

Vinegar: Its Production and Analysis

by Merle M. Moore

September 1st, 1911

Submitted to the Department of Chemistry of the
University of Kansas in partial fulfillment of the
requirements for the Degree of Master of Arts

VINEGAR, ITS PRODUCTION
AND ANALYSIS

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THESIS

VINEGAR, ITS PRODUCTION AND ANALYSIS.

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Science

Submitted to
The Graduate School
University of Kansas.
September 1911.

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R00107 47989

Lawrence, Kansas.
Sept. 1, 1911.

Faculty of the Graduate School,

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Dear Sirs:--

I wish to submit the accompanying
thesis for credit toward a degree in the Graduate School.

Respectfully yours,

Merle M. Moore.

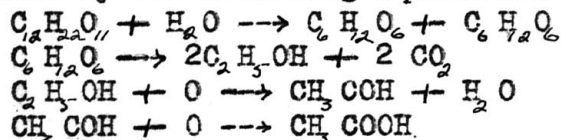
VINEGAR,
ITS PRODUCTION AND ANALYSIS.

Vinegar (vin aigre, sour or sharp wine), is quite ancient in origin; is, in fact, contemporaneous with wine, which was known to very primitive people, and from which it was first produced by accidental fermentation.

The fact that the ancients knew the solvent action of vinegar is illustrated by the story Pliny relates of Cleopatra. Also there are accounts given by Livy and Plutarch of how Hannibal overcame the difficulties which the Alps offered to the passage of his army, by the solvent action of this substance.

But although vinegar was used at a very early period, there was no knowledge of its mode of formation or cause of production. We are indebted to the Alchemists for the knowledge of its purification and increase in strength by distillation. Berzilius, in 1814, determined the exact chemical constitution of acetic acid; and in the same year, that of alcohol was determined. A few years later, after extended experiments, Pasteur demonstrated the relation of bacteria to the production of vinegar. In 1823, one year after the accepted theory of acetic fermentation had been established, the quick process for the manufacture of vinegar was introduced.

Vinegar may be defined as being a liquid resulting from the acetic fermentation of an alcoholic liquid. Sugar is a most important constituent for vinegar production. The fermentation of the sugar is due to yeast cells present in the air and results in the production of invert sugar which in turn changes into alcohol and carbon dioxide. Theoretically 100 parts sugar yield 51 parts alcohol, but in actual practice only about 47 parts are obtained. Acetic fermentation follows and the most necessary conditions are acetic bacteria, a large amount of air and a temperature of from 65° to 85° F. Theoretically 130 parts of acetic acid would be produced from 100 parts of alcohol, but the actual amount produced is only about 120 parts. The change from sugar to acetic acid may be represented by the following equations:



To the laity of the United States the term vinegar means cider vinegar, for as it was the first known and is a farm product made in all parts of the country the word is naturally associated with the product made from apples.

There are however a number of other kinds of vinegar; 7 of the most common and their distinguishing characteristics are given by Leach. These are: cider, malt, wine, sugar, glucose, distilled, and pyrolygenous vinegar. Malt vinegar is obtained from the alcoholic liquid which is made from the wort produced by washing malt or a mixture of malt and barley. It is brown in color and its odor is suggestive of sour beer. It varies in specific gravity from 1.015 - 1.035. The solids of malt are higher than those of cider vinegar but the acidity is about the same. It contains relatively large quantities of phosphates, dextrin, maltose and nitrogenous matter. Cream of tartar is absent.

1. For this and all subsequent references, see bibliography on rear pages.

Wine vinegar is produced from wine which has stood over wine lees; after which it is clarified by passing through beech wood shavings. It is then acidified by adding the wine slowly to the totally or partially acidified product, contained in a large open cask. The vinegar is drawn off as soon as produced. White wine vinegar is light yellow while that made from red wine is red in color. Wine vinegar varies in specific gravity from 1.013 - 1.021 and is slightly laevo-rotary with polarized light. Its acidity varies from 6 - 9 per cent and it usually contains free tartaric acid. Bitartrate of potassium is always present and serves as the chief characteristic of this kind of vinegar.

Glucose vinegar is produced from alcohol made from commercial glucose. It contains dextrin, dextrose, maltose and sulphate of calcium and is low in total solids. It usually has the odor and taste of fermented starch and both before and after inversion is dextro-rotary with polarized light.

Sugar or molasses vinegar as the name indicates is made from sugar-house wastes and low grade molasses. This vinegar is dextro-rotary with polarized light before, and laevo-rotary after inversion. Leach does not mention it, but sugar vinegar is now largely produced merely as a byproduct from the manufacture of yeast.

Distilled or spirit vinegar, which is erroneously called "white wine" vinegar is made from dilute alcohol which is usually prepared from grain. It is colorless, optically inactive with polarized light and ranges from 3 - 10 per cent in acidity. The specific gravity varies from 1.008 - 1.013. The amount of solids present is very small and only traces of ash are found. It often contains non-acidified alcohol and aldehyde.

Pyrolygenous vinegar which is also given by Leach is merely crude acetic acid. This however is now very little used and is not found upon the market its place being taken by distilled or spirit vinegar.

In the United States cider vinegar may be taken as the standard and is the most liable to adulteration. In England malt vinegar either plain or flavored is the most popular, while to the French and Germans the product made from wine is best known.

While cider, malt, wine, sugar, glucose, and distilled vinegar are the kinds known commercially, experiments have been conducted in which other sources for the raw material were used. Pernet³ used unsalable prunes for the manufacture of vinegar and obtained a product of good quality. Vinegar made from honey which was diluted with water containing mineral and nitrogenous matter was prepared by Vinson.⁴

The importance of the analysis of vinegar with reference to determining its purity, has been recognized for many years. In 1887, Davenport⁵ treated of the characteristics and adulterations of vinegar. Since cider vinegar is the principal subject of this paper it will be the only kind considered in giving a chronological resume of work in this line. The standard for cider vinegar is given by Davenport as 4.5 per cent acid and 2 per cent total solids. The acidity of vinegar, as expressed commercially, was formerly and is still often is given in grains of bicarbonate of soda, or of bicarbonate of potassium required to neutralize a fluid ounce of vinegar. To convert acid strength into commercial terms, if expressed as grains of sodium bicarbonate, multiply the per cent by 6.72. To convert to grains of potassium bicarbonate, multiply the per cent by 7.61.

Acidity and total solids, judging from Davenport's work, were the only quantitative determinations used about twenty-five years ago. The

qualitative tests used, by him, consisted of the appearance of the residue which is given as being always soft and viscid, with an acid and astringent taste and apple flavor. A small quantity burned on a loop of platinum wire, gives a violet colored flame only. The residue forms a fusible bead which has strong alkaline reaction and effervesces with acid. The presence of the quantity of acetic acid sufficient to serve as a practical adulterant will prevent the appearance of a pure violet flame, and the ignited residue will have a smoky taste or odor. The presence of free mineral acid will prevent the alkaline reaction of the fused bead. The presence of corn glucose will give the burned residue an odor of burned corn; and just at the close of the burning the odor of garlic will be detected, due to the sulphuric acid used in the production of the glucose containing arsenic incident to its manufacture.

But two of the above tests which Davenport gives are of very little or no significance today. In the first place acetic acid can now be obtained so pure that it would not interfere with the violet flame; but as a matter of fact the use of acetic acid to increase the acidity has given place to that of strongly acid distilled vinegar. Also due to revised methods in the production of glucose, arsenic is no longer present.

Other qualitative tests which are employed at that time consisted in the action of certain chemicals directly upon the vinegar. Hydrogen sulphide gas will not produce a discoloration in cider vinegar, and only very small amounts of calcium chlorides, and sulphates are present. Lead acetate solution produces a flocculent precipitate, which settling in about ten minutes leaves a clear supernatant liquid.

In 1897, Smith⁶ gave the result of a number of determinations of the ash of pure cider vinegar which he found to range from .30 to .50 per cent. Phosphate determinations were also made and it was found that from one-half to two-thirds of the total phosphates in pure cider vinegar are soluble in water. In other vinegars the proportion is lower, and in cider vinegar to which water containing calcium or magnesium has been added, the proportion of water soluble phosphates is also low, due to the formation of alkaline earth phosphates. Smith suggested that although mineral analysis is all that can be advantageously accomplished, the amount of the various organic constituents would be of value.

An important characteristic of the ash of cider vinegar is its alkalinity, the content of the alkaline carbonates being high. In determining this alkalinity the ash may be extracted with hot water and the solution titrated with an acid of known strength. The result may be expressed in cubic centimeters of decinormal acid required to neutralize the soluble ash from 100 c.c. of vinegar. Some vinegars, most frequently those known as distilled vinegars which are made principally from alcohol distilled from fermented grain, contain coloring matter. This, it has been claimed by some manufacturers adds flavor and "body" to the product. But in reality the coloration is for the purpose of selling the product as pure cider, malt, or wine vinegar, as the case may be. This fraud, however, is not as common as formerly since by recent requirement vinegar containing coloring matter must be so labeled. The most common coloring agent is caramel, a substance usually made either by burning glucose, or from an aqueous extract of charred grain. Caramel is most easily detected by separating it by means of paraldehyde, and identified by its bitter taste and reducing action on Fehling's solution.

Smith tabulates the analyses of 22 samples of pure cider vinegar, having obtained the acidity, total solids, ash, alkalinity, and phos-

phates. The calculated data obtained, are original solids which equal acetic acid times 1.5 plus total solids; ash per 100 parts original solids, and phosphoric anhydride per 100 grams original solids. The calculated data from the analysis of 27 samples of cider vinegar containing added water or added spirit vinegar, seem to give more constant values than those obtained from pure cider vinegar.

The fraud practiced in the sale of colored distilled vinegar, can easily be detected because of the very small amount of solids and ash. These simple determinations serve to prove the product other than pure cider vinegar. But in recent years the manufacturers wishing to sell a spurious product, have adopted the practice of adding material to bring the solids and ash up to the amount found in pure cider vinegar. It is possible often to detect this sort of adulteration by the appearance of the solids and ash, and the odor when burning. Smith claims that the solids of pure cider vinegar are often foamy in appearance, and when burned should at first give the odor of baked apples.

Brown, writing of the apple and its products, gives the chemical composition of 25 leading varieties of apples, of the juice from a number of them, of fermented cider, and of apple pumice. The comparison of the analysis of pumice with that of apples from which it was prepared shows the valuable constituents of the former. In discussing the uses to which pumice may be put, re-pressing after the adding of a small amounts of water, is considered the most profitable. Either jelly or vinegar may be made from the juice or "second pressings" obtained in this way. A large amount of valuable material is saved in this way, and the second pressing pumice may still be used for fuel, fertilizer or for feeding stock. The analysis of second pressing cider compared with that of first pressing from the same apples is interesting.

In discussing the acetic fermentation of cider, the slow cask fermentation is said to produce the best vinegar, both in flavor and acidity. Improper treatment often causes the production of a product which is practically worthless. To secure the best results, alcoholic fermentation must be completed before the acetic begins and hence mother or old vinegar ought never to be added to fresh apple juice. After the completion of the alcoholic fermentation, the acetic may be hastened by the use of mother or old vinegar. The containing casks should never be more than $\frac{3}{4}$ full, the bungs removed, and to secure the best results the temperature kept from 75° to 85° F.

Vinegar made by the generator process may be completely acidified in a fraction of the time required for cask fermentation. But it lacks the aroma and flavor produced by the slower process. This aroma is due to the presence of delicate and volatile esters which are lost in the more violent fermentation at higher temperatures.

Browne notes the extensive adulteration of vinegar the simplest form of which consisted merely in the addition of water to the product. But since the enforcement of the laws requiring a certain acidity and solid content, this form of adulteration has necessarily been discontinued. The requirement of 1.5 per cent solids, however, he considers too severe.

The next method of adulteration consisted in adding to distilled or so called "white wine" vinegar, which is quite cheap, such substances as molasses, caramel, sugar, glucose or cider jelly to bring the solids up to the requirement.

A very important characteristic of cider vinegar is its laevorotation

of polarized light. The presence of cane sugar or molasses will rotate the light to the right and after inversion with acid the rotation will be to the left. A marked increase in the reduction of copper from an alkaline copper tartrate solution is also obtained if these adulterants are present. Neither the rotation or copper reducing power is changed upon inversion of a pure cider vinegar. The presence of glucose is assured if a dextro-rotation is obtained after inversion.

The adulteration which is most difficult of detection consists in the addition of cider jelly. Then all the elements of the solids of pure cider vinegar are present. Likewise, an examination of the ash which is often conclusive evidence of other forms of adulteration is of no avail since the composition of the ash of cider jelly and cider vinegar is alike. But in a case of this kind the high percentage of reducing sugar with excessive laevo-rotation is good evidence of the nature of the product.

In concluding, Browne quotes an advertisement of a firm which sells acetic acid and the so called vinegar essence for the production of vinegar. The substances recommended by the firm for increasing the solids are sugar, starch and glucose.

Another adulterant which Browne does not mention is that of boiled cider. In this case as in that of the apple jelly the solids contain all the material of the solids from pure cider vinegar. About the only method of detection is in the large amount of sugar present, but it is often very hard to determine satisfactorily if the adulteration has been carefully done. Also apple wastes such as peeling, cores, etc. may be used to increase the total solids. In this case the per cent solids and ash is high and the solids are usually quite dark in color.

Doolittle and Hess⁸ give the constituents of the extract of pure cider vinegar as glycerol, albuminous substances, gums, organic acids, and mineral matter. This extract gives little or no reducing action with Fehling's solution, and no rotation with polarized light either before or after inversion, after being clarified with lead acetate. A complete analysis of the ash or mineral matter, shows it to be composed principally of potash, with small amounts of sulphuric anhydride, phosphoric acid, alumina, lime, and magnesia.

Frear and Beistle⁷ determined total solids by two methods. Those used were drying in a water oven at 100° C. and drying over sulphuric acid at ordinary room temperature. They obtained lower results by the first method due to volatilization of substances other than water, alcohol and acetic acid.

VanSlyke¹⁰ conducted quite an exhaustive study of apple juice and the resulting cider vinegar. He gives analyses of apple juice of a large number of American apples. A comprehensive study of the fermentation of these juices was then made. Upon the amount of sugar present largely depends the success of vinegar production. The quantity of sugar is dependent on the variety of apple and stage of ripeness; ripe apples containing more than green or over ripe. It was found that under ordinary conditions of cellar temperature most of the sugar is changed to alcohol in five or six months. The fermentation is more rapid at about 85° F. and still more so when yeast is added.

The acetic fermentation takes place rather slowly at cellar temperature the time required being about 18 months. The conditions most favorable for this process are a temperature of from 65° F. to 75° F., the addition of "mother" after the completion of the alcoholic fermentation and the separation of the sediment which forms as the process continues.

After the completion of the acetic fermentation the clear liquid should be drawn off and placed in well filled, closed barrels. This prevents the loss in acidity due to destructive fermentation of the acid by certain aerobic micro-organisms.

In determining acidity, the total acid was found by direct titration. To determine fixed acid a 10 gram sample was evaporated to dryness on a water bath, then about 5 cubic centimeters of distilled water added and the contents evaporated to dryness again. The addition of water and evaporation to dryness was repeated 2 or 3 times. The residue was titrated with decinormal sodium hydroxide and the result calculated as malic acid. This amount deducted from the total acid gave the volatile acid which was calculated as acetic.

Van Slyke worked upon the solids of pure cider vinegar, showing the change in the amount during the process of fermentation. Other data was also obtained, such as the increase in the amount of solids, in vinegar on long standing, due to evaporation, and the decrease due to destructive fermentation. The data shows that the solids may be less than 2 per cent when the acetic acid is above 4.5 per cent.

Most of the state laws, in giving the method for the determination of solids, use the expression, "Solids on full evaporation over boiling water". Some chemists in interpreting this clause maintain that constant weight is what is meant, while others claim that only such constituents as acetic acid, water and alcohol are to be evaporated. Different methods for determining solids were used and compared by Van Slyke. The methods used were: that of Leach and Lythgoe of drying one hour on a water bath; the official method which consists in drying to a syrup and then drying $2\frac{1}{2}$ hours in an oven at 100° C.; and that of drying on sand 8 hours in a steam oven. The results vary according to the stringency of the method, the lowest being obtained by drying on sand 8 hours in a steam oven, and the highest by heating for one hour on a water bath. It was clearly shown that solids estimated by one method would come to the standard while if another were used they would be illegal. The need of a uniform method for this determination is urged as was done by Frear and Beistle.

Van Slyke studied the behavior of malic acid during the fermentation of apple juice. His results show that within the 1st six months one third of the fixed acid had disappeared, and that the decrease is very marked between the seventh and eighth month. When good vinegar has been produced there is only a trace of fixed acid and all has disappeared in the case of some old vinegars. He also studied the effect of fermentation upon malic acid added to apple juice; (2) the effect of temperature upon the destructive fermentation of malic acid and; (3) the effect of sterilizing apple juice upon the decrease of malic acid. Van Slyke was able to obtain a white precipitate upon the addition of lead acetate to all of the pure cider vinegars with which he worked, even when no malic acid or malates were present. So having shown that malic acid decreased upon fermentation and that it was often entirely absent in old vinegars, Van Slyke went far to disprove the belief that malic acid was a characteristic of pure cider vinegar and that the action of lead acetate would indicate its presence.

In considering pure cider vinegar in relation to legal standards Van Slyke shows that if proper fruit is used and the fermentation conducted properly there is readily produced, in from 18 to 24 months, a product which contains above 4.5 per cent acetic acid. But the amount of solids may or may not be as high as 2 per cent depending to a great

extent upon the method employed for their estimation. When cider vinegar is produced which is below standard it is a result of one or more of the following causes -- (1) poor apple juice which may be due to unripe or over ripe fruit, fruit naturally low in sugar, addition of water to normal apple juice, or second pressing of water treated pomace, (2) conditions unfavorable to the fermentation process, such as unclean fruit or barrels, too low temperature or lack of air caused by closing the bung-hole, or filling the barrel too full, (3) lack of proper care after the production of the vinegar by allowing it to stand at too high a temperature with the bung open and the barrel only partially filled. Directions for successful home manufacture of cider vinegar are given in detail and in such a form as to be easily followed by any one.

Leach and Lythgoe² consider that 4.50 per cent acid and 2.00 per cent solids is not too high a requirement for a completely fermented cider vinegar. The quantity of the ash should be about 10 per cent of the solids. Some samples are lower than this but less than 8 per cent very rarely occurs. Less than 6 per cent should arouse suspicion of the authenticity of the sample. However the quality of the ash is much more important than the quantity. The ash of pure cider vinegar is alkaline due to the presence of organic potassium salts. One gram of ash requires about 90 cubic centimeters of decinormal acid to neutralize it so the number of cubic centimeters of decinormal acid required to neutralize the ash from 100 grams of vinegar will approximately equal the percentage of ash multiplied by 100. If the alkalinity falls below 65 per cent of this amount (per cent of ash times 100) the sample probably has been watered with hard water or other fruit juice added.

Another important ratio is the percentage of reducing sugar in total solids. Anything greater than 20 or 25 per cent should be questioned. Other characteristics are the polarization which should range from $-.1^{\circ}$ to -4.0° . Ventzke and malic which is determined quantitatively by the calcium chloride and lead acetate methods.

Browne¹ claims that laevulose may still be present in the solids of cider vinegar and that it is decomposed on heating. Admitting this to be a fact Tolman and LeClerc³ modified the method of drying vinegar so as to cause as little destruction of laevulose and other bodies as possible. They did this by drying in a vacuum oven at 70° C. for from 1 to $1\frac{1}{2}$ days. They modified Leach's original method for the estimation of solids and heated over live steam in a flat bottom dish for 2 hours instead of 1. They showed that samples determined in this way agreed very closely with those determined by heating in a vacuum oven for 1 day at 70° C. Their results show that 2 hours drying over live steam gives lower results than 1 hours drying; the vacuum oven samples agreeing more closely with the former. They conclude that it is quite possible to obtain a correct estimation of vinegar solids by drying in a flat bottomed dish over live steam for 2 hours, but that this length of time is necessary.

Tolman and LeClerc³ proved that the precipitate which formed in a cider vinegar on the addition of lead acetate was not lead malate due to due to the presence of malic acid. As has been stated Van Slyke proved that in a completely fermented cider vinegar malic acid if present at all, was in very small amounts. Also that a precipitate upon the addition of lead acetate would form in a vinegar in which there was no malic acid present. LeClerc and Tolman took the question up in a different way and after obtaining the precipitate of what had been thought to be lead malate they tested for phosphates. Upon further work they proved conclusively

that the precipitate was lead phosphate instead of lead malate.

It was also found upon investigating the composition of second pressing cider that among other things the amount of ash is much higher than in first pressing stock. Also that in polarization before and after inversion there is a slight increase in the laevo-rotation and an increase in the copper reducing power, indicating that some of the gums or other bodies are easily hydrolyzed. This increase in reducing sugar is not due to cane sugar for this would have been easily detected by the increase in dextro-rotation upon polarization. It seems that this would be true also of vinegar made from second pressing stock.

Crampton and Simons¹⁴ find the reducing action and paraldehyde test for caramel unsatisfactory. They prefer the use of Fuller's earth determining the per cent of color removed from the samples by means of a tintometer.

Dubois¹⁵ investigated the Fuller's earth test for caramel in pure cider vinegar. His results show that the color removed varies from none to 72 per cent. No one sample of Fuller's earth can be relied upon to give uniform results. One brand of the earth would indicate a certain sample of vinegar to be pure while with another which is known to be just as pure, 70 per cent of color would be removed. Again a vinegar would be indicated as pure with one sample of Fuller's earth while with another its quality would be questioned and if still another were used the vinegar would be condemned. The conclusion is that the method is unreliable and should be used as a preliminary test only. If no color or only a small amount, or on the other hand, if all is removed the analyst is reasonably safe in relying on the indication. But for the large majority of vinegars which lose from 25 to 75 per cent of their color, the Fuller's earth test is not final and must be followed by further examination.

From the work recorded above it will be seen that there is no reliable method for caramel detection which is applicable in all kinds of vinegar. The nature of this substance is such that conclusive evidence of its presence is very difficult to obtain. Owing to the difficulty of accurate estimation, any attempt at its determination is unsatisfactory in any but a distilled vinegar. Tests for caramel are then applicable and of value only in this product.

Hichey's¹⁶ report as referee on vinegar for the A.O.A.C. consisted of a number of analyses of pure cider and other vinegars. Special attention was given to the determination of the lead number, the modified method of Winton and Kreider being used. It was hoped that the lead number would prove a determination which would serve as a distinguishing characteristic of pure cider vinegar. It was found however, that the results varied over such a wide range that they are of little or no service.

Windisch¹⁷ claims that evaporation for two hours over live steam does not give the true amount of total solids in vinegar due to the incomplete volatilization of acetic acid. He advocates that in order to procure a true value for the extract, that a 50 cubic centimeter sample be evaporated in a platinum dish on a water bath, and the residue dissolved in 50 cubic centimeters of water. Then evaporate again on a water bath and dry $2\frac{1}{2}$ hours in a water oven and weigh. For the determination of the extract by the density method, the same procedure is advocated, with the difference that a porcelain dish be substituted for platinum, and the residue is finally made up to the original volume. Mohan¹⁸ advocates the determination of solids in vinegar by the density method. He suggests the use of a standard spindle which is accurate to .05 per cent.

Balcom,¹⁹ vinegar referee for the A.O.A.C. decided in regard to the lead number that no more information is to be gained as to the genuineness of the sample from the lead number than from noting the character and amount of the precipitate caused by adding lead acetate in a qualitative test. This precipitate is described as slight, normal or heavy as the case may be.

A calculated determination which is probably of more value than any other single determination is that of the non sugar solids. This is obtained by subtracting the total sugar from the total solids.

Analysis of a large number of samples are given, among which are two calculated data of interest; non sugar solids and per cent of ash in non sugar solids, the average amounts being respectively 1.90 and 1.88 per cent. Owing to the varying amounts of sugar, the above data varies less than total solids and per cent ash in total solids. /8.8

The referee also includes data which shows various adulterations of cider vinegar. The samples analysed consist of known pure cider vinegar, sugar and distilled vinegar, commercial samples of cider vinegar and known mixtures such as are often sold as the genuine product. The subtlety of skillful adulteration can be fully appreciated when this data is compared.

In regard to the ordinary determination of total solids the referee agrees with Windisch that too high a result is obtained. When the amount of solids is small the error due to the incomplete volatilization of acetic acid is small but it may amount to 0.1 per cent when the amount of total solids is large.

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Recently Silverman, suggested a method for the determination of pure cider vinegar by means of freezing point estimations. He thought that pure cider vinegars would lower the freezing point of a solution a uniform amount; that is, that values, obtained from this determination would vary to such a limited extent, that a standard could be established. It appears, however, that in the data collected, only one cider vinegar was used, so the method remains to be tested. But even should the method prove successful, it is doubtful if it would prove to be of great value; for it is open to the objection found in other methods. The probability is that it would not be long till an adulterant would be devised which would give the same results as those obtained with pure cider vinegar.

In concluding this chronological resume which we hope includes most of the principal points of the work in this line we would call attention to a Bureau of Chemistry report which is soon to be published. The report is said to deal exhaustively with vinegar and its analysis and is the result of extensive research work. It will undoubtedly be of much value to those interested in this subject.

In beginning my laboratory work in vinegar analysis, a comparative study of the principal kinds of vinegar now found upon the market was first made. The five kinds studied were known pure cider, red wine, tarragan flavored malt, glucose and sugar vinegar. The first sample was made in the Kansas Food Laboratory of known apples, the others were purchased in the market. Distilled vinegar was not used since acidity is practically the only determination to be made, the solids being low and only a trace of ash present. A complete analysis of these five kinds was made in order to show from this data the important characteristics of each kind.

Following the analysis of the various kinds, work was begun on a number of samples of known pure cider vinegar. These samples were prepared by the Kansas State Food Laboratory and data concerning their pro-

duction is first given. After determining the acidity which serves to show principally whether or not the samples are completely fermented, a special study of a method for the determination of total solids was made. The purpose of this study was to show the fallacy of the terms "full evaporation" or "evaporation to constant weight," and to more clearly point out the need of a more definite and uniformly adopted method for the determination of total solids. Further data covering this point is furnished by samples analysed in the Kansas State Food Laboratory. Following this, a partial analysis was made of a few samples produced under known conditions from Kansas apples. The object was to show the characteristics of vinegar from this source, as much as possible being accomplished in the given time. As far as we know there has never been a study made of vinegar produced from Western apples. It has been assumed that that made from Eastern apples would serve as a universal standard, and although this is very probably true, it has been lamented by those interested in the work that there were no analyses obtainable of vinegar produced from Western apples.

The object of the analysis is then not only to add further data to serve in better establishing characteristics of pure cider vinegar, but also to ascertain if the samples varied from vinegar produced in the East.

Experimental Work, Part I. Methods of Analysis.*

The following methods were used in the analysis of the five kinds of vinegar above mentioned.

1. Qualitative tests.
 1. Chlorides. Add a few drops silver nitrate solution to about a 15 C.C. sample.
 1. Sulphates. Add a few drops of 10 per cent barium chloride solution to about a 15 c.c sample.
 1. Calcium. Add a few drops 10 per cent ammonium oxalate solution to about a 15 c.c. sample.
2. Normal lead acetate test. Add 1 - 2 c.c. of 10 per cent normal lead acetate solution to about a 15 c.c sample. In this as in the qualitative tests the precipitate obtained is described and recorded as slight, medium or heavy.
3. Specific Gravity. Determined by means of a hydrometer which is graduated over a small scale (1.000 - 1.060).
4. Acidity, total as acetic. Titrate a 6 c.c sample which has been diluted with water, with decinormal sodium hydroxide solution, using phenolphthalein as the indicator. The number of c.c. of decinormal alkali divided by 10 gives the per cent of acetic acid.

*Since the specific gravity of vinegar is so near unity, samples may be taken by volume and the results given as grams per 100 c.c., except when otherwise stated.
5. Total solids. Evaporate a 5 c.c. sample in a tared flat bottomed porcelain dish of 5 cm. diameter, on live steam for two hours. Cool in a dessicator and weigh.

6. Total Ash. Burn the residue obtained from the evaporation of a 25 c.c. sample as given under "total solids" P.5, in a muffle. Heat very slowly at first, taking care that the residue does not catch fire. Burn at a low red, which must not be exceeded, until the ash is white or nearly so.
7. Alkalinity of Soluble Ash. Extract the total ash repeatedly with hot water; extract until washings give no test for alkalinity when a small test portion is treated with phenolphthalein. Titrate the extract when cool with decinormal acid using methyl orange as the indicator. Express the result in c.c. of decinormal acid required for the ash from 100 c.c. of vinegar.
8. Alkalinity of One Gram of Soluble Ash. Divide the alkalinity of the soluble ash by the soluble ash.
9. Insoluble Ash. Dry the filter paper containing the residue obtained from the extraction of the soluble ash, P.7, Char and burn in a crucible at a low red until free from carbon; cool and weigh.
10. Soluble Ash. Subtract the per cent of insoluble ash from the total ash.
11. Alkalinity of Insoluble Ash. Wash the insoluble ash into a beaker with the least amount of water. Add about 10 c.c. of carefully measured decinormal acid and heat on an iron asbestos gauze for a few minutes at about 65°C. with frequent shaking. Dilute with water and titrate with decinormal alkali using phenolphthalein. The number of c.c. of acid minus the number of c.c. of alkali gives the number of c.c. of decinormal acid required for the neutralization of the insoluble ash in the sample. Express the final result as the number of c.c. of decinormal acid required for the insoluble ash from 100 c.c. of vinegar.
12. Alkalinity of One Gram of Insoluble Ash. Divide the alkalinity of the insoluble ash by the insoluble ash.
13. Total Alkalinity of Ash. Add the alkalinity of the soluble ash to the alkalinity of the insoluble ash.
14. Alkalinity of One Gram Total Ash. Divide the alkalinity of the total ash by the total ash.
15. Soluble Phosphoric Anhydride. Determine the amount of soluble phosphoric anhydride in the neutral solution from the determination of the alkalinity of the soluble ash. Use the gravimetric method or determine volumetrically using the following solutions and methods.

Solutions; (a) Molybdate solution. Add 5 c.c. nitric acid (specific gravity 1.42) to 100 c.c. of the molybdate solution. Filter this solution before using. (b) Standard alkali solution. Prepare a decinormal sodium hydroxide solution. Each c.c. of this solution corresponds with .00025 grams of phosphoric anhydride, or .000111 grams of phosphorus. (c) Standard

Mineral Acid Solution: Prepare a decinormal hydrochloric acid solution. (d) Phenolphthalein: Dissolve one gram phenolphthalein in 100 c.c. of alcohol.

Determination: Add to the solution in which the phosphoric anhydride is to be determined, 15 c.c. concentrated nitric acid and nearly neutralize with ammonium hydroxide. Dilute to from 75 to 100 c.c., heat in water bath to from 60 to 75 C. and add 20 c.c. of freshly filtered molybdic solution. Stir, let stand about 15 minutes, filter at once and wash with cold water by decantation using 25 or 30 c.c. each time until free from acid, using litmus paper. Agitate the precipitate thoroughly each time and allow to settle. Place the filter in the vessel containing the main portion of the precipitate and add a known volume of decinormal alkali in excess of the amount required for solution. Boil the solution till all the ammonia is expelled, using litmus paper. Add a few drops of phenolphthalein and titrate with standare acid. Express the results as milligrams of phosphoric anhydride in the soluble ash from 100 c.c. of the vinegar.

16. Soluable Phosphoric Anhydride per One Gram Soluable Ash: Divide the number of milligrams of soluble phosphoric anhydride by the soluble ash.
17. Insoluable phosphoric anhydride: Estimate the insoluable phosphoric anhydride in the neutral solution from the determination of the alkilinity of the insoluable ash, using either of the methods given for soluble phosphoric anhydride, (Par. 12). If the gravimetric method is used, filter the solution to remove that portion of the ash which did not dissolve in the hydrochloric acid. Express the result as milligrams of phosphoric anhydride in the insoluable ash from 100 c.c. of vinegar.
18. Insoluable Phosphoric Anhydride ^{per} One Gram Insoluable Ash: Divide the number of milligrams of insoluable phosphoric anhydride by the insoluable ash.
19. Total Phosphoric Anhydride: Add the soluble to the insoluable phosphoric anhydride and express as given above.
20. Per Cent. Soluable Phosphoric Anhydride of the Total Phosphoric Anhydride: Divide the soluble by the total phosphoric anhydride and express as per cent.

(2) J. Am. Chem. Soc. 30; 1192.

(3) Ibid. 31; 390.

21. Per Cent. Insoluble Phosphoric Anhydride in the Total Phosphoric Anhydride: Divide the insoluble by the total phosphoric anhydride and express as per cent.
22. Phosphoric Anhydride Per One Gram Total Ash: Divide the total number of milligrams of phosphoric anhydride by the total ash.
23. Polarization, Direct: Clarify by means of Fuller's earth of known quality, for convenience using a centrifuge cream bottle of from 50 to 60 c.c. capacity. Add to the bottle about 10 grams of Fuller's earth and about 45 c.c. of vinegar. Shake well and allow to stand over night. Decant off the top portion and filter through the same filter till clear. Polarize in a 200 millimeter plain glass tube at room temperature. Express the result in degrees Ventzke.
24. Polarization, Invert: Add a 50 c.c. ^{sample} to a 100 c.c. graduated flask, 25 c.c. water and 5 c.c. concentrated hydrochloric acid. Invert by heating to 69 C. in 2.5 minutes and keep at this temperature for 7.5 minutes, the thermometer being immersed in the liquid. Cool and neutralize with concentrated sodium hydroxide (1:1) using a small piece of litmus paper placed in the liquid. Clarify and polarize as given in polarization, direct, Par. 15, with the exception that a 400 millimeter tube is used or the readings obtained from a 200 millimeter multiplied by two.
25. Reducing Sugars Before Inversion: Add a 25 c.c. sample to a 100 c.c. graduated flask, dilute with water and neutralize using small pieces of litmus immersed in the liquid. Make up to the mark and determine, reducing sugar by the Munson & Walker method,⁴ using 25 c.c. of the diluted solution for each determination. From the number of milligrams of cuprous oxide obtained, determine the number of milligrams of invert sugar from the tables. Multiply this result by sixteen to obtain the amount of sugar in 100 c.c. of the vinegar.
26. Reducing Sugar After Inversion: Add a 25 c.c. filtered sample to a 100 c.c. graduated flask, 20 c.c. water and 5 c.c. concentrated hydrochloric acid. Invert, cool and neutralize as given under polarization, invert, Par. ~~15.~~²⁴ Make up to the mark and determine reducing sugar as given under reducing sugar before inversion, Par. ~~17.~~²⁵

27. Reducing Sugar in Total Solids: Divide the amount of reducing sugar in a 100 c.c. sample by the total solids to obtain the per cent. of reducing sugar in the total solids.
28. Non-Reducing Sugar in Total Solids: Subtract the amount of sugar before inversion from the amount after inversion and multiply by 0.95 to obtain the amount of non-reducing sugar as sucrose in a 100 c.c. sample. Divide this amount by the amount of total solids and obtain the per cent. of non-reducing sugar as sucrose, in the total solids.
29. Non-Sugar Solids: Subtract the amount of sugar after inversion from the total solids and obtain the non-sugar solids.
30. Per Cent. Non-Sugar Solids of Total Solids: Divide the non-sugar solids by the total solids and express as per cent.
31. Ash in Non-Sugar Solids: Divide the amount of total ash by the amount of non-sugar solids and obtain the per cent. of ash in the non-sugar solids.

ANALYTICAL DATA
TABLE 1.
ANALYSES OF FIVE KINDS OF VINEGAR

: Kind	: Lead Acet.:	Qualitative Tests		
	: Prec.	: Ca''	: SO ₄	: Cl'
: Cider	: Heavy and*: : Flocculent:	: Small	: Small	: Small
: Glucose	: Small fin-: : ely div'd	: Moderate	: Small	: Moderate
: Malt	: Heavy and : flocculent:	: Moderate	: Moderate	: Heavy
: Sugar	: Small fin-: : ely div'd	: Moderate	: Moderate	: Moderate
: Wine	: Heavy : Flocculent:	: Small	: Moderate	: Small

: Kind	: Specific gravity	(1.) : Total acid	(2) : Total Solids. Gm. per 100c.c.	(3) : Total Ash Gm. per 100 c.c.
	: Cider	: 1.0165	: 5.56 : 5.58	: 2.19 : 2.21
: Glucose	: 1.0150	: 4.33 : 4.34	: 2.54 : 2.58	: 0.17 : 0.16
: Malt	: 1.0212	: 5.55 : 5.61	: 2.80 : 2.78	: 0.50 : 0.51
: Sugar	: 1.0065	: 2.93 : 2.94	: 0.50 : 0.51	: 0.10 : 0.09
: Wine	: 1.0175	: 7.20 : 7.16	: 1.59 : 1.60	: 0.22 : 0.23

: Kind	(4) : Alkilini- : ty, solu- : able ash.	(5) : Alkilini- : ty, lgm.so ² - : lb. Ash	(6) : Insoluble : Ash, Gm. : per 100c.c.	(7) : Soluable : Ash, Gm. Pr. : 100 c.c.
	: Cider	: 40.20 : 37.28	: 129.67 : 120.25	: 0.07 : 0.06
: Glucose	: 3.76 : 3.72	: 28.92 : 31.00	: 0.04 : 0.04	: 0.13 : 0.12
: Malt	: 38.00 : 41.24	: 92.68 : 103.10	: 0.09 : 0.11	: 0.41 : 0.40
: Sugar	: 6.68 : 6.72	: 95.43 : 96.00	: 0.03 : 0.02	: 0.07 : 0.07
: Wine	: 7.08 : 7.24	: 44.25 : 42.58	: 0.06 : 0.06	: 0.16 : 0.17

ANALYTICAL DATA
TABLE 1.
ANALYSES of FIVE KINDS of VINEGAR
(Continued)

	(8)	(9)	(10)	(11)
Kind	: Alkilineity: : Insoluable: : Ash	: Alkilineity: : 1 Gm. : Insoluable: : Ash	: Total : Alkilineity: : Ash	: Alkilineity : : 1 Gm. : Total : Ash
Cider	: 7.24 : 6.60	: 103.43 : 110.00	: 47.44 : 43.88	: 124.84 : 124.00
Glucose	: 4.72 : 4.76	: 118.00 : 118.99	: 8.48 : 8.48	: 49.88 : 53.00
Malt	: 8.12 : 9.92	: 90.22 : 90.18	: 46.12 : 51.16	: 92.24 : 100.31
Sugar	: 6.40 : 5.64	: 213.33 : 282.00	: 13.08 : 12.36	: 130.80 : 137.33
Wine	: 8.28 : 8.56	: 138.00 : 142.66	: 15.36 : 15.80	: 69.81 : 68.69

	(12)	(13)	(14)	(15)
Kind	: Soluable : P ₂ O ₅ : Mg. Per : 100 c.c.	: Soluable : P ₂ O ₅ Per : 1 Gm. Sol- : uable Ash	: Insoluable: : P ₂ O ₅ Mg : Per : 100 c.c.	: Insoluable : P ₂ O ₅ Per : 1 Gm Insol- : uable Ash
Cider	: 29.33 : 31.88	: 94.61 : 102.84	: 7.64 : 5.88	: 109.14 : 98.00
Glucose	: 5.08 : 4.84	: 39.07 : 40.33	: 8.16 : 9.40	: 204.00 : 235.00
Malt	: 76.92 : 78.28	: 187.61 : 195.70	: 46.32 : 48.32	: 514.66 : 439.27
Sugar	: 21.76 : 22.06	: 310.85 : 315.14	: 8.36 : 9.36	: 418.00 : 312.00
Wine	: 20.68 : 20.08	: 129.25 : 117.11	: 26.88 : 26.76	: 448.00 : 446.00

ANALYTICAL DATA
TABLE 1.
ANALYSES of FIVE KINDS of VINEGAR
(Continued)

	(16)	(17)	(18)	(19)
Kind	Total P ₂ O ₅ Mg Per 100 c.c.	Soluble P ₂ O ₅ in Total P ₂ O ₅ (Per Cent)	Insoluble P ₂ O ₅ in Total P ₂ O ₅ (Per Cent)	Mg Total P ₂ O ₅ Per L Gm Total Ash
Cider	36.97	79.33	20.67	97.28
Glucose	37.76	84.42	15.58	102.05
Mal	13.24	38.36	61.64	77.88
Malt	14.24	33.98	66.02	89.00
Sugar	123.24	62.41	37.59	246.48
Wine	126.60	61.82	38.17	248.29
	30.12	72.24	27.76	349.11
	31.42	73.39	26.61	301.20
	47.56	43.48	56.52	216.18
	46.84	42.87	57.13	203.65

	(20)	(21)	(22)	(23)
Kind	Polarization Direct	Polarization Invert	Reducing Sugar before Inversion Gm. Per 100 cc	Reducing Sugar after inversion Gm. Per 100 cc
Cider	-3.80°	-3.14°	1.19	1.21
Glucose	+4.01°	+3.98°	0.66	0.71
Malt	-1.95°	-1.65°	0.66	0.69
Sugar	-0.46°	-0.47°	1.03	1.04
Wine	+1.47°	+1.87°	1.03	1.05
			0.11	0.10
			0.11	0.09
			0.21	0.22
			0.21	0.22

ANALYTICAL DATA
TABLE 1.
ANALYSES of FIVE KINDS of VINEGAR
(Continued)

	(24)	(25)	(26)	(27)
: Kind	: Reducing Sugar in Total Solids (Per Cent)	: Non-reducing Sugar in Total Solids (Per Cent)	: Non-sugar Solids Gm. Per 100 c.c.	: Non-sugar Solids in Total Solids (Per Cent)
: Cider	: 54.03	: 1.24	: 0.98	: 44.76
: Glucose	: 53.86	: 1.45	: 0.99	: 44.79
: Malt	: 26.01	: 1.89	: 1.85	: 72.83
: Sugar	: 25.65	: 1.25	: 1.87	: 72.08
: Wine	: 36.61	: 0.33	: 1.76	: 62.85
	: 37.06	: 0.71	: 1.73	: 62.23
	: 21.76	:	: 0.39	: 78.00
	: 21.87	: Nil.	: 0.40	: 78.43
	: 13.49	: 0.36	: 1.38	: 86.79
	: 13.30	: 0.36	: 1.38	: 86.25

666	(28)
: Kind	: Ash in (Per Cent)
: Cider	: 34.24
: Glucose	: 34.77
: Malt	: 8.73
: Sugar	: 8.42
: Wine	: 29.00
	: 29.25
	: 23.00
	: 23.40
	: 16.05
	: 16.51

Where two sets of figures are given they represent the results obtained from duplicate samples.

*Leaves a clear supernatant liquid in about ten minutes.

Results recorded as the following: Nos. 1,17,18,24, 25,27, and 28 as per cent.; Nos. 2,3,6,7,22,23 and 26 as grams per 100 c.c. of vinegar; Nos. 4,8, and 10 as c.c. of decinormal acid required to neutralize the ash from 100 c.c. of vinegar; Nos. 5,9, and 11 as c.c. of decinormal acid required to neutralize 1 Gm. ash: Nos. 13,15 and 19 as milligrams per 1 Gram of ash; Nos. 20 and 21 as degrees Ventzke. Nos. 12,14, and 16 as milligrams Per 100 c.c. of vinegar.

OBSERVATIONS.

TABLE 1.

1. The lead acetate test results in a heavy flocculent precipitate being produced not only in the cider vinegar but also in the malt and wine. There is very little if any difference in the appearance of these precipitates, but the one in the cider vinegar tends to settle in a short time leaving a clear supernatant liquid, while the other two do not. The precipitates formed in the sugar and glucose vinegar are slight in amount and are finely divided.
2. The qualitative tests serve to show all five vinegars practically free from adulteration by water, provided hard water were used, and from acidity due to sulphuric and hydrochloric acids.
3. The specific gravities of the five vinegars agree closely with those given by Leach. He gives none for glucose or sugar but the others fall within the limits given.
4. The principal thing of note concerning the total acidity is the fact that the malt and wine vinegars run 5.58 and 7.18 per cent. respectively. These were choice samples, sold in small bottles at good prices and seem to contradict the statement advanced by manufacturers in this country, as the cause for watering their product; their plea being that people do not want to buy vinegar that is strongly acid. According to the Kansas law the sugar vinegar would be illegal since 4 per cent. acidity is required.
5. A small sample of 5 c.c. is preferred, for the method used for total solids, to a larger one, since in the latter there is a tendency to the formation of a viscid layer on the surface which protects the part underneath and keeps it from being properly evaporated. The residue of the cider vinegar was light brown, foamy and viscid, with a fruit like taste and odor. A small portion on a loop of platinum wire gave a pink flame and formed a fusible bead with strong alkaline reaction when burned. The color of the flame is violet if the yellow rays are excluded by a blue glass. The residue from the glucose was medium brown and comparatively smooth. That from the malt was very dark brown, rather smooth, and possessed a taste resembling that of American walnuts; the odor was quite pleasant, due to the tarragon with which the vinegar was flavored. Total solids from the sugar were light brown, with a smooth hard surface and very

little or no odor and taste. The wine vinegar gave a residue which was dark brown, foamy, and possessed a slightly bitter taste. The latter four samples when burned on a loop of platinum wire gave a yellow flame and did not form a fusible bead.

6. The residue from cider vinegar when first burned gave off a marked odor of baked apples. Care must be taken not to exceed a low red in burning the ash, in order to prevent volatilization. The final ash was almost pure white. When the glucose residue was charred the odor was at first similar to that of burnt sugar, and later to that of burnt corn. The final product was decidedly pink in color, due probably to the presence of iron. The malt residue when first heated gave off a very pleasant odor, due probably to the tarragon, later the odor was similar to that of toasted bread; the final ash was almost pure white, and contained a quantity of clear, transparent crystals. Upon burning the solids from the sugar vinegar an odor similar to that of burnt sugar was at first noticed. The extract from the wine vinegar when burned did not give any noticeably characteristic odor. A point of interest in the determination of ash is that often after the ash has been removed, the dish will be found to have been blackened. In the case of porcelain dishes the glaze is removed and the dish permanently discolored. In the case of platinum this black is removed by scouring with sea sand, but often with difficulty removed. No explanation is offered for the presence of this discoloration.
7. The presence of organic potassium salts is indicated by the relatively high alkalinity of the soluble ash from the cider vinegar. The soluble ash from the malt was also strongly alkaline; in this case the extraction was rather difficult, the alkaline salts being very slowly soluble. It was finally necessary to boil the ash for about an hour with 800-900 c.c. of water in order to complete extraction. This might have been due in part at least, to having heated the ash to too high a temperature and thus converted some calcium carbonate to calcium oxide. The alkalinity of the soluble ash from the sugar vinegar is comparatively high in proportion to the small amount of total ash.
8. When the alkalinity of 1 gram of soluble ash is calculated, it is seen that that of the cider vinegar is by far the most alkaline. The malt and sugar vinegars are also high in comparison with the other two. These calculations are especially interesting when compared with the results obtained by other workers.

9. The insoluble ash of the five vinegars agree with one another more closely in amount than does the total ash.
10. There is then more of a variation in the amounts of the soluble ash of the five kinds. This could be explained on the basis of the difference in the alkalinity of the soluble ash.
11. The alkalinities of the insoluble ash agree fairly closely, that of the malt and wine being higher than that of cider vinegar. That of the sugar is relatively high in proportion to the amount of insoluble ash.
12. The alkalinity of a gram of insoluble ash also furnishes interesting data. In this case that of the sugar is quite high. The cider and malt are however comparatively low in this respect. There is no very great difference between the alkalinity of 1 gram of soluble and 1 gram of insoluble ash of the cider and malt but the insoluble of the glucose is about four times as great as the soluble. The alkalinity of 1 gram of insoluble ash of the wine and sugar vinegar are respectively about 3 and 2 times as great as that of the soluble.
13. Considerable variation is observed in the total alkalinity of the ash from the five samples. The relation is about the same as that of the alkalinity of the soluble ash.
14. The alkalinity of 1 gram of total ash of the sugar and cider vinegars, show the highest results. The glucose is the lowest in this respect.
15. The relatively large quantity of soluble phosphoric anhydride in the malt vinegar is worthy of note. The amount in the cider is about normal, while that in the sugar vinegar is high, considering the small amount of total ash.
16. The amount of soluble phosphoric anhydride in 1 gram of soluble ash of the sugar is quite high, being over 3 times that found in the cider vinegar. The malt and wine are the next highest while the glucose shows only about one-seventh the amount found in the sugar vinegar.
17. The amount of insoluble phosphoric anhydride in the malt is also quite large. The cider has the lowest in proportion to the amount of the soluble phosphoric anhydride, which is more than two-thirds of the total

amount. An interesting fact is that the sugar vinegar also has two-thirds of the total phosphates soluble; which ratio has been considered especially characteristic of cider vinegar. It would seem that the relatively high phosphates in this sample must have been due to additions of this substance during the process of fermentation. Balcom found only a small fraction in the cider vinegar. The malt and wine are the next highest while the glucose shows only about one-seventh the amount found in the sugar vinegar.

18. The malt and wine and sugar vinegars contain large quantities of insoluble phosphoric anhydride in 1 gram insoluble ash.
There is only about half the amount found in the glucose as in the first three mentioned. The cider vinegar is the lowest in this respect and contains only about one-half as much as the glucose.
19. Also in total phosphates the amount found in the sugar vinegar is nearly the same as that in the cider. The maximum amount is found in the malt and the minimum in the glucose vinegar.
20. The per cent. of soluble phosphoric anhydride of the total phosphoric anhydride is greatest in the cider and sugar vinegars. These are not very much greater than the malt which is next highest. The wine and glucose do not vary greatly, the former having about one-half that shown by the cider vinegar. The per cent. of insoluble phosphoric anhydride of the total found in the different samples, varies, of course, inversely as the per cent. of soluble in the total phosphoric anhydride. In this case the cider and sugar are the lowest, and the glucose the highest in per cent.
21. The sugar, malt and wine vinegars show the greatest amount of phosphoric anhydride in 1 gram of total ash. That found in the cider is only about one-third the amount which the sugar vinegar contains. The glucose is the lowest in this respect, containing only about a fourth as much as does the sugar vinegar. Attention is called to the large amount of mineral salts found in the sugar vinegar. Since the total ash is low, this point is not clearly brought out except in results calculated from 1 gram of the ash. This data is then of considerable value in that it places the results upon a uniform basis from which they can be compared.
22. The polarization of the cider and glucose vinegars, both before and after inversion, is characteristic.

The malt is also laevo-rotary and is not materially changed by inversion. ⁷Leach gives sugar vinegar as being dextro-rotary before and laevo-rotary after inversion. As shown by the data a small laevo-rotary reading was obtained both before and after inversion. However, in crude cane products there are other optically active substances besides the sugars. They are not well known but it is as likely that they would be laevo as dextro-rotary. Also there are known to be reducing sugars present, and some would be laevo-rotary. As a matter of fact, little seems to be known at present about the polarization of vinegars from crude sugar house products.

⁸Leach gives wine vinegar as being slightly laevo-rotary with polarized light. The sample analyzed gave dextro readings both before and after inversion; the increase after inversion falls within experimental error. Grape sugar or glucose ($C_6H_{12}O_6$) is a monosaccharose and hence is not inverted by the process of fermentation or by the laboratory process of inversion. If glucose predominates in grapes it would seem reasonable to expect the vinegar to give dextro-rotary readings. As a further evidence of its authenticity, a sample of the wine vinegar was tested for potassium bitartrate and an estimation made. The method used was the one given by

⁹Leach and the amount found was .04 grams of potassium acid tartrate in a 100c.c. sample of vinegar. This amount seems to be a little low but serves as partial proof, at least, as to the source of the sample.

23. In all five vinegars there is but little difference in the amount of reducing sugars before and after inversion. There is a small increase after inversion of the cider, glucose, malt and wine samples, while the sugar vinegar shows a slight decrease. The cider and malt contain the larger amount of reducing sugar, the cider being greater. The lowest is that contained in the sugar vinegar.
24. The per cent. of reducing sugar in total solids is noticeably higher in the cider than in either of the other vinegars; with the exception of the malt, it contains more than twice as much as either of the others. The per cent. in the malt is about two-thirds of that in the cider vinegar. In all calculated results four decimal places obtained in the analysis are used.
25. The per cent. of non-reducing sugar in total solids in the cider and glucose samples are nearly the

(7) Leach, Food Insp. & Anal. p. 614

(8) Ibid, P. 612

(9) Ibid, p. 618

same, the latter being a little greater. This result in the other samples is quite small in comparison.

26. The non-sugar solids are noticeably higher in the glucose, malt and wine vinegars than in the cider. In the sugar vinegar they are quite small in comparison. The total solids of the glucose and malt are larger than in the cider, but not nearly as much so as the non-sugar solids. The total solids of the wine vinegar are less than the cider, but the non-sugar solids are greater. The non-sugar solids is thought to be a determination which will give more constant results than the total solids.
27. The per cent. of non-sugar solids in the total solids is greatest in the wine vinegar. The next greatest amounts are found in the sugar, glucose and malt, which decreases in the order named. The per cent. in the cider is about one-half that found in the wine, and about two-thirds that of the malt which contains the smallest amount with the exception of the cider. In the case of these results, the cider vinegar is notably different from the four other kinds. Since all of the samples are completely fermented and hence give the normal amount of non-sugar solids, it would seem that this calculation would give data which would serve as a distinguishing characteristic of pure cider vinegar.
28. The per cent. of ash in non-sugar solids in the cider is appreciably higher than that in the malt and sugar vinegar which are the next lowest. This result in the wine is about one-half that of the cider, and twice that of the glucose vinegar. The malt is comparatively high both in non-sugar solids and in ash, while the sugar vinegar is comparatively low in both these respects thus making the per cent. of ash in non-sugar solids nearly the same in these two samples.

PART 2.

Upon concluding the analysis of the various kinds of vinegar, work was begun upon twenty-nine samples of known pure cider vinegar. Data concerning the apples from which this vinegar was produced is found in Table 1.

The samples were quartered and pressed in a small hand press, as much force being applied as a man can give easily. The resulting juice was placed in partially filled 3 liter glass bottles, the stoppers being left out during the violent evolution of carbon dioxide. When acetic fermentation began, the liquid was filtered and placed in clean, stoppered bottles of about 3 liters capacity. The stoppers were removed occasionally to admit air for the oxidation process. In several cases water was added to the pumice obtained from the first pressing, and after being allowed to stand some time was repressed. The liquid obtained in this manner was then treated the same as the first pressing stock and was labeled "Pumice" and given the number of the sample from which it was obtained. All the samples were placed in the laboratory on a pyramid shelf underneath a large table. Thus some of them received more direct ~~light~~ light than others. An example of this is found in sample #10 which was put into two bottles, one of which was in the light, the other on the shady side of the shelf, #10(a) was in the stronger light and is considerably lighter in color than #10(b) which is a dark brown.

At the beginning of the work the samples were filtered from the large bottles into others of about 500 c.c. capacity, these being completely filled.

As indicated in the introductory paragraph, the first work done upon the samples of pure cider vinegar was the determination of acidity, which was made in March, 1911, or from 15 to 17 months after the date of pressing, (Table 1 following this, an extended study was made of total solid estimation, by means of evaporating two hours, over live steam, weighing, adding water and re-evaporating, (Tables 3 and 4). It has been claimed that heating for two hours does not give the correct amount of total solids due to the incomplete volatilization of acetic acid. In starting this work, it was our purpose to see if the samples lose weight upon re-evaporation, to study their various characteristics as the work continued, and in general to obtain as much data and information as possible.

ANALYTICAL DATA

TABLE 1.

DATA CONCERNING APPLES and the PRODUCTION of VINEGAR

No.	Variety	County of:	Kind of Soil
		Kansas	
	Wine		Very sandy on top, heavy
1.	Sap	Riley	black sand under.
	York		Very sandy on top, heavy
2.	Imperial	Riley	black sand under.
			Black loam bottom land.
3.	Geneton	Riley	
			Black loam bottom land
5.	Ben Davis	Riley	
			Rich black & red on bluffs
6.	Genton	Doniphan	near Mo. river.
			Black & red soil on bluffs
7.	Clayton	Doniphan	near Mo. river.
			Black & red soil on bluffs
8.	Black Twig	Doniphan	near Mo. river.
	York		Rich black & red soil on
9.	Imperial	Doniphan	bluffs near Mo. river.
			Heavy black bottom near
10.	Ben Davis	Doniphan	Mo. river.
			Black & red clay like soil
12.	Clayton	Doniphan	on bluffs near Mo. river.
	Winter		Black & red clay like over
14.	Greening	Lynn	gravel & stone.
	York		Red clay prairie over
15.	Imperial	Lynn	gravel & stone.
			Rolling prairie.
17.	Wine Sap	Douglas	
18.	Gano	Douglas	Rolling prairie.
19.	Geneton	Shawnee	Rolling prairie.
20.	Wine Sap	Shawnee	Rolling prairie.
			Dark sand, second bench,
21.	Geneton	Reno	Arkansas River bottom.
	Rome		Dark sandy soil, second bench
22.	Beauty	Reno	Arkansas River bottom.
			Dark sandy, second bench
23.	Wine Sap	Reno	Arkansas Valley
	Mammoth		Dark sandy, second bench
24.	Black Twig	Reno	Arkansas River bottom.
	Missouri		Dark sandy, second bench
25.	Pippin	Reno	Arkansas River bottom.

TABLE 1.
(Cont.)

No.	Weight of Apples ground (Killigrams)	Weight of Juice Ob'td. (Killigrams)	Used for Vinegar.	Date Pressed.
1.	11.145	7.082	Culls only used considerably for vinegar.	Oct. 13, 1909.
2.	12.800	5.721	Culls only used for cider & vinegar.	Oct. 14, 1909.
3.	13.358	8.902	Used more than any other variety.	Oct. 18, 1909.
5.	13.128	6.748	Culls only especially used for vinegar.	Oct. 29, 1909.
6.	9.169	5.547	Used considerably for vinegar.	Dec. 3, 1909.
7.	8.614	5.035	Not generally.	Dec. 7, 1909.
8.	13.156	8.220	Not generally. Culls used for vinegar.	Dec. 1, 1909.
9.	8.555	4.763	Culls considerably used for vinegar.	Dec. 2, 1909.
10.	10.295	5.479	Culls only used for vinegar.	-----/
12.	6.305	3.242	Not generally	Dec. 11, 1909.
14.	7.263	4.100	Not generally.	Nov. 24, 1909.
15.	7.431	4.206	Not generally	-----,
17.	12.283	8.473	Only culls used.	Nov. 1, 1909.
18.	9.734	4.-----	Only culls used.	Nov. 3, 1909.

TABLE 1.
(Cont.)

:No.	:Weight of :Apples Ground: :(Killigrams)	:Weight of :Juice Ob'td.: :(Killigrams)	:Used for :Vinegar.	:Date :Pressed.:
:19.	: 10.722	: 6.843	: Yes.	: Nov.5, : 1909.
:20.	: 6.680	: 3.149	: Only culls : used.	: Nov.10, : 1909.
:21.	: 16.807	: 8.973	: Yes.	: Nov.17, : 1909.
:22.	: 19.790	: 10.514	: Not much : used.	: Nov.11, : 1909.
:23.	: 16.710	: 9.123	: Only culls : used.	: Nov.15, : 1909.
:24.	: 13.204	: 8.039	: Culls used, but : only anlimt'd : quantity grown.	: Nov.29, : 1909.
:25.	: 7.971	: 3.818	: Not especially, : but culls used.	: Nov.18, : 1909.

TABLE 11.

PARTIAL ANALYSES of TWENTY-NINE SAMPLES PURE CIDER VINEGAR

:Sample:	Acidity	:Total	:	Sample:	Acidity	:Total	:
:	:	Solids.	:	:	:	Solids	:
:	:	(Gms. Per	:	:	:	(Gms. per	:
:	(Per Cent)	:100 c.c.)	:	:	(Per Cent)	:100 c.c.)	:
:Pumice:	1.96	: 0.91	:	:	3.34	: 2.27	:
:#1.	1.93	: 0.92	:	:#10(a):	3.37	: 2.31	:
:Pumice:	1.45	: 0.59	:	:	3.51	: 2.33	:
:#2.	1.46	: 0.60	:	:#10(b):	3.52	: 2.36	:
:Pumice:	1.21	: 0.76	:	:	3.80	: 8.65	:
:#3	1.21	: 0.80	:	:#12	3.82	: 8.74	:
:Pumice:	1.05	: 2.14	:	:	3.87	: 7.41	:
:#5	1.05	: 2.15	:	:#14	3.90	: 7.37	:
:Pumice:	5.43	: 5.13	:	:	3.42	: 5.16	:
:#12	5.42	: 5.19	:	:#15	3.46	: 5.13	:
:Pumice:	2.63	: 1.82	:	:	3.35	: 8.03	:
:#17	2.61	: 1.77	:	:#17	3.37	: 8.06	:
:Pumice:	3.76	: 4.90	:	:	3.86	: 7.79	:
:#18	3.81	: 4.92	:	:#17(a):	3.87	: 7.79	:
:Rot'n	2.26	: 2.59	:	:	6.08	: 3.19	:
:Appl's:	2.25	: 2.63	:	:#18	6.06	: 3.14	:
:Un-	4.81	: 5.61	:	:	5.12	: 5.32	:
:known	4.85	: 5.58	:	:#19	5.11	: 5.40	:
:	5.41	: 2.08	:	:	2.67	: 8.81	:
:#3	5.46	: 2.08	:	:#20	2.68	: 8.80	:
:	2.75	: 5.03	:	:	3.96	: 2.16	:
:#6	2.77	: 4.98	:	:#21	3.95	: 2.08	:
:	3.43	: 6.40	:	:	1.29	: 1.43	:
:#7	3.39	: 6.40	:	:#22	1.30	: 1.47	:
:	4.17	: 8.78	:	:	4.41	: 10.20	:
:#8	4.19	: 8.81	:	:#23	4.40	: 10.25	:
:	3.25	: 6.78	:	:	4.64	: 3.69	:
:#9	3.34	: 6.84	:	:#24	4.63	: 3.66	:
:	:	:	:	:	2.77	: 2.74	:
:---	---	---	:	:#25	2.75	: 2.76	:

1. Total solids recorded as grams per 100 c.c.
2. Acidity recorded as per cent.; total as acetic.

TOTAL SOLID DETERMINATIONS.

Sample	Number	Total	Total	Difference	Average
:	of	Solids	Solids	Total Sol-	Loss in:
:	Weighings.	at	at	ids First	Total
:	:	First	Last	to Last	Solids
:	:	Weighing.	Weighing.	Weight.	For ea-
:	:	(Gms Per	(Gms Per	(Gms Per	ch W'gt:
:	:	:100 c.c.)	:100 c.c.)	:100 c.c.)	(Gms Pr:
:	:	:	:	:	:100 c.c):
Pum-		0.91	0.85	0.06	0.02
ice#1	3	0.92	0.86	0.06	0.02
Pum-		0.59	0.57	0.02	0.01
ice#2	3	0.60	0.57	0.03	0.01
Pum-		0.76	0.72	0.04	0.01
ice#3	3	0.80	0.75	0.05	0.01
Pum-		2.15	1.64	0.51	0.04
ice#5	13	2.14	1.65	0.49	0.04
Pum-		5.13	4.24	0.89	0.04
ice#12	21	5.19	4.19	1.00	0.05
Pum-		1.82	1.57	0.25	0.04
ice#17	6	1.77	1.53	0.24	0.04
Pum-		4.90	3.98	0.92	0.04
ice#18	24	4.92	4.00	0.92	0.04
Rotten:		2.59	2.08	0.51	0.02
Apples:	24	2.63	2.09	0.54	0.02
Unkno-		5.61	4.58	1.03	0.04
wn	24	5.58	4.69	0.89	0.04
:		2.08	1.67	0.41	0.02
#3	22	2.08	1.70	0.38	0.02
:		5.03	4.25	0.78	0.03
#6	22	4.98	4.23	0.75	0.03

Total solids recorded in grams per 100 c.c.
of vinegar.

TABLE IV.

DETAILS of the DETERMINATION of TOTAL SOLIDS OF
SAMPLE # 6.

:Number :of :Weight : : : : :	:Weight of :Aluminum :Dish Plus :Total :Solids. : : :	:Loss in :Weight :of Total :Solids. :(Gms Per :5 c.c.) : : :	::Weight of ::Aluminum ::Dish Plus ::Total ::Solids. : : :	:Loss :in :Weight :of Total :Solids. :(Gms Per :5 c.c.) : : :
: 1	: 10.0523	:	:: 9.9248	:
: 2	: 10.0461	: .0062	:: 9.9187	: .0061
: 3	: 10.0454	: .0007	:: 9.9180	: .0007
: 4	: 10.0404	: .0050	:: 9.9127	: .0053
: 5	: 10.0358	: .0046	:: 9.9082	: .0045
: 6	: 10.0335	: .0023	:: 9.9070	: .0012
: 7	: 10.0330	: .0005	:: 9.9063	: .0007
: 8	: 10.0321	: .0009	:: 9.9050	: .0013
: 9	: 10.0274	: .0047	:: 9.9008	: .0042
: 10	: 10.0250	: .0024	:: 9.8980	: .0028
: 11	: 10.0242	: .0008	:: 9.8966	: .0014
: 12	: 10.0239	: .0003	:: 9.8968	: +.0002
: 13	: 10.0205	: .0034	:: 9.8940	: .0028
: 14	: 10.0221	: +.0016	:: 9.8946	: +.0006
: 15	: 10.0200	: .0021	:: 9.8937	: .0009
: 16	: 10.0176	: .0024	:: 9.8911	: .0026
: 17	: 10.0163	: .0013	:: 9.8898	: .0013
: 18	: 10.0150	: .0013	:: 9.8886	: .0012
: 19	: 10.0144	: .0006	:: 9.8880	: .0006
: 20	: 10.0137	: .0007	:: 9.8874	: .0006
: 21	: 10.0134	: .0003	:: 9.8874	: .0000

Results recorded in grams and obtained from
5 cubic centimeter samples.

TABLE V.

TOTAL SOLID DETERMINATIONS, (OFFICIAL METHOD).

Sample	Number	Total	Total	Difference	Average
:	:	of Solids	of Solids	in Total	Loss in
:	:	Weigh- at	at	Solids	Total
::	:	ings. First	Last	First to	Solids of
:	:	: Weighing:	Weighing	Last	Each
:	:	: (Gms Per:	(Gms Per	: Weighings.	: Weighing.
:	:	: 100 c.c.:	100 c.c.)	: (Gms Per	: (Gms Per
:	:	:	:	: 100 c.c.)	: 100 c.c.)
:	:	:	:	:	:
#7718	5	2.10	2.05	0.05	0.01
#7719	8	4.43	4.34	0.09	0.01
#7763	5	1.79	1.76	0.03	0.01
#7764	5	2.46	2.40	0.06	0.01
#7784	8	1.78	1.74	0.04	0.05
#9351	8	3.23	3.12	0.11	0.01
#9372	8	1.73	1.59	0.14	0.02

Samples with the exception of #7784 porposed^t to be Cider Vinegar, #7784 labeled Acid Vinegar.

Data obtained from Prof. H. L. Jackson, Kansas Food Laboratory.

Total Solids recorded as grams per 100 c.c.

TABLE VI.

ACIDITY of TOTAL SOLIDS.

(A)			(B)		
Sample	Acidity #1	Acidity #2	Sample	Acidity #3	
	No. c.c.	As Per		No. c.c.	
	N/10 Acid.	Cent. <i>Acetic</i>		N/10 Acid.	
	0.24	0.03	Pumice	3.21	
#22	0.26	0.03	#18	3.00	
	3.63	0.43	Rotten	2.70	
#23	3.94	0.47	Apple	3.62	
	3.36	0.40	Un-	4.76	
#24	3.43	0.41	known	7.25	
	0.84	0.10		2.49	
#25	0.81	0.10	#3	2.38	
				2.17	
			#6	2.07	
			Pumice	8.40	
			#12	---	

Acidity #1 and #3 expressed as the number of cubic centimeters of N/10 acid necessary to neutralize the solids from 5 cubic centimeters of vinegar.

Acidity #2 expressed as per cent. acid, calculated as acetic.

All samples in division (B) gave a decided test for caramel.

TABLE VII.

TOTAL SOLIDS and ACIDITY.

SAMPLE #18			
Time in Hours.	Total Solids. (Gms Per 100 c.c.)	Acidity #1 No. c.c. N/10 Acid.	Acidity #2. As Per Cent. Acetic.
	3.27	2.38	0.28
2	3.27	2.36	0.28
	3.21	1.89	0.23
3	3.18	1.87	0.22
	3.14	1.71	0.21
4	3.14	1.59	0.20
	3.03	1.55	0.19
5	3.02	1.49	0.18
	3.00	1.37	0.16
6	2.99	1.42	0.17
	2.94	1.45	0.17
7	2.94	1.35	0.16

Total Solids expressed as grams per 100 c.c. of vinegar.

Acidity #1 expressed as the number of cubic centimeters of N/10 acid necessary to neutralize the solids from 5 c.c. of vinegar.

Acidity #2 expressed as per cent. calculated as acetic.

TABLE VIII.

PARTIAL ANALYSES of FIVE SAMPLES of PURE CIDER VINEGAR.

	(1)	(2)	(3)	(4)	(5)
Sample	Acidity (Per Cent.)	Acidity (Per Cent.)	Alcohol (Per Cent. % Vol.)	Total Solids (Gms Per 100 c.c.)	Total Ash. (Gms. Per 100 c.c.)
		<i>Determined 4 months later than #1</i>			
#3	5.41 4.46	5.46 5.49	0.15 0.14	2.08 2.08	0.34 0.34
#6	2.75 2.77	2.67 2.69	2.46 2.44	5.03 4.98	0.35 0.35
#7	3.43 3.39	3.34 3.34	0.70 0.71	6.40 6.40	0.37 0.37
#8	4.17 4.19	4.26 4.25	0.14 0.15	8.78 8.81	0.37 0.35
Pumice	5.43	5.46	0.31	5.13	0.43
#12	5.42	5.50	0.33	5.19	0.42

	(6)	(7)	(8)	(9)	(10)
Sample	Alkalinity of Soluble Ash.	Soluble Ash. Alkalinity 1 Gm.	Insoluble Ash. Gms. Per 100 c.c.	Soluble Ash. Gms. Per 100 c.c.	Alkalinity of Insoluble Ash.
#3	40.20 37.28	118.88 133.74	0.07 0.06	0.27 0.28	7.24 6.60
#6	37.64 37.36	125.46 120.51	0.05 0.05	0.30 0.30	6.00 6.08
#7	39.20 38.68	122.50 120.87	0.05 0.05	0.32 0.32	2.80 2.48
#8	40.44 37.88	122.54 122.22	0.04 0.04	0.33 0.31	5.08 5.00
Pumice	41.20	121.82	0.10	0.33	6.08
#12	41.56	129.87	0.10	0.32	5.36

TABLE VIII.
(Cont.)

	(11)	(12)	(13)	(14)	(15)
Sample	Alkalinity : 1 Gm. in- soluble : Ash.	Total Alkalinity of : Ash.	Alkalinity : 1 Gm. total : Ash.	Reducing : Sugar : Before : Inversion. : Gms. Per : 100 c.c.	Reducing : Sugar : After : Inversion. : Gms. Per : 100 c.c.
#3	103.93 110.00	47.44 43.88	139.53 123.17	1.19 1.19	1.21 1.22
#6	120.00 121.60	43.64 43.44	129.68 129.11	2.73 2.72	2.75 2.75
#7	56.00 49.60	42.00 41.16	113.51 111.24	5.25 5.25	5.31 5.30
#8	127.00 125.00	45.52 42.88	123.03 122.51	4.17 4.16	4.20 4.21
Pumice	60.80	47.28	109.95	1.90	1.98
#12	53.60	46.92	111.71	1.92	1.98

	(16)	(17)	(18)	(19)	(20)
Sample	Reducing : Sugar in : Total : Solids. : (Per : Cent)	Non- : Reducing : Sugar in : Total : Solids. : (Per : Cent)	Non- : Sugar : Solids. : (Gms Per : 100 c.c.)	Non-Sugar : Solids in : Total : Solids. : (Per : Cent.)	Ash in : Non- : Sugar : Solids. : (Per : Cent.)
#3	56.85 57.00	1.24 1.45	0.87 0.87	41.82 41.82	39.50 38.74
#6	54.20 54.59	0.40 0.66	2.23 2.28	44.33 45.79	15.80 15.45
#7	82.02 82.02	0.95 0.83	1.09 1.09	17.03 17.03	34.25 34.36
#8	47.43 47.37	0.39 0.35	4.58 4.60	52.05 52.21	7.73 8.13
Pumice	37.10	1.11	3.14	61.21	10.26
#12	37.14	1.46	3.20	61.65	10.34

TABLE VIII.
(Cont.)

	(21)	(22)	(23)
Sample	Insoluable Ash in Total Ash. (Per Cent)	Alkalinity of Insoluable Ash in Alkalinity of Total Ash.	Ash Ratio.
&			
	19.55	14.09	1.38
#3	16.95	16.25	1.04
	15.54	13.74	1.13
#6	15.35	14.00	1.09
	13.54	6.65	2.03
#7	13.71	6.01	2.28
	12.09	11.16	1.08
#8	11.85	11.66	1.01
Pumice	23.70	12.85	1.84
#12	23.30	11.45	2.03

Results in Table VIII are recorded in the following manner:

Nos. 1, 2, 3 by volume.

Nos. 16, 17, 19, 20, 21 and 22 as per cent.

Nos. 4, 5, 8, 9, 14, 15 and 18 as grams per 100 c.c. of vinegar.

Nos. 6, 10, and 12 as the number c.c. N/10 acid required to neutralize the ash from 100 c.c. of vinegar.

Nos. 7, 11, and 13 as the number of c.c. of N/10 acid required to neutralize 1 Gram of the ash.

No. 23 as the quotient of No. 21 by No. 22.

After continuing these determinations ^{of total solids} for some time on a number of samples which were finally brought to practically constant weight, the series was concluded. Further information in this line is furnished by data obtained in the Kansas Food Laboratory, (Table V.). A few of these samples were then tested for caramel and acidity estimations made of the filtrate, (Table VI B). A few samples which had only been evaporated for two hours were also tested for acidity. (Table VI A).

Following this, another short series of total solid estimations was made. This time the samples were evaporated continuously, each being allowed to remain on the steam bath for a greater period of time than the one preceding it. The first sample was heated over live steam for 2 hours, while the last received 7 hours continuous heating. No water was added. It was our purpose to ascertain if the samples decreased in weight as had those run by the method of adding water and re-evaporating for two hour periods. The acidity of these samples was also obtained. (Table VII.)

Finally a partial analysis of five representative samples was made. (Table VIII.). In this connection it was our purpose to make those estimations which have been thought to give results especially characteristic of pure cider vinegar. The amount of sugar before and after inversion and particularly the calculated results obtained from these amounts, it was hoped would add valuable data to the present rather scanty knowledge of these points. In concluding the experimental data, a few calculated results are given which are obtained from the amounts of total and insoluble ash, and the alkalinity of the two.

METHODS of ANALYSIS.

- I. ACIDITY:--- Obtain the acidity of the vinegar as given in Part I, Methods of Analysis, par.4. Determine the acidity of the total solids by dissolving the solids from 5 c.c. of vinegar in from 150 to 200 c.c. of water and titrating with decinormal alkali, using phenolphthalein. (Table VI A and VII). In the case of the samples in Table VI B, the filtrate from the caramel estimation was used to determine the acidity. Pipette 10 c.c. of this filtrate into a beaker, dilute and titrate. Multiply the number of c.c. of decinormal sodium hydroxide by ten to obtain the total acidity of the solids from the 5 c.c. sample of vinegar. The results may then be given either as the solids from 5 c.c. sample of vinegar, or they may be expressed as acetic acid.

2. **TOTAL SOLIDS:**--- Determine the total solids according to the method given in Part One, Methods of Analysis, Par.5. To obtain the data in Table III this method was repeated a number of times; that is, after heating the 5 c.c. sample in a flat bottomed dish of about 5 centimeters in diameter, over live steam for two hours, placing in a dessicator and weighing, the process was repeated after the addition of 5 c.c. of water. Upon the addition of the water, the dish was slightly agitated so as to obtain a sample as nearly homogenous as possible. The dish was then heated as before, for two hours, placed in a dessicator, and re-weighed. As nearly as possible the re-evaporations were conducted on successive days. The number of re-evaporations and re-weighings are recorded; the weight of the first and last of the process is given, the total loss of a 5 c.c. sample is obtained, and from this and the number of weighings the average loss for each re-evaporation is obtained. The Details of this process are shown in Table IV, the loss at each successive weight being given.

Another method for total solid estimation is that of the Official method⁶. The general plan as to re-weighings as given above was followed, with the exception that no water was added to the samples at the beginning of each re-heating. The plan followed was such that two weights could be obtained each day.

The results for total solids recorded in Table VIII were obtained by heating over live steam for the number of hours given in the table. The process is simply a continuation of the first method given, the first sample receiving exactly the same treatment. The other five samples however were heated continuously over live steam for uniformly increased periods of time which are given in the first column. That is, the second sample was heated for 3 hours, cooled and weighed; the third for 4 hours before being weighed and so on, the seventh sample having received 7 hours continuous heating before being removed from the water bath. These samples were heated without any water being added.

3. **CARAMEL:**--- Caramel test were made upon the total solids which were run in the most extended series, the samples recorded in Table VI.B. These samples were washed into a 100 c.c. flask and water added to the mark. Then 50 c.c. of this solution was pipetted into a 250 c.c. flask to which had been added 25 grams of Fuller's earth. The flask was well shaken and allowed to stand for one-half hour and filtered. From a comparison of the color of this filtrate with that of the

original solution it is told whether there is caramel present in the total solids. It is the filtrate obtained in the above way that is used for the estimation of acidity, the data for which is recorded in Table VI B.

4. **ALCOHOL:---** In the determination of alcohol a Sprengel tube^a was used for obtaining the specific Gravity. The estimations were made at 15.56° C. Fill a 50 c.c. graduated flask at room temperature with the vinegar and pour carefully into the distilling flask, which may consist of a balloon shaped flask of about 500 c.c. capacity. Rinse the graduated flask with water, using not more than 25 c.c., or from one-fourth to one-half the volume of the sample, and add to the distilling flask. Neutralize the sample with sodium hydroxide solution (1-1), and use a small piece of litmus paper immersed in the solution as the indicator. Connect the flask with a condenser fitted with an adapter the end of which extends slightly into the original 50 c.c. graduated flask. Distill slowly, at least three-fourths of the original sample and fill the flask to the mark with water at room temperature. Fill the Sprengel tube with this solution at 15.56° C. and weigh. The weight of the alcohol divided by the weight of the water which the tube contains, which had been previously determined, gives the specific gravity of the alcohol. From reliable tables obtain the per cent. by volume of alcohol, knowing the specific gravity.

A precaution to be observed is in regard to neutralization. As the alkali is added slowly the flask should be well shaken, as the litmus paper has the tendency to "lag" in the strong alkali. As confirmation, it is well to remove a drop by means of a very small stirring rod and touch it to a fresh piece of litmus paper. As a final check the distillate after being made up to the original volume, should be tested with litmus paper to see that it is not acid.

5. **TOTAL ASH:---** Evaporate a 25 c.c. sample of vinegar in a platinum dish of about 5 centimeters diameter, over live steam to a solid or syrupy consistency. Carbonize in a muffle without letting catch fire; heat at first at the front of the muffle and gradually push farther in, and when the danger of catching fire is over push the dish to the back of the muffle which, is not above a dull red heat. Remove when completely carbonized, cool and leach out soluble ash with hot water. Leach three times, filling the platinum dish one-third full each time and pour the liquid on to an ash free filter in a small funnel which drains into a small beaker. Transfer filter paper to platinum dish and rinse funnel

with a small amount of water. Dry the contents of the of the platinum dish, that is the insoluable ash, either in an oven or at the front of the muffle, in which case care is to be used to prevent spattering. Ash and burn in the muffle at low red till all the carbon is consumed. Remove from the muffle taking care that no drafts of air disturb the ash and carefully pour the solution of the soluable ash into the platinum dish. Rinse the beaker twice with small portions of water and place the dish on live steam. When dry, heat for an instant at a dull red heat over a free flame, transfer to a dessicator, cool and weigh.

6. **INSOLUABLE ASH:---** To the total ash in a platinum dish add 40 c.c. of water and boil 2 minutes, being careful to avoid spattering. Filter through a small ash free filter and wash with water at the boiling point till the filtrate measshres 100 c.c. Transfer the filter paper to the original dish and dry in an oven. Ash and burn in a muffle at a low red till free from carbon. Transfer to a dessicator, cool and weigh. Rinse the funnel and reserve the filtrate, also insoluable ash.
7. **SOLUABLE ASH, Alkilineity of 1 gram of Soluable Ash, Alkilineity of 1 gram of Insoluable Ash, Alkilineity of 1 gram of Total Ash:---** Estimate this data as directed in Part 1, Methods of Analysis:
8. **ALKILINITY of SOLUABLE ASH:---** Transfer the filtrate from estimations of insoluable ash to a beaker if a graduated vessel has been used to receive the solution. Titrate and express as given in Part1, Methods of Analysis, par. 7.
9. **ALKILINITY of INSOLUABLE ASH:---** To the ash reserved from the determination of insoluable ash add a few c.c. of decinormal hydrochloric usually about 10 c.c., and 25 c.c. of water. Heat gently on an iron asbestos gauze to dissolve the alkaline salts, taking care to avoid volatilization. Transfer the contents of the platinum dish to a beaker and dilute to 150-200 c.c. Titrate and express as directed in Part 1.
10. **TOTAL ALKILINITY of ASH:---** Add the alkilineity of the water soluable to that of the insoluable ash.
11. **REDUCING SUGAR BEFORE INVERSION;--**Reducing Sugar after Inversion, Reducing Sugar in Total Solids, Non-reducing Sugar in Total Solids, Non-Sugar Solids, Non-Sugar Solids in Total Solids, Ash in Non-sugar Solids:-- Estimate all the above as directed in Part 1, Methods of Analysis.

12. INSOLUABLE ASH in TOTAL ASH:--- Divide the amount of insoluble ash by that of the total ash and obtain the per cent. of insoluble in the total ash.
13. ALKALINITY of INSOLUABLE ASH in the ALKALINITY of TOTAL ASH:--- Divide the alkalinity of the insoluble ash by the total alkalinity of the ash and obtain the per cent. of the alkalinity of the insoluble ash in that of the total ash.
14. ASH RATIO:--- Divide the per cent. of insoluble ash in the total ash by the per cent. of the alkalinity of the insoluble ash in that of the total ash.

OBSERVATIONS.

1. It will be seen from the table giving the data concerning the apples and the production of vinegar that samples were obtained from a number of counties. The varieties were also sufficiently diversified so that the Kansas Apple is fairly well represented. Besides the kind of soil, other data, such as the age of the trees was also obtained by the Kansas Food Laboratory but as this was necessarily so largely an approximation, it was omitted from the table.

A comparison of the weight of juice obtained from the weight of apples ground, between the different varieties, is interesting. It is found that the highest per cent. of juice from the weight of apples used is obtained from the wine sap, geneton and black twig, (Samples #1,3,6,8,17,19 and 24). In fact these varieties are the only ones which show over 60 per cent. of the apples as juice. The highest in this respect is the wine sap (Sample #17) with 68.7 per cent. juice, and the lowest of the above named samples is the geneton (Sample #6) with 60.5 per cent. juice, and the black twig (Sample #24) with 60.7 per cent. juice. The lowest per cent. juice of all the samples is the york imperial (Sample #2) with only about 44.6 per cent of juice; the average of all the samples is about 56 per cent. of juice. As regards counties and kinds of soil, there is not enough data to furnish comparisons. As far as general use for vinegar is concerned, of the three highest in juice per centage, only the geneton is used for this purpose it being more widely used than any other variety. It is seen from the table that the date of pressing from October 13 to December 11, 1909; the time over which the analysis extends is from March 6 to July 8, 1911, about 17 months after the date of pressing.

2. The acidity of the samples as a whole (Table 11) is rather low, only 8 being above 4 per cent. The results, omitting the pumice and rotten apple samples, show the maximum acidity to be 6.07 per cent., the minimum 1.30 per cent. and the average 3.81 per cent. However, in most cases of the first pressing samples in which the acidity is low, the solids are large enough to produce at least a 4 per cent. acidity if fermentation were completed. In fact, at the time of titration, samples number 6 and 7 showed evidence of alcoholic fermentation, a considerable quantity of carbon dioxide being eliminated. The total solid content is quite high, seeming to indicate incomplete fermentation in nearly all the first pressing samples. Sample number 23 seems to be exceptionally high in this respect, although the acidity is above 4 per cent. The total solids content, exclusive of the rotten apple and pumice samples, is maximum, 10.25; minimum 1.43; average 5.53 grams.
3. The total solids recorded at the first weighing of the series which was evaporated repeatedly, or such as would be obtained from the ordinary method, (Part 1, Methods of Analysis p.5), in routine work, represent a very wide range in amount. (Table 111). The samples having the smaller amounts were of course re-evaporated a less number of times in order to reach constant weight and hence the total difference between the first and last weight is also comparatively small. The reverse is of course true of those samples which have a large total solid content. The maximum difference between the first and last weight is 1.03 grams which may be taken as the maximum difference between evaporation for 2 hours on live steam and "full evaporation". It is true however, that this result was obtained on a sample in which the total solids were quite high. But taking a sample (Sample 3) which may be said to contain a more normal amount, it is seen that the total decrease is .40 grams. This may be taken as a significant difference for this amount of any sample which contained about 2 grams of extract would decide the legality of the sample according to the various state laws. In fact even one-half this amount of difference on a sample near the line between the legal and illegal would arouse the suspicion of the analyst and might cause the sample to be classified as the latter. It will be observed that the average decrease for each re-evaporation is .03 grams, the maximum being .05 and the minimum .01 grams. That is according to the above, three re-evaporations, (A total heating of 6 hours) would cause a decrease of .09 grams or practically .10 per cent

in the total solids. This however, does not hold since the decrease in weight is more rapid at the first of the process.

4. The data obtained from the re-evaporation of a 5 c.c. sample which is given in Table IV, shows the details of the results obtained by the method upon a representative sample. It is seen, as would be expected, that the decrease is greatest after the first re-heating, and in general the amount of this loss in weight decreases till a practically constant weight is obtained. Theoretically of course this loss in weight would continue to decrease indefinitely but a practical limit of one milligram was set, and when the samples failed to decrease this amount they were considered to be at a constant weight. In the sample recorded the limit was not strictly adhered to in order to observe the further rate of decrease.

It will be seen from the table that the rate of decrease is by no means strictly uniform. For instance at the third weighing the decrease was only .000% grams while at the second and fourth it was respectively .0062 and .0050 grams. Such irregularities as this were frequent throughout the series, even to the extent of an actual increase in weight as found in the fourteenth weighing and recorded with a plus sign. It will be seen however, that the duplicate samples check fairly closely as given in the table even in the apparently erratic variance in the rate of decrease in weight. These irregularities are hard to account for since the method was uniform in every respect, and as far as could be ascertained the increase in weight was due neither to material falling into the samples or to any possible deposit in the bottom of the dishes which were protected by pieces of filter paper during evaporation, and it hardly seems possible that differences in humidity could cause this variation.

From the table it will be seen that the first re-evaporation, that is a heating of two hours more than is required by the regular method, (Part I Methods of Analysis, p.5), caused a decrease of .12 grams. Also that the first three re-heatings caused a loss in weight of .24 grams. The first named variation caused simply by an additional heating of two hours is in itself strong argument for a fixed and uniform method for total solid determinations.

It has been considered that one characteristic of the solids of pure cider vinegar is the markedly foamy appearance. In our work we have found this to be true in general, but a number of samples were found which were quite smooth and at decided variance with the

general rule. After addition of water and re-evaporation the foamy appearance decreased quite appreciably and after the second or third repetition of this process, the sample entirely lacked this characteristic. As the process continued most of the solids became quite smooth with a glassy surface, but others became granular in appearance and assumed a wave-like formation. A marked change in color of the samples was observed as the process continued, the original solids which varied from a light to a dark brown all became gradually darker until at the close of the series all were a very dark brown, almost black in color. It was also observed that near the close of the process when the usual 5 c.c. of water were added prior to re-evaporation, that the liquid had the characteristic odor of caramel.

What has been said with regard to the data given in Table III, applies in general to that given in Table V. The samples used in the latter case were however, of unknown origin. This and the difference in the method used must then account for the much smaller decrease in weight. It will be seen that much more heating can be given the samples by this method than by the evaporation over live steam, without materially affecting the results. For instance, sample #7764 was re-heated four times, or after being evaporated to a syrupy consistence was heated 12.5 hours in an oven at the temperature of boiling water. One, and probably the primary cause for this difference in the loss in weight by the two methods, is in the addition of water. The total solids agitated in the 5 c.c. of water would seem to allow a more complete and rapid evaporation.

5. Table VIA. shows the 5 c.c. samples evaporated for two hours over live steam, still are quite noticeably acid. As has been pointed out, Van Slyke used this general method for determining fixed or malic acid assuming that all the acetic has been volatilized; while Windisch claims that the acetic is not completely evaporated. Comparing the original acidity (Table II) with that obtained from the total solids, it will be seen that they are practically proportional. The conclusion from this would seem to be that the acetic acid is not all volatilized by the two hours heating.

Table VIB. is quite interesting, showing the very appreciable acidity of the solids after being re-evaporated until they reached constant weight. All of these samples being first tested for caramel gave a decidedly positive test, indicating that a large quantity of this substance was present. The solids after being diluted to 100 c.c. were odorless and ^{almost} tasteless, the solutions

being a dark brown varying slightly in proportion to the color of the respective vinegars. After being treated with Fuller's earth the filtrates were all either clear and colorless or else a very light brown and slightly turbid. These solutions titrated for acidity did not, as shown in the table, always give concordant results as is noticeably true in the case of the unknown sample. An interesting fact in connection with the difference in degree of clarification of the filtrates and their acidity is that different brands of Fuller's earth were used. The same brand of Fuller's earth which may be called grade B, was used for the duplicate samples of numbers 3 and 6. For pumice #18 grades B. and C. were used; for the sample from rotten apples grades A. and C. were used. Where different grades of Fuller's earth were used for duplicate estimations of a sample not only the acidity of the filtrates varied but also their color. This lack of uniformity in the action of the various brands of Fuller's earth for caramel detection has already been referred to and renders its use impractical.

6. For the purpose of studying the action of total solids when heated continuously the method of heating a number of samples each for 1 hour longer than the preceding sample, was adopted. The data given in Table VII shows the results obtained. It will be seen that there was a decrease in weight from the second to the eighth hour of .33 grams. This is an average decrease, .07 grams per hour and goes to show that one extra hour of heating causes a difference of almost .10 grams in the total solids; the decrease from the second to the third hour period was .08 grams. In general the amount of decrease in weight becomes smaller the longer the samples are heated; that from the sixth to the seventh hour being .05 grams while from the second to the third it was .08 grams.

As seen from the table the acidity also decreases as the process is continued. The decrease in acetic acid is shown to be .05 per cent with the first additional hour of heating. The total decrease is .11 per cent which seems to be the maximum amount since the acidity remains the same from the sixth to the seventh hour of heating. This data shows conclusively that all of the acetic acid is not volatilized by two hours of heating of the total solids over live steam.

7. In Table VIII data showing the result of a partial analysis of five representative samples of pure cider vinegar is given. The second series of acidity determinations was made about four months later than the first series.

The results show that there had been little change either toward further fermentation and increased acidity or toward destructive fermentation. The alcohol estimations show that sample #6 contains the largest amount and samples #3 and #8 the smallest quantity of alcohol. It would take a long time for the .14 per cent found in sample #3 to disappear and it is probable that it would tend to the formation of organic esters rather than to conversion into acetic acid.

The total solids of all the samples except #3 are above normal and would seem to indicate incomplete fermentation. The ash, its amount and properties are, however, quite characteristic and may be taken as representative of pure cider vinegar. A point of interest and one which is worthy of noting in this connection is the comparatively large amount of total and insoluble ash of the sample made from the pumice. The total alkalinity is also greater than that of either of the other samples. From this data it would seem possible that vinegar made from second pressing stock might be detected provided it had not been tampered with in such a way as to cover these characteristics. Such adulteration could be accomplished by careful watering if the acid and total solid content permitted it; or if profitable by increasing these amounts by careful ~~careful~~ additions after watering.

The alkalinity of 1 gram of soluble ash furnishes data which is comparatively uniform. In fact, the results obtained from the five samples do not vary to any marked extent, no more than would be expected from different kinds of apples or the same apples grown under different conditions. The maximum and minimum results are respectively 120.26 and 129.87 which may be said to be approximately a 7 per cent variation, a smaller amount than is found in most of the calculated data. It will be seen that the duplicate samples of #3 and pumice #12 lack almost as great an amount of checking and still the duplicate determinations of both the alkalinity and the ash are within an allowable experimental error. Under these circumstances the maximum variation between samples seems comparatively small.

It is also worthy of note that the results obtained from the other kinds of vinegar (Part 1) show a much lower alkalinity. Therefore, judging by the results obtained from these five samples of known pure cider vinegar this determination would furnish valuable data in distinguishing it.

The alkalinity of 1 gram of insoluble ash varies to a much greater extent than does that of the soluble ash. The data given in Table 1 may be of value, at least it is of interest in this connection. Samples #7 and pumice #12 in which the alkalinity of 1 gram is only about one-half as great as that of the other three samples were both produced from Clayton apples.

The soil and the locality of production of the apples from which samples #6, 7, 8 and pumice #12 were made, is practically the same. But sample #6 was produced from Geneton apples while #8 was made from the Black Twig variety. Sample #3 which was also made from Genetons, but from apples that were grown in a different locality and in different soil, checks fairly well with sample #6. This would seem to indicate that the Clayton apple contained less of the insoluble alkaline salts that did the Geneton or Black Twig.

The data obtained from the calculation of the alkalinity of 1 gram of total ash is fairly uniform. Samples #7 and pumice #12 are again low in comparison with the others. This of course, is due to the low alkalinity of the insoluble ash which has been referred to in the preceding paragraph.

The sugar content, as might be expected from the large amount of the total solids, is quite high. With the per cent of sugar and of alcohol present in samples #6 and #7 the assumption would be that if completely fermented both would reach and probably exceed the legal requirements of 4 per cent acidity and still also contain sufficient total solid content. If the process of fermentation in sample #8 was completed it would be quite high in acidity. In each sample there was an increase in reducing sugar after inversion. This is, however not due to the presence of small quantities of sucrose, since it is known that the polarization of cider vinegar is the same before and after inversion. It is more probable that the increase in copper reducing power after inversion is due to the presence of small quantities of various gums or resins.

The per cent of non-sugar solids in the total solids is lowest in sample #7 being only about one-third as much as that shown by pumice #12. However, the non-sugar solids in the latter sample can not be compared with those of the other samples owing to the fact that it was made from pumice instead of being produced from first pressing stock. On the whole, the results obtained from this calculation do not check as closely as might be wished, owing in part, at least, to the unusually high content of total solids which some of the samples contain.

The other calculated results derived from the sugar content, speak for themselves. It was hoped that this data would be uniform to such an extent as to prove an important characteristic of pure cider vinegar.

It is however, not all that could be wished since the variation in each series seems to be too great to prove of any assistance in establishing limiting values for these results.

At the close of the experimental work an attempt was made to obtain calculated data from the relative proportion of total and insoluble ash and the alkalinity of the two. The per cent of insoluble ash in the total ash gives results which are comparatively concordant as does also the alkalinity of the insoluble ash in that of the total ash. In the latter series of results, sample #7 is at variance with the other samples, owing to the very low alkalinity of the insoluble ash. The ash ratio of the first series of results divided by the second seems also to give uniform data, with the exception of sample #7. It would be expected that pumice #12 would not check closely with the first pressing samples; the fact that it does not, is a point in favor of the series: However the number of samples analysed is insufficient to obtain enough data to serve as definite characteristics of pure cider vinegar. We had hoped to be able to obtain more data of this sort but owing to circumstances this was impossible. What has been given, especially that regarding the relationship of existing between the ash and its alkalinity may serve only as a suggestion for further work rather than being of any value in itself. It is hoped that in either case methods may soon be devised for obtaining data which will prove sufficient to differentiate pure cider vinegar from all the subtle adulterations which are sold as such.

SUMMARY.

The first of the thesis which was largely the result of library research, dealt briefly with the origin and early history of vinegar. The principal kinds were named and a brief outline of their characteristics were given. This was followed by a resume of the work done upon cider vinegar which being the original kind used in the United States, has received the most attention. In this connection it was our intention to give at least a brief record of the most important facts which has been brought out by work along this line. Special attention has been paid by most analysts to obtaining data characteristic of cider vinegar, which would serve to distinguish it from the many adulterations found on the market. In recording this work, care was taken to bring out as far as possible these characteristics by which pure

cider vinegar could be identified. At the close of the chronological resume the results of our own experimental work are given. This may be conveniently divided into two parts, the first consisting of a complete analysis of the five principal kinds of vinegar, and the second, of special estimations and study of pure cider vinegar. Sufficient data is obtained from the first part of the work to enable one to make comparisons of the five kinds in nearly every important point. The characteristics of the various kinds are clearly brought out and if each represents its kind, which seems to be the case in many important particulars, these would enable one to distinguish one vinegar from another.

The value of the work done upon the known pure cider vinegar is probably somewhat lessened by the fact that the samples were not completely fermented and hence a number of results obtained from them could not stand as representative of samples in which the process of fermentation had been completed. However, it is hoped that the work done upon the total solids will be of some value. It was our purpose to show the necessity of a uniformly adopted method for this determination and from the data obtained it seems to us that this point is clearly shown. Its importance was dwelt upon at some length and for the purpose of food inspection work, is not likely to be over emphasized.

The latter part of the work which was devoted to a partial analysis of five representative samples, shows a number of important characteristics. Owing to incomplete fermentation the total solid and sugar content were not normal, but the ash, its proportional amounts and alkalinity is peculiar to pure cider vinegar.

CONCLUSION.

In concluding, we wish to acknowledge the debt we owe the Kansas Food Laboratory for the samples of known pure cider vinegar. These were prepared by Professor H. Louis Jackson of the laboratory, and given to us ~~for~~ the purpose of this work. We also wish to acknowledge Professor Jackson's courtesy and help in planning the work and in furnishing facilities for its completion.

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