

1936

Preparation and preservation of apple pectin extracts

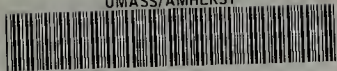
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PREPARATION AND PRESERVATION
OF APPLE PECTIN EXTRACTS

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PREPARATION AND PRESERVATION OF APPLE
PECTIN EXTRACTS

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Thesis submitted for
the degree of
Master of Science

Massachusetts State College, Amherst

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I. INTRODUCTION

A suitable method for the preparation and preservation of pectin extracts and concentrates from grade C or cull apples, and apple pomace, would provide a use for materials otherwise largely wasted. Apple pectin concentrates if easily prepared would effect a saving for farm and orchard owners, farm factory and cider mill operators, and even for the housewife who might not wish or be able to pay the prevailing high prices asked for the commercial pectin extracts now on the market. There is a great abundance of literature pertaining to methods of extraction of pectinous juices, but the great diversity of procedures recommended and the lack of uniformity in the results obtained are misleading and confusing. On the other hand, very little work has been done on the clarification, preservation and storage life of pectinous extracts of fruits.

It is the purpose of this thesis to check the extraction methods now recommended and determine which is most satisfactory, to find a workable means of clarifying the extracted juices, and to discover insofar as possible, their storage life under various conditions. The work on methods of extraction of fresh apples will be largely a check on the previous work of Fellers (1928), pertaining to ratios of fruit to extraction water, time and temperature

of extraction, and the effect of the addition of organic acids. The extraction of pomace from the same apple variations likewise will be employed. The clarification of the pectinous juices will be attempted by methods believed to be in commercial practice, and storage life will be determined by measuring the deterioration of variously prepared pectin extracts stored under usual warehouse or farm conditions.

II. REVIEW OF LITERATURE

Pectin was discovered in 1825 by Braconnot, when he made artificial jellies with a pectin which he extracted from carrots. Since that time many others have worked on pectin, attempting to determine its chemical constitution, its part in jelly making, and the optimum conditions for its extraction from various raw materials, until today the literature on the subject is very voluminous. For the purpose of this problem only the literature bearing on the extraction, clarification and preservation of pectin was considered. Many have recommended a wide variety of extraction methods but few have considered the problem of clarification; all have considered the preservation of pectinous extracts to be a simple matter, but no one has ever determined their deterioration during storage.

A. Methods of Extraction

Many patents have been granted in various countries for methods of pectin extraction. Douglas (1915) received a British patent for extraction of apples or other fruits or vegetables by removing the natural sugars, e.g. expressing the juices of the fruit, and treating the pulp with a solvent such as hot water, which may contain acid. Practically all methods are variations or modifications of this one, but many are more explicit. The Douglas patents were used exclusively by the Certo Corporation of Rochester, New York, the largest manufacturers of liquid apple pectin in the United States. Hunt (1918) described a method of extracting pomace, cull apples, cores and peelings by three extractions of one part of raw material in three or four parts of boiling water; the pectin was obtained in solid form by precipitation with $(\text{NH}_4)_2\text{SO}_4$ (25 grs. per 1000 c.c. extract). Barker (1918) freed raw material containing pectin from acids by leaching with cold water or by neutralization. Pectous substances were then rendered soluble by treating the raw material with steam, and were subsequently removed with hot or cold water; such a neutralized extract may be concentrated and preserved with sugar without jellying. In 1921 two U.S. patents were granted, one to Barker (1918) for the

method described above, and one to Doell and Maes (1921) for the extraction of pectin from fruit rind, peel or core by boiling in water containing citric or other acid and a bleaching agent such as cellite and boneblack.

In 1920 the Food Investigation Board of the Department of Scientific and Industrial Research of Great Britain reported that by the action of heat and acid more pectin may be obtained from fruit tissue than is originally present in the soluble state. This report stated that the greatest amount of pectin obtainable from any raw material is that extracted by the action of hot dilute acid; steam pressure extraction is less efficient than hot dilute acid, but is better than non-acid hot water extraction. Beylik (1922) received a U. S. patent for a method of preparation of pectous material for jelly making; he treated the raw material with alcohol, ether, or chloroform to dissolve substances other than pectous and cellular tissue, and then dried the insoluble pulp. In the same year Monti (1922) received a patent for a method of extracting pectin with water and concentrating in vacuo at a low temperature (40° C. or below) to preserve the natural fruit juice flavor.

Paine (1922) stated that the alcohol precipitate method for determining pectin content may not give con-

cordant results when used on different fruits because of the precipitation of other pectous but non-jellying substances. Jelly quality may therefore vary with different pectins. Leo (1921) was granted an English patent for a method of producing a mixture of pectous substances in raw material containing pectose by treatment with a solution of pectase derived from a non-acid vegetable such as the carrot. Crawford (1924) received a U. S. patent for removing pectous substances from raw material by treatment with hot lactic acid after sugars had been removed with hot water. Davidson (1925) reported a method of pectin extraction by treatment of fruit pulp, after expression of natural juices, with a 0.07 - 0.15 per cent acetic acid solution. To remove sugars the extract was subjected to alcoholic fermentation, which was stopped before the pectin was destroyed; the fermentation product was then concentrated until the acetic acid content reached 0.65 per cent, when the product was bottled and sealed.

Mehlitz (1926) stated that boiling for ten hours causes decomposition of apple pectin solution of 16 to 20 per cent, most of which occurs during the first few hours. Bell and Wiegand (1925) gave directions for obtaining the " - largest possible extraction of pectin - ."

They parboiled fruit at temperatures not over 212° F. with

the addition of a very small amount of water for berries, and of about one-half the weight of the fruit for material high in cellular matter (apples, etc.) either with or without the addition of citric or tartaric acid. Nanji (1927) was granted a U.S. patent for a method involving the treatment of material containing pectin with dilute solutions of salts, such as ammonium tartrate, which are capable of rendering soluble substantially all of the pectin present. A British patent was granted Schwartzauer, Honigwerke and Zuckerraffinerie (1927) for a means of removing soluble coloring substances, sand and other impurities by alternately stirring the raw material in cold weakly acidulated water, and pressing; pectin was then removed by a hot acid extraction of the pulp.

Fellers (1928) determined that the optimum fruit to water ratio is 3:2 where sliced apples are used, and 3:3 where chopped apples are used. He obtained maximum yields of juice, pectin and acid when he used two extraction periods of 15 minutes each at 212° F. Tartaric acid added to the extraction water in concentrations of 0.2 - 0.4 per cent, slightly increased jelly yield, flavor, color and consistency. A French patent was issued to the Pectinerie du Kervor (1927) for a method of removal of sugars and salt from apples or apple waste by treatment with water containing dilute inorganic acid.

Myers and Baker (1929) determined that the jelly grade of pectins reached a maximum value when the pectin was extracted at a pH of approximately 2.40; lower pH values resulted in a sharp decline in the jelly grade of the extracted juice. They reported that jelly grade decreased with the time of boiling, although the yield of the extracted pectin increased with the boiling period. The same workers (1931) found that as the extraction temperature increases the pectin yield also increases, but that in general, the higher the extraction temperature the lower the jelly grade of the extracted juice. Darling and MacMillan (1931) received a U.S. patent for the removal of pectin from apple pomace, after the extraction of palatable juices under pressure, by the addition of 10 per cent by weight of hot water and the application of much higher pressure. In the same year a patent was issued to the California Fruit Growers' Exchange (1930) for the extraction of pectin from substances containing it, particularly cellulosic material, by treatment with hydrochloric acid (pH 1.5 - 1.9) at 50° - 70° C. for a short period.

A German patent was issued in 1928 to "Deutsches Pektinges" (1928) for the removal of soluble substances and calcium salts by treatment with water, not above

40° C. and containing a little SO₂. Pectin was then extracted with hot water. Mithoff (1934) was granted a U.S. patent for treatment of pectin, prior to extraction, with dilute tertiary butyl alcohol at about 60 - 88° C., and subsequent washing; this treatment removes coloring and flavoring material. Baker (1934) reported that tannins, sugars and alcohol soluble salts may be removed from apple pomace by twice leaching, or by soaking four parts of the pomace in one part of 90 per cent alcohol and pressing. Enzyme activity is also immediately stopped by this treatment. Practically all the methods described are variations of the method of removing sugars and salts with a suitable solvent, and extracting the pectin with hot water, usually weakly acidified.

B. Methods of Clarification

As early as 1916 Douglas (1916) received an English patent for the clarification of pectinous extracts by treatment with malt extract or a starch-converting enzyme; however, since that time, but little work has been done on this phase of pectin production. Caldwell (1917) described a method of concentrating pectin extracts by freezing and separating the liquid by use of a cream separator or by straining; he accomplished

preservation by the addition of one ounce of calcium carbonate to seven or eight gallons of the water extractions. Magoon and Caldwell (1918) reported a means of removing matter suspended in pectinous extracts by precipitation, by the addition of commercial alum, ammonia water, or heat treatment. Crawford (1924) treated pectinous extracts with a proteolytic enzyme to convert proteins to soluble substances; it is doubtful whether much was accomplished by this treatment. Perrier (1924) proved that starch is present in pectinous extracts in largest amounts in those made from either dried fresh apples or dried pomace, since the drying treatment used renders starch almost completely soluble. Juices from fresh apples contained less starch than juices from fresh pomace.

A British patent granted to the California Fruit Growers' Exchange (1929) and various workers, among them Bell and Wiegand (1925), Bender (1929), and Ripa (1929), have reported results obtained by centrifuging pectinous extracts or by filtration with or without pressure, with the aid of some highly porous material such as "Celite" or "Filter-Aid"; the last named investigator used ground nut shells.

Bender (1929), treated apple pectin extracts with solid casein, separated the clear liquor from the

sediment, treated it with diastatic enzyme, then with activated carbon and finally filtered it. Baker and Kneeland (1935) stated that diastatic action had a detrimental effect on viscosity and jelly grade of pectin solutions, but that hydrolysis was least at a pH value of 3.3. Pistor (1935) received a German patent for the clarification of pectin solutions by treatment with tannin, followed by filtration; the tannin combines with the starch in solution and is added in a proportion about twice that of the starch.

This literature review reveals the large amount of research that has been conducted on pectin and on extraction methods. There is great industrial activity in this field as witnessed by the large number of patents issued. However, as yet no one has proposed a satisfactory method for the small scale preparation of apple pectin.

III. EXPERIMENTAL

A. Raw Material Survey

Fresh Apples

All the fresh apples used were Baldwin Grade C culls, kept in cold storage for from two to six months. Once removed from the cold storage temperatures, they were used as quickly as possible.

Pomace

The pomace used was the residue from cold-pressed King and Baldwin apple-cider, dried to a moisture content of 6 to 8 per cent and stored in covered wooden boxes at room temperature. The drying was accomplished by spreading the wet pomace, immediately after its removal from the cider press, in thin layers on screens which were then placed in a dryer equipped with three electric heating units and a rapidly moving fan. In this manner a swift current of air at a temperature of 120° F. passed continuously over the pomace, and drying was accomplished in 48 to 60 hours.

Two extractions of part of the dried pomace in cold water, to remove sugars, were then made. Eight pounds of pomace were well broken up, added to 96 pounds of water at 80° F. and allowed to soak for twenty minutes. At the end of this time, the extraction water was poured off

through a heavy corded cloth of the type used in cider pressing, and as much more as possible was pressed from the pomace in a hand operated cider press. A second similar extraction and pressing were then made on the same pomace, adding the same amount of water as in the first extraction. Saccharometer readings were made on the first and second extractions and on the two combined; altogether 165 pounds of extraction water containing 2.45 per cent soluble solids were pressed from the soaked pomace. The pressed pomace was then returned to the dryer, dried as before, and stored at room temperature.

B. Determination of Optimum Extraction Methods for Raw Materials Used

It is well known that juices pressed cold from fruits contain little or no pectin, and therefore cannot be used in jelly-making. The reason for this is that pectin is held in the cell-walls of fruit and is not released until the cell-structure is broken down. This disintegration of the cell-membranes may be accomplished most readily by heat. For this reason, only heat extracted juices were considered in this study of the extraction problem. Both pomace and fresh apples were successively extracted three times at temperatures of 190° F., 212° F. and 240° F. for from 15 to 30 minutes

and the total yields of juice, their jelly strength or sugar supporting capacity, their soluble solids content and the total yield of jelly was determined for each extraction separately, for the first and second extractions combined and for all three extractions together. For fresh apples, the ratio of fruit to extraction water varied from 3:4 to 3:2; for pomace extractions the ratios varied from 1:6 to 1:16. The effect of the addition of different concentrations of tartaric acid to the extraction water was also noted. Chlorinated tap water was used in all extractions.

Fresh apples were first sliced in a hand slicing machine adjusted to give slices $1/4$ inch in thickness, and pomace was broken into flakes less than one inch in diameter. The desired amounts of the raw material and extraction water were then put together in the containers used (depending on the extraction temperature), the desired temperature obtained as quickly as possible, and the heat regulated to maintain this temperature for the duration of the extraction period. For extractions at the boiling point or below, 10 quart aluminum pans equipped with close fitting covers were found convenient; in the former case heat was obtained from ordinary gas stove burners and in the latter from a temperature con-

trolled water bath. For extraction at 240° F. steam pressure cookers were employed. When the extraction period was completed, the juice and pulp were poured into a cheesecloth, allowed to drain for a minute or two and thoroughly squeezed out by twisting the ends of the cloth in the hands for several minutes. The pulp was returned to the pan, the desired quantity of water added, and the second extraction made; in like manner a third extract was obtained. Examination of the extracts was made immediately.

1. Measurement of Jelly Strength and Yield of Extractions

Present quantitative methods in general use the alcohol precipitation method for determining the pectin content of extracts. In all alcoholic precipitates there are present certain amounts of pectinous compounds that do not possess jelling power. At the present time we have no method which differentiates jellifying pectin and non-jellifying pectin. Such a procedure is greatly needed. In these experiments no analysis was made by chemical methods for pectin determination. Instead, a method evolved by Myers and Baker (1927) was employed to find the jelly strength of the pectinous juices. The jelly strength of a pectinous extract is defined as being

its sugar supporting capacity and is expressed in the units of sugar jelled by one unit weight of juice to give a jelly of definite strength. These investigators found that jelly strength of a pectinous juice is a function of its viscosity, this relationship being independent of the concentration of the pectin. Since the yield of jelly is dependent upon the amount of added sugar and the amount of added sugar is dependent on the relative viscosity of the pectin solution, the relative viscosity of the solution determines the jelly yield. Baker (1934) has evolved a general empirical formula for converting the relative viscosity value of a pectin extract into terms of its jelly strength:

$$\lg y = .833 x + .195$$

where y = the relative viscosity

and x = the unit weights of sugar jelled by one unit weight of juice

$$\text{In terms of } x : x = \frac{\lg y - .195}{.833}$$

For measuring the viscosity of the extracted juices, a simple "jellmeter" originally devised by Baker (1934) was used. It consists of several inches of capillary tubing (.02 to .04 inches) sealed to a small glass cylinder of several c.c. capacity; the time required for the juice to flow from one etched mark on the glass

cylinder to another below divided by the time required for water at the same temperature gives the relative viscosity of the juice. In these determinations the jellmeter was held vertically in a clamp and the flow at 76° F. was timed with a stop watch. From the data so gathered the jelly strength of the extracted juice, the yield of jelly per unit of juice, and the theoretical total yield of jelly obtained from the extractions, separate and combined, was computed by Baker's formula. The computation of jelly yield per unit of juice and total jelly yield was made on the basis of a finished jelly with a sugar content of 67 per cent, which Fellers (1928) found most satisfactory. Some of the extracted juices were used to make jellies according to Baker's jellmeter and formula, and were so constant in giving satisfactory results that later in the experiment the practice was largely discontinued, and yields and jelly strength values merely figured. Readings of soluble solids content (largely sugar) were made on an Abbe refractometer at room temperature.

2. Fresh Apple Extractions

a. Effect of Variations in Time

In Figure I and Table I are shown the results of successive extractions of 15 and 30 minutes each at 212° F. using 3 pounds of sliced Baldwins and 3 pounds of water. It is seen that in the 15 minute period each of the three extractions produced a juice successively lower in jelly strength, jelly yield per unit of juice and soluble solids content, that the strength of the first two combined was slightly lower than that of the first, and that the three together were much lower than the first and second extractions combined. However, jelly yield is not dependent only on the jelly strength of an extracted juice, but also on the total yield of juice. Considering the relative value of the extractions on this basis, the first and second extractions together produced a large yield of juice of a fairly high jelly strength, which resulted in the greatest total jelly yield, and entirely overbalanced the apparent advantage which the first extract alone would possess if only jelly strength were considered. Although the addition of the third extracted juice substantially increased the total juice yield, it so weakened its sugar supporting capacity that the result was to decrease the total jelly yield. The first 30 minute

Figure I. Jelly Strength of Extractions of 3 Pounds of Fruit in 3 Pounds of Water for 15 and 30 Minutes at 212° F.

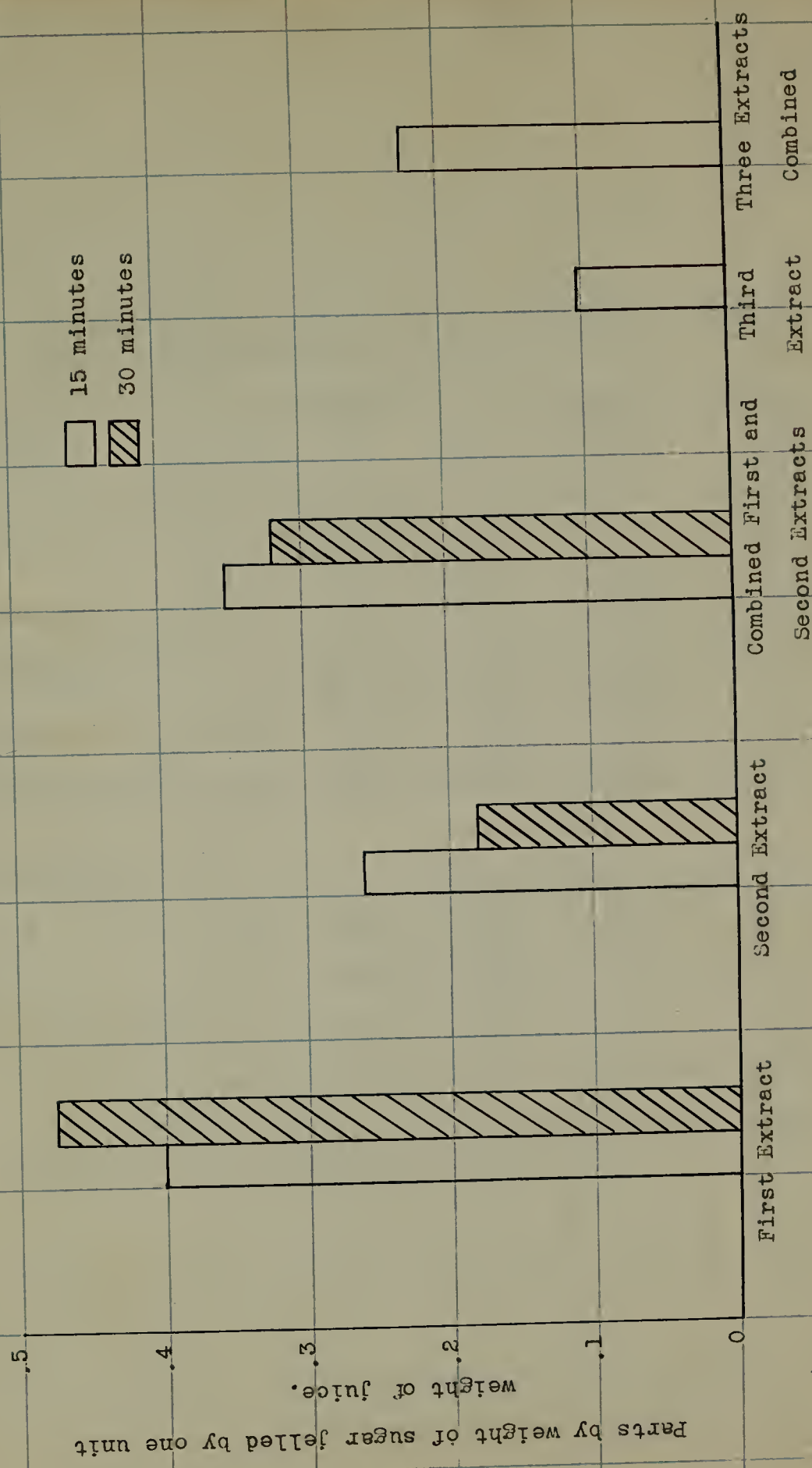


Table I. Extractions of 3 Pounds of Apples and 3 Pounds of Water for 15 Minutes at 212° F.

15 Minute Extraction	Relative Viscosity	Yield of Juice ounces	Jelly Strength*	Parts of Jelly per One Part Juice	Total Jelly Yield ounces
First	3.36	55.75	.3977	.5935	33.09
Second	2.58	51.35	.2600	.3880	19.92
First and second combined	3.08	107.61	.3523	.5258	56.58
Third	1.91	36.25	.1032	.1540	5.58
Three combined	2.42	140.42	.2266	.3380	47.49

30 Minute Extraction					
First	3.94	52.00	.4807	.7174	37.30
Second	2.23	34.66	.1840	.2746	9.52
First and second combined	2.91	86.60	.3227	.4816	41.70

* Jelly strength is expressed as parts by weight of sugar jelled by one unit weight of juice.

extraction at 212° F. greatly exceeded the first 15 minute extraction in relative viscosity, jelly strength and yield, and soluble solids content, but the second was almost as poor in the same respects as the third of the successive 15 minute extraction periods. Together or singly, they did not equal the combined first two 15 minute extracts in strength and yield.

b. Effects of Variations of Temperature

Myers and Baker (1931) state "As the extraction temperature increases the yield of pectin increases when the extraction period remains constant. It is therefore impossible to obtain as great a yield at any definite temperature as may be obtained, under optimum conditions at a higher temperature.----- As the temperature of the extraction period increases, the jelly grade of the resulting pectin decreases when the extraction period remains constant." The results obtained in this experiment are in agreement with the statement of these investigators.

Table II and Figure II picture the results of two successive extractions of 3 pounds of apples with 3 pounds of water for 15 minutes at 10 pounds of steam pressure (240° F.) and at 190° F., as compared with the two 15 minute extractions using the same fruit to water ratio and a temperature of 212° F.

Figure II. Comparison of Two Extractions for 15 Minutes at 212° F. and 240° F. and for 30 Minutes at 190° F.

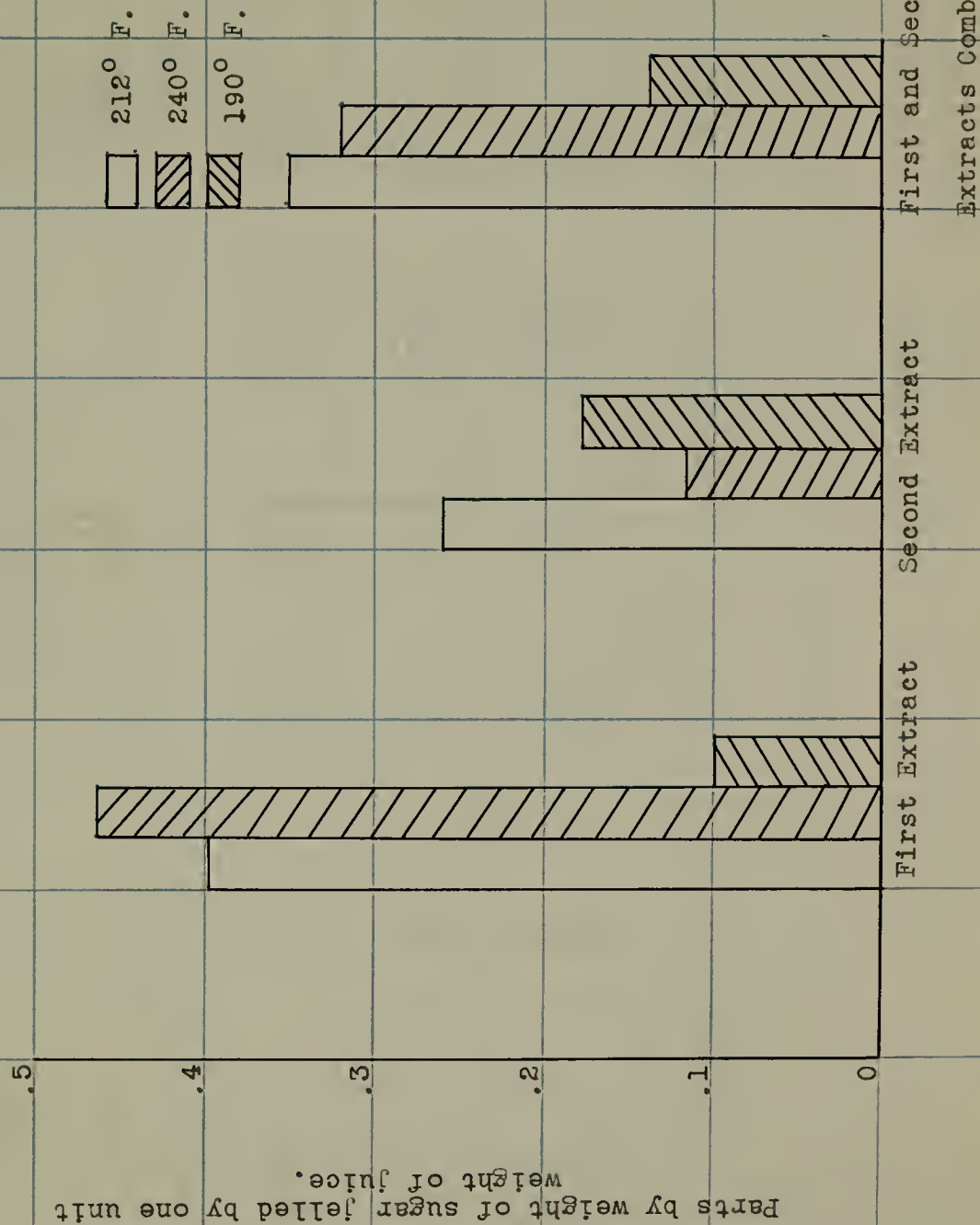


Table II. Extractions at 190° F. for 30 Minutes and at 240° F. for 15 Minutes Compared with Extractions at 212° F. for 15 Minutes, Using a Fruit to Water Ratio of 3 to 3 by Weight

Extraction	Extraction Temperature	Relative Viscosity	Yield of Juice	Jelly Strength*	Parts of		Total Jelly Yield
					Jelly per One Part Juice	ounces	
First	degrees F.		ounces				
	212	3.36	55.75	.3977	.5935		33.09
	240	3.83	60.5	.4660	.6940		41.99
	190	1.89	49.20	.0977	.1458		7.17
Second	212	2.58	51.35	.2600	.3880		19.92
	240	2.00	37.5	.1272	.1898		7.12
	190	2.10	47.65	.1767	.2637		12.56
First Two Combined	212	3.08	107.61	.3523	.5258		56.58
	240	2.91	98.0	.3227	.4816		47.19
	190	2.04	97.20	.1376	.2053		19.95

* Jelly strength is expressed as parts by weight of sugar jelled by one unit weight of juice.

The temperature of 190° F. proved to be the least efficient of all extraction temperatures tried. The soluble solids content of the combined first and second extracts was only 54.1 per cent of that of the two, 15 minute, 212° F. extractions, and the jelly strength was but 38 per cent as high. Since the yield of juice was approximately the same, the total jelly yield was only 35 per cent as great.

In respect to juice yield, jelly strength and soluble solids content, the first of the 15 minute extracts at 240° F. surpassed any of those taken at the 15 minute at 212° F. period; the second, however, produced a lower yield so weak in sugar supporting capacity, that the two combined could produce but 83 per cent as much jelly as the first two 15 minute extracts at 212° F.

c. Effects of Added Acid

Tartaric acid was added to the first extraction water in quantities equal to 0.2 or 0.4 per cent of the combined weight of apples and extraction water, and the same amounts added again to the water used in the second extraction. The period employed was 15 minutes at 212° F. using a fruit to water ratio of 3 to 3 by weight. Of all methods attempted, this was most efficient, as can be judged by the comparison of the results with those obtained in the

same extraction without the added acid. Figure III and Table III. No great difference was apparent between the yield and strength of the combined juices extracted with 0.2 per cent acid and those in which 0.4 per cent was used; a slight advantage lay with the 0.2 per cent extraction. The greatest gain over the non-acid extractions occurred in the first extracted juice; the first 0.2 per cent extract was 27.4 per cent, and the first 0.4 per cent extract was 18.7 per cent higher in jelly strength than the first non-acid juice. The second 0.2 per cent extract was slightly weaker, and the second 0.4 per cent extract was approximately 30 per cent stronger than the corresponding non-acid extract. The total jelly production of the combined 0.2 per cent acid extracted juices was 15 per cent greater than that of the non-acid juices; the jelly yield of the 0.4 per cent acid extractions was slightly less than that of the 0.2 per cent extractions. A deeper reddish "apple" color and a more pronounced apple flavor than in the non-acid extractions were distinctly noticeable in the acid extracted juices and in the jellies made from them.

Figure III. Comparison of Two Extracts for 15 Minutes at 212° F. Using
Fruit to Water Ratios of 3:3, 3:2 and 3:4, and Ratios of
3:3 Containing 0.2 and 0.4 per cent Tartaric Acid.

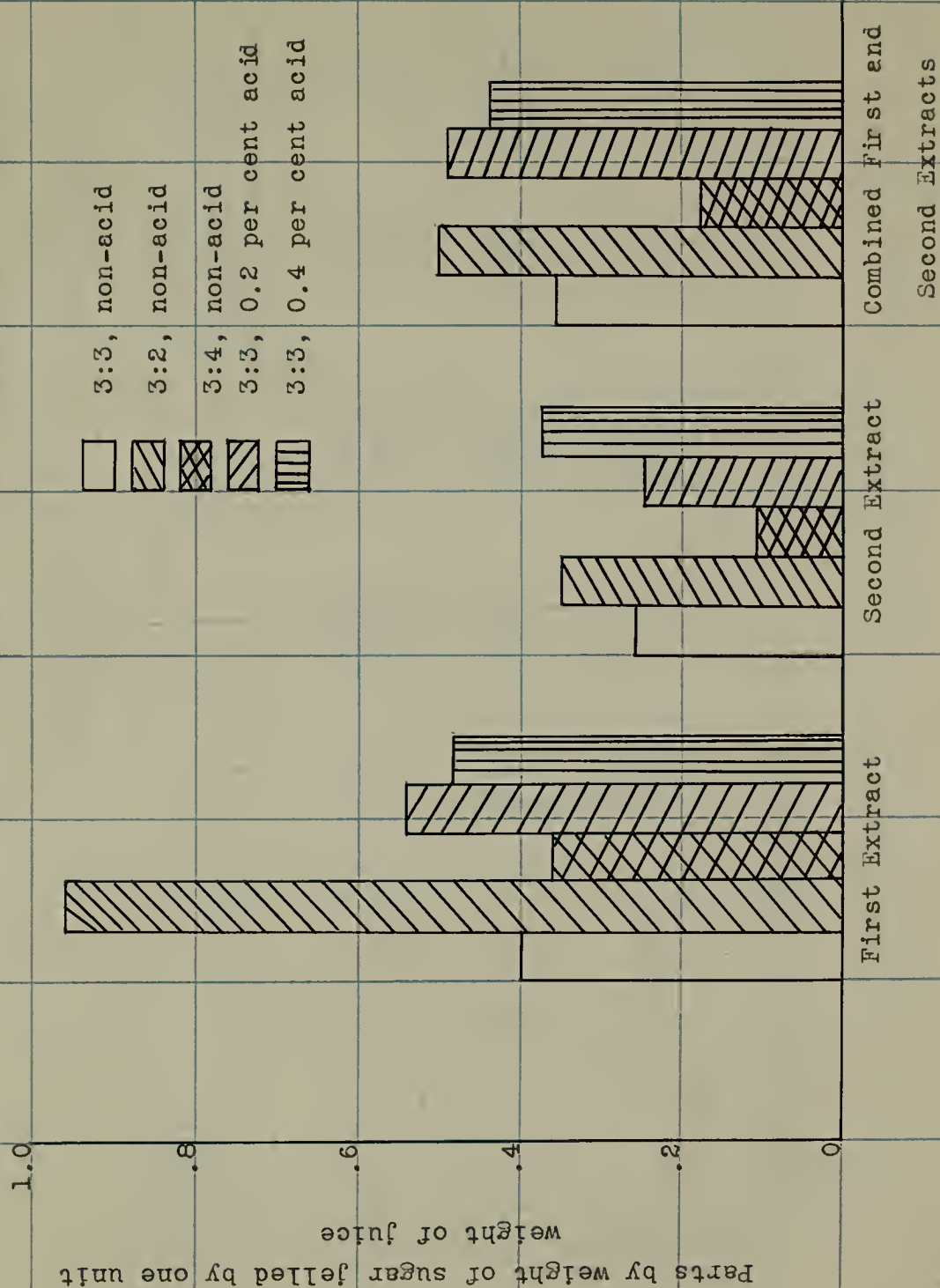


Table III. Comparison of 15 Minute Acid and Non-acid Extractions at 212° F., Using a Fruit to Water Ratio of 3 Pounds to 3 Pounds

15 Minute Extractions	Relative Viscosity	Yield of Juice	Jelly Strength*	Parts of Jelly per One Part Juice	Total Jelly Yield
					ounces
First	No acid				
	0.2 per cent	3.36	.3977	.5935	33.09
	0.4 per cent	4.48	.5477	.8174	40.91
Second	No acid	3.97	.4883	.7288	45.26
	0.2 per cent				
	0.4 per cent				
First Two Combined	No acid	2.58	.2600	.3880	19.92
	0.2 per cent	2.51	.2457	.3667	16.78
	0.4 per cent	3.23	.3771	.5628	24.57
First Two Combined	No acid				
	0.2 per cent	3.08	.3525	.5258	56.58
	0.4 per cent	4.02	.4912	.7331	68.10
First Two Combined	No acid	3.66	.4423	.6601	65.68
	0.4 per cent				

* Jelly strength is expressed as parts by weight of sugar jelled by one unit weight of juice.

d. Effects of Variations in Fruit to Water Ratios

The 3:3, fruit to extraction water ratio, was varied to 3:2 and 3:4, and extractions were made at 212° F. for 15 minutes. The 3:4 extraction was a failure, for although the juice yield was high, its jelly strength was low, and the total jelly yield was but 66 per cent of the yield from the 3:3 ratio at the same temperature and time. The 3:2 ratio was much more satisfactory; the sugar supporting values of the extracts were relatively very high, but the juice yields were low, so that the total jelly yield was actually 10 per cent less than in the 3:3 extraction. The results are pictured in Table IV and Figure III.

The ultimate test of any method for the extraction of a pectinous juice, however, is the total amount of jelly of a definite grade which the extracted juice will produce. In this experiment 100 grams of the extracted juices were added to the required amount of sugar, dependent upon the relative viscosity of the juice and computed by Baker's (1934) formula. The solution was put in a small aluminum pan and boiled until the sugar concentration reached 67 per cent. The jelly was then immediately poured into two-ounce glasses, and allowed to stand for 24 hours. In all but one or two instances the resulting

Table IV. Comparison of Extractions Made at 212° F. for 15 Minutes Using a Fruit to Water Ratio of 3:2, 3:3 and 3:4 Parts by Weight

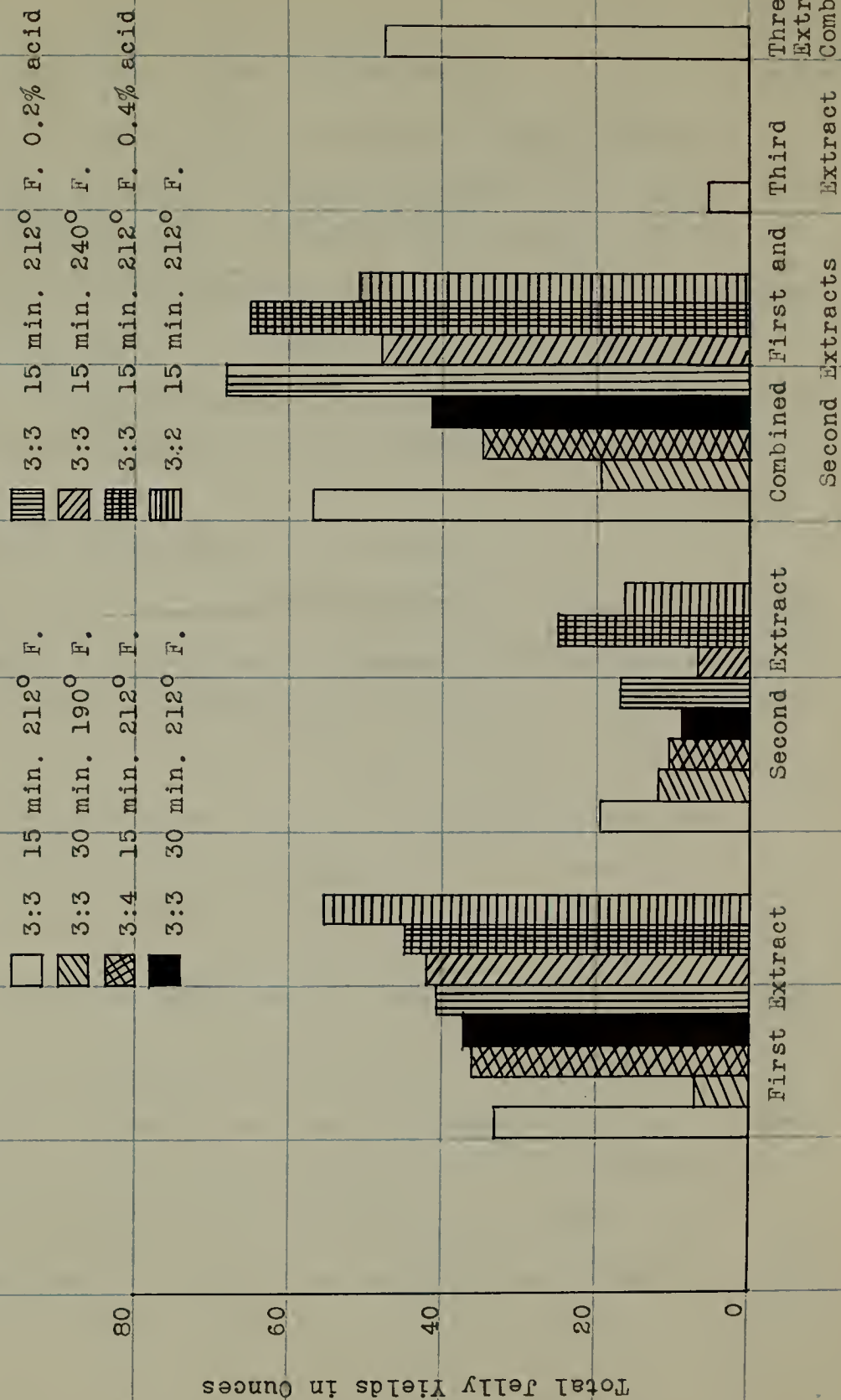
Extractions	Ratio of Fruit to Water	Relative Viscosity	Yield of Juice	Jelly Strength*	Parts of Jelly per One Part Juice	Total Jelly Yield
			ounces			ounces
First Extract	3:3	3.36	55.75	.3977	.5935	33.09
	3:2	10.00	38.5	.9663	1.422	54.74
	3:4	3.07	70.36	.3507	.5234	36.83
Second Extract	3:3	2.58	51.35	.2600	.3880	19.92
	3:2	3.07	30.7	.3507	.5236	16.07
	3:4	1.94	60.72	.1114	.1622	10.09
First and Second	3:3	3.08	107.61	.3523	.5258	56.58
	3:2	4.26	67.25	.5094	.7603	51.13
	3:4	2.21	131.2	.1793	.2676	35.09

* Jelly strength is expressed as parts by weight of sugar jelled by one unit weight of juice.

jelly was of a fairly firm consistency that retained its shape on being removed from the glass. If the 100 gram portion formed a good jelly, then the total jelly which could be obtained from the whole batch of extract was calculated by figuring the amount of sugar which would be required to give a content of 67 per cent sugar in the jelly made from the extract.

In Figure IV are pictured the yields of jelly obtained from the extracts of three pounds of apples under the various extraction conditions already described. In only one instance, that of the 3:2 extraction for 15 minutes at 212° F., was the jelly yield from the first two extracts combined less than either the first or second singly. The three best jelly yields were obtained from the combined first and second 3:3, 15 minute, 212° F. extractions. The best of those was the extract to which 0.2 per cent tartaric acid was added, the next the one containing 0.4 per cent tartaric acid, and third the non-acid extraction. Extraction at a temperature of 190° F. gave the poorest results, and the 30 minute extraction at 212° F., the 240° F. extraction, and the 3:4 and 3:2 fruit to water ratios were intermediate. Third extractions yielded but little jellying pectin and only served to lower the total jelly yields.

Figure IV. Average Total Jelly Yields Obtained from Various Extractions
of Fresh Apples



3. Pomace Extractions

In attempting to determine the optimum methods for obtaining pectinous extracts from dried apple pomace, the same variations of time and temperature as used in the fresh apple extractions were employed, and the effects of added tartaric acid were noted, but the ratios of pomace to extraction water varied from 1:6 to 1:16 by weight. The reason for this large proportion of extraction water to the raw material is that the pomace, being dried to a moisture content of less than 8 per cent, absorbs much water during the first extraction, greatly decreasing the yield and enhancing the danger of scorching. In the 1:6 extractions one-half pound of pomace was added to 3 pounds of water; in the 1:12 extractions one-third of a pound of pomace was used with 4 pounds of water, and in the 1:16 extraction one-quarter pound of pomace was extracted in 4 pounds of water.

a. Effects of Variations in Time and Fruit to Water Ratio

In the pomace, as in the fresh apple extractions, no third extraction at any time or temperature proved of any value, but served only to lower the jelly grade of the juice and greatly reduce the total jelly yield. In summarizing the results, the jelly strength values of the

different extracts proved of little comparative worth, due to the large differences in juice yields obtained by the various methods attempted. Thus, the jelly strength of the combined first and second juices extracted from a pomace to water ratio of 1:12 at 212° F. for 15 minutes was but 38.8 per cent as high as that of the two combined juices extracted under the same conditions from a 1:6 pomace to water ratio (Figure V). But since the yield of juice in the former case was 2.6 times as great as in the latter (Table V) the ultimate total yield of jelly was slightly greater (Table V and Figure VI). The true criterion for the value of an extraction method, especially in pomace extractions, is therefore, the total jelly yield obtained. Total jelly yields obtained under varying conditions are compared in Figure VI.

b. Effects of Added Acid and Variations in Temperature

The combined first and second extracts in all cases yielded the largest amounts of jelly. Of the different pomace to water ratios attempted, the 1:12 was most efficient, the 1:16 least efficient, and the 1:6 intermediate. The 30 minute period in all cases produced a lower yield of juice of a poorer jelly grade than the corresponding 15 minute period. The extraction made at 190° F. was a total

Figure V. Comparison of Jelly Strength of Successive Extractions of Pomace for 15 and 30 Minutes at 212° F. Using Pomace to Water Ratios of 1:6, 1:12 and 1:16

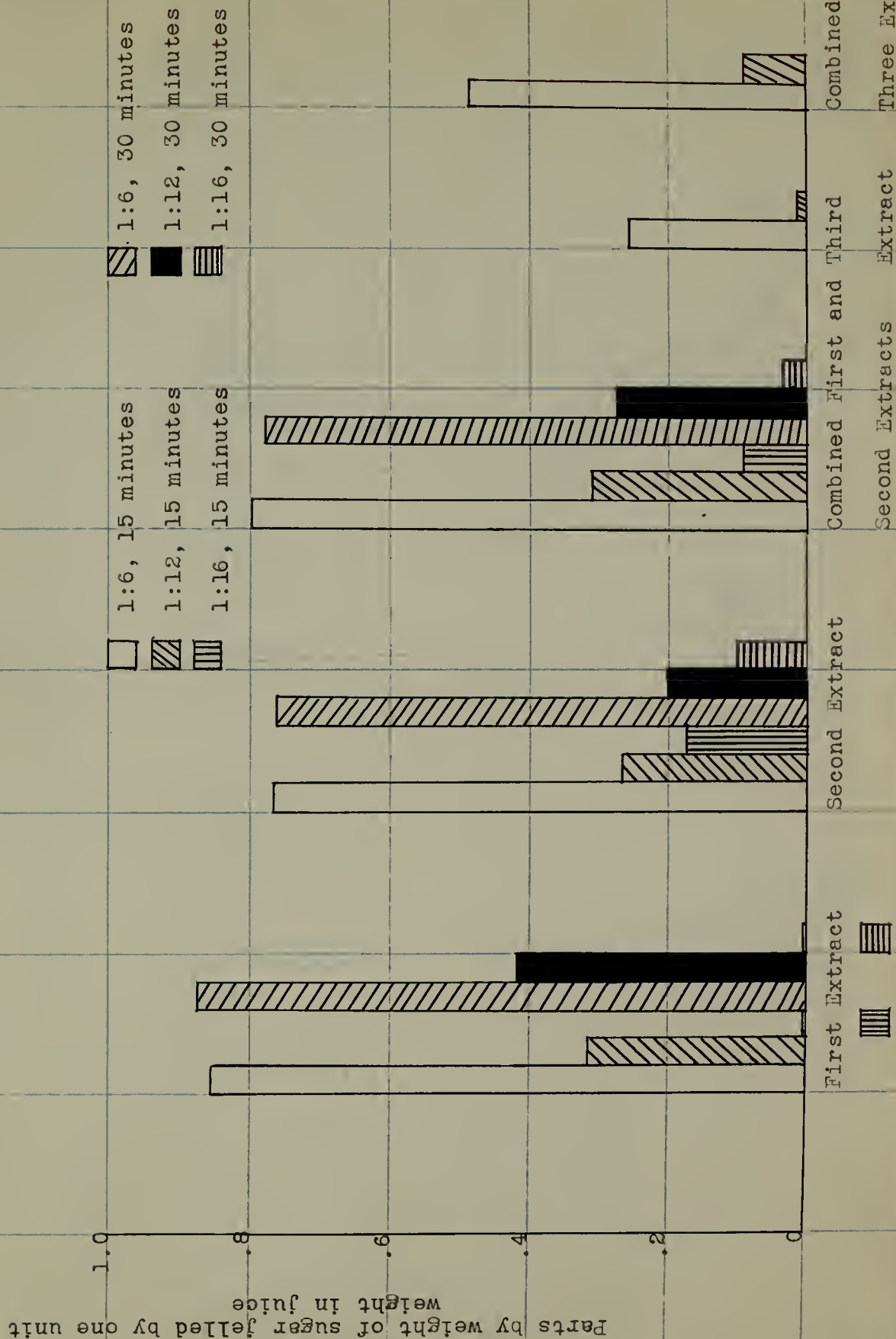


Figure VI. Total Jelly Yields from Successive and Combined Extractions of Pomace Obtained Under Varying Conditions.

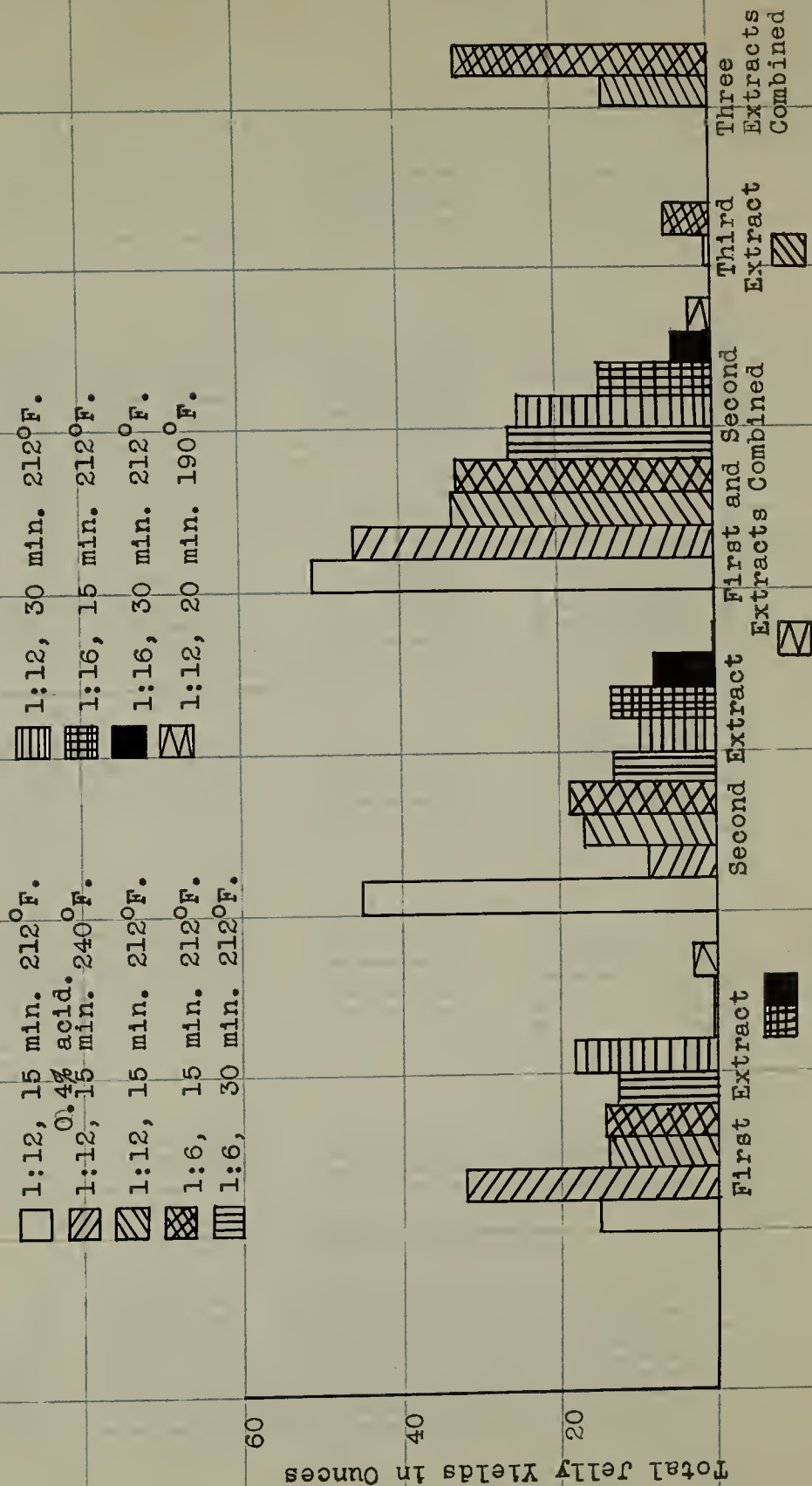


Table V. Comparison of Successive Extractions of Pomace for 15 and 30 Minutes at 212° F. Using Pomace to Water Ratios of 1:6, 1:12 and 1:16

First Extraction	Pomace to Water Ratio	Relative Viscosity	Soluble Solids per cent	Juice Yield ounces	Jelly Strength*	Total Jelly Yield ounces
15 Minutes	1:6	8.10	7.00	11.50	.8565	14.89
15 Minutes	1:12	2.89	4.62	30.37	.3192	14.38
15 Minutes	1:16	1.63	2.10	51.40	-	-
30 Minutes	1:6	8.42	6.50	10.22	.8767	13.27
30 Minutes	1:12	3.60	3.20	29.50	.4217	18.76
Second Extraction						
15 Minutes	1:6	6.91	2.6	16.25	.7736	18.75
15 Minutes	1:12	2.66	1.92	41.37	.2759	17.03
15 Minutes	1:16	2.10	1.12	52.80	.1767	13.92
30 Minutes	1:6	6.74	1.84	12.17	.7606	13.81
30 Minutes	1:12	2.30	1.12	33.22	.2001	9.91
Combined Extractions						
15 Minutes	1:6	7.24	4.25	27.72	.7980	33.01
15 Minutes	1:12	2.83	2.65	73.00	.3082	33.58
15 Minutes	1:16	1.88	1.42	103.12	.0950	14.61
30 Minutes	1:6	7.07	3.92	22.41	.7856	26.25
30 Minutes	1:12	2.65	1.84	62.10	.2732	25.32

* Jelly strength is expressed as parts by weight of sugar jelled by one unit weight of juice.

Table VI. Comparison of 15 Minute Extractions at Various Temperatures, Using a Pomace to Water Ratio of 1:12 by Weight

Extraction	Extraction Temperature	Relative Viscosity	Soluble Solids	Juice Yield	Jelly Strength*	Total Jelly Yield
	degrees F.		per cent	ounces		ounces
First Non-acid	212	2.89	4.62	30.37	.3192	14.38
First Non-acid	240	3.91	5.20	23.75	.9052	32.08
First Non-acid	190	1.77	3.25	34.10	.0635	3.23
First 0.4 per cent Acid	212	3.54	3.55	52.20	.4249	15.98
Second Non-acid	212	2.66	1.92	41.37	.2759	17.03
Second Non-acid	240	2.07	2.23	42.00	.1452	9.10
Second Non-acid	190	1.59	1.50	46.75	.0076	.514
Second 0.4 per cent Acid	212	7.08	2.12	38.76	.7863	45.46
Combined Non-acid	212	2.82	2.65	73.05	.3082	33.58
Combined Non-acid	240	3.86	3.40	65.75	.4699	46.11
Combined Non-acid	190	1.65	2.70	80.42	.0269	3.22
Combined 0.4 per cent Acid	212	4.40	3.05	63.85	.5383	51.29

* Jelly strength is expressed as parts by weight of sugar jelled by one unit weight of juice.

failure, and the 212° F. 0.4 per cent acid extraction produced juices giving the largest yield. One difference noted between fresh apple and pomace extractions was that the pomace at 240° F. yielded more juice of a higher pectin grade than under otherwise similar conditions at 212° F., whereas fresh apples at 240° F. yielded less juice of a lower grade than corresponding extractions at 212° F.

A standing period of 10 minutes during which the pomace soaked in the extraction water at about 50° F. before the extraction temperature was attained proved of no value whatever, the juice yields, soluble solids content and jelly grade being exactly the same as in the corresponding extraction without the standing period. The only difference between extractions of pressed and unpressed pomace was in the soluble solids content, the pressed pomace containing on an average 75 per cent less than the unpressed pomace.

C. Clarification

1. General Discussion

Pectinous juices extracted by heat from apple pomace, and more particularly from fresh apples, contain in colloidal suspension a large amount of very finely divided material, besides the pectin, which is not removed when

the juices are strained through cheesecloth or through a felt or flannel filter. This suspended material, mostly starch and proteins, causes a heavy turbidity or cloudiness in the extract which becomes more pronounced when the extract is concentrated. Such a cloudy juice is unattractive and cannot be used in jellies without causing a scummy surface and loss of clarity. After one or two months storage of the extracts, especially at room temperatures, the suspended material settles down and becomes a muddy, thick, and sometimes even gelatinous sediment on the bottom of the container. In this experiment great difficulty was experienced in measuring the viscosity of the concentrated extracts, and the results obtained in these measurements were often irregular. It was observed in certain instances in which an extract showed a viscosity much above the average for juices extracted and concentrated under similar conditions, that the viscosity might not be due to pectin alone, but also partly to the suspended starchy materials. This observation may have been the correct explanation of the matter, for when jellies were made of some of these concentrated juices of unreasonably high viscosity, it was found that their relative viscosity values were not true indications of their jelly strength, and that the amounts of sugar

necessary to add according to Baker's (1934) formula were not jellied, being in all cases excessive. For these reasons attempts were made by various methods to clarify the pectin extracts.

2. Clarification Methods

a. Filtration

Filtration through cheesecloth and through felt and flannel bags being unsuccessful, an attempt was made at clarification by passing the extract into a filter flask through "Filter-Cel" held in a Buchner funnel by a circular piece of canton flannel covering the funnel bottom. Suction was provided by an aspirator attached to a sink faucet and connected to the flask with a heavy rubber hose. By this method, some of the turbidity was removed, but the juices still remained cloudy, and a more or less heavy layer of sediment settled from them after about two weeks storage.

b. Yeast Fermentation

Half-liter amounts of unconcentrated fresh apple extract of known viscosity and soluble solids content were placed in Erlenmeyer flasks of about one liter capacity, inoculated with pure cultures of four different

strains of yeast, the tops of the flasks plugged with cotton, and the whole incubated at room temperature for from 5 to 7 days, or until fermentation had ceased. The fermented extracts were then filtered through "Filter-Cel" in a Buchner funnel, and tested again for viscosity and soluble solids content.

The juices treated with yeast in this manner possessed a dull, translucent sort of clarity, but lacked sparkle and brilliance, and after several weeks storage a thin layer of sediment settled from them. The average relative viscosity value before the yeast fermentation, 3.52, had fallen to 3.23 when the fermentation had ceased. This difference is equivalent to a loss in jelly strength of the extract of .0466, a negligible amount which seems to indicate that yeast does not destroy pectin. The soluble solids content fell from an average of 5 per cent to 0 per cent as determined with a Brix saccharimeter.

Clarification by yeast fermentation was entirely unsatisfactory. In eight attempts, using pure cultures of four different strains of yeasts, not a single extract was clarified more successfully than by filtration alone.

c. Enzyme Treatment

Since the cause of the cloudiness was believed to be largely colloidal starch, it was decided to attempt the conversion of the starch into more soluble sugars by enzyme action. Starch is converted by the enzyme diastase into maltose. For this purpose Medicinal diastase of malt, U.S.P. was purchased, and two preparations intended expressly for purposes of fruit juice clarification in commercial practice were furnished by their manufacturers, "Protozyme Px" by Jacques Wolf & Co., and "Clarex" by the Takamine Laboratory, Inc. Specifications for the use of Protozyme Px were followed closely, and its action compared with that of the other two preparations under the same conditions. Later, variations were introduced into the methods by using all three.

According to Bayliss (1925) and Waldschmidt (1929), the end products of enzyme action do not depend upon the concentration of the enzyme present. However, the rate of the reaction does depend partly on the concentration and also on the temperature at which the reaction takes place. The greater the concentration of the enzyme, and the higher the temperature at which the conversion occurs, up to a certain degree, the quicker the reaction. Temperatures above 60° C. readily destroy diastase. The

producers of Protozyme Px recommended as the optimum conditions for the use of their preparation a concentration of 0.1 per cent on a weight basis, and a temperature of 120° F. They state that conversion will occur at this temperature in 40 to 75 minutes, depending on the amount of starch present in the solution, or at a temperature as low as 74° F., the reaction being slower at the lower temperatures. If the juice is to be filtered after treatment, it is not necessary to extract the enzyme from the substrate carrying the enzyme usually bran or a similar substance. However, it will go into solution more quickly if it is first leached for 15 to 30 minutes in about four times its weight of the juice. This procedure was followed in all treatments in this experiment. The enzyme was destroyed at the end of each conversion period by heating quickly above 160° F. The end point of the reaction should be indicated by the "characteristic reddish-brown color" appearing when the sample is treated with a dilute iodine solution. This color reaction was indistinguishable in the treatment of the apple pectin extracts, however, perhaps because of their natural reddish-brown color.

Extracts of fresh apples, pomace and pressed pomace were treated (both unconcentrated and after concentration

to 1/5 of their original weight) with all three enzyme preparations for periods varying from 35 to 110 minutes, and at temperatures of 120° F and 90° F. The extract to be treated was poured into an aluminum pan and raised to the desired temperature by immersing it in a temperature-controlled water bath. The leached enzyme was then added and the whole held at the same temperature for the duration of the conversion period, at the end of which it was heated to boiling temperature and twice filtered through Filter-Cel in a Buchner funnel. Soluble solids and viscosity measurements were taken at 76° F. before the enzyme treatment, and immediately after the final filtration.

The results obtained in the clarification of pectinous extracts by treatment with medicinal Diastase Merck, with the Jacques Wolf product "Protozyme Px," and with the Takamine product "Clarex," were equally very satisfactory. In general, it was observed that fresh apple extracts required a longer time for conversion than pomace extracts, and that clarification is more complete and more easily accomplished on the unconcentrated extracts than on the extracts after concentration. Thus, unconcentrated pomace extracts which were treated for 50 minutes at 120°F. filtered to a clear, brilliant liquid, and no sediment had formed on the bottom of the

container after three weeks storage at room temperature. Extracts which were concentrated to $1/5$ of their original weight and then treated for 50 minutes with enzymes equal to 0.1 per cent of the weight of the extract before concentration were slightly cloudy, and showed a slight sediment after three weeks storage. A period of 75 minutes at the same temperature was required to accomplish as complete conversion as the 50 minute period for the unconcentrated extract. For unconcentrated fresh apple extracts, any period of enzyme treatment less than 70 minutes at 120° F. and for concentrated fresh apple extracts any period less than 90 minutes at 120° F. failed to give a perfectly clear juice from which no sediment settled during three weeks storage. Another factor in favor of enzyme treatment before concentration is the difficulty of filtering a concentrated pectin extract as such a viscous liquid filters very slowly, almost drop by drop when cold, even though a high vacuum be drawn on the filter flask. When hot it flows faster, but boiling or foaming up in the flask and resultant loss of liquid is very likely to occur as filtration continues and the vacuum in the flask increases.

Under conditions where it is impossible or impractical to maintain an exactly regulated temperature of 120° F., conversion may be just as satisfactorily accomplished at

temperatures of from 80° F. to 90° F. by allowing a longer time for the completion of the reaction. The enzyme treatment at 90° F. and subsequent filtration resulted in a well clarified juice when a 90 minute period was allowed for the reaction in unconcentrated pomace extracts, and when a 110 minute period was allowed for unconcentrated fresh apple extracts. In some cases in which the reactions, both at 120° F. and at 90° F., were allowed to continue for shorter periods complete conversion, as judged by the clarity of the filtered juice, was accomplished; in others, clarification was imperfect, varying degrees of turbidity and sediment remaining. The periods noted in Table VII constituted the shortest reaction times allowed for the different temperatures, in which there were no failures.

Difficulty was encountered in attempts to clarify extracts of pressed pomace, and acid extracted juices. The clarified unconcentrated pressed pomace extracts were fairly clear, but when the same had been concentrated after clarification they developed within several days an opaque greenish-black discoloration. It was believed that this might be due to imperfect filtration, but after refiltering the extract retained the discoloration. The unconcentrated acid extracted juices were not as completely clarified as the juices extracted

Table VII. Time Required for Enzyme Clarification of Pectin Extracts Treated at 120° F. and 90° F.

Extraction	Time at 120 F.	Time at 90 F.
	minutes	minutes
Concentrated Pomace	75	
Unconcentrated Pomace	50	90
Concentrated Fresh Apple	90	-
Unconcentrated Fresh Apple	70	110

without acid, but remained rather cloudy. This cloudiness was more pronounced when the extracts were concentrated after clarification. Within 24 hours after concentration and bottling, approximately a quarter of an inch of a heavy, white, partly crystalline sediment had settled to the bottom of the half-pint jars used as containers, and the supernatant liquid remained cloudy. No acid extractions were successfully clarified. No explanation was found for either of these difficulties.

Viscosity measurements were made on all extracts before enzyme treatment and after the final filtration, and in no case was the viscosity lowered by the action of any of the three preparations used. The average gain in soluble solids content during enzyme treatment was 0.75 per cent in the unconcentrated extracts, and 1.1 per cent in the concentrated extracts. Some of the clarified extracts were stored unconcentrated, while others were first concentrated 1 to 5. All retained their brilliance and clarity after concentration. The results of the various times and temperatures of the conversion periods used on the different extracts are given in Table VIII.

Table VIII. Results of Enzyme Clarification of Apple Pectin Extracts with Diastase Preparations in a Concentration of 0.1 per cent by Weight

Extract	Conversion Temperature	Period Time	Pressed Pomace	Pomace	Fresh Apples
Not Concentrated	120° F.	35 Minutes	-	Slightly cloudy; slight sediment	Cloudy, sediment
		50 Minutes	Fairly clear, but dark; slight sediment	Very clear; no sediment	Slight cloudy; slight sediment
		70 Minutes	-	-	Very clear; no sediment
Concentrated	120° F.	50 Minutes	-	Slightly cloudy; sediment	Very cloudy; heavy sediment
		75 Minutes	Very cloudy, heavy sediment	Very clear, no sediment	Slightly cloudy; sediment
		90 Minutes	Very cloudy and dark; heavy sediment	-	Fairly clear; slight sediment
Not Concentrated	90° F.	75 Minutes	-	Cloudy, sediment	Cloudy; heavy sediment
		90 Minutes	-	Very clear; no sediment	Fairly clear; no sediment
		110 Minutes	-	-	Very clear; no sediment

D. Preservation

In order to determine the deterioration which the pectinous juices might suffer during storage, each extract was bottled in half-pint jars and preserved by thermal treatment or by the addition of sodium benzoate. Preservation of the extracts, particularly of those which had been concentrated, was discovered to be a simple matter. This was probably due to their acidity, the unconcentrated juices having an average pH slightly above 4.0, and the concentrated juices a pH between 3.5 and 4.0.

1. Required Thermal Treatment

Of the unclarified extracts, those which remained unconcentrated were filled into clean half-pint jars immediately after extraction, and those which were concentrated 1 to 5 immediately after concentration. In all cases the jars were immediately processed either at 212° F. for 10 minutes or pasteurized at 170° F. for 10 minutes in a temperature controlled water bath, or sealed and stored without the further application of heat. The clarified extracts were heated to approximately 200° F. after the final filtration, filled into the jars immediately and then given one of the same treatments as the unclarified juices.

In none of the juices which were pasteurized or preserved was there any sign of fermentation or mold growth after four months storage either at 40° F. or at room temperature. The concentrated extracts which were filled hot and sealed without heat treatment all kept without spoilage, but among the twenty-three jars of unconcentrated extract preserved in the same way, eleven showed a surface mold growth within the first three weeks storage. There was no apparent difference between the keeping quality of fresh apple extracts and that of pomace extracts, or between clarified and unclarified extracts.

2. Benzoate Preservation

The maximum concentration of sodium benzoate in foods allowable by law is 0.1 per cent by weight. In the attempts at preservation of the pectin extracts by the use of benzoate, concentrations of .05 per cent and .1 per cent were used. The benzoate was added to the warm concentrated and unconcentrated juices, thoroughly stirred into complete solution, and the juices immediately bottled. Within ten days storage at room temperature both concentrated and unconcentrated extracts containing .05 per cent benzoate had a thin pellicle of mold growth on their

surfaces. The viscosity of these extracts had not been reduced. A sodium benzoate content of 0.1 per cent did prevent mold growth in the concentrated extracts, and in about half of the unconcentrated extracts for the three weeks during which the juices were in storage. However, after the first ten days, the viscosity values of all these extracts, both concentrated and unconcentrated, had been reduced by approximately one-half, seriously impairing their jelly strength. In general, preservation by the addition of sodium benzoate was unsatisfactory and is not recommended.

E. Storage Life

1. General Discussion

There is no material in the voluminous literature on pectin which pertains to the effects of storage under various conditions on jellying pectin. It has been the experience of some, however, who have attempted the preservation of pectinous extracts, either as a jelly stock or as a pectin concentrate, that the pectin undergoes during longer or shorter periods of storage a deterioration in quality; that is, that it loses jelly strength, or in other words, that its sugar supporting

capacity is decreased. It was therefore undertaken as a part of the problem of pectin extraction and preservation, to determine as far as possible the extent of this deterioration, and to discover under which storage conditions it is least serious.

In this experiment pectinous juices of known viscosity were stored, clarified and unclarified, concentrated and unconcentrated, at a temperature of 40° F. and at room temperature, 76° F. At the end of one, two and three months storage they were examined for jelly strength, using Baker's (1934) jellmeter and formula. The viscosity measurements of the unclarified concentrated extracts were in several instances obviously inaccurate and not truly indicative of the jell strength of the extract; in these cases the viscosity was greater after one or two months storage than when the extract was first made. When jellies were made of these extracts, sugar was added according to their indicated jell strength, and was in no case jelled, being always excessive. As already explained in the discussion on the clarification of pectinous extracts, these inaccuracies were probably due to a coagulation of the starchy substances which were not removed by filtration immediately after extraction. When an extract which showed a gain in viscosity during storage was filtered

through Filter-Cel in a Buchner funnel, and again measured in the jellmeter, its viscosity measurement was close to the average determined for other juices extracted and stored under similar conditions. When the filtered extract was made into jelly, the viscosity measurement was found to be the true indication of the jell strength of the juice, for in all cases the amount of sugar required by Baker's (1934) formula was jelled. No similar difficulty was experienced in viscosity measurements on clarified extracts.

2. Storage Life of Unclarified Extracts

During two months storage of the unclarified extracts, loss of jelly strength was greatest in the unconcentrated pomace extract; in all cases the extracts stored at 40° F. lost less jelly strength than those kept at room temperature. Thus, the average jell strength value of the unconcentrated pomace extracts kept at 76° F. showed a loss of 64 per cent after two months, while the same extracts kept at 40° F. lost an average of 37 per cent of their original strength during the same period. The concentrated pomace extracts during two months lost 4 per cent of their original strength at 76° F., but only 2 per cent at 40° F. The difference between the original jelly strength lost

at these two temperatures was plainly evident but less pronounced in the fresh apple extracts. The figures are shown in Table IX.

An indication of the rate of deterioration during storage of the jellying pectin can also be obtained from the figures of Table IX. The concentrated extracts, both pomace and fresh apple, show a greater average decline during the second months than during the first; the difference is greater in the extracts kept at 76° F. than in those stored at 40° F. On the other hand, the unconcentrated extracts, both fresh apple and pomace, kept at both cold storage and room temperatures, showed the greatest decline in jelly strength during the first month, the average loss being approximately twice as great as during the second month. Only the concentrated fresh apple extracts were stored for three months, and their average loss during the third month was less than during the second. As far as may be judged from the results of this experiment, it would seem that deterioration of unclarified pectinous extracts is a more or less serious matter; furthermore, the greatest deterioration during storage of concentrated extracts occurred during the second month, while unconcentrated extracts show the greatest loss during the first month.

Table IX. Average Deterioration During Storage of Unclearified
Extracts Indicated by Loss of Jelly Strength

Extract	Storage Temperature Degrees F.	Jelly Strength *				
		Original	After Storage for 30 days	Per cent Loss in Storage for 60 Days	After Storage for 90 Days	Per cent Loss in Storage for 90 Days
Fresh Apple Concentrated	76	1.2885	1.2825	0.47	1.2623	4.64
	40	1.1717	1.1585	1.13	1.1465	2.16
Pomace Concentrated	76	1.1647	1.1361	1.60	1.1127	4.45
	40	1.2132	1.2062	0.58	1.2025	1.71
Fresh Apple Not Concentrated	76	.3867	.3723	3.73	.3673	5.02
	40	.3667	.3783	2.17	.3739	3.06
Pomace Not Concentrated	76	.3369	.1863	44.11	.1220	63.80
	40	.3899	.2837	27.24	.2436	37.53

* Jelly strength is expressed as parts by weight of sugar jelled by one unit weight of juice.

3. Storage Life of Clarified Extracts

In this discussion only the extracts clarified by enzyme treatment are considered, since all other methods failed. Although none of the clarified extracts remained in storage longer than one month, the viscosity measurements at the end of that time gave a good indication of the effects of clarification on the storage life of pectinous extracts. Not only the completely clarified juices, but also those upon which the attempts at clarification were more or less unsuccessful were stored. Some of these were equally as cloudy and after storage contained a layer of sediment as deep as any unclarified extract. Others were less cloudy than the unclarified extracts, but still were not totally clear, remaining rather turbid and opaque. A "completely" clarified juice is one which has no turbidity and no sediment, but is absolutely clear and almost transparent, one that "sparkles" when held to the light.

In general, it was found that clarified extracts retained jell strength better, at least during the first month in storage, than did the unclarified. Apparently the storage temperature made no difference in the loss or retention of jell strength. In several instances, as shown in Table X, juices clarified in

Table X. Comparison of Deterioration of Representative Extracts Clarified Under the Same Conditions and Having the Same Initial Jelly Strength During Thirty Days Storage at 40° F. and at 76° F.

Extract	Storage Temperature	Original Jelly Strength *	Jelly Strength After 30 Days Storage	Per cent of	
				Original Jelly Strength Lost	During Storage
Degrees F.					
Tomace, Not Concentrated	76	.2720	.2720	0.00	
	40	.2720	.2720	0.00	
Pomace, Not Concentrated	76	.2952	.2498	15.38	
	40	.2952	.2498	15.38	
Fresh Apple, Not Concentrated	76	.2952	.2863	3.02	
	40	.2952	.2863	3.02	
Fresh Apple Concentrated	76	1.177	1.142	3.24	
	40	1.177	1.142	3.24	
Pomace, Concentrated	76	1.507	1.357	9.96	
	40	1.507	1.357	9.96	

* Jelly strength is expressed as parts by weight of sugar jelled by one unit weight of juice.

the same manner and stored at 40° F. and at 70° F. having the same initial viscosity, at the end of the month showed exactly the same loss. The loss of jelly strength of the various extracts did, however, to a certain extent coincide with the degree of completeness of clarification accomplished, the more complete the clarification the less the deterioration suffered by the extract, and viceversa. The completeness of clarification was not the only factor, however, for, as in the unclarified extracts, the average unconcentrated pomace extract lost a greater percentage of its original jelly strength than any of the others. There was no difference in the storage life of the extracts clarified under the same conditions with the different diastase preparations used; the determining factor seemed to be not the particular brand of diastase used, but the completeness of the clarification accomplished.

Table XI shows the average loss in jelly strength suffered during one month storage by extracts of pomace and fresh apples treated with enzyme under the different conditions which resulted in the varying degrees of clarification described. In all treatments a concentration of enzyme of 0.1 per cent of the weight of the unconcentrated extract was used, and the extracts were filtered after the conversion period as described in the

Table XI. Average Loss of Jelly Strength During Thirty Days' Storage of Extracts Treated with Enzyme to Varying Degrees of Clarification

Extract	Extent of Clarification of Extract	Original Jelly Strength* Storage	Jelly Strength After 30 Days' Storage	Per cent of Original Jelly Strength Lost During Storage
Pomace Not Concentrated	Very cloudy; heavy sediment Cloudy; slight sediment Very clear; no sediment	.2252 .2952 .2743	.1546 .2680 .2743	31.38 9.20 0.00
Pomace Concentrated	Very cloudy; heavy sediment Slightly cloudy; sediment Very clear; no sediment	.7146 1.0177 1.3120	.6050 .9287 1.309	15.34 8.26 .225
Fresh Apple Not Concentrated	Very cloudy; heavy sediment Slightly cloudy; sediment Very clear, no sediment	.4297 .4707 .4725	.3822 .4405 .4674	11.06 6.37 0.70
Fresh Apple Concentrated	Very cloudy, heavy sediment Slightly cloudy; sediment Very clear, no sediment	1.6295 1.2047 1.1668	1.4206 1.1563 1.1623	12.82 4.02 .395

* Jelly strength is expressed as parts by weight of sugar jelled by one unit weight of juice.

discussion on clarification. The figures of Table XI, when compared with those of Table IX, which shows the loss of jell strength during storage of unclarified extracts, clearly indicate the advantages of clarification of a pectinous extract before storage. Whether the minute deterioration suffered by completely clarified extracts during the first month in storage indicates an ultimate smaller total jelly strength loss than in the unclarified extracts, or merely a slower rate of deterioration, is open to conjecture. Whichever is the case, the advantage is still with the clarified extracts. When clarification was not complete, however, no advantage was gained; on the contrary, the initial loss of jelly strength inexplicably surpassed that of the unclarified extracts.

IV. DISCUSSION OF THE POSSIBILITIES OF HOME PREPARATION AND UTILIZATION OF APPLE PECTIN EXTRACTS

The extraction, clarification, and preservation of apple pectin extracts by the methods found most advantageous in the work done on this research problem is a fairly simple procedure, one which requires no great technical skill or training. The most difficult part of the preparation of the extracts is likely to be the clarification, because it requires the use of special materials and equipment, namely, an enzyme preparation, Filter-Cel, and a filter pump. For the housewife who desires to prepare relatively small amounts of pectin, sufficient only for her own general use, it might be more practical to omit the enzyme treatment and store the extracts unclarified, although such an extract will lose jelly strength during storage, and because of its cloudiness cannot later be used in the preparation of good quality products. But to the orchard owner and to the farm factory or cider-mill operator for whom the extraction of pectin provides a valuable use for otherwise waste products, an investment of the equipment and material required for clarification is essential. Without clarification his pectin is inferior and cannot compare with commercial extracts and con-

concentrates now on the market; clarified, it is their equal.

There are endless ways in which pectin extracts and concentrates can be utilized to advantage in the home; probably their most general use is in jam and jelly making. Pectin concentrates are used in the production of synthetic sugar-acid-pectin jellies artificially colored and flavored, but their more general home use is in bolstering the jelly strength of juices extracted from fruits of low pectin content. Pectinous concentrates can also be used to increase the jelly yield of an extracted juice and reduce the cost of the product; the more pectin present the larger the amount of sugar that will be jelled, and therefore the greater the yield. Furthermore, with a large amount of sugar present a small evaporation of water will be required to bring the sugar concentration to the desired amount, thereby reducing the cooking time and improving the color and flavor. However, the addition of too much extra pectin and sugar will mask the flavor of the finished jelly. Jams, conserves or marmalades of a naturally weak or watery consistency can be thickened by the addition of small amounts of pectin.

The amount of pectin necessary to add to any juice to be used for jellies can be most easily determined by viscosity measurements of the juice made with Baker's (1934) jellmeter. The jellmeter is cheap and easy to use, and, for the sake of convenience, with each is included a chart showing the amounts of sugar necessary to add to, and the final yield of jelly obtained from a unit weight of juices of varying viscosities. In this manner, a pectin concentrate, regardless of its viscosity, can be added to any juice until the viscosity of the juice reaches the desired level.

Jelly formation depends not only on the sugar and pectin concentration but also on the acidity, a pH below 4.0 being required for jellation. The pH of juices could not be determined in the home without the use of expensive, specialized equipment, and if added acid is needed, the proper amount can be determined only by trial. Tartaric acid is cheap, but if unavailable, lemon juice may be used.

Jelly, however, is only one of the many products in which pectin can be profitably used. In the home manufacture of candies, pectin may be used in making jelly centers and bases for dipped or coated pieces. When properly made such candies keep fresh without

becoming sticky, are tender and firm, and of clear and sparkling color.

Any kind of liquid sauce or fruit juice cocktail is benefitted by the addition of pectin. Sauces for puddings or ice cream dishes become thicker and heavier, conveying the impression of richness, when they contain pectin. Fruit juice cocktails receive added body and an improved physical appearance on the addition of pectin. A small concentration of pectin in beverages which contain solid material in suspension, such as tomato juice cocktails or chocolate flavored liquids, acts as a protective colloid and, according to Baker, has a definite stabilizing effect, increasing the resistance of the product to separation into layers of solid and liquid.

Pectin is believed to improve the texture, moisture holding capacity, and yield of baked food products, definitely slowing the staling process. Too much pectin in such products would decrease the total solids content, and, instead of slowing, would probably accelerate the rate of staling. The texture and time of set of meringues and frosting could probably be improved by the use of pectin. As a stabilizer pectin has a definitely established place in the commercial manufacture of ices and sherbets, improving their texture and body.

Since ices and sherbets are best adapted to home production of any of those products classified as "Ice Cream", another method for home utilization of pectinous concentrates is added to the already generous list.

Difficulty is often experienced in dissolving dried pectin preparations, the pectin having a tendency to clump together in a gelatinous mass. No such difficulty is encountered in the use of liquid concentrates; a thorough mixing always suffices. The limiting factor in the use of apple pectin concentrates is the flavor imparted by its addition. In jellies or jams this is not noticeable or objectionable, but in some of its other uses, such as in baked food products, ices and sherbets, and beverages, the apple pectin concentrate may cause a decided off-flavor if used in too large amounts. However, in such products the desired concentration is very low and probably would not affect flavor to a very noticeable degree.

The fact that an otherwise waste product can be utilized in the easy and inexpensive production of apple pectin concentrates, and the variety of opportunities for its utilization in the home would seem to make its preparation a profitable undertaking for the housewife, the farm and orchard owner, and the farm factory or cider-mill owner.

V. SUMMARY AND CONCLUSIONS

1. Determination was made of the optimum conditions for the extraction of pectinous juices from cull and grade C Baldwin apples, and from apple pomace from Baldwin and King apples. The extraction conditions considered varied as to fruit to water ratio, time and temperature of extraction, and the addition of organic acids.
2. Determinations of pectin were not made by analytical methods; instead measurements were taken of relative viscosity, of which, according to Myers and Baker (1927) jelly strength and dependent jelly yield are functions. The method proved very successful, the viscosity being in all cases a relatively accurate indication of the jelly strength or sugar supporting capacity of the pectinous extracts.
3. In fresh apple extractions a fruit to water ratio of 3:3 and two 15 minute extractions at 212° F., with the addition of 0.2 per cent by weight of tartaric acid produced a combination of large juice yield and high jell strength, which resulted in the greatest total jelly yield.
4. The addition of 0.4 per cent of tartaric acid to the extraction water was slightly less efficient under

otherwise similar extraction conditions than a concentration of 0.2 per cent acid. The non-acid extraction under conditions the same as employed in the acid extractions was less favorable for jelly yield than either of the acid extractions.

5. Three extraction periods, two periods of 30 minutes each, fruit to water ratios of 3:2 and 3:4 and extraction temperatures of 240° F. and 190° F. were all far less efficient than the acid extraction. The last mentioned resulted in a total jelly yield approximately 30 per cent less than the 0.2 per cent acid extraction.
6. In pomace extractions the same variations as in fresh apples were employed, but the pomace to water ratios were 1:6, 1:12 and 1:16. Two 0.4 per cent acid extractions of one part pomace in 12 parts water, for 15 minutes at 212° F. produced a juice having the largest jelly yield. The yield obtained from the same pomace to water ratio twice extracted at 240° F. for 15 minutes without the addition of acid was only 10 per cent smaller.
7. A third extraction served only to seriously reduce the total jelly yield, except in the 15 minute 212° F.

extraction of a pomace to water ratio of 1:6; the yield from the three combined extracts in this case was only 3 per cent less than that obtained from the first two combined.

8. From two extractions of a ratio of 1:12 a jelly yield was obtained only 2 per cent greater than from two extractions of a 1:6 ratio under otherwise similar conditions. The 190° extraction, the 1:16 pomace to water ratio and the 30 minute extraction all were less than 50 per cent as efficient as the acid extraction.
9. Methods of clarification of apple pectin extracts were attempted. The use of diastase preparations was found very satisfactory in removing cloudiness due to starchy material in suspension. Medicinal diastase, U.S.P. and two commercial preparations, "Protozyme PX" and "Clarase" succeeded equally in clarifying under similar conditions.
10. Although fermentation by yeasts did not reduce the viscosity of the pectin extracts, as a means of clarification it was unsuccessful.
11. Processing at 212° F. for 10 minutes or pasteurizing at 170° F. for 10 minutes were successful in all instances in effecting sterilization of the extracts

bottled in half pint jars. Of the extracts which were filled hot, sealed and stored without further thermal treatment, the concentrated kept unspoiled, but about 75 per cent of the unconcentrated showed a surface mold growth within three weeks.

12. Preservation with sodium benzoate was a failure. Addition of 0.05 per cent sodium benzoate failed to prevent surface mold growth after ten days. A concentration of 0.1 per cent sodium benzoate prevented mold growth in all concentrated extracts and in half of the unconcentrated extracts, but after ten days' storage the viscosity in every case was reduced by about half.
13. Viscosity of unclarified pectin extracts decreased during two months' storage, the average loss in concentrated extracts and unconcentrated fresh apple extracts being approximately 5 per cent when kept at room temperature and 3 per cent when stored at 40° F. Unconcentrated pomace extracts, however, lost an average of 63 per cent of their original viscosity at a storage temperature of 76° F. and 37 per cent at 40° F.
14. Concentrated extracts suffered the largest part of their total loss in viscosity during the second

month in storage, while the unconcentrated suffered the greatest decrease during the first month.

15. Concentrated and unconcentrated extracts completely clarified with any of the three diastase preparations used, lost less than 1 per cent of their original viscosity during one month in storage. Extracts which were not successfully clarified, however, suffered a greater loss during the first month in storage than the unclarified extracts.
16. The storage temperature made but slight difference in loss of viscosity of clarified extracts. Several instances were noted in which extracts having the same initial viscosity suffered identically similar losses during the same periods of storage at 76° F. and at 40° F.
17. The possibilities of home preparation and utilization of apple pectin extracts were briefly discussed. It was concluded that, owing to the ease of preparation of pectinous extracts and the variety of ways in which they may be used, their preparation would be practical and profitable for the orchard owner, the farm factory or cider-mill operator and even for the housewife.

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