CANNING OF THE JUICE
OF THE BITTER ORANGE
(C. Aurantium)

by

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CANNING OF THE JUICE OF THE BITTER ORANGE
(C. Aurantium)

CHAPTER I
INTRODUCTION

Northwest Frontier Province (Dominion of Pakistan),
the writer's home, lies between 32 and 35 North latitude.
The maximum summer temperature reaches up to 120°F and the
minimum winter night temperature comes down to 29°F. The
soil varies from loamy to heavy clay. The average annual
rainfall is between 13 and 20 inches.

Owing to the recent development in irrigation
systems from snow-fed rivers, and the variety of climates
the province enjoys, fruit tree plantations of all kind,
deciduous as well as citrus, have been increasing yearly
during the last 10 to 12 years. It has been observed that
the farmers plant the Seville orange (sour orange, or
bitter orange) around their orchards, on the sides of the
roads in the orchards, to serve as windbreaks, to give
shade and to add to the beauty of the orchard. On the
other hand the cylindrical shape of the tree, its brightly
colored reddish oranges and its lush, dense, dark green
foliage has made it a prominent ornamental plant, which is
planted in every public and municipal park. It is planted
near bungalows and in back-yards just as holly is grown in
The seed of the fruit is used for the raising of the seedlings which serve as stocks for the budding of varieties of sweet oranges and the rest of the fruit is discarded. In some cases the rind is used for flavoring cooked rice after the rind is sliced and dried in the sun. In other cases the juice is used on "Kabab" (a meat preparation similar to hamburger) in the same fashion as lemon is used in this country on fish. The price fetched by the fruit is so low that in the majority of cases the bare expenses of the harvesting and transportation charges are not realized with the result that the fruit is left on the trees until it falls by itself and gets rotten on the ground.

It is thus clear that if new planting of the fruit continues to increase as it has in recent years, it will not be long until the wasting of this fruit will be a great economic loss to the growers. Utilization of this fruit, as in juice, would be of much value in the light of the following facts.

From the literature it is evident that the food chemists have very recently realized that this fruit is a rich source of vitamin C. The detailed discussion of the different investigators, who have worked on the subject, will be made in its proper place in this paper but it will
suffice to say here that the juice of this species usually has more vitamin C than common sweet oranges. This is the most needed vitamin at present for the people of the province already mentioned, where the absence of this vitamin in diet has resulted in rickety children, with swollen gums and bad teeth.

A curious reader may suggest to use the fruit for the preparation of marmalade for which this fruit has been so well known by the English. The reasons for not doing so are not far to be sought. The climate of the province in summer is very hot and hence the use of cold drinks has become one of the necessities of life while to use marmalade is supposed to be a luxury. The purchasing power of the common man is so low that it is difficult for him to use marmalade, at least for some time to come.

Also, the population is 99 per cent Moslem who keep one month fast a year. To break the fast in the evening, every person rich or poor, prepares some sort of sweet drink to quench his thirst.

Taking all these facts into consideration it was decided to tackle the problem in such a way that the producers of the fruit and the consumers of the juice are equally benefited, that the producer of the fruit may find some market for his fruit and the consumer may be able to pay for it. This was done by partial de-acidification of
the juice and then adding sugar to bring it to the desired sugar-acid ratio, and secondly to make a syrup by the addition of sugar to the juice and then diluting it with water before drinking. The former will serve as a juice and the latter as a beverage. These two products were then canned and bottled.
CHAPTER II
REVIEW OF LITERATURE

The bitter oranges (C. Aurantium) have been used for the manufacturing of marmalade for a long time. The seed obtained from the fruit has been used for the purpose of rootstock for the sweet oranges. The literature, whatever the writer could study, deals with these subjects. It seems that no systematic study has been made on the canning of the juice of this species of oranges. The processing methods here used in the canning of the juice are those used for the common oranges and hence the literature discussed will be that pertaining to canning of sweet orange juice.

A. Behavior of Oranges in Cold Storage

Technically speaking, the storage study of the oranges does not come within the scope of the title of the thesis. This part of the work was included for two reasons. First, every juice industry, large or small, has to sometime store fresh fruit for a certain period of time due to unavoidable circumstances. For example, in the state of Texas grapefruit is attacked by fruit fly if it is harvested after May first. The fruit is therefore picked before that date so as to avoid the attack of the pest.
The lemons in California are ready for harvesting in the winter season or the fall, while the fruit is needed in summer for consumption. In that state the fruit is picked and stored until needed. In some places the fruit is to be shipped in refrigerated cars for a number of days before it is utilized. Strikes in the factories may sometimes force the management to store the fruit until conditions are normal. Many examples can be cited.

The second reason for the inclusion of this part of the work in the study was that in the knowledge of the experimenter no work seems to have been done on this phase and hence it would serve as useful information for any worker in the future.

Miller (46) reports that citrus fruits are very sensitive to low temperatures. He states that certain disorders like aging, pitting and watery breakdown occur only at low temperatures and will not occur if the fruit is kept at higher temperatures. The most dangerous zone for occurring of these maladies as stated by the same author, lies between 32° to 40°F in the case of oranges.

Stahl and Camp (61) working on oranges, found slight loss of acid and no significant loss of total solid in a period of two weeks to two months. But in the period of four months storage these authors found a slight increase in hydrolizable sugar, total sugar and pH. This work was
Harvey and Rygg (28) found in the peel of Washington Navel oranges an increase of hydrogen ion concentration and an increase in the glucoside hesperidin. They found a decrease of the soluble solids in the peel. This experiment was conducted for a period of seven weeks. They conclude that the changes accelerated at higher temperatures.

Huelin (30) and Hamersma (26), found no loss of vitamin C in the case of the late and mid-season oranges in a period of two weeks to three months storage period. Brately (9), working on tangerines in the New York cold storage market, found a marked loss of vitamin C. He reports that the loss of vitamin C was higher at higher temperatures. Sergree (58) found no loss of vitamin C in the bitter oranges in a period of one to one and a half months. Bracewell and Zilva (8), working on Jaffa oranges, report that no marked variation in vitamin C content of the fruit was observed when it was kept at 15°C for two months time.

In the literature there does not seem to be an agreement among the various workers as to the most suitable temperature for the storage of oranges. This is because climatic conditions, soil variation and agricultural practices vary and these factors have a profound effect on the behavior of the fruit in cold storage. Thus for long
storage of oranges the U.S.D.A. (55) recommends cold storage temperature of 34° to 38°F. On the other hand Overholser (50) recommends temperature of 36° to 38°F for oranges to be shipped to the orient from California. State workers in Florida (1) recommend temperature of 37½°F and those in California recommend temperature of 38° to 40°F. Miller (57) recommends 85 to 90 per cent relative humidity for oranges.

Stahl and Camp (61) working on the maturity of oranges best for cold storage, state, "From the standpoint of metabolic changes, it is best to store slightly under normal mature fruit." They have concluded that mature oranges do not hold up well and take on storage tastes rapidly. Miller (46) reports that the relative humidity in cold storage is related to the incidence of rind breakdown in citrus fruits. Low relative humidity accelerates the development of low temperature injuries, such as pitting, weight loss and rind breakdown, of oranges. Stahl and Cain (62) exposed oranges to dry atmosphere by storing over sulfuric acid and found that the fruit lost weight and firmness and that an increase in pitting resulted.

Miller (46) summarized his discussion as follows: 
".....Some of the factors that have been reported to pre-dispose citrus fruits to these low temperature injuries are a high percentage of potash fertilizers, a relatively high
content of moisture and organic matter in soil, the susceptibility of specific varieties, harvesting fruit after relatively high mean temperature, storing fruit from the outside branches of the tree, or fruit that is physiologically immature, processing in the packing house and low relative humidity in storage rooms."

B. Citrus Juice Canning Procedure

It has been stated in the previous discussion that in the absence of any special method for the canning of bitter orange juice in the literature, the procedure of canning sweet orange juice will be reviewed. Selection of variety and the suitable maturity of the fruit will not be considered as they were not a part of this investigation. Thus the following points will be reviewed: 1. de-oiling, 2. extraction, 3. straining, 4. de-aeration, 5. pH and acidity, 6. pasteurization, 7. canning versus bottling, 8. vacuum sealing, and 9. type of can.

1. De-oiling

The peel of oranges contain oil glands and at the time of extraction of the juice by either burring or reaming the glands are pressed or ruptured, resulting in the incorporation of oil in the juice. The presence of a
limited amount of this oil is essential for the aroma of the juice, but an excessive amount has been found by some workers, to be responsible for the development of sharp terpene-like flavor during storage. Lueck and Pilcher (44), recommend that the peel oil content in the juice be maintained below 0.03 per cent. Riester et al (54) have made an extensive study on the matter and have found that if the oil content of the juice is more than 0.05 per cent an objectionable terpene flavor develops within one month at room temperature.

These authors have found that if the oil content in the Florida Valentia orange is within the limit of .03 to .04 per cent terpene flavor would not develop but the juice would have slightly more orange flavor and aroma than samples containing less peel oil. Boyd and Peterson (7) reported that the oil desired in orange juice be .01 per cent. They were able to keep the orange juice having .007 to .001 per cent of peel oil for a period of 18 months at room temperature without any off-flavor. They however found that if the oil content was absolutely excluded by removing the peel from the oranges and then extracting the juice, the result was that the juice was "flat" and lacking in aroma. They recommend that the oranges be given a hot bath for a period of at least one minute in water at 180°F or more. Scott (57) reports the same method for
grapefruit. Shearon and Burdick (59), use water at temperature below boiling and recommend 30 seconds immersion. Curl and Veldhuis (24) very recently repudiated the findings of all these authors and state that, "The suspended matter, which includes the fatty fraction, was the principal contributor to the off-flavor of aged orange juice. Peel oil appears to have no particular effect except that of masking the off-flavor to some extent. The essence fraction had little or no effect. Only a small amount of off-flavor was attributable to the water-soluble, nonvolatile fraction."

2. Extraction of the Juice

In the processes of juice extraction, pressure and tearing forces are exerted upon the various parts of the orange. As a result the extracted juice contains more or less macerated material from these tissues in the form of solid particles. In addition to the torn tissues, the (freshly extracted before straining) juice contains the seed from the oranges. According to Higby (29) all the varieties of the sweet orange contain, to a varying degree, a non-bitter, water-soluble parent substance which liberates an intensely bitter principle on hydrolysis. Working on Washington Navel oranges Higby was able to separate and identify the bitter principle, from the edible portion, and
named it isolimonin. He considers it to be an isomer of limonin and citrolimonin. He found that the parent substance diminishes as the fruit matures. Hall (25) has concluded that hesperidin is the most hydrolyzable complex substance in the orange fruit and it may be isolimonin. Joslyn and Hohl (41) report that the seed and the carpel-lary membranes of the oranges, have bitter glucosides and some other constituents which if allowed to stand in the juice will give it a bitter taste. For all these reasons the removal of the seed and the coarse particles from the juice immediately after extraction is very important.

Selection of best variety and maturity, and the extraction of the juice by methods which do not incorporate the bitter principle in the juice need careful consideration.

3. Straining

The separation of the seed and the coarse particles which have been found to be responsible for the development of the bitter taste in the juice in storage is important no doubt, but the complete removal of the suspended material from orange juice is not desirable from many points of view. Thus Tressler et al (65), Lueck and Pilcher (44), Joslyn and Marsh (39), Joslyn and Hohl (41), state that the flavoring principle which gives a characteristic flavor and aroma to the citrus juice is found in chromatophores which
are incorporated in the juice at the time of extraction and which remain in the suspended form. Joslyn and Hohl (41) suggest that the diameter of the perforation for screening of the juice should not be more than .03 inches. Vilté and Coustou (69) report as follows:

"Vitamin C content in filtered juice decreased to traces after 29 days. In unfiltered juice 90 per cent of the original vitamin C was present after 37 days. Addition of the filtration residue to the filtered orange juice resulted in the retention of 80 per cent of vitamin C content after 28 days. Pectic and protein substances seem to have protective effect on vitamin C."

Beattie et al (3) report that in the juice the vitamin C is oxidizable while the pigments are reducible and it is possible that the two may react at the expense of vitamin C.

Whether the loss of vitamin C in the citrus juices is due to the presence of the carotinoids or not the trend of the public is toward cloudy juices and hence the juice might beneficially have some percentage of the suspended matter in it.

4. De-eration

Oxygen is generally reported to be responsible for the loss of vitamin C and the loss of color and flavor in
the citrus juices at the time of extraction and during storage. According to Boyd and Peterson (7) the citrus juice has about 0.18 to 0.2 per cent of oxygen from the intercellular spaces. In the processes of extraction more oxygen from the air is incorporated in the juice. Thus Pulley and Von Loesecke (53) report that in the commercial juices the oxygen found before de-aeration was 2.41 to 4.67 cc per liter. Tressler et al (65) state that oxygen is present on the surface of the fruit particles. Some of it is dissolved by the juice, and at first only a small portion is fixed by the constituents of the juice as peroxides. Therefore, if the juice is subjected to high vacuum, just after extraction, a great proportion of the oxygen as well as other gases will be removed.

Joslyn (34) working on the vitamin C content of fruit juices stated that the vitamin C was stable to high temperature provided there was no oxygen in the juice or in the container. He attributed the loss of vitamin C to the vitamin C oxidase enzymes which should be inactivated. Humberger and Joslyn (31) state that in the orange juice there are other reducing substances which may retard the immediate loss of vitamin C by oxidation. This is not the case with lemon juice which does not seem to have these reducing substances.
5. pH and Acidity

In the sterilization of food and food products pH plays an important role. An arbitrary line on the pH scale has been drawn which divides food and food products into acid and low-acid foods. This line is at pH 4.5. Foods or juices having pH above this line are classed as low-acid foods while the ones having pH value below this figure are classed as acid foods. The sterilization time and temperature of a food depends largely upon the pH of the food or the juices. Foods or juices having pH value below 4.5 can be sterilized at the temperature of boiling water in a few minutes while the foods having pH value above this figure must be sterilized under pressure.

Citrus juices fortunately come under the acid foods and hence they can be sterilized at the temperature of boiling water. Enzyme inactivation in citrus depends much upon the pH of the juice. Joslyn and Sedky (40) working on the inactivation of the pectin enzymes in Valentia and Navel orange juices found that at pH 4 the enzymes were more active than at pH 2.2. Lowering the pH reduced the time required for the inactivation of the pectic enzymes; conversely an increase in pH increased the time.

Gruess et al (23) found that the preservative action of sodium benzoate depended upon the pH of the juice in which it was used. Summarizing their investigation the
The concentration of sodium benzoate, sodium salicylate, potassium acetate (acetic acid) and sodium sulfite (sulfurous acid) required to prevent the growth of molds, yeasts, and bacteria were much greater at pH 5 to pH 9 than at pH values in the distinctly acid range of 2.0 to 4.5. The concentration of sodium benzoate required to preserve several food products was found to depend upon pH value. In some cases more than 200 times as much preservative was required at neutrality as at pH 3 or less.

Tressler et al (67), stressing the importance of pH in the destruction of microorganisms, stated:

"Pure canned or bottled carrot, spinach, lettuce, and other vegetable juices of slight acidity must be heated in a pressure sterilizer at 240° to 250°F in order to destroy spores of some of the more heat resistant organisms. When heated to this temperature they sometimes acquire more or less disagreeable taste resembling that of scorched or over-cooked vegetables. If acidified so that pH value is 4.2 or less they may be pasteurized at 180° to 212°F without danger of spoilage by putrefactive organisms."

Experiments performed by Cruess (17) have proved that peas, string beans, corn, and fish inoculated with spores of Bacillus botulinus were sterilized perfectly in one hour at 100°C; by heating in dilute brine acidified
with lemon juice to approximately 0.2 per cent acidity expressed as citric. The same products not acidified developed a vigorous growth of the organism, even after three hours boiling in sealed containers. When fed to guinea pigs this product was proved to be very toxic, the victim showing typical signs of botulism.

6. Pasteurization

In the case of citrus juices the purpose of pasteurization is two fold. The first is to kill the spores of molds and yeasts and the heat resistant bacteria and the other purpose is to inactivate the enzymes like the pectic enzymes and the peroxidase enzymes.

Tanner (63) reports that the pulp of the citrus fruit is sterile. It means that if proper care is taken in selecting sound fruit, if it is washed thoroughly, and the equipment is kept clean, pasteurization is a simple procedure in juice canning.

To decrease the surface organisms from the fruit and the equipment Shearon and Burdick (59) use wetting agents and sanitizers like the quaternary ammonium compounds at the rate of 0.1 per cent for washing the oranges. The equipment is kept clean with chlorinated water.

In the sterilization of citrus juices the molds and the yeasts can be easily destroyed but the inactivation of
the pectic enzymes will need high temperature. If these enzymes are not inactivated they will convert the pectic substances into the insoluble pectic acids with the result that it and other suspended substances settle and make the juice clear.

The first attempt made in this connection was by Cruess (14), who reported that the citrus juice would, if not pasteurized, become clear in two days but pasteurized citrus juice would not settle and would remain cloudy. He pointed out that by heating the juice in bottles at 185°F the settling of the juice is prevented for about seven months.

Extending the work of Cruess, Joslyn and Sedky (40) found that the inactivation of the pectic enzymes depended upon time, temperature, pH and the nature of the enzymes. By keeping the pH constant these authors concluded that the enzymes in the case of Valentia oranges were more active than the Navel oranges, that the enzymes of the lemons are the least active of the three juices. They recommended the short time and high temperature for the first time. These authors stated that Valentia orange juice, if pasteurized at 82.5°C for ten minutes, will remain cloudy for 513 days.

Keeping the temperature constant and raising or lowering the pH of the different juices like Valentia, grapefruit and lemon, these authors found that inactivation
of the pectic enzymes could be accomplished in shorter time at lower pH than at higher pH.

Loeffler (42) working on the problem of the maintenance of cloud in citrus juice reported that the cloud was actually increased by pasteurization and not merely stabilized. He states that the loss of cloud in citrus juices in storage is not entirely due to the pectic enzymes since heavily clouded samples pasteurized at comparatively high temperatures will lose an appreciable portion of their cloud. He recommends homogenization before pasteurization. Phaff and Joslyn (51) state that in citrus juices, pectin esterase acts on the pectin so that it no longer acts as a protective colloid to prevent the settling out of the solid in freshly packed juice.

As to the method of pasteurization all workers agree that short time and high pasteurization temperature is the most desirable for the keeping of the best aroma and flavor of the juice and for the retention of vitamin C. The old holding method of pasteurization has been replaced by flash pasteurization exclusively. In the literature temperatures of 190° to 192°F (59) for one minute, 225° to 240°F (7) for a few seconds, have been recommended.

The objection to the holding methods are the loss of the original flavor, the loss of vitamin C and the cooked flavor, mentioned by Lueck and Pilcher (44), Riester et al
and many others.

For the pasteurization in the containers, Magoon and Culpepper (45) summarized their investigations that the factors affecting the rate of change of temperature in the center of the can are: the diameter of the container, the conductivity, the thickness and the radiating power of the walls of the container; the temperature, conductivity and mobility of the content of the container; the temperature conductivity and the movement of the medium surrounding the container. Biglow et al (4) pointed out that the heat penetration is influenced by the size of the container, the initial temperature of the content of the container, the temperature of the retort, the nature and the consistency of the food, and the presence of the forced convection current due to the rotation of the container during the processing.

Irish et al (33) extended the work of these authors by working on the glass containers and using syrups and concluded that in the case of low concentration of sugar in the syrup there was only a slight retarding effect of the heat penetration but an appreciable retarding effect was observed when the balling degree reached above 50. Marked decrease in the heat penetration was observed when the syrup was made of 60 to 70 balling. They found that juice syrups having a high quantity of pectin and gums retarded
the heat penetration to a marked degree. They summarized that viscosity and diameter of the glass containers have the most appreciable effect on the heat penetration.

7. Canning Versus Bottling

The apparent advantage of the bottling of citrus juices is that the consumer sees the color of the product easily and can decide whether or not the juice is to be purchased. The other advantage is that the bottles can be reused. However, its great disadvantages over canning are the high transportation charges, high breakages in handling, and the loss of vitamin C.

Moore et al (48) have found that citrus juice, if kept in bottles at room temperature for a period of six months, will have less ascorbic acid than the same juice canned. They found, however, that the flavor of the bottled juice was better than the canned juice, though the difference was not significant after six months. The cans used in this experiment were plain.

Joslyn and Marsh (35) have found that the color of the glass-packed orange juice became darker on storage at 80° to 100°F but that of the canned juice did not. They found no change of color in the bottled juice if the temperature was low. They concluded, however, that although the color of the juice in the case of the bottled juice
changed, there was no change in the flavor.

Von Loesecke et al (70) have confirmed the investigation of Joslyn and Marsh and concluded that the enamel-canned orange juice had definitely better flavor than the juice preserved in the plain cans and bottles. They reported that the change of flavor was found only when the temperature was 80°F or higher. It did not occur at the storage temperature of 60°F or lower. They confirmed the same investigation as stated by Joslyn and Marsh that the canned juice had better color and retained more vitamin C than the bottled juice. Loeffler (43) summarized his findings as follows:

"...less than two months storage at hot summer temperature could make unpalatable the best quality of glass-packed orange juice but that the flavor of the freshly bottled juice could be retained almost indefinitely if kept at 40°F."

8. **Vacuum Sealing**

The purpose of vacuum sealing is to remove the air from the headspace of the container. Lueck and Pilcher (44) have mentioned the unpublished work of Clark and Lachele, who found that if more than 0.1 per cent by volume of oxygen is present in the citrus juice, packed in enamel cans, disruption of the bond between the enamel and the tin
takes place, causing a disagreeable flavor. Joslyn and Marsh (36) reported that the decrease in iodine number in the citrus juice was caused not so much by heating or sodium benzoate as by oxygen in the headspace of the container.

Reister et al (54) did not find any significant difference of vitamin C content between the cans from which the oxygen of the headspace was removed and ones from which no oxygen was removed. They found, however, that the removal of the oxygen from the headspace reduced the corrosion of the tin plate by about 50 per cent after a period of 13 months at room temperature.

Joslyn (34) states that citrus juice deteriorates in color and flavor on exposure to air, especially during heating.

Cruess (18) objected to the presence of air in the can for the reason that it acts as a depolarizer and thus accelerates the corrosion of the can. He further stated, "A second object is to produce a vacuum so that the ends of the can on the grocer's shelf will be concave and thus indicate to the prospective purchaser that it is in sound condition. Convex or bulged ends usually indicate gaseous spoilage."

9. Enamel Versus Plain Cans

The necessity of the enamel cans was felt by the
juice industry when it was found that the highly acid juices like citrus, corroded the plain cans in storage. The juice was often found to acquire what was termed a "tinny" flavor. It was also observed that the juice with much anthocyanin pigment would lose its color in the plain cans. Since then a considerable amount of work has been done on the comparative merits of the two types of cans.

Cruess (19) explained the theory of can corrosion with acid foods and stated that in the presence of oxygen the anthocyanin pigments accelerate the corrosion of the can. He further stated that the temperature and agitation of the can also adds to its corrosion.

Joslyn and Marsh (38) reported that the taste of the juice in the citrus enamel can was better than that in the plain can. They found that all the enamel cans were not equally good.

Reister et al (54) found no difference in the ascorbic acid content nor in the taste of the juice packed in the two types of cans. Their report indicated that the members of the taste panel were persistent in their opinion but the divergence of the opinions on the preference of the special type of can did not permit them to draw any definite conclusion.

Von Luessecke et al (70) report that the flavor of the juice in the enameled can was better than the juice
packed in the plain can. They state, however, that the enamel can was not found to resist the corrosion after six months storage. Boyd and Peterson (7) report as follows:

"...The corrosion of the tin is, to some extent, dependent on the oxygen in the can. This slight change in color and flavor is not seriously objectionable, but is at least undesirable in a product that must compete, to some extent, with fresh juice. The use of enameled cans prevents the loss of color and the development of flavors due to metallic contact; with some enamels the terpene flavor may be less, due apparently, to some absorption of terpene in the enamel. With a given oxygen content more oxidation of the juice constituents occurs in enameled cans than in plain cans."

C. De-acidification

A certain percentage of acid is, no doubt, essential for the flavor and keeping quality of the juice, but an excessive amount of acid in a juice has been found to be objectionable from the consumers point of view. Partially neutralizing the acidity to the extent that it will be palatable to the consumer has been practiced for some time in the food industry.

In the dairy industry sour cream is neutralized partially to the desired taste and flavor. The common
neutralizers used in the dairy industry as mentioned by Wilster (71), are soda and lime. Commercial neutralizers are sold under the name of "Peerless," "Allwood," "Perfection," and others.

For the de-acidification of grape juice Cruess (20) states: "It is possible to neutralize all or most of the acid of grape juice by the addition of calcium carbonate or calcium hydroxide. Insoluble calcium tartrate is formed and can be separated from the juice by settling, racking, and filtering...."

For the de-acidification of other juices Cruess (15) mentioned the method as follows: "Some juices are improved by de-acidification by removing a portion of the acid before concentration. This is true of grape, sorgham, and apple juice. The acid is removed by chalk."

The method described is to neutralize three-fourths of the juice at the rate of one ounce of chalk per three-fourths gallon of juice. Heat the juice to boiling, keep it for 24 hours, filter and add the one-fourth of the juice to the neutralized juice. Tressler et al (66) have recommended the de-acidification of rhubarb juice. They have described the method of partial de-acidification of the juice by the addition of calcium carbonate at the rate of 0.32 per cent. The juice is first heated to 180°F and the required amount of calcium carbonate added. The juice is
allowed to stand for 30 minutes, chilled, and allowed to stand overnight. The calcium oxalate settles and the juice is then decanted.

Worsley (72) concentrated the juice of the sour orange under vacuum. The juice was neutralized by sodium carbonate and no loss of vitamin C was found. He reports that a palatable concentrate was obtained by this method.

In the United States Fruit and Vegetable Chemistry Laboratory at Los Angeles, California (10), western grapefruit juice is being treated with sodium bicarbonate and is brought to the desired sugar acid ratio by the addition of sugar to the neutralized juice. The method of neutralization has not yet been published.

D. Syruping

The term syrup is applied to the fruit juice which has been concentrated by the removal of water by evaporation or freezing or by the addition of 50 per cent or more of cane sugar and which contains 33 1/3 per cent of pure fruit juice (1).

Fruit juice syrups have been in use for some time but systematic study and their preparation in this country seems to have started very recently. From the literature it appears that Cruess (16) was the first who stressed the utilization of the surplus fruits in this way.
Irish (32) made some investigations on making orange syrup by the addition of sugar and he concluded that citrus juices when preserved by heat pasteurization, do not retain their fresh flavor satisfactorily. By the addition of heavy sugar or concentration before pasteurization, the fresh flavor is retained. He also found that the use of hard water in the making of syrup caused cloudiness due to the precipitation of the minerals of the water with the constituents of the syrup. He recommended the use of distilled water or the use of a water softener like zeolite. In his work he used 100 gallons of orange juice with 700 pounds of sugar. He recommended the dilution of this syrup with four parts of water.

Extending the work of Irish, Cruess and Irish (21) found that the addition of one part lemon juice with four parts orange juice resulted in a better syrup. They made 40° and 50° balling syrups by adding cane sugar. They bottled the syrup and pasteurized the 40 Brix at 165°F for 30 minutes, while the 50 Brix was pasteurized at 175°F for 30 minutes. They also found that the use of sodium benzoate at the rate of one-tenth of one per cent would keep the syrup in a fresher flavor than the preservation by heat.

Cruess and Irish (22) made further research on the syruping of the juices of strawberry, loganberry, etc and
found that bottled syrups would keep their flavor better than the canned syrups. They also recommended that heating in sealed containers was the best method for the preservation of the syrups. They confirmed the findings of Irish that tap water (they likely mean hard tap water) caused flocculent deposits in orange syrup. Their recommendation for the 65 Brix orange syrup was 30 minutes at 175°F in case the syrup was not carbonated. If the syrup was carbonated then 150°F for 30 minutes was sufficient.

Tressler et al (68) recommend the proper pasteurization of the juice used for syrup and the use of pectin or other stabilizers to keep colloidal particles in suspension. Blumenthal (5, 6) suggested that the suspension in the syrups can be kept best if use is made of Viscogum (trade name for a pectin containing stabilizer). Preichett and Stevens (52) suggest the use of 300 to 500 ppm of sodium polyphosphate to be added to base syrup.

Joslyn and Marsh (37) have suggested that browning of the syrup is due to the oxidation of the juice at the start. The primary product of oxidation is supposed to undergo further condensation reaction, probably of amino-acid sugar reaction which may be responsible for the darkening of the syrup and the juices. These authors recommend the use of antioxidants like sulfur dioxide etc.

Charley (11, 12, 13) has made extensive study of the
manufacturing of syrups from different fruit juices. His conclusions are that addition of too much sugar to the juice will make it so sweet that the flavor of the final beverage will be lost, due to the excessive dilution necessary when it is used. On the other hand, too little addition of sugar to the juice will result in the loss of flavor and color on prolonged storage. He preferred the addition of 50 per cent sugar to strawberry juice rather than 30 per cent, and sulfur dioxide as a preservative rather than sodium benzoate. His objection to the use of sodium benzoate was that it does not stop oxidation while sulfur dioxide does.
CHAPTER III
EXPERIMENTAL

A. Material and General Procedure

The bitter oranges used in this experiment were imported from the Riverside Experimental Station of the University of California. They were received April 7, 1948 and were placed immediately, in their original shipping cases, in cold storage, average temperature 32.5°F and relative humidity approximately 90 to 95 per cent.

The total weight of the oranges was 250 pounds and as reported by Dr. E. R. Parker, Horticulturist of the said experimental station, the variety was "Standard" budded on rough lemon.

For the purpose of extraction of the juice for analysis, 10 fruits were taken at random, weighed, de-oiled by washing in 180° to 185°F water for two to three minutes. The de-oiled fruit was then washed with cold water and dried with paper.

The fruit was cut with a stainless steel knife. For the first few days the juice was extracted with an electrically operated hand-fed juice extractor of the burring type. Since the juice extracted by this machine was found to be gummy, and burring caused the scooping of the albedo, it was replaced by a hand operated juice press.
The extracted juice was strained through three-fold cheesecloth and measured to calculate yield ratio.

B. Analytical Methods

The vitamin C content was determined by the use of a Fisher electro-photometer. The method used was the Oregon State College modified method, as follows: 15 mg of 2 to 6 dichlorobenzene indophenol was dissolved in 500 ml of distilled water. This dye, when diluted with an equal volume of buffer, would read approximately 75 degrees on the photometer, with B 525 m/u filter. Throughout the experiment the dye was used only for one week and then discarded. This standard stock dye was kept in cold storage.

The three per cent metaphosphoric acid used as an inhibitor for blending the juice in this experiment was prepared by dissolving 30 gr of the chemical in 1000 ml of water. This solution was kept in cold storage.

The buffer solution used in the determination of vitamin C was prepared by dissolving 96 gr of metaphosphoric acid (sticks) in three liters of distilled water. Thirty-five gr of sodium hydroxide was dissolved in one liter of distilled water and to this was added 92 gr of citric acid crystals. The two solutions were mixed and the solution adjusted to pH 3.6 by the use of sodium hydroxide or acetic acid.
To run a vitamin assay 10 ml of juice was blended in an electrically operated Waring blender, with 50 ml of three per cent metaphosphoric acid for about one minute. The solution was then filtered through folded filter paper, discarding the first 10 ml of the filtrate.

The dial of the photometer was adjusted to zero by testing with distilled water, then 10 ml of the sodium citrate metaphosphoric acid buffer (already mentioned) was placed in a well cleaned photometric tube. To this was added one ml of distilled water and 10 ml of the dye was run through a rapid delivery pipette. The dye was then decolorized by a crystal of ascorbic acid and the reading taken again. The readings thus obtained were designated $D_1$ and $D_2$.

Similar procedure was followed using one ml of the blended filtered juice, in place of water. The two readings were designated $X_1$ and $X_2$.

Using the formula $(D_1 - D_2) - (X_1 - X_2)$ the value obtained was multiplied by the factor 0.00258, the factor for the B 525 m/u filter used in the photometer. Usually three determinations were made of the same aliquot and an average figure used.

The acidity of the juice was determined as done by Moore et al (48). Five ml of the juice was taken in a 125 ml Erlenmeyer flask. To this was added 25 ml of distilled
water and the mixture was shaken. The acidity was determined as anhydrous citric acid by titrating against standard sodium hydroxide, which had been standardized against potassium acid phthalate, which met the United States Bureau of Standards requirements. The procedure for standardization of the sodium hydroxide was as described in A.O.A.C., Sixth edition, page 803 (49). Usually three determinations were made and the average taken. The soluble solids were determined by the laboratory hand refractometer, made by Bausch and Lomb Optical Company. The range of this refractometer was from zero to 60° Brix. The readings were adjusted to the temperature of the thermometer attached to the refractometer.

A Beckman glass electrode pH meter was used to determine the pH in all samples.

C. De-acidification

For the partial de-acidification of the juice to certain pH, sodium carbonate (purified anhydrous) was used. For this purpose a measured volume of juice was taken in a beaker and the glass electrodes of the pH meter were dipped in the juice. Dry sodium carbonate was gradually stirred in until the desired pH was obtained. The difference between the first weight of the sodium carbonate and that after the desired pH was obtained, was recorded as the
weight required for the de-acidification. The residual acidity in the partially neutralized juice of the desired pH and the vitamin C value of this juice were obtained by methods previously described.

D. Syruping

For the purpose of making syrup the juice was weighed, its soluble solid determined by the refractometer, and the required amount of sugar added in the cold juice to bring it to the desired soluble solids content. Sugar was dissolved by stirring with a glass rod. All the syrups were made by the addition of pure cane sugar in this experiment. It was assumed throughout that the sugar content was approximately equal to that of the soluble solids as determined by the refractometer.

E. Canning and Bottling

The cans used were half pounds, "C" enamel, diameter 3 7/16 inches, and height 2 1/4 inches. All the cans were filled with cold juice or cold syrups and vacuum sealed.

The bottles used were one-half pint, screw capped. They were filled with cold juice, keeping a little space for the expansion of juice or syrup.

Both the canned and the bottled products were
pasteurized for 20 minutes in water heated in a steam jacketed kettle to 180°F. The cooling was done gradually by addition of lukewarm water and later cold water so that the bottles would not break due to the sudden change of temperature.

Samples were kept at room temperature until further examination.
CHAPTER IV
RESULTS AND DISCUSSION

A. Behavior of the Oranges in Storage

It will be mentioned again that although the title of the thesis does not cover the storage study of oranges, this part of the work was undertaken for several reasons. Little work seemed to have been done on the storage of this species of orange. Every industry, compelled by unavoidable circumstances has to store the raw products until they can be processed. The inclusion of this part of the work in the problem, therefore, becomes obvious and worth consideration.

1. Cold Storage

The cold storage study of the oranges in question started April 7, 1948 when the fruit was placed in cold storage, and continued up to May 28, 1948 when the last of the fruit was utilized. During this period of time both the chemical as well as physical changes in the juice and the fruit were taken into consideration. For the chemical changes the chemical analysis for vitamin C, acidity, pH, and total soluble solids in the juice were taken into account. In the physical changes of the fruit the loss of weight, changes in the texture and the attack of diseases,
like molds, were studied.

Table I gives a complete record of the chemical analysis of the juice extracted from the fruit from time to time. Considerable variation in vitamin C and in acidity was found while the pH and the soluble solids varied very little throughout the storage study. The highest vitamin C was the sample analyzed on April 17, when 46 mg/100 ml was found. The highest acidity observed was 4.24 per cent (anhydrous citric acid). The average of all the figures for vitamin C, acidity, pH and the total soluble solids are given in the table.

As far as the physical changes are concerned, the writer did not see any change in volume, nor any shrivelled fruit due to desiccation for the period of storage. For a period of one and one-half months no attack of any fungus was seen. After that mold colonies were observed round about the stem end of the oranges. Only two oranges were found badly attacked by the green mold at the end of the storage. No rind breakdown was observed for one and one-half months but in the last few days the disorder was found very pronounced in the oranges that had thin rinds and that had comparatively smooth rinds. Oranges having a yellowish tinge, seemingly on the under mature side, did not suffer from the rind breakdown, nor did the ones that had coarse and thick skin.
### TABLE I

<table>
<thead>
<tr>
<th>Date</th>
<th>No. of Strained Fruits</th>
<th>Vol. of Fruit Juice</th>
<th>% Acidity as Citric Acid</th>
<th>Vit. C</th>
<th>Refractometer Reading mg/100 ml</th>
<th>pH</th>
<th>Weight of Fruits</th>
<th>Reading of Refractometer</th>
<th>Weight of Fruits</th>
<th>Reading of Refractometer</th>
</tr>
</thead>
<tbody>
<tr>
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<td>10</td>
<td>3-7</td>
<td>180</td>
<td>2.75</td>
<td>3.2</td>
<td>37.9</td>
<td>8.0</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>10</td>
<td>10</td>
<td>3-8</td>
<td>185</td>
<td>2.74</td>
<td>3.2</td>
<td>38.7</td>
<td>8.5</td>
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<tr>
<td>13</td>
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<td>3-10</td>
<td>187</td>
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<td>40.2</td>
<td>9.0</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>14</td>
<td>10</td>
<td>3-6</td>
<td>184</td>
<td>2.75</td>
<td>3.8</td>
<td>39.5</td>
<td>9.0</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>15</td>
<td>10</td>
<td>3-4</td>
<td>175</td>
<td>2.75</td>
<td>3.64</td>
<td>40.24</td>
<td>8.5</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>17</td>
<td>10</td>
<td>3-7</td>
<td>182</td>
<td>2.7</td>
<td>4.24</td>
<td>46.4</td>
<td>8.5</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>May  18</td>
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<td>3-4</td>
<td>181</td>
<td>2.72</td>
<td>4.0</td>
<td>43.3</td>
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<td>26</td>
<td>10</td>
<td>2-14</td>
<td>178</td>
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<td>3.65</td>
<td>35.6</td>
<td>9.0</td>
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<td>27</td>
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<td>2-10</td>
<td>174</td>
<td>2.7</td>
<td>3.5</td>
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<td></td>
</tr>
<tr>
<td>28</td>
<td>10</td>
<td>3-2</td>
<td>190</td>
<td>2.7</td>
<td>3.8</td>
<td>44.9</td>
<td>9.2</td>
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<td></td>
</tr>
<tr>
<td>Avg.</td>
<td>10</td>
<td>3-4</td>
<td>181.6</td>
<td>2.71</td>
<td>3.68</td>
<td>40.15</td>
<td>8.74</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

2. **Changes at Room Temperature**

One box of oranges from the lot was removed from the cold storage on April 19 and kept at room temperature. This was to observe what changes may take place in oranges when taken from cold storage and kept at room temperature. Chemical analysis of the juice from the fruit was made periodically. Observations of the texture of the fruit,
changes of volume and the attack of mold were recorded.

Table II gives detailed chemical information on the juice of the fruit kept at room temperature. It will be seen that no appreciable changes occurred as far as vitamin C, acidity, and the soluble solids are concerned. A noticeable drop from 2.7 to 2.45 in pH was found after two days of storage at room temperature. The experimenter was unable to explain the reasons for this drop.

### TABLE II

**ANALYSIS OF RAW BITTER ORANGE JUICE FROM FRUITS STORED AT ROOM TEMPERATURE**

<table>
<thead>
<tr>
<th>Date</th>
<th>No. of fruits used</th>
<th>Weight in lb oz</th>
<th>Vol. of strained juice in cc</th>
<th>% Acidity as citric acid mg/100 ml</th>
<th>Refractometer reading pH (Anhydrous) ml</th>
<th>Vit C reading</th>
<th>Avg.</th>
</tr>
</thead>
<tbody>
<tr>
<td>April</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>19</td>
<td>10</td>
<td>3-7</td>
<td>180</td>
<td>2.7</td>
<td>4.0</td>
<td>41.7</td>
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<td>4.09</td>
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<td>10</td>
<td>3-1</td>
<td>180</td>
<td>2.45</td>
<td>3.91</td>
<td>41.0</td>
<td>9.0</td>
</tr>
<tr>
<td>24</td>
<td>10</td>
<td>2-14</td>
<td>188</td>
<td>2.46</td>
<td>3.89</td>
<td>37.7</td>
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<td>27</td>
<td>10</td>
<td>2-13</td>
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<td>2.45</td>
<td>3.91</td>
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<td>9.0</td>
</tr>
<tr>
<td>Avg.</td>
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<td>3-4</td>
<td>190</td>
<td>2.54</td>
<td>3.95</td>
<td>40.4</td>
<td>9.0</td>
</tr>
</tbody>
</table>

During storage there was some loss of weight but the volume of juice was not much affected. Shrivelling of the rind was observed after two days and the attack of mold.
increased as storage progressed. Green mold was the common trouble.

To find the extent of loss in weight at room temperature, 20 oranges were individually weighed from the lot of the cold storage and kept at room temperature. They were again weighed after 15 days. Table III gives the loss which occurred between the two weighing periods. In this length of time two oranges completely rotted. All of the other damages were attacks by molds, some slightly, but others considerably attacked. The oranges had become shrivelled badly and when cut the pulp had contracted from the rind. Juice extraction was difficult and the yield small. Here again the thin-skinned smooth-rind oranges were found more shrivelled than the thick-skinned coarse-rind ones.

Discussion:

In the literature various investigators have given different ranges for the vitamin C content of bitter oranges. Thus Bacharach et al (2) analyzing bitter orange juice (Seville orange) on December 21 and again on January 30, reported the range of vitamin C content to be 36 to 56 mg/100 ml and 22 to 46 mg/100 ml respectively. He attributed the variation of vitamin C content in oranges to climatic conditions, seasonal variation, sunshine, rainfall,
TABLE III
LOSS OF WEIGHT OF BITTER ORANGES STORED AT ROOM TEMPERATURE FOR A PERIOD OF 15 DAYS

<table>
<thead>
<tr>
<th>Date stored</th>
<th>Weight of fruit in gm</th>
<th>Date weighed</th>
<th>Weight of fruit in gm</th>
</tr>
</thead>
<tbody>
<tr>
<td>May 27</td>
<td>116.0</td>
<td>June 11</td>
<td>99.5</td>
</tr>
<tr>
<td></td>
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<td></td>
<td>103.4</td>
</tr>
<tr>
<td></td>
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</tr>
<tr>
<td></td>
<td>129.5</td>
<td></td>
<td>109.5</td>
</tr>
<tr>
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<td>133.2</td>
<td></td>
<td>113.5</td>
</tr>
<tr>
<td>Average</td>
<td>132.6</td>
<td>Average</td>
<td>114.6</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Percentage Loss</td>
<td>13.5</td>
</tr>
</tbody>
</table>

humidity, variety, maturity, cultural practices, and size of the fruit. Sergree (58) reported the average vitamin C content in bitter oranges to be 48.5 mg/100 ml. He states that larger size oranges have more vitamin C than the smaller ones. Miller and Winston (47) reported that as the fruit matured the ascorbic acid decreased and the carotinoids increased. Harding and Thomas (27) working on grapefruit found that vitamin C content in that fruit was more
when the fruit was grown directly in the sunshine, and when
the stock used was that of rough lemon, as compared to the
fruit grown in the shade and when budded on the sour orange
stock.

Worsely (74) has reported vitamin C content of bitter
oranges from 35 to 45 mg/100 ml and the results of the
writer are in close agreement with this worker. Ross (56)
and Abbott (l) have reported correlation between acidity and
vitamin C in oranges. Bacharach et al (2) found no core-
lation between pH and the vitamin C content in orange juice
and the findings of the author are supporting this state-
ment.

The results obtained by the writer further agree with
the results obtained by Stahl and Camp (61) that oranges
undergo very little change in cold storage for a period of
two months as far as vitamin C, pH, acidity and soluble
solids are concerned.

B. Preparation and Testing of Partially
De-acidified Sweetened Juice

The raw juice of the bitter orange, due to the high
acidity and low sugar-acid ratio, is very sour and unpalat-
table. To make it a palatable juice, it was decided to
partially de-acidify it and sweeten it with sugar to the
desired sugar-acid ratio. Higby (29) has said that
limonin (the bitter principle of the Valentia orange) and isolimonin (the bitter principle of the Navel orange) are changed to tasteless compounds by the addition of alkali. Apparently any alkali used to reduce the acidity of bitter orange juice would also reduce the bitterness.

Sodium carbonate was selected for the de-acidification or partial neutralization of the juice because it was inexpensive and was available in the pure form.

1. **Determination of pH Range Most Acceptable for Bitter Orange Juice**

For the partial neutralization of the acid, the pH, rather than the final acidity of the juice was selected as the important measurement. This was done because pH is known to be the important factor in determining the time required for the sterilization of fruit juices. The buffering capacity of the juice was not known and hence the addition of the calculated amount of sodium carbonate to reduce the acidity to the desired point was not possible. Levels of 3.5, 4.0 and 4.5 pH were chosen as practical. Juice was adjusted to these levels to study the effect which the addition of alkali might have on the stability of vitamin C, the color and the acidity.

Preliminary tests showed that when pH was brought up to 4.5 there was a detectable taste of alkali in the juice.
This level was discarded in the subsequent trials.

For the effect on vitamin C actual titrations were made. Tables IV and V give the detailed information in this respect. It will be seen from these tables that there is very little effect on the vitamin C of the juice, but the unfortunate situation is that very little acidity is reduced. In the opinion of the writer, in the juice of this species of orange, there are some easily oxidizable substances other than vitamin C which may be responsible for the stability of the vitamin C in the juice. This has been explained by Sherman (60), who states:

"....It is also to be remembered that oxidation hazards are not all from without; for if cabbage juice (to mention a single instance) is carefully acidified to the same pH as tomato juice, and the two are heated with the same precautions in all the respects above mentioned, the vitamin C value will still fall more rapidly in juice of cabbage than in that of tomato, because of higher oxidation potential than in the tomatoes."

It was seen that the addition of the sodium carbonate improved the color of the juice. The original color of the juice of the bitter orange was yellowish-orange but the addition of the salt made it more orange than the original.
TABLE IV
ANALYSIS OF THE RAW JUICE OF BITTER ORANGES

<table>
<thead>
<tr>
<th>Date</th>
<th>Juice taken in cc</th>
<th>Na₂CO₃ used in gm</th>
<th>pH</th>
<th>% Acidity as citric acid (Anhydrous)</th>
<th>Vitamin C mg/100 ml</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Before Neutralization</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>April 14</td>
<td>170</td>
<td>none</td>
<td>2.75</td>
<td>3.8</td>
<td>39.5</td>
</tr>
<tr>
<td>15</td>
<td>120</td>
<td>none</td>
<td>2.75</td>
<td>3.64</td>
<td>40.24</td>
</tr>
<tr>
<td>17</td>
<td>120</td>
<td>none</td>
<td>2.70</td>
<td>4.24</td>
<td>46.40</td>
</tr>
<tr>
<td>19</td>
<td>120</td>
<td>none</td>
<td>2.70</td>
<td>4.00</td>
<td>41.70</td>
</tr>
<tr>
<td>27</td>
<td>120</td>
<td>none</td>
<td>2.45</td>
<td>3.91</td>
<td>43.30</td>
</tr>
<tr>
<td></td>
<td>After Neutralization to pH 3.5</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>April 14</td>
<td>170</td>
<td>0.9</td>
<td>3.5</td>
<td>3.2</td>
<td>39.3</td>
</tr>
<tr>
<td>15</td>
<td>120</td>
<td>0.6</td>
<td>3.5</td>
<td>3.14</td>
<td>---</td>
</tr>
<tr>
<td>17</td>
<td>120</td>
<td>0.613</td>
<td>3.5</td>
<td>3.40</td>
<td>43.3</td>
</tr>
<tr>
<td>19</td>
<td>120</td>
<td>0.611</td>
<td>3.5</td>
<td>3.36</td>
<td>37.9</td>
</tr>
<tr>
<td>27</td>
<td>120</td>
<td>1.038</td>
<td>3.5</td>
<td>2.96</td>
<td>42.5</td>
</tr>
</tbody>
</table>

2. Determination of Desirable Sugar-Acid Ratio

Since reduction of acidity by use of alkali did not reduce it to an acceptable level it was necessary to examine other measures of adjusting acidity to a palatable level. This might be done by adjustment of its sugar-acid
### TABLE V

**ANALYSIS OF THE RAW JUICE OF BITTER ORANGES**

<table>
<thead>
<tr>
<th>Date</th>
<th>Juice taken in cc</th>
<th>Na$_2$CO$_3$ used in gm</th>
<th>pH</th>
<th>% Acidity as citric acid (Anhydrous)</th>
<th>Vitamin C mg/100 ml</th>
</tr>
</thead>
<tbody>
<tr>
<td>Before Neutralization</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>April</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>21</td>
<td>100</td>
<td>none</td>
<td>2.70</td>
<td>4.09</td>
<td>40.2</td>
</tr>
<tr>
<td>22</td>
<td>100</td>
<td>none</td>
<td>2.45</td>
<td>3.90</td>
<td>38.7</td>
</tr>
<tr>
<td>23</td>
<td>100</td>
<td>none</td>
<td>2.45</td>
<td>3.91</td>
<td>41.0</td>
</tr>
<tr>
<td>After Neutralization to pH 4.0</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>April</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>21</td>
<td>100</td>
<td>1.7</td>
<td>4.10</td>
<td>2.10</td>
<td>40.0</td>
</tr>
<tr>
<td>22</td>
<td>100</td>
<td>1.65</td>
<td>4.00</td>
<td>2.10</td>
<td>37.0</td>
</tr>
<tr>
<td>23</td>
<td>100</td>
<td>1.65</td>
<td>4.00</td>
<td>2.10</td>
<td>40.2</td>
</tr>
</tbody>
</table>

To determine the acceptable sugar-acid ratio levels, two samples of commercial orange juice were analyzed for sugar-acid ratio. Table VI gives the results of the two juices. It will be seen that the sugar-acid ratio in both these juices is 11:1. Both juices were sweetened with sugar.

Since citrus juices of 8:1 sugar-acid ratio are also sold in the market (64) it was thought advisable to make some juices with this low sugar-acid ratio. Juices of 8:1, 9:1, 10:1, 11:1 and 12:1, were prepared from the partially
## TABLE VI

**ANALYSIS OF COMMERCIAL JUICES BOUGHT FROM CORVALLIS MARKET**

<table>
<thead>
<tr>
<th>Name and type of juice</th>
<th>% Acidity as citric acid</th>
<th>Sugar-acid ratio</th>
<th>pH</th>
</tr>
</thead>
<tbody>
<tr>
<td>Standby grapefruit-orange blended juice, sugar added</td>
<td>10.00</td>
<td>0.91</td>
<td>11:1</td>
</tr>
<tr>
<td>Delmonte orange juice, sugar added</td>
<td>11.00</td>
<td>1.03</td>
<td>11:1</td>
</tr>
</tbody>
</table>

All samples were #2 plain cans.

de-acidified juices of pH 3.5, and pH 4.0. They were taste-tested. All tasters preferred the juices prepared from the juice of pH 3.5.

From the pH 3.5 juices of different sugar-acid ratio the one of 10:1 sugar-acid ratio was preferred by the majority while other tasters gave preference to 9:1 sugar-acid ratio. Giving value to the majority, the 10:1 sugar-acid was decided to be tried further.

Discussion of the reduction of acidity by dilution method will be found on page 50.

### 3. Comparison of Canned Juice Versus Freshly Prepared Juices

To see if processing by heat had any detectable and objectionable effect on the juice partially neutralized to
pH 3.5 and sweetened to 10:1 sugar-acid ratio, organoleptic tests were arranged. To compare with the canned juice, freshly prepared juices of pH 3.5, 3.8, and 4.1, all of 10:1 sugar-acid ratio, were included. The canned juice had been held at room temperature for a period of eight days. This period of eight days storage at room temperature was considered to be sufficient for any chemical changes which might take place due to heat processing.

The juices were tasted by six judges and most of them were renowned for their taste ability. That heat processing had some effect on flavor of the juice was shown by the fact that two members were able to distinguish the canned juice of pH 3.5 from freshly prepared juice of pH 3.5 and gave their opinion in favor of the fresh juice of the same pH. The other four members gave preference to the canned juice of pH 3.5.

Of the juice of pH 3.8 and 4.1 all gave their opinion in favor of the former.

The conclusion drawn is that there is some slight change in flavor due to heat processing and eight days storage at room temperature, but the change was not objectionable and it was even preferred by many. Another conclusion was that heat processing had no effect on color of the juice for the period of storage.
C. Preparation and Testing of a Syrup of Bitter Orange Juice

The adjustment of acidity of bitter orange juice by the dilution method requires the addition of sugar to make an acceptably balanced beverage. Highly acid juices such as pomegranate, grape, berry and citrus are often made into syrups by a large addition of sugar. This may even be canned at the point when the sugar acts largely as the preservative but normally heat is the preservative of syrups. When the amount of sugar is correct only water needs to be added to prepare a fine acceptably balanced beverage.

Sixty Brix fruit juice syrup was prepared from bitter orange juice by the addition of sugar. Beverages were prepared from this syrup by dilution with various amounts of water. The dilution of 4:1 was preferred by tasters but when it was put to taste, not in comparison with the other dilutions, some contended that the sweetness had masked the orange flavor.

It was thought that the 50 Brix syrup with a dilution of 4:1 might prove satisfactory. When put to taste again some individuals preferred the 60 Brix dilution of 4:1. The majority of the tasters preferred the 50 Brix syrup when diluted with four parts of water.
1. **Comparison of 50 Brix Bottled Syrup with 50 Brix Freshly Prepared Syrup**

The bottled syrup was prepared and pasteurized for 20 minutes at 180°F. It was kept at room temperature for ten days before dilution and tasting. Fresh bitter orange juice syrup was prepared about an hour before the syrups were put to taste. The dilution of the syrups was 4:1 (four parts water and one part 50 Brix syrup). Duplicates of both the bottled and the freshly made syrups were made to test the accuracy of the tasters. They were requested to express their opinion on the general acceptability of the beverages thus made.

The opinions of the judges varied considerably. Two of the five tasters could not accurately designate the duplicates. Two members were able to select the freshly prepared beverage but they did not raise any objection to the bottled beverage. One member preferred the bottled.

When analyzed, the bottled beverage had 16:1 sugar-acid ratio and the pH was found to be 2.8. The freshly prepared beverage had 17:1 sugar-acid ratio with the same pH. The difference in the sugar-acid ratio may be explained due to the variation of acidity in the oranges. It was of interest to find that the dilution did not change the pH to a great extent. The original pH of bitter orange juice may be safely taken to be 2.6 to 2.7.
2. Comparison of the 60 Brix Canned Syrup with 60 Brix Freshly Prepared Syrup

The object of this test was to learn whether canning had any effect on the quality and the general acceptability of the beverage made from canned juices. The canned syrup was prepared eight days before tasting and held at room temperature. The cans were vacuum sealed, pasteurized at 180°F for 20 minutes, and cooled under tap water.

The freshly prepared syrup was made from the freshly extracted juice about an hour before the tasting. Dilution in both cases was 4:1. Duplicates of both the canned and the freshly prepared beverages were offered the tasters to check their decisions.

Four tasters tested the beverages. Two tasters preferred the beverage made from the canned syrup and two liked the beverage made from the freshly prepared syrup. No member was able to find any fault in the canned beverage as far as the general acceptability was concerned.

For the period of the short storage of eight to nine days no appreciable difference was found between the processed and the freshly prepared syrups. Quality of the syrup was as well retained by canning as by bottling. A word of caution can be given that this conclusion was based on a very short storage period. Longer storage would have been necessary for further investigation on the subject.
The syrup density to be recommended will depend largely upon the people for which the syrup or beverage is to be prepared. Further it depends upon the initial acidity of the bitter orange juice. It can be said, however, that sugar-acid ratio of about 16 or 18 to one has been found to be suitable and desirable for bitter orange juice. The beverage resembles lemonade and many tasters have expressed the similarity.

Acceptability of Different Citrus Beverages:

To find out how well the partially neutralized sweetened bitter orange juice of pH 3.5 compared with commercial citrus juices, grapefruit and orange blended juice, with added sugar, was selected. Also included in the test were the 60 Brix canned and bottled syrups. The dilution in the case of the syrup was 4:1. Sugar-acid ratio of the juice was, of course, 10:1.

The tasters were four staff members whose tasting ability was well known. Two of them preferred the beverage made from the bottled syrup. The other two preferred the neutralized juice made from the bitter orange juice. It was interesting that no one preferred the commercial juice. This was because the naringin (bitter principle) in the grapefruit juice is not liked by many persons. The only objection of the tasters to the bitter orange beverage was
that it was too sweet.

D. Bottling Versus Canning

In the beverage industry bottles are used exclusively while, in the juice industry canning of the juice is the common practice. A small scale experiment was conducted to learn whether use of different containers had any material effect on the vitamin C content of the juice or the syrups of bitter oranges.

The cans were vacuum sealed while the bottles were cold packed and the two containers were given the same subsequent treatment of sterilization.

Both the juice and the syrups, were analyzed for vitamin C and pH before and after the processing. The results are tabulated and given in Table VII.

It shows that there is some difference in the vitamin C content of the two types of containers but the difference does not seem to be of any material importance. It further shows that the use of more sugar does not seem to have any additional preservative effect on the vitamin C content.

The samples analyzed were not sufficient in number to lead one to form a definite conclusion. The data give some idea of retention of vitamin C in the case of both types of containers.
<table>
<thead>
<tr>
<th>No. of samples used</th>
<th>Container used</th>
<th>Juice or syrup</th>
<th>Before canning</th>
<th>After canning</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>Vit. C</td>
<td>pH</td>
</tr>
<tr>
<td>3</td>
<td>can</td>
<td>syrup</td>
<td>33.2</td>
<td>2.7</td>
</tr>
<tr>
<td></td>
<td>bottle</td>
<td>syrup</td>
<td>33.2</td>
<td>2.7</td>
</tr>
<tr>
<td>2</td>
<td>can</td>
<td>syrup</td>
<td>30.18</td>
<td>2.5</td>
</tr>
<tr>
<td></td>
<td>bottle</td>
<td>syrup</td>
<td>30.18</td>
<td>2.5</td>
</tr>
<tr>
<td>2</td>
<td>can</td>
<td>juice</td>
<td>37.0</td>
<td>3.5</td>
</tr>
<tr>
<td>1</td>
<td>bottle</td>
<td>juice</td>
<td>37.0</td>
<td>3.5</td>
</tr>
</tbody>
</table>
CHAPTER V
SUMMARY AND CONCLUSIONS

In the Northwest Frontier Province (Dominion of Pakistan), bitter orange (C. Aurantium) trees are grown around the orchards, in the municipal parks and other private and public places to serve as windbreaks and as ornamental plants.

In the United States and throughout the world the seed obtained from bitter oranges is used by nurserymen for raising seedlings to be used as stock for sweet oranges. The whole fruit has very little market value due to the sour and bitter taste of the juice. The increased planting of orchards will result in simultaneous increase of the planting of this species of oranges in the province.

This project was therefore undertaken to see if the juice of the fruit might be canned. Along with this project the study of the behavior of the oranges in storage was included.

On arrival from the Riverside Experimental Station the oranges were placed in cold storage, with average temperature of 32°F and relative humidity of 92 to 95 percent. The cold storage period of the oranges lasted for 51 days. During the period of study chemical, physical and physiological changes in the fruit juice and the fruit were taken into consideration.
The chemical analysis of the juice consisted of determination of vitamin C, acidity, pH, and the total soluble solids. The physical and physiological changes studied were the loss of weight, the change in shape, the attack of molds and the change in texture of the fruit.

Results obtained indicate that very little change occurs in the chemical composition of the juice of the fruit when kept in cold storage at the above mentioned temperature and humidity. Different samples showed a wide range of variation in vitamin C content and acidity. The highest vitamin C value was 46.4 and the lowest figure was 34.8 mg/100 ml. The average of 10 determinations came to 40.15 mg/100 ml. The range of acidity was found to be from 4.2 to 3.2 per cent (expressed as anhydrous citric acid) with an average figure of 3.68 per cent. Practically speaking the pH and the total soluble solids did not change very much. The average figures for the pH and the soluble solids were 2.7 and 8.7 respectively.

As to the physical and physiological changes in cold storage, molds were noticed after a period of one month and a half of storage. The first attack was confined to the stem end which was found spreading gradually to the sides of the fruit. Rind breakdown was observed in the last few samples. This attack was noticed on the fruits having smooth and thin skin. The affected fruits were found to be
pulpy, soft, and with depressions in the rind on the affected side. Fruits having rough surface and thick skin and on the under side of maturity did not suffer from this trouble.

The effect of the room temperature on the fruit was studied too, after 18 days in cold storage.

In the short period of eight days, the writer did not see any appreciable change in the vitamin C content, acidity, and total soluble solids. The only change observed was in pH which dropped from 2.7 to 2.45.

As to the physical and physiological changes in the fruit, mold attack was found progressing. The last samples were found shrivelled and soft and they had lost 13.5 percent in weight. The extraction of the juice from the fresh fruit was found to be easier as compared to fruit from cold storage.

In the preparation of juice partial de-acidification was brought about by the addition of sodium carbonate to the desired pH. Different pH like 3.5, 4.0, and 4.5 were tried. Neutralization of the juice to pH 4.5 gave a foreign flavor. In the partially de-acidified juices of pH 3.5 and 4.0 several sugar-acid ratios, namely, 8:1, 9:1, 10:1, 11:1 and 12:1 were tried. Many organoleptic taste panels were arranged. Juice of pH 3.5 with sugar-acid ratio 10:1 was found to be the most desirable and acceptable
but the exact ratio would depend upon the purpose and circumstances of use. The cans and bottles were sterilized at 180°F for 20 minutes. The processed juice, after storing at room temperature for a week, was compared with the freshly prepared juice of the same pH and same sugar-acid ratio. Tasters could not make an easy distinction between the freshly prepared and the processed juice. Nor could they find any distinctive difference in the flavor between the canned and the bottled juice.

Comparison of bitter orange juice, prepared as mentioned above, was made with the commercial grapefruit-orange juice blend with added sugar. Tasters preferred the bitter orange sweetened juice over the grapefruit-orange samples purchased.

Bitter orange syrup was prepared by the addition of cane sugar to the juice. Both 60 and 50 Brix syrups were given trial. Many dilutions were tried, and the prepared beverage was subjected to organoleptic taste panels. The dilution of one part of syrup with four parts of water was preferred. The 50 Brix syrup with dilution of 4:1 was the most commonly agreed upon. Analysis of the beverages have shown that the sugar-acid ratio of 16 to 18:1 is commonly agreeable to the majority of the tasters.

The syrups were canned and bottled and sterilized at 180°F for 20 minutes. After a short storage of eight days
at room temperature, the canned and the bottled syrups were compared with freshly prepared syrups. Dilution of the syrups was 4:1. Members of the taste panels could not find any objectionable flavor in the beverages made from canned or bottled syrups.

To find the vitamin C retention in the canned as well as in the bottled products analyses of the juice and the syrups before and after the processing were made. The canned product had retained slightly more vitamin C than the bottled one. It may be mentioned that the cans used were "C" enamel and were vacuum sealed. The bottles were cold packed and both the cans and the bottles were sterilized for 20 minutes at 180°F.

Conclusions:

1. Bitter oranges can be profitably stored at storage temperature of 32° to 35°F with relative humidity of 92 to 95 per cent for a period of one month and a half.

2. Storage at room temperature, after the fruit has been in cold storage, has been found to be uneconomical and wasteful. This is true if the fruit is to be kept at room temperature for more than a week.

3. Oranges with smooth surface and thin skin have been found to be more susceptible to rind breakdown than the rough surfaced and thick skinned ones. Similarly over
mature fruits are more susceptible to rind decay than the under mature oranges.

4. Good palatable juice can be prepared from the juice of bitter oranges when the acidity is neutralized to pH 3.5 by sodium carbonate and the sugar-acid ratio is adjusted to 10:1 by addition of sugar. Sterilization by heat does not seem to have any disagreeable effect on the flavor of the juice. Canning has been found as good as bottling.

5. A good palatable beverage, tasting like lemonade, can be prepared by making 50 or 60 Brix syrups and diluting one part of syrup with four parts of water. Heat sterilization of 180°F for 20 minutes has not been found to have any effect on the general acceptability of the products. It is true both for the canned and the bottled products.

6. With proper precautions of filling the bottles, "C" enamel cans, vacuum sealed, had but very slightly more vitamin C retention than the bottles.

7. Finally, all these deductions are drawn for a storage period of not more than seven to ten days. Storage behavior of the processed products needs further study.
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