Introductory

SUGAR   MANUFACTURE

And

ANALYSIS

By

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INTRODUCTION

The sugar industry ought to be one of the important industries in the Arab countries of the Near East, Iraq, Syria, and Lebanon; for the climate is suitable for the cultivation of sugar cane or sugar-beet; Yet, hundreds of thousands of pounds worth of sugars and saccharine materials are imported annually into these countries.

The following table '1' gives the quantities and value of the imported sugars into Iraq alone during the years : 1936 - 1942

Material Imported	1936		1937	
	Quantity in tons	Value in I. Dinars.	Quantity in tons	Value in I. Dinars
1. Sucrose				
a. In loaves cones, or Sugar candy blocks	22,025	251,829	23,931	292,846
b. Granulated or powder	14,885	121,293	17,828	146,203
c. Unrefined	1	10	1	19
2. Other Sugars (Glucose, maltose, Lactose etc.)	85	855	55	985
3. Molasses	1	11	-	-
4. Confectionary	461	18,449	728	30,075
5. Other Saccharine Materials	13	605	22	1,138
Total		393,052		471,466

'1' Foriegn Trade Statestics
Department of Customs and Excise, Ministry of Finance, Government of Iraq.

Imported Material	1938		1939	
	Quantity in Tons	Value in I.Dinars	Quantity in Tons	Value in I.Dinars
1. Sucrose				
a. In leaves, cones or blocks and candy	24,642	334,069	23,770	341,554
b. Granulated or powdered	19,204	170,796	21,400	212,219
c. Unrifined	6	94	1	17
2. Other Sugars (Glucose, Maltose, Lactose, etc.)	15	268	18	395
3. Molasses	—	—	✓	—
4. Confectionary	660	26.685	477	18,708
4. Other Saccharine Materials	23	1,162	32	1,922
Total		~~506,389~~ 533,074		~~555,107~~ 564,810

Imported Material	1940		1941	
	Quantity In Tons	Value in I.Dinars	Quantity in Tons	Value in I.Dinars
1. Sucrose				
a. In leaves, cones or blocks and candy	17,116	366,275	7,462	193,357
b. Granulated or powdered	23,855	383,433	32,034	632,147
c. Unrefined	1	20	⃰	5
2. Other Sugars: (Glucose, Maltose, Lactose etc.)	23	561	29	772
3. Molasses	—	—	—	—
4. Confectionary	203	13,313	66	6,826
5. Other Saccharine materials	10	803	13	1,452
Total		767,405		834,559

Imported Material	1942	
	Quantity in Tons	Value in I.Dinars
1. Sucrose		
a. In leaves, canes or blocks and candy	1,039	41,992
b. Granulated and powdered	13,904	385,743
c. Unrefined	109	4,049
2. Other Sugars (Glucose, Maltose, Lactose, etc.)	9	354
3. Molasses	—	2
4. Confectionary	77	12,240
5. Other Saccharine materials	16	5,057
Total		449,437

These mentioned figures illustrate the necessity of establishing sugar factories better than any other way

The Iraq government started the research to ascertain the suitability of Iraq climate and soil for the cultivation of sugar-beet or sugar-cane. The results of the experiments showed that sugar-beet grows successfully in the northern part of Iraq.

When Dr.N.D. Constan suggested to the Iraqi Government that I take up the study of sugar manufacture for my Master of Arts degree, my Government welcomed the idea and I was asked to study the methods of sugar manufacture as far as available in this University,

The practical part of my work consisted of sugar analysis
and visits to sugar factories. Many different samples of
saccarine materials were analyzed such as row sugar, sugar-cane,
molasses, jams, etc. the results of the analysts are not included
in this theses as they mean little or nothing, to the reader,
or, in view of any originality.

I worked for two weeks in Tripoli Sugar Factory studying
the manufacture of cane sugar

Comercial glucose was studed in the Cipa Glucose Plant, and
experiments in the laboratory were made to that end.

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Parts I, II of this thesis present a condensed review
of the methods of sugar manufacture and refining and the
manufacture of commercial glucose, stressing on the theory of
the process and explaining it whenever possible.

The discription of the different types of machinary was
avoided except for the very general type which was the basis
for any further developpment.

Part IV is devoted to the general methods of sugar anal-
ysis. The first chapter, the qualitative analysis, summarises
the most important chemical reactions of sugars in general,
besides describing the important procedures for the qualitative
identification of sugars. The methods on the quantitative anal-
ysis of sugars described in the second chapter of part IV are
only those which I used in my work.

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CHAPTER. 1.

INTRODUCTION.

The term sugar was originally employed and intended to classify all substances having a sweet taste, and thus came to be used almost indiscriminately for cane sugar, fruit sugar, lead acetate (so-called sugar of lead) and other compounds possessing this property.

At present, in a general sense, it is reserved almost exclusively to denote crystallized sugar from the tropical cane or the sugar beet, which is sucrose, or cane sugar. The commercial sugar whether derived from the sugar cane, sugar beet, the maple tree, the palm or some other sources, is almost pure sucrose,

History of sugar:- Etymologically, sugar would seem to be of Indian origin, the earliest from of the word seem to be "Çarkara" in Sanskrit and "Sakkara" in Prakrit hence it may be traced through all the Aryan and Semitic languages, as "Shakar" in Percian, " Sukkar " in Arabic, "Suicar" in Assyrian and Phenician, " "Saccharum" in Latin, "Azucar" in Spenish and Portoguese, Z "Zuchero" in Italian, "Sucre" in French, "Sucker" in German etc..

The cultivation of the genuine sugar cane" "Saccharum officinarum" appears to have been common in India and China in very remote times, but there is no documentary evidence on this point earlier then Herodotus. Frequent mention of the "Sweet cane" occurs in the Scriptures but the plant refered to is doubtful. An Indian reed yielding honey is alluded to by Strobo and a similar statement concerning an Egyptian reed is made by Thiophrastus; while Dioscorides actually gives the name "Saccharum" to a kind

of honey obtained from reeds in Arabian Felix and India; Both he
and Pliny accurately describe the product as being white and brittle
and of salt like consistency.

Later it seems to have been generally termed "Indian Salt"
among the Greeks and the Romans, by whome it was obtained in small
quantities at great expense from India, and used medicinally.

The introduction of sugar cane in the Mediterranean basin
must have occurred as an early date; for it was found growing at
Assouan, on the Nile in upper Egypt, in 714 A.D. and was carried
into Spain by the Moors in 766 while Sicily engaged in the culture
about 1060 to 1090. During the religious wars of the middle ages,
the "Sweet honeyed reed" called "Zucra" which abounded in the
meadows about Tripoli, were consumed by the Crusaders; and it is
evident that sugar making in that neighbourhood was conducted in
a whole sale and systematic manner. From Cyprus and Madeira, the
industry extended, in 1500 to 1600, to most of the West Indies
where it was carried on by Spanish and Britich colonists; but
there is strong evidence in favor of the supposition that several
kinds of sugar cane are indigenous to both the West Inies and to
almost the whole continent of South America.

Establishment of Refineries:- From the extensive grouth of
sugar in the western tropics there resulted large importation of
the raw article into Europe. The introduction of tea and coffee
about the same time created a general and wide demand for what
had hitherto been regarded as a medicine rather than a nutritive
article of diet. Sugar refining appears to have been copied from

the Arabs by the Venetians. Refineries were established in England and Germany in the 16th century, and in Holland soon after, and utilized the imported raw sugar.

Sugar from Beets:- Up to this time sugar derived from cane was the only kind known in commerce. But in 1747, A.S.Margroff, Director of the physical class of the Academy of Science at Berlin, demonstrated the existance of about 6 % of sugar in beet root and recommended the cultivation thereof; and in 1795 Franz Carl Archard, Director of the physical Class at the Academy of Science at Berlin, succeeded in manufacturing beet sugar on his farm in Silesia, Germany, and presented loaves of refined sugar to Frederich Willian the III of Prussia in 1799, and established the first beet sugar factory at Steinan, Germany. About 10 years later Napolion used extraordinary efforts to porter the production of native-grown sugar, and grapes, plums, maize, sorghum, carrots, etc. were also experimented on. The results obtained did not excell those from beets, and the first French factory for making beet-sugar was established at Lille, in 1810 by Crespel-Delisse. Today Europe alone produces annually nearly 7 million tons of beet-sugar.

Chief Saurces of Sugar:- From the preceeding statements, it may be seen that the worlds sugar supply is obtained almost exclusively from two plants, the sugar-cane and the sugar-beet. Only a comparatively small quantity of palm sugar is produced, and the small quantity of maple sugar made in America and Canada is consumed in the raw state.

Chapter 2

CANE SUGAR

The Sugar Cane:-   "Saccharum Officinarum" is a very large grass; its stalk is round and pointed. Normal cane are never hollow. The exterior color varies with the variety. The stalk, of the variety usually grown for sugar production are of a greenish yellow, or purple, or purplish red color, or have stripes of red or purple and green. In certain varieties, the green is so light that the canes are after spoken of as being white. The hight to which the cane grows varies greatly with the soil and climatic conditions and range from 2 to 6.5 meters, seldom attaining the latter hight. The cane grown in this country, Lebanon and Syria, never reaches full maturity, it attains a hight of about 2 - 3 meters . The Cuban cane under favorable conditions reaches a hight of from 3.5 to 4 meters. The leaves of the sugar cane are ribbed, and in different parts of the plant thier length varies from few centimeters to 2 meters. Sugar cane is a tropical plant though it grows successfully in subtropic reageons. Outside the tropics it rarely reaches full maturity and produces seed.

Composition of Cane and Cane Juce:-   The composition of the cane is extreamely variable and is influenced by the variety, soil, the climate, and the age of the plant. According to Dr.C.H. Browne Jr. of the Louisiana Sugar experiment Station, the composition of the purple variety of Louisiana cane as indicated by the results of many analyses at the time of harvesting, in November-December, is as follows:

	Percent
Water	74.5
Ash	0.5
Fiber	10.0
Nitrogenous bodies	0.4
Sugars (like sucrose,glucose,and fractose)	14.0
Fat,wax,pectin (Gum) free acids, combined acids, ...	0.6
Total	100.0

The reaction of cane juce with litmus is always acidic.

The preceeding analysis refers only to that part of the cane barvested for sugar making. The unripe top joints of the stalk are always lifts in the field.

The average sucrose content in the juce of extracted in the sugar factory in Tripoli was 16 - 17.5 % , and reducing sugars were 0.3 %.

Coefficients used in Sugar Manufacture

For convenience and bravity, several expressions, or coefficints, are used by sugar makers and chemists in describing the condition of the juce and products at various stages of the manufacture. These coeffecints may be difined as follows:-

Coefficient of Quotient of Purity:- The coefficient or quotient of purity shows the relation between the sucrose and the total solids contained in the material; it is the percentages of the sucrose in the total solid material in the substance. In calculating the "apparant coefficient", that is the coefficient ordinarily employed, the total solids are determined by a Brix hydrometer and the sucrose by the direct polorization. In the actually "cane true coefficient of purity" the total solids are determined by actually drying the material.

The true cane coefficient is used in comparative laboratory tests and in special researches but not usually in conducting the manufacture.

The apparant coefficient is meant when the word "true" is not prefixed, and usually only the word "purity" is used to designate this coefficient. Purities are commonly expressed with the degree mark instead of the percentage sign, or both signes are omitted.

The purity coefficient gives approximate indication to the amount of crystalizable sucrose, as every part of solid non sucrose prevent the crystalization of one and a half part of sucrose, approximatily . So in a juce of purity 90, which is con- sedered very high, there are 10 parts of solid non sucrose which binders the crystallization of 15 parts of sucrose having the crystallizable sucrose 75 or 75/90 = 85% of the sucrose present. In a juce of purity 80 , which is considered very low, the crystallizable sucrose is $\frac{80- (20 \times 1.5)}{80}$ = 63% of the sucrose present

The purity of the juce in Tripoli Sugar Factory ranged between 85 - 87,   that is, this crystallizable sucrose ranged form 74 - 78% of sucrose present.

2. Glucose Coefficient or Glucose Ratio :-

The glucose coefficient is the ratio of the glucose (redu- cing sugars are termed glucose by sugar manufacturers) to the sucrose multiplied by 100. An increase in this coefficient from a stage of manufacture to another is usually indicative of inversion of sucrose. This is not necessarily the cose however, since the

sucrose may not be removed in like proportions in the process of manufacture and the mechanical loss may not balance each other.

5. Saline Coefficient:-

The saline coefficient is the ratio of the percentage of ash to that of sucrose. It is little used in the cane sugar industry.

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MANUFACTURE OF CANE SUGAR

The various stages of the manufacture of cane sugar may be briefly outlined as follows:-

1. The extraction.

   a. Milling process.

   b. Diffusion process.

2. The purification of the juce. That is defacation and clarification.

3. The concentration of the juce to a syrup.

4. The crystallization of the sugar

   a. Crystallization in the vacum pen.

   b. Crystallization at rest.

   c. Supplement crystallization with motion.

5. Curing of the Sugar:-

   a. Centrifuging and washing

   b. Drying or granulation of the sugar

   c. Formation of cubes, loaves , etc.

EXTRACTION OF THE JUCE.

Milling process:-

The oldest and the most employed method of extracting
the juce from the sugar cane is by means of mills.

The most common forms of mill is the three roller type or
combination of three-rollers mill. Two of the rollers are on the
same level and turn in the same direction. The first is called
"feed-roller" and the second"bagasse roller". The third roller is
placed above the other two and turns in the opposite direction.
It is called "top-roller". As a rule the rollers of the first
mill are placed in such a way that the cane can enter easily and
is but slightly squeezed, but the space between the top and the
bagasse roller is so small that only the mill squeezed cane can
pass, and it is here that the actual crushing takes place. The
distance between the top roller and the feed-roller and between
the former and the bagasse-roller of the second mill is smaller
than those in the first, and still smaller the third one in order to
obtain maximum extraction.

The extracted juce falls from the rollers on to the hed
plate of the mill which has the form of a collecting trough with
perforated bottom, which keeps back the coarser particles of
cane and allows the juce to pass.

In the extraction of the juce in "Tripoli Sugar factory" two -
and three roller mills are used . The whole cane are introduced
into the first mill the roller of which are provided with V shaped
teeth, which are placed so close togeather that the cane passing
through them are slightly squeezed. The emerging cane is crushed

soft and goes to the second mill, the rollers of which are quite
close togeather with small teeth about 9 to the square inch where
the cane are crushed further and the bagasse emerges in the form
of small chips.

The sugar content of the bagasse was about 5% and sometimes
went up to 6.5%.

To extract this sugar left, the bagasse is macerated with
water and recrushed. This procedure is not adopted in Tripoli
Sugar Factory.

Usually in three mills extraction the bagasse is macerated
twice. The first time in the second mill and the juce from the
first and second mills go to the defacator. The second maceration
is in the third mill and the juce obtained is used for maceration,
instead of water, in the second mill.

Maceration can be carried with hot water insted of cold
water, but experiments showed that there is no difference between
the two methods inthe extracting power

The bagasse is used as a fuel and seldom as catile food,
and it usually have the following average composition:-

	Percent
Fiber	46.5
Water	47.5
Sucrose	4.5

Of course, higher or lower values may occur, depending on
the quality of the crushing and the hardness of the cane fibers.

Diffusion Process

As the diffusion process is used exclusively in beet-
sugar manufacture, and only to a very limited extent in cane

sugar factories, it will be described fully in the section on beet-sugar industry. A brief outline of the process as applied to cane, however, will be given here

The cane is reduced to fine shreds or match-like fragments and is then introduced into a series of cylendrical vessels. Hot water at 85 $C_{\circ}^{o}$ is introduced into the first vessel which contains the exhausted cane and then to the second, then the third until it reaches the last one which contains the fresh cane. The juce and cane move on the counter-current principles, the juce becoming more and more concentrated by passing from one vessel to the other and the cane becomes more and more exhausted. When the cane in the first vessel is completly exhausted it is refilled with fresh cane and attached at the end so the second vessel becomes the first and so on.

Owing to the scarcity of water and the high price of fuel, on most plantations and to the difficulty of disposing of the resedue or exhausted chips. This process is little used in cane factories. From the point of view of the extraction of sugar, however, it is an ideal process. For example, the loss in sucrose (unextracted) by the best of mills is never less then 5% and usually about 10% while by diffusion the loss in sucrose is 0.97%

Another advantage is the absence of gums and pectins and waxes expressed with the juce due to the use of the heavy press - ure in milling, yet the big amounts of water used, the inability to use the bagasse as fuel, due to the high water content, without drying, the fixed capacity of the extracting vessels which does not permit variations to meet the necessities which might be encountered during the other stages of manufacture, renders the diffusion process impractical in cane-sugar industry.

THE PURIFICATION OF THE JUCE.

The turbid, viscous juce from the mills is not fit to be worked up into sugar without clarification, and the first step is to submit the juce to such treatment as will remove the viscous and gummy constituent. As sucrose is very liable to decomposition by micro-organisms, which results in sourness, inversion and fermentation, and as the cane juce is very suitable medium for their development, it is necessary to boil the juce immedeately; whereby the albuminoids are coagulated and the further development of the micro-organisms checked. It is also necessary to remove all impurities, both dissolved and suspended, so for as it is practical without spending too much on clarifying agents, which may cost more than the results are worth.

The purpose of calrification is, therefore, to free the juce as quickly as possible from all constituents except sucrose, without delay , since it is only in concentrated solution that sugar is unaffected by ferments and micro-organisms.

Besides heat, which coagulates the albuminoids and kills the germs, the ragent which has been used for clarification from the earlist times is lime. This precepitates gummy and albuminoid matter which forms a muddy sediment, and a top layer of scum, bitween which the bulk of the juce is clear and limpid.

The method of clarification may be devided into three kinds:-

1. Defecation with the right amount of lime so there is no excess lime left.

2. The juce is heavily limed and the excess lime is removed by :-

   a. Carbon dioxide.

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b. Sulfer dioxide.

3. The juce is treated with sulfer dioxide and then lime
    is added to neutrality.

1. The quantity of lime used on the defecation process is just
sufficient to neutralize the free acids of the juce, preventing
them from afterwords causing inversion; further, the lime combines
with the albumin, which had previously been suspended in the
juce in a gelatinous form, but is now coagulated both by the
action of lime and heat. The acid calcium phosphates are preci-
pitated as floculant tribasic phosphate, as well as a part of
the pectin. All these floculant precepitates envelop the suspen-
ded particles of bagasse, wax, clay etc. and partly rise to the
surface and partly sink as soon as the juce begins to boil.

The amount of lime necessary for tempering is ascertained
in many ways. Some use as much lime as necessary for the juce to
give a faint alkaline reaction to litmus paper. Others ascertain
in the laboratory how many cubic centimeters of milk of lime are
needed to clarify one liter of the juce and use that proportion
in the factory. The best method is to add milk of lime in the
approximate proportion, filÅter some of the juce after boiling
and add few drops of a solution of saccharate of lime '1' [1].
If a precipitate is formed then too little lime was added so the
lime is increased in the next batch. If no precipitate is formed
then either too much lime was added or just the right amount to
make sure of this point the quantity of lime is decreased in the

'1' The solution is made by shaking up 20% solution of sucrose
    with lime and filtering the solution.

following batches gradually until a precipitate begins to form
on the addition of saccharate of lime solution then the lime is
increased slightly and that proportion adopted as the correct
one.

The milk of lime used is a 15 B° suspention or 150 grm.
calcium oxide per liter.

Owing to the variable composition of the raw juce the
defacoted juce should be constantly tested in the laboratory for
the amount of lime added and changing it whenever necessary.

After the addition of lime, in the liming tanks, the limed
juce is pumped to the boiling tanks where the juce is heated
just to boiling by a closed steam pipes in the bottom of the
tank. When the limed juce starts to boil, the steam is shut off
and the juce transferred to the settling tanks, usually situated
at the top of the factory so the clear juce can be transfarred
to the storage tarks and then to the evaporators by the action
of gravity .

In the settling tanks the heavy sediment sinks to the bottom
and the light sediments floats. It is left for some time, ½ -
2 hrs. depending on the quality of the juce, and then is drown
to the storage tanks through a pipe in the bottom slightly
elevated so no sediment may be drown with the clear juce.
The sediment and scum is then sent to the filter press where
the clear filtrate is sent to the storage tanks.

2. Juce treated by the defocation process still contains so many gummy constituents that it is very defficult to filter, and is therefore never quite clear. The manufacture of white granulated sugar demands more care and necessitates filteration of the clarified juce as well as of the syrup, and consequently a more thorough elemination of impurities than the simple defocation process permits of. Such purification is brough about by adding a large excess of lime which precepitates albumin and gummg and decomposes redusing sugars. When this action of lime taken place at the temperature of boiling juce the lime salts are dark colored, viscous, and very troublesome, but if the lime acts upon glucose at a temperature below 55 C_e^o the lime salts abe partly insoluble and easily removed, the remainder being colorless and very clear. As soon as the lime has finished its purifying action, the excess is removed by carbon dioxide or sulfer dioxide, which combines with the lime to form the insolububle calcium carbonate or calcium sulfite.

a. The saturation with carbon dioxide may be affected in one or in two operations and according as this is done, the process may be distinguished by the name of single or double carbonation, both of which have thier advantages as we shall see.

Description of the carbonation process:-

The raw juce, from the mill, is mixed with as much lime cream as is though necessary in large tanks provided with stirring apparatus, and then passed throug juce heater where it is wormed to 40 - 50 C_o^o. From the heater; it enters the carbonation tanks which are only half filled with the juce so as to allow for the

violent frothing which occurs during saturation with the gas.
Some times these tanks are open, but in most cases they are
covered with an iron plate, provided with a lid permitting
samples to be taken from time to time, and allowing enterence
to the tank for cleaning it. A chimney carries off the escaping
gases. A coil, into which steam can be admitted, serves for
heating the juce, and a perforated iron coil or cross for in-
troducing the carbon dioxide gas. .

As soon as the tank is half full with the heated juce,
carbon dioxide is turned on care being taken that the temperature
shall not exceed 55 C_o^o.

At first the carbon dioxide is completely absorbed but
the juce soon becomes thick and start to froth violently. Some
oil sprayed on the surface helps to minimize frothing. When the
juce becomes gelatinous the absorption of the gas becomes very
slow until it becomes liquid again and the absorption becomes
more complete until a time comes when a sample taken, the preci-
pitate settle quickly.

Up to this point the procedure for single or double car-
bonation is similar, but this is no longer so.

Single Carbonation:-

In the single carbonation the carbon dioxide is admit-
ted until the juce gives a faint pink reaction to phenolphthalein
then the supply of the gas is slowly shut off. The juce being just
neutral. It is then heated by the steam coil to 90 C_o^o to decom-
pose any calcium bicarbonate formed, and filtered or decanted,

after settling.

As a rule, 1 % of lime to the weight of the juce is used or 70 liters of lime cream at 15 B$_e^o$. on 1000 liters of juce.

Double Carbonation:-

In the double carbonation process the supply of carbon dioxide is stopped as soon as the juce settles rapidly; it then possesses an alkalinity of 0.05 % CaO.

The juce is now filtered without heating. The filtered alkaline juce is pumped again to the saturation tank which can be filled to the top now as there is no more frothing. A slight amount of lime is added and saturation repeated up to neutrality; then the juce is boiled to decompose any calcium bicarbonate which may have formed and filtered or decanted after settling

In the double carbonation process more lime is used usually 1.5 % to the weight of the juce; or 100 liters cream of lime of 15 B$_e^o$. to 1000 liters of juce.

Third Saturation with Sulfer Dioxide:-

In many cases it is impossible to obtain quite neutral juces by saturation with carbon dioxide as either the lime or the juce may contain some alkali metals, which become carbonates and thus keep the juce alkaline after saturation. In order to prevent this, and to obtain still greater decoloration, a third saturation, this time with sulfer dioxide, is sometimes used in factories working with double carbonation process. To this end the saturated juce is pumped again into the saturation tanks, and sulfer dioxide is admitted to neutrality to phenolphthalein, and remains so after being boiled, after which is it filtered.

The effect of lime on the sucrose of the juce in the car-
bonation process is negligable as the temperature is kept low
and the heating period is short. But its effect on the reducing
sugars is twofold, destructive and transforming.

Alkalies decompose reducing sugars into organic acid and
other complex substances. But as the temperature is not high
enough in the carbonation process, the reducing sugars merely
undergo an intramolecular rearrangement or break up into few
pieces so the principal products are lactic acid and saccharic
anid, the calcium salts of which are insoluble in the neutral
medium and are precepitated

b. The excess of lime used for defacation may be removed
as the insoluble calcium sulfite by using sulfer dioxide insted
of carbon dioxid.

In this process excess of lime is added and the juce is not
heated but is saturated with sulfer dioxide. As calcium sulfite
is more soluble than calcium carbonate and as its solution is
alkaline and not neutral therefore the saturation is done in
the cold and the sulfer dioxide gas is shut when the juce gives
a very faint red color with phenolphthalein $

After filteration or decantation the juce is boiled to
coagulate the remainder of the albuminoids.

This method is quite simple and unexpensive and is superior
to the direct defacation process as the juce obtained is clearer
and easier to filter, yet it is very inferior to the carbonation
process regarding the quality of the sugar produced and ease of
operation.

As the calcium sulfite is more granular and easier to filter than calcium carbonate, some manufacturers use sulfer dioxide along with carbon dioxide in the carbonation process. Both gases being shut simultaneously as soon as the juce settles rapidly so it is still alkaline, so that no danger can exist of the carbonate being converted into the soluble bisulfite.

3. In Tripoli Sugar Factory still another process is used for defacation and calrification of the juce.

The raw juce is pumped directly after screening from the mills to the top of a tower about 5 meters high, and as it trickles down it meats an ascending current of sulfer dioxide (generated by burning sulfer) . It reaches the bottom nearly saturated with sulfer dioxide and there it is mixed with lime cream in such proportion that the liming juce after boiling and settling has a pH of 6.8 - 7.2.

The limed juce is then pumped to tanks heated by steam coils where it is heated first to boiling and is left to settle It is then convayed to the storage tanks then to the evoperators.

The owner of the factory claims that this process gives better quality of sugar than other processes.

ADVANTAGES AND DEFECTS OF DIFFERENT PROCESSES OF CLARIFICATION.

The advantages of carbonation over defocation are that the juce is better purified and settled from gummy constituents, it is easier to filter, and yields a more fluid molasses, Yet when we lime the juce cold, and before heating remove the

excess of lime with sulfer dioxide, a great improvement in this
direction results. Another considerable advantage is that the
clarification is affected in a single operation, for immediately
after saturation, the liquid is divided into a clear juce cary
to filter, and a precipitate which settles rapidly; whereas,
in the case of defecation with or without sulfer dioxide the
precipitate settls slowly and filters badly, whilst the clari-
fied juce is gummy and opalescent.

Independent of the kind of cane crushed, we can employ alike
quantity of lime for a long time at a stretch, because if an
excess of lime has been used the carbon dioxide neutralizes it
afterwords, while the quantity of lime required in defecation
is very variable if canes from two different fields are crushed
togeather, and therefore one is not sure of using it in proper
quantity, which is very important point in this operation.

Owing to the carbonated juce being purer, brighter, and
more easily worked, the quality of the sugar is better than from
similar juce clarified by the defecation process.

Against thse advantages must be set certain drawback, the
chif of which is the greater expense, because more lime is req-
uired; the cost of the lime-kiln and fuel, togeather with that
of carbon dioxide pump and other plant, which increases the cost
price of the sugar.

If we do not expect more from the carbonation process than
a more perfect subsidation and clarification of impure juces and
do not want to make white sugar, the single carbonation process
is amply sufficient. But even then it is too expensive because

we can obtain this by ~~having~~ liming the cold juce heavely and saturating
it still cold with sulfer dioxide, which is very convinient
and inexpensive process.

If on the other hand we want to make sugar for direct
consumption then every thing aprox. to cause coloration should
be avoided, and we must have recoverse to the double carbona-
tion process. This requires much lime and carbon dioxide, and
thus causes considerable expenses but this is more than com-
pensated for by the higher ~~Juce~~ price such sugar fetches when composed
with raw sugar.

In short, we are justified in saying that when making
refining crystals or yellow sugar simple defocation and sulfita-
tion are quite sufficient, but that the manufacture of white
sugar requires double carbonation, so that in the present stage
of cane sugar manufacture there is no longer any scope for single
carbonation.

Before concluding this part we should mention something
about the qualities of the lime used.

It should be the purest obtainable . The following is
the maximum permiceable impureties:- '1'

	Percent
Iron oxide and aluminium oxide	2
Sulfer trioxide	0.2
Magnesium oxide	2
Silica	2
Carbon dioxide	2
Moisture	2

Water and carbon dioxide through inoffensive, decrease
the affect of a given quantity of lime, the same thing aplies for
insoluble impurities as sand and clay. So the other has fixed
the limits of these impurities as given above. Iron oxide and

aluminium give rise to the formation of scale in the evaporators.

Magnisia is a very troublsome impurity in lime, when present in appreciable quantity. It discolors the juce and retards subsidation, so that the decanted juce always contains much suspended matter, which forms scales in the evaporating vessels.

Sulfates also scale the evaporators and should not occur in any appreciable quantities in the lime used for clarification. Alkalies, on the other hand, are harmless and occur usually, in small proportions in lime stone, and may be totally absent.

'1' Cane Sugar and its manufacture by H.C.Drinson Geerligs P.160

CONCENTRATION OF THE JUCE AND CRYSTALLIZATION

Up to the stage now reached all the operations, the juce undergone, have had for thier object the removal of the non-saccharine matter and the clarification of the juce. This being accomplished, the sucrose now exists in a solution containing about 15% of dry subâtances including those impurities which clarification failed to remove. The purpose of the next opera-tion is, therefore, to convert the sucrose into a solid in order that it may then be saparated from the impurities still remaining in solution in the mother liquor or molases. This is affected by evaporating water from the juce until super saturation where the sucrose will crystallize oñt and can be saparated from the molasses thus obtained in a pure state.

This removal of water by evaporation must be made at once and without delay, as in a dilute solution the sucrose is very liable to decomposition by micro-organisms, which cause inversion and formentation, alcoholic and acetic, and therefore give rise to considerable loss. It is therefore necessary to deprive the clarified juce of the great bulk of its water as rapidly as possible, so as to bring it to a condition in which it is no longer liable to such decomposition, i.e. a crystalline mass containing 8 - 10 percent of moisture.

Since sucrose is liable to decomposition at high temperature it is desirable to keep the temperature as low as possible during evaporation and at the same time to reduce the time during which it remains at that temperature. This is affected by boiling the juce under reduced pressur and by means of which its boiling

point falls. Powerful pumps are used to drew off the vapors and memtain a vacuum.

The concentration of the juce is ordinarily affected in two stages, viz " evaporation) which concentrated the juce to syrup containing about 50 % of solid matter, and boiling which concentrates the syrup stile further and transfers it into a crystalline mass, conteing only from 8 - 10 % of moisture.

The sole object of evaporation is to concentrate the juce, as soon as possible, with a minimum waste of steam and at a low temperature.

In boiling (during which the sucrose first assumes a crystalline form), additional care is necessary in order to obtain the maximum yield of crystallized sucrose inas pure a state as possible and in such a condition as to allow it to be saparated from the mother liquor with the minimum loss and trouble.

EVAPORATION:-

The Brix degree of the their juce is about 25 and it must be concentrated to 55 - 60 Bx. before it can be handled easily in the crystallizer becouse:-

1. The juce contains too large quantity of water and requires too long a time to be evaporated in the crystallizer or vacuum pan.

2. The juce contains a considerable quantity of subs- tances which separate from the solution when boiled down to a syrupy condition and should be removed by filteration before concentration or otherwise they increase the ash content of white granulated sugar.

3. Concentration usually increases the alkalinity of the juce which should be corrected to the proper degree, since too high alkalinity renders the crystallization of sucrose difficult.

It is for these reasons that the juce is first concenration to a syrup, corrected for alkalinity, and filtered before it is sent up to the vacuum pan for crystallization.

Construction of Evaporators:-

There are several forms of evaporators:- one body (single effect) two-body (double effectes), three-body (triple effects), four-body (quadruple effects), five-body (quintuple effects),. Those evaporators consisting of more than one body are called multiple effects.

The most commonly used kind is the vertical quadruple effect which it will be described brieffly.

The evaporator consists of four bodies, each body consists of two sections of large cost-iron cylinders. The lower section is the heating chamber, and the upper section is the vapor chamber. The heating chamber is pierced through by numerous brass tubes which serve as heating surface as well as for the passage of the juce.

The heating chambers are filled to about three quarters full of juce and the steam turned on.

The first body is heated by the exhaust steam of the factory and is regulated such that the juce in it boils about 104 - 105 C., and at a pressure of 2.5 lbs. per sq. inch.

The vapor developed from the juce in the first chamber passes over to the heating chamber of the second body where it heats the juce there so it boils at atmospheric pressure and at about 100 - 101 C..

The vapors developed from the second body serve in heating the third body which should boil at 85 C^o and a vacuum of about 6 inches of mercury.

The fourth body is heated by the vapors developed from the third body. The boiling temperature 60, C^o and 23 inches of vacuum.

The low pressure in the third body is mentained by a condenser while in the fourth body it is produced by a strong air-pump and a condenser.

The juce enters the first body and moves to the second then the third and to the fourth increasing in concentration all the time until it reaches the proper concentration and then is pumped to the storage tanks.

The advantage of the multiple effect evaporators may be explained from the fact that '1'

1 Kg. of steam evaporates 0.96 Kg. of water in a single effect

         and   1.90 "   "   "   souble   "

          "   2.85 "   "   "   triple   "

          "   3.79 "   "   "   quadruple "

          "   4.72 "   "   "   quintuple "

The advantage of a multiple effect may also be seen from a comparison of the coal consumed in evaporating the juce obtained from 10,000 metr. of beet '1'

With single effect .... 1929.7 Coal metr.

  "   double   "   .... 975.0   "

  "   Triple   "   .... 650.0   "

  "   quadruple "   .... 488.8   "

  "   quintuple "   .... 392.5   "

The syrups is left to settle, the pH corrected to 7 by some phosphoric acid, filtered and is sent to the vacuum pan.

'1' Beet-sugar making and its chemical control. Mikaido P.223.

BOILING.

During the boiling process the concentration of the clarified syrup is continued up to the point where there is not sufficient water for the sugar to remain dessolved, and it becomes partially crystallized.

The rapidity with which sugar crystallizes out from a solution depends, other circumstances remaining the same, on the purity of the solution, i.e. on whether much or little foriegn substances are present along with the sugar. When the sugar solution is fairly pure, crystallization takes place as soon as the concentration has become too great for all the sugar to remain in solution. When an impure juce, such as molasses, is evaporated, crystallization takes place only very slowly, and no crystals are visible even when the concentration is much greater than corresponds with the solubility of sucrose.

The hot concentrated liquid is allowed to cool, and after some time sucrose crystallizes out, partly becuse it is less soluble in cold water than in hot, but chiefly because the super-saturated state slowly gives place to the normal state of solubility.

Boiling may therefore, be performed in two ways, one of which is followed when boiling syrup, and the second when boiling molasses.

The syrup obtained by the evaporation of the juce usually has a density of 55 - 60 Bx. at 17,5 C. and contains about 40 - 45% water. If only the matter of the further concentration of the syrup were involved, it could be readily and economically accomplished in a multiple effect. but the crystallization of the

sugar must also be consedered. In modern factories, the sugar
is crystallized, or grained, in a single effect vacuum evapora-
tor, termed a vacuum pan.

Vacuum Pan.

A vacuum pan consists of cylinderical cost-iron vessel,
lined from the inside with copper as its is less reactore.
The vessel has a conical bottom and a domed shape top. It is
heated by steam coil, and has a large opening at the bottom for
discharging the contents. An air-pump and condenser are used to
produce the low pressure 25 - 26 inches of vacuum.

TERMS USED IN SUGAR BOILING.

Syrup: The clarified juce evaporated to a density of 55 -
60 Bx.

Grain: The sugar crystals in the masscuite. This word is
also used as a verb, to indicate the process of
forming the crystals.

Proof: That stage of the process when the liquor has been
concentrated to a state of sugar saturation with
sugar.

Boiling to Grain: The boiling of strike in which the
sugar is crystallized in the vacuum pan.

Boiling Blank, or to String Proof: The boiling of a strike
in which the crystallization of the sugar is left
to subsequent operation.

Masscuite: The thick mixture of the sugar crystals and
the mother liquor; also, where strikes are boiled
blank or to string proof, the heavy material ready
for subsequent crystallization of the sugar, is

called massecuite. The word is also, for convin-
ience, applied to the material in the pan at all
stages of the process. The massecuite, are numbered
serially, according to their origin, beginning with
that from syrup, which is called first massecuite.

Mixed Massecuite: A massecuite formed partly of syrup and
partly of molasses. The molassed is said the
"boiled in" .

Strike of Sugar or Massecuite:- The completed massecuite
from a boiling is called a strike and the operation
in called "boiling a strike of sugar or massecuite".
The word strike is also applied, in the same way,
to molasses.

Cut:- The part of the strike that is left in the pan as
a nucleus on which to boil another strike. This
is used also as a verb. Many sugar makers use the
word cut to indicate the portion of the strike that
removed from the pan.

Final Molasses:- The liquid residue from which more sugar
cannot be profitably extracted.

METHODS OF SUGAR BOILING

Grained Strikes:-

The syrup is evaporated until it becomes super-saturated
and deposits tiny crystals of sucrose. When this stage is reached
fresh syrup is admitted gradually and evaporation continued so
that the sugar in the syrup added deposits on the crystals alrea-
dy formed. This process is continued until the crystals grow to
the size required. In the latter stages of the boiling the supply

of fresh syrup is stopped and the contents of the pan evaporated
so the final massecuite has an avarage water onntent of 7%.

The heating is then stopped, and air admitted to normal
pressure and the contents are discharged to be cured.

Cut Strikes:-

When a cut strike is to be boiled, a part of the massecuite
from a previous strike is left in the pan, and its crystals take
the place of the grain that otherwice have to be formed. This
method produces a very large-grained sugar.

Blank strikes or Crystallization at Rest:

In sugar boiling the termes "string strikes", "smooth
strikes", and "fellied strikes" have practically the same meaning
a blank strikes. In boiling the blank strikes, the molasses are
first heated to dissolve the fine crystals it usually contains.
It is then drawn into the pan and concentrated under as high as
a vacuum as can be obtained with the apparatus. The evaporation
is usually stopped when a test sample held between the thumb
and a finger produces a strong thread when these digits are
saparated. The test is often made by pouring the test sample
from the proof strik and noting the appearance of the "string",
when it breaks. The strikes boiled to a "string proof" are run
into small sugar wagons or into large tanks and are placed in
a hot-room and are allowed to remain there for a week or more at
a temperature of about $38°C$., this time depends on the purity of
the molasses. This method is termed "crystallization at rest"
to destinguish it from the more modern method of "crystallization
at motion" which will be described later.

The products made from blank strikes are termed "molasses sugars". These sugars have a low polariscopic test, which usually ranges from $80^{\circ} - 90^{\circ} V_{\bullet}$. Two grades of molasses sugars are usually made, namely seconds and thirds. The third sugar is boiled as the close of one grinding seaon and left until the biginning of the next or for a period of 2 to 3 months, before centrifuging.

Crystallization in Motion

This method does not increase the yield of crystallized sugar but produces more uniform crystals in a shorted time than when employing crystallization at rest.

The crystallizer is a horizantal cylendrical tank with an inlet and outlet doors and is provided with a spiral stirrer.

The blank strike after boiling is run into the crystallizer and is stirred slowly and continuously till no more sugar deposits on the crystals.

Crystallization in motion is used when boiling molasses with massecuites using the crystals of the last a nuclie. After boiling to the proper concentration the strike is run into the crystallizer and is stirred while it cools gradually. The sugar in the molasses deposit on the crystals already present, as the motion keeps the crystals in contact with all the different parts of the solution. So by this method the production of new small crystals, colled false grain, is avoided as their presence offers many difficulties afterwords as they clog the holes in the centrifugal sieve and prevent the compleat separation of the crystals from the mother liquor, apart form the loss in sugar as these timy crystals connot be

separated and utilized.

To sum up, the crystallization processes, we can say that generally, the operations follow the sequence:-

1. Grained strike where the purest sugar is produced by using pure syrup throughout the whole crystallization.

2. Grained strike too, but here molasses are added towards the end of the boiling and the product is pure sugar too. This procedure is adopted until the purity of the resulting molasses falls from about 70 to 50.

3. The cut strikes are employed only in special cases where large sugar crystals are desired.

4. Blank strikes and crystallization in motion are employed to extract the last amounts of crystallizable sugar from the molasses resulting from previous operation. The yield is brown sugar polorizing $85° - 90°V_s$, and is used in sugar refineries. The exhausted molasses have a purity of about 28.

CURING

The operations now to be described have for their object the mechanical separation of massecuite into crystallized sugar and molasses. It is evident that the most advantageous way to affect this saparation is that in which the maximum of crystalli-zed sugar is obtained at once as first product, because of portions of it become again mixed with the molasses, they can only be recovered after much trouble and expence (and not even than entirely) in the less valuable after products. It has been proved that the curing is most easily affected, and with a minimum of less, when the sugar crystals are well developed, and the mother-liquor lumpid and free from so-called "false" grain.

Saparation of Sugar From Syrups

The separation of sugars from syrup is accomplished by centrefuging the massacuite in a centerfugal machine . The machine is simply a short, wide brass cylinder with perforated walls lined from the inside with a fine brass screen in order to keep the crystals from passing out of the cylinder. It is suspended by means of a steel rod from the bottom and is enclosed in a cast-iron shell. It is usually driven by means of a belt at a speed of 1200 r.p.m. In operating the centrifuge, it is filled with massacuite about one third full and set in motion, where-upon the centrifugal force will drive the mass aginst the walls of the machine and force the syrup out through the pores in the screan, while the sugar crystals remain clinging to the inner surface of the screan. The last trace of syrup adhering to the crystals is worked off by sprinkling water while the machine is

still in motion. The syrup produced by washing is called "wash syrup". Wash syrup is diluted to about 60 Bx., filtered and used for boiling white sugar mixed with thick syrup.

Drying and Bagging of Sugar

When the washing is completed, the sugar is dumped into a trough siturated directly below the centrifuge. The sugar in the trough is forworded by means of a scroll to one end of the trough where it is transfarred by means of a bucket elevator, up to a sugar storage box located on the upper floor. The storage box is provided with a ropper which directs the sugar into a sugar drying machine called a "granulater". A granulator is simply a large, long cylinder made of sheet-iron supported on a series of roller-wheels, inclining toword the outlet end,(at the bottom) and rotated by means of a wheel gear. The intirior of the granulator is heated by a steam pipe passing through the center of the cylinder throughout the intire lenth. At the outlet end, a brass screan is attached to the cylinder so that any lumps of sugar may be kept from going into the bagging room. The sugar is admitted from the storage box at such a rate that the sugar comming out from the granulator is perfectly dry. The sugar is then received in the bagging room where it is weighed and bagged, ready for transportation.

"Blueing" of Sugar

When the sugar is quite centrifuged and washed, it is sometimes blued in order to neutralize the last traces of yellow color. Sometimes a suspenssion of ultramarine blue in water is drawn into the vacuum pan at the graining point in order that

the neucleus of each crystals may contain sufficient blue to
neutralize the yellowish tint of crystals when fully grown. In
other cases, a little of a little dilute suspenssion is poured
into the centrifuge after the wash water is separated. The
finely divided ultramarine blue remains between the crystals
importing to them a bluish tint which pleases the eye of the
purchaser. Some employ aniline blue or indanthrene but ultra-
marine is the most universal dye for white sugar in refineries,
as also in factories where white sugar is made directly from cane
duce.

Lump Sugar

Instead of sugar in loose crystals, some factortes make
sugar in lumps, resumbling the lump sugar of the refineries.

This class of sugar is made from fine graned massecuites,
intended for white sugar and cured as usual, The moist white
sugar is mixed with pure syrup and treated in special centrifuges,
having small boxes attached to the interior of the drums, the
bottom of which consists of the centrifuge gauze. The mixture
of crystals and syrup fills the boxes, the syrup escapes, and
the sugar is heated with wet steam, which causes the crystals
to stick togeather in a hard lump. As soon as the syrup escap-
ing is colorless, the sugar is discharged in the form of square
white lumps which now only need drying.

In the same way slabks of sugar may be made which are
cut into cubes by means of an automatic scisors.

14 %

16 %

16.8 %

17.1 %

16.5 %

16 %

14 %

10 % ?
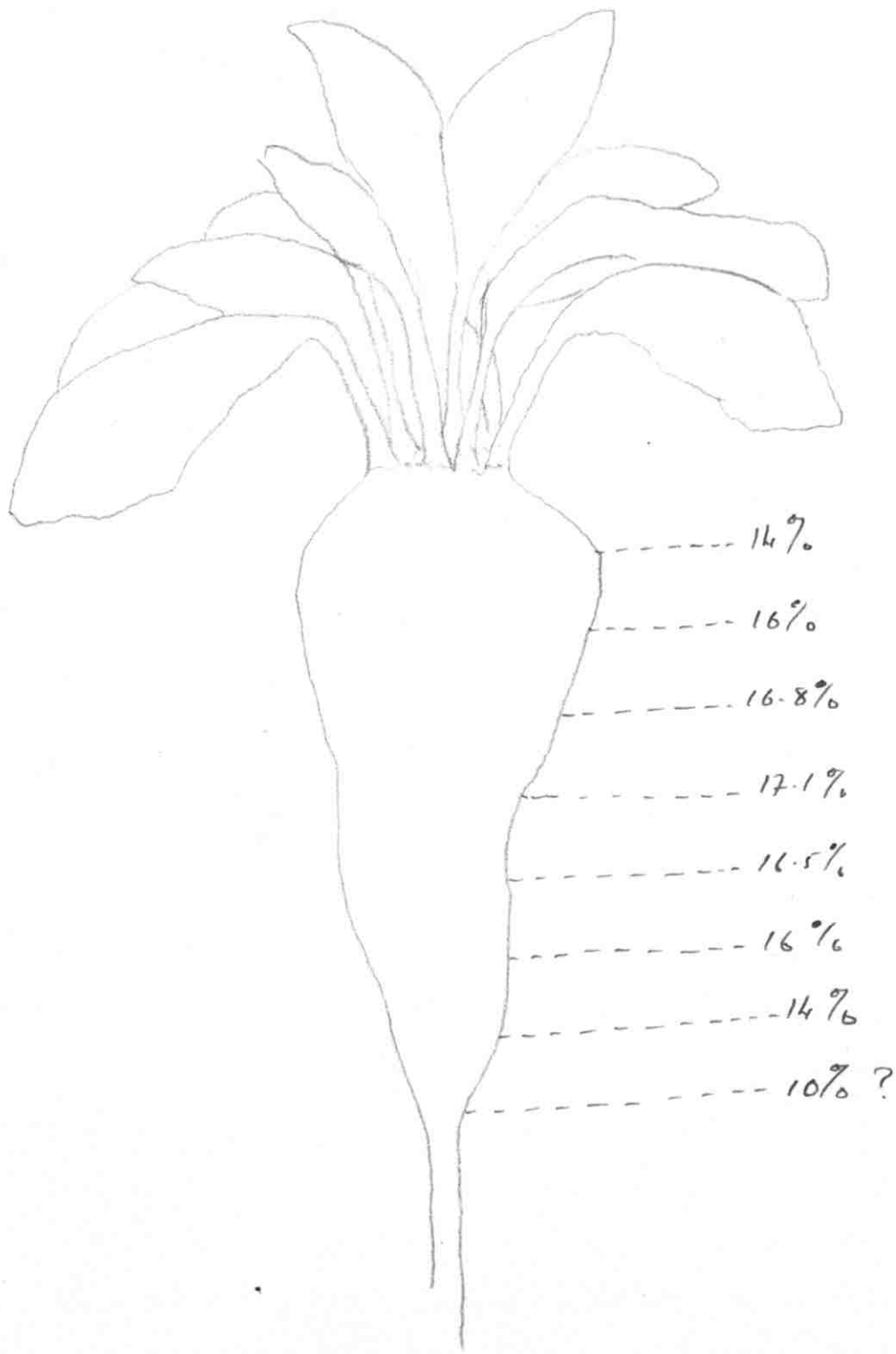
fig (1)

CHAPTER III.

THE MANUFACTURE OF BEET - SUGAR .

The Plant

The several varieties of sugar-beets now known have been derived by selecting roots that possessed valuable sugar producing qualities. All the varieties are similar to those of the common garden beet, or "Beta Vulgaris".

The most favorable climate condition for the sugar-beet are found where the mean summer temperature is 70° F,. It requires an abundance of sunshine for its proper development as a saurce of sugar.

The question of rain-fall is also of great importance. Summer-rains are very desirable, but heavy autumn rains, especially following a dry summer, are extremely unfavorable to the sugar contcut of the beet. Late rains cause a second growth of the plant at the expence of the sugar. Cold winter without alternate frezing and thawing are desirable for the proper keeping of the roots in storage preparatory to the manufacture

A typical beet, so far as the shape is concerned, is shown in Fig. 1. This beet is large and tepering, with only a small neck, or crown and with broad, spreading foliage. These conditions of shape facilitates harvesting. The long top root penetrates the soil deeply in search of moisture; the spreading leaves shade the adjacent soil, thus conserving the moisture and preventing the frowth of weeds; and the small neck, which must be removed in harvesting, results in little waste,

Beets weighing more than 5 pounds are usually of inferior
quality, but this depends more or less on the variety of seed,
the suitability of the soil and the method of cultivation. As
a general rule, however, the larger beets are poorer in quality,
so far as the sugar content is concerned, than the smaller or
medium sized ones.

Composition of the sugar beet.

The sugar beet usually contains from 3 to 5 percent. or
more of "marc" or matter that is insoluble in water. As a rule,
only two sugars, sucrose and raffinose are present in the nature
beet, though it may contain invert sugar, which is always found
in the immature root.

The following table 'l' gives the percentage composition of
there varieties of sugar beet:

	French Variety	German Variety	Czecoslovakia Variety
Percentage of sucrose	17.0	16.7	18.3
Density of the juce in Bx.	21.3	20.5	22.1
Percentage of reducing sugars in the juce	0.26	0.29	0.24
Percentage ash in 100 cc.	0.80	0.71	0.61
Apparant purity	87.10	86.60	88.10
Period of growth in days	183	175	212

In addition to sucrose, raffinose and chlorophyl, the beet
contains a large variety of compounds namely:
Organic acids, Oxalic, Formic, Citric, Malonic, Succinic, Aconilic
Tricarballicylic, oxicitric, maleic, and tartaric asids.

Nitrogenous bodies: Betaine, asparagine, glutamine, le
leucine, legumine, tyrosine, xanthine, guanine, hypoxanthine,

adenine, and coniferine.

Non-Nitrogenous bodies: Lecithine, and pectose.

Mineral constituants: Salts of potassium, sodium, rubidium, vanadium, calcium, magnesium, iron and manganese.

The bases are combined with hydrochloric, sulfuric, phosphoric, nitric and silicic acids, and the organic acids named in the preceding list.

Faffinose is present in small quantities and accumulates in the molasses. This sugar forms needle chaped crystals and is not injurious in the manufacture.

The greatest concentration of sugar in the beet root is around the middle and is about 17%, the top contains about 14% and the end about 15%.

'1' The establishment of Sugar industry in Iraq.
     The Ministry of Economics Baghdad, 1941.

     The sixth leaflet of the Department of Industrial Research.

THE MANUFACTURING PROCESSES.

The various stages of the manufacture of beet sugar may be outlined as follows:-

1. Washing the beets
2. Weighing the beets
3. Slicing the beets
4. Extracting the juce by the diffussion process
5. Purification of the juce
6. Concentration of the juce to a syrup
7. Crystallization of the sugar

    a. In the vacuum pan
    b. Crystallization at rest
    c. Supplemental crystallization with motion

8. Curing the sugar.

Washing the Beets

On receiving the beets in the factory they are carried up from the first floor into a beet washing machine by means of a spiral lift, called a "Beet Scroll" . The beet washer is simply a rectangular tank provided with paddles attached to a shaft which agitates and forwords the beets from one end of the washer to the other. On reaching the further end of the washer, the beets are lifted by means of a wider paddle and thrown into a bukkets elevator called " Beet Elevator", which carries the beets up to the upper most floor of the factory building.

Weighing the Beets:

On arriving at the top of the elevator, the buckets empty the beets into an automatic scale. The automatic scale is

usually so arranged as to weigh 1000 lbs. in one weighing. When filled with the required quantity of beets, the scale automatically closes the bottom of the hopper and empties beets into another hopper which directs them into the beet slicing machine.

Beet Slicer :

At the present time the diffusion process has replaced all others in the extraction of the juce from beets. The first step in this process, after the beets have been thoroughly washed and cleaned, is to cut the roots into thin slices termed, "Cossettes", and it is of great importance that these be of uniform thickness. The cossettes are usually 1:8 inch thick and 5:8 inch wide.

A beet slicer consists of a cylinder for holding the beets and a circular disk provided with about 20 rectangular openings towards the rim for receiving knife-holders,. Knife-holders are rectangular frames exactly fit in the openings. The knives are fixed on the holders by means of balts. The knives blades are of such a form that the cossette has a V shaped cross-section. The beets are piled in the cylinder and press on the knives by their own weight and as the disk which holds the knives is revolved, the beets are sliced and received on a belt conveyor called a "Cossette Conveyor" and are sent to the diffusion batteries.

Extraction Of The Juce

The juce was formarly extracted from the beets by means of hydraulic press. This method, however, has been entirely rep-

laced by the diffusion process, This process was devised by
Jules Robert, in France, and received its first practical
application in Austria.

The extraction of the juce from beets depends on the
phenomenon known as Osmosis' or "Diolysis" . Beet roots consists
of a great number of cells. These cells are alongated their
diameter varying from 0.014 mm. to 0.022mm. and their lenght
from 0.054 mm. to 0.089 mm. the cell walls are porous and
consist mainly of cellulose and pectine matter. The substances
dissolved in the cell-sap, can be devided into two kinds
according to crystallizability and deffusability through a
membrane; the first kind is the crystalloids and the second the
colloids. The former pass through a membrane more readily than
the latter. Sugar and salts belong to the crystalloids; albumins
to the colloids.

If we surround beet slices composed of a great number of
cells, whose contents are all nearly alike, with water; an
inter-change of water and soluble matter will take place. First
between the surrounding water and the outermost row of cells.
When the compositions of the contents of the first row of cells
becomes different from that of the second row, then the soluble
part in the second row pass to the first row and to the water,
and so on from the different rows until the composition of the
surrounding water becomes like the composition of the cells sep
after diffusion, then no more diffusion takes place as equilibr-
ium is established. For this reason beets are sliced into thin
slices so that this stage of equilibrium is quickly established.
There are, however, in sliced beets, about 6.4 % of torn and

bushed cells. The contents of such cells are simply washed away.

Temperature is an important factor in the process of diffu-
sion. Although its influence varies according to different substan-
ces, a higher temperature always causes mor or less rapid diffu-
sion. The diffusion of sugar from beet chips does not take place
in cold water but begines at 50° C. The following explanation has
been offered for the necessity of heating diffusion water: the
outer cell wall is lined from the inside by a layer of protoplasm
which resembles the cellulose wall in structure although it differs
in chemical composition. The protoplasm wall is much finer and the
sugar molecules are too large to pass easily through it. Heating
kills the protoplasm and changes the structure compleatly and it
becomes full of holes so it will no more hinder the passage of
sugar molecules. The temperature of the diffusion juce should be
raised to a point just enough to soften the cossets and not to
coock them, so to speak, as this will render the cossettes too
soft and cause them to lie one on another, closeing the screen
at the bottom of the diffuse, and obstructing the circulation of
the juce. Another objection to too high temperature is that it
causes more non-sugars to be diffused and makes the process of
crystallization more diffecult.

A suitable temperature for the diffusion process varies
according to the qualty of the beets; usually it is kept within
the limits between 78° and 85° C. when the beets are sound and the
purity of the juce is high, the temperature . ~ ~ ~ ~ ~

After drawing a quantity of juce from the ninth cell, the
first diffuser is cut out of the series and is emptied. Usually

may be kept as high as permissible. When the beets are unsound,
that is, fermented, unripe, or frozen, the temperature must be
kept at the lower limit.

Diffusion Apparatus.

The apparatus employed today for the extraction of the
juce from beets is called a " Diffusion Battery " consisting
of several Diffusors. The batteries commonly used consists of
10,12, or 14 diffusors. A usual form of a diffuzor os a cylinder
ending with cones, and provided with powerful covers. The
lower cone and cover are lined with screens so that the cossettes
may not pass/out of the diffusor. The lower cover is opened or
closed by means of hydrolic press use; after being closed, it
is held up tight by means of a latch. Each diffusor is provided
with a heating chamber called a " Calorisator ", and each
diffusor is provided with water pipes, valves for admitting wat-
water or juce or drowing water or juce.

In biginning the work, the first diffusor is filled with
fresh cossettes, and then warm water is poured into it through
the pipe at the bottom, while air aand gases are vented by
means of a cock in the cover. While the first diffuser is being
filled with water, the second is receiving cossettes, and so on.
When the first diffusor is filled with water, or as it may now
be termed, juce, this juce is passed through the heater of the
first diffusor into the heater of the second diffusor and into
the second diffusor; and when this is filled the juce is passed
to the third diffusor and so on. When nine cells of a battery,
made up of twelve cells, have been filled, and measured quantity
of juce is drown from the last, the circulation of the liquid

is being mentained by passing air or water into the first
diffusor of the series.

After drawing a quantity of juce from the ninth cell,
the first diffuser is cut out of the series and is emptied.
Usually all the diffusers but three are in operation at the
same time. After cutting off the first diffuser, a new one,
filled with fresh cossettes is attached last and the second
diffusor takes the place of the first and so on, the cossettes
in each diffusor remain in contact with the diffusion juce of
various densities for an average *time* of about 1 hour and 20 minutes,
or longer, the concentration of the juce increases from diffusèr
to diffusor, that of the first vessel contains only a small
fraction of a percent of sucrose, and the juce of the last cell
has a density of about four-fifths that of the normal juce.
Under favorable working conditions; the exhausted cossettes
contain from 0.15 to 0.25 % of sucrose, in terms of the weight
of the beets.

PURIFICATION OF THE JUCE

Straining or Depulping, the Juce

As it is drawn from the diffusion battery, the juce
contains large quantities of minute fragments of beet pulp.
These must be removed *prior* to the clarification, since the
pectin bodies would be decomposed, and at least in part, be
rendered soluble. The straining or depulping *is* and accomplished
by filtering the juce by a fine metal screen or perforated
metal sheet.

From now on, that is, the clarification, concentration,

crystallization, and curing of the juce and sugar are exactly
as described in the chapter on cane-sugar manufacture.

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C H A P. IV

SUGAR REFINING.

The second and third grade sugar i,e. sugar produced from mixed molasses and from molasses alone, is not fit for direct consumption and should undergo refining to be converted into white sugar.

The important point in sugar refining is the bleaching of the syrup, made by dissolving the raw sugar, by decolorizing charcoal and the subsequent crystallization of the pure syrup produced.

If the sugar is not very impure, then proper working will remove the coating of impurities and leave the pure crystals behind.

Sugar Refining Process:

The centrifugal sugar is mixed with syrup in a "Magma " Machine" so as to form a magma of about the consistency of massecuite. The syrup softens the coating of umpurities on the crystals and prepares them for washing in the centrifuge. The crystals are thoroughly washed in the centrifuge and the resulting sugar is very light straw, yellow color and of a poloriscope test of about 99° V.

If pure sugar is derived, this washed sugar is dissolved in the melters forming a solution containing about 50 % of sugar. The melted sugar is limed to slight alkalinity in the blow up tanks, and then sufficient phosphoric acid is added to combine with the lime. After thoroughly blowing up the syrup with

steam and skimming it, it is filtered usually through bag
filters.

The wash water and the syrup of the magma is pumped to
a separate blow up tanks as this syrup contains most of the
impurities.

From the bag filters, it is conveyed to a clear filter,
where it is passed under pressure through decolorizing charcoal.
The resulting colorless liquor is boiled in vacuum pans and
the crystals are cured in the centrifuge as usual. The molasses
produced are boild again and when the sugar produced is no
more pure, it is treated again as was described in the beginning
of the process, i.e., melted blown - up and decolorized etc....

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PART II.

THE MANUFACTURE OF

GLUCOSE

Hydrolysis Products Of Starch:

Starch is a highly condensed hexose carbohydrate of the formula ($C_6H_{10}O_5$)$_x$, consisting of approximately 100 anhydride groups which can be resolved by suitable hydrolytic agents into as many equivalents of dextrose, provided the hydrolysis is sufficiently prolonged. Dilute acids will produce complete hydrolysis, the rate depending on the kind of acid and varieng approximatly as the concentration, but increases rapidly with rise of temperature.

The profess of hydrolysis, or conversion of starch manifists itself by characteristic chemical and physical changes. The thick starch paste loses its colloidal nature and becomes more limpid, the concentration of the solution increases, and the solution becomes distinctly sweeter in taste.

If tested with a very dilute aqueous solution of iodine the deep blue color given by the original starch past changes as the hydrolysis proceeds, passing into violet then to a rose red which in turn changes to a reddish brown which grows lighter and lighter until justtbefore complete hydrolysis it disappears altogeather. A few drops of the solution poured into alcohol gives a copious white precipitate during the the early stages of conversion; as the hydrolysis continues the amount of precipitate becomes less until it disappears near the end.

Glucose.

The term " glucose " as used to define this product must not be confused with dextrose or the pure granulated mono-saccharide, but has referrence to a spacial commercial syrup which is always sold under this name. The name " corn - syrup " , which has been suggested fits better, as is the German " starkezucker Sirop." It is a thick, viscid syrup, practically colorless and clear, or of light amber tint, and is the product of partial hydrolysis of starch . Its composition varies somewhat but the average product has a specific rotation about 140°, with a Fehling reducing value of about 47% that of dextrose.

The above corresponds to a composition peculiarly well adapted to the commercial requirements of glucose which are; that it be practically " neutral " (flavorless) sweet syrup, uncrystallizable when highly concentrated and capable of forming uncrystallizable mixtures with cane sugar when the latter is added in large proportions. The syrup must also be capable of being refined so that it is practically colorless. This properties are achived by hydrolysing starch only so far that at least 25 % of dextrin still remains as such. If less dextrin is present the product tends to crystallize when highly concentrated and it will not prevent the " graining " of its mixture with cane-sugar. If more dextrin is present the product will be less sweet and looks cloudy due to the less soluble dextrin.

Manufacture Of Glucose.

A suspension of starch in water, having a density of about 20° Bé. is mixed with an acid, hydrochloric or sulfuric are the most generally used acids, the proportion of the acid is from 2 - 5 % on the basis of the dry starch.

The mixture is then led into a converter i.e. a vessel capable of withstanding internal pressurre to about 6 atmospheres. The mixture is heated by indirect steam in coils and is boiled under otmospheric pressure for about 10 minutes and then the convertor is closed and the pressure raised to about 2 otmospheres i.e. 30 lbs/in_{e}^{2} . A test sample is drown from time to time and tested with iodine, when the iodine gives only a faint brown color, in about 30 minutes, the steam is shut and the pressure released quickly so that the escaping gases will carry any smell due to the hydrolysis of glutines always present in the starch used in the makufacture of glucose. The batch is nexet cooled and the acid newtralized by using sodium carbonate if the acid used was hydrochloric or calcium carbonate if sulfuric acid was used.

The impurites which saparate in the neutral solution are filtered off in a filter press and the resulting clear liquid is concentrated in under reduced pressure to ad a density of 32° Bé.

On concentrating the solution the dissolved calcium sulfate become less and less soluble and saparates out, this is filterd off and the thin syrup is sent to be decolorized.

The decolorization is affected either by mixing the worm syrup with 2 % of decolorizing charceal stirred and then filtered

or is filtered through char-filters as was discribed in the part dealing with sugar rifining (Part I Chap. IV)

The colorless syrup is next concentrated to the final concentration usually $42°$ Be'. in a vacuum pan. The resulting syrup must be clear and colorless and shows no crystalliation on standing.

I made many trials to prepate glucose from starch and quite a number of them were successful.

The first difficulty I met with was the convertor, I used a pressure cooker instead but as the maximum pressure reached in, it was 20 lb / in^2 and I had no means of drawing test samples to follow the progess of hydrolysis; I tried different batches and heating for different times I started from 2 hrs. and decreased the time 10 minutes at a time.

Next came the question of the acid to be used, I had to use a higher proportion so as to affect conversion in a reasonable short time due to the low pressure, as increasing the time of heating resulted in a bitter almost black mass.

For thses reasons the use of hydrochloric acid wass unpractical because of the relatively high concentration of sodium chloride, in the final product, in fact the samples prepared in this way had a stronger salty taste than sweet. So the acid used was sulfuric acid and the best proportion used was found by experience to be 7 cc. of the conc. H_2SO_4 for every $\frac{1}{2}$ kg. starch and the amount of water was $2\frac{1}{2}$ lt.

This mixture was boiled under otmospheric pressure for

10 mins. the starch was mixed with part of the water to form
a thin paste and was added to the boiling acidulated water at
such a tate that boiling did not stop in order to prevent
lump formation.

The mixture in a copper kettle, was next put in the
pressure cooker and heated to the maximum pressure obtained
i.e. 20 lb. / in$_2^2$. The first time the heating was kept for
2 hrs. at the highest pressure and the result was a black
bitter solution. Then on decreasing the time of heating, the
right period of heating was 30 mins. after the pressure reaches
the 20 lb. mark.

Calcium carbonate was used for neutralization and no
difficulties wore met in filteration as the precepitate was
floculant and settled quite rapidly.

The decoloration was affected in two stages, the first
directly after neutralization and filtration and 1% of "Darco"
was used at a temperature about $30 - 40°$ C and the mixture was stirred
for 1/2 hr. and then filtered, this gave a practically water clear
and colorless solution.

Concentration was made in an ordinary round bottom 2 lit.
pyrex flask and the vacuum was obtained by the use of an
ordinary suction pump which gave a vacuum, even more than
necessary, which caused the solution to boil between $40 - 45°$ C

The volume of the syrup was reduced from about 2½ lit. to
1 lit. and was mixed with 1% of "Darco" and kept worm for ⅓ hr.
and then filtered with suction, this removed the dark yellow
color and the precipitated calcium sulfate. The resulting syrup

was next concentrated again to final valume of about 400 cc.
which gave a very pale yellow and clear syrup of a density
about 40° Be'.

The earlier samples, which were hydrolyzed for a period
of 1 hr. showed crystallization of standing and in a monthes ti
time the whole became one solid mass of glucose hydrate which
were dried in a dessicator and pulverized, with difficently, to
a powder.

PART III

GENERAL METHODS FOR

SUGAR ANALYSIS

CHAPTER 1

Qualetative Analysis.

Probably no other class of organic compounds gives such a variety of reaction or form so large a number of chemical derivatives as the sugars. Owing to the great extent of the field only the most important reactions will be mentioned here.

For convinience in identification, sugars will be classified under two general groups;

      1. Reducing Sugars

      2. Non-reducing sugars

The reducing sugars are distinguished by the fact that they form a precipitate of cuprous oxide when heated with alkaline fehling solution while the non-reducing sugars do not form a precipitate or form it to a very slight extent and after prolonged boiling. The reducing sugars constitute by for the largest group. The mono-saccharides and many of thier derivatives reduce fehling solution. Most of the disaccharides including moltose and lactose exhibit this property. The best known nonreducing sugars are sucrose, a disaccharide, and raffinose, a trisaccharide.

GENERAL REACTIONS OF REDUCING SUGARS

The general tests for reucing sugars may be devided, for convience, into the following general groups of reactions.

  I. Reducing reaction with solutions of metallic salts or of organic compound.

  II. Color reactions with alkalies, acids, phenols, and other organic compounds.

  III. Hydrozone and osazone reactions with phenyl-hydrozine and its substituted derivatives.

  IV. Miscelanious reactions .

1. Reducing reactions of Sugars:

The simple sugars and certain of the disaccharides have the property of reducing alkaline solutions of many metallic salts such as those of copper, silver, mercury and bismuth. This reaction which is common to most aldehydes is due to the reduction of the metallic base and precipitating it in the form of the suboxide or the metallic form.

In the simplest case the aldehyde groups is oxidized to the carboxyl group.

If an alcohol group is also present further oxidation converts it into the carbonyl and further into the carboxyl as in glycol aldehyde

$$\begin{matrix} H_2 - COH \\ | \\ HC = 0 \end{matrix} + 2Ag_2O \longrightarrow \begin{matrix} COOH \\ | \\ COOH \end{matrix} + 6Ag + H_2O$$
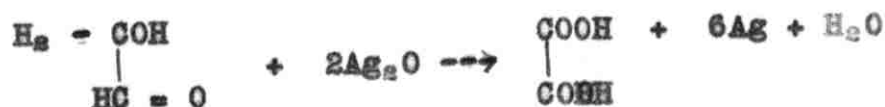
This oxidation, in the case of the higher mono-saccharides, is usually attended by breaking down of the carbon chain as

by the oxidation of glucose in ammonical silver solution.

$$C_6H_{12}O_6 + 9Ag_2O \longrightarrow 3(COOH)_2 + 18Ag + 3H_2O$$

The reaction between sugars and alkaline salts of metals as carried out, gives rise to a number of monobasic and dibasic acids (formic, oxalic, etc.) in varying proportions according to the conditions of the experiment. It is not possible, therefore, to express the reaction by chemical equations in a very general way.

The most common of the alkaline salt solutions employed in testing sugars are those of copper. The sulfates and acetates of copper are the salts most generally used, and sugar literatures is filled with modifications for making the tests, Only a few of the important solutions will be described here.

Fehling's Copper Solution:-

This is the more common chemical reagent employed in testing sugars. As ordinarily prepared, the reagent consists of two solutions; solution 'A' containing 34.64 grms. $CuSO_4$. $5H_2O$, in 500 cc. water and 'B' containing 173 grms. Rochelle salts and 50 grms; sodium hydroxide in 500 cc. water

By mixing equal volume of A and B, and boiling it with a sugar solutions a brick-red precipitate of cuprous oxide forms if reducing sugars are present. The intensity of coloration and amount of precepitate is proportional to the amount of sugar present. The test is sensitive to about 0.01 mg. of glucose per 1.cc..

When solutions A and B are mixed and cupric hydroxide is formed which forms a complex with the hydroxy acid present thus

by the oxidation of glucose in ammonical silver solution.

$$C_6H_{12}O_6 + 9Ag_2O \longrightarrow 5 (COOH)_2 + 18Ag + 5H_2O$$

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When solutions A and B are mixed and cupric hydroxide is formed which forms a complex with the hydroxy acid present thus

decreasing the concentration of the cupric ions below the requirements of the Ksp for cupric-hydroxide. When the solution is boiled with a reducing sugar the free cupric ions are removed in the form of the insoluble cuprous oxide which causes the complex to furnish more free cupric ions and so on.

$$\begin{array}{c} COONa \\ | \\ HC\text{-}OH \\ | \\ HC\text{-}OH \\ | \\ COONa \end{array} \quad + \quad CO(OH)_2 \quad \underset{\longleftarrow}{\longrightarrow} \quad \begin{array}{c} COONa \\ | \\ HC\text{-}O \\ | \quad \quad \rangle Cu \\ HC\text{-}O \\ | \\ COONa \end{array} \quad + \quad 2H_2O$$

Products obtained by heating reducing sugars with Fehling's soln.

The chemical reactions which take place in the oxidation of sugars by means of Fehling's solution is exceedingly complex. Nef '1' who made the first systematic standers in this field, found that in l-arabinose the oxidation proceeds along three separate lines

    1. From 10 to 25 % are oxidized to form pentonic acids

$$C_5H_{10}O_5 \; + \; O \; \longrightarrow \; C_5H_{10}O_6$$

    2. From 35 to 45 % of sugars are oxidized to form formic and trioxybutyric acid.

$$C_5H_{10}O_5 \; + \; 2O \; \longrightarrow \; HCOOH \; + \; C_4H_8O_5$$

    3. From 30 to 38 % of sugars are oxidized to form formic and glycolic acids.

$$C_5H_{10}O_5 \; + \; 3O \; \longrightarrow \; HCOOH \; + \; 2C_2H_4O_3$$

With the *hexose* sugars, d-glucose, d-mannose, and d-fruc-tpse. Mef obtained analogous reactions with *the* formation of carbonic formic, glycolic, glyceric, trioxybutyric, and hexonic andd. The formation of all these products has been explained on the basis of the fact that the reducing sugars are not stable in alkaline solutions, but undergo molecular rearrangement and are partly broken up into compounds with shorter carbon chain. The cupric oxide in Fehling's solutions oxidizes the reaction products, with the formation of the acids named above. The primary effect of alkalies on sugars will be described more fully later.

Benedicts' Copper Solution

Instead of the sulfate- tartrate, solutions, of other copper solutions have been employed in testing for sugar.

Benedicts' reagent is '2' prepared by dissolving 173 gm. of sodium citrate and 100 gm. of anhydrous sodium carbonate in about 800 cc. of water and 17.3 gms. $CuSO_4.5H_2O$ in about 100 cc. water and making the volume to 1 lit. The solution have an advantage over Fehling's solution and not being reduced by creatine, creatinine, uric acid, or simlar substance so it is especially adopted for *the* testing of urine.

Badfords' Solution '4'

The reagent contains 13.3 gms. crystalline cuprric acetate (neutral) in 200 cc. water and 5 cc. of 38 % acetic acid. Mono-saccharides only reduce this solution but not disaccarides such as lactose.

'1' Ann. 357, 214, 312, (1907)
'2' J. Bid. Chem. 5, 465 (1909) Samson J.Am.chem. Soc. 61, 2389 (1939)
'3' Ber., 15, 1655, (1882); 16, 921 (1883)

Tollɵɪn's Silver Solution

The most sensitive ot metallic - salt solutions for detecting sugars is ammonical silver solution, first employed by Tollens and hence usually known as Tollens reagent. This; is prepared by dissolving one part of silver nitrate in 10 parts of water; a second solution is then made containing, one part sodium hydroxide in 10 parts of water.

Before making the test equal parts of the two solutions are mixed and then ammonia is added drop by drop until the precepitate of silver oxide is completely dessolved. A solution containing one part glucose in 1000 parts of water will cause a strong reduction of Tollen's reagent in the cold, a mirror of silver being deposited within 5 minutesⱡ A solution containing one part glucose per 100,000 parts water also produce a perceptible reduction in the cold but the solution should be kept for 1 to 2 days.

Tollen's silver reagent is also reduced by all aldehydes; it is affected not only by the sugars which reduce Fehling's solution but also by sucrose, raffinose, and all other soluble carbohydrates.

Even the alcohol derevatives of the sugars produce reduction, glycerol, for example, causing the formation of a silver mirror, the readucers (readiness) with which ammonical silver solutĭon is reduced by soluble organic non-sugars., has proved a serious objection against the use of this reagent in ordenary analytical work.

'2' J.Biol. Chem. 5, 485(1909;); Semson J.Am.Chem.Soc.61,2387 (1939).

'3' Ber., 15, 1635 (1882); 16, 921 (1883).

Reduction of Nitro-Compounds:

Among the reagants used for detecting sugars on the basis of thin reducing effect, are also included a number of organic compounds.

Picric acid test; Picric acid $C_6H_2(NO_2)_3.OH$, is reduced to amino-nitro-phenol $C_6H_2(NO_2)_2.NH_2OH$, which has a deep red color. The reaction first observed by Baun '4' has been widely used for detecting sugars in boiler feed water. The water sample is boiled with few drops of hydrochloric acid to invert the sucrose and 2 to 3 drops of an alcoholic solution of picric acid is added. The reaction gives posetive results at a sugar concentration of 1:5000.

O-Dinitrobenzene test. This is carried out as follows according to Bose '5', One drop of 1 % solution of the reagent in alcohol is mixed with 2 cc. of 25% solution of sodium carbonate and 1 cc. of the sugar solution is added. Upon heating the mixture for 15 to 20 secs. a deep violet color appears which fades gradually. The reaction is very sensitive, permitting the detection of 6 parts per 1,000,000 of glucose, fructose, galactose, mannose, and lactose or 3 parts per 1,000,000 of arabinose. Non reducing sugars do not give the test.

Other nitro compounds such as m-dinitrobenzene, m-or/p-nitro bfnzaldehyde and dinitro salicylic acid, give simler reactions.

'4' Z. anal. Chem. 4, 185 (1865m).
'5' Z. anal. Chem. 87, 110 (1932).

II. COLOR REACTIONS OF SUGARS WITH ALKALIES, ACIDS, PHENOLS, ETC.

As a second general reaction of reducing sugars may be mentioned certain color effects which nearly all soluble carbohydrates give when, brought into contact with different reagents. The reagents employed may be devided into three groups:

      a. Alkalies

      b. Concentrated mineral acids.

      c. Phenols and other orgainc compounds.

Color Reactions of Sugars with Alkalies:

All reducing sugars have the property of coloring solutions of the hydroxides of alkalies and alkaline earths metals yellow, the applecations of heats darkens the color to brown. The exact nature of the coloring matter formed is not understood. Considerable oxygen is absorbed during the reaction, and various products of an acid nature are among the substahces formed.

Products Obtained by Heating, Reducing Sugars with Alkalies:

Lactic acid is produced in considerable amount by the action of alkalies on mahy reducing sugars such as xylose, arabenose#, glucose and fractose. The presence of caletum lactate in certain sugar-cane molasses is explained by the action of an excess of lime during clarification upon the reducing sugars of the juce. Formic, acetic, and oxalic acid have also been found.

Nef '6' has developed a theory explaining the formation of these various reaction products. It is based on the suggestion originally advanced by Wobe and Neuberg, that sugars may react as enediols.

The alkalie causes a splitting of the ring, the aldehyde

or keto form is produced and the enediols are formed by the migration of hydrogen atoms. d- Glucose, forinstance, may form the following three isomers:

$CH_2OH - CHOH - CHOH - CHOH - COH = CHOH (1,2 \text{ enediol})$

$CH_2OH - CHOH - CHOH - COH = COH - CH_2OH (2,3 \text{ enediol})$

$CH_2OH - CHOH - COH = COH - CHOH - CH_2OH (3,4 \text{ enediol})$
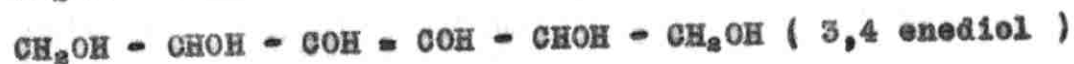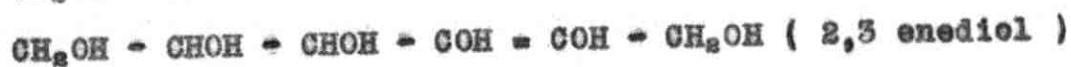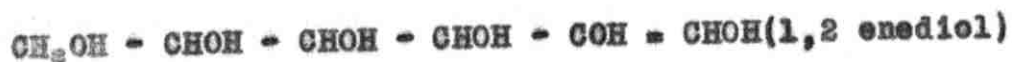
Increase in the alkali concentration shifts the equilibrium from the 1,2 enediol toward the 3,4 enediol.

These reactive substances undergo further transformation in various ways, such as:

1. Splitting of enediols:

When the enediols split at the double bond they form the corresponding aldehydes. The resulting pentoses, tetioses and trioses may form again the enediol, split further into corresponding aldehydes. In the presence of an oxidizing agent this form the corresponding acid. This is the cause of the reduction of Fehlin's solution and other related reagents.

2. Shifting of a hydrogen atom:

By the shifting of the hydrogen atom of glucose 1,2 enediol, it may change into either fructose or mannose as all the three sugars have the same spatial confeguration in the last four carbon atoms counting the carbon atom of the carbonyl group of glucose as No. 1.

The action of alkalies on reducing disaccharides is the same, namely the formation of enediols although the reactions

folloing that are much more complecated but yet theygive the aldehyde groups by breaking at the double bond which reduces Fehling's solution.

Color Reactions of Sugars with Mineral Acids

Treatment of solutions of sugars with concentrated mineral acids gives rise to a number of decomposition products with a development of a color the intensity of which depend on the kind or sugar, the concentration of the acid used and the temperature. By heating a sugar solution with concentrated hydrochloric or sulfuric acid a brown color develops which is due to the formation of an insoluble so-called humus substances of relatively high carbon content ($C = 62 - 67 \%$ and $H = 3.5 - 4.5 \%$) the percentage carbon and depth of color increasing on the concentrations of the acid used.

In addition to the insoluble humus, a number of soluble and volatile compounds are produced, such as formic acid, levulinic acid, furfural, methyl furfural, hydroxymethyl - furfural, and a number of dextrin like condensation or reversion products.

The ketoses are much more easily decomposed by strong mineral acids than oldoses, and their solutions give rize to color reactions with correspondingly greater ~~faintely~~ facility. This offers a mean of distinguishing between ketoses and aldoses or detecting ketoses in the presence of aldoses.

If concentrated sulfuric acid is added to a cold solution of a sugar in a test tube so that the acid will flow on the sides of the test tube and form a layer at the bottom, a rose-

color to brown ring is produced as the junction if the solution contains a ketose such as fructose or sorbose or if the sugar gives rise to a ketose on hydrolysis such as, sucrose, raffinose or inulin. With glucose, lactose, maltose and the aldoses in general the color will develop only slowly or not at all

As if a few of the sugar crystals are placed on a wad of asbestos moistened with concentrated hydrochloric acid in a test tube, the crystals will be colored violet in a short time if the sugar is a ketose or gives a ketose on hydralysis. Although the latter sugars give the test a little more slowly. Aldoses develop only a light yellow color in 2 to 3 hours.

Color Reactions of Sugars with Phenols.

The most distinctive color reactions of the sugars are those obtained by treatment with different phenols in the presence of concentrated sulfuric or hydrochloric acid. The development of color in this case is due to the formation of condensation prodects between the phenol derivatives and the decomposition products obtained from the sugars, particularly furfural and its derivatives. a-Naphthol, thymol, resorcinol, orcinol, naphtharesorcenol, phloroglucinol, cresol, and lysol are among the more important phenolic compounds used for making color reactions with sugars. A typical example of the conden- sation product obt between furfural and the phenols is di-a- naphthafurylmethane, $C_{25}H_{18}O_3$, which was obtained by Bredereck from a-naphthol and furfural. It is collrless, but dissolved in concentrated sulfuric acid with a deep violet color.

The color reactions with the phenols are performed in various ways, the test with a-naphthol, Molisch test, for example,

which is perhaps used more frequently than any of the others, is made as follows: 1 to 2 cc. of the sugar solution is heated in a test tube with 1 to 2 drops of 10 to 20 percent alcoholic solution of α-naphthol . A few cubic centimeters of concentrated sulfuric acid are then carfully added so as to flow down the walls of the tube to the bottom.

If sugars containing a ketone group are present a violet ring will form instantly as the junction of the two liquids; in the presence of aldoses a gentle warming of the test tube is usually nedessary to bring out the full intensity of the color. The α-naphthol test, which is of extreme delicacy is frequently employed in sugar houses and refineries in testing the condene sation water form the vacuum pans for presence of sucrose lost by entrainment.

The test with phenols and hydrochloric acid are usually made by warming a few cubic centimeters of the sugar solution with a solution of the phenol in concentrated hydrochloric acid. the coloration thus obtained are usually very brlliant, varying in tent from a bright red to a bluish violet. The colors formed are not permanent, however, they rapidly darken and the clear colored solution soon becomes turbid with the precepitation of a dark colored condensation product.

Glacial acetic acid may be also used instead of hydrochloric acid to dissolve the phenols employed in these tests. Under these conditions the solution remain clear and can be used direc- tly for spectroscopic examination or for colorimetric comparison.

III. HYDRAZONE AND OSASONE REACTIONS OF REDUCING SUGARS

WITH PHYNYL.- HYDRAZINE AND ITS SUBSTITUTED DEREVATIVES

In many respects the most impodtant of the quali-
tative tests for sugars are those obtained with phenyl hydrazine
and its substituted derevatives. Phenylhydrazine was introduced
as a reagent in sugar chemistry by Emily Fipher '7' in 1884;
it has been of immerse service not only as a means of saparation
and identification but also in first opening a way to a thorough
inderstanding of the molecular constitution of sugar.

Hydrazone Reaction: The reaction with phenylhydrazine is
limeted to such sugars as contain a free carbonyl group and
proceed two phases with the production of two entirely different
classes of compounds. The first phase of the reaction is common
to all aldehydes and ketones the oxygen of the carbonyl group
combining with the two atoms of hydrogen of the amine group
in the phenylhydrazine with the formation of a group of compounds
called hydrazones.

The hydrazone reaction is carried out by treating the sugar
solution in the cold with a solution of phenylhydrazine in
dilute acetic acid,, Or the sugar solution may betreated with
crystals of phenylhydrazine-hydrochloride with some solid
sodium acetate. After the above treatment the hydrazone of the
sugars will saparate sooner or later as well-defined crystalline
compounds, the length of time for saparation depending upon the
solubility of the hydrazone formed. The phenylhydrazone of
mannose, for example, being very insoluble, will saparate
aomost immediately, those of ketones such as fructose, and

rhamnose also deposit readily; the phenylhydrazone of glucose
on the other hand, being very soluble in water, may require
1 or 2 days for its precepitation. By filtering off the hydrazones
as they are form, and saparation of the sugars in mixtures may
often be acomplished.

In place of phenyl hydrazine any of its substituted
derevatives may be used for the purpose of precepitating sugars.
The substituted phynylhydrazines yield in many cases character-
istic hydrazones with sugars and their use in sugar chemistry
has been of greater service. Of the various substituted phenyl-
hydrazines the following are among the most important.

Methyl, Ethyl, Amyl, Allyl, Phenyl, Benzyl, p-Bromo and
p-Nitro phenylhydrazines and Naphthylhydrazine.

The reactions with the substituted phenylhydrazines are
usually carried out in alcoholic solutions, the hydrazones
formed being for the most part much less soluble than those of
ordinary phenylhydrazine. A table of melting points will then
identify the hydrazone of the sugar.

The sugar can be regenerated and saparated from the
hydrazone either by treatment with concentrated hydrochloric
acid, as employed by Fisher, where the hydrazone is changed into
phenylhydrazine hydrochloride, which is insoluble in concentrated
hydrochloric acid, and the sugar is set free.

The other method is by refluxing the hydrazone a 50%
alcoholic solution of the hydrazone with an aldehyde. Benzaldehyde
is used with phenylhydrazones and formaldehyde with the substitu-
ted hydrazones. The hydrazones of the aldehydes being insoluble
are removed and the sugar removed by crystallization.

'7' Ber, 17, 579 (1884).

Asazone Reaction:

While the hydrazone reaction is of perminent value in the
isolation of sugars, the asazone test with phenylhydrazine is
usually of more qualitative significance owing to the greater
insolubility of the osazone in water and the consequent greater
rapidity and ease of thin saparation as compared with the
hydrazones.

The reaction is carried out by heating in a boiling water
both a solution of the sugar with excess of phenylhydrazine,
and acetic acid. first the hydrazone is formed then the hydroxyl
group of the α- carbon atom is oxidized into a carbonyl group on
the expense of one molecule of phenyl hydrazine which is reduced
to aniline and ammonia. A third molecule of phenyl hydrazine then
reacts with the new carbonyl group to form the osazone.

The time of formation of the osazone, crystal structure,
and melting point are used to identify the sugar

One of the important limitations of the use of the
osazones in identifying sugars is that, whith sugars which
differ only in the spacial arrangement of the first two carbon-
atoms such as d-glucose, d-fructose and d-mannose, will yield
identical osazones

Methylphenylhydrazine forms osazones readily with ketones
but not with aldoses so it will partly help in eleminating
the above mentioned limitstion of used along with phenylhydrazine.

REACTIONS OF THE NON - REDUCING SUGARS .

The comparatively small number of sugars which do not reduce Fehling's solution all belong to the higher di-tri- and tetra- saccharides and include sucrose, difructose anhydride, raffinose and other rare sugars, some of them not completely studied yet. The soluble polysaccharides such as, dextrin, inulin, and glycogen, although not classified as sugars, are sometimes included, for convinience, in the group of non reducing sugars.

A free aldehyde or ketone group, to which the reducing sugars owe thein peculiar reactivity, is lacking in the non-reducing sugars so they do not reduce Fehling's solution and the related reagents not from hydrazones and osazones or react with dilute alkaline or show other reactions typical of reducing sugars.

The non-reducing sugars give many of the color reactions with phenols. But as previously explained these reactions are not given by the non-reducing sugars as such, but by the reducing sugars derived from them due to the hydrolytic action of the acid used in making the reaction.

Some of the special tests which may be used to detect the most commonly encountered non-reducing sugar, sucrose, are described here:

Raybin's Test '8'

If 40 to 50 mg. of sucrose are dissolved in few cubic centimeters of 0.05 N sodium hydroxide solution, is shaken at about 10 10° 6e in a stoppered test tube with 7 to 10 mg. of diazourasil, until the latter dies dissolves , a blue-green

color developes in few minutes. In the presence of soluble mag-
nesium salt and stable blue precepitate forms.

This test is not given by glucose, fructose, or most other
sugars, but by sugars characterized by the fructofuranose-
glucose linkage such as raffinose.

Pictet Test for Sucrose.   Pictet '9' observed that a mixture
of cold, saturated solutions of sucrose and of copper sulfate
in water, after standing for several hours, deposits microscopic
needles of a double salt $C_{12}H_{22}O_{11}$. $CuSO_4.4H_2O$. This reaction
is not given by any of the other common pentoses, hexoses, di and
tri-saccharides or by the synthetic sucrose isomers. But in
mixtures, at least 10 % of sucrose must be present to produce
a positive test.

Schlemmis' Test for Sucrose in the Presence of Inverted Sugar

This is based on the fact that reducing sugars are dest-
royed by boiling with milk of lime, and do not give the usual
sugar reactions after this treatment. 20 cc. of the solution to
be tested containing not over 2 % of total sugar is mixed in
a test tube with 2 cc. of 10 % milk of lime and the tube is
placed in boiling water bath for 10 - 12 minutes. The tube is
now centrifuged, the supernatant liquid pipetted off and
tested with naphthol or thymol. If the color reaction connot be
readily seem because of the yellow tint of the treated sugar
solution, a dichromate filter is used for the observation.
This is easily accomplished by placing the test tube in which

'8' J.Am.Chem. Soc. 55, 2603 (1933); 59, 1402 (1937).
'9' Helv. chem. Acta, 16, 144 (1933).

the α-naphthol test is carried out, in a beaker filled with a
0.25 to 0.5 % solution of potassium dichromate to make^(match) the color
of the treated sugar solution. Under thses conditions a positive
test for sucrose may be obtained at a concentration of 0,001 %
Raffinose and reversion products of reducing sugars also give
this test, also commercial glucose which always contain reversion
products. The test is therefore specific only in the absence
of these other carbonhydrates.

————————————

SYSTEMATIC PROCEDURE FOT THE DETACTION OR IDENTIFICATION
OF CARBOHYDRATES

In order to facilitate the identification of carbohydrates which the chemist may encounter, various schemes for a systematic procedure have been developed. The identification may be based on chemical reaction, on fermentation with yeast, fungi, or bacteria, or on the effect produced by enzymes. The example given here is based on chemical reactions.

Identification by Chemical Reactions.

Dehn Jackson and Ballard '10' have compiled a table of chemical reagents used for the detection of carbohydrates and another table showing the tests given with these reagents by a number of carbohydrates. These are reproduced in tables 1, and II. The meaning of the figures and signs in table II are explained under "Remarks" in table I, and that of the abbreviations in a footnote below table II.

'10' Ind. Eng. Chem., Anal, Ed., 4, 413 (1932).

TABLE I   REAGENTS

Reagent	Solvent	Amt. of reagent	Time heated at 100C Mins.	Elapsed time before reading Mins.	Remark.
		Percent			
a. Water	(1) H_2O				Cellulose insoluble
b. + K + I	H_2O	3	1	1-5	Heat to expel free iodine; cool.
c. Nylanders (1)	8% NaOH	6	1	1-5	(+) Reagent catalyzed with $AuCl_3$; bk. pp't's; (wh) white pp't's
d. Disch's	(3)		30	30 +	Pentoses brown; hexoses etc. blue
e. Orcinol	(4)		1	1-5	See table I
f. Phloroglucinol	(5)		1	1-5	See table II; boiling gives CHR, with Pectin, gum arabic and gum tragacanth
g. Na_2CrO_4	H_2O	10	1	1-30	Simple sugars green; pectin, red solution
h. Sodium nitro-prusside	H_2O	1	1 +	?	Add NH_4OH and heat; (+) green while hot; arabinose green without NH_4OH
i. $KMnO_4$	H_2O	1-0	"	1-60	(+) immediate decolorization; (#) decolorization in 1 hr.
j. Fehling's(c)	H_2O	"	"	60	Add $FeCl_3$; brown, negative; green, positive; maltose; blue.
k. $K_3Fe(CN)_6$	H_2O	0-5	1	1-5	Add NH_3 squares inplate depth of color (red).
l. Picric acid	H_2O	sat.	1	1-5	Yellow to amber to brown to black. ber = depth of color
m. H_2SO_4	H_2O	35	1	1-60	Yellow to amber to brown to black. ber = depth of color
n. $HClO_4$	H_2O	20	1	1-60	Yellow to amber to brown to black. ber = depth of color
o. H_2SO_4	H_2O	50	1	160	Yellow to amber to brown to black. ber = depth of color
p. HCl	H_2O	20	1	1-60	Yellow to amber to brown to black. ber = depth of color.
q. KOH (+)	H_2O	5	1	1-60	Nos = depth of color; deep yellow.
r. $(NH_4)_2MoO_4$	H_2O	5	"	60	PPP carbohydrate; then tar.
s. HNO_3	H_2O	25	"	-	H_2SO_4. Nos = depth of color; rahamose green.
t. $H_2S_2O_3$	50% H_2SO_4	10	1	1-60	Nvan. 2/3 vol. for muoid ace. Nos = depth of red.

TABLE I REAGENTS

Reagent	Solvent	Amt. of reagent Percent	Time heated at 100C Mins.	Elapsed time before reaction Mins.	Remark.
a. Water	(1)				Cellulose insoluble
b. + KI	H₂O	3	~	~	Heat to expell free iodine; cool.
c. Nylanders (1)	8% NaOH	6	1	1-5	Reagent catalyzed with AuCl; (+) bk. ppt's; (wh) white ppt's
d. Disch's	(3)		30	30+	Pentoses brown; hxoses etc. blue
e. Orcinol	(4)		1	1-5	See table 1
f. Dhkoroglucinol	(5)		1	15	See table 1; boiling gives CHR, with pectin, gum arabic and gum tragacanth
g. Na₂CrO₄	H₂O	10	1	1-30	Simple sugars green; pectin, red solution
h. ...oprusside	H₂O	1	1+	?	Add NH₄OH and heat; (+) green while hot; arabinose green without NH₄OH
i. KMnO₄	H₂O	0-1	~	1-60	(+) immediate decolorization; (*) decolorization in 1 hr.
j. Fehling's	H₂O		~	60	
k. K₃Fe(CN)₆	H₂O	0-5	1	1-5	Add FeCl₃; brown, negative; green, positive; maltose, blue.
l. picric acid	H₂O	sat.	1	1-5	Add NH₃ numbers indicate depth of color (red).
m. H₃PO₄	H₂O	85	1	1-60	Yellow to amber to brown to black. Num- ber = depth of color
n. HClO₄	H₂O	60	1	1-60	Yellow to amber to brown to black. Num- ber = depth of color.
o. H₂SO₄	H₂O	50	1	1-60	Yellow to amber to brown to black. Num- ber = depth of color.
p. HCl	H₂O	20	1	1-60	Yellow to amber to brown to black. Num- ber = depth of color.
q. KOH (+)	H₂O	5	1	1-60	Nos. = depth of color; pectin yellow.
r. (NH₄)₂MoO₄	H₂O	5	~	60	Add carbohydrate, then ½ the vol. code. H₂SO₄. Nos = depth of color; rahmnose green.
s. HNO₃	H₂O	25	~	-	Evap. 2/3 vol. for mucic acid.
t. H₂S₂O₈	50% H₂SO₄	10	1	1-60	Nos = depth of red.

Table I Contd.

u. Reagent	Solvent: A wt. of reagent (8) Percent	Time heated at 100C Mins.	Elapsed time reagent Mins.	Remark.
t. Na₂O₃				Nos # relative speed of decolorization of Na₂O₃
v. Heroine	H₂SO₄	1	#	15	Mostly ambers to brown black (9)
w. Codiene	H₂SO₄	1	#	15	Mostly rose to brown black (10)
x. AgNO₃	Dil. NH₄OH	1	1	1-60	(+) black ppt's; or silver mirror; some give brown solution.
y. p-tolylhydrazine Dil. HCl	1	#		1-60	Boiling gives for (+) dichromate color; levulose and sucrose give color and ppt.

1. Mono-, di-, and triy saccharides are sweet and soluble in cold water. Polysaccharides, dissolve in hot water. Starch, pectin, glycogen, and gum tragacanth dissolve in hot water to give opalescence and foam. Gum arabic gives clear solution and foam. Dextrin an inulin give clear solution but no foam.

2. Nylander's reagent: 2 g. of bismuth subnitrate and 4 g. of Rochelle salt dissolved in 100 g. of 8% NaOH

3. Prepare 10% solution of diphenylamine in alcohol and add 1 volume to 4 volumes of acetic acid and 5 volumes of HCl.

4. To 5 cc. of saturated aqueous solution of ercinol, add 50 cc. of HCl and dilute to 90 cc.

5. to 0.1 g. of phloroglucinol in 10 cc. of alcohol, add 50 cc. of HCl and dilute to 90 cc. Solution of ercenol or phloroglucenol in dil. HCl, will indicate carbohydrates containing levulose.

6. Fehling's reagent. (1) solution of 36.44 g. $CuSo_4.5H_2O$ in
   water diluted to 500 cc. (2) solution of 125 gr. KoH and
   173 g. of sodium potassium tartrate in water diluted to 500 cc.

7. Usually called Moor's test.

8. Prepare solutions (a) 10 gr. of $NiS0_4.7H_2O$ in 1000 cc. of
   H_2O and (b) 4.8 gr. of $K_2S_2O_8$ and 6 gr. of KOH in 1000 cc.
   H_2O.Use equal parts and let stand. Black Ni_2O_3 precepitates.
   Carbohydrated decolorize this.

9. With varied times and temperatures. carbohydrates give changing
   colors. A check with a known carbohydrate $\overset{is}{and}$ revealing and
   disirable. Perhaps in these reactions, formoldehyde is split
   off, because color closely follows Kobert's test for codiene
   etc.

_____

TABLE II

EFFECTS OF VARIOUS REAGENTS ON CARBOHYDRATES

Carbohydrate	a	b	c	d	e	f	g	h	i	j	k	l	m	n	o	p	q	r	s	t	u	v	w	x	y
1. Cellulose	•	•	•	Bl	•	•	•	•	•	•	•	Br	•	•	•	•	•	8	•	•	•	R	r	•	•
2. Starch	+	+	wh	Bl	•	•	+	•	•	•	•	Br	1	•	•	•	•	6	•	•	•	r	r	•	•
3. Double Starch	+	+	wh	Bl	•	•	•	•	•	•	ros-	Br	4	•	•	•	•	6	•	5	•	•	•	•	•
4. Glycogen	+	+	wh	Bl	•	•	•	•	•	+	1	G	1	•	9	3	•	5	•	1	1	PV	Br	•	•
5. Dextrin	+	+	wh	Bl	Am	•	•	•	+	+4	2	G	•	•	9	•	•	5	•	1	3	Br	Br	++	•
6. Pectin	+	+	•	Br	R	•	•	+	+	+	2	G	1	•	•	+	+	•	•	1	1	Br	Br	+	•
7. Levulose	+	+	+	Bl	Br	Br	+	+	+	+	9	G	9	9	9	9	9	9	•	9	4	Br Bk	Br Bk	+	•
8. Mannose	+	+	+	Bl	•	•	+	•	+	*	1	G	1	1	3	1	1	7	•	5	7	V Vp	V Vp	•	•
9. Maltose	+	+	+	Bl	Y	•	+	+4	++	+	3	Bl	1	3	5	1	2	6	•	3	9	Am r	F-+	+	•
10. Rhamnose	+	•	•	Br	•	Or	+	+	+	•	2	G	5	6	•	2	2	4	•	8	4	Or R	Br	•	•
11. Arabinose	+	•	•	Br	Bl	Ch R	+	+	+	•	1	G	2	2	•	7	7	7	•	5	8	f R	R	+	•
12. Xylose	+	•	•	Br	Bl	ChR	+	+	+	*	1	G	•	7	•	3	5	5	+	7	8	R R	R R	+	+
13. Galactose	+	+	+	Bl	Am	•	+	+	+	+	4	G	+	3	•	3	4	6	+	6	5	R R	R	+	+
14. Lactose	+	+	+	Bl	Am	•	+	+	+	•	•	G	2	3	•	6	6	6	+	7	2	•	R	+	+
15. Raffinose	+	•	+	Bl Bl	Br	Bl	+	+	+	+	2	Br	5	6	•	5	7	7	+	8	2	Am Bk	Am Bk	•	+
16. Sucrose	+	•	•	Bl	Br	Bk	+	+	+	+	•	Br	8	8	•	7	8	8	•	8	2	Br Am	Br Am Bk Bk	+	•
17. Inulin	+	•	•	Bl	Br	Bk	+	+	+	+	•	G	9	7	•	6	9	5	•	5	2	Am r	r	+	•
18. Glucose	+	+	+	Bl	Y	•	+	+	+	+	•	G	2	8	•	2	3	8	•	2	6	•	•	•	•
19. Gum arabic	+	•	+	Am Bl Bk	•	•	•	+	+	+	se	G	50 se 2	•	•	5	2	6	+	5	1	r	r	•	•
20. "Tragacanth"	+	•	•	wh Br Gk	•	•	•	+	+	+	•	G Tr-Tc se se	1	•	4	•	4	4	•	6	1	•	•	•	•
21. Agar agar	+	•	•	Am Bl Bk	Br Br Bk	•	•	•	+	•	•	G	4	4	•	4	5	8	+	5	•	Br VR R	Br VR	•	+

Abbreviations: Am= Amber: Bk,black; Bl,blue; Br,brown; Ch,cherry; G, green; Or, orange;
P, purple; R, red; r, rose; V,voilet; Wn, white; Y, yellow.

CHAPTER II

QUANTITATIVE ANALYSIS.

The methods used for the quantitative determination of sugar fall in two different groups:

    1. Physical methods.

    2. Chemical methods.

Physical methods for the Quantitative Determination of Sugars:

There are many methods in use which depend on the physical properties of sugars and sugar solutions., such as specific-gravity, viscosity, optical activity, etc.

Of these, the methods based on the optical activity of sugar solutions are by far the most important, in accuracy and in the scope of application.

All sugars are optically active due to the presence of un-symmetrical carbon atoms in the molecule, and the degree to which the plane polorized light is rotated in a sugar solution depends, other factors remaining the same, on the kind of sugar and the concentration of the solution.

The polorizing power of sugars is expressed as specific-rotation, or specific rotary power, by which is ment the calculated angular rotation which a solution, containing the mass of 1 gr. active substance in a volume of 1 cc. and is polorized in a tube 1 dem. long, gives to the plane of polorized light. The specific - rotation (α), can easily be calculated from the angular rotation " a " of the substance by means of the equation:

$$(\alpha) = \frac{100\ a}{c \times 1}$$

In which c is the concentration of the substance (grammes mass in 100 cc. of solution) and 1 the length of the observation tube in decm.

As the angular rotation depends on the temperature and on the wave-length of the light used, so the standerd is set at 20° C and the light of sodium (the line D in the spectrum) so the specific rotation is written as:

$$[\alpha]_D^{20} = \frac{100\ a}{c\ x\ l}$$

The following table '1' gives the specific rotations of some of the common sugars:

Sugar	$[\alpha]_D^{20}$
Arabinose	+ 104.5
Xylose	+ 19.0
Rhmmose	+ 8.5
Galactose	+ 80.5
Glucose	+ 52.5
Fructose	- 92.5
Invert Sugar	- 20.0
Sucrose	+ 66.5
Lactose	+ 52.5
Maltose	+ 138.5
Raffinose	+ 104.4

The above formula for the specific rotation cam be modified in the following way:

$$[\alpha]_D^{20} = \frac{100a}{p\ x\ d\ x\ l}$$

Where p is parts by weight of the substance in 100 parts by weight of solution and d is the density of the solution.

Salving the about equation for p we get

$$p = \frac{100\ a}{\alpha\ x\ d\ x\ l}$$

which gives us a simple way to calculate the percentage of a sugar in its solution (in the absense of other optically active substances) from knowing the angular rotation of the given solution,

the specific rotation of the sugar, the density of the solution
and the length of the observation tube.

The following formulas '1) are given for calculating the
concentration (grms. per 100 cc.) of different sugars from
the angular rotation (α) in a 2 - dem. tube

1.	Arabinose	c	= 0.4785 a	
2.	Xylose	c	= 2.6316 a	
3.	Glucose	c	= 0.9470 a	
4.	Fructose	c	= 0.5405 a	(left degrees)
5.	Galactose	c	= 0.6173 a	
6.	Sucrose	c	= 0.7519 a	
7.	Maltose	c	= 0.3623 a	
8.	Lactose	c	= 0.9524 a	
9.	Raffinose	c	= 0.4785 a	

The different kinds of polarimeters are adopted to the
examination if all optically active substances; in sugar chemistry
however, a modified form of a polarimeter, called a Saccharimeter,
is used. For convinience the scale of the saccharimeter instead
of expressing angular rotation is replaced by one graduated
according to percentage.

Owing to the many difficulties and inconviniences connected
with the use of sodium or other monochromatic light in practical
work, the French physiset Soliel was led in 1845 to device a
means by which ordinary light or lamplight canld be used for
measuring the optical rotation for sugar solutions. This invention
known as the " Quartz-Wedge Compensator", is the characteristic
feature of most saccharimeters.

In the quartz-wedge saccharimeter, the polorizer and the
analyzer are both stationary; the rotation of the sugar solution
is measured by shifting a wedge of optically active quartz between

81' Sugar analysis. Browne and Zerhan. P. 265
'1' Sugar. anal. B. and Z. P. 295.

the solution and analyser until the rotation of the wedge system
at a certain thickness exactly neutralizes or compensates the
rotation of the sugar solution. By means of a scale attached to
the quartz wedge the rotation of the sugar in solution is measured
in percentage.

The selection of quartz for compensation is based upon
the fact that it has almost exactly the same rotation dispersion
as cane sugar; i.e., a section of quartz and cane sugar solution
of equal rotation for light of one wavelength will have very
nearly equal rotation for light of all other wave lengths. The
small disturbances due to the slight difference in rotation
dispersion between cane sugar and quartz are elliminated by a
dichromate filter

The Sugar Scale and Normal Weight of Saccharimeter:

The 100 point of a saccharimeter scale is usually based
upon the rotation of a difinite weight (the so-called normal
weight) of chemically pure sucrose dissolved in water to 100 mm.
at a specific temperature and polorized at the same temperature
in a 200 - mm. tube.

The greatest confusion has prevaled in saccharimetey in
the past and unfortunately still prevails, not only to the size
of the normal weight of sugar to be taken for a specified scale,
but also as to the conditions of volume and temperature under
which its normal weight is to be polorized.

French Sugar Scale:

The 100 point of the sugar scale employed upon sacchrimeters
of French manufacture was originally based upon the rotation in

sodium light of a plate of dextrarotary quartz 1 mm. thick cut exactly perpendicular to the optical axis.

Owing either to mistakes of polorimetric measurements or to defects in the quartz (through natural imperfection or mistake in cutting), the rotation of the 1 mm. plate has been given defferent values from time to time, the result ranging from + 20.98 to 22.67 and the normal weight of sucrose for such a saccharimeter from 16.000 grm. to 16.471 grm.

The value accepted now is 16.29 grm.

Ventzke or German Sugar Scale

The sugar scale most gernerally used outside of France and the one imployed upon all German saccharimeters is that of Ventzke. This scale as originally devised by Ventzke, was based upon the rotation of a solution containing 25 % of sucrose by weight but this was later changed to that of a solution of 1.1 sp.gr. 17.5°C nead in a tube 234 mm. long. It was found later that it is incon-vienient to base the normal weight upon the sp. gr. of a solution and the amount of sugat contained in this solution was adopted as the normal weight. This weight was determined to be 26.048 gm. weighed in air with brass scales and dissolved at 17.5°C to 100 cc. . The length of the tube was reduced to 200 mm.

With the introduction of the Mohr cubic centimeter (the volume of 1 grm. of water at 17.5°C weighed in the air with brass weights), the normal weight was recalculated to be 25.9872 grm.

The standard based on the Mohr cubic centimeter was abondened later and the temperatures changed to 20°C and this necessiated the reculculation of the normal weight and the new volue was found to be 26.0082 gms. which was changed later to 26.0000 gr,

and was called the Bates-Jacobson scale.

The adopted normal weight for the Ventzke scale now is 26.026 grm.

The Bidecimal Sugar Scale.

In 1896, it was proposed to adopt a so-called international sugar scale, based upon the normal weight of 20 grm. Among the advantages proposed for such a scale are:

1. It is a compromise between the French and the German scales.

2. Calculations are simplified by use of decimal weights.

3. Aliquotes of 50, 25, 20, 10, and 5 cc. of the 100 cc. solution contain even gram quantities.

4. The normal weight, its fraction, and its multiples are always available in one-piece unit.

The saccharimeter used in this university has such a scale and its normal weight is difined as " 20 gm. of pure sucrose dissolved in 100 ml. of solution and polorized in a 200 mm. tube at 20° C."

The following table '1' gives the normal weight to Ventzke scale for various sugars:

Sugar	Normal wet.
Sucrose	26.026 gr.
Glucose	32.398 gr.
Fructose	18.416 gr.
Invert Sugar	86.333 gr.
Lactose (+ H_2O)	32.972 gr.
Maltose	12.528 gr.
Raffinose (+$5H_2O$)	16.574 gr.
Raffinose (Anhyd.)	14.062 gr.

For the Bates-Jacobson scale with the normal weight of suc-rose as 26.000 gr. the above volues are multiplied by 0.999. and for the Bidecimal scale by 0.763

'l' Sugar Anal. B.and Z. p 298.

CHEMICAL METHODS FOR THE QUANTITATIVE DETERMINATION OF SUGARS

The principal chemical methods for determining sugars are based upon the property which all aldehydes and ketones gave of reducing alkaline solutions of certain metallic salts.

Of the verious alkaline solutions of metals, those of copper are employed most generally in sugar analysis.

Fehling in 1848 first worked out the details of the alkaline copper methods, as they now stand, and the copper sulfate and alkaline tartarate reagent had simce been called by his name.

All the different methods using alkaline solutions of metallic salt are based on the same principle i.e. the oxidation of the sugar by the metallic ion which is reduced in its turn to the free metal of to the lower insoluble oxide; but they differ in the way the quantity of sugar used is calculated. Some are based on the weight of the cuprous oxide (copper solutions) formed which bears a relation to the quantity of reducing sugars used and can be found from given tables, and others finde the quantity of unreduced copper from which, and the original quantity of copper, the part reduced is found whmth gives the quantity of sugar used.

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For the Bates-Jacobson scale with the normal weight of sucrose as 26.000 gr. the above values are multiplied by 0.999. and for the Bidecimal scale by 0.763

'1' Sugar Anal. B.and Z. p 298.

CHEMICAL METHODS FOR THE QUANTITATIVE DETERMINATION OF SUGARS

The principal chemical methods for determining sugars are based upon the property which all aldehydes and ketones have of reducing alkaline solutions of certain metallic salts.

Of the various alkaline solutions of metals, those of copper are employed most generally in sugar analysis.

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was the case with my work, so naturally I chosed a method based on the second way. The simplest way to determine unreduced copper is volumetrically although it can be found by electrolysis or gravemetrically. Again I wanted a method which is quick with a short heating period and at a temperature easily obtainable with the equipment I had; and in my work I had to analyze mixtures of sugars which almost all of them were composed of sucrose, glucose, and fructose so a solution which had equal reducing power towards glucose and fructose and which would permit the quantitative determination of fructose in the presence of glucose was necessary.

These properties were found in the modified Luff-Schoorl method.

Modified Luff-Schoorl Method [1]

The copper reagent is prepared as follows:

1. 388 g. of crystalline sodium carbonate ($Na_2CO_3.10H_2O$) or its equivalent of the anhydrous salt are dissolved in 300 - 400 cc. of luke worm water in 1 lit. volumetric flask and to this is added with stirring a solution of

2. 50 g. Citric acid in 50 cc. of water, after all effervescence has ceased add gradually with constant vigorous shaking a solution of:

3. 25 g. crystalline copper sulfate ($CuSO_4 .5H_2O$) dissolved in 100 cc of cold water . Cool, Make up to volume and let stand for few days; decant the clear portion and store; the solution will keep indefinitely and shows no auto-reduction even upon boiling.

Place 25 cc of this reagent and 25 cc. of the sugar solution in

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(1) Sug. Anal. B. and Z. P. 832

a 300 cc. erlenmeyer flask, add few pieces of pumice stone and attach a water-coold reflux condenser. Heat over a free flame so that the contents will start to boil in about 2 min. then heat over an asbestos centered wire gauze and let it boil gently for exactly 10 mins. longer. Remove the flame and cool immediatly in running water. Add 5 gm. of potassium iodide and then add slawly with stirring 20 cc. of 25 % hydrochloric acid. Titrate the liberated iodine with standard sodium thiosulfate solution using starch indicator towerds the end. Rumn a blank end with 25 cc. of the reagent and 25 cc. of distilled water following the same procedure exactly. The difference between the two titrations is equivelent to the reduced copper and to the quantity of reducing sugars present which is found from the following table:

0.1N Thiosulfate	Glucose Fructose Invert sugar	Ahydrous Lactose	Anydrous Maltose
Vol. cc.	Mg.	Mg.	Mg.
1	2.4	3.6	3.9
2	4.8	7.3	7.8
3	7.2	11.0	11.7
4	9.7	14.7	15.6
5	12.2	18.4	19.6
6	14.7	22.1	23.5
7	17.2	25.8	27.5
8	19.4	29.5	31.5
9	22.4	33.2	35.5
10	25.0	37.0	39.5
11	27.6	40.8	43.5
12	30.3	44.6	47.5
13	33.0	48.4	51.6
14.	35.7	52.2	55.7
15	38.5	56.0	59.8
16	41.3	59.9	63.9
17	44.2	63.8	68.0
18	47.1	67.7	72.2
19	50.0	71.7	75.5
20	53.0	75.7	80.9
21	56.0	79.8	85.4
22	59.1	83.9	90.0
23	62.2	88.0	94.6

The same table may also be employed if larger or smaller quantities of the copper reagent than the 25 cc. spcefied are used. As for example, 50 cc. reagent plus 50 cc. sugar solution. The titer found is devided by 2, the corresponding milligrams of sugar found from the table, and the result multiplied by 2. Thus a titer of 20 under there conditions indicate 25.0 x 2 or 50 mg. of glucose and not 53.0 mg. The same thing is applied is smaller volume is used, namely find the milligrams of sugar which corespond to 25 cc. of reagent and then convert to the volume used.

Two points should be boven in mind, the final volume of reagent plus sugar solution must always be twice the volume of the reagent, use distilled water to accomplish this point, and second, after boiling, the solution sheild show a blue color indicating the presence of unreduced copper, if no blue color is present, repeat the determination using smaller volume of sugar and make up for the difference with distilled water.

Clarifying Agents

A clarifying agent is a substance added to the solution of the sugar or the sugar containigg material which causes the coagulation and the precepitation of the gummy, albuminoids and waxy materials present, which results in a solution clear and easy to filter.

The most used clarifying agents are:

1. Lead subacetate. It is prepared by mixing 300 gr. lead acetate and 100 gm. lead oxide (litharage) in one liter of water stirred and heated gently for about 2 hours. It is left to settle and the clear solution decanted.

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The most used clarifying agents are:

1. Lead subacetate. It is prepared by mixing 300 gr. lead acetate and 100 gm. lead oxide (litharage) in one liter of water stirred and heated gently for about 2 hours. It is left to settle and the clear solution decanted.

2. **Neutral lead acetate;** It is a saturated solution of lead acetate in water.

3. **Dry basic lead acetate:** (Horne Powder) It is obtained as a dry powdered salt and contains 72.8% Pb, which corresponds to the composition of $3Pb(C_2H_3O_2)_2 \cdot 2PbO$.

4. **Alumina cream:** Prepare a cold saturated solution of potassium aluminium alum in water. Add conc NH_4OH with constant stirring until the solution is alkaline to litmus; let the precipitate settle and wash by decantation with water until the wash water gives only a slight test for sulfate ions with $BaCl_2$ solution. Pour off the exass of water and store the residual cream in a stoppered bottle.

Clarifying agents are added before completing to volume except the dry Horne powder which is added after competing the volume. As the basic lead acetate precepitates the fructose as lead fructosate, therefore in the presence of reducing sugars to be determined the use of neutral lead acetate or alumina cream for clarifying is imperative.

Hints on Clarification and Filteration

After the addetion of the clarifying agents (about 3 or 4 cc. per 100 cc. of the solution) add one drop of ether to clear the foam before completing to the mark. Shake thoroughly and let stand for about 10 mins. Pour into a fluted filter paper capable of holding at least half the quantity at one time, collect the clear filterate after rejecting at least the first 25 cc., as the addsorption power of filter paper to water and sugars is not proportional to the composition of the solution.

If lead solutions has been used for clarification, the excess

lead chould be removed by addition of dry, powdered anhydrous
sodium carbonate to the filterate and refiltering ; as the excess
of lead will cause a change in the rotation of the sugar solution
and will react with the copper sulfate to form the insoluble
lead sulfate and with the hydrochloric acid used in inversion
forming the precipitate lead chloride.

THE QUANTITATIVE DETERMINATION OF ~~GLUCOSE~~ SUCROSE

1. In the Absence of other Sugars:

Weight out one normal ~~wight~~ weight exactly, transfare it to a 100 cc. volumetric flask, add about 70 cc. water, stirr to dissolve, add the proper clarifying agent, complete to the mark and polorize in a 200 mm. tube, Reading is percentage sucrose.

2. In the Presence of Mono-Saccharides:

Prepare a normal solution as directed above and find direct polorization reading. Invert part of the solution as fallows:

Transferre 50 cc. of the normal solution to a 100 cc. vol. flask, add 25 cc. water and 5 cc. conc. HCl, while shaking, heat in a water bath at such a rate that the internal temperature reaches 67° - 69° C in 2 - $2\frac{1}{2}$ mins. Keep at that temperature for 7 mins. longer. Cool, rinse in the thermometer, make up to volume and polorize in a 200 mm. tube. This gives the " Invert reading" , Calculate percentage sucrose in the original sample from the following formula.

$$S = \frac{100\ D}{142.66 - \frac{20}{t}}$$

where S = percentage sucrose

D = Direct polorization - 2 invert polorization

t = Temperature of polorization in degrees centigrade.

It is very important that the two polorizations should be made at the same temperature although not necessarly 20° C, but it is preferrable to be near 20° C.

This method depends on the change in poloriscope reading due to the inversion of the sucrose; as the mono-saccharides sulffer no change at the used concentration of acid and at the used temperature, then any change in the rotation is due to the invert sugar produced

In calculating D in the above formula, twice the invert reading was taken because the invert solution is $\frac{1}{2}$ normal. If the invert reading is negative then it is added to the direct reading, of caurse.

THE QUANTITATIVE DETERMINATION OF GLUCOSE

1. In The Absence Of Other Sugars

When only glucose is known to be present, then it can be determined either by the use of a saccharimeter or chemically.

a. For the determination by the saccharimeter, follow the procedure given under the determination of sucrose (Method-1) using the normal weight of glucose as given in the beginning of this chapter

b. For determining glucose chemically, prepare a solution containing about 0.5 - 1 percent of solid matter and follow the Modified Luff-Schoorl method, after proper clarification.

2. In The Presence Of Non-reducing Sugars

Follow the procedure given above (method 1, b.)

3. The Determination of Commercial Glucose in Other Sugar Products

Method of A.O.A.C. '1'

Prepare an inverted half-normal solution of the substance, do not cool after inversion, make neutral to phenol-phthaline

'1' Methods of Analysis, A.O.A.C. 5th ed; p 498, 1940.

with sodium hydroxide solution, slightly acidify with dilute
HCl (1+5) and treat with 5 to 10 cc. of alumina cream before
making to the mark. Filter and polarize at 87°C. in a 200 mm.
jacketed metal tube, preferably silver. Calculate the quantity
of commercial glucose solid polorizing $+211°$ V by the formula

$$S = \frac{200\ R}{196}$$

where S, =is the quantity of glucose as discribed above,
and D the saccharimetric reading.

The normal weight here referres to that of sucrose and
not to glucose

The cause for using a high temperature here is because
of the fact that invert sugar is optically inactive at 87°C.
because the rotation of fructose decreases rapidly with rise
of temperature so that at 87°C the levo rotation due to fructose
is compensated by the dextro- rotation due to the equivalent
amount of glucose. So this method of high temperature polori-
zation eliminates the effect of any present sucrose or invert
sugar or originally present.

This method is used to detect the adulteration of honey
and other similar products with commercial glucose.

THE QUANTITATIVE DETERMINATION OF FRUCTOSE

1.   If it is the only reducing sugar present, then the pro-
cedure is that described for the determination of reducing
sugars or as described for glucose. (1, b.)

2.   In The Presence Of Other Sugar:

The selective determination of fructose in a mixture of

sugars is carried either chemically or physically. Thus, *chemical* method depends on the property of fructose of suffering a big change in the rotation with change in temperature.

Prepare a normal solution, clarify, filter, and find the direct polerization at 20° C in a 200 mm. tube and polerize again at 87° C in a 200 mm. tube also. Calculate the weight of fructose in the normal weight by the formula:

$$F = \frac{R_{20} - 1.0315\ R_{87}}{2.3919}$$

where F is the weight of fructose in the normal weight

R$_{20}$ is the Direct polerization at 20° C.

R$_{87}$ is the direct polerization at 87° C.

b.- Chemically

This method depends on the fact that, fructose being a ketose (the only common ketose in ordinary sugar analysis) is more diffecult to oxidize than other common sugars which are aldoses. The oxidizing agent is sodium hypoiodite.

Kolthoff - Kruishesr Method For Determining Fructose '1'

Reagents:

1. 4 N. Sodium hydroxide solution

2. 4 N. Sulfuric acid solution

3. Iodine solution containing 13 grm. iodine and 15 grm. potassium iodide in 100 cc.

4. 20 percent sodium sulfite solution

5. 2 percent sodium sulfite solution

'1' Sug. Anal. B. and Z. p. 902.

Procedure:

Dissolve a suitable quantity of the product representing from 1.75 to 3.5 grms. total solids, in water in a 100 cc. vol. flask, calrify, complete to volume and filter. Pipette 25 cc. of this solution into another 100 cc. vol. flask, add 25 cc. water and then 5 cc. of 4 N. sodium hydroxide. Run in at once 16 cc. of the iodine solution or enough to impart a distinctly brown color to the solution. Let stand for 5 to 7 mins. and add 3 cc. of 4 N. sulfuric acid. Remove the excess iodine first with 20 percent sodium sulfite solution, and then carefully with 2 percent sodium sulfite solution. Add starch indecator twards the end and continue the titration till the blue color is discharged. Add 4 N. sodium hydroxide till the solution is slightly acid to methyl orange. Complete the volume and determine the fructose in a 25 cc. aliquote by the modified Luff -Schoorl method. Calculate the percentage of fructose in the original sample.

In the presence of sucrose it is advisable to first invert the solution, neutralize, and determine the fructose after inversion. The fructose produced by the inversion of sucrose is 52.63 percent of sucrose, as found by double polorization.

ANALYSIS OF MIXTURES OF SUGARS

The most important mixtures of sugars encountered in everyday analysis are those composed of sucrose, glucose, and fructose.

1. Sucrose and any one of the other two sugars present a

very simple mixture the sucrose being determined by double polorization and the other reducing sugar by the copper-solution.

2. A mixture of glucose and fructose can be analysed by finding the total reducing sugar in the original sample and after the oxidation of glucose as discribed in the method for determining fructose selectivly. The difference between the two values represent the glucose, From this point, it is easy to understand why the copper reagent employed must have the same oxidizing power towords glucose, and fructose.

3. A mixture of the three sugars togeather can be analyzed by combining the above menthioned two procedures, i.e. Sucrose, by double polorization, fructose by the hypo-iodite method, and glucose by difference.

MOISTURE IN SUGAR CONTAINING PRODUCTS

The water content in a sugar containing product can be determined either by drying a sample at 100° C in an oven under reduced pressure or by extracting the water with a volatile immicible solvent. Generally Xylol is used but as there is the danger of fructose decomposing at the boiling point of xylol 138° C, toluene, with the lower boiling point of 110.6° C is used instead, otherwise the procedure is exactly as described in books on commercial quantitative analysis.

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