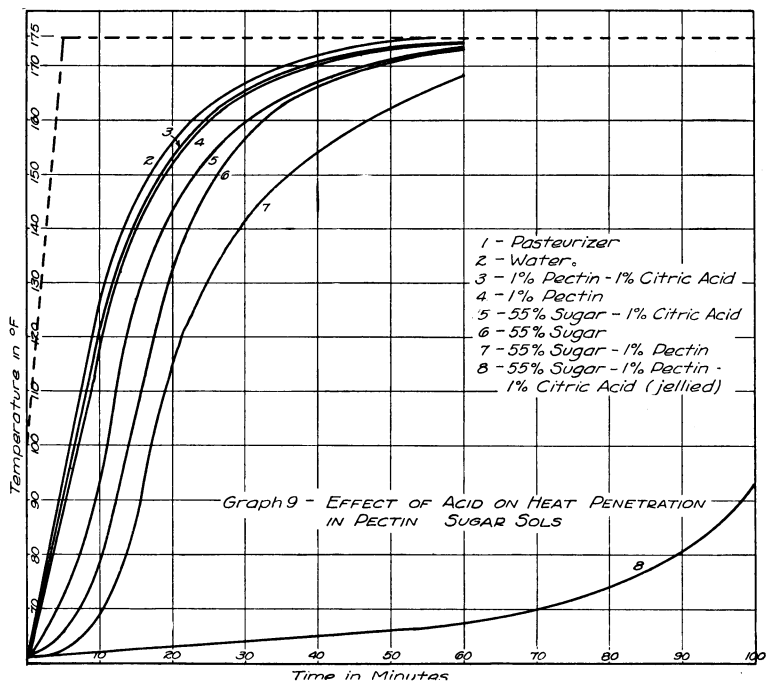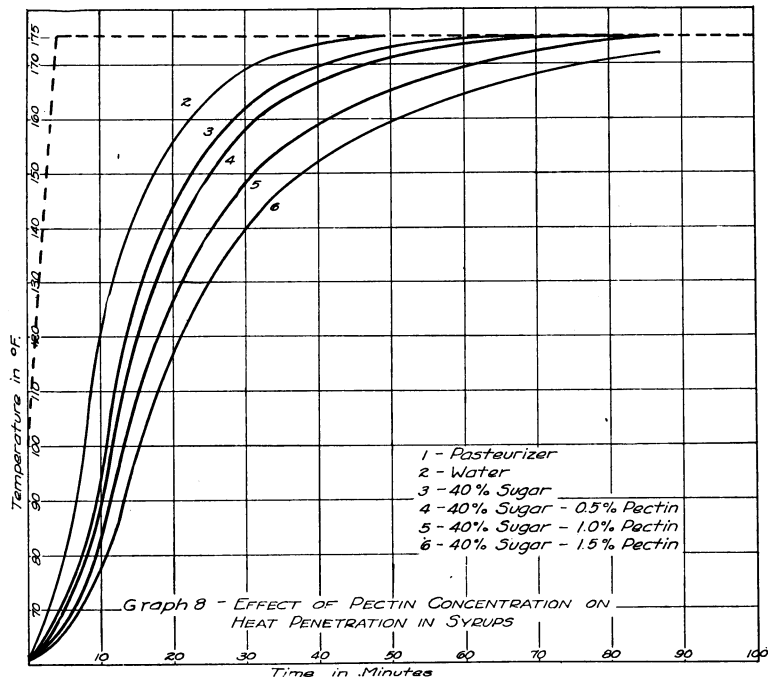
In our second experiment with pectin, 0.5 per cent, 1.0 per cent, and 1.5 per cent of pectin were added to 40° Balling and 55° Balling simple syrups. To one series 1 per cent of citric acid was also added. These materials were incorporated in the manner previously described to yield syrups of maximum viscosity. They were pasteurized in gallon bottles.

1 - Pasteurizer
2 - Water
3 - 40% Sugar, 1% Citric Acid
4 - 40% Sugar, 1% Pectin, 1½%
   Citric Acid - Prepared by
   Heating and Stirring
5 - 40% Sugar, 1% Pectin,
   1% Citric Acid - not Heated

Graph 7 - EFFECT ON HEAT PENETRATION OF MANNER
   OF PREPARING PECTIN SOLS
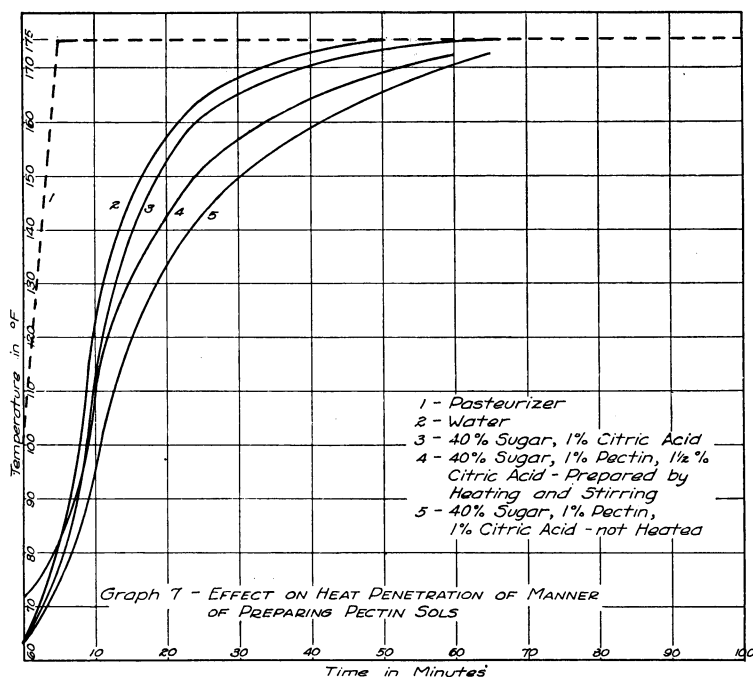
The results obtained showed that pectin markedly retarded heat penetration in syrups. This retardation increased with increase in concentration of pectin (graph 8). Acid, where it did not cause jellying, reduced this retarding effect, probably because it reduced the vicosity of pectin sol (graph 9).

This is in accordance with the work of Ohn,,[19] who found that the relative viscosity of sols made from pectin, citric acid, cane sugar, and distilled water depended upon the proportion of pectin, acid and sugar present. Within certain limits, if the hydrogen-ion concen-

[19] Ohn, Asta. Viscosity of pectin sols. J. Ind. Eng. Chem. 18: 1295–1297. 1926.

1 - Pasteurizer
2 - Water
3 - 40% Sugar
4 - 40% Sugar - 0.5% Pectin
5 - 40% Sugar - 1.0% Pectin
6 - 40% Sugar - 1.5% Pectin

Graph 8 - Effect of Pectin Concentration on Heat Penetration in Syrups

Temperature in °F.

Time in Minutes

1 - Pasteurizer
2 - Water.
3 - 1% Pectin - 1% Citric Acid
4 - 1% Pectin
5 - 55% Sugar - 1% Citric Acid
6 - 55% Sugar
7 - 55% Sugar - 1% Pectin
8 - 55% Sugar - 1% Pectin - 1% Citric Acid (jellied)

Graph 9 - Effect of Acid on Heat Penetration in Pectin Sugar Sols

Temperature in °F.

Time in Minutes

tration is sufficient, any increase in the amount of pectin or sugar added caused a noticeable increase in the relative viscosity. If the hydrogen-ion concentration is low, such increases cause only a slight change in the relative viscosity.

In this series of experiments, the retardation in the rate of heat penetration due to the presence of pectin was greater than in the former series (graph 7) for the reason stated above, i.e., that the pectin in this second series was dissolved without heating in order to secure maximum viscosity.

The consistency of a jelly is such as to prevent convection currents. Heat penetration, being caused only by conduction, is very slow (graph 9). When the solution jellied, the heat penetration was not influenced markedly by change in pectin and in acid content. Also, it was found that changing the sugar content from 55 per cent to 70 per cent did not noticeably affect the heat penetration in a jelly.

That the foregoing results could be predicted from a consideration of the viscosity of the solutions is evident from the following table. Unfortunately sugar syrups of 55° and 70° Balling containing pectin were too stiff at room temperature to permit determination of viscosity by the instrument used.

TABLE 7

RELATIVE VISCOSITY OF PECTIN-SYRUPS AT ROOM TEMPERATURE

Per cent pectin	Per cent sugar	No citric acid added	One per cent citric acid added
		Relative viscosity	
0 (water)	0	1.00	....
0	40	2.00	1.67
0.5	40	3.20	2.61
1.0	40	7.50	6.45
1.5	40	9.25	8.95

COORDINATION OF MOST IMPORTANT FACTORS

Graph 10 makes it possible to compare readily the pasteurizing time for various syrups in various containers. There are two series of curves in this graph. In one, the time necessary for the temperature of the syrup to reach 170° F in various bottles under the pasteurizing conditions previously described is plotted as abscissas against the concentration of sugar as ordinates. In the other the relative viscosity of the syrup as abscissas is plotted against the concentration of sugar as ordinates.

The use of these curves will be seen by applying them to the following examples.

Example 1.   Suppose one desires to pasteurize 40° Balling simple syrup in gallon bottles and wishes to know the pasteurizing time necessary.   By consulting the curve for the gallon bottles, it will be seen that it requires approximately 45 minutes for the temperature at the center of the container to reach 170° F.   If 170° F is the
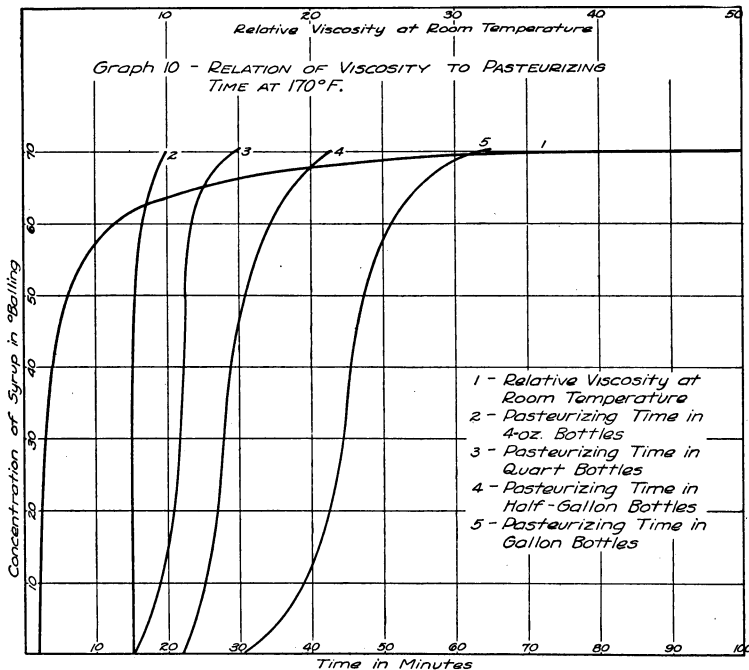
pasteurizing temperature this will be the approximate time required for pasteurization.

Example 2.   Suppose it is desired to pasteurize a fruit syrup of 40° Balling in gallon bottles and it is suspected that the viscosity of this syrup is greater than that of 40 per cent simple syrup.   Let us assume that it is found that its viscosity is roughly five times that of water.   From the viscosity curve, it is seen that the concentration of simple syrups having this viscosity is approximately 60 per cent. The time required for the temperature at the center of a gallon bottle of syrup of 60° Balling to reach 170° F is then found (as in example 1) to be approximately 55 minutes.   If 170° is the pasteurizing temperature, this will be the approximate pasteurizing time.

However, the reader must be cautioned against splitting hairs in using this graph. It will give the pasteurizing time only to within five minutes. Other factors than viscosity enter the case also; such, for example, as thickness of the walls of the container; agitation of the water in the pasteurizer and the shape of the container. The graph is presented principally to illustrate the very great effect of viscosity on heat penetration in liquids.

SUMMARY

1. The rates of heat penetration in bottled syrups as affected by sugar concentrations, viscosity of liquids, size of bottle, and variety of syrup were determined.

2. It was found that sugar exerted only a slight retarding effect at low concentrations but an appreciable retarding effect at concentrations above 50° Balling. A very marked decrease in rate occurred between 60° and 70° Balling. This was proved to be due to increase in viscosity of the solutions with increase in sugar concentration.

3. Differences in the rate of heat penetration in containers of different sizes were not very great in small containers such as pint, 8-ounce and 4-ounce bottles, but a marked difference was found in the rate between the gallon and the smaller containers. This last fact probably accounts for the greater loss by molding of fruit juice in gallon containers than in small containers; most manufacturers give little or no greater time to gallon-size than to smaller containers.

4. Syrups rich in pectins and gums transmitted heat very much more slowly than those poor in these constituents. This was proved to be caused by the effect of pectin on the viscosity. Pectin very greatly increased the viscosity of the sugar solutions of fruit juices and thereby greatly reduced transmission of heat by convection.

5. Where the juice, syrup, or concentrate actually jellied, heat penetration became very slow, but was not noticeably affected by increase or decrease of the sugar content, provided the sugar content was not reduced below that required for jelly formation.

ACKNOWLEDGMENT

The titles of the Technical Papers of the California Agricultural Experiment Station, Nos. 1 to 20, which HILGARDIA replaces, and copies of which may be had on application to the Publication Secretary, Agricultural Experiment Station, Berkeley, are as follows: