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Improvements in the Manufacture and the Preservation of Grape Juice

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ABSTRACT

The results of studies on methods of preservation of grape juice, especially that made from Concord grapes, are presented. Attention is given particularly to methods of preservation by pasteurization, although temperature storage, freezing storage, germ-proof filtration, storage under carbon dioxide pressure, concentration of juice, and the use of chemical preservatives are briefly discussed.

The preparation of grape juice is described, noting particularly that the pasteurization both for carboy storage and for bottling may be carried out at 165° to 170°F if care is taken to remove the foam and suspended solids and to sterilize properly the corks as well as the containers.

The main causes of deterioration of grape juice are ascribed to the action of micro-organisms, the effect of enzymes, and the effect of air. Deterioration due to growth of micro-organisms or to enzyme action may be overcome by proper pasteurization and handling. Harmful effects due to air may be retarded considerably by eliminating air from the bottle and by proper storage of the juice. This is accomplished most easily by filling the bottles full at temperatures slightly above the pasteurization temperature.

Methods for removing argols or crude tartrates are presented with particular reference to rapid methods, such as the use of "Pectinol" clarification and freezing and thawing procedures.

Finally, the carbonation of grape juice is discussed describing briefly a method of carbonating by the use of dry ice.
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IMPROVEMENTS IN THE MANUFACTURE AND THE PRESERVATION OF GRAPE JUICE

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INTRODUCTION

Grape juice is one of the few fruit juices in which the flavoring substances are so well balanced that it is in itself a pleasing and refreshing drink. This is particularly true of juice prepared from Concord grapes or closely related varieties. Unfortunately, the methods of preparing grape juice are so little understood that many undesirable changes may take place prior to its consumption. Because of this many grapes are unused which might well be converted to unfermented grape juice. A study of the factors essential to the preparation of an attractive beverage which will not change in color, clarity, aroma, and flavor seemed of importance in increasing the utilization of grapes.

A large amount of the grape juice manufactured is prepared from Concord grapes or closely related varieties. The juice of Concord has that combination of qualities, such as color, aroma, flavor, and acidity, which makes a refreshing and thirst-quenching beverage. It has also that flavor so typical of native grape, an after-taste suggestive of tannin, a flavor which may be desirable if not too pronounced. The juice, if properly prepared, lends itself remarkably well to dilution with water or carbonated beverages or blending with other fruit juices, altho addition of sugar is usually necessary. Concord juice has become the most common grape juice not only because of these desirable qualities, but because of the productivity of the grapes. From 95 to 98 per cent of the commercial grape juice consumed is prepared from the Concord grape. New York is the leading state in production of grapes for unfermented grape juice. On the other hand, California and Arizona produce more grapes than all other states combined. Western grapes are ordinarily higher in sugar and lower in acid and therefore juices prepared from them are sweet and pleasing, but they do not have the distinctive flavor and refreshing qualities of New York grapes.

When these investigations on grape juice were started 6 years ago, the commercial product on the market was of varying quality. Some of the freshly pasteurized juice had a bright purplish red color
and a fine grape flavor and made a pleasing bottled beverage. On the other hand, much of the juice, especially if it had stood in the warehouse or upon grocers' shelves, did not present a particularly appetizing drink. It was thought that this defect accounted for the low consumption of this grape by-product and that a comprehensive study of the factors involved in the deterioration of bottled Concord grape juice would be well worth while. The results of these researches have been gratifying and have indicated methods which permit the storage of bottled juice for considerable periods of time without material loss in quality. Certain phases of these studies have already been discussed from a technical viewpoint,¹ and the present paper is a resumé and general discussion of the application of the results expanded to include certain additional studies and observations.

Altho the studies have dealt primarily with juice made from Concord grapes and, to a lesser extent, from Catawba grapes, many of the facts noted hold true for juice prepared from other varieties as well as for juices from other fruits.

METHODS OF PRESERVATION OF GRAPE JUICE

There are a number of methods used in the preservation of grape juice which may be summarized briefly, as follows:

1. Preservation by pasteurization is the common method used for unfermented juice. Since this is the method of preservation which was studied in greatest detail, the discussion will deal primarily with this method.

2. The juice may be preserved by holding in cool storage, that is, at a temperature of 28° to 30°F. The juice after pressing is chilled to 28° to 30° as rapidly as possible by passing thru a series of coolers. Altho at this time there may be from 10,000 to 20,000 living microorganisms per cc present in the juice, these die off fairly rapidly.² After from 3 to 6 months' storage, the number of micro-organisms may be reduced to below 100 per cc. Unfortunately, mold may develop to some extent at this low temperature so that it is necessary to keep air from contact with the juice. The argols are precipitated rapidly at this low temperature. The juice itself has a pleasing

¹Pederson, C. S. The preservation of grape juice: I. Pasteurization of Concord grape juice. Food Res., 1, 9-27. 1936.

²Pederson, C. S. The preservation of grape juice. III. Studies on the cool storage of grape juice. Food Res., 1, 301-305. 1936.

flavor, but it is essential to pasteurize it in bottling just as with regularly prepared juice.

3. Cold or freezing storage is used to some extent, particularly for juices that do not have a high tartrate content. It has many of the advantages of cool storage, but the disadvantage of causing too much sedimentation resulting in a clear juice without the desirable body so characteristic of Concord juice. Experiments conducted upon Concord juice indicate that it is essential to allow some of the tartrates to redissolve before bottling juice in order to compensate for the extra precipitation of the tartrates at the low temperature and to give the juice the normal Concord flavor.

4. Preservation by filtering thru a germ-proof filter such as the Seitz EK, is used to a certain extent. The use of this type of filter has been studied at this Station. A sparkling clear juice is produced, and if clarity is desired, this method has its merits as the organisms present are readily filtered out. Sufficient heating or pasteurization to inactivate the enzymes however, is necessary for retention of quality. Elimination of air is very essential since it has been noted that the clear juice obtained by Seitz filtration will show oxidative changes more rapidly than ordinary juice. By filling the bottles full, the clarity is retained for a fairly long period. This effect of oxidation upon grape juice is discussed more fully under pasteurization.

5. Storage of the juice under a high pressure of carbon dioxide gas has been used to some extent.

6. Concentration of the juice to such a degree that the total solids present are high enough to prevent the development of organisms has been used. Ordinarily, since mold may be a troublesome factor in spoilage of such concentrates, they should be bottled with little or no air present.

7. A certain quantity of juice is preserved by means of chemicals, particularly for the manufacture of grape by-products such as wine. Sulfur dioxide and sodium benzoate are the two materials most commonly used. The latter has little to be said in its favor, while the former is used for wine musts and much of it may be removed by aeration previous to fermentation. The use of chemical preservatives has also been found to affect the temperature required for pasteurization of the juice. Grape juice can be pasteurized at lower temperatures when even traces as low as 50 p.p.m. of sulfur dioxide or similarly small quantities of sodium benzoate are added to the juice. Sodium benzoate also has been found to increase the rate of killing of micro-organisms when juice is preserved by holding in cool storage.

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5Pederson, C. S. The preservation of grape juice: I. Pasteurization of Concord grape juice. Food Res., 1, 9-27. 1936.

6See footnote 2.
Altho all of these methods are in use to some extent, the following discussion will deal primarily with the first method, *viz.*, pasteurization, since it is that method which is in common use in the preparation of unfermented grape juice.

THE PREPARATION OF GRAPE JUICE

The quality of grape juice depends to a large extent upon the variety of grape used and upon the condition of the fruit when pressed for juice. Ordinarily, the sugar content increases and the acid decreases as the fruit ripens. It is essential, therefore, that the grapes be left on the vines until they are fully ripe. A Brix reading of the juice of 18° (Fig. 1) indicates that the grapes are ripe. The sugar content of Concord grapes may vary 4 per cent or more from year to year. Usually, in warm seasons with plenty of sunshine, the sugar content is higher and the acidity is lower than in colder seasons.

Preparing grape juices according to the usual commercial procedure involves three essential steps or processes. First, the juice is pressed from the grapes, pasteurized, and bottled or placed in carboys. This is done during the grape harvesting season. Second, the bottled or carboyed juice is stored to allow crystallization of argols or the crude tartrates and sedimentation of the solids. Third, the stored juice is freed from its sediment and then rebottled for consumption. Since these processes are more or less distinct, they will be treated separately in this discussion.

PRESSING, PASTEURIZATION, AND BOTTLING OF THE JUICE

There are two methods in common use in pressing grapes for juice, *viz.*, hot pressing and cold pressing. Hot pressing methods are more commonly used in the preparation of unfermented grape juice, particularly from Concord grapes. The heating of the grapes brings certain substances into solution, particularly pigments, tartrates, and tannin-like compounds. Cold pressing of grapes is used to a large extent in preparing juice for fermented beverages, particularly when wines of light color are desired.

When used in preparation of an unfermented beverage, the juice is sometimes consumed without the subsequent precipitation and repasteurization processes mentioned above. Juice pressed hot is favored by most consumers, and furthermore, this process gives a higher yield of juice.
HOT PRESSING OF JUICE

The fully ripened grapes are picked and allowed to stand a short time before pressing since it appears that this procedure mellows the juice. The grapes should not be crushed or bruised, since this not only allows the development of molds and yeast which multiply very quickly at ordinary temperatures, but it allows the natural enzymes of the fruit to produce decay more rapidly. Ordinarily, as grapes are harvested, the total number of organisms will seldom exceed 400,000 per cc. Grapes which have been bruised or crushed will show counts as high as 10,000,000 per cc within several hours. The micro-organisms, as well as the enzymes, will alter the flavor and quality of the juice and make sterilization more difficult.

The grapes are washed and stemmed, crushed, and heated to 140° to 145°F until the bright purple red color of the grape skins is diffused throughout the heated mass. The time and temperature used varies somewhat depending upon the quantity of pigment present in the grapes. Commercial packers try to prepare juice of uniform color and quality and therefore allow only experienced men to judge the amount of heating necessary. Heating may be accomplished within an open steam-jacketed kettle (Fig. 2), in preheaters (Fig. 3) or, if in small quantities, in an ordinary aluminum kettle. A large double

FIG. 1.—HYDROMETER AND CYLINDER USED FOR DETERMINING SPECIFIC GRAVITY OF GRAPE JUICE.

The instrument is graduated in a Brix scale which indicates the approximate total solids present in the juice. The juice from Concord grapes when picked should give a reading of about 18° Brix indicating approximately 16 per cent sugar. Apparatus may be obtained from any laboratory supply house.
boiler may be desirable for small quantities of juice. This heating, altho not high enough to kill all of the organisms present in the grapes, does result in the death of the large majority. The molds particularly survive this temperature, but these are comparatively few in number and do not multiply as rapidly as do the yeasts.

Hot pressing of grapes loosens or dissolves the jelly-like layer on the inner surface of the skins of the grapes, particularly of the Concord type. Certain other extractives are obtained so that if heated too long or at too high a temperature the juice takes on a harsh tan-
Fig. 3.—A Tubular Heater for Stemmed Grapes.

Steam circulates around the inside tubes heating the grapes to approximately 145°F. The temperature is controlled automatically.

nin-like flavor. Apparently some of this harshness comes from the seeds. The juice from the skins is highly colored and is high in acid, tannin, and minerals but is low in sugar. The proper extraction of the color results in a pleasant-appearing and highly flavored and colored product.

The heated grapes are pressed immediately. In factories, this is
usually accomplished by large hydraulic presses (Fig. 4), the grapes being placed on heavy cloths on racks which are folded over and built up one on top of another. There are a number of presses of various types available, not only for the large-scale manufacturer, but also for the small packer and for home use. Presses which have been used for the making of cider may be used, but they will absorb much

![Hydraulic Press](image)

Courtesy Hydraulic Press Manufacturing Co.

**Fig. 4.—Hydraulic Press.**

For pressing the juice out of the grapes. Similar presses are manufactured by the Dunning and Boschert Press Co.; A. B. Parquhar Co., Palmer Bros.; Shriver & Co.; and others.

of the color of the grape and impart it to cider that may be pressed later. It is essential that no metallic parts other than aluminum or special alloys, such as stainless steel, come in contact with grape juice.

The first juice, usually called the free-run juice, contains less solids, color, and body than the pressed juice. Hard pressing increases the amount of color and total solids. Pressing should be accomplished by a uniform pressure which is increased slowly.
COLD PRESSING JUICE

The cold pressing of juice is utilized particularly in the preparation of light-colored juices and juices intended for fermentation. The method differs from hot pressing, only in that the grapes are not heated previous to pressing. Since the color of Concord grapes is concentrated near the inner surface of the skin and since heat is required to dissolve this material, in cold pressing very little color is extracted. Washing tends to dissolve some of this color so that juice prepared from washed Concord grapes pressed cold is light red in color. The juice from unwashed grapes may be colorless. Some varieties of grapes, such as Ives, produce a dark red juice even when pressed cold. Naturally, the degree of pressing affects the color and consequently the harder the grapes are pressed, the more color is obtained.

After cold pressing, the juice is handled in the same way as hot-pressed juice. Since the juice is not heated, there are, however, a greater number of organisms than in hot-pressed juice. These organisms may develop and ferment the juice if it is not handled immediately. On the other hand, in the northeastern states, grapes are harvested in late fall and are often so cold when pressed that the juice is not warmed sufficiently during the pressing process to allow rapid development of the yeasts. Varieties of grapes which are harvested early in the season when the weather is still warm have been known to begin to ferment while still in the presses.

PASTEURIZATION AND BOTTLING OF THE JUICE

Any method of preservation of grape juice should be a safeguard against deterioration in any form. Pasteurization has a three-fold purpose. First, the yeasts, molds, and bacteria which are in the juice and are capable of fermenting or otherwise spoiling the juice, must be killed. Secondly, the enzymes of the grape which will cause deterioration of the juice must be inactivated. Finally, the juice must be bottled hot so as to exclude air, since oxidation, as will be pointed out later, has a detrimental effect on the juice.

The best method of preservation to accomplish these ends is pasteurization, altho other methods, such as cool and cold storage, may retard or entirely stop the activity of micro-organisms and enzymes or the effect of oxidation to such an extent that they may also be used successfully.
Improperly pasteurized and bottled juice may spoil or deteriorate in any one of a number of ways. The effect of the growth of yeasts is noted in that a considerable amount of gas is formed. This may be sufficient to blow the stopper or burst the container; or when the container is opened, it may cause the juice to froth or effervesce forcing a considerable portion out of the container. Bacteria are not an important factor in spoilage of juices prepared from tart Eastern grapes. Altho numerous types of bacteria are introduced into grape juice, especially from the grapes themselves the vinegar bacteria are about the only type that can develop in grape juice. Their presence may be recognized by the vinegar flavor and slimy growth of the organisms. Spore-forming bacteria are often present in the juice and may survive pasteurization, but they are unable to grow in the juice and are therefore harmless. The presence in pasteurized juice of either yeast or bacteria usually results from recontamination of the juice either from the container or failure to fill and close the container while still hot.

In the pasteurization process death of yeast and bacteria may start at temperatures as low as 120°F and very few organisms survive heating to 155°F (Table 1). The few types of bacteria that survive this temperature are fortunately unable to grow in the juice.

Molds are readily recognized since they form a film on the surface of the juice. This film is usually blue-green in color and of the consistency of wet paper. Altho the juice is altered by mold growth, it may often be used. In nearly all such cases, sufficient enzyme is produced to clarify the juice. This results in a dark-colored clear product. It has been found that this clarification is brought about by any one of a large number of species of molds. The presence of mold on grape juice has usually been found to be due to failure to stir the juice during pasteurization, to failure to remove the foam which forms on the surface of the kettle of juice during pasteurization, or to recontamination from the closure or the container. In a large number of cases, spoilage by mold has been directly traceable to contamination from the cork used to close the bottle or to small pieces of material, particularly cork, which may have been left in the bottle. It is almost impossible to sterilize cork, as well as similar materials that float, since it is not only difficult to contact the material, but cork in itself is a good heat insulator. Corks used for closures may be dipped in hot paraffin immediately before use in order to sterilize them and to enclose mold spores which otherwise might develop
Table 1.—The Reduction in Number of Living Micro-organisms During the Pasteurization of Grape Juice in Open Kettles for Filling into Carboys.

| Sample No. | Juice No. 1 | | | Juice No. 2 | | | Juice No. 3 | | |
|------------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|
|            | Time of heating, min. | Temperature of juice, °F | No. of living micro-organisms per cc | Time of heating, min. | Temperature of juice, °F | No. of living micro-organisms per cc | Time of heating, min. | Temperature of juice, °F | No. of living micro-organisms per cc |
| 1          | 0           | 116         | 30,000      | 0           | 119         | 51,000      | ...          | 146         | 16,000                  |
| 2          | 2           | 118         | 30,000      | 4           | 129         | 30,000      | ...          | 146         | 11,000                  |
| 3          | 4           | 124         | 27,000      | 5           | 132         | 30,000      | ...          | 148         | 3,200                   |
| 4          | 6           | 133         | 20,000      | 6           | 136         | ...         | ...          | 156         | 38                      |
| 5          | 8           | 142         | 10,000      | 7           | 139         | 17,000      | ...          | 162         | 0                      |
| 6          | 10          | 151         | 200         | 8           | 143         | 12,000      | ...          | 168         | 0                      |
| 7          | 12          | 160         | 0           | 9           | 146         | 3,800       | 5†           | 172         | 0                      |
| 8          | 14          | 168         | 0           | 10          | 150         | 180         | 6            | 173         | 0                      |
| 9          | 16          | 178         | 0           | 11          | 153         | 17          | 7            | 176         | 0                      |
| 10         | 18          | 187         | 0           | 11½         | 155         | 0           | 8            | 181         | 0                      |
| 11         | 20          | 192         | 0           | 12          | 156         | 0           | 9            | 185         | 0                      |
| 12         | 12½         | 158         | 1*          | 10          | 189         | 0           | 10½          | 192         | 0                      |
| 13         | 13          | 160         | 0           |              |             |             |              |             | 0                      |
| 14         | 14          | 163         | 0           |              |             |             |              |             | 0                      |

*Mold.
†Kettle filled after 5 minutes, 5 seconds.
in the juice. Even then the paraffin may crack and permit the mold spores to escape. Definite strands of mold growth from pores in corks to the surface of juice have been observed.

It has been noted that the yeast on the grapes as they come from the vineyard is not as resistant to heat as that which recontaminates the product in the processing procedures. It is essential, therefore, that the equipment, especially in commercial plants, be kept scrupulously clean at all times to prevent development of yeasts.

The enzymes of the grape which may cause deterioration of the product are inactivated at relatively low temperatures. Their effect on the juice is to cause a form of deterioration resulting in a flavor like that of an overripe grape. This effect is extremely difficult to demonstrate since juice which is insufficiently pasteurized to inactivate the enzymes usually spoils rapidly by yeast fermentation. This flavor has been noted in a juice pasteurized at only 150°F and in juice preserved against the growth and action of micro-organisms by other means.

The effect of oxidation is similar to the effect often attributed to overheating. In pasteurization of juice for carboy storage, all of the air is forced out and the bottle is filled full so that changes due to oxidation seldom occur. This form of deterioration takes place more often in juice bottled for the trade and therefore will be discussed in more detail later.

Until recently grape juice has been pasteurized in many commercial plants at 190° to 195°F, while in smaller scale operations the juice has even been boiled. Such pasteurization is wholly unnecessary, is detrimental to the product, and results in a cooked flavor. Juice bottled or carboyed at this high temperature is difficult to cool rapidly because of the danger of breakage of glass. As previously stated, few yeasts survive pasteurization at 155°F, while molds, tho slightly more resistant, fail to survive at 160°F. With ordinary precautions, pasteurization at 165°F has always been found sufficient.

Pasteurization of grape juice at 165°F has been tried on a large commercial scale both for the original bottling of juice as well as rebottling with no losses due to fermentation or mold growth. The detrimental effects of heating on the flavor of grape juice are noticeable after temperatures of 180° or higher are attained, the degree of change, of course, being more or less proportional to the temperature.

In effecting pasteurization, the juice is heated in a steam-jacketed
kettle (Fig. 5) or in tubular pasteurizers to 165° to 175°F, stirred constantly with a stirring device or a wooden spoon or paddle, and poured into hot, clean jugs, carboys, or other suitable containers, making certain that the juice is at least 165°F and that the container is filled to the top. Samples of grape juice pasteurized without stirring may show living organisms present when temperatures up to 170°F are recorded, while similar juice stirred thoroly during pasteurization may be entirely freed of micro-organisms at 155° to 160°F. Temperature differences as great as 25° have been noted between different parts of a 1-gallon laboratory kettle of juice which was not stirred during pasteurization. In large kettles even greater differences in temperature may be observed. A thermometer should always be used to determine these temperatures since overheating has a detrimental effect while underheating will not insure sterilization. The foam and solid particles which rise to the surface in heating should be carefully skimmed from the juice. It is impossible to heat this scum adequately and, therefore, organisms survive in it and are a source of recontamination and spoilage of the juice.

In filling the container, sufficient juice should be added to force the foam formed during filling over the top and out of the container. Less foam is formed if the containers are filled by tubes leading to the bottom of the containers. If a cork is used for closure, it should be
dipped in hot paraffin about half way up immediately before use and pushed in above the paraffined part of the cork. The containers should then be stored in as cool a place as available without actually freezing the juice.

In 4 months or more, tartrates and tannin-like substances, as well as solid particles, will have precipitated and formed a heavy sediment or sludge at the bottom of the container. The time required for this precipitation to take place will depend to a considerable extent upon the temperature. The cooler the temperature, the more rapid the rate of precipitation and the less chance there is of a precipitate forming in the rebottled juice. Furthermore, the cooler the temperature of storage, the more tartrates are removed and, therefore, the resultant juice will be sweeter. Factors effecting the precipitation and removal of argols will be discussed subsequently.

The temperatures of pasteurization recommended above are applicable to juices prepared from the tart grapes of the eastern states, such as the Concord. Grape juice of greater acidity naturally will not require a higher temperature since the acidity has not only a preserving effect but aids in the killing of micro-organisms. Grape juices of less acidity, such as that prepared from California grapes and possibly from a few varieties of eastern grapes of low acidity, undoubtedly require higher pasteurization temperatures.

REMOVAL OF ARGOLS OR CRUDE TARTRATES FROM GRAPE JUICE

Since the crystallization of the potassium bitartrate and the deposition of the other components of the sludge, commonly known as "argols", require a cool storage in carboys of 4 to 6 months, a number of means of hastening this crystallization and deposition have been suggested, as follows:

1. One procedure proposed was that of freezing and thawing the juice and then siphoning the cold juice from the crystalline sludge formed during freezing.

2. A second procedure involves storing the grape juice in carboys or other suitable containers at temperatures just above the freezing point of the juice, i.e., 28° to 30°F.

3. Another method, patented in 1931, involves the addition of calcium acid malate, lactate, or acid phosphate to the juice. On standing, a rapid precipitation is said to occur.

4. Removal of the tartrates is said to be hastened by the addition of dipotassium tartrate. This reacts with the free tartaric acid, thus increasing the concentration of the less soluble potassium bitartrate and so hastens its deposition.

5. Another procedure is based on the fact that the removal of protective colloids facilitates crystallization of solutes from supersaturated solutions. The method involves treatment of the juice with "pectinol", followed by a short storage period in a cool place and then filtration. This method can be used only when a clear grape juice is desired.

The conclusions presented below are based primarily on the first, second, and fifth methods, the ones studied in this laboratory.

The rapid crystallization and separation of argols from the juice by freezing, thawing, and decantation or filtration is applicable to all of the varieties of grape juice tested. The freezing method is recommended wherever the very rapid elimination of the argols is desired or where a sufficient number of carboys is not available to permit the usual period of storage. The freezing may be carried out in carboys, barrels, casks, or tanks. The temperature employed may be as high as 20°F or as low as —5°F.

Other conditions being the same, juice that has been frozen, thawed, and filtered will be clearer than that which has been given the usual carboy storage treatment, since the freezing causes the deposition as sediment of much of the colloidal material that causes the natural cloudiness of the juice. Because of its clarity, the development of a haze or cloud due to oxidation is more easily noticed in either this type of juice or that clarified by pectinol.

Another noteworthy point is that more argols are thrown down by the freezing treatment than by storage at 45°F for 6 months. Freshly thawed juice is often too low in acid to please the average person; therefore, unless the juice is allowed to stand for a few days after thawing so that sufficient acid may redissolve to give the desired degree of tartness, the frozen and thawed juice will be too bland. This point is usually attained after thawing by holding the juice for 2 days at 45°F, if frozen in carboys, or for 4 days at this temperature if barrels have been used as containers.

In general, the procedure found most suitable for the rapid removal of tartrates by freezing and thawing is as follows: Run the cool juice into carboys or barrels taking care to fill them not more than ninetenths full, thus allowing for expansion, as otherwise the containers will break during freezing. Place the barrels or carboys in a sharp
freezer at approximately 0°F. Carboys should remain 4 days and the barrels 7 days. At the end of this freezing period remove the frozen juice to a cool room, preferably one maintained at 45°F or thereabouts. It is advisable to place a number of electric fans in the room to assist the circulation of air. After the ice has thawed, allow the carboys to remain in the 45°F room for 2 days and the barrels for 4 days. Continue the use of the fans so as to bring the juice rapidly up to the temperature of the room, then siphon the juice into other containers taking care not to disturb the sludge on the bottom. Combine the various lots of sludge remaining in the containers, add about 2 or 3 per cent of a filter aid, such as Hyflo Super Cel, and filter thru canvas in a wood or aluminum plate and frame filter press.

Bottle and pasteurize the juice according to the procedure suggested in the next section of this bulletin. It is especially important to eliminate all of the air in the headspace of the bottles, since in the clear juice prepared by freezing and thawing procedures, clouding and sedimentation due to oxidation is more noticeable than in cloudy juices.

Since the crystallization and subsequent precipitation of argols is dependent upon the temperature, cool storage or storage at 28° to 30°F, which is just at the freezing point of grape juice, results in a more rapid removal of argols than storage at a higher temperature. The method has certain advantages as well as disadvantages over other procedures. Argols are not precipitated as rapidly nor as completely as they are by the methods just described. The juice, therefore, is not as bland. Yeast and bacteria present in the juice slowly die, but mold may develop. By keeping air away from the juice so that mold cannot develop, therefore, the first pasteurization of juice may be omitted.

PECTINOL CLARIFICATION AS AN AID TO DEPOSITION OF ARGOLS

The simplest rapid way of eliminating argols from Catawba grape juice is to treat the juice with a commercial pectic enzyme preparation, such as "Pectinol", which is used at the rate of 20 ounces to each 100 gallons. Enzyme preparations such as pectinol may be prepared from mold such as are occasionally found growing on grape juice. Their effect is the same as that produced by mold development upon juice in that the colloidal matter settles out leaving a clarified "dark" juice. Store in a cool room, 47°F, and after 3 days siphon off the supernatant juice, heat to 170°F, cool, and filter. This product, when bottled according to the procedure devised to eliminate air
in the headspace (page 22) remains clear and does not deposit argols, provided it is not stored below 47°F.

In Concord juice treated according to this procedure, argols are deposited if the bottled juice is stored in a cool place; therefore, this treatment is not recommended as a means of eliminating argols from Concord grape juice unless the cool storage period is extended to 6 or 8 weeks. Even then it possesses some advantages as its use cuts the time required for elimination of argols approximately in half. However, clear juice is obtained, and, as has been indicated above, such juice is more difficult to keep without apparent change.

**BOTTLING AND PASTEURIZATION**

As previously stated, the three principal causes of deterioration of grape juice are the growth of micro-organisms, mold, yeasts, and bacteria; the action of enzymes; and oxidation changes. In rebottling grape juice from carboys after precipitation of the argols, the juice is recontaminated with micro-organisms so that repasteurization of the juice to kill these micro-organisms becomes necessary. The enzymes have previously been inactivated if the juice is pasteurized for carboy storage, but if it was held in cold storage, pasteurization is also necessary for inactivation of the enzymes. The inhibition of oxidation effects due to air are still an important consideration; in fact, due to methods in general practice for rebottling grape juice, it is far more of a factor than in the first pasteurization.

The deterioration occurring during storage which this work has shown to be caused by oxidation was previously ascribed to various other causes, including the cooking effect of pasteurization, high storage temperatures, the action of light, and reaction of the acids of

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**Fig. 6.—A Partly Filled Bottle of Grape Juice Lying on Its Side to Show the Effect of Oxidation.**

Note the deposition of colored sediment on the sides and bottom of the bottle.
the juice with the alkalies of the glass. The effect of oxidation is first noted by a clouding of the juice, especially the clear samples. Shortly afterward a brown sediment begins to be deposited, and in advanced deterioration a subsequent clearing takes place leaving an amber-colored juice with a very heavy sediment on the bottom of the bottle (Figs. 6 and 7). In some instances all of the bottle in contact with the juice may be stained because of deposition of colored sediment.

Simultaneously with the clouding and sedimentation, the color of the juice changes, first from reddish purple to a dull brick red and then to a dull brown. Changes in flavor and aroma are more difficult to describe but are just as definite. At first there is a loss of flavor then the juice takes on a flavor similar to that of over-cooked grape jam, and still later, that of a poor grape butter. If the bottle contains an excess of air, e.g., is only half filled, the flavor of juice finally becomes very unpleasant because of the development of off-flavors. From a chemical standpoint only slight changes occur in the juice. The main changes observed are a decrease in protein and acidity with an increase in total water-insoluble sediment. The deterioration as noted by flavor and color

Fig. 7.—Difference in Volume of Bottled Grape Juice.

*Left*, bottle filled cold and pasteurized at 170°F; and *right*, a bottle filled at 175° and pasteurized at 170°F. The former procedure does not exclude the air, therefore the juice deteriorates. Note the slight haziness at the bottom of the left hand bottle which is sediment due to the presence of air. Also see Fig. 6.
is far more marked than changes in chemical constitution of the juice.

**IMPORTANCE OF FILLING BOTTLES COMPLETELY**

Studies of the pasteurization and storage of grape juice previously reported\(^8\) have shown clearly that, assuming that the juice was pasteurized at a sufficiently high temperature to sterilize it, the degree of deterioration which may occur in the bottled juice is directly proportional to the amount of air left in the headspace. Other factors, such as temperature of storage and kind of light to which the juice is exposed, may affect the rate of deterioration, but they have no effect on the extent of the deterioration. Thus, it has been clearly shown that, if juice is bottled in such a way that no air remains in the

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**FIG. 8.—DIFFERENCES IN FILL OF BOTTLED GRAPE JUICE.**

Bottles of grape juice showing: A, a bottle filled at 200° and pasteurized at 195°F; B, a bottle filled at 185° and pasteurized at 180°F; C, a bottle filled at 165° and pasteurized at 160°F; and D, a bottle completely filled. The difference in fill of bottles A, B, and C is due to the difference in contraction on cooling.

\(^8\)See footnote 1.
headspace, the product may be left in a sunny window for a year or more without noticeable changes in clarity, color, flavor, or aroma.

The simplest way of obtaining bottled grape juice substantially free from air in the headspace is to fill the bottles completely at a temperature a degree or two higher than that employed in subsequent pasteurization and then close the bottles immediately with a closure which will not leak air subsequently (Fig. 8). The juice is likely to take on a cooked flavor if it is held in an open kettle at the pasteurization temperature for considerable periods of time unless the bottle filling operations are carried out rapidly. Open-kettle pasteurization is the method commonly employed (page 14) in filling carboys and is satisfactory for that purpose because the filling operation is carried out rapidly and the juice does not remain exposed to air at the pasteurization temperature for a long period of time. A much more satisfactory system of preheating is that employing a tubular pasteurizer or heat interchanger (Fig. 9). After filling, the bottles of juice may be held at the pasteurizing temperature in a holding type pasteurizer (Fig. 10).

![Tubular Pasteurizer](image)

**Fig. 9.—Tubular Pasteurizer.**

The grape juice flowing thru the inner tubes is heated by steam or hot water for filling into bottles. This is a continuous-flow pasteurizer suited for bottling at constant temperature.
The advantage of filling the bottles hot is that, assuming a hermetic seal, when the juice cools a vacuum forms in the headspace with very little or no air present. If cold juice is put into the bottles, they

![Diagram](image-url)

**Fig. 10.—Automatic Pasteurizer for Bottled Juice.**

The bottles of juice are placed in baskets which travel thru consecutive compartments which preheat, pasteurize, and finally cool the juice. Since it is recommended that bottles be filled with hot juice, the preheating section would be operated at the pasteurizing temperature. A similar pasteurizer is manufactured by the Liquid Carbonic Company.
cannot be completely filled without causing them to break during subsequent pasteurization. It is possible to eliminate a large part of the air in the headspace by filling cold to the level now commonly employed commercially and then closing under a high vacuum, but this system requires a much more complicated closing machine and even then does not attain the high vacuum obtained by the hot-filling procedure described above. Furthermore, when bottles are filled hot, a long period of pasteurization in bottles is unnecessary to bring the

![Graph](https://via.placeholder.com/150)

**Fig. 11.—Rate of Transmission of Heat to the Center of Bottles of Concord Grape Juice Immersed in Water Held at 160°F.**

It is readily seen that the killing temperature for organisms is not attained until sometime after immersion of the bottles of juice in the water.
temperature of the juice to that of the water bath. This time varies from 18 to 30 minutes, depending upon the size of container (Fig. 11). Furthermore, when the bottles are filled with a cold juice, allowance must be made for expansion of the juice and therefore, as complete a fill is never obtained (Fig. 8). The amount of headspace in bottles depends not only upon the difference between the temperature of pasteurization and the temperature of the juice when cool, but also upon the original fill of the bottles.

TEMPERATURE OF PASTEURIZATION

If the juice is bottled hot under the conditions described above for eliminating air in the headspace, there is relatively little danger of the development of “cooked” flavors during pasteurization, and temperatures as high as 180°F may be employed for pasteurization without appreciable injury to the quality of the juice.

However, if air is permitted to remain in the headspace, it is important to pasteurize the bottled juice at the lowest temperature and for the shortest time in which complete sterilization is assured, as otherwise the juice will lose some of its fresh flavor and aroma and will take on the characteristics of a very slightly cooked juice. Studies of the destruction by heat of micro-organisms during pasteurization have shown that heating of the juice in small bottles, e.g., half-pints, for 15 minutes at 160°F is sufficient to sterilize it (Table 2). It will be noted that during pasteurization at 150°F and 155°F a few organisms persist throughout the holding period of pasteurization. This is not true when the juice is pasteurized at 160°F. Under commercial conditions, however, it is wise to employ a temperature of 165°F to 170°F for 20 to 30 minutes, the length of time depending upon the size of the bottle. This time may be adjusted in automatic pasteurizers (Fig. 10) by varying the speed of operation. Attention to details is more essential if juice is bottled cold since it has been shown that a temperature of 160°F is not attained until 18 to 33 minutes have elapsed, depending upon the size of the container (Fig. 11).

As a matter of precaution, if a low temperature of pasteurization is employed, it is advisable to make sure that the crowns used on the bottles are not contaminated with molds and that they are put on tight without cracking the paper liner. The paper liner serves to protect the juice from coming in contact with the cork. The corks used for closure have been found to harbor mold spores which may
<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Pasteurization at 150°F</th>
<th>Pasteurization at 155°F</th>
<th>Pasteurization at 160°F</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Time, min.</td>
<td>Temperature, °F</td>
<td>No. of living microorganisms per cc</td>
</tr>
<tr>
<td>1</td>
<td>0</td>
<td>40</td>
<td>50,000</td>
</tr>
<tr>
<td>2</td>
<td>3</td>
<td>60</td>
<td>50,000</td>
</tr>
<tr>
<td>3</td>
<td>4</td>
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</tr>
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<td>4</td>
<td>10</td>
<td>100</td>
<td>50,000</td>
</tr>
<tr>
<td>5</td>
<td>12½</td>
<td>110</td>
<td>50,000</td>
</tr>
<tr>
<td>6</td>
<td>14½</td>
<td>120</td>
<td>3,000</td>
</tr>
<tr>
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<td>16½</td>
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</tr>
<tr>
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<td>19</td>
<td>135</td>
<td>100</td>
</tr>
<tr>
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<td>20</td>
<td>140</td>
<td>4</td>
</tr>
<tr>
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<td>145</td>
<td>15</td>
</tr>
<tr>
<td>13</td>
<td>15½</td>
<td>150</td>
<td>2</td>
</tr>
</tbody>
</table>

**In Preheater**

**In Bottle**

* Bottles of juice were removed from pasteurizer at five-minute intervals in all tests.

Those marked with an asterisk were fermented or molded.
be a source of spoilage if they come in contact with the juice. The cork of the crown is a poor conductor of heat and this makes the destruction of the molds therein difficult.

If temperatures above 180°F are used for effecting pasteurization of juice containing air in the headspace, rapid oxidation occurs during the pasteurization process and the juice takes on a flavor of grape jam.

**STORAGE OF BOTTLED JUICE**

Ordinary commercially bottled grape juice which contains air in the headspace should be stored in a cool, dark place but not cooler than the cellar in which the carboys of juice were stored during elimination of the argols. A temperature of 45° to 47°F may be recommended, but less change in flavor has been found to occur at 30° to 32°F. However, there is danger of the deposition of argols if the juice is held there for considerable periods of time.

If it is desired to display juice containing air in the headspace in windows or on shelves where it is exposed to light, the bottles should be made of glass which will not transmit the short wave lengths of light (violet and ultraviolet) or should be wrapped in paper or sheet cellulose treated in such a way as to prevent the passage of this light. As has been previously pointed out, the short wave lengths of light merely accelerate oxidation, and if there is no oxygen in the juice or the headspace over it, there can be no oxidation even in the presence of the light.

As indicated above, if Concord grape juice is bottled and pasteurized under conditions which eliminate the air in the headspace, it keeps very well under all ordinary conditions of storage. However, it should not be stored for long at temperatures lower than that which was employed in the carboy storage for the elimination of argols because of the danger of further crystallization of these salts. Moreover, under no circumstances should bottled grape juice be permitted to freeze. Bottles filled more than 92 per cent full will break during freezing, and bottles containing less than this amount of juice, altho they will not break, will be found to contain argols and coagulated colloidal matter after freezing.

**CARBONATED GRAPE JUICE**

Altho numerous carbonated grape beverages are being successfully marketed no one has been successful with a carbonated grape juice. Carbonation adds much to the flavor of Catawba juice, and, if the cost of its manufacture is not too great so that the price asked
would be too high, it would seem to offer possibilities. Carbonated Concord juice is not as pleasing a beverage as that prepared from Catawba juice. It is also more difficult to prepare so that it will not deposite sediment during storage.

Nevertheless a good carbonated Concord grape juice should find favor with those who prefer sparkling fruit juices to the still products. It should also be a good mixer in certain types of punches and mixed fruit drinks.

In selecting juice for carbonation, it is important to use juice from which the colloidal matter has been removed (a) by long standing in carboys (3 or 4 years) in a cool place, (b) by freezing and thawing, or (c) by treatment with the commercial pectic enzyme preparation, "Pectinol," and subsequent filtration. Moreover, care should be taken during carbonation to eliminate air from the headspace of the bottle. Also, the pasteurization temperature and time employed should be just sufficient to sterilize the juice.

Grape juice can be carbonated either by passing carbon dioxide into the juice contained in equipment especially devised for the carbonation of beverages, or by shaking the cold bottled juice with a weighed amount of solid carbon dioxide. The former procedure is that employed in all large scale operations involving the carbonation of fruit juices.

Two precautions must be taken if a first class carbonated juice is to be obtained. First, the carbonator must be kept substantially sterile. This can be accomplished by cleaning it out with a sterilizing solution and then rinsing with distilled water just before use. If micro-organisms are allowed to grow in either the carbonator itself or its fittings, they will be introduced into the juice during the carbonation operation and the product will be more difficult to sterilize. It is also important to operate the filler in such a way as to eliminate the air from the headspace of the bottle. Therefore, the "sniffting" operation should be carried out so as to blow out the air from the bottle. If air is permitted to remain in the bottle, the juice will cloud during storage, and eventually sediment will be deposited on the bottom of the bottle.

Grape juice is very corrosive to most metals; moreover, contact with iron causes the discoloration of the juice. Stainless steel and aluminum are not affected to any considerable extent by grape juice; therefore, grape juice factory equipment is usually constructed of one or the other of these metals and the carbonation equipment should not be an exception to this rule. Silver-plated equipment is
sometimes used but does not last, as it is almost impossible to get the silver plating free from pin holes.

If the carbonation operations are conducted on a small scale, it may be simpler to use solid carbon dioxide for the purpose. If this is done, the following precautions must be observed:

1. Use only very cold (40°F or under) grape juice.
2. Allow a headspace at least 10 per cent of the volume of the bottle.
3. Weigh out the solid carbon dioxide accurately to 0.1 gram.
4. Use an amount of solid carbon dioxide which will give not more than 3 volumes of carbonation (Table 3).

Table 3.—Amount of Solid Carbon Dioxide Required for Carbonation of Grape Juice.

<table>
<thead>
<tr>
<th>Weight of Solid Carbon Dioxide, Grams</th>
<th>Volume of Juices, Fluid Ounces</th>
<th>Size of Bottle, Fluid Ounces</th>
<th>Carbonation, Volumes</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.0</td>
<td>7</td>
<td>8</td>
<td>1.8</td>
</tr>
<tr>
<td>2.0</td>
<td>14</td>
<td>16</td>
<td>1.8</td>
</tr>
<tr>
<td>4.0</td>
<td>28</td>
<td>32</td>
<td>1.8</td>
</tr>
<tr>
<td>1.5</td>
<td>7</td>
<td>8</td>
<td>2.8</td>
</tr>
<tr>
<td>3.0</td>
<td>14</td>
<td>16</td>
<td>2.8</td>
</tr>
<tr>
<td>6.0</td>
<td>28</td>
<td>32</td>
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<td>7</td>
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</tr>
<tr>
<td>8.0</td>
<td>28</td>
<td>32</td>
<td>3.8</td>
</tr>
</tbody>
</table>

5. Wear gloves. Do not pick up the solid carbon dioxide with the fingers or it may freeze the skin.
6. Allow just enough time after the addition of the carbon dioxide to permit the gas evolved to sweep out the air from the headspace and then cap quickly. This can be told by the appearance of a fog in the neck of the bottle.
7. Immediately after capping wrap the bottle in a piece of burlap or canvas and shake vigorously and constantly for at least 3 minutes or in any case it must be shaken until all of the solid carbon dioxide has sublimed. The bottle should then be placed in a corrugated fiber-board shipping container.

Two-and-a half to 3 volumes of carbonation is the amount which will please the average person. Higher carbonation covers up the flavor of the juice and makes pasteurization difficult as only the very best bottles will stand the pasteurization temperature at high carbonation.

If sterile juice is employed (that obtained from a freshly opened carboy), pasteurization for 30 minutes at 150°F will be sufficient for 8-ounce bottles, while 35 minutes and 40 minutes will be required for 16 and 32 ounce bottles, respectively.

The carbonated juice stands up best if stored at 45° to 50°F.