PRACTICAL . . .
WHITE SUGAR
MANUFACTURE

H. C. PRINSEN GEERLIGS, Ph. D.

In alkaline juices this inversion is not to be feared; but then, especially at high temperatures, the reducing sugars are rapidly decomposed with the formation of dark-coloured organic acids, which combine with the alkali and therefore produce a neutral solution after the alkali has exerted its action. Therefore, the stronger the alkaline reaction, the more reducing sugar is decomposed. At low temperatures, *i.e.*, below 55° C, the products of decomposition are colourless, but at higher temperatures, the products of the reaction possess a brown colour and have the disadvantageous property of being broken up gradually into acid secondary decomposition products which may afterwards give rise to inversion; while gaseous products may be formed as final products, which are apt to cause the so-called "frothy fermentation" of the after-products.

Any prolonged treatment of juices, syrups, massecuites, molasses, etc., at a high temperature should be avoided as much as possible, and the temperature of such products should never exceed 70° C. Acid juices and syrups should be closely watched when maintained at a high temperature, while heating strongly alkaline cane juices above 55° C should at any rate be avoided.

Coloration of Juices by Iron Compounds.—Finally, juices and sugars may assume a very unpleasant dark coloration by the formation of soluble iron compounds. During its treatment in the factory the sugar juice comes constantly into touch with iron surfaces such as those of defecating pans, eliminators, subsiding tanks, filter-presses, juice-heaters, evaporators, etc., which may lead to an absorption of iron salts, all the more so in acid as compared with neutral reactions. It is true that much may be done to guard against that contingency by having part of the plant made of copper or by protecting the inside of tanks or pipes with a coating of paint or varnish, but such precautions do not entirely prevent the dissolving of traces of iron oxide from the vessels in which the juice is treated or through which it runs.

This formation of iron compounds exerts, however, a detrimental influence on the colour of the sugar crystals only if the iron is dissolved in the highest state of oxidation, *i.e.*, in the ferric form (Fe_2O_3), and when the reaction of the juice is neutral. If the iron is present in the lowest state of oxidation, the ferrous condition (FeO), or if the juice is acid, no co-crystallization of ferric saccharate with the sugar crystals is to be feared and the iron remains in the molasses. In this case the sugar crystal is not given a dark colour by iron salts, so that all coloration by iron can be avoided by treating the juice with sulphurous acid, which first of all reduces the iron to the ferrous stage of oxidation, and then imparts an acid reaction to the juice.

(B) Clearness of the Juice.

A second very important factor in the manufacture of white sugar is the complete removal of the floating impurities, both those arising from the hard particles of the cane passing into the juice, and those precipitated from the juice by the clarification process and during evaporation. It is necessary not only to precipitate impurities during clarification but also to remove them from the juice, while the silica, calcium phosphate and calcium oxalate, which become insoluble during concentration, and are apt to make the syrups cloudy or even opaque, must be got rid of before the sugar starts crystallizing. The fine particles of insoluble matter left floating in the syrup will form the nuclei of sugar crystals, or will be incorporated between the lamellae of the growing crystal which thereby assumes a greyish hue, not to be removed by washing, the commercial value of the finished product thus being impaired. Apart from the necessity of having the juice as lightly coloured by dissolved substances as we can possibly make it, we have also to take into consideration the necessity of simultaneously making the juice and syrups as clear as we can, and every endeavour should be made to combine these two requirements in the many clarification processes proposed for the preparation of white sugar from cane juice.

(C) Decomposition of Sucrose by Acids and Ferments.

As already stated, the sucrose in juices, syrups, massecuites and molasses is very liable to become transformed into invert sugar by the action of acids, especially at high temperatures.

The crystallizable sucrose is thereby transformed into a mixture of glucose and fructose, which, although sugars, do not crystallize, thus constituting a total loss for the practical manufacturer; hence any such inversion is to be avoided as resolutely as possible.

From A.M. Duperu, 1874-1944.
Ref. Mgr. C.& H. Sug. Ref. Corp.,
Crockett, California. 1915-1932
See "Chemical Engineering", Apr. 1945. P181.

July. 1915

PRACTICAL . .
WHITE SUGAR
MANUFACTURE

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MANUFACTURE

Or the Manufacture of Plantation White Sugar directly from the Sugar Cane.

BY

H. C. PRINSEN GEERLIGS, Ph. D.

Author of "Cane Sugar and its Manufacture," "The World's Cane Sugar Industry—Past and Present," "Methods of Chemical Control in Cane Sugar Factories."

LONDON:
NORMAN RODGER,
St. Dunstan's Hill, E.C.

1915

PREFACE.

During the last ten years the question of manufacturing so-called "Plantation White Sugars" has come very much to the front and each year more of this grade of sugar is finding its way into the markets of Asia, and even of Europe and America.

Numerous are the processes used, and their modifications suggested in the various cane sugar producing countries, and it occurred to the author that the moment had come in which an attempt might be made to review them and to express an opinion of their merits.

In the completion of this work the author has been greatly assisted by the courtesy of the many inventors and patentees of the new methods, who have not hesitated to put at his disposal an authentic description of the processes and samples of the chemicals used, thereby ensuring the use of first-hand information.

The author is in this respect much indebted to the courtesy of the following, to whom he expresses his gratitude: Messrs. the Anilin und Sodafabrik, Ludwigshafen; N. B. Bach; E. E. Battelle; Wladimir Guerrero; M. Weinrich; and Dr. A. Wijnberg.

Further, the author has to express his thanks to the following firms, who have loaned him blocks for illustrations: The Harvey Engineering Co., Ltd., Glasgow; The Hersey Manufacturing Co., Boston; Mr. Norman Rodger, London; Messrs. Stork & Co., Hengelo, Holland; and Messrs. Watson, Laidlaw & Co., Ltd., Glasgow.

Finally, the author has to express his thanks to Mr. James P. Ogilvie, Technical Editor of the *International Sugar Journal*, for his share in checking the manuscript of this work, as regards the technical and chemical nomenclature most generally in use in English and American sugar literature.

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As in the author's book, "Cane Sugar and its Manufacture," Fischer's nomenclature for the different sugars has been followed in this work, the terms used for them having the following meanings:

SUGAR: The commercial product.

SUCROSE: The chemical body, the principal constituent of the commercial product.

GLUCOSE: The chemical body, also called dextrose.

FRUCTOSE: The chemical body, also called levulose.

INVERT SUGAR: The mixture of exactly equal proportions of glucose and fructose.

REDUCING SUGARS: The mixture of equal proportions of glucose and fructose.

The analytical methods in Part III. are those employed at the Java Sugar Experimental Station.

H. C. PRINSEN GEERLIGS.

AMSTERDAM,
March, 1915.

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PRACTICAL
WHITE SUGAR MANUFACTURE.

INTRODUCTION.

HISTORICAL.

Far from being a modern invention dating from the present generation, the manufacture of white sugar directly from cane juice without the intermediary of a refining process is one of the oldest achievements of the sugar industry.

Very probably the oldest form in which cane sugar was manufactured, in those early periods of our civilization in which the use of earthenware or metal boiling vessels was first practized, was that of cane juice evaporated to a dry mass, such as is nowadays still produced in every cane-growing country to a total amount of hundreds of thousands of tons per annum.

It is likewise probable that very soon after the art of sugar making by evaporation of the cane juice had developed, it was discovered that if juices were not concentrated to the utmost degree of dryness, the mass after being cooled down changed into a solid mass of crystals, from which the adhering brown molasses could be drained off rather easily, leaving behind a whiter and better product than the original one.

The draining off of the molasses and the production of a dry and white sugar could be promoted by giving a conical shape to the receptacle into which the hot, concentrated sugar liquor was run to cool down, while the last portions of the molasses could be induced to separate from the sugar crystals by allowing a slow and regular stream of water to percolate the sugar, thereby removing the impure coloured molasses and leaving behind the white network of concrete sugar crystals.

In every part of the world in which cane sugar is produced, we know of methods for making white sugar by covering the surface of coherent masses of brown sugar with the moist leaves of water-plants or with a mixture of clay and water,[1] and it cannot be wondered at that such a simple and effective method of turning brown sugar into a white product was already known in the infancy of this art, so that the production of white sugar directly from cane juice must have preceded that of the art of refining properly speaking.

The earliest forms of white sugar recorded in history, as to which we have knowledge, are those of candy and loaves, of which the latter was the most common. At the same time we find mention made of powdered sugar, derived in every instance from crushed loaves, which had been damaged or broken and could not be shipped as ordinary good quality loaf sugar. Whereas sugar candy was manufactured at the outset from raw or from white sugar which had been dissolved and recrystallized, and may therefore be reckoned to belong to the class of refined sugars, the loaves of antiquity and of the early middle ages were the product of raw sugar factories, having been obtained from juice and washed white without being first redissolved.

In perusing the classical work of von Lippmann, "Die Geschichte des Zuckers," we find numerous instances between the years 1250 and 1400, of sugar exported in loaves from Cyprus, Rhodes, Syria and Alexandria. In many cases these loaves were made from sugar only boiled once, or from washed raw sugar; but in other instances we find that such sugars had been boiled twice and even several times, and therefore represented really refined sugar.

The sugar in loaves from Brazil and the West Indian islands, which was shipped to Europe after the establishment of the sugar industry out there by the European settlers, consisted, however, exclusively of raw sugar, from which the molasses had been drained off through an opening at the bottom of the conical mould in which the concentrated syrup had been allowed to crystallize.

After crystallization had taken place, the surface of the mass in the moulds was levelled, a piece of paper was laid flat on the smooth surface, and then a mixture of clay and water was poured on the top. The water trickled slowly through the contents of the mould, washed off the adhering molasses and thereby improved the quality of the sugar. Finally the loaves were taken out of the moulds, divided into three parts: viz., the

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[1] For particulars as to these primitive methods of white sugar manufacture, see H. C. Prinsen Geerligs' "The World's Cane Sugar Industry" (1912), pages 50, 74, 101, 235.

uppermost whitest part, "blanco"; the middle part, in which some molasses had been left, "muscovado"; and the undermost or brownest part, "panela." The pieces of sugar of each of the three portions were crushed and packed; the white sugar could be used for consumption without being refined, while the others required further treatment in a refinery before being delivered to consumers.

The refining industry in Europe soon attained a high degree of perfection, the great care bestowed on the clarification of the sugar clairces (liquors), and on the boiling of the sugar and the finishing of the loaves and after-products, accustomed sugar consumers to the use of refined sugar to such an extent that the white portions of the raw sugar loaves disappeared from the market as far as direct consumption was concerned, and were only used as raw material for the refineries. The refining industry continued steadily to improve its methods, the quality of the refined sugar was flawless, even in the seventeenth century, and has remained so ever since, while the manufacture of the raw cane sugar remained in its original primitive state down to the last quarter of the nineteenth century.

By that time the cane sugar manufacturers in some of the countries of production felt the necessity of availing themselves of the beneficent results which scientific research had accomplished in the beet sugar industry. They saw how the application of science in manufacture had wrought wonders in building up a splendid industry, and so they wanted to follow that example. Gradually the cane sugar industry remodelled its methods and studied the circumstances under which it had to work; and the result is an organization no whit less brilliant and well-organized than that of the sister industry itself.

When it was found possible in raw beet sugar factories to manufacture without the use of animal char, and by mechanical contrivance alone, a white, odourless, crystallized product which was very little inferior to a first-class refined article, and which is at this moment used in huge quantities for direct consumption, the cane sugar industry could not refrain from trying to do likewise.

Instead of making only refining crystals as a raw material for sugar refineries, manufacturers of raw cane sugar have gone to work to make in their sugar houses a white sugar for direct consumption, without remelting or using charcoal, and have succeeded in producing a beautiful quality of white cane sugar, without being involved in any extraordinary expense or excessive loss of sugar during manufacture.

In this book it is proposed to discuss the different methods of attaining that end, and the several advantages or disadvantages of the various processes and their modifications will find complete consideration.

SOME PRELIMINARY CONSIDERATIONS.

The manufacture of white sugar from sugar cane juice is often said to be a relatively easy operation, since it is not very difficult to remove the colour of the juice, and, since further, the juice of the sugar cane does not possess an unpleasant smell, as is the case with that of the sugar beet. It is therefore believed to be unnecessary to remove the last traces of colouring and aromatic substances from the juice, and it is even claimed that minute amounts of the latter in cane sugar add to its merits and increase the sweetening power as compared with that of a really refined article.

This is not the place to enter into a discussion on the relative merits of white beet or cane sugar, or refined sugar of either origin, and this the more so because we do not agree with the opinion just stated, viz., that impurities left behind in the white cane sugar play a favourable rôle in the finished product. On the contrary we must make our product as white and as pure as possible, converting it to a state in which no difference at all can possibly be detected between the dietetic and other merits of raw or refined cane or beet sugar.

It is not only our aim to make a good and dry white sugar, but also to have it in such a condition that it will remain white and dry after storage; and therefore it is necessary to remove the last traces of substances other than sucrose as far as possible. A small percentage of glucose left in the sugar may cause it to attract moisture from the atmosphere, while some colouring matter derived from the rind of the cane, or formed during manufacture and temporarily bleached may reassume its original colour and give the article a greyish hue after it has been stored for some time, thereby in either case decreasing its value.

Instead of being rather an easy task, only requiring a little additional machinery over that used in a common raw sugar factory, the manufacture of Plantation White Sugar demands not only a good knowledge of general sugar manufacture but also special skill on the part of manager, employees and workmen, and above all a large capacity in all departments of the sugar-house and a proper arrangement of the whole plant.

Our greatest enemies are inversion and coloration, and we have to suppress these two to the utmost of our power. Therefore no juice or syrup or massecuite should be allowed to stand any longer than can possibly be helped, and, in the second place, any probability of overheating should be avoided in every way.

No massing together of heterogeneous types of machinery should be allowed, but each item of the whole plant should be well calculated and be in relation to the rest, so that at no stage will the regular course of manufacture meet with an obstacle to prevent it from turning out the maximum yield of brilliantly white sugar from the cane juice in the shortest time possible.

The disposition of the plant and the ability of employees and workmen count for much more in the manufacture of white sugar than the choice of any one of the many processes in use. It may be possible to make a good product with either of them, provided the plant is well designed and well constructed, and the staff and workmen understand their work ; but even the best process will fail where the machinery is inadequate or the men incapable. The plant may be purchased and erected in any spot in the world where cane grows, but if it be desired to establish a white sugar factory in a place where the workmen are not trained to that task or are unwilling to become so, the best process and the best plant will not prevent the result from proving poor and disappointing.

The careful education and training of the workmen is indispensable for any good issue, and the fault of having overlooked this point has already repeatedly been the cause of failure where success was anticipated.

PART I.

CLARIFICATION OF THE CANE JUICE.

CHAPTER I.

PRINCIPLES.

The clarification of the cane juice is of supreme importance in the manufacture of plantation white sugar and this for two reasons; it is necessary to make the juice as devoid of colour and at the same time to render it as clear as can possibly be done.

(A) Colour of the Cane Juice.

The colour of the juice may have a twofold origin: It may be a consequence of colouring matter from the cane itself, passed over into the juice on extraction; it may also be the result of the action of lime or of heat or of both combined, on the constituents of the juice.[1]

Colouring Matter from the Cane.—The sugar cane contains in the rind cells a mixture of two colouring matters which are represented there in mutually widely varying proportions according to the colour of the cane.

One of these, the *chlorophyll*, is the regular constituent found in every green cell of plants. It is insoluble in water and in sugar solutions, and is in suspension in the cane juice, for which reason it passes into the scums, not interfering any further with the process of manufacture.

The second colouring substance present in the rind cells is *anthocyan*, a violet-coloured substance which is found in the outer cells of dark-coloured canes, but is almost absent in white or yellow-coloured varieties. In contrast with chlorophyll, anthocyan is soluble in water and in sugar solutions, which explains why the juice of canes having a dark-coloured rind is so much more coloured than that of green, white or yellow canes, and also why the last mill juice of coloured canes in which the juice from the hard rind is strongly represented is so much more coloured than the first mill juice in which chiefly juice from the soft parenchyma cells is contained.

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[1] For chemical properties and reactions of the constituents of sugar cane, see H. C. Prinsen Geerligs' "Cane Sugar and its Manufacture" (1909), Pages 3-54

The purple colour of the anthocyan solution is changed into a dark green by the addition of lime, but the body is not precipitated until a strongly alkaline reaction is obtained by addition of more lime.

Sulphurous acid is incapable of bleaching anthocyan solutions; it decolorizes to some extent, but after exposure of the treated solution to the oxygen of the air the original colour returns.

A third colouring matter, *saccharetin*,[1] is found impregnated in the fibre of the cane. Neither water nor sugar solutions extract this colouring matter from the fibre, but as soon as these media are rendered alkaline with lime or any other alkaline body, the hitherto colourless saccharetin becomes yellow and is extracted by the liquid, which extraction proceeds only slowly, as saccharetin and fibre appear to be very closely united. Saccharetin occurs in the raw juice attached to the fine particles of bagasse suspended in the juice and dissolves to some extent as soon as the juice is limed. A thorough separation of the "cush-cush" will therefore prevent much saccharetin entering into the clarified juice, where after all it is comparatively harmless, as it only assumes a yellow colour in alkaline media, and becomes colourless again as soon as the reaction is neutral or acid.

Coloration of the Juice by Decomposition of its Constituents.— When maintained at a high temperature, solutions of both sucrose and the two reducing sugars, glucose and fructose, assume a dark coloration. Of the three sugars under consideration, the fructose is decomposed most quickly, next comes glucose, and finally sucrose, but at any rate a prolonged spell at a high temperature is to be avoided, if one desires to keep the cane juice as lightly coloured as possible. The coloration of the solutions of the sugars after being heated is even perceptible in their pure solution in water; but it becomes much more apparent where neutral salts or nitrogenous bodies such as asparagin, aspartic acid, or ammonium salts are simultaneously present, especially in the case of the reducing sugars, an occurrence which is regularly the case in the sugar juices met with in practical working.

The coloration of solutions of sucrose and glucose or fructose having an acid reaction is not so strong as in neutral solution, but acid reaction is apt to cause the decomposition of sucrose to invert sugar at a rate increasing with the temperature of the juice.

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[1] International Sugar Journal, 1912, 53.

In alkaline juices this inversion is not to be feared; but then, especially at high temperatures, the reducing sugars are rapidly decomposed with the formation of dark-coloured organic acids, which combine with the alkali and therefore produce a neutral solution after the alkali has exerted its action. Therefore, the stronger the alkaline reaction, the more reducing sugar is decomposed. At low temperatures, *i.e.*, below 55° C, the products of decomposition are colourless, but at higher temperatures, the products of the reaction possess a brown colour and have the disadvantageous property of being broken up gradually into acid secondary decomposition products which may afterwards give rise to inversion; while gaseous products may be formed as final products, which are apt to cause the so-called "frothy fermentation" of the after-products.

Any prolonged treatment of juices, syrups, massecuites, molasses, etc., at a high temperature should be avoided as much as possible, and the temperature of such products should never exceed 70° C. Acid juices and syrups should be closely watched when maintained at a high temperature, while heating strongly alkaline cane juices above 55° C should at any rate be avoided.

Coloration of Juices by Iron Compounds.—Finally, juices and sugars may assume a very unpleasant dark coloration by the formation of soluble iron compounds. During its treatment in the factory the sugar juice comes constantly into touch with iron surfaces such as those of defecating pans, eliminators, subsiding tanks, filter-presses, juice-heaters, evaporators, etc., which may lead to an absorption of iron salts, all the more so in acid as compared with neutral reactions. It is true that much may be done to guard against that contingency by having part of the plant made of copper or by protecting the inside of tanks or pipes with a coating of paint or varnish, but such precautions do not entirely prevent the dissolving of traces of iron oxide from the vessels in which the juice is treated or through which it runs.

This formation of iron compounds exerts, however, a detrimental influence on the colour of the sugar crystals only if the iron is dissolved in the highest state of oxidation, *i.e.*, in the ferric form (Fe_2O_3), and when the reaction of the juice is neutral. If the iron is present in the lowest state of oxidation, the ferrous condition (FeO), or if the juice is acid, no co-crystallization of ferric saccharate with the sugar crystals is to be feared and the iron remains in the molasses. In this case the sugar crystal is not given a dark colour by iron salts, so that all coloration by iron can be avoided by treating the juice with sulphurous acid, which first of all reduces the iron to the ferrous stage of oxidation, and then imparts an acid reaction to the juice.

(B) Clearness of the Juice.

A second very important factor in the manufacture of white sugar is the complete removal of the floating impurities, both those arising from the hard particles of the cane passing into the juice, and those precipitated from the juice by the clarification process and during evaporation. It is necessary not only to precipitate impurities during clarification but also to remove them from the juice, while the silica, calcium phosphate and calcium oxalate, which become insoluble during concentration, and are apt to make the syrups cloudy or even opaque, must be got rid of before the sugar starts crystallizing. The fine particles of insoluble matter left floating in the syrup will form the nuclei of sugar crystals, or will be incorporated between the lamellae of the growing crystal which thereby assumes a greyish hue, not to be removed by washing, the commercial value of the finished product thus being impaired. Apart from the necessity of having the juice as lightly coloured by dissolved substances as we can possibly make it, we have also to take into consideration the necessity of simultaneously making the juice and syrups as clear as we can, and every endeavour should be made to combine these two requirements in the many clarification processes proposed for the preparation of white sugar from cane juice.

(C) Decomposition of Sucrose by Acids and Ferments.

As already stated, the sucrose in juices, syrups, massecuites and molasses is very liable to become transformed into invert sugar by the action of acids, especially at high temperatures.

The crystallizable sucrose is thereby transformed into a mixture of glucose and fructose, which, although sugars, do not crystallize, thus constituting a total loss for the practical manufacturer; hence any such inversion is to be avoided as resolutely as possible.

In a pure solution of sucrose in water even the least traces of a mineral acid will occasion rather rapid total inversion at the temperature of boiling water. However, as soon as the salts of such organic acids as in their free state only possess a weak invertive power are present in the solution, the hydrolysing power of mineral acids is replaced by that of the much weaker organic acids, and that is why impure sugar solutions, such as cane juice, can stand with impunity a fairly lengthy spell of heating in a slightly acid reaction; whereas pure sucrose solutions would certainly become strongly inverted. This fortunate circumstance should not, however, lead us to count too far, and we should, therefore, at all events limit the heating of acid juices to the shortest period possible and watch the hot acid juices or syrups with the greatest care.

Besides acids, ferments and micro-organisms are apt to attack the sucrose, an action which reaches its maximum at temperatures of about 30° C. The ubiquitous yeast-cells find in the acid and luke-warm cane juice, as it comes from the mills, an excellent medium for their development, and they very rapidly decompose cane juice in which they are allowed to propagate and exercise their rôle.

The first action of the yeast is to break up the sucrose into glucose and fructose, and afterwards these two sugars along with those already present in the juice are transformed into alcohol and carbonic acid, while at the same time some other bodies such as glycerin and non-volatile organic acids are formed.

A second organism, *Leuconostoc mesenterioides*, causes the so-called "dextran fermentation" of the juice, by the action of which large masses of insoluble gummy substance are formed from the sugars of the juice; and this mucilaginous body may choke up pipes and plugs and cause great trouble in the factory apart from the loss of sugar thus occasioned. An alkaline reaction is favourable for the development of *Leuconostoc*, but its growth is a slow one in acid and neutral juice.

A third organism, called *Bacillus levaniformans*, has been detected in cane juices, where it produces another gummy substance bearing the name of *levan*.

Lactic acid bacteria, which are found in large numbers in every soil, also attack sucrose at ordinary temperatures and more rapidly at somewhat higher ones, thereby forming invert sugar and acid, and thus constituting a certain source of loss.

All these micro-organisms exert their action on the juices and syrups, in short, on all saccharine liquids of a lower dry substance content than 70 per cent. The reports of moulds or yeasts attacking syrups or molasses of a higher density have as yet not been fully confirmed, so that it is sufficient to concentrate the sugar juices in order to prevent the further action of micro-organisms. They are also killed by heat, and therefore any loss of sucrose by the action of micro-organisms, and the ferments which they secrete, is best avoided, first, by observance of the greatest cleanliness in the gutters, strainers, measuring tanks, etc., through which the raw juice flows; and, second, by using short gutters of limited capacity, small sand-catchers or none at all, in short, by making the period during which the juice has to pass between the mill and the juice-heaters as short as possible.

(D) Requirements for the various Processes of Clarification.

We find therefore the following indispensable conditions for the practical execution of clarification in making white sugar :—

The juice should be heated as soon as possible after being extracted by the mill, so as to prevent all action of micro-organisms.

The reaction of the juice when at a high temperature should be either neutral or only slightly acid or alkaline, in order to prevent inversion by an acid reaction as well as coloration by an alkaline one.

A comparatively large amount of lime should be used for clarification in order to remove as much as possible of the colouring matter derived from the rind of the cane; and in order to avoid the solution of the saccharetin from the floating particles of bagasse by the action of the lime, the juice should be carefully deprived of "cush-cush" by fine-meshed strainers.

If a large amount of lime has been used in the clarification process with a view to removing a very large amount of the impurities, the alkaline reaction of the juice should be eliminated before it is heated.

The whole clarification process should be accelerated from the moment the juice flows into the measuring tanks till it is pumped into the evaporators, so as to avoid as far as possible all decomposition and coloration of the juice by too prolonged an exposure to high temperatures.

The methods available for the clarification of cane juice with a view to preparing it for the manufacture of white sugar may be divided into three distinct classes:—

1. Those of the first category have this characteristic in common that clarification is effected with a comparatively small amount of lime, and they are summarized under **Defecation Methods.**
2. The second category is based on the action of a large amount of lime on the constituents of the cane juice, and as the excess of lime is afterwards removed by carbonic acid these processes are known as **Carbonatation Methods.**
3. A third category includes processes aiming at the clarification or decolorization of juices or syrups or even of dissolved sugars by various agents and contrivances, and these will be summarized under the heading of **Special Methods.**

CHAPTER II.

DEFECATION METHODS.

When clarifying cane juice in the manufacture of raw sugar, we may confine ourselves to adding only so much lime to the mill juice, as will neutralize the free acids and precipitate the acid phosphates in the form of flocculent tribasic calcium phosphate. The application of heat causes the dissolved albuminoids to coagulate, and the combined heavy precipitate envelops the floating particles of cane fibre, the waxy substance and part of the gums, thereby leaving a clear juice which may be decanted after subsiding from the layer of mud.

When crushing, however, a variety of cane, having a dark-coloured rind, or in general when crushing cane in a very powerful mill, so much colouring matter and so much of the gummy matter from the fibre will pass over into the juice, that a good quantity of lime is required for obtaining a thorough clarification and a clear juice. If in such a case only so much lime is added to the juice on tempering that a faint alkaline reaction is obtained, the clarified juice syphoned off from the mud in the subsiding tanks will be so much coloured and will contain so much gummy matter, that it could not well be used for the manufacture of white sugar. If more lime be used, the colouring matter and the gums will be precipitated to an increased extent, and the juice will be rendered more suitable for white sugar manufacture; but the excess of lime has to be removed before the juice be heated, in order to avoid coloration by the action of the lime on reducing sugars at high temperatures.

It is therefore necessary to temper the juice cold (i.e., at the temperature at which it comes from the mills) with the proper amount of lime; to allow a certain interval for the action of the lime on the impurities to be complete; to neutralize the excess of lime with some acid that produces an insoluble lime-salt; to heat the neutral or slightly acid or alkaline juice to boiling point in order to coagulate the albuminoids; and then to separate the scums from the clarified juice by decantation combined with filtration, or if possible by filtration only.

Possibly it is not so much the action of the lime on the gums and colouring matter which causes them to precipitate, but rather the amount and the character of the precipitate formed from the lime and the acids, for in many cases the order of treatment with the reagents is exactly reversed, the acid being added first and the lime afterwards; and although with this procedure there may never be an instant during which the lime was in excess to exert any special action, yet the impurities will be precipitated as completely as when lime had been added first and the acid afterwards.

It is very probable that the beneficial effect of the removal of the colloidal gummy, slimy and coloured matters is chiefly due to the formation of a precipitate in the heart of the juice itself, which fixes those gelatinous bodies by surface attraction and drags them to the bottom; and, seen in that light, it is immaterial whether the lime is added first and the acid afterwards, or *vice versa*. The chief consideration is that the precipitate be formed in the juice and that it be of such a character that it will attract the colloids.

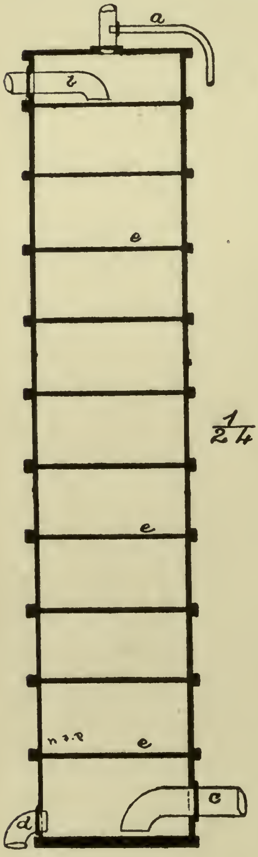

FIG. 1.
Sulphur Box.

While lime is the universal main ingredient used in clarification, the acids employed in the defecation methods are either sulphurous acid, or phosphoric acid, or combinations of both, while in some processes the action of the reagents on the colloids is emphasized by the addition of other bodies, such as phosphates, kieselguhr, fuller's earth and the like.

The principal acid reagent is sulphurous acid or sulphur fumes, while the application of phosphoric acid is much more restricted and indeed is only used as a complementary addition; we shall therefore consider first the various modes of application of sulphurous acid, generally called Sulphitation Methods. $\frac{1}{24}$

(A) Sulphitation.

Continuous Sulphitation.—The oldest method of sulphiting cane juices dates from the beginning of the last century and is still used in a great many sugar-houses. It is carried out in the so-called "sulphur box," (Fig. 1), a wooden upright vertical chamber about 12 ft. high. At about every foot of height are fastened horizontal perforated plates *e* or some other device intended to divide the flow of the falling juice into a shower. The sulphur fumes enter the box at the bottom through a pipe *c* and are drawn upwards through the box by a jet of steam *a* at the top of the box, which acts as an aspirator. The cold mill juice flows into the box at the top, falls on the topmost plate, where it is divided into a shower, trickles down on to the next plate, and so on till it reaches the bottom charged with the sulphurous acid from the upward current of sulphur fumes which it has encountered.

A more modern and more compact apparatus consists of a tank, some 4 to 5 ft. high, in which the sulphur fumes are conducted through a wide pipe ending a few inches from the bottom. The fumes are forced into the juice by means of an injector, while the juice enters through a pipe at the bottom and overflows through a second pipe three feet over the level of the first one. During its passage through the tank it encounters the current of sulphur fumes, absorbs the sulphurous acid and flows out charged with the proper amount of gas, which may be regulated by the velocity of the flow of juice or by the cock of the injector.

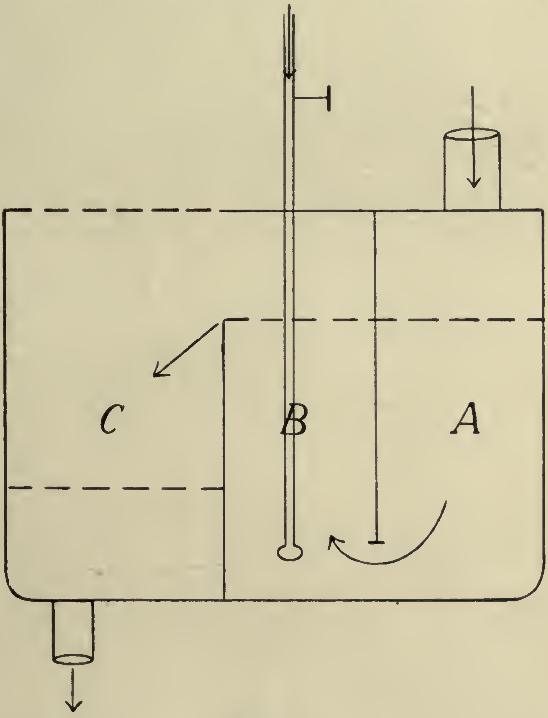

Fig. 2.
Continuous Sulphitation Tank.

In another device the juice is sulphited in a tank (Fig. 2), in which two baffle plates are fitted, which force the juice, pumped into the tank, to flow first down and afterwards upwards. These plates divide the tank into three compartments: the juice enters in the first, *A*, passes through the opening between the bottom and the first baffle plate into the second compartment *B*, where the current of sulphur fumes is forced into it. Together with the bubbles of gas the juice passes through the opening between the top and the second baffle plate into the last compartment *C*, whence it is discharged by a pipe at the bottom saturated with sulphurous acid.

The Quarez system of continuous sulphitation (Fig. 3) makes the juice itself regulate the rate of flow of the sulphur fumes which it needs. Here the juice from the mills runs through the pipe *B* into a tank *A*, which is divided into two compartments by a plate *C* almost reaching to the bottom; from here it is forced by means of a Worthington pump *D* through the injector *E*, which is in direct communication with the supply pipe from the sulphur furnace *G*. By this arrangement the amount of sulphurous acid absorbed by the cane juice during its course through the sulphitation column *K* may be regulated by the rate of flow of the juice itself, while no draught takes place and no sulphur is uselessly burnt if the current of juice is interrupted for some reason or other. The juice itself is pumped back into the second compartment of the tank *A* and flows out by the overflow *M*.

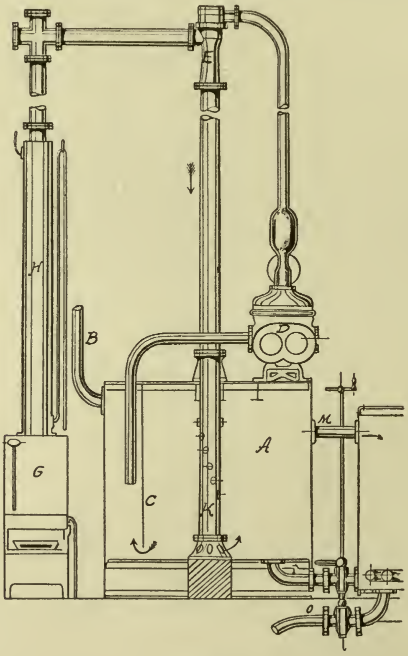

Fig. 3.
Quarez Sulphitation Apparatus.

Although the juice which is injected through the Quarez apparatus has previously passed through a series of copper strainers which deprived it of the greater part of the "cush-cush," yet this does not at all preclude some fine particles of cane fibre passing along with the juice and at the end choking the apertures of the injector. We therefore recommend that two injectors be fitted to every apparatus, so that the operations may be continued with the spare one, in case the other has become choked and needs dismantling and cleaning.

When working with the Quarez sulphitation apparatus, the flow of juice and of gas is, as nearly as possible, regulated in such a manner that the juice coming out of it has a sulphurous acid (SO_2) content of about 0·06 per cent., equivalent to 300 grms. of sulphur per ton of cane.

In the older apparatus just mentioned, the absorption of sulphurous acid can not be pushed so far; the average amount of sulphur, which after being burnt can be incorporated in the juice, does not exceed 100 to 150 grms. per ton of cane.

The large amount of sulphurous acid absorbed in the juice in the Quarez apparatus is certainly sufficient for the proper decolorization of the juice and for the production of an abundant precipitate with the lime added afterwards, while at the low temperature of the raw juice the degree of acidity mentioned above, if caused by the sulphurous acid, the hydrolysing power of which is relatively weak, is not high enough to be dangerous.

The sulphited juice is run or pumped into liming tanks, where as much milk-of-lime is added to it during its inflow that its reaction is neutral or very slightly acid.

The liming should be conducted so far that the juice does not impart a pink coloration to phenolphthalein paper, nor a red one to blue litmus paper, in which case there is neither an excess of lime, nor an excess of free sulphurous acid sufficient to cause inversion during the subsequent heating on defecation.

As neutral alkali sulphites will cause the colour of litmus paper to turn blue, the fact of a sulphited and limed cane juice not changing the colour of litmus paper is not a proof of its being just neutral. On the contrary, owing to the action of the dissolved sulphites, a cane juice which, after the treatment indicated, possesses an exactly neutral reaction to litmus paper is slightly acid by free sulphurous acid, and that is just what is required in the manufacture of white sugar.

The liming should be performed or at least completed in large tanks, provided with a stirring apparatus which keeps the contents in slow movement. These tanks should have the capacity of half-an-hour's juice supply, so as to facilitate the proper liming, which in this case has only to be finished once every thirty minutes.

This continuous sulphitation should be adopted in every case where it is thought advisable to sulphite first and lime afterwards. It is evident that, notwithstanding the greatest care, it is not always possible to regulate the flow of juice and gas, and the sulphurous acid content of the latter, so strictly that the juice always comes out with the same acid content; and therefore it is not advisable to make use of this method of sulphitation if it is necessary just to neutralize a limed juice, as in such a case it might become either too acid or be left still alkaline. Where the juice is sulphited first and neutralized afterwards with lime, there exists no necessity for a constant acidity and it is of no consequence whatever if the acid content varies in the sulphited juice to some extent, since later it is entirely neutralized by the lime in the liming tank.

If, however, the juice is to be limed first and sulphited afterwards, it is better to use intermittent sulphitation, as in that case the limed juice must be neutralized exactly by the acid.

Intermittent Sulphitation.—In the intermittent sulphitation process the limed juice is pumped into iron tanks, in which a current of sulphur fumes from a sulphur furnace is introduced, by which operation the excess of lime is neutralized, and can then be tested with phenolphthalein paper. As soon as the point of neutrality is reached, the juice is discharged from the tank, which is filled again with a fresh portion of limed juice, so that every tankful is treated separately.

Usually the sulphitation plant (*Plate* 1) consists of four or five iron cylindrical or rectangular tanks, each having a capacity equivalent to the amount of juice extracted per hour. A factory crushing 1000 tons of cane per 24 hours therefore requires a total sulphiting capacity of 900 gallons. The tanks are each covered with a lid fitted with a flue to allow the escape of the surplus gas. Further, they are provided with valves for the ingress and egress of the juice, a special valve for the sulphur fumes with lead and rubber fittings, and a perforated iron distributing pipe reaching to the bottom of the tank. A small cock fitted a few inches below the level of the juice, when the tank is full, permits of occasionally taking a sample of the contents in order to check the course of the neutralization.

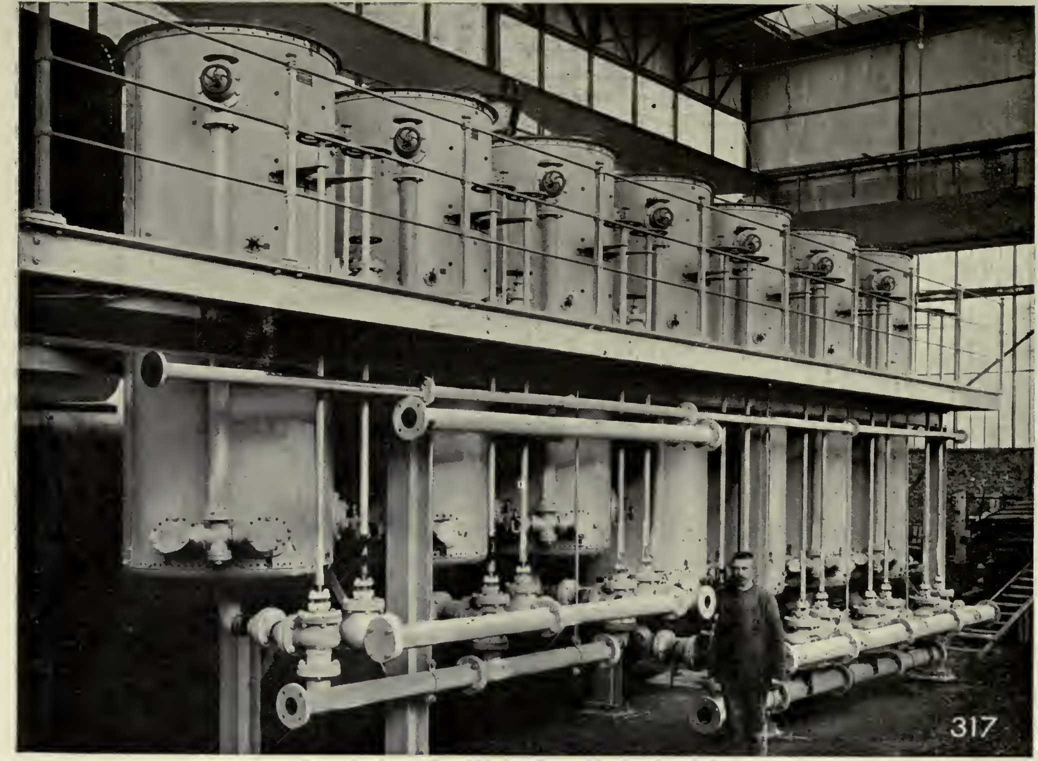

Plate 1.
SULPHITATION TANKS FOR JUICE.

The raw cane juice is tempered with about 10 or 12 parts per thousand of its volume with milk-of-lime at 15°Bé., pumped through a juice-heater, which brings it at a temperature of 60° C into the sulphitation tanks, taking care only to fill one at a time. During the inrush of the juice, the supply valve of the sulphur fumes is opened, and the current of gas is still maintained after the tank is full, until a sample of the contents taken from the test-cock and placed on a strip of phenolphthalein paper, or mixed on a porcelain tile with a drop of phenolphthalein solution, produces only a faint pink coloration. If at that point the supply of gas is slowly stopped, the last trace of alkaline reaction will then be saturated, and the juice will be neutral or slightly acid, though not yet imparting any red coloration to blue litmus paper.

The current of gas enters into the sulphitation tank through a perforated supply pipe which reaches to the bottom, and the contents of the tank are kept constantly in movement by the stream of gas and air, thus rendering any special stirring apparatus superfluous.

It has been seen that with the continuous process of sulphitation we strongly advised the reader to keep the temperature of the juice low during sulphitation, whereas with the intermittent process we advised him to heat the juice up to a temperature not exceeding 60° C. The reason for this distinction is, that in the former case the juice is rather strongly acid during some of the time, while it is always alkaline in the second instance. The sulphurous acid might invert the sucrose if the juice was heated, but in the alkaline juice the sulphurous acid is at once neutralized without being able to exert any inverting action. Next, it has been proved that at temperatures not exceeding 60° C, the lime at the concentration used here does not colour the glucose-containing juices, so that this temperature is not high enough to do any harm. On the other hand, a higher temperature favours the rapid absorption of the gas by the limed juice and at the same time promotes the precipitation or the crystallization of the calcium sulphite formed by that action. The precipitate is therefore formed in the tanks, while when performing the saturation at lower temperature the formation of calcium sulphite partly takes place in the juice-heaters, thus causing a rapid incrustation and even choking up the tubes of these vessels.

(B) Clarification with the Aid of Phosphoric Acid and its Salts.

Besides employing sulphurous acid for the neutralization of the alkaline reaction and for the formation of a flocculent precipitate capable of carrying down the floating impurities in the form of a heavy, rapidly-subsiding mud, much use is made of phosphoric acid, either in the form of the free acid, or of acid and neutral phosphates.

Phosphoric acid and acid phosphates act upon lime just as does sulphurous acid, namely by combining with the free lime, thus neutralizing the alkaline reaction, while at the same time the tribasic calcium phosphate precipitated envelops the colloidal and other substances and leaves the clear, supernatant juice free from suspended impurities.

Phosphoric acid and the acid calcium phosphates have, for many years past, found an extensive application in every cane sugar producing country. Formerly crude superphosphate, used also as a fertilizer, was thrown into the juice, and later this rough method was somewhat improved upon by using an aqueous extract of superphosphate, but such crude procedures, which have the disadvantage of bringing not inconsiderable quantities of the very objectionable gypsum (calcium sulphate) into the juice, are very seldom met with nowadays.

We prefer to use one or other of the many pure preparations, which after reacting disappear totally from the juice without leaving behind any impurities.

Pure phosphoric acid and acid calcium phosphates are sold under various trade names, such as "soluble phosphoric acid," "Ehrmannite," "Albuslite," "Claryphos," "Snowflake," etc., etc. These are all pure, and all of them contain either phosphoric acid only or compounds of that acid with lime, which after being neutralized by the lime in the juice, yield up all their phosphoric and lime content in the shape of the flocculent tribasic calcium phosphate, thereby clarifying the juice without leaving anything detrimental in it.

Some manufacturers prefer the use of sodium phosphate instead of the acid phosphates, especially in cases where they only want to obtain a heavy precipitate without increasing the acid content. We do not approve of such a substitute, unless it is administered in so minute an amount as to be equally harmless and ineffective, and this for two reasons. In the first place, the sodium phosphate, that is to say the common crystallized salt of commerce, though it has a neutral reaction to litmus paper, is in reality an acid salt, in which one-third of the phosphoric acid content is free. If this enters into reaction with a neutral calcium salt, so that tribasic phosphate is formed, the acid becomes free and in such a case the sodium phosphate does not fulfil its principal requirement. This is, however, not the worst characteristic as, after all, some dicalcium phosphate might be formed which is also insoluble ; but the greatest objection is that without any necessity sodium salts are carried into the juice, which can never be eliminated, and which increase the amount of molasses to be expected from the juice and consequently also the loss of sugar in the molasses. If one really wants to form the phosphate precipitate without increasing the acid content, it will be useful to add, simultaneously or at least after a short interval, phosphoric acid or acid calcium phosphate and the proper amount of milk-of-lime to neutralize that acid again.

In many factories only phosphoric acid preparations are used, while in others they are employed in conjunction with sulphurous acid in order to supplement the action of the latter. The application of the phosphoric acid solution remains pretty much the same in both instances, though sometimes the amount to be added may be modified, but these quantities depend so much on the character of the juice and also on the personal opinion of the operator that no fixed rules can be given on that point.

If the juice be sulphited first and limed afterwards, or if no sulphur is used and the phosphoric acid be added first and the lime next, the phosphoric acid solution is run into the juice at the rate of about 150 to 200 grms. of the actual acid per ton of cane before it is pumped into the liming tank. If one wishes to lime first and to sulphite afterwards, or if no sulphitation is wanted and only phosphoric acid is to be used to increase the precipitate or to promote its subsidence, the phosphoric acid may be added to the juice mixed with the milk-of-lime, while the quantity is regulated entirely by the rapidity with which the mud settles in the subsiding tanks and by the rate of filtration in the filter-presses. If the subsiding is too slow, or the filter-press cakes are too soft, an effort may be made to improvee th defects by the addition of phosphoric acid and lime to the juice, and the amount of both will depend entirely on circumstances. But, excepting what has already been mentioned on the subject, no fixed figures can be given.

The only item of interest is that usually the solution containing the phosphoric acid is made up to a density of 20°Bé., though it is evident that any other concentration will have the same effect, provided that the total quantity of acid remains the same.

Recently a new phosphoric acid preparation has attracted some attention, the so-called "phospho-gelose," which according to the claims of the inventor consists of dicalcium phosphate and kieselguhr or infusorial earth, while some pamphlets also mention the presence of certain iron compounds which are made responsible for the conversion of glucose into carbonic and oxalic acids. We leave this last statement for what it is worth, and only take into consideration the action of the phosphate and the kieselguhr. The process of treatment is very simple, as it only consists in mixing the cane juice, which comes out of a sulphitation apparatus charged with sulphurous acid, with sufficient milk-of-lime to give it an alkaline reaction, and immediately afterwards running in the "phospho-gelose," and if necessary some additional kieselguhr. The mixture is kept in motion in large tanks by a stirring apparatus for 15 to 20 minutes in order to give the reagent an opportunity to do its work, after which the juice is heated in the usual way.

According to the recommendation of the inventor, the necessary amount of "phospho-gelose" is about 14 lbs. per 1000 gallons of juice for purities between 85° and 95°, and about 22 lbs. for purities between 75° and 80°. From other communications we also learn it is well to add some more kieselguhr, the quantity being about half the weight of the "phospho-gelose."

The action of this new preparation is therefore identical with that which has ruled for many decades, namely, the formation of tribasic calcium phosphate from the acid phosphate and lime, while the subsidence is facilitated by the presence of kieselguhr, which moreover has the property of attracting slimy matter in its pores and therefore renders the juice clearer.

It has often been recommended to mix the cane juice with kieselguhr, fine clay, fuller's earth, and the like, in order to promote subsidence, and in one case even the use of finely ground pumice stone was strongly advocated; but all these admixtures enjoyed only a short run of popularity and were dropped for a time to be resurrected once more some later day.

(C) Heating the Tempered Juice to the Boiling Point.

When treated in one or other of the above mentioned ways with lime and acids or with only one of them, the juice is either cold or only lukewarm, and has next to be brought to the boiling point with a view to a thorough separation and coagulation of the totally or only partially precipitated impurities which are liable to be thrown down.

This is done most expeditiously in one of the many designs of juice-heaters or other devices in which a rapid current of juice is pumped through pipes which are grouped in a vessel heated by steam, the juice being thus rapidly and economically heated to a temperature above 100° C. After a few minutes' treatment, the juice is heated to the proper point at which the impurities acquire their maximum density and are rendered as capable as possible of being separated from the juice by decantation, followed by filtration, or even by filtration alone.

Yet, however rationally these apparatus for the heating of the juice may be constructed, it would be imprudent to depend on them alone in cases where juices loaded with so heavy a precipitate have to attain the proper temperature during their passage through their tubes. It might arise that owing to a very heavy incrustation of matter separating from the juice in the tubes, the transmission of heat would get so strongly hampered that, although the heater stood under the full steam pressure, the juice rapidly passing through the pipes would not be heated to the boiling point, and would offer an obstacle to the proper coagulation and hardening of the scums. As a rule heating will be sufficient if the area of the heating surface is well chosen in relation to the amount of juice and its initial temperature; but as one is never quite sure, and as too low a temperature in defecation is no longer reparable, making itself felt during the whole course of manufacture and being greatly detrimental to the making of perfectly white sugar, it is advisable not to depend entirely on the juice-heater. We are safe if we allow the juice coming from the heater to pass through a common iron defecation pan, where the juice is heated by means of a steam coil. The juice enters through a pipe in the pan, is heated to boiling point and flows out again in an intermittent current through the outlet pipe in the bottom. The juice boils fiercely, a process which can be actually seen and watched at a distance, making sure that the proper temperature has actually been reached; while at the same time all gas and air escape and so cannot occasion any disturbance in the settling tanks, whereas if they had remained in the juice, the continuous settling of the mud layer might have been interfered with by the bubbling up of some escaping gas or air.

It has been found that the incrustation or scale forming in the tubes of juice-heaters on heating sulphited juices (which principally consist of calcium sulphite and sulphate together with silica, calcium phosphate and fibre) adheres much more rigidly to iron than to brass tubes, while steel tubes become only slightly more incrustated than brass ones. This is a point which should not be lost sight of when ordering juice-heaters for factories in which the juice is sulphited. Further, it has been observed that when slightly acid sulphited juice is passed through a heater the scaling is much less than in the case of an alkaline sulphited juice, and again that calcium sulphite crystallizes much more rapidly from a hot than from a cold sugar solution. Calcium sulphite is an insoluble salt and in aqueous solution it is precipitated at once on neutralizing sulphurous acid solution with lime or on treating a calcium salt with a solution of an alkali sulphite; but in sugar solutions this precipitation is retarded, all the more when the temperature of the liquid is low. When the cold juice in which the calcium sulphite is formed, but from which it has not yet precipitated owing to the inhibiting action of the sugar, is heated, the precipitation will occur suddenly and, indeed, that is just what is witnessed in juice-heaters, in which cold sulphited juice is being treated. Frequently, even after a rather short period during which the cold sulphited juice has been pumped through the juice-heaters, thick layers of scale collect in the tubes, which sometimes acquire such dimensions that the passage of juice is blocked and the juice-heater can no longer be worked.

It has been found that if sulphitation of the limed juice is performed at a higher temperature and the size of the tanks is ample enough to allow a rather long stay of the sulphited juice before it is pumped through the heaters, the precipitation of the calcium sulphite takes place in the tank, and therefore the tubes of the heater are not so thickly scaled as when the juice is sulphited at a lower temperature.

In the preceding pages it was shown that a temperature of 60° C is still low enough to be used in sulphiting the limed juice, and indeed the reason why we advise sulphiting at that temperature is only that it is a remedy against incrustation in the tubes of the heaters.

However careful one may be in operating in this way, it will yet be impossible altogether to avoid scaling the tubes, and some incrustation must always be expected. It is therefore necessary to possess a juice-heater in reserve so as to have always another one clean and ready to take up the work, as soon as one of those in use loses in efficiency, owing to the scaling of the tubes.

(D) Separation of the Precipitated Impurities from the Clarified Juice.

Many a trial has been made, and numerous have been the expectations aroused by the introduction of some new clarification process that would succeed in filtering all the juice clarified by the defecation process. Now and then we hear reports of factories where all the defecated juice can be filtered through filter-cloth at once without subsiding, and in such cases we have always envied those manufacturers, for in our lengthy career in many cane sugar producing countries, we have never witnessed the act of juice extracted by powerful mills from ordinary sugar cane being filtered right away through cloth after having passed through a defecation process only. We can fully believe that when the extraction at the mills is poor, so that only a little gummy matter from the cane fibre passes along with the juice, it may be possible to filter the juice in filter-presses of great capacity, but we should not like to take the responsibility for establishing a sugar factory and relying upon the possibility of filtering all the juice without first subsiding and decanting off the greatest part of the clarified juice, which may then pass through some other filter, but not combined with the scums.

The new "phospho-gelose" process claims to clarify the juice according to the method just indicated, in such a way that the juice from the heaters may be filtered at once through filter-presses and may be separated there into clarified juice and hard scum-cakes of about 50 per cent. moisture. The filtering surface required is reckoned to be 600 to 700 square feet for juices from 85° to 95° purity and from 800 to 900 square feet for juices of 75° to 80° purity per 100 tons of cane crushed per 24 hours.

In some places this filtration has been performed without a hitch, though we are not sure of its success with every kind of cane or with every degree of extraction by the mills ; but perhaps we are too pessimistic on that point.

At any rate, up to now the usual way of handling the juice coming from the heaters has been to allow it to subside either in an uninterrupted operation or intermittently in subsiding tanks. The clear juice on being decanted or syphoned off is carried to the storage tank of the evaporators, on the way to which it either passes through a mechanical filter or through a sieve of finely-meshed bronze gauze.

The muddy layer at the bottom of the tanks or drawn through the cocks of the continuous subsidence apparatus is steamed and pumped through filter-presses, in which the scums are pressed together in the form of hard cakes, while the juice is expressed and is united with the clarified juice in the storage tank of the evaporators.

It is in most cases impossible to wash the scum-cakes in the filter-press ; while steaming them in order to recover as much of the juice contained in them is, in general, also a failure with the scums obtained in the defecation process. The loss of sugar on 100 parts of scums is therefore rather considerable, but is fortunately counterbalanced by the fact that the amount of scums obtained on 100 parts of cane is relatively small.

Details as to subsidence, decantation, treatment of the scums and filtration may be found in any text-book[1] on cane sugar manufacture, and as there is nothing new in them in respect to the speciality of white sugar manufacture, we think we are justified in referring our readers to those works, and so refrain from repeating the information here.

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[1] Cf. "Cane Sugar and its Manufacture," by H. C. Prinsen Geerligs.

CHAPTER III.

CARBONATATION METHODS.

All carbonatation methods of clarifying cane juice have the common principle of the addition of a large excess of lime to the juice and the neutralization of that excess by carbonic acid, the result being that the whole volume of juice may be passed through filter-cloth without any subsidence.

The methods of carbonatation may be divided into two categories, viz., those in which the glucose is left intact as much as possible, and those in which the total destruction of every trace of reducing sugars in the juice is effected.

The methods may also be divided into single and double carbonatation systems, according as the saturation with the gas is completed in one continuous operation or is split up into two partial saturations, separated by the operation of filtering the scums formed during the first period.

(A) Methods in which the Glucose is left intact as far as possible.

It is a well-known fact that reducing sugars, glucose as well as fructose, are rather rapidly transformed into organic acids under the influence of alkaline reagents and high temperatures. Lime will form a great many organic acids on being heated with a solution containing reducing sugars and will form lime-salts with those acids. The action is the more rapid, the higher the temperature, while the character of the acids formed varies likewise with the temperature. At the ordinary temperature of the atmosphere the action will be rather slow, and colourless products will be formed, such as saccharinic acid, lactic acid and the like. At temperatures over 55°C, the reaction will be much more rapid, while a multitude of dark-coloured products will be produced, which cannot be precipitated by the carbonic acid, and therefore will remain in the juice, being afterwards broken up into simpler compounds, such as acetic and formic acids, humin substances, carbonic acid, etc.

The inference from these observations is that if it be desired to keep the reducing sugars in the juice as intact as possible, and to prevent any formation of lime-salts in general and of objectionable dark-coloured lime-salts in particular, the temperature of the juice must be kept low so long as the alkaline reaction is still a strong one, work at the same time being accelerated so that the excess of lime does not act for too long a period on the glucose in the juice.

Yet the limed juice should not be saturated at ordinary temperature, for in that case the absorption of carbonic acid is only slow, while the precipitate formed in the cold liquid is much more difficult to filter than in hot working. In order to save time and filter-press capacity in carbonatation it is necessary to saturate at a somewhat elevated temperature, and in order to prevent too great a decomposition of the reducing sugars with the formation of dark-coloured products, it is wise to heat to as low a temperature as possible, choosing the highest temperature which is still harmless, viz., that of 55° C.

The chief action of the lime on the colouring matter from the rind, the gums, and the albuminoids is again a precipitating one, while the carbonic acid gas causes a precipitate with the lime, which envelops the floating impurities and forms so thick a layer of calcium carbonate that the slimy particles are incorporated among the hard granules, forming together a mass which is readily separated from the juice by filtration through cloth in presses. The cakes formed there are so permeable, that they can be washed with water and exhausted by steam, so that the loss of sugar is only small. In fact, notwithstanding the large percentage of carbonatation scums per 100 parts of cane, the sugar content in the washed cakes is so small that the total loss of sugar is generally the same as that occurring in the defecation mud, which is richer in juice, but considerably less in quantity.

The only criterion of proper liming is the manner in which the juice issues from the filter-presses. If it runs out freely, we might try to decrease the lime and see whether the same effect is still produced with a smaller amount, while if it flows from the presses with some difficulty, it is advisable to increase the addition to the next lot of juice and thus improve upon the earlier work.

It is therefore evident that the amount of milk-of-lime cannot be stated once for all; it greatly depends on the purity of the juice and its contents of gummy and slimy matter. Within wide limits, however, we may give the quantity of milk-of-lime to be employed as 7 to 10 per cent. by volume of the juice using milk-of-lime having a density of 15° Bé.

Intermittent Carbonatation. The cane juice from the mills, after having passed through the strainers which deprive it of the greater part of the "cush-cush," is heated to about 55° C, and pumped into the carbonatation vessels through an open gutter, into which is run the milk-of-lime so that the juice is limed during its passage from the pump to the carbonatators.

These last are iron tanks (*Plate* 2) sometimes open, and sometimes covered with an iron plate provided with a lid, permitting samples of the contents to be taken from time to time and allowing entrance to the tank for cleaning it. A chimney carries off the escaping gases, while a coil, into which steam may be admitted, can serve to heat the juice if necessary, and iron perforated pipes in the form of a coil or cross are provided for introducing the carbonic acid. A pipe of large diameter reaching to the bottom forms the inlet for the limed juice, while the saturated liquor with its precipitated calcium carbonate and impurities is discharged at the end of the operation by means of a valve at the lowest part of the inclined bottom of the tank.

As soon as the limed juice flows into the carbonatation tank, the carbonic acid supply is opened and the saturation starts at once. Care is taken to fill the tank only half full, for in the first stage of the saturation the carbonic acid combines with lime and with sucrose to form a very viscous combination, termed "hydro-sucro-carbonate of lime" which offers such a resistance to the bubbles of carbonic acid as to cause the formation of much froth, which fills the whole empty space left in the carbonatation tank. Even if the precaution has been taken to fill the tank only half full, this frothing may be so violent that the mass would overflow the tank if the lid were not well-closed, and sometimes would even rise into the chimney, causing the inner surface of the latter to be coated with cane juice and calcareous mud. At the same time the absorption of carbonic acid is very slow, so that much of the gas escapes unutilized through the viscous mass and up the chimney. After some time, however, the gelatinous compound is gradually broken up, because an increased amount of the calcium hydrate is turned into the carbonate by combination with carbonic acid, the juice becomes more liquid, and the carbonic acid is again better utilized, until a moment arrives when the precipitate settles rapidly, and a sample taken from the tank separates into a bright coloured alkaline liquid, and a greyish-yellow, rapidly subsiding precipitate.

Single Carbonatation.—Up to the stage reached here, the procedure is the same in both single and double carbonatation, but after that there is a difference. In single carbonatation the carbonic acid is admitted until a sample of the juice gives a faint pink reaction to phenolphthalein paper, after which the supply is slowly shut off, so that the juice becomes just neutral to the test paper. There is no fault in slightly over-carbonatating the juice at first, because there is always a danger of particles of unslaked lime present in the milk-of-lime gradually slaking and giving off free lime to the juice, causing an alkaline reaction, and leading to trouble in the filter-presses. It is therefore advisable to allow the saturated juice to remain in the carbonatation tank for a few minutes after the point of neutrality has been reached. It is then tested anew by means of phenolphthalein paper, and if the coloration obtained by the juice is stronger than a very faint pink tinge, the current of gas should be turned on till the dark pink reaction has reverted to a very feeble one. In this case the lime is almost entirely saturated and the ideal degree of alkalinity is attained. The juice is heated by means of the steam coil to a temperature between 50° and 55° C, and is filtered through presses without subsiding or decanting.

Notwithstanding all efforts to have a milk-of-lime as free from unslaked lumps as possible, and notwithstanding the precaution taken to leave the saturated juice standing a few minutes to allow those lumps to be slaked and yield their lime, it is not impossible that some calcium hydrate will still become dissolved during saturation and impart a rather strongly alkaline reaction to it. So long as the temperature is not over 55° C, this alkaline reaction will do no harm whatever, but it will certainly darken the colour of the juice by decomposing reducing sugars when the juice is heated before entering into the evaporators, and naturally in the evaporators too. It is therefore wiser to transfer the filtered juice to a sulphitation tank, in which it is brought into contact with sulphur fumes which neutralize the last traces of alkaline reaction and communicate an acid reaction ; this is, in the opinion of many sugar manufacturers, a favourable condition for the manufacture of white sugar.

We use here the term " alkaline reaction " and not " lime " because there will always be some alkaline reaction in carbonatated juice, which is due to potash and cannot therefore be neutralized by carbonic acid

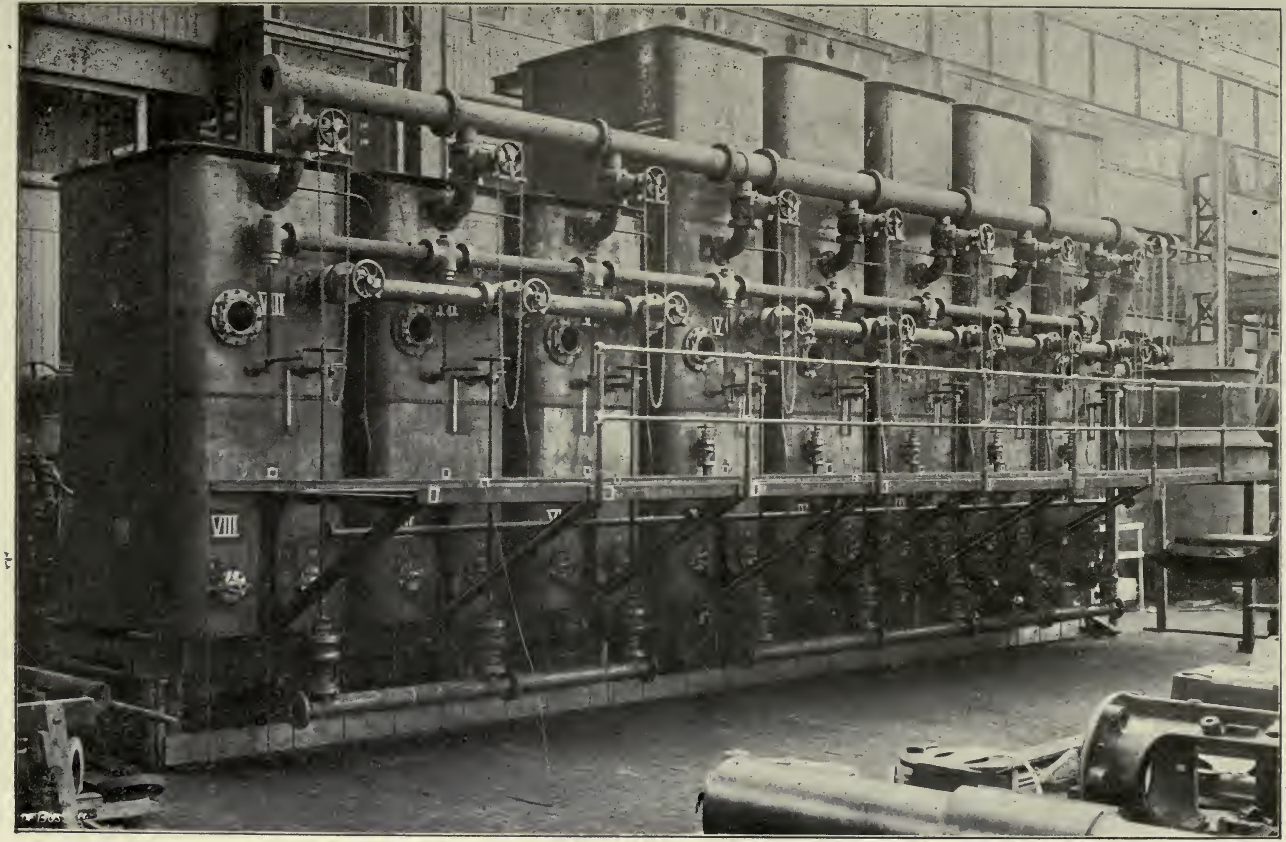

Plate 2.

Carbonatation Tanks.

The five right-hand ones for the first carbonatation, and the three left-hand ones for the second

unless over-carbonatation has taken place and potassium bicarbonate has been formed. As, however, this combination is again broken up by heating, the alkaline reaction of the potassium carbonate will reappear and do its detrimental work at the high temperature.

Cane juice always contains some potash combined with acids capable of giving insoluble calcium salts, among which phosphoric acid is foremost and perhaps also sulphuric acid, as the juice will contain potassium hydrogen phosphate and potassium sulphate. Lime combines with those acids and liberates the potash. This alkaline body remains in the juice with the large amount of lime added during the process, but after saturation the latter is thrown down by the carbonic acid, while the potash remains in the caustic state as long as there is any free lime left. As soon as the last portions of free lime have been neutralized, there are present in the liquid both lime-salts and potash, which latter becomes potassium carbonate and keeps the juice alkaline so long as it is not over-carbonatated.

It is therefore not only the unslaked lime which afterwards may render the juice alkaline again, but it may also be the potassium bicarbonate, which is broken up into carbonic acid and potassium carbonate, and accounts for the return of the alkaline reaction.

Both are to be avoided, and this is best done by allowing the filtered juice, as it comes from the filters in a luke-warm condition, to pass through a continuous sulphitation tank, just as has been described on page 15. The juice flows into the tank and sulphur fumes are admitted at the same time, while a test-cock placed in the wall of the tank a few inches below the juice level permits the taking of samples in order to test the degree of acidity of the juice.

It is not feasible to use litmus or phenolphthalein paper here, as, first of all, the atmosphere is too much charged with acid fumes to keep the paper in good condition, and furthermore many manufacturers desire to give the juice a fixed degree of acidity, so that a chemical analysis is necessary to estimate the proper acidity.

The sulphitation should be conducted so far that the juice is neutral to litmus paper, in which case it is in reality acid, for when it is titrated with N/100 potassium hydroxide solution it will need an appreciable number of c.c. of the standard solution to make it neutral to phenolphthalein as an indicator. This phenomenon is due to the fact that, after sulphitation, all the potassium sulphite and some of the calcium sulphite are dissolved, and produce a rather strong alkaline reaction to litmus, but are neutral to phenolphthalein. When complete neutrality to phenolphthalein is reached yet more sulphurous acid is admitted, the acid reaction of that body to litmus will neutralize the alkaline reaction of the salts, so that a moment comes when the liquid is neutral to litmus, but is in reality acid. This point is the very one which is most suitable for the manufacture of white sugar, for then the reaction is not too acid to cause inversion during heating. We therefore sulphite the juice till complete neutrality towards litmus, but, as mentioned above, the exact point of this neutrality cannot be ascertained in the vicinity of the tanks or sometimes in bad light, so we ascertain once for all with what real acidity this point coincides, and next sulphite till we have reached that point, in the manner discussed in Part III.

Double Carbonatation.—It has been observed that when the limed juice is treated with carbonic acid till a neutral reaction occurs, some impurities which had been thrown down by the lime, and were insoluble in the alkaline juice, are redissolved in the juice from which they had been removed in the first instance. This phenomenon is clearly shown by the aspect of the precipitate, which in the alkaline state, at the moment of its rapid settling, is gelatinous and yellowish, but becomes grey and granular, as soon as the point of neutrality is attained, while dark-coloured bodies are dissolved from the scums and pass into the juice.

Many manufacturers therefore prefer first to filter the great bulk of the precipitate in a still alkaline state and remove the last parts of the lime in a second carbonatation tank, so as to make the juice neutral without any risk of dissolving already precipitated impurities, which process bears the name of *Double Carbonatation.* At the same time the danger of contamination of the clarified juice by particles of lime slaking subsequently is totally avoided. Even if an inferior kind of milk-of-lime has been used, containing a large portion of grit and hard lumps which will slake after saturation and pass into the juice, these only increase the alkaline reaction of the filtered juice. But this is, at any rate, entirely done away with in the usual course of the second carbonatation.

Especially in those days before the second saturation with sulphurous acid had been thought of, double carbonatation had for the last named reason a distinct advantage over the former process, though this has now decreased to some extent ; but since the first reason has lost none of its significance double carbonatation still finds many adherents.

The limed juice is treated exactly as has been described on page 29, and is carbonatated until a sample of the juice poured into a cylinder glass rapidly separates into a brilliant layer of juice and a rapidly settling precipitate. It is advisable to have a row of such glasses and occasionally fill one with a sample from the tank in order to judge of the precipitation.

This rapid precipitation occurs as soon as the alkalinity of the juice has come down to about 500 mgrms. of lime per litre of juice; but as the proper point might be exceeded, in general an alkalinity of 400 mgrms. of calcium oxide (CaO) per litre is met with in practical working. At that point the juice, which has a temperature of about 55° C or has been warmed up to that degree, is discharged into a storage tank and then filtered in presses.

The alkaline juice coming from these is pumped for the second carbonatation into tanks which differ in construction from those for the first carbonatation. They are not covered, as the juice does not froth and for that same reason they may be filled to the brim. A coil or cross distributor for the introduction of carbonic acid reaches to the bottom, while the contents may be warmed by means of a steam coil or a Witkowitz heating apparatus. The use of the last-mentioned apparatus is to be preferred over that of a coil, since the juice has to be heated from 50° to about 100° C within a short time and therefore the Witkowitz heater (Fig. 4) is better suited to the task because of its large heating surface.

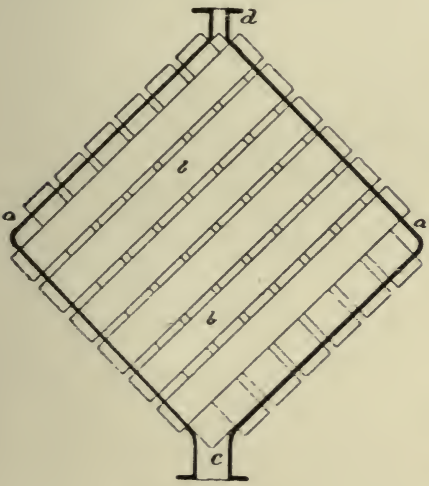

Fig. 4.
Witkowitz Heater.

Sometimes a very small amount of milk-of-lime is added to the juice, and sometimes this is omitted. Next the carbonic acid supply is turned on and steam is admitted into the heating apparatus or into the coils, so that the juice becomes simultaneously hot and almost neutral.

The carbonatation is continued till the juice only gives a faint pink coloration to phenolphthalein paper and immediately afterwards it is discharged from the tank into a storage tank, from which it is passed through filter-presses.

The juice coming from these, owing to the high temperature which it possesses, cannot contain any bicarbonate; yet it may still have an alkaline reaction as a consequence of its containing potassium carbonate, just as has been shown on page 31. In order to make the juice neutral or even acid, it can be passed through an apparatus for continuous sulphitation where it is sulphited exactly in the same way as was discussed under the heading "single carbonatation."

The time necessary for the saturation of the lime in the tanks for single carbonatation, or in those of the first stage of double carbonatation, depends on the amount of lime, on the carbonic acid content of the gas, on the shape of the tanks and also on the character of the juice, so that no fixed rules can be given as to the duration of "gassing." When calculating the carbonatation capacity one is, however, justified in allowing 25 minutes for carbonatation up to the settling point, the time required for filling and emptying included, so that the volume of these tanks (viz., those for single carbonatation and those for the first saturation in the double process) must be equivalent to the amount of juice produced in half an hour's time. The second carbonatation only requires a quarter of an hour, also inclusive of filling and emptying, so that the capacity of the tanks for the second carbonatation need only be half that of those for the first one. In this case it should be understood that by "capacity" is meant the actual working capacity and not the total capacity. This makes no difference for the second carbonatation, in which the total volume of the tanks may be utilized, but in the first carbonatation it must be remembered that the tanks are only half filled, so that the total space that is actually used and that required on account of froth must be taken as equivalent to the amount of juice extracted per hour.

Sometimes the juice of canes from certain fields, especially those having a soil rich in humus or heavily manured, or juice from fallen or from immature canes, containing an enormous amount of albuminoid matter, can become so thick and froth so violently that saturation is seriously protracted. In such cases the greater part of the carbonic acid passes unutilized through the liquid in large bubbles, a feature which prolongs the saturation time to twice or thrice its ordinary duration. Little can be done to prevent this excessive foaming, though theoretically it might be combatted by an increased addition of lime. The usual expedients resorted to in the factory to eliminate the froth, namely, pouring a little oil on top of it or beating it down by means of a jet of steam blown on its surface, are quite insufficient to keep that heavy foam within its limits; while the resort to heavy liming, which might combat the evil is out of question in a practical way, because the cost is too high. Moreover, the saturation of the excess of lime requires extra time, while finally the larger amount of press-cake involves a larger loss of sucrose in it.

The best way of treating such refractory juices is when crushing to mix the cane which yields them with cane from other normal fields and thereby diminish the influence of the impurities in the troublesome juice.

De Haan's Modification.[1]—The tiresome loss of time, of carbonic acid, and of space, induced de Haan to modify the method of adding the lime to the juice. Instead of adding all of it at once and having a strongly alkaline juice to saturate to a slightly alkaline reaction, in course of which the gelatinous body is formed, de Haan adds the lime and the carbonic acid during the whole run of the saturation, so that the alkaline reaction remains constantly slight, and the objectionable gelatinous compound is not formed at all. Hence the frothing period is avoided; the slow absorption of the carbonic acid is not experienced; loss of that acid does not take place; and the whole of the capacity of the carbonatation tank is utilized.

After passing the raw juice through juice-heaters until it reaches a temperature of 55°-60°C, it is run into the carbonatation tank, and as soon as the latter is filled, a certain volume of milk-of-lime at 20° Bé is run in slowly, the gas being then turned on. Through a test-cock small amounts of the contents of the tank are continuously drawn off, and tested with a titrated paper[2] of an alkalinity of 20 mgrms. of lime (CaO) per litre, so that when this special paper gives a neutral indication, it means that the alkalinity mentioned has been reached, though ordinary phenolphthalein paper will, of course, be coloured strongly red. This indication being obtained, more milk is run in, and "gassing" continued, always keeping the alkalinity of the juice neutral to the special paper. The workman has therefore only to regulate the flow of the milk-of-lime according to the indication of his test-paper, while the current of gas is kept on continuously. When the total amount of milk-of-lime found to be necessary has been added to the juice, the operation is finished by allowing the gas still to pass in till the juice is neutral to the ordinary phenolphthalein

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[1] *Archief voor de Suikerindustrie in Nederlandsche-Indië*, 1913, 1541; International Sugar Journal, 1914, 131, 438.

[2] Paper soaked in a solution of phenolphthalein and oxalic acid of a certain concentration and dried.

paper, after which it is treated just as has been described before in discussing the ordinary carbonatation methods.

It was found that by this modification the form of the calcium carbonate is more crystalline than in the usual process, whereby a better filtration is obtained and less milk-of-lime required to produce a readily filtering precipitate, while the purification of the juice is better, owing to the better preservation of the reducing sugars as a consequence of the slightly alkaline reaction.

The great advantage, however, is the avoiding of the frothing period, by which tank space and time are saved, so that the capacity of the carbonatation tanks may safely be half that of those used in the older process.

Weinrich's Process.[1]—Weinrich has patented a process in which the cane juice is submitted not only to double carbonatation, but is enriched with sugar from former operations which has been separated from the molasses in the after-products.

To this end the second and third sugars of the factory or, if it is desired, also raw first sugar is mixed with raw cane juice, in a suitable horizontal mixer, into a magma. Neither the mill juice nor the sugar should be heated previously, so that the temperature of the mixture does not exceed say 30° C. This magma is run continuously from the horizontal mixer into a vertical one, provided with agitators. This mixer bears at the bottom a wide goose-neck pipe, which has a height of about 3 ft. to provide for a continuous outflow, regulated by the inflow of the magma. To the magma is added continuously through a screen or sieve a certain amount of powdered quicklime, the quantity of which varies according to the amount and quality of the juice and-the raw sugar. If, for instance, 1000 tons of cane are crushed in 24 hours and 100 tons of sugar mixed with the juice, the total amount of lime-powder to be used should be from 14 to 20 tons. This amount of lime when added to the magma will give it a whitish colour, and turn it into a rather stiff mass. Owing to the small amount of water, it will slake very slowly, so that the mass will still contain some unslaked lime when being pressed out of the goose-neck pipe, which will take place 15 to 20 minutes after it first enters into the mixer. Through the action of the slowly slaking lime, the temperature of the mass will be raised during this time to 45° or 50° C, thus remaining below that of the destructive action of lime on glucose.

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[1] U.S. Patent, 1,084,771, January 20, 1914.

The mass, treated as above, is run continuously into a series of carbonatation tanks provided with stirrers, which tanks have been previously filled about 4 ft. high with raw juice, having a temperature of about 35° C. As soon as the mass begins to drop into the raw juice, the stirrers are set in motion and the introduction of the magma is stopped after a layer 1¼ to 1½ ft. deep has entered, raising the level to 5¼ to 5½ ft. As soon as the desired quantity of the mass has been mixed with the juice or a few minutes before, carbonic acid is pumped into it till a sample of the same taken out settles rapidly; and after this the juice is treated in the ordinary way by the double carbonatation process. The density of the juice enriched by the sugar will be 25° to 30° Brix.

Simultaneous Saturation with Carbonic and Sulphurous Acids.—Calcium sulphite crystallizes in a coarser form than the carbonate and, therefore, it has been tried with success to replace part of the calcium carbonate in the precipitate by sulphite, thereby rendering the scums more easily filtered and permitting a decrease in the amount of lime. This may be done by introducing some sulphurous acid fumes into the carbonatation tank along with the large amount of carbonic acid. It is, however, understood that this modification may only be used so long as the juice is still alkaline and on no account may it be continued with a neutral juice which at the same time contains the precipitated carbonate.

When using it in the double carbonatation process, the sulphurous acid should be passed in with the carbonic acid and turned off together with the latter, as soon as the precipitate settles well and while the liquid is still alkaline.

When applying it in single carbonatation, the supply of the sulphurous acid should be shut off the moment the juice settles rapidly and is still alkaline. No sulphurous acid should be applied after that stage, but the saturation should be completed by means of carbonic acid, while finally the last traces of alkaline reaction may be removed by sulphurous acid *after* the precipitated calcium carbonate has been filtered off.

It is evident that if one continues the supply of sulphurous acid to the muddy juice till a neutral reaction is obtained, then the sulphurous acid would attack the carbonate, liberate carbonic acid, and be completely wasted without any compensating advantages whatever.

Continuous Carbonatation.—Instead of treating every tankful of juice separately, attempts have been made to saturate the limed juice during its flow through an apparatus from which it would emerge properly saturated in a constant stream.

The raw juice from the mills, after having passed the strainers and measuring tanks, is conducted into a large tank where it is limed with an amount necessary to allow of a good filtration of the scums afterwards. The limed juice is pumped into a carbonatation tank into which it enters at the bottom, meets the current of carbonic acid and runs off from an overflow pipe. This carbonatator (Fig. 5) is a cylindrical vessel 5 ft. in diam. which has a juice outlet 15 ft. from the bottom. The current of the juice is upwards, but in order to prevent it from rising at once to the exit a couple of baffle plates are fitted in the cylinder, which change the direction of the flow and prolong communication with the gas. In this case also no trouble is experienced with the formation of froth; the juice enters the tank, becomes saturated, and runs out through the pipe with its proper degree of alkalinity. A man in charge tests the outflowing juice with a strip of phenolphthalein paper; if the colour is quite white he slackens the flow of the gas and when the coloration imparted to the test-paper by dipping it into the stream is more than just pink he opens a little valve. It is feasible to regulate the alkalinity by using special standardized acid phenolphthalein paper, which becomes red at a given degree of alkalinity, and so use continuous carbonatation in the single or the double method at pleasure.

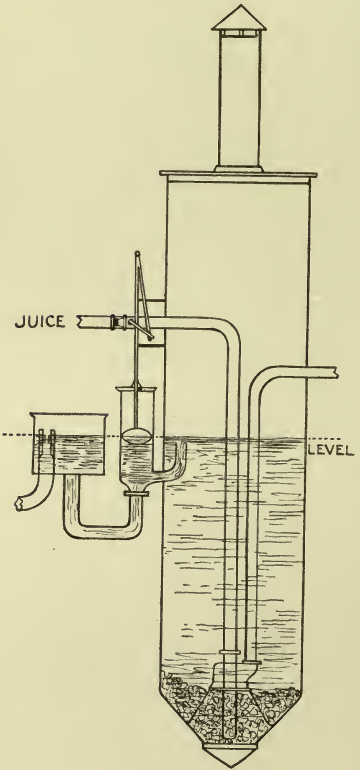

Fig. 5

Apparatus for Continuous Carbonatation.

There are as yet only a few factories using this continuous process for the first carbonatation, but there are many in which it is employed for the second carbonatation.

There the juice from the filters of the first carbonatation is pumped into a tank, entering at the bottom and leaving by a pipe at the top, while during the passage through the tank it is saturated by the carbonic acid admitted into it. The alkalinity is again regulated by the cock or valve of the supply pipe of the carbonic acid, just as has been mentioned above, with strips of phenolphthalein paper to test the proper saturation. The juice should leave with a very faint pink reaction, and is filtered through presses, after which the filtered juice may be neutralized or rendered faintly acid by means of sulphur fumes in the way already described earlier.

(B) Method in which the Glucose is totally destroyed.

All the methods mentioned up to now aim at a good clarification of the juice with the preservation of the reducing sugars as much as possible. It is true that while the juice remains at an elevated temperature in contact with lime, there will always be some decomposition of glucose and formation of lime-salts, and this will be greater in the methods where all the lime is added at once than in those where a slightly alkaline reaction is maintained during the whole operation.

There is, however, also a method which aims at a total destruction of the glucose by the lime, and is intended to cause the great bulk of the lime-salts to be removed by carbonatation, so that a clear and comparatively colourless juice ensues. This is—

The Battelle Process.[1]—In the Battelle process the cane juice is boiled with an excess of lime or of calcium saccharate till a sample no longer contains any reducing sugars; afterwards the alkaline juice is treated with carbonic acid till the precipitate settles rapidly, just as has been described under the heading of "Double Carbonatation." The only difference (which is, however, immaterial) is that the contents of the first carbonatator passes through a heater where it is brought again to 100° C, previous to filtering. The filtered juice is drawn into one of the second carbonatators, where a small amount of lime may or may not (this is optional) be added. Again carbonic acid is applied to precipitate the remaining free lime in solution, and again it is passed through a heater to increase the temperature to boiling point, being then filtered through the second presses dressed with the same material as the first ones (No. 8 or No. 10 duck canvas).

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[1] International Sugar Journal, 1913, 47, 535.

The clear juice from the second presses is evaporated to about 65 per cent. of solids, sulphited to slight alkalinity or neutrality, again filtered through presses dressed with the same material as the thin-juice presses, and the resulting syrup, which is clear, brilliant and rather free from colour, is ready to be boiled for a strike of white sugar.

We see at once that the small differences in the carbonatation and sulphitation of the juice in Battelle's process are an outcome of the fact that the glucose has been destroyed to begin with, so that heating in an alkaline state later on cannot do any harm.

In the processes described above, care must be taken not to heat the alkaline juice for fear of coloration, and that is why the juices are kept cold or rather lukewarm so long as they are alkaline. They are filtered cold after the first carbonatation, and the last traces of alkalinity sulphited out before evaporating or the juice even kept slightly acid, all for the same reason. As in Battelle's process the glucose is destroyed at the commencement, it is quite immaterial whether it be heated and evaporated with a faint alkaline reaction, since sucrose in that case is not attacked at all, as is known from the experience of the beet sugar industry. In fact, the clarification by Battelle's process has, practically speaking, turned the cane juice into beet juice.

The great difference as compared with the older methods is that in their case every precaution is taken to keep the glucose intact, and if it is necessary to have the juice alkaline, the temperature is maintained so low that the formation of coloured decomposition products is avoided as far as possible.

The result however of Battelle's experiments is that only in the case of the incomplete destruction of glucose by lime at high temperatures will the saturated juice contain in solution the dark-coloured lime-salts, whereas if there is an excess of lime and the boiling is pursued so long that all the glucose has been attacked, the greater part of the lime-salts and in fact all the coloured ones will be precipitated during the first carbonatation, thus leaving a colourless and glucose-free juice flowing out of the filters from the first saturation, which may be treated and exhausted in just the same way as with beet juice.

We must confess that this bold way of doing away with the glucose in the juice has impressed us very much, after our long experience with cold carbonatation and with the trouble encountered in the factory if, perchance, the temperature in the carbonatation tanks had risen over 60° C for a not too brief period. After having seen, however, the almost colourless juice quite free from glucose obtained by boiling cane juice with an excess of lime and saturating it to an alkalinity of about 500 mgrms. of CaO per litre, we were convinced of the possibility of obtaining a clarified juice which would be at the same time neutral, colourless, and free from glucose.

The principal point is that the excess of lime must be considerable and the boiling be continued long enough to have all the glucose decomposed. The amount of lime greatly depends on the percentage of glucose in the juice and may vary between 1·5 and 3·5 per cent. of the weight of juice. As an indispensable factor with this method is the extraction of the molasses by Steffen's process, in which sucrose is precipitated from the dilute molasses solution in the form of calcium saccharate, there is always a good portion of that material in the factory, which is used instead of quicklime.

As soon as the saccharate comes into the juice, it is broken up into sucrose, which unites with that amount already present, and also into lime, which at the high temperature attacks the glucose and also throws down all such impurities from the juice as may be precipitable by that reagent.

The raw juice is strained, then limed in a receiving tank with only so much lime as will exactly neutralize the acid reaction, after which it passes through a juice-heater or a Deming superheater, and is run into the carbonatation tanks to be mixed with the necessary amount of calcium saccharate from small tanks above each carbonatator. After having been exposed to the high temperature for a sufficient time to break up the glucose, the saturation is started as indicated.

(C) Separation of the Clarified Juice and the Scums.

The carbonatation methods are characterized by the fact that all the juice is filtered through cloth without subsiding or decanting, so that all floating impurities are withheld by the filtering material and are not dragged along with that part of the liquid which is only syphoned off and is sent to the evaporators without any filtration at all, or which may undergo some filtration through mechanical filters, never with complete success.

The filters used for separating the scums from the saturated carbonatation juices are either Beeg, Kroog, Kelly or some other system, while the cakes are usually so well permeable, that a thorough washing is possible, the sugar content of the washed cakes being reduced to a very satisfactory degree.

As we have showed before, this desirable result can only be obtained if the cakes are pressed dry in the presses, and whether this can be done depends entirely on the work of the sugar-house. If the amount of lime has been deficient, it is seen that the precipitate does not become firm enough, and that is why the tempering of the juice with lime has to be regulated according to the velocity with which the juice leaves the filter-presses.

If by some inadvertence, a carbonatation tank is discharged before the rapid settlement of the scums begins, it is obvious that also in this case a bad filtration will follow, since then the gelatinous hydro-sucro-carbonate of lime will not have been completely decomposed, through which fault in the work not only is the flow of the juice hampered and a too large amount of juice left in the scums, but, also, no inconsiderable quantities of sucrose enter into the insoluble compound and therefore add to the total loss of sugar. If however the amount of lime is well chosen, the saturation conducted to the proper point, the pressure of the pump strong and especially regular enough, and the cloths clean without being incrustated with dirt from former operations, one may be certain not only of a good filtration but also of a press-cake capable of being exhausted as far as desired.

It must not be forgotten that the recovery of the sugar from the scum-cakes requires the use of water which has to be evaporated again and thereby may burden too much the evaporating capacity. It is therefore wise to restrict the washing to such an extent that the expense and trouble of evaporating the wash-waters is not greater than the price of the sugar recovered.

In order to avoid the accumulation of too large amounts of water to be evaporated, the washing of the scums in the press is done on a rational basis in many factories by only adding the first denser sweet-waters to the juice and using the later and more dilute washings for the washing of the next press. To this end the presses are washed first with the dilute wash-waters from a former operation and the juice coming out is added to the clarified juice so long as it has a content of 4° Brix or over.

Next the press is washed with fresh water till the juice has a density of 1° to 2° Brix, then the latter is run into a tank, from whence the water is taken with which to wash the new presses. In this way the cakes are sweetened off to a small percentage of sugar and yet the dilution which the juice undergoes is not a serious one.

In some factories the scums are taken from the first carbonatation presses without washing them, after which they are transferred into a revolving drum and therein mixed with water until they are made into a thin paste. This is filtered in a second set of filter-presses and the sweet-water used for the maceration of the bagasse in the mills. The scum remaining in the presses is discarded from the factory as exhausted.

The foregoing discussion only relates to the scums filtered from the single carbonatation, and from those of the first carbonatation in the double process, but has no bearing on the filtration of the juice from the second carbonatation nor from the sulphurous acid saturation after the carbonatation process. The scums arising therefrom are so insignificant that the filters may run hours at a stretch without any necessity for changing the cloths, and then it is sufficient to wash the same a little in the press to have the loss of sugar reduced to a minimum. The loss on filtration lies with the heavy scums and should be well controlled there, while it is comparatively harmless in the filtration of the second carbonatation juice.

CHAPTER IV.

SPECIAL METHODS.

Besides the two kinds of clarification methods for the removal of the greater part of the impurities and for the obtaining of a clear light-coloured juice, there are also a large number of special processes intended to take away colouring or slimy substances from the already partially clarified juice and make it more adaptable for the purpose of white sugar manufacture.

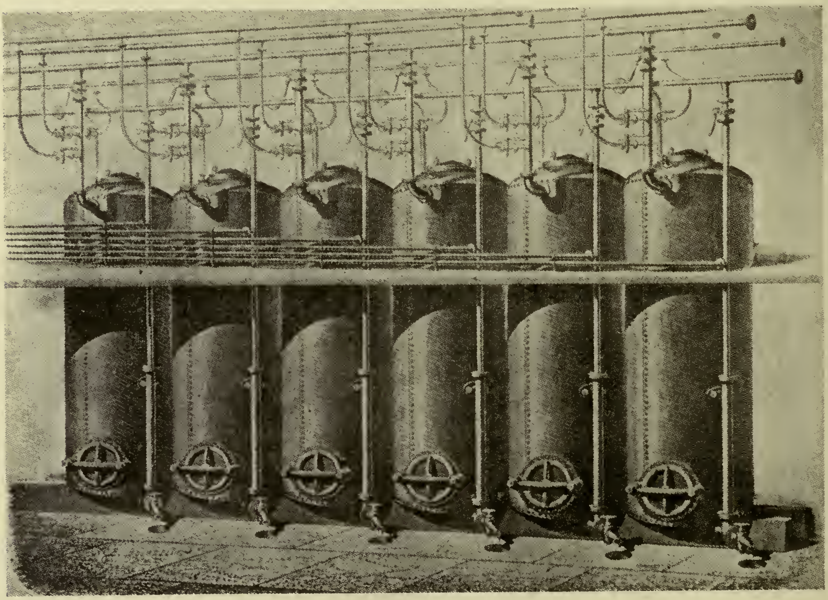

Fig. 6.

Char Filters.

These may be applied to the clarified juice, to the syrup, to the massecuite or even to the molasses, and they will be considered here according to the stage at which they are used.

(A) Methods for the further Purification of the Clarified Juice.

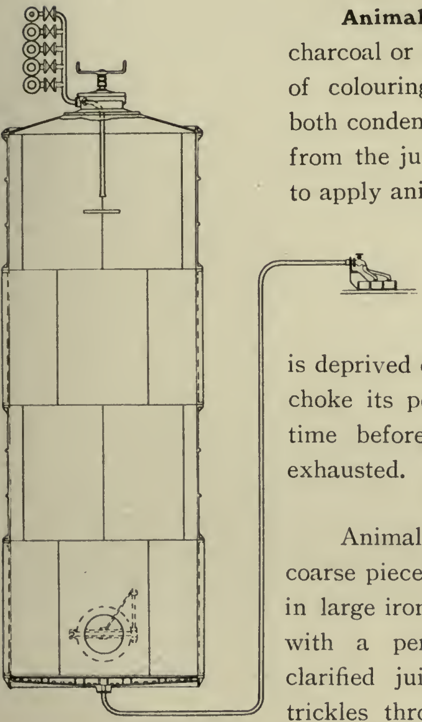

Fig. 7.

Section of Char Filter.

Animal Charcoal or Bone Black.—Animal charcoal or bone black is an excellent absorbent of colouring and gummy matters, which are both condensed in its pores and thereby removed from the juice. Although it might be possible to apply animal charcoal to the raw juice, from a practical standpoint it is indispensable to use it only for the juice which has been clarified beforehand as far as possible, and is deprived of all floating particles, which might choke its pores and render it inactive a long time before its real absorptive power was exhausted.

Animal charcoal is used in the form of coarse pieces free from dust, which are packed in large iron cylinders (Fig. 6 and 7) provided with a perforated false bottom. The hot clarified juice is poured over the char, and trickles through it, losing its colouring matter and some of its odour, while its soluble sulphates are for the greater part transformed into soluble calcium sulphate. The decolorized juice runs from a cock at the bottom of the char cistern, and is sent at once to the evaporators.

Gradually the animal charcoal over which the coloured juice has passed for a long time becomes saturated with the colouring matter and other impurities and is incapable of absorbing more, and so a revivifying process is applied to render it active again. To this end the char in the cisterns is washed with water till the density reaches 1–2° Brix, while the wash-waters are collected with the juice. After that, it is washed further and the last washings discarded.

After this operation it is taken out of the cisterns and boiled with a solution of sodium carbonate which dissolves any colouring and slimy matter present and transforms the calcium sulphate into calcium carbonate. It is washed again (Fig. 8), and then treated with a rather dilute solution of hydrochloric acid, which dissolves the calcium carbonate as calcium chloride. This treatment with hydrochloric acid has to be done very carefully, and certainly not more of the acid should be employed than is strictly necessary to re-dissolve the calcium carbonate formed from the gypsum. In its natural state animal charcoal contains some calcium carbonate which should be preserved, since it is an indispensable constituent for the action on colouring matter. If, therefore, by too copious a use of hydrochloric acid all the calcium carbonate be dissolved, the revivified product would not serve the purpose so well as it would have done if some calcium carbonate had been left in it.

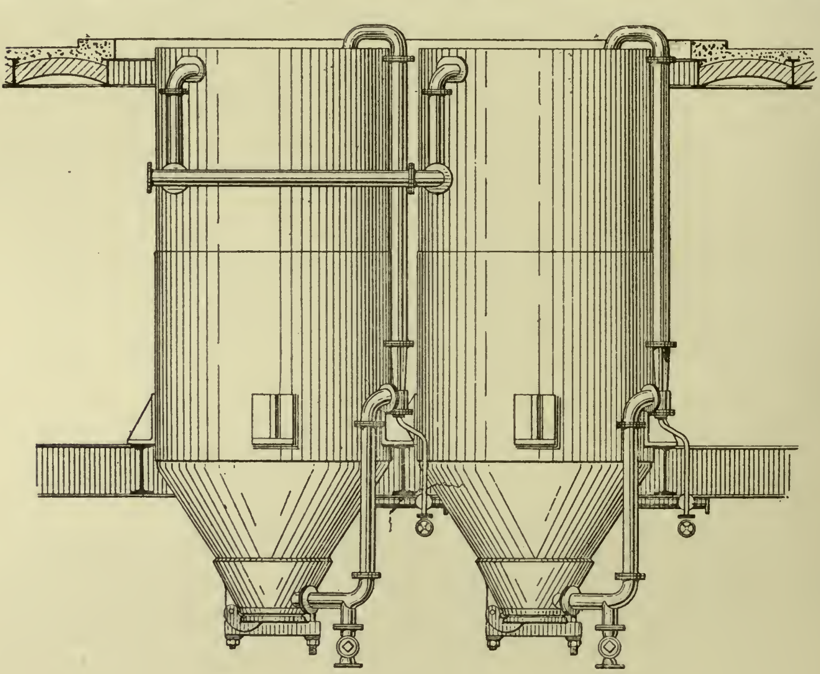

Fig. 8.

Charcoal Washers.

The purified material is washed with water from the hot-well till the calcium chloride has been removed, afterwards being conducted to a dryer in the form of a cascade or trip, and heated by the flue gases of the calcination oven. It is next passed into this calcination oven (Fig. 9), and heated by a coke fire in absence of air to a certain temperature, being discharged after a certain time through refrigeration tubes shut by automatic valves which allow the revivified and cooled char to be with-

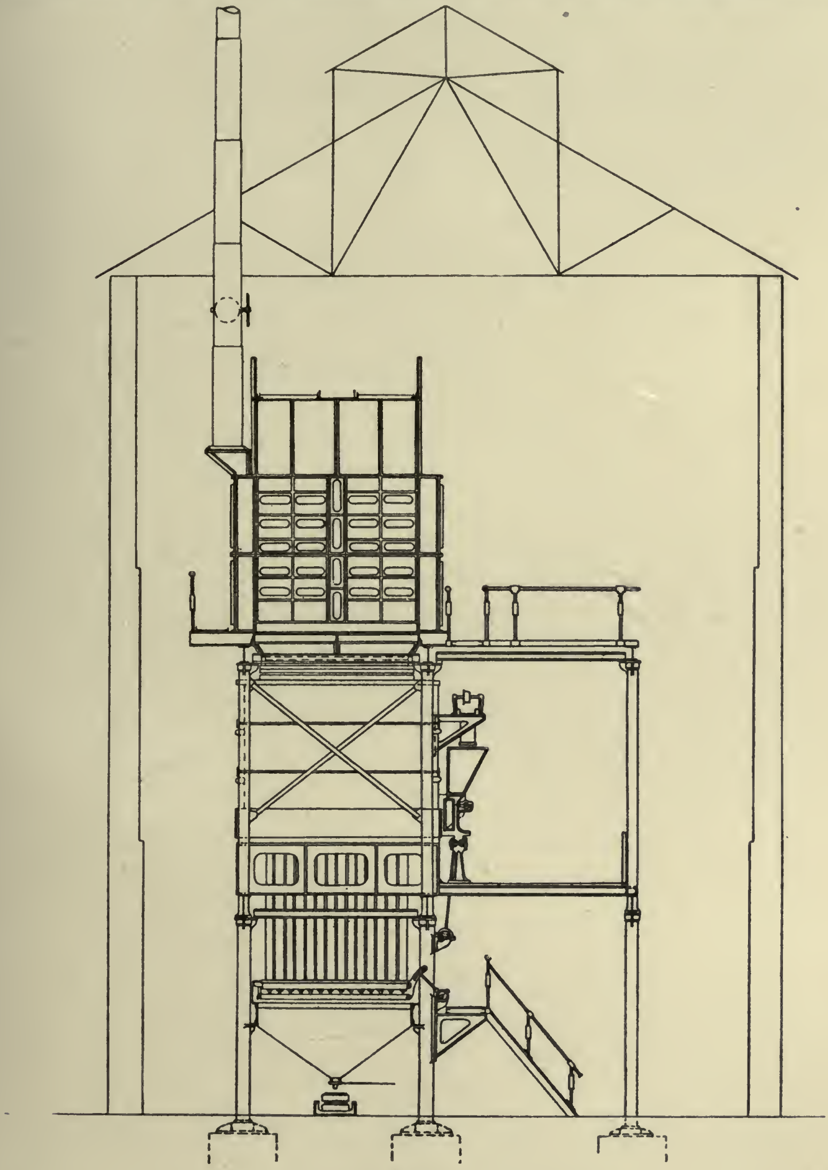

FIG. 9.
Char Revivification Kilns. (End Elevation).

drawn from time to time without admitting air into the oven. White spots on the char may show that notwithstanding the precautions taken air has entered the hot oven through leaks in the ironwork, which defect must therefore be remedied.

The revivified animal charcoal is sieved in order to remove the dust, after which it may be used again, although it has not its full decolorizing power, which now may be reckoned to be three-quarters of what it originally was.

The amount of char necessary for the decolorization of juice depends largely on its quality and on that of the juice, but if its weight be made up with fresh material so that the quality remains pretty well the same, we may take the quantity required to be 25 per cent. of the weight of the sugar expected from the juice.

Decolorizing Carbons: "Eponit," "Norit," Noir épure, etc.—The decolorizing material in animal charcoal is chiefly the carbon which it contains, although, as has been mentioned before, the inorganic constituents are not without some action. As the amount of carbon in the animal charcoal only amounts to about 12 per cent., it is obvious that the same decolorizing effect could be obtained with a much smaller amount of some material principally consisting of carbon. Further, the revivification of the char is a tedious operation, especially as the drying and calcination require much time, attention and fuel, while, finally, its use is a hindrance to the sale of sugar thereby decolorized, if it is intended for the Indian market because of the religious prejudices of the Hindoos.

For these three reasons the use has been recommended of preparations of vegetable or mineral origin, having a high carbon content, and being either so cheap that revivification is unnecessary or else capable of revivification without being dried or carbonized. Among the many decolorizing powders and preparations we might call attention to "Eponit," "Norit" and Noir épuré, which are all highly carbonaceous, very finely powdered preparations, the origin and manufacture of which are kept strictly secret by the patentees. Strohmer[1] describes experiments made with "Eponit" in the decolorization of refinery liquors (clairces) upon which he found decolorizing carbon to exert a much stronger decolorizing power than animal charcoal, but since that publication sugar literature has contained little on this subject. Dr. Wijnberg has taken

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[1] *Österr.-Ungar, Zeitsch, Zuckerind.*, 1910, 687.

out patents[1] for the decolorization of cane juice with these kinds of decolorizing carbons, and chiefly for the revivification of the rather expensive material, from which patents we quote the following particulars:

The cane juice clarified in the usual way with lime is saturated with sulphurous acid until 100 c.c. of the juice require 10 c.c. of N/100 alkali for neutralization to phenolphthalein. To this juice is added from 0·3 to 0·5 per cent. of decolorizing carbon, after which the mixture is boiled or heated by perforated steam coils for a quarter of an hour. The juice is now filtered in a filter-press, by which operation a colourless liquid is obtained, which is sent to the evaporators.

The decolorizing carbon is taken from the filter-press and boiled with a solution of sodium carbonate or bicarbonate or of caustic soda, the concentration of which is 10 per cent., but may be stronger or weaker. The liquid is filtered again and the carbon in the press washed first with water, next with a dilute solution of hydrochloric acid to eliminate the last traces of alkali, and finally with water again till the washing water has only a neutral or faintly acid reaction. The carbon remaining in the press after that treatment is immediately ready to be added again to a fresh portion of the sulphited clarified juice which is to be decolorized.

Sometimes the finely divided carbon passes through the filter-cloth with the current of juice, and has the disadvantage of causing a troublesome dark-coloured haze. This may be avoided by mixing the carbon beforehand with kieselguhr (infusorial earth) or other substance of a similar nature. If one fears to contaminate the carbon with the kieselguhr, the latter may also be mixed separately with the already decolorized juice and filtered through a mechanical filter.

According to the patents, the very fine carbon is mixed with water or cane juice to a homogenous thin paste, which is carried by a screw conveyor to large mixers from which it is added to the slightly acid cane juice. The mixture thus obtained flows into a receiving tank, from whence it is pumped through juice-heaters into a storage tank, and there kept hot till it is pumped through the filter-presses. The residue remaining behind in the presses is discharged into a gutter, from

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[1] United Kingdom Patents, No. 11,860, of May 18th, 1912; No. 21,204, of Sept. 26th 1911. International Sugar Journal, 1912, 720.

which an Archimedian screw propels it into a vessel where it is boiled with soda solution. Filtration through presses follows, in which the carbon is washed with water, with acid, and with water again, and finally is discharged in the first-mentioned mixer to start its journey anew.

The inventor claims that his carbon, "Norit," may be revivified an unlimited number of times, and that it is only necessary to add new carbon in order to make up that portion which is lost during filtration in order to continue to work indefinitely.

The process has been tried a few times, when it appeared that the absorbent really collected so many slimy substances from the juice together with the colouring matter that the juice could be filtered through cloth. As yet, the process has not been applied in the regular work of a factory where powerful mills extract the juice from the cane; but it will, we understand, be given a trial in 1915 in Java.

(B) Methods for the Clarification of the Syrup.

During evaporation almost every cane juice, even that which had been filtered through cloth in presses after having been clarified by the carbonatation process or by animal charcoal, assumes again some turbidity, while in general the colour is darker than can be ascribed to the concentration alone. When comparing a syrup after its dilution to the density of the thin-juice with the same thin-juice from which it originated, the colour of the former will always be found to be somewhat darker as a consequence of the overheating and caramelization of some of its constituents at the elevated temperature. At the same time, constituents which had been dissolved in the great bulk of thin-juice at the high temperature of the clarification process may crystallize out for three reasons; firstly, because of the diminished quantity of the solvent (in this case water) which has been evaporated; in the second place, because of many lime-salts being more soluble in weak than in concentrated sugar solutions; and in the third place, because the temperature in the last vessel of the evaporating plant is much lower than in the clarifiers or in the filters, so that many bodies also find an opportunity of crystallizing out. Most of those impurities deposit on the tubes of the evaporators, but a good part remains in the syrup, making it turbid.

Syrup from carbonatated juices, or in general from every juice which has been filtered through cloth during the clarification of the cane juice, may be filtered again through mechanical filters or through filter-presses dressed with cloth; but syrups from juices which in their dilute state have been syphoned off without having passed through filter-cloth cannot be so treated.

It is clear that one of the first requirements for a syrup intended to yield white sugar is that it should be clear and free from floating particles which might form a nucleus for the crystals or might crystallize with the sugar, giving it a dark tinge. This requirement is as necessary as a light colour, and perhaps it is the more important of the two, and therefore much care ought to be taken to free the syrup entirely from insoluble impurities. Those juices which, owing to their content of gummy matter, cannot be filtered through cloth should be allowed to subside just in the same manner as was shown in the description of the defecation process. The syrup coming from the evaporators is boiled in an eliminating pan and run into settling tanks where the impurities subside and the clear supernatant syrup is decanted off. The scums are pumped to receivers for the raw juice which has passed through the measuring tanks, thus undergoing again the whole course of clarification; while the clear decanted or filtered syrup may still be clarified, decolorized or bleached by one of the following methods for the further treatment of the syrup.

Filtration over Animal Charcoal.—The syrup which either by filtration or by decantation has been deprived of its floating impurities is run into the char cisterns described when dealing with animal charcoal filtration of the thin-juice (page 45). The best and most economical way of employing the char is to use the filters filled with the fresh material first for the syrup and afterwards for the clarified thin-juice, and this for two reasons; first, to give the thick-juice the advantage of the most active material; and next, to saturate the exhausted char with the most diluted juice and thereby save wash-water at the revivification stage. The amount of char for the decolorization of syrup is about 5 per cent. of the weight of sugar to be expected.

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[1] It has been observed that the most favourable density of the syrup lies between 26° and 28° Bé. measured hot. If the density is higher the subsidence is too slow and too difficult, while at a low density the scums settle rapidly as far as they are formed but the quantity separating from the juice is too small, so that the purification is incomplete.

Sulphitation of the Syrup.—Whereas animal charcoal actually removes the colouring matter of the syrup, sulphurous acid only bleaches it temporarily, but at the same time it reduces the ferric salts in the juices and syrups to ferrous salts which are colourless and do not crystallize together with the sugar in an acid syrup, so that a syrup sulphited to a rather considerable acid reaction will yield on further concentration white crystals which after having been washed will remain colourless a long time after delivery.

For this purpose the reaction of the syrup should be distinctly acid, and although the inverting action of the sulphurous acid and of the organic acids which it liberates from their combinations is not a great one, yet it is prudent to keep the temperature of the sulphited syrup comparatively low, *i.e.*, below 70° C.

Filtering and subsiding require on the contrary a high temperature for the proper separation of the dirt, and so the syrup should be filtered or decanted first at a temperature as close to the boiling point as possible, after which it should be cooled (if it has not become cold enough during subsiding) previous to its being exposed to the action of the free sulphurous acid during sulphitation.

This cooling may take place in tanks provided with coils through which cold water flows and carries off the surplus heat, or the syrup may be pumped through a juice-heater, the casing of which is filled with water ; but at any rate the syrup must come into the sulphitation tanks with a temperature between 65° and 70° C, never exceeding the last mentioned point.

This sulphitation may be performed either intermittently or continuously. In the first method, sulphitation tanks of the same design as those used for the sulphitation of the raw juice are employed, which have as the only difference a stirring apparatus (*Plate* 3). The raw juice is thin and mobile enough to be kept in constant motion by the current of gas which induces circulation and complete admixture of the contents, so that one part of the juice cannot become over-saturated while another is still alkaline.

The syrup, however, and still more so the molasses, are much thicker, and when sulphiting these it is well to have a stirrer operating in the tanks in order to distribute the current of gas throughout the liquid and prevent its passing out in a partly unutilized stream.

Plate 3.

Sulphitation Tanks for Syrup and Molasses.

The syrup was already neutral or slightly acid at the moment it entered the sulphitation tank, so that in this case the reaction of the juice fails to give an indication as to how far to proceed with "gassing." We therefore regulate the sulphitation according to the amount of sulphurous acid in the syrup, which is determined by titration with N/100 iodine solution, and regulated by the decoloration of the syrup.

It is not feasible to give fixed directions as to the amount of sulphurous acid to be used in the treatment, since the variety of cane, the method of clarification, and even the design and shape of the evaporators, greatly influence the amount of colouring matter to be bleached. The degree of sulphuring is regulated in the simplest way by experimenting now and then with one tank, which is constantly watched during the operation. From time to time samples are taken from the tank during the influx of the gas and poured in a test-tube, all of which samples are placed in a rack. The colour of the samples is inspected, and in the one in which a further addition of sulphur fumes causes no further decrease of coloration, the amount of sulphurous acid is determined. In the following tanks sulphitation is continued until the syrup has absorbed the same amount of gas previously shown to exert the maximum decolorizing power with the smallest amount of sulphurous acid. When passing on to another cane variety or in case of any great change in the tempering of the raw juice, the amount of sulphitation required should be determined again, but if no change has occurred, the figure found once will hold good a long time. Although, as has been said, no fixed rules can be given herein, yet we may say that for the manufacture of white sugar, sulphitation is usually pursued till an acid content of from 1 to 1·5 grm. of sulphur dioxide (SO_2) per litre has been attained.

The continuous sulphitation of the syrup is described by Harloff and Schmidt[1] as follows:

"The continuous sulphuring process of concentrated juice is effected by means of two saturation tanks. In the first tank the filtered or subsided juice is roughly sulphured to just below the required point for the maximum bleaching, while but little sulphurous acid is admitted to the second tank in order clearly and easily to observe the final reactions. The supply of sulphurous acid to the first tank may remain unaltered, while the supply stop-cock of the second is turned on or off according to requirement. In both tanks the juice should be admitted from below and run off at about

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[1] "Plantation White Sugar Manufacture," page 109.

the same level. From the second tank it simply runs to the syrup supply tanks."

The proper point to which the admission of sulphur fumes should be continued is determined again by titration with N/100 iodine solution, as will be described in Part III, Section IX.

The temperature of the acid syrup is never above 70° C and in most cases will be lower; in the vacuum pan the temperature should also be below 70° C, so that there need be very little fear of inversion of the sulphited and rather acid liquor. Yet it should not be omitted to determine daily the glucose ratio of the unsulphited syrup and of the first massecuite, provided that there has not been any return of molasses between these two stages. If the glucose ratio is found to have risen, there should be great suspicion of the syrup having been too acid, and steps should be taken at once to remedy this possible error. Besides, the vapours coming from the vacuum pan in which the sulphited syrup has been boiled contain sulphurous acid, which at the high temperature and in combination with water vapour and oxygen from the air will form sulphuric acid, so that the pipes, domes, save-alls, and in short all the ironwork of the dome of the pan and the save-all and also of the condenser will be corroded. This is more keenly felt in factories where a so-called "central condensation" is in use, because there will be in that case much more opportunity for oxygen to enter into the piping than where every pan is only connected to its own condenser and air-pump. Much may be done by painting the inside of domes, pipes, condensers and save-alls with a coating of paint or enamel proof against acid and hot vapour, this being renewed every year; yet there is no security that the paint will not break off at some such exposed spot, when both an inverting action on the sucrose and a corroding action on the ironwork of the pans and conducts may occasionally prepare unpleasant surprises for both chemist and engineer.

Hydrosulphites: "Blankite."—It should not be a matter of surprise that where the powerful bleaching action of the sulphurous acid is recognized and appreciated, endeavours should have been made to avail one's self of those properties without running the risks connected with the acid nature of that chemical; and that is why the introduction of the very strong reducing and bleaching agent, sodium hydrosulphite or "Blankite," which bleaches in a neutral medium as well as in an acid one, was hailed with enthusiasm, notwithstanding its very high price.

Sodium hydrosulphite oxidizes very rapidly when moist, and still more rapidly in solution, forming sodium sulphite and sulphate, so that it must be stored dry in drums or barrels and never kept in solution. It is advisable to purchase the hydrosulphite in such small packages that one is sufficient for four or five days only. In this way it only remains for a short time exposed to the moist atmosphere and can be entirely used up before it becomes seriously deteriorated.

The bleaching action of the chemical, though a very powerful one, is not lasting, since the colour, as in every case of bleaching with sulphurous acid preparations, re-establishes itself again after exposure to the oxygen of the atmosphere. Since the hydrosulphite is extremely expensive, any return of the colour eliminated at a high cost must not be made possible, and therefore this agent should be added to the syrup at the very last moment, namely in the vacuum pan just when the grain is formed.

The proper charge per pan is weighed out (care being taken to use a dry spatula) mixed with a good proportion of syrup, and drawn into the pan through the butter-cock, after which the cock is washed with some water, which is also drawn into the pan. The solution should not be too concentrated and should not contain undissolved hydrosulphite, for if a strong solution or even part of the powder comes into contact with the uncovered copper coils or the calandria in the vacuum pan, it might be decomposed and form a patch of copper sulphide, which would be detached by the crystals in the pan, imparting a black instead of a white colour to them.

The charge stated by the manufacturers is one part of the hydrosulphite per ten thousand parts of dry sugar obtained, but in many instances double that amount is necessary to obtain a good decolorization.

Sodium hydrosulphite has been introduced into the sugar industry by the Badische Anilin und Sodafabrik of Ludwigshafen. A French preparation, called "Rédo," is calcium hydrosulphite, and is claimed to possess the same properties as the sodium salt; up to the present it has chiefly found employment in a number of French sugar factories.

Bach's Process.—Another method of combining the bleaching effect of sulphurous acid with a neutral reaction, to which is added the possibility of filtering the syrup from a juice clarified by the defecation process, is the syrup sulphitation method advocated by Bach.

In this process the cane juice is clarified in the usual way as if intended to yield ordinary raw sugar. It is therefore tempered with just sufficient lime to produce a good separation of clarified juice and mud when the former is decanted off, the latter filtered in filter-presses, and both decanted and filtered juices concentrated to syrup without any elimination, filtration or addition of chemicals.

The hot syrup coming from the evaporators is mixed with lime at the rate of 2 to 2·5 per cent. milk-of-lime of 15° Bé. on the volume of the syrup and sulphited at once to a neutral reaction. The copious precipitate thus formed occludes the floating and slimy impurities in such a thorough manner that the syrup may be filtered through cloth in filter-presses. The syrup comes out freely with a light colour and great brilliancy, while the press-cake of calcium sulphite, calcium sulphate, silica, and slimy, gummy and colouring matters is so coarse, that it may be washed to a sugar content of 2–3 per cent. of sugar with an amount of wash-water equal to 1 per cent. of the volume of the original raw juice; and, if it were considered necessary to proceed further with the washing, practically all the sugar could be recovered from the scums. It was found that a large excess of lime was indispensable, for if the amount of lime added to the syrup was reduced below a certain maximum, which has to be ascertained by experiment, the proportion between the calcium sulphite and the slimy matter becomes such that the scums do not filter well.

It is not so much a specific action of the lime which accounts for the formation of the good filtering precipitate, but the formation in the liquid itself of a precipitate capable of enveloping the colloidal impurities. If, for instance, the juice is sulphited first to the equivalent of 2 to 2·5 per cent. of milk-of-lime by volume, *i.e.*, to a percentage of 3–4 grms. of sulphur dioxide (SO_2) per litre, and the milk-of-lime immediately added until a neutral reaction is obtained, the same result is produced; and if one fears that the action of that quantity of sulphurous acid would be too strong, the acid and the lime may be added slowly and simultaneously, if only care be taken that the amount of lime does not fall below the limit ascertained, and that at the end the reaction is neutral. The syrup is filtered through cloth and may afterwards be sulphited to a slight acid reaction in order to obtain the best white sugar. As filtration is conducted without special heating, and therefore at the temperature at which the syrup left the evaporating plant, no cooling is required previous to the after-sulphitation, but the syrup from the filters is pumped through an apparatus for continuous sulphitation, where it is "gassed" to a content of 0·6 to 0·9 grm. of sulphur dioxide (SO_2) per litre. This is a much smaller quantity than that used in ordinary syrup sulphitation and perfectly harmless both as regards the dangers of inversion and corrosion.

(C) Methods for the Purification of the Molasses.

The molasses spun off the first or second sugars in the centrifugals, from which a further growth of crystals is expected, always contains suspended impurities, which ought to be removed before a new crystallization takes place, for the reason already mentioned several times, namely that they might become occluded in the crystal and impart to it an undesirable coloration. And even if they do not enter into the crystal, but remain suspended in the mother-liquor they can then do harm, because they make the thick molasses viscous and it is difficult to spin off the sugar crystals. Further, molasses from a badly boiled massecuite containing much false grain, or molasses spun off hot, may contain a multitude of fine sugar crystals, which ought to be dissolved before the molasses is used for further crystallization, either by being concentrated for a second boiling or returned to a first boiling for dilution or desaccharification. In the first case when the molasses is boiled smooth, the minute crystals will settle on the coils and become charred, or they will cause an irregular crystallization being mixed with crystals which form during cooling. If returned into first massecuites, the small crystals will find much larger ones already formed, and although they may grow during the last stage of the boiling process, their size never reaches that of the larger ones, so that here again a very irregular product will ensue. For these reasons all molasses which serve for further crystallization should be reclarified, and to this end they are run into an eliminating pan and heated by a jet of steam entering through a perforated pipe. The apertures of the pipe should be made downwards in order to prevent their being choked by subsiding dirt during the time that no steam is admitted. A greasy froth which contains all the floating and suspended impurities of the molasses appears on the surface and is skimmed off. At the same time the steam and the resulting dilution dissolve the fine crystals present in the molasses, so that a clear liquor is obtained having a density of about 70° Brix.

Molasses from syrups which have been filtered previously may be filtered again at this stage of the process, which may be done in Danêk or similar mechanical filters; but, on the whole, the molasses is not fit to be filtered, and the skimming of the steamed and diluted product will in most cases remain the sole purification to be applied.

In some factories the molasses is sulphited after its clarification. The clarified molasses is treated in a sulphitation tank, just as is used in the sulphitation of syrup, but in this case the use of a stirrer is quite indispensable, as otherwise the gas would merely pass through the liquid without mixing with it or affecting it. There are no rules to be given as to how far this sulphitation should be pursued; in factories sulphiting their molasses this process is continued only for a couple of minutes; and as, in general, the decolorization obtained at so late a stage of the manufacturing process is very insignificant, it does not make any notable difference if the molasses sulphitation be totally omitted; but the elimination, dilution and skimming, and if possible filtration, should be scrupulously observed.

CHAPTER V.

ADVANTAGES AND DISADVANTAGES OF THE DIFFERENT PROCESSES.

In discussing the advantages and the defects of the various processes described in the foregoing chapters, we must make a distinction between the great majority of methods in which the glucose (or reducing sugars) is kept intact as far as possible, and the ones in which it is deliberately and purposely destroyed.

It is a well-known fact that the final molasses of cane sugar products can be reduced to a far lower quotient of purity than that of beet sugar and it is also well-known that the presence of the large amount of glucose in the molasses accounts for that phenomenon. It is not the place here to dwell lengthily on the subject, which has been treated extensively in other works,[1] and we will content ourselves with the statement of the fact that in the beet sugar industry the limit of crystallization of the molasses is about 60 quotient of purity, whereas it is about 40 in the cane sugar industry.[2] That is to say that on every 40 parts of non-sugar in the clarified juice 60 parts of sucrose must be lost in the case of beet juice; while with cane juice the unavoidable loss of sucrose in the molasses is 40 on every 60 parts of non-sugar present in the clarified juice.

On eliminating the glucose from the juice the quantity of non-sugar diminishes; but, strange as it may appear, not that of the sucrose lost in the molasses per part of non-sugar. It is just the part of the non-sugar which remains in the juice which is the strong sucrose binder, while the glucose, on the contrary, by its action causes sucrose to crystallize out.

The removal of the non-sugar, glucose, is therefore the cause of more sucrose remaining in the juice than when it had been left there, which fact will be clearly understood from the following reasoning.

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[1] Cf.: H. C. Prinsen Geerligs' "Cane Sugar and its Manufacture."

[2] These values 40° and 60° represent the real purity obtained by dividing the actual sucrose content by the dry substance and multiplying by 100.

We shall for convenience' sake leave out of the question the sugar which crystallizes out and only consider the molasses, and find the composition of a typical cane molasses to be as follows:—

Sucrose	32		per cent.
Reducing sugars	23	48	,,
Other non-sugars	25		,,
Water	20		,,
	100		
Quotient of purity	40°		

On 48 parts of total non-sugar 32 parts of sucrose are lost, and supposing that a method of eliminating all the glucose or reducing sugars without any formation of soluble lime-salts or decomposition products, were found, the non-sugars other than glucose remain what they were, viz., 25 parts, but its sucrose binding power is that of the non-sugars in the beet molasses, with which it now shares the same character. As 40 parts of that non-sugar are known to immobilize 60 parts of sucrose, the 25 parts of the non-sugar immobilize $25 \times 60 \div 40 = 37{\cdot}5$ parts of sucrose. Instead of the 32 parts of sucrose immobilized by the 48 parts of total non-sugar from the cane juice, there are now 37·5 parts of sucrose immobilized by the 25 parts of the much more active non-sugar remaining, which fact shows that the removal of the reducing sugars, even if it has been done as favourably as possible, has the effect of bringing more sugar into the molasses than if we had left the reducing sugar unaltered.

This destruction of glucose is intentional in the Battelle process, and the inventor completes his scheme by extracting all the sucrose from the molasses by the Steffen process. This latter cannot be used for molasses containing reducing sugars as these interfere, but as soon as the glucose is removed, which is the effect of the Battelle clarification, all the sucrose can be extracted from the cane molasses too, which more than makes up for the larger sucrose content left in them. When combining therefore the sugar extraction by Steffen's process with the destruction of glucose by the Battelle process, much more sugar can be made than when the glucose is left intact and the molasses is thrown away unutilized. The Battelle process should therefore not be judged from its merits alone, but the subsequent extraction of sugar from the molasses should also be taken into account.

It is beyond the scope and subject of this present work to deal at any length with the Steffen process, and we shall confine ourselves to a brief description which may give an idea of the work to be done.

The molasses which is free from glucose or only contains a few per cent. of that constituent is diluted to a density of about 10° Brix, then containing about 6 per cent. of sucrose. The solution is cooled artificially to a temperature of 3° to 5° C, and milk-of-lime is added to an amount of about 20 per cent. of dry lime of the weight of sucrose. Next very finely powdered quicklime is added to the cold, dilute molasses solution by means of a shaking screen, at the rate of about 110 per cent. of the weight of sucrose in the solution, thus making the total amount of lime added about 130 per cent. of the weight of sucrose. The quicklime combines with the sucrose to form insoluble calcium saccharate which is at once separated from the solution in filter-presses, always maintaining the indicated low temperature. The cold liquid coming from the press is brought to the boil by passing through a heater to precipitate the part of the saccharate remaining dissolved, while the white cake of saccharate is washed with water of low temperature. As the wash-water gradually dissolves the cake, it should not be thrown away, but be collected and heated together with the first liquid in a juice-heater, by which process the saccharate, which had been dissolved, is precipitated again and may be separated in a filter-press and washed with hot water. The liquor from the hot presses is practically free from sugar and is sent to the sewer. The cakes both from the cold and the hot presses are carried to the sugar factory and are used for liming the cane juice as mentioned on page 41.

It will depend on the cost of lime and ice, and on the construction of the houses where all the cold work is performed, as to whether this process may be carried out in an economical way in the tropics; but theoretically there is no objection, all the more because in the hot summer months the whole of the exhausted beet molasses from the sugar factories in California is extracted by means of the Steffen system.

But, as we said before, the Battelle process of clarification cannot be employed unless at the same time the molasses extraction process be used, and up to now every trial in a tropical country on a commercial scale has lacked means for the extraction of the cane molasses, so that with our limited knowledge of facts we cannot compare the process with others. Therefore we have treated it here without any attempt at comparison.

When asked to choose between the many different processes for the clarification of cane juice intended for the manufacture of white sugar, we should be guided in our considerations by different observations. If we are sure to dispose of our whole product as white sugar, it is advisable to arrange the whole sugar-house for the manufacture of that special article and have the best machinery and equipment for that purpose. If, however, we make say half our crop in whites in one year without knowing whether in a subsequent season the market will be favourable for whites or perhaps be better for raw sugar, it is preferable to choose a system in which the main lines of manufacture run parallel and only some relatively small alterations are sufficient to turn out either raw sugar or plantation whites. In countries where limestone is scarce or even lacking, so that this bulky material has to be carried long distances at considerable cost, it is folly to think of carbonatation, while factories producing for the British Indian market find the use of animal charcoal absolutely excluded. It is therefore very difficult to state once for all, generally, which of the many processes is to be recommended, although, of course, such a choice is easy enough for each concrete case, in which the various factors are known and can be taken into consideration.

The general aim of the different methods for white plantation sugar is the obtaining of a clear and, if possible, of an uncoloured juice; and of these two the first desideratum is of foremost importance, the colour dissolved in the juice not being so objectionable as floating or suspended dark substances.

All the other points, the importance of which is often mentioned when discussing clarification, are of minor significance.

One always hears when some new defecating powder or chemical is offered to the public that the use of even a small quantity of the speciality will tend to increase the quotient of purity of the juices treated with it by several points, but in almost every instance the complementary particulars remain unmentioned, namely, which of the non-sugar constituents will be eliminated from the juice by the action of the chemical, since the addition of some non-sugar without a much larger removal of some other body could not give rise to any noticeable increase in the quotient of purity.

It must be borne in mind that the clarification of cane juice does not eliminate from the juice any great amount of impurities, but that it is more the improvement of the physical properties which is effected, as for example, the removal of the small amount of colloidal matter which hinders the juice from being filtered and keeps afloat the suspended ingredients.

If the raw juice is not too muddy, so that there is not much earth and clay in suspension to indicate too high a figure for the dry substance, the improvement in purity on clarification will not rise higher than a few points, and if it is much more elevated it is certain that there is something abnormal in the state of the raw juice, in all probability an uncommonly high mud content.

It is well understood that in this statement the real purity is meant, *i.e.*, the quotient of the real sucrose content and the dry substance, and not the single polarization and the degree Brix by spindle, for if the apparent purity is ascertained a rather large increase in the figure may be found with those carbonatation processes, in which the juice is left during a long time in contact with lime. The reducing sugars, which in fresh juice consist of glucose and fructose in such amount that the rotation of the mixture is levo-rotatory, change on contact with lime into a mixture of the two sugars in a different proportion with a small amount of mannose, which mixture has no rotatory power, the rotation of the several constituents neutralizing one other. If we only take into consideration the polarization and not the real sucrose content, the levo-rotation of the reducing sugars disappears, for which reason an increase in polarization is observed and is in that case wrongfully attributed to an increase in sugar content.

Although the quotient of purity should be ascertained daily in the raw and in the clarified juices, the increase in the figure should not attract any undue attention and on no account should it be used as a criterion for the value of one system or other of clarification for white sugar manufacture.

In general, that system of clarification is best which allows of complete filtration through cloth, together with a light colour in the syrup and a reaction which is as little acid as possible. Where these three desiderata are combined in a syrup, it constitutes a material having the best qualities with which to make white sugar. From a technical point of view, all the carbonatation processes with saturation of the last juice by means of sulphurous acid, Bach's process, and the decolorization processes with animal charcoal and decolorizing powders (provided that the juice clarified by them is filterable through cloth) suit that purpose, while the sulphitation processes, which do not yield a syrup which will pass through cloth, only occupy a secondary place. The strong sulphitation of the syrup has still the disadvantage of giving off strong acid vapours which may corrode the iron piping of the vacuum pan of the condenser and the dome, and can also cause inversion if the reaction should be too acid and the temperature in the pan too high.

From a financial point of view, the sulphitation methods are to be preferred as being the cheapest to instal and the cheapest in use. A carbonatation plant is rather expensive and does not offer any advantage if it is not used the whole season and every season, so that one is only justified in using that system when sure of finding a steady sale for white plantation sugar and thus making full use of an expensive plant.

If therefore the sugar factory is to be arranged for double use, *i.e.*, to turn out, according to the conditions of the market, raw as well as white sugar, one of the many sulphitation methods, or sulphitation combined with the addition of phosphoric acid preparations, or decolorizing carbons must be chosen, as these require the least change in the installation. In the case of sulphitation the best choice will be Bach's process, as therein the whole process up to syrup remains exactly the same both for making raw and for making white sugar. Only beyond the stage of syrup do we get any great difference; in the first case the syrup is eliminated, subsided and sent immediately to the vacuum pans, while in the latter case it is sulphited, limed and filtered.

It is not necessary to enter into the merits and defects of the various processes, as these have already been treated in the discussion on their application (pages 13–56). The following brief remarks in which they are reviewed according to their place in the list should prove sufficient:—

Sulphitation.—This is the simplest process which may be used in a factory where both raw and white sugar are to be produced. Neither the juice nor the syrup are fit for filtration, and the colour of the product cannot be relied upon. In this process a heavy incrustation of the juice-heaters is to be feared if the temperature of the juice on sulphitation is too low, while the acid vapours arising from the juice in the evaporators and distilling over into the condensation water may attack the plates of the boilers.

Sulphitation combined with the use of Phosphates.—This method is better than the first-mentioned, and is to be used in the same cases. The inventor of Guerrero's "phospho-gelose" procedure claims that juice treated with that clarifying agent may be filtered through cloth, and if this is confirmed in practice the sugar obtained has every chance of being of a very good quality. The acidity of the juice being less, there is less danger of acid condensation water.

Single Carbonatation.—This process is advisable for factories which are definitely intended for white sugar manufacture. It is a rather simple and not very expensive method, but there is a danger connected with it, *viz.*, that if the milk-of-lime contains unslaked lime it may slake afterwards in the filter-presses and give rise to an alkaline reaction in the clarified juice, which is most objectionable when in the manufacture of white sugar the alkaline juice is afterwards heated. This may be totally overcome if the filtered juice is sulphited after filtration, so that the alkaline reaction is quite removed, but then the difference from double carbonatation is not great, having only the distinction that the alkaline reaction in this case is removed by carbonic and by sulphurous acids in one stage, while in double carbonatation followed by sulphitation it is performed in two stages.

Double Carbonatation.—This is the classical white sugar making process, in which the juice is clarified and made fit for filtration in the surest and most reliable way. It requires a costly plant, and should therefore only be resorted to when the factory does not make anything else than white sugar. The action of the carbonic acid should be completed by sulphurous acid only to neutralize the last traces of alkaline reaction after the second carbonatation.

De Haan's Modification.—This modification of the carbonatation process is most rational, and should at all events supersede the old-fashioned way of adding the lime at once. By the gradual addition of lime together and simultaneously with the carbonic acid, time, space, lime and carbonic acid are saved, thus doing the same good work at less expense in shorter time.

Continuous Carbonatation.—What has been said of de Haan's modification can also be said of continuous carbonatation, which, if properly controlled, also saves time, space and lime.

Harloff's Acid Thin-juice Process.—The saturation of the juice from the filters after the first single carbonatation, to such degree of acidity that no danger of inversion is incurred and only a little sulphurous acid will be distilled off into the condensation water in the hot-well, is a good substitute for the second saturation or for the second half of the double process. It requires fewer carbonatation tanks and filters, also less heating and pumping, but requires close supervision of the acidity of the juice, and also of the condensation water intended for feeding the boilers. When using this process the water in the hot-well should be repeatedly tested with litmus paper to ascertain whether it is acid or not, and in the former case should be neutralized with sodium carbonate solution.

Weinrich's Process.—This procedure does not contain much of use for common sugar factories in which no second or third sugars are remelted and where no raw sugar is bought from outside and worked up together with the juice. Apart from the refining of these sugars, the process does not differ much from common double carbonatation.

Battelle's Process.—This is quite a new process and fundamentally different from all the others. It has great advantages, but requires a very expensive plant and an exceedingly well-trained body of workmen and overseers, because it cannot be used save in connection with an establishment for sugar extraction from molasses.

All these variations of the carbonatation process are, of course, only advisable for factories in which white sugar is the regular product made and not the occasional one.

Filtration over Bone Black.—This is rather an expensive, but at any rate an effective process for removing colouring and floating matter from the juice. It cannot be used where the product is intended for the British Indian market, while the revivification of the char is an expensive and troublesome operation.

Decolorizing Carbons.—These fulfil the same rôle as does animal charcoal, *i.e.*, they absorb colouring matter, and also slimy substances. Juice treated with them may be filtered through cloth, and revivification is feasible by simply washing with soda solution and hydrochloric acid without carbonizing. Their use has not yet gone beyond the experimental stage in cane sugar factories.

Sulphitation of the Syrup.—This is rather a dangerous operation if not extremely well controlled. Sulphitation is continued till the maximum effect is attained, but at that point the acidity may have become so great that there is danger of inversion in the event of the temperature in the pan going too high, while the ironwork may get corroded by the acid vapours emitted during concentration in the pan.

Blankite.—The use of Blankite has the same bleaching effect as has sulphitation of the syrup inasmuch as it decolorizes powerfully when applied at the right moment, *i.e.*, as late in the process of manufacture as it possibly could be. It has not the disadvantages of the former, because it is a neutral body and so does not invert or give off acid vapours. It may also be used as an aid in the other processes of decolorization and offer the final touches to the effect accomplished by the others.

Bach's Process.—A good rational process intended to give a bright, light-coloured and not too acid syrup, capable of ready filtration. It can be carried out without great expense in an apparatus which is simplicity itself.

All the five last-mentioned processes may be used in factories where it is required occasionally to make white sugar and at other times the common raw product. Of all those mentioned here with that intention, the Bach process next to the procedures in which decolorizing powders are used appears to be the most effective, although also in this case practical experiments are still too few in number for us to be able to give a definite opinion on its merits.

CHAPTER VI.

COSTS AND YIELDS OF THE DIFFERENT CLARIFICATION PROCESSES.

It is very difficult to submit any fair comparison of the costs of the various clarification methods, since the prices of the different materials and, be it remembered, the number and the pay of the workmen in the different countries, where sugar cane is cultivated and cane sugar is manufactured, offer such widely varying differences that figures for comparison, which hold good for one country, may be absolutely worthless for another.

We ourselves shall abstain here from giving figures as to the money involved in acquiring the plant for the processes, as in many a case where an existing raw sugar factory goes in for white sugar manufacture, a good part of the old plant can be incorporated in the new establishment, while in other instances everything has to be purchased brand-new. The cost of the installation has to be considered in every special case, and therefore no figures could be given in a general treatise like the present one, intended as it is for no special country. For the same reason we shall also abstain from giving an *exposé* of the daily cost necessary for the carrying on of the work, but we give here a list of materials required for the treatment of a given amount of juice by each of the several systems. When making a comparison anybody may fill in the prices of the materials in his country, and so see which will be the best for his use ; and as to the number of workmen we regret not to be able to give any data, because the work done by one man in one country or another differs so enormously that it is useless to quote figures and numbers from a given country as an example.

We shall only give one example from Java as a model specification of the different items, in order to draw attention to all the points entering into consideration for comparison.

We take as the unit *the juice extracted from 1000 tons of cane* and understand here *the English or long ton* of 20 cwts. or 2240 lbs. or 1016 kg.

Sulphitation of the raw juice: 0·80–1·5 tons of lime, ½ ton of sulphur.

Sulphitation followed by treatment with phosphates: 1 ton of lime, 200 lbs. of sulphur, 200 lbs. of phosphoric acid.

Sulphitation followed by treatment with "phospho-gelose": 1 ton of lime, 200 lbs. of sulphur, 1–2 tons of "phospho-gelose."

Single carbonatation and continuous double carbonatation: 35 tons of limestone, 4 tons of coke, 400 lbs. of sulphur.

Double carbonatation: 40–45 tons of limestone, 4·5 tons of coke, 400 lbs. of sulphur.

De Haan's modification: 23 tons of limestone, 2·5 tons of coke.

Harloff's acid thin-juice process: 30 tons of limestone, 2·7 tons of coke, 600 lbs. of sulphur.

Battelle's process: 250 tons of limestone, 30 tons of coke.

Animal charcoal: 25 tons of char (which can be used several times, requiring acid and soda for regeneration).

"Norit": 3–5 tons of the Norit (which can be used several times, requiring acid and soda for regeneration).

Sulphitation of the syrup: 250–300 lbs. of sulphur.

"Blankite": 20–40 lbs.

Bach's process: 2 tons of lime, 1 ton of sulphur.

In this table neither the capacity of the filter-presses nor the number of times the juices are filtered is taken into account, so that the costs for filter-cloth are not taken into consideration. Nor is the amount of dilution brought along with the milk-of-lime and in the washing of the filter-press cakes calculated, so that the cost of evaporating all the surplus water is not mentioned. In the Battelle process there is a big item for cooling the diluted molasses which should not be forgotten either. It would carry us too far and give the work an undesirable extension if we tried to calculate all these items for the different processes. After what has been said about them, the expense can be easily calculated for every concrete instance, and the two cases mentioned hereafter are only quoted merely as an instance of how to do so.

COMPARISON OF THE COST OF CLARIFICATION BY THE SULPHITATION AND DOUBLE CARBONATATION PROCESSES.[1]

(Per 1000 tons of cane.)

I.—SULPHITATION.

A. Materials.	£	s.	d.
Lime, 0·86 ton @ £2 12s. 2½d. per ton … …	2	4	10
Sulphur, 800 lbs. @ 1d. per lb. … … … …	3	6	8
Filter-cloths, 2 pieces @ 8½d. … … … …	0	1	5
	£5	12	11

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[1] Transcribed into English weights and currency from the Java examples of de Haan in the *Archief voor de Suikerindustrie in Nederlandsch-Indië*, 1911, 1354.

B. Wages per 1000 tons of Cane for such work as differs from that in the Carbonatation Process.

				£	s.	d.
Preparation of the milk-of-lime	2 coolies	@	5d.	0	0	10
Sulphitation of the raw juice	2 foremen	@	1/-	0	2	0
Subsidence of the sulphited juice	2 ,,	@	1/-	0	2	0
Do. do.	8 coolies	@	5d.	0	3	4
Decantation of the clarified juice	2 foremen	@	1/-	0	2	0
Treatment of the scums	2 ,,	@	1/2	0	2	4
Filter-presses	2 ,,	@	1/2	0	2	4
Do.	12 coolies	@	5d.	0	5	0
Clarified juice pump	2 foremen	@	1/-	0	2	0
Mending filter-cloth	3 women	@	3d.	0	0	9
Transport of scums	6 coolies	@	5d.	0	2	6
Purification of the syrup	2 foremen	@	1/-	0	2	0
Do. do.	10 coolies	@	5d.	0	4	2
Carrying away the scums after the grinding season				0	5	7
			Total wages	£1	16	10

II.—DOUBLE CARBONATATION.

A. Materials.

Limestone, 43·67 tons @ 7s. 4d. per ton	16	0	3
Coke, 3·42 tons @ £3 2s. 5d. per ton	10	13	5
Sulphur, 460 lbs. @ 1d. per lb.	1	18	4
Filter-cloths for the second carbonatation and the thin-juice filtration, 32 pieces @ 8½d.	1	2	8
Filter-cloths for the first carbonatation, 24 pieces @ 1s. 2d.	1	8	0
	£31	2	8

B. Preparation of the Materials.

Breaking limestone, 43·67 tons @ 2d.	0	7	3
Breaking coke, 3·42 tons @ 1s. 4d.	0	4	7
Carrying stone and coke to the kiln	0	2	6
	£0	14	4

C. Wages per 1000 tons of Cane for such work as differs from that in the Sulphitation Process.

				£	s.	d.
Weighing limestone and coke ...	2	foremen	@ 8d. ...	0	1	4
Do. do. ...	12	coolies	@ 5d. ...	0	5	0
Lime-kiln	2	foremen	@ 1/4 ...	0	2	8
Do.	12	coolies	@ 5d. ...	0	5	0
Preparation of the milk-of-lime ...	2	foremen	@ 1/- ...	0	2	0
Do. do. ...	8	coolies	@ 5d. ...	0	3	4
Engine drivers at the lime-kiln ...	2	foremen	@ 1/4 ...	0	2	8
Measuring the milk-of-lime ...	2	foremen	@ 1/- ...	0	2	0
Do. do. ...	2	coolies	@ 5d. ...	0	0	10
Carbonic acid pump...	2	foremen	@ 1/4 ...	0	2	8
First and second carbonatation ...	2	,,	@ 1/2 ...	0	2	4
Do. do. ...	8	coolies	@ 5d. ...	0	3	4
Pump for the raw juice	2	foremen	@ 1/- ...	0	2	0
Scum pumps	2	,,	@ 1/- ...	0	2	0
Pumps for the washing water of the presses	2	,,	@ 1/- ...	0	2	0
Filter-presses	2	,,	@ 1/2 ...	0	2	4
Do.	38	coolies	@ 6d. ...	0	19	0
Transport of scums	2	foremen	@ 1/2 ...	0	2	4
Do.	16	coolies	@ 5d. ...	0	6	8
Mending the filter-cloths	16	women	@ 3d. ...	0	4	0
Sulphitation of the thin-juice ...	2	foremen	@ 1/- ...	0	2	0
Purification of the syrup	12	coolies	@ 5d. ...	0	5	0
Carrying away the scums after the grinding season ...				2	2	11
			Total wages	£6	3	5

We find therefore the total expenses coming into consideration here for the comparison of the two systems (the other expenses of labour, etc., being the same and those for the fuel remaining outside the scope of the problem) to be the following per 1000 tons of cane:—

	Sulphitation.			Double Carbonatation.		
	£	s.	d.	£	s.	d.
Materials and their preparation... ...	5	12	11	31	2	8
				0	14	4
Wages	1	16	10	6	3	5
Total Expense coming into consideration in this comparison	£7	9	9	£38	0	5

Similar calculations could be made for every process and for every country, and therefrom we can see what expense will be incurred in the execution of the various modifications of the methods for white sugar manufacture; and, if likewise, the amortization and interest and the wear and tear of the machinery are taken into account, a reliable survey can be obtained of the financial demands of the several methods.

On the other hand, we have not only to take into account the quality of the white sugar, both at the moment of its being made and also after having been stored for a long time, but also the probable yield of sugar by the method used and the reliability with which it works.

It is evident that every process in which the colouring matter is removed either by carbonatation or by animal charcoal and carbonaceous powders, or by filtration in some way or other, is far more reliable and will offer a better prospect of sugar which keeps its quality, than processes in which the colouring matter is only bleached and may return to some extent some time under certain conditions. Processes in which all the juice is filtered give more certainty of obtaining a juice and a syrup therefrom, in which no coloured particles will impart a grey colour to the sugar crystals, than processes in which the juice is only decanted from a subsided layer of dirt, which might inadvertently be stirred up and contaminate the clarified juice.

These advantages or disadvantages are not capable of exact estimation. No fixed data are obtainable respecting them, and the individual tastes of the manufacturer count here greatly in the choice of a system.

But apart from the different merits as to *quality*, which have been extensively treated in the foregoing chapters, we can only obtain full information as to the *quantity* of sugar yielded by a given amount of juice, after its treatment by the different systems.

Supposing we start from a given quantity of juice of a certain analysis and supposing we obtain at the end of manufacture a white sugar of exactly the same polarization, apart from the shade of colour and the brightness of the crystals, then we have solely to consider the factors of the loss on filtration, the loss by inversion and by entrainment, and the loss in the molasses, since all the other losses are the same in every system, and properly speaking the loss by entrainment might perhaps be eliminated also as there is little chance of a juice clarified by one method frothing more than in another, save in the foaming in the first carbonatation tanks where some juice might be driven out of the chimney.

Van der Want[1] calculated the losses sustained in Java factories where white sugar was made, and extended his calculations over a great number of factories working with the sulphitation of the raw juice and with the carbonatation process. The results are tabulated as follows, all figures being calculated on 100 parts of sucrose entered in the raw juice.

	Sulphitation.	Carbonatation (de Haan's method).
Clarification of the raw juice...	1·35	1·414
Evaporation	0·810	0·675
Clarification of the syrup ...	0·014	0·064
Molasses	8·03	8·71
Total loss on manufacture ...	10·204	10·863

There is therefore a difference to the advantage of the carbonatation process of about 0·66 parts on 100 of sucrose entered into the sugar-house, and it has to be considered whether this quantity is large enough to justify the establishment of a carbonatation plant, provided that all the other factors remain the same.

We have already seen that the loss in scums does not differ considerably; in the sulphitation process we have only a little scum, but of a high sugar content, while in the carbonatation process we have a large amount, which however is of a nature to be sweetened off till a low sugar content is reached, the total loss thereby coming out at the same net figure.

The larger losses of sugar during evaporation and on clarification of the syrup may find an explanation from the fact that usually the sulphited juice is of a stronger acid reaction than the carbonatated one, and therefore may have become inverted during evaporation and especially during the long stay at high temperature in the syrup subsiding tanks, while the carbonatation syrup is quickly filtered and boiled.

The loss in molasses in the defecation process is certainly somewhat larger than in the carbonatation one and, in short, than in all processes where the juice is filtered. The slimy matter which remains in the juices and syrups which are only decanted off exerts its detrimental influence during the whole manufacturing process, and especially in the crystallization of the last products. As a rule, a sticky and gummy molasses cannot be so thoroughly desaccharified as a clear one, and this accounts for the larger loss of sucrose in the molasses of defecated juices of the same quality as carbonatated ones.

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[1] *Archief voor de Suikerindustrie in Nederlandsch-Indië*, 1914, 1084.

Most of the processes described in the earlier chapters have not been sufficiently long in practical operation to permit us to give reliable figures regarding the output of white sugar from the juice. The Norit, Battelle, Phospho-gelose and other processes are only in their infancy, so that no reliable data can be gathered at so early a stage as the present, and we therefore confine ourselves to the figures given by van der Want in his above quoted article, where he compares the yield of a given quality of white sugar from juice of the same analysis treated in practical working by four methods in factories where the processes under consideration were in regular use. Taking the amount obtained by the common sulphitation process as 100, the outputs by the others are quoted as percentages of that amount.

Yield by the usual sulphitation process ...	100·00
,, double carbonatation	100·32
,, de Haan's modification	100·64
,, Bach's process	100·96

All these figures have relation to data on white sugar only, as they serve for the comparison of the merits of the different processes for the manufacture of that article, but it is perhaps not without interest to complete the modes of comparison by giving a very simple method for the conversion of one assortment of sugar into another, in order to enable the manufacturer to compare the yield of white sugar to be expected when going in for white sugar manufacture with that of the raw sugar to which he has hitherto been accustomed.

It is evident that even if the losses in scums, by inversion, by entrainment, and so on, are absolutely the same in both processes, and the molasses is exhausted to exactly the same real purity, yet the yield of the product in a white sugar factory will be smaller than that in a factory where raw sugar is turned out, for the simple reason that where white sugar only consists of pure crystallized sucrose, these crystals of raw sugar are coated with a layer of adhering molasses. Even if in both cases exactly the same weight of sucrose had crystallized out and had been obtained, the weight of the product in the case of the white sugar is lower than in that of the raw sugar, because of that layer of molasses. It results from this observation that the relation between the two products could not be established by the simple comparison of the polarization figures of the product, as in the case of raw sugar part of the polarization is produced by the sucrose in the molasses, which does not belong to that part of the product taken into account here.

In order to obtain a trustworthy comparison between the quantities of raw and of white sugar turned out by the different processes, we must only consider the amount of real crystallizable sucrose present in the product.

For the white sugar, which is washed quite free from molasses, this question is easily solved by assuming the amount of crystallizable sugar to be equal to its polarization.

Raw sugars may be considered to consist of a mixture of crystallizable sucrose and molasses having a purity of 33⅓. We know that in reality the molasses covering the crystals in raw sugar is of a higher purity, but in that case the molasses could be desaccharified in a refinery, and it is not our aim here to determine the actual crystallized sugar in the product or its crystal content, but only the maximum percentage of crystallizable sucrose there.

The reason why we take the quotient of purity at the figure of 33⅓ is because that figure is about the lowest on record, and further because of the simplification of calculations since by using it the ratio between sugar and non-sugar is exactly 1:2. When we want to find the amount of crystallizable sucrose in a given product we have only to subtract half the difference between the dry substance and the polarization from the polarization figure to find what we are looking for.

If, for instance, a raw sugar has a polarization of 96·2° and a moisture content of 0·72 so that the dry substance is 99·28, then the content of non-sugar is 99·28−96·2=3·08. These 3·08 parts of non-sugar immobilize half their weight or 1·54 parts of sucrose, and prevent it from crystallizing out; and therefore the amount of sugar capable of crystallization is 96·2−1·54 = 94·66.

If therefore we are accustomed to make 10·0 per cent. of 96·2° sugar from our cane, we cannot expect more crystallizable sugar therein than 10·00 × 0·947 = 9·47 per cent., and for that reason we cannot expect more than 9·47 per cent. of white sugar instead of the 10·00 of raws, even if we do not suffer additional loss during the much more delicate work.

Although the reckoning is very easily made, we give here a table in which the crystal contents of sugars can be found if their moisture content and their polarization are known, taking the minimum purity of the molasses to be 33⅓.

If we have a sugar polarizing 97·4° with a moisture content of 0·7, we look up in the horizontal line 97·4, in the vertical one 0·7, and we find at the intersection 96·45, which is the amount of crystallizable sucrose in 100 parts of that raw sugar.

We are no sticklers as to that figure of 33⅓; if it be thought too low, anyone is free to employ another, but in that case a new table will have to be composed with the higher figure as a basis.

Table for the Percentages of Crystallizable Sucrose Polarization and from 0 to 2 per cent. of Moisture, exhausted to 33⅓ degrees

Polarization	Moisture Content.										
	0·0	0·1	0·2	0·3	0·4	0·5	0·6	0·7	0·8	0·9	1·0
99·9	99·85	99·90	..	..	..	..	..	..	..	..	..
99·8	99·70	99·75	99·80	..	..	..	..	..	..	..	..
99·7	99·55	99·60	99·65	99·70	..	..	..	..	..	..	..
99·6	99·40	99·45	99·50	99·55	99·60	..	..	..	..	..	..
99·5	99·25	99·30	99·35	99·40	99·45	99·50	..	..	..	..	..
99·4	99·10	99·15	99·20	99·25	99·30	99·35	99·40	..	..	..	..
99·3	98·95	99·00	98·05	99·10	99·15	99·20	99·25	99·30	..	..	..
99·2	98·80	98·85	98·90	98·95	99·00	99·05	99·10	99·15	99·20	..	..
99·1	98·65	98·70	98·75	98·80	98·85	98·90	98·95	99·00	99·05	99·10	..
99·0	98·50	98·55	98·60	98·65	98·70	98·75	98·80	98·85	98·90	98·95	99·00
98·9	98·35	98·40	98·45	98·50	98·55	98·60	98·65	98·70	98·75	98·80	98·85
98·8	98·20	98·25	98·30	98·35	98·40	98·45	98·50	98·55	98·60	98·65	98·70
98·7	98·05	98·10	98·15	98·20	98·25	98·30	98·35	98·40	98·45	98·50	98·55
98·6	97·90	97·95	98·00	98·05	98·10	98·15	98·20	98·25	98·30	98·35	98·40
98·5	97·75	97·80	97·85	97·90	97·95	98·00	98·05	98·10	98·15	98·20	98·25
98·4	97·60	97·65	97·70	97·75	97·80	97·85	97·90	97·95	98·00	98·05	98·10
98·3	97·45	97·50	97·55	97·60	97·65	97·70	97·75	97·80	97·85	97·90	97·95
98·2	97·30	97·35	97·40	97·45	97·50	97·55	97·60	97·65	97·70	97·75	97·80
98·1	97·15	97·20	97·25	97·30	97·35	97·40	97·45	97·50	97·55	97·60	97·65
98·0	97·00	97·05	97·10	97·15	97·20	97·25	97·30	97·35	97·40	97·45	97·50
97·9	96·85	96·90	96·95	97·00	97·05	97·10	97·15	97·20	97·25	97·30	97·35
97·8	96·70	96·75	96·80	96·85	96·90	96·95	97·00	97·05	97·10	97·15	97·20
97·7	96·55	96·60	96·65	96·70	96·75	96·80	96·85	96·90	96·95	97·00	97·05
97·6	96·40	96·45	96·50	96·55	96·60	96·65	96·70	96·75	96·80	96·85	96·90
97·5	96·25	96·30	96·35	96·40	96·45	96·50	96·55	96·60	96·65	96·70	96·75
97·4	96·10	96·15	96·20	96·25	96·30	96·35	96·40	96·45	96·50	96·55	96·60
97·3	95·95	96·00	96·05	96·10	96·15	96·20	96·25	96.30	96·35	96·40	96·45
97·2	95·80	95·85	95·90	95·95	96·00	96·05	96·10	96·15	96·20	96·25	96·30
97·1	95·65	95·70	95·75	95·80	95·85	95·90	95·95	96·00	96·05	96·10	96·15
97·0	95·50	95·55	95·60	95·65	95·70	95·75	95·80	95·85	95·90	95·95	96·00

contained in Raw Sugar from 99·9 to 97 per cent. supposing the Molasses around the Crystals to be Quotient of Purity.

Polarization	Moisture Content.									
	1·1	1·2	1·3	1·4	1·5	1·6	1·7	1·8	1·9	2·0
99·9	..	..	..	..	..	..	..	..	..	..
99·8	..	..	..	..	..	..	..	..	..	..
99·7	..	..	..	..	..	..	..	..	..	..
99·6	..	..	..	..	..	..	..	..	..	..
99·5	..	..	..	..	..	..	..	..	..	..
99·4	..	..	..	..	..	..	..	..	..	..
99·3	..	..	..	..	..	..	..	..	..	..
99·2	..	..	..	..	..	..	..	..	..	..
99·1	..	..	..	..	..	..	..	..	..	..
99·0	..	..	..	..	..	..	..	..	..	..
98·9	98·90	..	..	..	..	..	..	..	..	..
98·8	98·75	98·80	..	..	..	..	..	..	..	..
98·7	98·60	98·65	98·70	..	..	..	..	..	..	..
98·6	98·45	98·50	98·55	98·60	..	..	..	..	..	..
98·5	98·30	98·35	98·40	98·45	98·50	..	..	..	..	..
98·4	98·15	98·20	98·25	98·30	98·35	98·40	..	..	..	..
98·3	98·00	98·05	98·10	98·15	98·20	98·25	98·30	..	..	..
98·2	97·85	97·90	97·95	98·00	98·05	98·10	98·15	98·20	..	..
98·1	97·70	97·75	97·80	97·85	97·90	97·95	98·00	98·05	98·10	..
98·0	97·55	97·60	97·65	97·70	97·75	97·80	97·85	97·90	97·95	98·00
97·9	97·40	97·45	97·50	97·55	97·60	97·65	97·70	97·75	97·80	97·85
97·8	97·25	97·30	97·35	97·40	97·45	97·50	97·55	97·60	97·65	97·70
97·7	97·10	97·15	97·20	97·25	97·30	97·35	97·40	97·45	97·50	97·55
97·6	96·95	97·00	97·05	97·10	97·15	97·20	97·25	97·30	97·35	97·40
97·5	96·80	96·85	96·90	96·95	97·00	97·05	97·10	97·15	97·20	97·25
97·4	96·65	96·70	96·75	96·80	96·85	96·90	96·95	97·00	97·05	97·10
97·3	96·50	96·55	96·60	96·65	96·70	96·75	96·80	96·85	96·90	96·95
97·2	96·35	96·40	96·45	96·50	96·55	96·60	96·65	96·70	96·75	96·80
97·1	96·20	96·25	96·30	96·35	96·40	96·45	96·50	96·55	96·60	96·65
97·0	96·05	96·10	96·15	96·20	96·25	96·30	96·35	96·40	96·45	96·50

TABLE FOR THE PERCENTAGES OF CRYSTALLIZABLE SUCROSE POLARIZATION AND FROM 0 TO 2 PER CENT. OF MOISTURE, EXHAUSTED TO 33⅓ DEGREES

Polarization	Moisture Content.										
	0·0	0·1	0·2	0·3	0·4	0·5	0·6	0·7	0·8	0·9	1·0
96·9	95·35	95·40	95·45	95·50	95·55	95·60	95·65	95·70	95·75	95·80	95·85
96·8	95·20	95·25	95·30	95·35	95·40	95·45	95·50	95·55	95·60	95·65	95·70
96·7	95·05	95·10	95·15	95·20	95·25	95·30	95·35	95·40	95·45	95·50	95·55
96·6	..	..	95·00	95·05	95·10	95·15	95·20	95·25	95·30	95·35	95·40
96·5	..	..	..	..	94·95	95·00	95·05	95·10	95·15	95·20	95·25
96·4	..	..	..	..	94·80	94·85	94·90	94·95	95·00	95·05	95·10
96·3	..	..	..	..	94·65	94·70	94·75	94·80	94·85	94·90	94·95
96·2	..	..	..	..	..	94·55	94·60	94·65	94·70	94·75	94·80
96·1	..	..	..	..	..	94·40	94·45	94·50	94·55	94·60	94·65
96·0	..	..	..	..	..	94·25	94·30	94·35	94·40	94·45	94·50
95·9	..	..	..	..	..	94·10	94·15	94·20	94·25	94·30	94·35
95·8	..	..	..	..	..	93·95	94·00	94·05	94·10	94·15	94·20
95·7	..	..	..	..	..	93·80	93·85	93·90	93·95	94·00	94·05
95·6	..	..	..	..	..	93·65	93·70	93·75	93·80	93·85	93·90
95·5	..	..	..	..	..	..	93·55	93·60	93·65	93·70	93·75
95·4	..	..	..	..	..	..	93·40	93·45	93·50	93·55	93·60
95·3	..	..	..	..	..	..	93·25	93·30	93·35	93·40	93·45
95·2	..	..	..	..	..	..	93·10	93·15	93·20	93·25	93·30
95·1	..	..	..	..	..	..	..	93·00	93·05	93·10	93·15
95·0	..	..	..	..	..	..	..	92·85	92·90	92·95	93·00
94·9	..	..	..	..	..	..	..	..	92·75	92·80	92·85
94·8	..	..	..	..	..	..	..	..	92·60	92·65	92·70
94·7	..	..	..	..	..	..	..	..	..	92·50	92·55
94·6	..	..	..	..	..	..	..	..	..	92·35	92·40
94·5	..	..	..	..	..	..	..	..	..	..	92·25
94·4	..	..	..	..	..	..	..	..	..	..	92·10
94·3	..	..	..	..	..	..	..	..	..	..	..
94·2	..	..	..	..	..	..	..	..	..	..	..
94·1	..	..	..	..	..	..	..	..	..	..	..
94·0	..	..	..	..	..	..	..	..	..	..	..

CONTAINED IN RAW SUGAR FROM 96·9 TO 94 PER CENT. SUPPOSING THE MOLASSES AROUND THE CRYSTALS TO BE QUOTIENT OF PURITY.

Polarization	Moisture Content.									
	1·1	1·2	1·3	1·4	1·5	1·6	1·7	1·8	1·9	2·0
96·9	95·90	95·95	96·00	96·05	96·10	96·15	96·20	96·25	96·30	96·35
96·8	95·75	95·80	95·85	95·90	95·95	96·00	96·05	96·10	96·15	96·20
96·7	95·60	95·65	95·70	95·75	95·80	95·85	95·90	95·95	96·00	96·05
96·6	95·45	95·50	95·55	95·60	95·65	95·70	95·75	95·80	95·85	95·90
96·5	95·30	95·35	95·40	95·45	95·50	95·55	95·60	95·65	95·70	95·75
96·4	95·15	95·20	95·25	95·30	95·35	95·40	95·45	95·50	95·55	95·60
96·3	95·00	95·05	95·10	95·15	95·20	95·25	95·30	95·35	95·40	95·45
96·2	94·85	94·90	94·95	95·00	95·05	95·10	95·15	95·20	95·25	95·30
96·1	94·70	94·75	94·80	94·85	94·90	94·95	95·00	95·05	95·10	95·15
96·0	94·55	94·60	94·65	94·70	94·75	94·80	94·85	94·90	94·95	95·00
95·9	94·40	94·45	94·50	94·55	94·60	94·65	94·70	94·75	94·80	94·85
95·8	94·25	94·30	94·35	94·40	94·45	94·50	94·55	94·60	94·65	94·70
95·7	94·10	94·15	94·20	94·25	94·30	94·35	94·40	94·45	94·50	94·55
95·6	93·95	94·00	94·05	94·10	94·15	94·20	94·25	94·30	94·35	94·40
95·5	93·80	93·85	93·90	93·95	94·00	94·05	94·10	94·15	94·20	94·25
95·4	93·65	93·70	93·75	93·80	93·85	93·90	93·95	94·00	94·05	94·10
95·3	93·50	93·55	93·60	93·65	93·70	93·75	93·80	93·85	93·90	93·95
95·2	93·35	93·40	93·45	93·50	93·55	93·60	93·65	93·70	93·75	93·80
95·1	93·20	93·25	93·30	93·35	93·40	93·45	93·50	93·55	93·60	93·65
95·0	93·05	93·10	93·15	93·20	93·25	93·30	93·35	93·40	93·45	93·50
94·9	92·90	92·95	93·00	93·05	93·10	93·15	93·20	93·25	93·30	93·35
94·8	92·75	92·80	92·85	92·90	92·95	93·00	93·05	93·10	93·15	93·20
94·7	92·60	92·65	92·70	92·75	92·80	92·85	92·90	92·95	93·00	93·05
94·6	92·45	92·50	92·55	92·60	92·65	92·70	92·75	92·80	92·85	92·90
94·5	92·30	92·35	92·40	92·45	92·50	92·55	92·60	92·65	92·70	92·75
94·4	92·15	92·20	92·25	92·30	92·35	92·40	92·45	92·50	92·55	92·60
94·3	92·00	92·05	92·10	92·15	92·20	92·25	92·30	92·35	92·40	92·45
94·2	91·85	91·90	91·95	92·00	92·05	92·10	92·15	92·20	92·25	92·30
94·1	..	..	91·80	91·85	91·90	91·95	92·00	92·05	92·10	92·15
94·0	..	..	91·65	91·70	91·75	91·80	91·85	91·90	91·95	92·00

PART II.

BOILING, CURING AND FINISHING WHITE SUGAR.

CHAPTER I.

BOILING AND COOLING.

After the juice has reached the stage of clarified syrup the further treatment for converting the saccharine liquid into white sugar is always the same whatever the method of clarification followed to reach this stage may have been.

For this reason the methods for turning the syrup into white sugar will start from syrup only, without further reference to the clarification process to which it has previously been submitted.

As a general rule boiling in white sugar manufacture should be done exclusively to grain, even with the after-products; string-proof boiling should entirely be discarded and if the purity of first or second molasses is too low to build up grain from them, they should be returned to a pan where a pied-de-cuite of sugar, or first massecuite, is already present, so as to be desaccharified by the existing crystals to the desired point. Not only does boiling to grain produce a better shaped and more easily curable crystal and give better yields for that reason, but string-proof boiling requires a tedious cooling in crystallization tanks, where owing to the acid reaction, there is much danger of inversion and loss of sucrose.

We saw in the Introduction that one of the foremost requirements in the manufacture of white sugar is the rapidity and the uninterrupted run of all the manipulations, and this same maxim should guide us too in the boiling, cooling and crystallization of the after-products.

White sugar destined for direct consumption should not only be white and brilliant, but should also possess a regular form and a rather large size; this latter desideratum makes it preferable to start the building up of the grain from a well-developed seed. In order to obtain this without too much waste of time and of pan capacity, it is advisable to have all the pans connected with one another by large pipes, provided with valves which permit the contents of one pan to be drawn over into another, while one pan or all of them should have a wide pipe through which a magma of sugar crystals and syrup may be sucked in from outside in order to serve as "seed."

Not only does the arrangement of making the "seed" in one pan and distributing it over the others save a great deal of time, as the formation of the grain is a slow operation bristling with difficulties, but it also permits the use of calandria pans for white sugar making, which would not be feasible if the grain had to be formed each time afresh.

If coarse crystals are to be made, which is the case in the manufacture of granulated plantation whites, the graining must be done "low down," in order to give the crystals time and space to grow to the required size. Graining low down cannot be done in a calandria pan, unless special arrangements are made, *e.g.*, the calandria could be constructed in two pieces with an empty space between them, but a common calandria occupies so much room and reaches so high, that a rather large amount of syrup must be in the pan when steam is admitted to the calandria, to prevent the hot surface from protruding above the level and charring the drops of syrup drying there.

In a coil pan this difficulty is overcome by only admitting the steam in those coils which are covered by the syrup, and then one is able to grain as low down as desired, by only admitting the steam in the lowest coils and leaving the others cold.

It is clear that owing to this procedure the greater part of the heating surface remains unemployed and that both time and pan capacity are wasted; but if the grain is made in a special pan and has attained a certain size, the whole mixture may be drawn over into the vacuum pan, coil or calandria pan, until the whole heating system is submerged. This can then come into action with its full capacity, and thereby the whole power of the pan at its best is utilized.

When boiling last-product massecuites, which are strongly concentrated, the natural circulation occasioned by the boiling is not strong enough to ensure the proper admixture of the pan's contents, and hence the pan in which the after-product massecuites are boiled should be provided with a stirring apparatus. In most cases this consists of a screw revolving in a wide central tube open at both ends. The stiff mass is raised at the centre and induced to flow downwards at the sides while the empty space in the centre is filled by the adjacent mass from the sides, thus creating a slow movement of the contents of the pan.

This circulation is most necessary for several reasons. First, the massecuite remaining stationary at the same place may cake against the coils, be locally overheated, and become coloured by the too elevated temperature. A second reason is that the crystals in the stiff mass are not free to move, and thereby cannot grow regularly, which is one of the first requirements for a good white sugar for direct consumption. Finally the lack of circulation may give rise to the circumstance that when molasses is returned into the massecuite, as is always the case when after-products are boiled to grain, large portions of the former do not mix with the latter whereby the desired effect of the desaccharification process is totally lost, the result being that a final molasses of too high a quotient of purity is discarded by the factory. For these and yet other reasons the circulation in the vacuum pan should be artificially promoted, for which purpose a stirrer as described above will render good service.

Pans in which first massecuites are to be boiled, and in which the fluidity of the mass is maintained throughout the whole process, do not need a stirrer ; only those for last massecuites require one, but in order to be free to choose whatever pan one likes for the after-products and not be hampered in one's movements when wanting to use also another pan than the one usually reserved for after-products, it is safe to have every pan provided with the stirring apparatus which may be used or left idle at will.

Instead of a screw revolving in the pan, circulation is sometimes promoted by a perforated copper coil at the bottom of the pan, through which dry low-pressure steam is blown into the boiling mass. By this operation the vacuum is not perceptibly lowered and the massecuite is kept in gentle movement. The perforations of the coil should be made on its underside in order to prevent them being choked with crystals when the pan is discharged. Another good device for promoting circulation is to introduce the syrup and molasses through a bent pipe extending nearly to the bottom of the pan, so that the thin liquids are compelled to force their way upwards through the massecuite and so become thoroughly mixed with it, which is not the case if the molasses is introduced at the top of the massecuite upon syrup already there. The same end is sometimes gained by drawing in the syrup and molasses through a perforated coil at the bottom of the pan. To prevent choking, this coil is likewise perforated on its underside.

These last devices will render good service in mixing the molasses with the massecuite from syrup, but they are useless after the mixture has already been performed and the mixed massecuite has to be concentrated. That is then where the stirring apparatus comes in, and keeps the contents of the pan in motion till the end of the concentration.

In many processes for the making of white sugar the syrups and molasses to be concentrated have rather a high acidity, part of which is volatile, so that they give off acid vapours which may attack the ironwork of the pan, the dome, the piping, the save-all, the condenser, etc. It has been suggested to coat the inside of the iron pan with thin copper-plates but this measure does not appear to have found much encouragement, at least beyond a few remarks as to its introduction here and there, after which nothing more has been heard of the proposal. The method most generally in use is to protect the inside of all those iron parts exposed to the action of the acid vapours by means of a very adhesive coating of heat-and-acid-proof varnish or paint, and to make all bottoms inclined so as to prevent every accumulation of acid condensation-water in the save-alls and tubes.

Most of the massecuites to be made will consist of mixtures of a primary massecuite from syrup and of molasses having a certain quotient of purity, which both together have to make up the massecuite wanted. The quantities of the two constituents are determined by their purities, and that of the mixed massecuite desired, using a very simple calculation, so that the percentage of the primary massecuite which should be in the pan before the molasses is added is estimated beforehand. In order to facilitate the work, a scale should be painted on the outside of the pan, showing percentages of the contents, so that the pan-boiler can know exactly how far he has to fill his pan with the primary massecuite to be sure of a final massecuite of good composition suitable for the use for which it is intended.

The density of the syrup is a matter of great consequence in the white sugar process. We saw (on page 51) that the density of syrup on subsidence and filtration had to oscillate between 26° and 28° Bé., measured hot, and this was also the best density for obtaining large and well-shaped crystals. A too low density is unnecessary and retards the boiling process, while at higher densities the crystals will not grow regularly. There is always a great danger of formation of false grain by using a too highly concentrated syrup.

The molasses to be added afterwards to the primary massecuite do not part so easily with their sugar content, thereby offering a far smaller opportunity for the crystallization of fine sugar crystals; and therefore these may be drawn into the pan with a higher density, even up to 38° Bé., measured hot.

Athough the density of the molasses is rather immaterial, its temperature at all events should be at least equal and preferably superior to that of the massecuite in the pan, for, if it does not itself easily drop sugar crystals, it might cause the liquid in the pan to do so. By its lower temperature it decreases the temperature of the mother-liquor in the pan so far that it could no longer keep all its dissolved sugar in solution, in which case the introduction of cold molasses might cause formation of false grain. As we said, this may be totally prevented by keeping the molasses warm before it is drawn into the massecuite in the vacuum pan.

The methods of boiling for white sugar do not differ from those in use in ordinary sugar manufacture, and therefore we shall abstain here from dwelling on that subject, which is extensively treated on in standard works on sugar manufacture.[1]

In Chapter III will be found a couple of schemes according to which we may go to work, in order to obtain all the sugar capable of crystallizing out, either in the shape of white granulated first-product, or divided into a percentage of first granulated and soft white seconds, or as first white granulated and dark brown after-product. The market conditions may lead to the final decision which one may make; every one of them may be followed, but it is strictly necessary to boil all the massecuites to grain, even the last ones, and in this case a pied-de-cuite from first massecuite from syrup, or from a magma of after-product sugar and syrup, should be used as " seed " while the desaccharification of the molasses is performed by boiling this along with the already existing grain.

In order to be sure of obtaining exactly that quality of massecuite and of molasses one would expect from the system selected, we must carefully mix our massecuite from syrup with the clarified molasses so as to obtain a mixed massecuite of a known quotient of purity; for if this is not obtained the scheme has to be altered and we are not at all certain of obtaining a thoroughly exhausted molasses.

The following list of purity quotients of mixed massecuites and the corresponding values of the molasses spun off from them without cooling

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[1] Cf. H. C. Prinsen Geerligs, "Cane Sugar and its Manufacture."

may be a reliable guide in our calculations, in so far that if we require a molasses of 60° purity we have to make the foregoing massecuite to have one of 80° and to spin that hot. This list only relates to uncooled massecuites, and the purities are the apparent values, found by dividing 100 × polarization by the degrees Brix.

Massecuite of	90°	purity yields	a molasses of	75°	quotient.
,,	88°	,,	,,	71°	,,
,,	85°	,,	,,	65°	,,
,,	82°	,,	,,	62°	,,
,,	80°	,,	,,	60°	,,
,,	70°	,,	,,	50°	,,
,,	65°	,,	,,	45°	,,

If the mixed massecuite of 60° purity is cooled in motion to a temperature of 45° C, the purity of the molasses will come down to about 30°, and then it will be exhausted.

We know at the commencement of a boiling the quotient of purity of the syrup, and also that of the molasses to be drawn in ultimately, and from these figures we can find by a simple equation the percentage of massecuite from syrup and from molasses to form a panful of massecuite of the desired quotient.

Suppose that we have a syrup of 85° purity and a molasses of 65°; that we want to make a second massecuite of 75°; and that we get this by mixing x parts of the syrup massecuite with $100 - x$ parts of the molasses massecuite. We then have the following equation :—

$$85\,x + 65\,(100 - x) = 100 \times 75$$
$$85\,x - 65\,x = 7500 - 6500$$
$$20\,x = 1000$$
$$x = 50$$

This means that the pan has to be filled half full with syrup massecuite before drawing in the molasses.

These proportions have, properly speaking, only a bearing on the dry substance of the different materials, and in case of these having a different water content, they have also to be taken into account. Here this is not necessary, since the mixed massecuites have, of course, ultimately the same water content, and therefore the relationship remains the same. It is a good thing to have tables made showing the relation of syrup massecuite for every degree of purity of syrup and of molasses for the different massecuites entering in the scheme.

If, for instance, the scheme is to have one first massecuite of 85°, one second of 70°, and one final of 60°, we use the three tables on pages 87-90, while when other standard purity values are taken as a basis of the scheme, similar tables may be calculated and given to the pan-boiler in order to guide him in his work. The figures in the tables are calculated to the nearest unit from the formula given above.

After the necessary amount of syrup has been boiled to a massecuite, the molasses is gradually drawn in and boiling continued to evaporate the surplus water and allow the sugar crystallizing out by the evaporation to adhere to the already formed grains, which increase in size and, owing to the good circulation maintained in the boiling mass, grow regularly and evenly, so that at the last the large and well-developed sugar crystals are evenly divided in a clear molasses, coating and surrounding them evenly without hard lumps or crystals caked together by desiccated molasses.

When finishing the boiling, those massecuites from which molasses is expected, which has still to give up more of its sugar in a subsequent treatment, are boiled less closely than those from which only exhausted molasses is expected. The Brix[1] of the first category should be about 93 or 94, while that of the latter kind may be concentrated much farther and go up to 96 or 97. The reason is that if the massecuites give up molasses which is not exhausted and is to be drawn into other boilings to be totally desaccharified, the proper degree of purity can much more easily be obtained by the exact mixing of syrup massecuite and molasses than by concentrating to the utmost, and if we want to have a molasses of a certain purity this can much more economically be obtained by using another proportion between the two constituents than by forcing more sugar to crystallize out. As a more openly boiled massecuite is ready sooner, runs faster out of the pan, and offers less trouble in transporting and curing, it is better to boil those massecuites open, the molasses of which is later desaccharified. The last massecuites, however, from which molasses to be discarded from the factory is expected, and which therefore should be as far exhausted as it possibly can, are highly concentrated in order to induce all sugar, which can eventually crystallize out, to do so. The trouble encountered in transportation, cooling and curing must be taken into the bargain, but when acting as prescribed, it is only part of the massecuites which causes the extra trouble, while the rest is worked off smoothly.

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[1] The degree Brix must not to be confounded with the actual dry substance, which is considerably less.

PURITY OF THE MIXED MASSECUITE = 60.

Purity of the Molasses.	Purity of the Syrup.																								Purity of the Molasses.
	70	71	72	73	74	75	76	77	78	79	80	81	82	83	84	85	86	87	88	89	90	91	92	93	
40	67	65	63	61	59	57	56	54	53	51	50	49	48	47	45	44	43	43	42	41	40	39	38	38	40
41	66	63	61	59	58	56	54	53	51	50	49	48	46	45	44	43	42	41	40	40	39	38	37	36	41
42	64	62	60	58	56	55	53	51	50	49	47	46	45	44	43	42	41	40	39	38	37	37	36	35	42
43	63	61	59	57	55	53	52	50	49	47	46	45	44	43	41	40	40	39	38	37	36	35	35	34	43
44	62	59	57	55	53	52	50	48	47	46	44	43	42	41	40	39	38	37	36	36	35	34	33	33	44
45	60	58	56	54	52	50	48	47	45	44	43	42	41	39	38	38	37	36	35	34	33	33	32	31	45
46	58	56	54	52	50	48	47	45	44	42	41	40	39	38	37	36	35	34	33	33	32	31	30	30	46
47	57	54	52	50	48	46	45	43	42	41	39	38	37	36	35	34	33	33	32	31	30	30	29	28	47
48	55	52	50	48	46	44	43	41	40	39	37	36	35	34	33	32	32	31	30	29	29	28	27	27	48
49	52	50	48	46	44	42	41	39	38	37	35	34	33	32	31	31	30	29	28	28	27	26	26	25	49
50	50	48	45	43	42	40	38	37	36	34	33	32	31	30	29	29	28	27	26	26	25	..	..	..	50
51	47	45	43	41	39	38	36	35	33	32	31	30	29	28	27	26	26	25	..	..	..	..	..	..	51
52	44	42	40	38	36	35	33	32	31	30	29	28	27	26	25	..	..	..	..	..	..	..	..	..	52
53	41	39	37	35	33	32	30	29	28	27	26	25	..	..	..	..	..	..	..	..	..	..	..	..	53
54	38	35	33	32	30	29	27	26	25	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..	54
55	33	31	29	28	26	25	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..	55
56	29	27	25	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..	56

Purity of the Molasses.	Purity of the Syrup.											
	71	72	73	74	75	76	77	78	79	80	81	82
40	97	94	91	88	86	83	81	79	77	75	73	71
41	97	94	91	88	85	83	81	78	76	74	72	71
42	97	93	90	88	85	82	80	78	76	74	72	70
43	96	93	90	87	84	82	79	77	75	73	71	69
44	96	93	90	87	84	81	79	76	74	72	70	68
45	96	93	89	86	83	81	78	76	74	71	69	68
46	96	92	89	86	83	80	77	75	73	71	69	67
47	96	92	88	85	82	79	77	74	72	70	68	66
48	96	92	88	85	81	79	76	73	71	69	67	65
49	95	91	88	84	81	78	75	72	70	68	66	64
50	95	91	87	83	80	77	74	71	69	67	65	63
51	95	90	86	83	79	76	73	70	68	66	63	61
52	95	90	86	82	78	75	72	69	67	64	62	60
53	94	89	85	81	77	74	71	68	65	63	61	59
54	94	89	84	80	76	73	70	67	64	62	59	57
55	94	88	83	79	75	71	68	65	63	60	58	56
56	93	87	82	78	74	70	67	64	61	58	56	54
57	93	87	81	76	72	68	65	62	59	57	54	52
58	92	86	80	75	71	67	63	60	57	55	52	50
59	92	85	79	73	69	65	61	58	55	52	50	48
60	91	83	77	71	67	62	59	56	53	50	48	45
61	90	82	75	69	64	60	56	53	50	47	45	43
62	89	80	73	67	62	57	53	50	47	44	42	40
63	87	78	70	64	58	54	50	47	44	41	39	37
64	86	75	67	60	55	50	46	43	40	38	35	33
65	83	71	62	56	50	45	42	38	36	33	31	29
66	80	67	57	50	44	40	36	33	31	29	27	25
67	75	60	50	43	37	33	30	27	25	..	..	..
68	67	50	40	33	27	25	..	..	..	..	..	..
69	50	33	25	..	..	..	..	..	..	..	..	..

MIXED MASSECUITE = 70.

Purity of the Syrup.											Purity of the Molasses.
83	84	85	86	87	88	89	90	91	92	93	
70	68	67	65	64	63	61	60	59	58	57	40
69	67	66	64	63	62	60	59	58	57	56	41
68	67	65	64	62	61	60	58	57	56	55	42
68	66	64	63	61	60	59	57	56	55	54	43
67	65	63	62	60	59	58	57	55	54	53	44
66	64	63	61	60	58	57	56	54	53	52	45
65	63	62	60	59	57	56	55	53	52	51	46
64	62	61	59	58	56	55	53	52	51	50	47
63	61	59	58	56	55	54	52	51	50	49	48
62	60	58	57	55	54	53	51	50	49	48	49
61	59	57	56	54	53	51	50	49	48	47	50
59	58	56	54	53	51	50	49	48	46	45	51
58	56	55	53	51	50	49	47	46	45	44	52
57	55	53	52	50	49	47	46	45	44	43	53
55	53	52	50	48	47	46	44	43	42	41	54
54	52	50	48	47	45	44	43	42	41	39	55
52	50	48	47	45	44	42	41	40	39	38	56
50	48	46	45	43	42	41	39	38	37	86	57
48	46	44	43	41	40	39	37	36	35	34	58
46	44	42	41	39	38	37	35	34	33	32	59
43	42	40	38	37	36	34	33	32	31	30	60
41	39	38	36	35	33	32	31	30	29	28	61
38	36	35	33	32	31	30	29	28	27	26	62
35	33	32	30	29	28	27	26	25	..	..	63
32	30	29	27	26	25	..	..	..	..	..	64
28	26	25	..	..	..	..	..	..	..	..	65
..	..	..	..	..	..	..	..	..	..	..	66
..	..	..	..	..	..	..	..	..	..	..	67
..	..	..	..	..	..	..	..	..	..	..	68
..	..	..	..	..	..	..	..	..	..	..	69

Purity of the Mixed Massecuite = 85.

Purity of the Molasses.	Purity of the Syrup.							
	86	87	88	89	90	91	92	93
40	98	96	94	92	90	88	87	85
41	98	96	94	92	90	88	86	85
42	98	96	93	91	90	88	86	84
43	98	95	93	91	89	88	86	84
44	98	95	93	91	89	87	85	84
45	98	95	93	91	89	87	85	83
46	98	95	93	91	89	87	85	83
47	97	95	93	90	88	86	84	83
48	97	95	93	90	88	86	84	82
49	97	95	92	90	88	86	84	82
50	97	95	92	90	88	85	83	81
51	97	94	92	89	87	85	83	81
52	97	94	92	89	87	85	83	81
53	97	94	91	89	86	84	82	80
54	97	94	91	89	86	84	82	79
55	97	94	91	88	86	83	81	79
56	97	94	91	88	85	83	81	78
57	97	93	90	88	85	82	80	78
58	96	93	90	87	84	82	79	77
59	96	93	90	87	84	81	79	76
60	96	93	89	86	83	81	78	76
61	96	92	89	86	83	80	77	75
62	96	92	88	85	82	79	77	74
63	96	92	88	85	81	79	76	73
64	95	91	88	84	81	78	75	72
65	95	91	87	83	80	77	74	71
66	95	90	86	83	79	76	73	70
67	95	90	86	82	78	75	72	69
68	94	89	85	81	77	74	71	68
69	94	89	84	80	76	73	70	67
70	94	88	83	79	75	71	68	65
71	93	87	82	78	74	70	67	64
72	93	87	81	76	72	68	65	62
73	92	86	80	75	71	67	63	60
74	92	85	79	73	69	65	61	58
75	91	83	77	71	67	62	59	56

Although we advise never to boil string-proof, it might be that manufacturers still desire to concentrate the last molasses without grain, and in that case the clarified and diluted molasses should be boiled in a pan till the required density is obtained, after which the cuite is struck in large cisterns and allowed to crystallize there for a couple of weeks or even months.

This is, however, an exception, and we shall refrain from further occupying ourselves with that old-fashioned way of working, which wastes both time and sugar.

As a rule those massecuites which will, after centrifugalling, yield sugar and molasses giving up yet further sugar after being returned into a second boiling are not cooled but are cured hot for the same reason as for their not being highly concentrated. We want a molasses of a certain purity and can get that either by cooling and depressing thereby the quotient, or by taking in a little more molasses into the mixture. Since that latter operation is very easily performed and cooling requires time and space, it is much better to calculate the proportion of the two constituents of the mixed massecuite on the basis of hot-cured molasses than to calculate it on the cold-cured base and take the trouble of cooling.

Yet the receivers of the first and second massecuites, although they do not serve as coolers, have to be provided with a stirring apparatus to keep the mass in movement during its journey from the pan to the centrifugals. The massecuite is hot; it contains rather rich molasses from which sugar will crystallize out if it gets cold, and if that sugar crystallizes out in a stiff mass, without circulation, it will not adhere to already existing crystals but will form an after-crystallization of minute sugar crystals, which make the molasses turbid and stiff, which interfere with its proper separation from the large crystals, and which are spun through the centrifugal linings. With a view to keeping the molasses limpid and clear, and also ensuring that the after-crystallization is available for the first product, the massecuite coming from the pan must be kept in the same gentle motion as it was while boiling; and this action must be maintained till it comes into the centrifugal basket.

It is advisable to have large iron troughs under the pans connected by means of wide, open inclined gutters, so that every pan can discharge its contents into any of the troughs desired. The outlets of the troughs are in their turn connected with the receivers for the centrifugals by open gutters and screws or pumps, but in order to crush the crystals as little as possible, the transport of the massecuite through the trough to the centrifugals should preferably be done by gravity, and not by chain-pumps and the like.

The troughs should be of a size to contain the contents of a whole pan with some space over, if it is necessary to mix the massecuite with diluted molasses, water or sugar crystals.

A spindle with stirrers keeps the contents in continuous gentle movement and impels them slowly towards the outlet.

For the same reason as mentioned when discussing the fittings of the pan, the inside of the troughs should be painted with a good adhesive coating of heat-and-acid-resisting varnish or paint. The massecuites may be acid and may attack the iron plates which might give rise to an unpleasant colour in the sugar crystals, with a corresponding reduction in the value of the finished sugar.

The last massecuites, which are to be separated into sugar crystals and exhausted molasses, need a thorough cooling in order to force all the crystallizable sugar to assume the crystalline form, for if the molasses in which it is contained were not cooled to a low temperature, it might hold sugar in solution which had been disposed to crystallize out, but now is thrown away in the exhausted molasses causing loss.

The coolers for the last massecuite may be of the open or of the closed type; they may be provided with a water-jacket or none, all this being immaterial and to be decided according to the fancy of the manufacturer. The principal thing is that they be large enough to hold the contents of a whole pan at one time with some space over to dilute the mass with water or with diluted molasses, while the stirring apparatus must be strong enough to move the stiff mass properly without getting bent or broken.

The requirement that each cooler should hold the contents of one vacuum pan is necessary in order to be able to treat every panful individually, and to prevent the proper crystallization and cooling being disturbed by any addition of hot massecuite from a subsequent boiling.

The possibility of adding water or diluted molasses to the highly concentrated last massecuite is an excellent aid to promote crystallization of the last portions of sugar still to come out from the hot molasses; and it further enables the mixing of the cooling mass to be carried out without undue stress on the stirring gear, while finally the curing of the dilute molasses in the centrifugals goes on much better than when in a strongly concentrated condition.

The concentration in the pan of the last massecuite is conducted much too far as regards the proper water content of the molasses, but it is necessary in order to induce the sugar to crystallize out during the short time of its stay in the pan. As soon as this advantage is reached, the sugar crystallizing out slowly in the cooling mass will come out too, if the molasses surrounding the crystals is somewhat diluted; in fact the crystallization is in many cases even a little better in a moderately diluted molasses. For all these reasons it is wise to dilute the massecuite in the coolers to a density of 96° Brix (equivalent to one of 85° in the molasses, which corresponds to 80 per cent. of dry substance by the refractometer).

The dilution has to be done very carefully so as not to form pools of water in one part of the cooler in which whole portions of the massecuite get dissolved, while other parts remain intact. The water should be added to the massecuite through a perforated pipe at the bottom of the cooler, or hot diluted molasses of 70° Brix should be poured on the top of the massecuite. This hot molasses readily mixes with the highly concentrated massecuite, so that the mixture becomes more fluid and in proper condition for the crystallization of the last portions of sugar and for the subsequent curing in the centrifugals.

The cooling must not be pushed further than 45° C, and should remain preferably a couple of degrees above, as at that temperature crystallization is finished, and below it the natural viscosity of the molasses increases so considerably that cooling down to a lower point only causes trouble without any compensating advantage.

CHAPTER II.

CURING AND FINISHING.

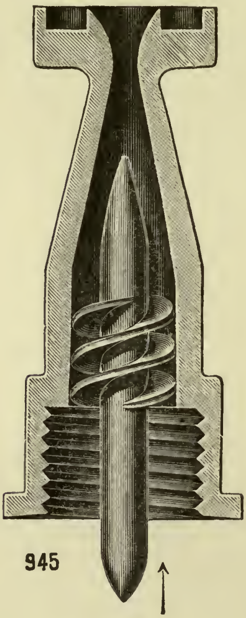

Fig. 10.
Steaming Nozzle.

The molasses is spun off the sugar crystals by running the massecuite from the troughs or the coolers into centrifugal machines, which are made to revolve at high speed. The molasses is forced through the meshes of the lining by centrifugal force, while the crystals are held back, being at the end of the operation taken out of the basket. Yet, however long the spinning may be continued, there will always remain a thin film of molasses around the crystals, which makes them sticky and colours them, and which can only be removed by washing their surface with water, dilute syrup, or steam.

By this washing, or as it is also called, purging or covering, no small amount of sugar is dissolved in company with the molasses and thereby its quotient of purity is unduly raised, while portions of sugar which had already been obtained from that molasses will return to it once more, which is not an economical way of working.

When making white sugar the sugar crystals should be washed freely in order to deprive them as far as possible of the adhering layer of coloured molasses. If the whole of that washing water or syrup, with all the sugar dissolved, is to be prevented from mixing again with the mother-liquor just spun off, one or other of the many devices must be used which have been invented with the aim of separating during curing the mother-liquor from which the sugar has been crystallized and the much richer washings or purgings.

The first requirement of a good separation is of course to machine the crystals as dry as possible in the centrifugals, so as to leave the smallest possible amount of mother-liquor adhering to the crystals and require the least amount of water for its solution in the washing water or syrup.

When the molasses layer is being washed off and the washing water is being spun off by centrifugal force, great care should be taken to prevent these two liquids getting mixed.

The simplest way of performing this separation is to provide two gutters behind the centrifugals, one for the molasses proper, the other for the pure washings. A movable outlet from the centrifugal discharges the molasses into that gutter to which it belongs by simply inclining it to one side or the other. This, however, is not quite sufficient, as the viscid molasses has not entirely left the outer drum of the centrifugal when purging has started, so that a partial mixing of the two cannot be avoided. Moreover, the outlet is not shifted automatically, and there is consequently risk of this operation being sometimes omitted. Another method is an arrangement in the outer casing of the centrifugal, which necessitates the basket being arranged to turn in either direction by means of an alternation of the driving gear.

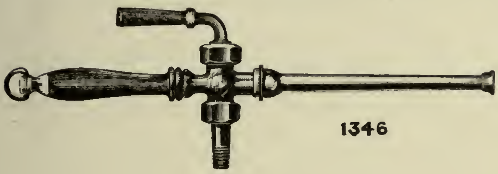

Fig. 11.
Spraying Nozzle.

A third device is a centrifugal having two casings, one inside the other, each of which is provided with a discharge outlet. One casing is fixed, as in the ordinary type, while the inner casing may be shifted up or down by means of a lever. Whilst the true molasses is being separated, the movable casing is raised, so that the molasses is caught in the fixed one, and passes through its discharge pipe to its respective receiver. As soon as purging commences the movable casing is lowered, and catches the purging syrup, which escapes through the other discharge pipe into a separate receiver.

All these appliances are very ingenious and probably work well when properly handled, but there is always a chance of mishaps, and that is what one cannot afford to risk in white sugar manufacture. The slightest tinge in the sugar will reduce its commercial value and as at so late a stage of the manufacturing process no mistakes could be made good again, we are obliged to go the safest way.

This is undoubtedly the double curing in two sets of centrifugals In the first the poor molasses is separated and the sugar crystals spun as dry as possible, after which they are taken out of the basket, mixed with purging syrup in a mixer or pug-mill and cured in the second set of machines. There they are washed with a fine spray of water till all the adhering molasses is removed. The runnings obtained are partly used to pug fresh quantities of dry sugar from the first set, while the surplus is carried back to the receiver of the clarified juice, or to that of the first molasses, depending on the quality of the sugar or on the kind of massecuite which has been cured.

The figures 12 and 13 show diagrammatically how this double curing might be effected. In both cases there are four centrifugals for the first curing, the so-called "foreworkers," and six for the second spinning, the "after-workers." In the first example, the dry crystals of raw sugar from the first set are mixed in a pug-mill with the rich syrup and conveyed into the feeding trough of the second set by means of a chain pump. In the second example, the dry sugar from the first set is carried into the mixing mill above the second centrifugals by means of an elevator. So it is seen there are many possibilities of performing the admixture of the dry sugar from the fore-workers with syrup from the after-workers, and of conveying the magma obtained into the after-workers for the final curing.

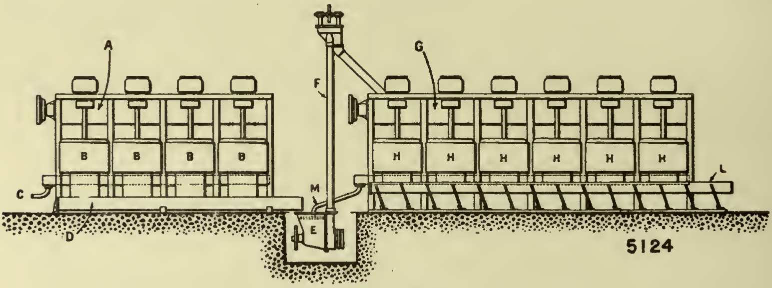

Fig. 12.
Arrangement with Chain Pump.

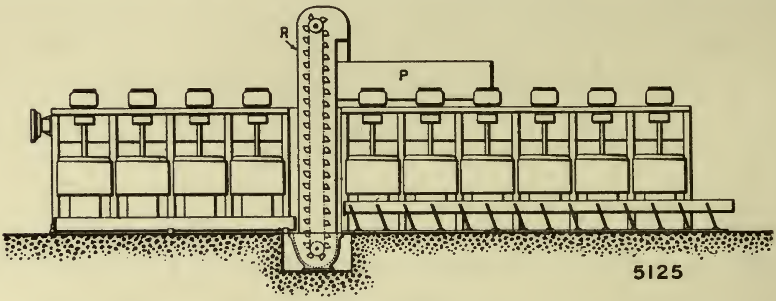

Fig. 13.
Arrangement with Elevator.

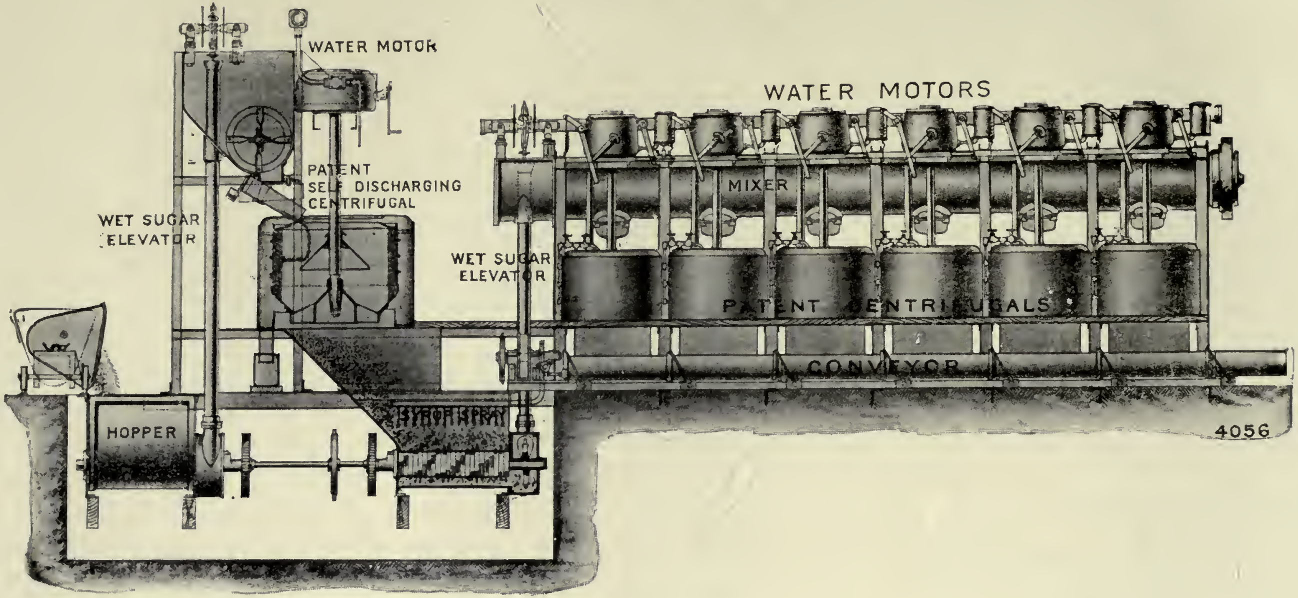

Plate 4.

Centrifugal Arrangement for White Sugar with one Fore-worker of Special Design.

The proportion between the number of fore and after-workers is not a fixed one, as this greatly depends on the character of the massecuite to be treated.

It is wise to maintain a certain elasticity in the ratio of the numbers of the two kinds of centrifugals in order always to be ready for every emergency. Therefore the centrifugals which are all of the same size and pattern are arranged in one continuous row and have two feeding troughs *A*, *B* placed behind them, as shown in Fig. 14. These have two openings opposite each centrifugal, so that massecuite can be fed into any centrifugal from either feeding trough according to how the attendant has set the valve.

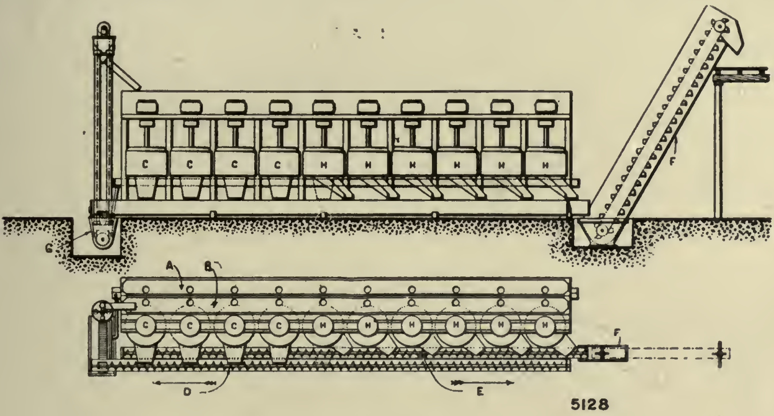

FIG. 14.
Arrangement with a variable number of fore and after-workers.

If, therefore, feeding trough *A* receives the original massecuite, as many centrifugals as may be thought necessary for use as fore-workers can be set to draw their supply from it, and in the figure the centrifugals *C* have been so arranged. The sugar from the centrifugals is guided by swivel shoots into one or other of the two conveyors *D* and *E*. The conveyor from the first set takes the sugar of the fore-workers to the mixing mill, from which it is elevated to the feeding trough *B*, which has been set to supply the other centrifugals *H*. These are the after-workers, the sugar from them being discharged on the other or white sugar conveyor, while the molasses is discharged through swivel spouts. The fore-workers deliver into one gutter and the after-workers into another.

If it is found that the proportion of fore and after-workers is not correct, a simple alteration of the valves of the feeding troughs, the swivel shoots under the centrifugals, and the swivel molasses discharge pipes, will allow any other desired grouping. This arrangement has an incidental advantage in that, if it is desired to cure two different grades in the ordinary way, it is possible to use all the machines for one kind of massecuite and of sugar at the same time. It is evident that although the fore-workers need not be provided with appliances for washing the sugar with water, all of them should be so provided in case they are all used for making a sugar which requires washing. And save for that reason, it would be good to have a hose connected with each centrifugal in order to wash out the linings occasionally, as in the meshes some sugar might be caked and dried up and so impede the regular outflow of the molasses.

In some factories the set of fore-workers has been replaced by one big machine of special design, which is open at the bottom (Fig. 15.) The massecuite enters it from the mixer when the centrifugal is running at full speed, the sugar clinging to the walls of the basket, while the molasses passes through. By slackening the speed of the machine, so as to reduce the centrifugal force, the sugar is caused to slip down until it falls through the open base of the basket into a second pug-mill, where it is mixed with purging syrup and then finally cured in the ordinary centrifugals (*Plate* 4.)

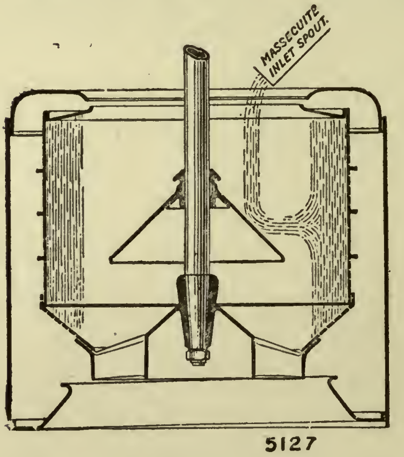

Fig. 15.
Self-discharging Centrifugal.

The washing in the second set of centrifugals, the after-workers, may be done by means of a spray of water, or by a fine jet, or by steam, which condenses on the crystals, washes off the layer of adhering molasses, and is spun off as a sugar solution. In case of covering with water, condensation water from the hot-well should be used after having been cooled down, and on no account should water from a ditch or river be employed as it might convey dirt or noxious micro-organisms or fungi into the white sugar, a danger which should be carefully avoided for obvious reasons.

The mother-liquor is clarified and boiled again if it is not exhausted, while the final molasses, spun off from the sugar crystals in the last massecuites without covering, is thrown away.

The purging syrups are partly used to pug fresh portions of the raw sugar from the fore-workers, while the balance is returned to the juices or first molasses. In order, however, to prevent this syrup or portions of it remaining constantly in the centrifugalling plant, all the purging syrup is from time to time sent back to the juices, and the purging started again with water or with fresh syrup from the evaporators.

The purging syrup which is not wanted for the pugging of new portions of raw sugar is sent back to the clarified juice in the case of first massecuites, and to the clarified first molasses if it has come from after-product sugar, so as to bring it back to the same stage of purity from which the sugar originated.

It is not wise to return the purging syrup of first sugar to the syrup, because, being of quite a different density, this might confuse the pan-boiler who prefers to work with syrup of uniform density, especially when boiling massecuite for white sugar. It is therefore better to return it to the clarified juice, after which it is concentrated to the usual density of syrup in the evaporators.

In cases when the sugars from the first and the second massecuites have to be mixed together to form one grade of white sugar, the centrifugalling may be facilitated by not curing the sugar from the fore-workers of the second massecuite in the after-workers, but by bringing it in the troughs into which the first massecuite had been struck. That sugar had been deprived as far as possible of its adhering mother liquor, and when stirred in the troughs with the first massecuite the thin layer of molasses still sticking to the crystals mixes with the purer molasses from the first massecuite. This mixing has the same effect as if the sugar had been mixed in a pug-mill before being cured a second time, while the small amount of low grade molasses does not affect considerably the purity of the first molasses.

In other cases the last sugars, called "molasses sugars," are only spun dry in the fore-workers and are not submitted to a second curing in the after-workers. They are either sold as molasses sugars or they are pugged with syrup into a magma which serves as seed wherewith to start a new boiling. The syrup washes off the layer of molasses surrounding the crystals, so that the mixture, which is drawn into the vacuum pan as seed, consists of pure sucrose crystals in a saccharine liquid having the purity of the syrup from which a portion of the sugar has already crystallized out, *i.e.*, the identical constitution of a syrup in the pan after the graining has taken place and the grain has attained the same size as that of the crystals used for seed. There is therefore no objection whatever against starting a new massecuite for white sugar in this practical way.

When the sugar is quite cured and purged, it is sometimes blued in order to neutralize the last traces of yellow colour. The classical material which has been used since times immemorial to make yellowish sugar appear sparkling white is ultramarine blue. A packet of that blue dye-stuff is mixed in a large pail of water and well stirred, and as soon as the purging syrup is entirely spun off a cupful of the ultramarine emulsion is poured into the centrifugal basket over the sugar. The particles of blue are held between the sugar crystals and the water passes through. When the sugar is discharged on the conveyor we see parts of the sugar coloured distinctly blue while others are not touched at all, but the sugar crystals are so well mixed on the conveyor and in the sieves or screens, that at the moment when the sugar is bagged, the colour has become quite even without any blue patches being visible. Ultramarine, though possessing a beautiful blue colour which admirably mixes with the slightly dirty yellowish tinge of white plantation sugar to produce a good neutral white tone, has the serious disadvantage of not being acid-fast and, what is still worse, of being decomposable by acids with the formation of evil smelling sulphuretted hydrogen.

When white plantation sugar blued with ultramarine is used for the sweetening of lemonade, wine, or in short any preparations into which acid juices enter, the blue dye gives off this bad smelling constituent, thereby spoiling the fine flavour of the liquors and diminishing their value as beverages.

In order to obviate this, much use has been made of late of an acid-fast and quite insoluble dye-stuff, called *indanthrene.* It is sold either as a paste or precipitated on dextrin as a dry powder, which is diluted in water just like ultramarine and is employed in exactly the same way. As has been said, it is insoluble, quite harmless, and colours everything very strongly just as does ultramarine, but it is acid-fast and does not impart an unpleasant smell to the condiments or aërated drinks made with it; on the other hand the colour is not so pure a blue as are specially selected brands of ultramarine for sugar-house use, and does not give so beautiful a neutral tone with the yellowish tinge of white plantation sugars.

As a rule only granulated sugars are blued, while pilé, cubes, and white seconds do not undergo any such treatment at all, but are sold in their natural state of coloration.

Up to now the treatment of the different kinds of white sugar has been the same, while the finishing touches only show such differences as will follow from the observations given here.

Granulated Sugar.—After the granulated sugar has been cured, purged, and, if desired, blued, it has next to be dried. The best way of drying that sugar is by a jet of dry steam in the centrifugal and not in a special dryer or granulator, in which raw sugar may be treated with success. The brilliant sugar crystals lose their lustre in a Hersey or other dryer, owing to their being rubbed against each other and to the sugar dust being driven by the air over the moist surface of the crystals entering into the dryer. Thereby the commercial value is diminished unnecessarily, and that is why we advocate drying plantation whites by steam in the centrifugal.

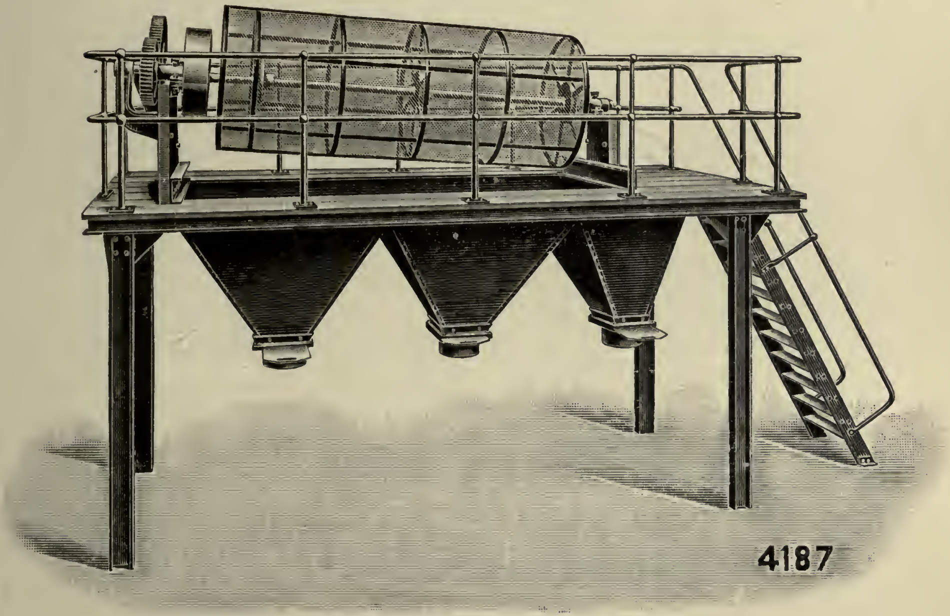

Fig. 16.
Sugar Sifter.

The steam ought to be dry and superheated by passing it through a small heater previous to its being introduced into the centrifugal. The condensed water formed is vaporized again in the furnace, so that the steam is quite dry when it strikes the sugar grain and takes away the moisture without impairing the brilliancy of the crystals. The steamed sugar after being discharged from the centrifugal is passed through a sieve

(Fig. 16) where big lumps or pieces of rust or, in short, everything not belonging to the pure sugar crystals, is held back and during that sieving it cools so that it may be bagged quite dry and cool. Sugar should never be bagged when still warm, for in that case it may cake together, forming hard masses in the bags to the detriment of its value.

Second Boiling Granulated.—In some countries there is a ready demand for a white, moist second-product sugar, which should be extremely fine-grained. This sugar is obtained by boiling first molasses of about 70° quotient of purity on grain, which grain is built up from the molasses and not from a pied-de-cuite from syrup. A pied-de-cuite of very small-grained molasses sugar may also do, but the requirement is a small size of grain. so that a pied-de-cuite of syrup does not suit the purpose. This massecuite is concentrated to a Brix of about 93, cured double, and in the last set of centrifugals only treated with purging syrup, without covering with water or drying. The sugar comes out moist, which moisture must consist of the last remnants of the glucose-containing purge syrup and not of water. The reason for this is that the purchasers want a moist, soft sugar, which must remain so even after being stored for a long time. If the moisture consists of water, or rather pure sugar solution, it will evaporate and the mass in the bags will become a hard lump, while if that moisture consists of syrup containing sucrose and glucose, it keeps moist, owing to the hygroscopicity of the solids, and thereby the sugar retains its softness. Many instances of moist white seconds having become unsaleable after having been stored for some months in a dry warehouse have been found to be due to a too extensive removal of the white purging syrup. Such sugar should contain at least 0·25 per cent. of reducing sugars, while lower quantities may give rise to the objectionable phenomenon just referred to.

Ground Second Sugar.—In some cases a very fine, white sugar is wanted, which is obtained by passing a well-dried white after-product sugar through a disintegrator. The sugar must be quite dry in order to be ground to a fine powder, while, owing to the crushing of the crystals, the colour of the finely divided sugar is much whiter than that of the larger crystals from which it has been made.

Lump Sugar.—Sometimes the moist white second sugars are pressed into blocks of a few pounds' weight and sold; but this method of distribution has not met with much success, because the block of sugar becomes so hard tha it is difficult to break it conveniently for use.

Pilé or "Broksuiker."—The pilé sugar as is made in South America and in Spain is a white sugar, having the form of irregular pieces of a very fine, grainy, crystalline mass, while the corresponding "broksuiker" of Java is of the same quality and only occurs in pieces in the form of bricks or broken divisions of the same. Both sugars are made from a very finely-grained first massecuite which is centrifugalled in the ordinary way, after which it is washed with water till the purging syrup is quite white. After that washing, steam is admitted to the centrifugal, so that the surface of the crystals gets wet and sugar is dissolved. If the steaming is stopped at the same time as the centrifugal, the sugar will come out, as we have shown, in loose crystals, but if the steam is stopped, say, five minutes before the centrifugal, the sugar dries in the basket owing to the air passing through the mass, and this drying causes the film of dissolved sugar which surrounds the crystals to solidify and bind the whole mass together into a solid lump (Fig. 17). In the case of pilé, the sugar is dug out of the basket with a *machete* or a crowbar, and put through a breaker, the lumps being sorted out into different sizes.

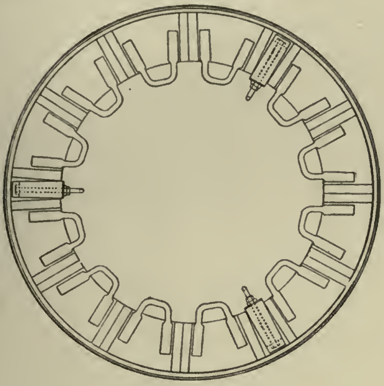

Fig. 17.
Centrifugal for Pilé Sugar.

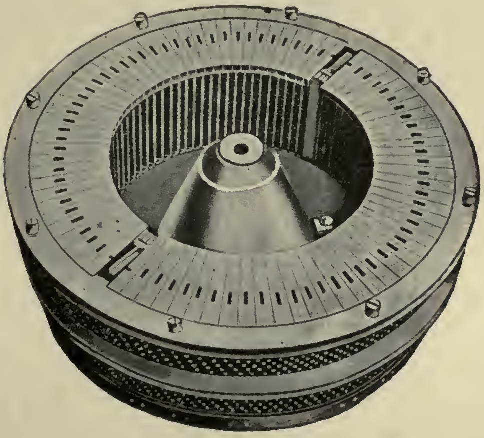

Fig. 18.
Centrifugal for Cube Sugar.

The "broksuiker" of Java is always moulded in blocks. The way of making it is the same as has already been described, but the basket is filled with moulds of the size desired, so that when the centrifugal is stopped the moulds can be withdrawn separately and the sugar preserved in neat handy blocks. In some factories the bottoms of the moulds are made of centrifugal gauze with a monogram or trade mark fixed in the middle, so that the blocks of sugar have a neat design stamped on them; but in other cases the moulds simply lie against the centrifugal shell, which is lined with a perforated copper plate. The sugar bricks are dried in the sun or in a special hot-air chamber, after which they are ready for sale.

Cubes.—Instead of bricks, moulds having the form of slabs are sometimes fitted in the centrifugal, so that the centrifugalled sugar there assumes the form of rectangular plates of the desired thickness (Fig. 18). These are purged and steamed just like pilé sugar, and after having been dried are cut in pieces by means of automatic shears (Fig. 19).

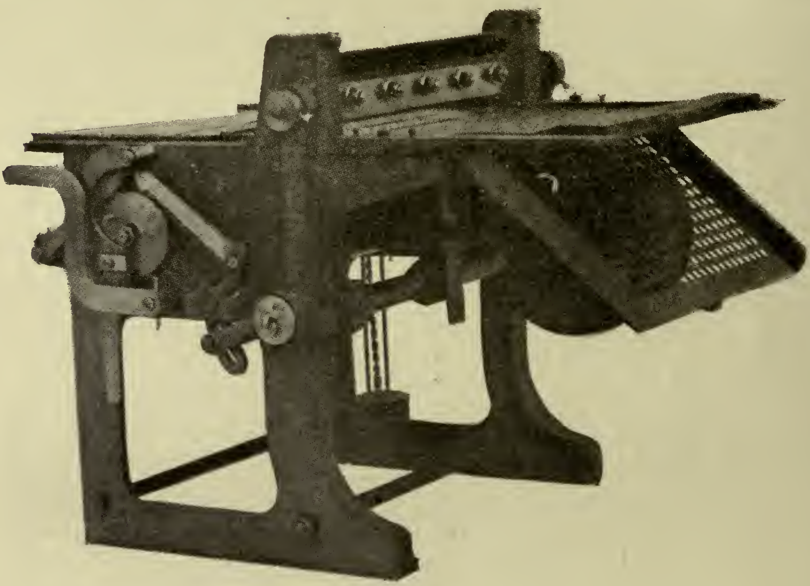

Fig. 19.
Cutting Machine for Sugar Cubes.

A second form of cube sugar is the Hersey, made by pressing a mixture of very fine white sugar and a thick sugar syrup in cube pieces by means of a drum with square openings through which the thick magma is pressed. The moist cubes are collected on a carrier and slowly transported through a hot-air chamber from which they emerge quite dry and ready for sale (Fig. 20).

Granulated first and second sugars are packed in double bags, the moist white seconds in single bags, while the packing of fancy sugars differs according to circumstances.

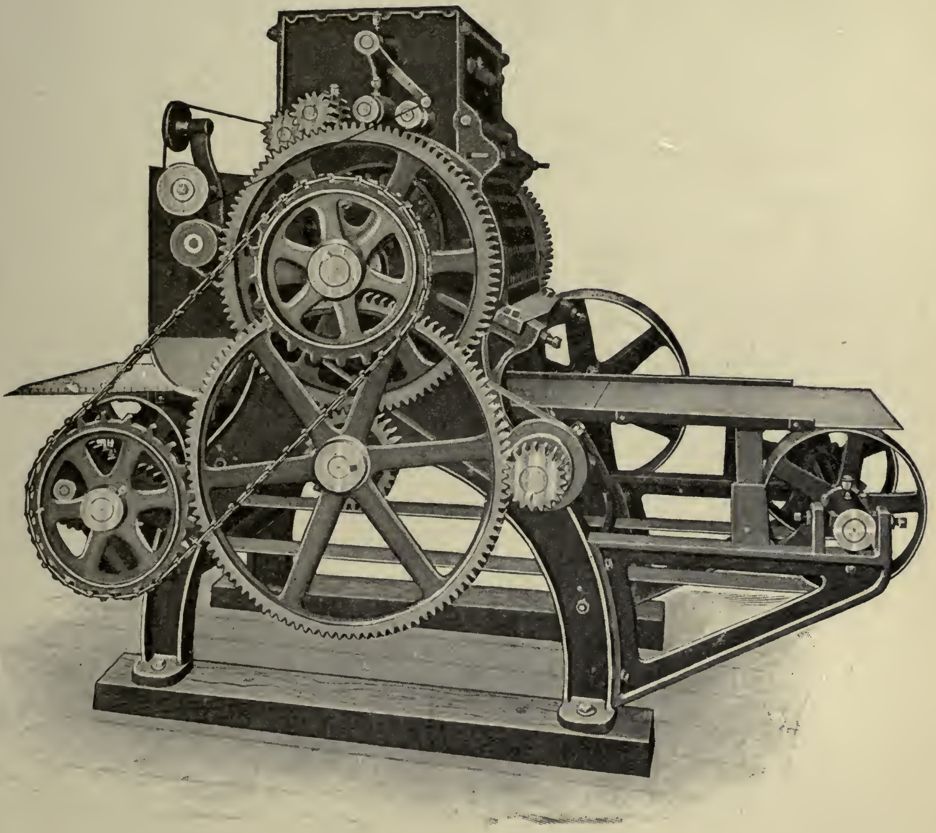

FIG. 20.

Hersey Cube Sugar Press.

CHAPTER III.

SCHEMES FOR THE MANUFACTURE OF WHITE SUGAR OF DIFFERENT DESCRIPTIONS, AND THE NECESSARY CAPACITY OF THE PLANT REQUIRED.

We saw in the foregoing chapters that several kinds of white sugar may be made from syrup according to the demands of the market, and for convenience' sake we give here a couple of schemes by which all the crystallizable sugar from the syrup may be obtained as a white product within three or four days' time simply by boiling to grain, so that six days after the last canes have been crushed all the available sugar may be in the bags.

I.—Scheme for turning out one sort of White Granulated.

The syrup is boiled to grain, and centrifugalled hot in the fore-workers.

Impure White Sugar.

It is pugged in a pug-mill or mixer with purging syrup, centrifugalled in the after-workers, washed, blued and dried with dry steam.

White Granulated.

Purging Syrup.

It is partly used for pugging fresh portions of the impure white sugar, while the balance goes back to the clarified juice.

First Molasses.

This is clarified as indicated, and boiled on a pied-de-cuite of molasses sugar and syrup to a second boiling of 70° purity, of large grain. This is cured hot in the fore-workers without washing.

Impure White Sugar.

This is carried to the troughs in which the first boiling is struck, and treated along with this to be turned into **White Granulated.**

Second Molasses.

This is boiled to grain on a pied-de-cuite of syrup to a purity of 60°. The massecuite is highl concentrated, cooled and cured without washing.

Molasses Sugar.

Is sold as such or is melted in the juice or mixed with syrup to serve as "seed" for the second massecuite.

Exhausted Molasses.

II.—Scheme for turning out White Granulated and White Seconds.

The syrup is boiled to grain and centrifugalled hot in the fore-workers.

Impure White Sugar.

It is pugged in a pug-mill or mixer with purging syrup, centrifugalled in the after-workers, washed, blued and dried with dry steam.

White Granulated.

Purging Syrup.

It is partly used for pugging fresh portions of the impure white sugar, while the balance goes back to the clarified juice.

First Molasses.

It is clarified, diluted, boiled to grain without any pied-de-cuite of syrup or molasses sugar. The grain should be small. The massecuite is highly concentrated, cooled for a long time, and cured without washing.

Impure Second Sugar.

This is mixed with purging syrup in a mixer or pug-mill, centrifugalled in the after-workers and washed if required.

White Second Sugar.

Purging Syrup.

This is partly used for pugging fresh portions of impure white second sugar, while the balance goes back to the clarified first molasses.

Molasses.

This is discarded from the factory as being exhausted, or may still be boiled smooth in order to effect some crystallization. It is then separated into—

Black After Products.

Exhausted Molasses.

When calculating the capacities of the pans and the coolers and also of the centrifugals for making white sugar, by one or other of the two schemes given above, we may state everything on 1000 tons of cane per 24 hours and assume an extraction of 78 per cent. and a density of the undiluted juice of 18° Brix. The quotient of purity of the syrup is taken as 85°, the density of the massecuite as 94° Brix, the purity of the three massecuites respectively at 85°, 70° and 60°, while the purity of the sugar from the fore-workers is assumed to be 97·5°.

The amount of sugar redissolved and returned to the syrup on curing in the after-workers is neglected, just as is the loss in the filter-press cakes and the unaccounted loss.

Scheme I.—The final molasses is, from the list on page 85, as low as 30°, while the intermediate molasses have purity values of 65° and 50° respectively.

1000 tons of cane yield, with an extraction of 78 per cent., 780 tons of undiluted juice, which with a Brix of 18° represent 140·4 tons of dry substance in the syrup or in the primary massecuite.

When curing a massecuite of 85° purity in a hot state and obtaining a raw sugar of 97·5° purity and a molasses of 65° we obtain (calculating for the moment everything on the dry substance basis) 61·5 per cent. of sugar and 38·5 per cent. of first molasses from the well-known formula:

$$\text{Sugar} = \frac{\text{Quotient massecuite} - \text{Quotient molasses}}{\text{Quotient sugar} - \text{Quotient molasses}} \times 100$$

$$\text{Sugar} = \frac{85-65}{97{\cdot}5-65} \times 100$$

Sugar = 61·5 per cent.; and molasses = 100 − 61·5 = 38·5 per cent.

In the same way calculation will show that 100 parts of the massecuite of 70° purity yield 42 parts of sugar of 97·5° purity and 58 parts of molasses of a purity of 50°, it being well understood that everything is calculated as dry substance for the moment.

100 parts of second massecuite of 70° purity require 75 parts of molasses of 65° purity to form a mixed massecuite of 70° purity according to the second well-known formula:

$$\text{Syrup} = \frac{\text{Quotient mixed massecuite} - \text{Quotient molasses}}{\text{Quotient syrup} - \text{Quotient molasses}} \times 100$$

$$\text{Syrup} = \frac{70-65}{85-65} \times 100$$

Syrup = 25 per cent.; and molasses to be added = 100 − 25 = 75 per cent.

In the same way we find that when making a mixed massecuite of 60° purity from syrup of 85° and molasses of 50°, we want 28·6 parts of dry substance from syrup and 71·4 parts from the molasses. With those figures we arrive at the following calculation:

100 parts massecuite of 85° purity yield 38·5 parts of molasses of 65°;
100 parts massecuite of 70° purity yield 58 parts of molasses of 50°;
100 parts massecuite of 70° purity require 75 parts of molasses of 65°;
100 parts massecuite of 60° purity require 71·4 parts of molasses of 50°.

The molasses for those 100 parts of massecuite of 60° is obtained from $\frac{71{\cdot}4 \times 100}{58} = 123$ parts massecuite of 70° purity.

The molasses of these 123 parts of massecuite of 70° is obtained from $\frac{123 \times 75}{38{\cdot}5} = 240$ parts of primary massecuite of 80°.

We have therefore:

100 parts of massecuite containing 28·6 parts of primary massecuite;

123 parts of massecuite containing $\frac{123}{100} \times 25 = 30{\cdot}7$ parts of primary massecuite

240 parts of primary massecuite = 240·0 parts.

463 parts of total mixed massecuites containing 299·3 parts of the primary one.

The 140·4 tons of dry substance in the syrup become therefore by the first scheme under consideration $\frac{140 \times 463}{299{\cdot}3} \times 100 = 217$ tons of dry massecuite or, with a Brix of 93°, equal to $\frac{217 \times 100}{93} = 234$ tons of massecuite with its proper moisture content to be boiled per 24 hours.

We assume the boiling time to be the same for every category of massecuites, which will not be far amiss, and if we take the time for boiling, emptying, steaming out, etc., at 8 hours, the total *vacuum capacity* should be 80 tons working contents.

The 234 tons of massecuite are subdivided into 122 tons of 85°, 62 tons of 70°, and only 50 tons of 60°. The two first-mentioned boilings are cured hot, while the last one needs cooling. When allowing the last massecuite to occupy the coolers during three days the *cooler capacity* should be 3 × 50 = 150 tons.

The weight of massecuite to be cured is as follows:

In the fore-workers—

122 tons of first massecuite of 85° purity yielding per 100 $61{\cdot}5 \times \frac{93}{98} = 71$ tons.

62 tons of second massecuite of 70° purity yielding per 100 $42 \times \frac{93}{98} = 25{\cdot}7$,,

50 tons of third massecuite of 60° purity.

25·7 tons of moist sugar from the second massecuite returned into the troughs of the first massecuite.

259·7 tons.

In the after-workers—

71 tons of moist sugar from the first massecuite.

25·7 tons of sugar from the second massecuite returned in the first ones.

96·7 which with the syrup used for pugging half that amount means a total weight for the after-workers of about 150 tons.

The *centrifugalling capacities* per 24 hours are therefore 260 tons for the fore and 150 tons for the after-workers.

The work in the former is much easier done than in the latter, save for the last massecuite, which is very sticky. Fixed figures as to the time of centrifugalling cannot be given, as these depend on too many factors to be duly considered.

Scheme II.—The data concerning the weight of cane, the purity of the juice, etc., remain the same, the final molasses also having a purity of 30°. The purity values of the two massecuites are 85° and 65°.

We saw from the former example that when boiling a massecuite of 85° purity and centrifugalling hot we obtained 61·5 parts of raw sugar, and 38·5 parts of first molasses of 65° purity. When this molasses is boiled to grain, highly concentrated, cooled during a long time and cured hot we obtain a molasses of 35°, and from the equation we find a proportion of 48 parts of impure sugar against 52 parts of molasses, everything computed on the dry substance basis. We come to the following results:

100 parts of primary massecuite yield 38·5 parts of molasses and 61·5 parts of sugar.

Those 38·5 parts of sugar yield 20 parts of molasses of 35 and 18·5 parts of sugar.

The 20 parts of molasses are boiled smooth and cooled for a long time. They are cured when work permits.

100 parts of primary massecuite therefore make: 100 + 38·5 + 20 = 158·5 parts of dry total massecuite.

The 140·4 tons of dry substance in the syrup therefore become from the second scheme under consideration: 140·4 × 158·5 ÷ 100 = 222·7 tons of dry massecuite or, with a Brix of 93°, equal to 240 tons of massecuite with its proper moisture to be boiled per 24 hours.

These are subdivided into 151 tons of first, and 58 tons of second massecuite, which are boiled to grain, while the remaining 31 tons are boiled smooth. We shall assume again the boiling time of the first ones to be 8 hours, but that of the latter to be much shorter, namely 2 hours.

The total *vacuum pan capacity* is therefore one-third of 151 + 58 = $\frac{209}{3} = 69{\cdot}6$ and one-twelfth of 31 or 2·5, so that the total vacuum pan capacity required is 72·1 tons.

The first massecuite is not cooled at all and the last is cooled in cisterns, so that only the second is cooled in coolers during the course of manufacture. This amounts to 58 tons, and when allowing for four days we find the necessary *cooler capacity* to be 230 tons.

The weight of massecuite to be cured is as follows:

In the fore-workers—

151 tons of first massecuite of 85° purity yielding per 100

$$61{\cdot}5 \times \frac{93}{98} = 88{\cdot}1 \text{ tons}$$

58 tons of second massecuite yielding per 100

$$48 \times \frac{93}{98} = 26{\cdot}4 \text{ tons}$$

—

209 tons.

In the after-workers—

88·1 tons of sugar from the first massecuite
26·4 tons of sugar from the second massecuite

—

114·5 tons of moist sugar which with the syrup used for pugging half that amount means a total weight for the after-workers of 170 tons.

The *centrifugalling capacities* per 24 hours are therefore 209 tons for the fore-workers and 170 tons for the after-workers.

PART III.

MATERIALS EMPLOYED IN THE MANUFACTURE OF PLANTATION WHITE SUGAR.

I. LIME.

Requirements.

In those factories where the juice is clarified by the defecation process, either followed or not by sulphitation or any other process in which only moderate amounts of lime are used, it is customary not to burn the lime on the estate, but to purchase it from outside.

The amount of lime consumed in such factories is, as we saw on page 19, relatively small; and since lime is the most active clarifying agent, it is well to bestow some attention on its quality so as to use the very best and purest obtainable, for any attempt at economy involving the purchase of an inferior product will invariably result in subsequent trouble and loss.

Lime readily absorbs moisture and carbonic acid from the atmosphere, for which reason it should be kept in closed packages, for instance in iron drums or in sealed tins, but not in baskets, and much less in open heaps in a shed.

Lime used for clarification should answer the following tests:

When mixed with half its weight of water it should become very hot within a few minutes; the slaked lime after the addition of ten times its weight of water should form a soft cream, which on being passed through a fine sieve should not leave behind more unslaked particles than one-tenth of the original weight, and most of these particles should become soft after an hour's standing in a moist condition. The lime, after being slaked, should dissolve in hydrochloric acid without appreciable effervescence, and not leave more than 2 per cent. of insoluble matter.

The maximum impurities should be:

Iron oxide and alumina	2	per cent.
Sulphuric acid	0·50	,,
Magnesia	2	,,
Silica	2	,,
Carbonic acid	2	,,
Moisture	2	,,

If burnt at too high a temperature it sinters somewhat and slakes with difficulty or not at all, so that it loses its efficiency, and when used in the single carbonatation process may cause a very objectionable after-slaking and alkaline reaction in the carbonatated juice in the filter-presses.

Pure lime, however, requires such a high temperature before being over-burnt as can hardly be obtained in an ordinary lime-kiln, and lime which in reality becomes over-burnt at a relatively low temperature is generally impure. For if the limestone (from which the lime has been prepared) contains silica together with alumina or iron oxide, fusible silicates are formed, and the lime is liable to become over-burnt. Usually the appearance of the pieces of lime indicates whether it has so suffered, but those pieces are generally picked out by the burner, so that the buyer very seldom finds them in the product supplied.

Water and carbonic acid, though not harmful, decrease the effect of a given quantity of lime, which explains why there are fixed limits for these constituents and also for the insoluble impurities which consist of sand or clay. Iron oxide and alumina give rise to the formation of scale in the evaporators; those caused by alumina being remarkable because formed from soluble aluminates during evaporation.

Magnesia is a very troublesome impurity in lime, when present in appreciable quantity. In the defecation process it gives the juices an unpleasant colour and retards subsidence, so that the decanted juice always contains suspended matter which forms scale in the evaporators. In the single carbonatation process the carbonic acid first converts the magnesia into magnesium carbonate which is insoluble but, on further addition, the soluble magnesium bicarbonate is formed. Later on, after filtration, the magnesium bicarbonate acts upon the lime-salts with the formation of free lime, so that the former neutral reaction of the filtered juice becomes alkaline again, which phenomenon at the high temperature in the evaporators gives rise to a dark coloration of the juice. If the juices are treated by double carbonatation or by sulphitation after single carbonatation, these detrimental effects are obviated, but in order to be safe the permissible percentage of magnesia in clarification lime must be restricted to a maximum of 2 per cent.

Sulphuric acid present in the lime (as calcium sulphate) also scales the evaporators, and should not occur in any appreciable quantity. Alkalis, on the other hand, are harmless, and usually occur only in small amount.

Methods for the Analysis of Clarification Lime.

Sampling.—From the lot of lime purchased, a number of pieces, both small and large, are chosen at random; they are broken smaller to the size of an egg by means of a hammer. The pieces are piled up into a heap, from which one section is taken to be piled up into another heap and subdivided till a subsample of about 2 lbs. is obtained. This is crushed into a coarse powder and kept in a stoppered bottle. As lime rapidly attracts carbonic acid and moisture from the atmosphere, the sampling should be done very quickly.

Matter Insoluble in Hydrochloric Acid.—5 grms. of the coarse lime powder are placed in a beaker; about 50 c.c. of water are added and after the lime has slaked, so much hydrochloric acid is gradually added till no more dissolves. A few c.c. of nitric acid are run into the solution, which is boiled and filtered through an ash-free filter into an Erlenmeyer flask of about 300-400 c.c. capacity. The insoluble part remaining on the filter is washed with hot water, till the filtrate shows a neutral reaction, after which it is dried and burnt in a platinum crucible. The weight of the ash is that of the insoluble matter from 5 grms. of lime.

Silica.—The filtrate from the foregoing operation, which together with the wash water will occupy a volume of 200-300 c.c., is heated to boiling point and nearly neutralized with ammonia, leaving the liquid only faintly acid and absolutely clear. This solution is boiled gently for one or two minutes and a few drops of ammonia added, rendering it slightly alkaline. After boiling for a short time, it is then allowed to subside and is filtered into a litre flask, the filter being washed thoroughly. The precipitate consists of silica, together with alumina, iron oxide and perhaps traces of lime; it is dissolved from the filter in dilute hydrochloric acid, and the paper washed with water.

The solution together with the wash water is evaporated in a porcelain dish to dryness and heated in an air-bath at a temperature of 120–140° C for one hour. The dry residue is moistened with a few drops of strong hydrochloric acid, diluted with water, heated and filtered through an ash-free filter which is washed. The filter with its contents is dried, and incinerated in a platinum crucible. The weight of the ash is that of the silica in 5 grms. of lime.

Iron Oxide and Alumina.—The filtrate and wash waters from the silica determination are heated to boiling in an Erlenmeyer flask and nearly neutralized with ammonia so that the liquid remains only faintly acid and completely clear. It is boiled gently for one or two minutes and rendered alkaline by means of a few drops of ammonia. The precipitate is collected on an ash-free filter, washed, dried, and incinerated in a platinum crucible. The weight found on this occasion is that of the iron oxide and alumina in 5 grms. of lime.

Calcium Oxide.—The filtrate and wash waters of the foregoing operations, which may contain some lime-salts, are added to the liquid in the litre flask which had been filtered off from the first precipitation with ammonia. The liquid is cooled to room temperature, made up with water to the mark and well shaken (Solution A). 50 c.c. of this are diluted with about 200 c.c. of hot water in a beaker or an Erlenmeyer flask; a little ammonia is added, and the mixture brought to the boil, after which the lime is precipitated by a solution of ammonium oxalate. The liquid is stood in a warm place for a couple of hours, preferably on a hot air-bath, and at the end of that time filtered through an ash-free filter, after ensuring that any further addition of the ammonium oxalate reagent produces no further precipitation. The precipitate on the filter is washed with water, till the washings no longer become turbid with nitric acid and silver nitrate. The lime may be weighed either as oxalate or as carbonate.

In the first case, the precipitate is filtered through a dry tared paper, that is a paper which has previously been placed in a drying-bottle, dried at 100° C in an air-bath and weighed. The washed paper containing the precipitate is replaced in the drying-bottle, which is again heated in the air-bath at 100° until its weight is constant.

The weight found for the calcium oxalate with its water of crystallization is multiplied by the factor 0·3836 in order to find the weight of the calcium oxide in 0·250 grm. of lime.

In the second method the dried filter with the calcium oxalate is incinerated in a platinum crucible till the filter is completely burnt; the residue is moistened with a few drops of ammonium carbonate solution, the excess of which is expelled by heating on the water-bath, any calcium oxide being thus converted into the carbonate again. As soon as the mass in the crucible has become dry, it is carefully heated for about five minutes by a very small flame, cooled and weighed. The weight found multiplied by the factor 0·56 is that of the calcium oxide in 0·250 grm. of lime.

Magnesia.—400 c.c. of Solution A are transferred into a 500 c.c. measuring flask, treated with ammonia until alkaline, and with ammonium oxalate solution until all the lime is precipitated. In order to make sure that this has really taken place, it is advisable to add 6 grms. of ammonium oxalate dissolved in 50 c.c. of hot water. The liquid is cooled to room temperature, made up to the mark, shaken and filtered; 250 c.c. of the filtrate are concentrated on the water-bath to a volume of about 50 c.c., and hydrochloric acid is added till the reaction is distinctly acid in order to dissolve again some magnesia which might have become insoluble during the evaporation. The liquid is rendered alkaline again by ammonia, is next filtered, and the insoluble residue washed with a little water.

The solution and the wash water are united and precipitated with sodium phosphate, the liquid being stirred with a glass rod, which is softly rubbed against the inside of the beaker. After a quarter of an hour about one-fourth of the volume of ammonia is added, and the mixture is allowed to stand for 12 hours in a cold place.

The precipitate is collected on an ash-free filter, and washed with 3 per cent. ammonia solution until the washings no longer become turbid with nitric acid and silver nitrate. Sometimes the precipitate adheres so strongly to the glass that it cannot be removed with a glass rod bearing a rubber cap. In that case as much of the precipitate as possible is collected on the filter, and the rest dissolved in a drop of hydrochloric acid; this solution is rendered alkaline with 3 per cent. ammonia and the precipitate thus obtained is combined with the first after standing for 2 hours. The filter with the combined precipitate is washed several times with the dilute ammonia solution, and dried in an air-bath.

The dry precipitate is transferred as completely as possible into a platinum crucible, while the filter with the precipitate which could not be removed is incinerated on the cover of the crucible and the ash added to the original quantity in the crucible, the whole being heated first over a small flame and finally over the blow-pipe. The complete whiteness of the heated matter may be promoted by moistening it during a pause in the incineration with, at most, two or three drops of nitric acid—on no account more.

The residue obtained is magnesium pyrophosphate, the weight of which is calculated to magnesia by multiplying with the factor 0·3624, which gives the amount of magnesia in 1 grm. of the lime.

Sulphuric Acid.—200 c.c. of Solution A are precipitated while boiling with a hot solution of barium chloride, placed on a hot air-bath for a couple of hours, and filtered through an ash-free filter. The precipitate is washed with water until the washings no longer become turbid with silver nitrate, after which it is dried and incinerated. The residue is removed from the paper as completely as possible to the platinum crucible while the paper is burnt on the cover. Both portions are combined and strongly heated, after which the crucible is cooled and weighed. The weight found, multiplied by the factor 0·3432 is that of the amount of sulphuric acid (SO_3) in 1 grm. of the lime.

Carbonic Acid.—A portion of the sample is ground in a mortar to a fine powder and 3 grms. weighed into a Geissler apparatus. This apparatus is first weighed empty, after which about 3 grms. of the lime are placed in the flask, the apparatus being weighed again to ascertain exactly the weight of the material employed. 10 c.c. of water are added to the lime, the closed funnel is filled with strong hydrochloric acid; and the bulb through which the carbonic acid gas escapes is filled with just so much concentrated sulphuric acid as to prevent back-suction. The whole apparatus is wiped dry on the outside, put aside for half-an-hour, and then weighed.

Next the hydrochloric acid is allowed to flow from the funnel into the flask gradually by carefully opening the cock, and as soon as all the lime is dissolved, the flask is carefully heated over a very small flame till the liquid commences boiling. A narrow rubber tube is connected with the opening, through which the carbonic acid has escaped; the cock of the hydrochloric acid funnel is opened; and a slow current of air is sucked through the apparatus till the gas no longer tastes of carbonic acid.

The apparatus is left to cool, and is weighed again, preferably by leaving the original weights on the right-hand pan while the difference is made up by placing small weights on the same pan as the apparatus.

The decrease in weight represents the carbonic acid (CO_2) in the amount of lime examined.

Alkalis.—Into a large flask containing 1500 c.c. of water 150 grms. of the coarsely powdered lime are added in small quantities, and the mixture shaken continuously till all the lime is slaked. As soon as this point is attained, the liquid is filtered through a dry paper and 1000 c.c. of the filtrate are measured.

A current of carbonic acid is conducted through the hot solution, which is heated, the precipitate filtered off, and the solution evaporated almost to complete dryness. The last traces of calcium oxide are precipitated by a few drops of ammonia and ammonium oxalate as insoluble calcium oxalate; the liquid is filtered from the precipitate into a platinum dish, and the filter washed. Filtrate and wash waters after being rendered acid with hydrochloric acid are evaporated to dryness, and gently heated over a small flame to drive off the ammonium chloride. The residue is weighed as potassium and sodium chloride, and after multiplication by the factor 0·6319 yields the amount of alkali, calculated as potash (K_2O) in 100 grms. of lime.

Water.—As the water is chemically combined with the calcium oxide, it cannot be driven off at 110° C, and a much higher temperature must be employed. 3 grms. of the coarse powder are heated in a porcelain or platinum crucible, covered with a lid for a quarter of an hour over a gas or spirit lamp, cooled in a desiccator and weighed. The loss sustained on heating is the weight of water in 3 grms. of lime.

The crucible is preferably of platinum and is narrow in shape. It must remain covered with the lid while heating, for if this is not done the lime may absorb carbonic acid from the products of combustion, the water content thus being found too low, and sometimes actually an increase in weight is observed.

The results of the analysis may be stated in the following form:

Calcium oxide	95·32
Magnesium oxide	0·07
Carbonic acid	0·91
Insoluble in hydrochloric acid	0·46
Silica	0·05
Iron oxide and alumina	0·18
Sulphuric acid	none
Alkalis	0·02
Water	2·89
Undetermined	0·10
	100·00

Rapid Method for the Estimation of the Magnesia in Clarification Lime.—The determination of magnesia being rather a lengthy one, a rapid method for detecting approximately whether the lime contains too much magnesia or not is often employed, especially as it is not always necessary to know the exact magnesia content. It is only needed to ascertain whether the lime is above or below 2 per cent. 1 grm. of the finely powdered lime is mixed with 10 c.c. of water and heated; hydrochloric acid is added drop by drop until almost all the lime is dissolved and the liquid is alkaline. It is then boiled, filtered, and ammonia added to the filtrate. If now a white gelatinous precipitate is formed, the lime contains over 2 per cent. of magnesia and should not be used; if a flocculent cloudy precipitate is formed, the content is between 1 and 2 per cent.; while an amount of less than 1 per cent. escapes detection by this rough method of estimation.

II. MILK-OF-LIME.

Preparation.

The lime used for clarification is in almost every case added in a mixture with water known as milk-of-lime or lime-cream.

This is prepared by placing lumps of lime in a shallow iron tank and throwing water on them till they are just submerged. The lime combines with the water, the pieces swell up considerably and crumble to powder with the development of much heat. More water is gradually added till the lime is quite soaked, and forms a thick paste. It is not advisable to throw the pieces of lime into a tank full of water as they are thereby cooled too much, and are slaked only very slowly.

After the action is over, the still hot pasty mass is diluted with water and passed through a strainer in which any large pieces of unslaked lime, stones, sand or other coarse impurities are removed; subsequently it is passed through finer metal sieves which keep back smaller grit; and, finally, it is run into large tanks where it is diluted to the required density.

In some places the pieces of lime are thrown into a rotating drum where they are slaked and whence the pieces of unburnt or over-burnt lime and the stones are discarded automatically.

At all events it is well to mix the lime with the water some time previous to using it, so that the hard particles may soften and be slaked. As we have already seen, this is especially necessary in the single carbonatation process, in which large quantities of lime are added to the juice, and if the milk contains unslaked grit, this remains unsaturated in the juice and may re-dissolve afterwards in the filter-presses, imparting an alkaline reaction to the clarified juice.

The carbonatation process requires a very large plant for the slaking, mixing and straining of the lime-cream, but in factories using defecation methods, three slaking and mixing tanks are sufficient.

It is advisable to use pure water for slaking lime. In factories working with the defecation process, the amount of water contained in the milk-of-lime is insignificant and does not impose any great additional strain on the evaporating plant. On the other hand, factories using the carbonatation process add such a large quantity of water to the juice in the form of lime-cream that it is worth the trouble of devising measures to reduce it. Trials, utilizing the sweet-waters from the filter-

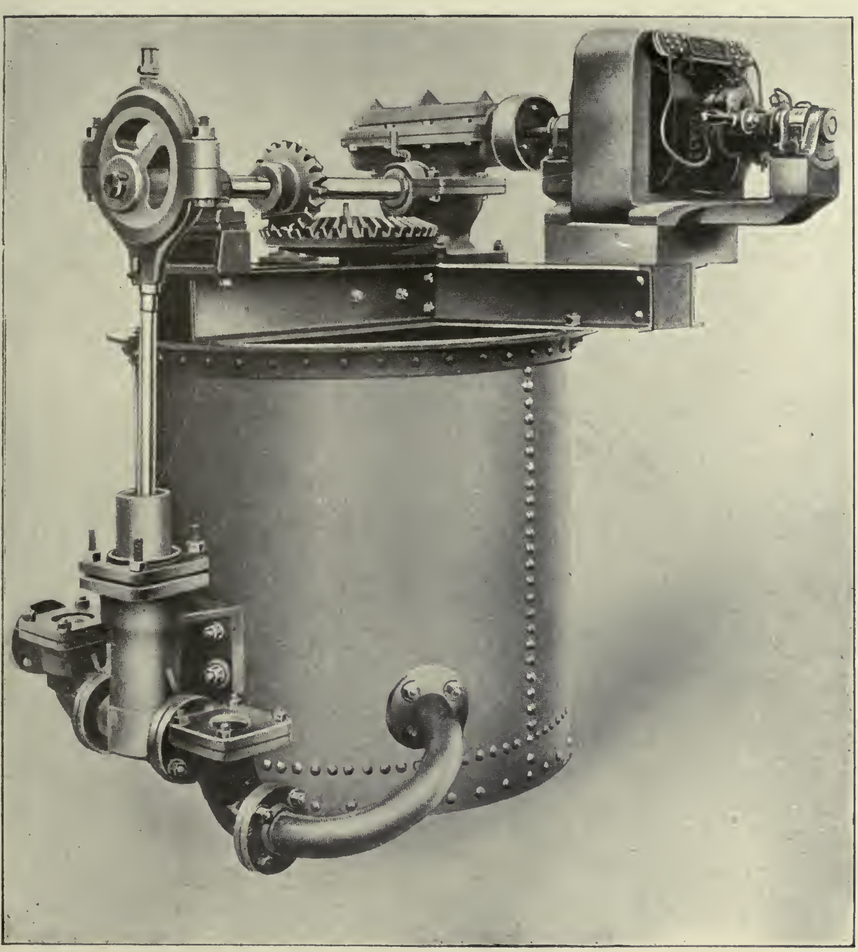

Plate 5.

Milk-of-Lime Mixer.

presses for slaking and diluting the lime, have often been made, but the result has not been satisfactory. If the milk-of-lime made from lime and sweet-water is allowed to stand for a couple of hours, the supernatant liquid contains a dirty brown scum consisting of products of decomposition of the reducing sugars by the action of the strong lime. These scums are viscous and render the juice thick, and so retard the saturation.

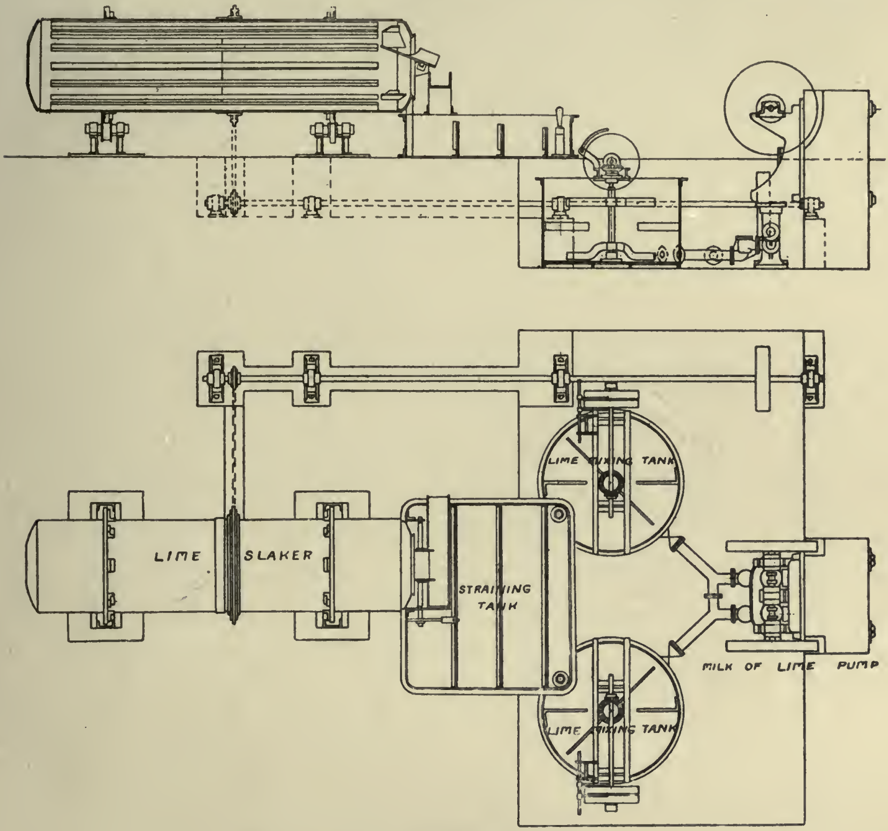

FIG. 21.
Milk-of-Lime Plant.

This waste of time by unsatisfactory saturation is, of course, as unfavourable to the work as is the long retention of the diluted juice in the evaporators.

The slaked lime, mixed with pure water to the consistency of a thick cream, is poured through strainers into two tanks which are filled and emptied alternately. When one of these tanks is filled and diluted to the desired density of 15° or 20° Bé., this tank is drawn from until empty. A fresh supply is meanwhile prepared in the second tank, so as to be in readiness when the first is empty ; this ensures only fresh and well-slaked lime being added to the clarifiers.

The contents of the tanks must be kept in steady motion to prevent the lime subsiding. In factories where only a little lime is used this may be done by stirring the milk with a paddle, but in factories using a great amount the lime-cream is constantly pumped through a pipe which passes near the quarter where it is mixed with the juice and returns to the supply tank. The milk-of-lime is thus forced from this tank through the pipes and back again. When lime has to be drawn off and added to the juice, a cock in the pipe is opened, otherwise it continues on its way back to the supply tank and is thereby kept in constant motion, no opportunity being thus given for any subsidence.

Determination of the Lime Content in Milk-of-Lime.

The lime content of lime-cream corresponding to the readings of the Beaumé hydrometer is as follows :

DENSITY OF MILK-OF-LIME.

Degrees Beaumé.	Weight of 1 Gallon, lbs.	Pounds of CaO per Gallon.	Per cent. of CaO.	Degrees Beaumé.	Weight of 1 Gallon. lbs.	Pounds of CaO per Gallon.	Per cent. of CaO.
1	10·07	0·075	0·75	16	11·25	1·590	14·13
2	10·14	0·166	1·64	17	11·33	1·700	15·00
3	10·21	0·259	2·54	18	11·43	1·810	15·85
4	10·29	0·360	3·50	19	11·52	1·930	16·75
5	10·36	0·459	4·43	20	11·61	2·060	17·72
6	10·43	0·559	5·36	21	11·70	2·180	18·61
7	10·51	0·650	6·18	22	11·80	2·290	19·40
8	10·59	0·745	7·08	23	11·90	2·420	20·34
9	10·66	0·839	7·87	24	11·99	2·550	21·25
10	10·75	0·940	8·74	25	12·10	2·680	22·15
11	10·83	1·039	9·60	26	12·20	2·810	28·03
12	10·91	1·150	10·54	27	12·30	2·950	23·96
13	10·99	1·260	11·45	28	12·41	3·090	24·90
14	11·07	1·370	12·35	29	12·52	3·240	25·87
15	11·16	1·480	13·26	30	12·62	3·390	26·84

In order to determine the density of the thick liquid as exactly as possible, the lime-cream is poured into a wide, glass cylinder, the hydrometer placed carefully in the liquid and induced to sink by gentle taps with the forefinger. As soon as the instrument is stationary the degrees are read off and the percentage or the weight of the milk-of-lime is ascertained from the table above. The temperature should not deviate much from 30° C, on which the figures are based.

If one wants to determine the density with greater accuracy, the milk-of-lime being examined is diluted with its own volume of water and the density determined with a Brix hydrometer. The table given underneath shows the number of degrees Bé. and the lbs. of calcium oxide (CaO) per gallon of the undiluted lime-cream, which correspond with the degrees Brix read in the mixture of the milk with an equal volume of water:

Degrees Brix of the Diluted Lime-Cream.	Pounds of CaO per Gallon of the Original Lime-Cream.	Degrees Beaumé.	Degrees Brix of the Diluted Lime-Cream.	Pounds of CaO per Gallon of the Original Lime-Cream.	Degrees Beaumé.
9·0	0·931	9·9	14·5	1·535	15·5
9·5	0·980	10·4	15·0	1·590	16·0
10·0	1·029	10·9	15·5	1·645	16·5
10·5	1·084	11·4	16·0	1·700	17·0
11·0	1·139	11·9	16·5	1·755	17·5
11·5	1·195	12·4	17·0	1·810	18·0
12·0	1·250	12·9	17·5	1·870	18·5
12·5	1·300	13·4	18·0	1·930	19·0
13·0	1·360	13·9	18·5	1·995	19·5
13·5	1·420	14·4	19·0	2·060	20·0
14·0	1·480	15·0	19·5	2·120	20·5
			20·0	2·180	21·0

These figures have, however, not only relation to the amount of really active lime in the milk-of-lime, as, of course, all the unslaked—and for other reasons inactive—portions of the lime are represented in the figure for the total calcium oxide content derived from the readings of the hydrometer.

The real content of active lime may be found by dissolving the lime from the lime-cream by means of phenol, which readily combines with calcium oxide to form a clear solution which can be filtered off, leaving the other constituents, which are of no value, on the paper. In the clear solution the amount of calcium oxide is found by titration with normal acid and phenolphthalein as indicator. Details are as follows:

The milk-of-lime is well mixed and 10 grms. are weighed in a weighing dish and transferred with 5 per cent. phenol solution into a 250 c.c. measuring flask. The calcium oxide dissolves at once, and after making up to the mark with the phenol solution, the liquid is shaken, filtered through a dry paper, and 100 c.c. of the filtrate titrated with normal hydrochloric acid, using phenolphthalein as indicator. The number of c.c. necessary for neutralization is multiplied by 0·7 to give the grms. of CaO in 100 grms. of the lime-cream.

III. LIMESTONE.

Requirements.

Factories in which the juices are clarified by the carbonatation process produce the necessary lime locally in a kiln, not only from an economical standpoint, but also because the carbonic acid which is formed on burning the limestone is required for the saturation. The process of lime-burning supplies therefore both reagents, the lime, and the carbonic acid.

In selecting the limestone yet other factors than that of the chemical composition have to be considered, now that such large quantities are required; for example, the distance of the quarry from the factory, the means of transport, the hardness of the stone, etc. The harder the stone, the more trouble is experienced in breaking it into suitable pieces and burning it in a kiln. It will therefore be generally more economical to buy a soft limestone found in the vicinity than a hard one which has to be conveyed from a distance, even if the analysis of the latter is more favourable.

Limestone exists in the forms of marble, calcspar, amorphous sedimented stone and fossil or new coral. The marble and calcspar are very pure, but the former sometimes contains a vein of dolomite (magnesium carbonate) while the latter has the defect of being very hard and very difficult to burn. The fossil coral is a soft stone, in many cases pure, but sometimes interspersed with ferrous strata, which render it unsuitable for the production of pure lime.

Fresh coral from the seashore should be exposed to rain before use, in order to wash out the salt.

In the best limestone the amount of moisture is so trifling that it may be neglected, but sometimes limestone is so porous and soft that it may contain very appreciable quantities of moisture. Owing to its firmness, the hard limestone may be kept in the open air, but it is advisable to store the more porous material under cover.

Apart from the water content, the maximum impurities allowable in a good limestone are:

Insoluble in hydrochloric acid	1 per cent.
Silica	1 „
Iron oxide and alumina	1 „
Magnesia...	1 „
Sulphuric acid (SO_3)	1 „
Alkalis	0·5 „

The content of dry calcium carbonate in the dry limestone should not be under 95 per cent.

Methods of Analysis of Limestone.

Sampling.—The sampling should be done exactly as has been described under *Lime* with this difference, that the last subsample should be ground to a very fine sample in a mortar. As limestone attracts neither moisture nor carbonic acid from the atmosphere, the sampling need not be done so hurriedly as with clarification lime.

The determination of the matter insoluble in hydrochloric acid, iron oxide and alumina, calcium oxide, magnesia, silica and sulphuric acid is made exactly in the same way as already described for lime. The only difference is that in this case 10 grms. of the limestone are weighed, transferred into a beaker, 50 c.c. of water added, and then a short stemmed funnel placed in the beaker so that the end of the stem does not dip into the liquid. Hydrochloric acid is poured into the funnel in small quantities, till the effervescence has stopped, after which the funnel is washed and removed, and the analysis proceeds as indicated. The weights found for the various constituents have, of course, now to be calculated on 10 grms. of the material, instead of on 5 grms. as in the former case.

Carbonic Acid.—The carbonic acid is determined in the Geissler apparatus, in which from 1 to 2 grms. of finely powdered limestone are weighed. Only 10 c.c. of approximately 20 per cent. hydrochloric acid need be used in the closed funnel.

Alkalis.—The direct estimation of alkalis in the limestone is very difficult, and therefore the stone has to be burnt first. 100 grms. of limestone are burnt and the alkalis determined in the quicklime obtained, using the method indicated on page 118.

Moisture.—5 grms. of the finely powdered stone are dried for two hours at 120° C, and weighed when cool. The loss of weight in grms. multiplied by 20 represents the percentage of moisture in the stone.

The results of the analysis may be collected as follows:

Calcium carbonate	96·77
Calcium oxide	54·50
Carbonic acid	42·27
Magnesium oxide	0·56
Insoluble in hydrochloric acid	0·82
Silica...	0·38
Iron oxide and alumina	0·79
Sulphuric acid	none
Moisture	0·42
Undetermined (alkalis, etc.)	0·26
	100·00

Rapid Method for the Estimation of the Magnesia in a Limestone.

For the same reason as given in the section on *Lime*, it is well to be able to make a rapid test for the magnesia content of the limestone, to enable us to ascertain without any lengthy analysis whether the limestone to be used contains more magnesia than the permissible limit or not. To this end 2 grms. of the finely powdered limestone are boiled with 10 c.c. of water and so much dilute hydrochloric acid that the liquid is neutral and still contains a very small quantity of the undissolved stone remaining unaffected by the acid; a few c.c. of lime-water are next added to throw down any iron oxide and alumina which may be present, when the liquid is filtered and ammonia added to the filtrate. In case of more than 1 per cent. of magnesia being present in the limestone, a gelatinous precipitate of magnesium hydroxide is formed, while a smaller quantity only gives a slight flocculation, or no separation at all.

IV. COKE.

Requirements.

Coke is used for burning with the limestone in the kiln. To that end it should not only be hard, but also of a uniform hardness, so that it is not crushed to powder by the heavy load on top of it in the lime-kiln. The coke should not contain more than 6 per cent. of moisture, for if stored in a moist condition it loses part of its fuel value; and when burnt with too high a water content, it may form carbon monoxide, a highly poisonous gas. It is therefore well, not only to see that coke with no more than the permissible water content is purchased, but also that it is stored under cover. A further requirement is that the coke shall contain only a minimum of tarry matter, because the latter would distil off during the heating in the kiln and contaminate the gas; it has even happened that so much tar had distilled over that the valves of the air-pump had become clogged.

The coke used in sugar factories is either foundry coke or gas coke. The former is purer, containing less ash and less tarry substance, so that, in order to obtain the same effect, 20 per cent. more gas coke is required than foundry coke; but in tropical countries where gas works exist, the difference is more than counterbalanced by the much lower price of the gas product. The sulphur content should also be low, and harmful sulphur compounds should not exceed 1·5 per cent.

The following analysis of a gas coke gives a good insight into the composition of such a product, while that of a foundry coke is added for comparison:

	Foundry coke.	Gas coke.
Ash	8·20	10·80
Moisture	0·68	2·20
Loss on heating in a current of nitrogen	1·44	3·72
Total sulphur...	0·45	1·84
Harmful sulphur	0·23	1·39

Methods of Analysis.

Sampling.—Lumps of coke are taken at random from the heap and broken to pieces of the size of an egg. The sample thus obtained is well mixed and piled in a heap; a segment is taken from it which is mixed again and piled in a smaller heap, till a subsample of about 2 lbs. is obtained. This is beaten to small pieces which are again mixed, while finally a portion of the last subsample is ground to a very fine powder and kept in a stoppered bottle.

Moisture.—5 grms. of the fine powder are dried to constant weight at 100°–105° C in an air-bath, which generally requires 2 or 3 hours to complete. The loss of weight multiplied by 20 represents the moisture in grms. on 100 grms. of coke.

Ash.—3 grms. of the very finely powdered coke are spread in a thin layer on a platinum dish and heated first on a small flame and afterwards at gradually increasing temperatures, until at last the heating is completed at a very high temperature. The carbon is consumed and the ash remains behind, but to make sure that really all the carbon has been burnt, the ash which has been cooled and weighed is treated with a little strong alcohol. If carbon is still present, this floats and then the alcohol is evaporated and combustion pursued till all the carbon has vanished. The weight of the ash is divided by 3 and multiplied by 100 in order to find the grms. of ash on 100 grms. of coke.

This determination must be made in a muffle-oven. The dish with the 3 grms. of coke is placed in the cold muffle, after which the lamp is lighted and the temperature gradually raised.

Sulphur.—Sulphur occurs in the coke in two forms, *viz.*, as sulphates (gypsum) or as sulphides (chiefly as pyrites). It is principally the sulphur occurring as sulphides which has a harmful action in the kiln, since it forms sulphurous acid on combustion which attacks the pipes and the pump. This sulphur is called the "harmful sulphur" in distinction to the sulphur in the form of sulphate which is harmless.

Total Sulphur.—0·5 to 1 grm. of the finely powdered coke is thoroughly mixed in a platinum crucible with 1 grm. of magnesium oxide and 0·5 grm. of anhydrous sodium carbonate; the crucible is placed at an angle and heated during several hours till all the carbon is burnt. The mixture is stirred now and then with a platinum wire.

After being cooled down, the contents of the crucible are transferred with a jet of water into a beaker, and bromine solution is added till the yellow colour remains, after which the mixture is boiled with an excess of hydrochloric acid. The harmful sulphur which had been oxidized to sulphites during the combustion is oxidized to sulphate by the bromine and dissolved by the hydrochloric acid. The liquid is boiled till the odour of bromine is driven off, filtered, the residue washed out with water, and the sulphuric acid precipitated with barium chloride, just as has been described on page 117.

By multiplying the amount of barium sulphate weighed by the factor 0·137, the total sulphur in 1 grm. of coke is found.

It is understood that both the magnesia and the sodium carbonate used must not contain any sulphate, and if that is not so, then the sulphuric acid is determined in the amount of the reagents used, an allowance being made in the calculation.

Harmless Sulphur.—This is determined by heating the ash of 5 or 10 grms. of coke with hydrochloric acid and a few c.c. of bromine solution; this latter addition serves to re-oxidize that part of the sulphate which, on combustion, might have been reduced to calcium sulphide. The liquid is filtered, boiled till no odour of bromine can any longer be detected, after which the sulphuric acid is determined in the usual way.

Harmful Sulphur.—This is found by subtracting the percentage of the harmless sulphur from that of the total sulphur in the coke.

Loss of Weight on Heating in a Current of Nitrogen.—5 grms. of coke are heated in a Rose crucible for 2 hours at 110°–120° C to drive off the moisture. The crucible is weighed after cooling, after which it is heated for 15 to 30 minutes over a large flame, while a slow current of dry nitrogen gas is conducted through the crucible. After cooling, the crucible is weighed again, the loss of weight representing the tarry substance in 5 grms. of coke.

The nitrogen gas is prepared by heating a solution of 2 parts of potassium nitrite and 1 part of ammonium chloride in a large flask. The flask is connected with a gasholder by a wide tube.

When determining the loss on heating, the gas is conducted first through a wash-bottle filled with concentrated sulphuric acid, and the current is regulated in such a way that only one gas bubble passes per second.

The Lime-kiln.

The kilns used in sugar factories are of the "shaft" and "Belgian" types, fed with a mixture of limestone and coke. The fact that the burned lime is thereby contaminated with the ash of the coke has long ago been recognized as of no importance, since this ash is almost entirely insoluble both in water and in milk-of-lime and thus cannot impart impurities to the juice. The burnt lime contains about 2 per cent. of ash derived from the coke, the greater part of which forms a fine powder on the surface of the lumps of lime, and falls off during the handling of the lime without causing any trouble during manufacture. Generator kilns (in which the lime is heated by the flame from inferior fuel burnt in special furnaces, so that no ash or tar can contaminate the lime) are not used in cane sugar factories; so coke, which does not contain much tarry substance, and which is hard enough to stand the pressure of the limestone without becoming pulverized owing to too great a brittleness, must be used.

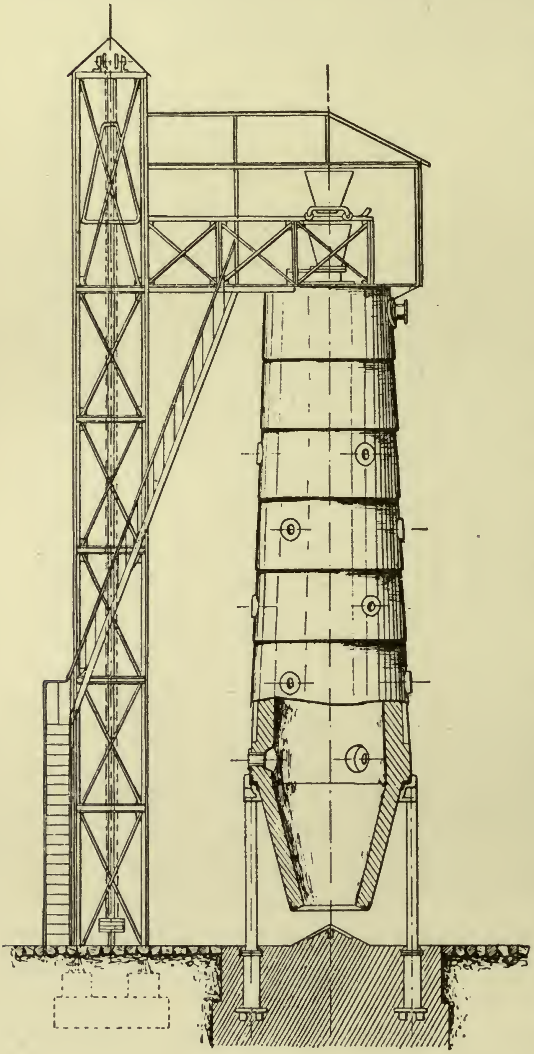

Fig. 22.
Lime-kiln.

The shaft kiln is a truncated cone of masonry, lined on the inside with refractory brick. The top terminates in an iron funnel closed by a cone, which may be raised or lowered by moving a lever. When charging the kiln the cone is lowered, and the charge falls through the space between the cone and the funnel into the interior of the kiln, the bottom of the latter being provided with three large openings closed by iron doors, through which the burnt lime may be withdrawn from time to time. The openings for air are also at the bottom, while the temperature of the different parts of the kiln may be inspected through sight-holes at various heights, and closed with plugs. Finally a flue, built of masonry, at a height of about three-quarters of the kiln, is connected with the gas-suction pipe, conveying the gas to the carbonic acid pump and washer.

The Belgian kiln (Fig. 22) is made of firebrick cased with iron plates, whilst the space between the brick and the outer casing is filled with non-conducting material to retain the heat. It has the form of two obtuse cones, joined together at their bases, similar to the shaft kiln. The upper and longest one is provided with an iron funnel and cone at the top, which opens to allow the introduction of fresh charges. The undermost inverted cone is open and its lower edge is about 2 ft. from the ground, the kiln being suspended in an iron ring supported by six iron columns. Under the bottom opening a small cone of masonry is built upon which the burnt lime falls from the kiln. The fallen lime partially obstructs the opening between the kiln and the ground and thus prevents more lime from dropping. From time to time the cooled pieces of lime are removed, allowing the contents of the kiln to fall a little, and these, in turn, are removed as soon as they are sufficiently cooled.

The kiln should be lighted a few days prior to the grinding season, since it takes some time to become sufficiently heated to yield good lime and carbonic acid. In the Belgian kiln a wall of coarse pieces of limestone is made round the bottom in order to support the firewood, which is thrown in at the top. In the case of a shaft kiln the doors are merely closed, no wall of limestone being required. Some two tons of burning firewood having been thrown into the kiln, a layer of limestone is spread upon it in order to distribute the flame of the fire evenly throughout. Above this is added a layer of coke and then alternately layers of limestone, and coke in their usual proportion. The funnel is left open until the wood is all consumed and the lowest strata of the kiln alight, after which the funnel is closed with the iron cone and the carbonic acid pump started, the latter draining off the gases and producing a vacuum in the kiln. This causes air to flow in from below, which passes through the red-hot stones and coke, and thus maintains the draught necessary for combustion.

The usual charge consists of 1 part by weight of coke to 9 parts of limestone; and as the weight of the same volume of limestone is thrice that of coke the proportion of the two materials by volume is 1 : 3; so that for every 3 baskets of limestone 1 basket of coke is added. These materials are commonly hoisted up in baskets containing 100 to 120 lbs. of limestone and thrown down into the kiln through the funnel at the top. It is advisable to alternate regularly the two components in order to obtain a homogeneous mixture in the kiln and not local heaps of either limestone or coke.

This proportion of 3 parts of stone to 1 part of coke is not intended to be a fixed one, but may be varied according to circumstances; for example, the size of the pieces of stone and coke, the hardness or softness of the stone and the quality of the coke. As will be shown on page 134, the analysis of the gases and the temperature of various parts of the kiln will give sufficient indication as to whether it is necessary to add more of one or other of the ingredients. The limestone should be broken into pieces of the size of one's fist and the coke in pieces of half that dimension. Though many mechanical breakers have been invented it is advisable to break limestone and coke by hand, in order to prevent too much waste. If the pieces of limestone are too large they burn slowly, while if too small the draught in the kiln is obstructed and the lime does not slake well afterwards. It is evident that in case of a soft stone pieces of larger dimensions may be taken than when a hard crystalline stone has to be burnt.

When the kiln burns well its contents shrink considerably at the commencement, because of the rapid combustion of bulky firewood. Charging is continued, always keeping the level of the contents at about one foot below the flue for the carbonic acid, and as soon as the contents cease to sink rapidly the layer of unburnt stones is withdrawn at the bottom. When no more unburnt limestone falls out, but well burnt lime appears at the doors or at the bottom of the kiln, the regular working of the kiln commences, all that is then necessary being to continue charging the kiln with limestone and coke, and to remove the burnt lime, all the time keeping the pump working steadily to maintain draught and combustion. The lime should be removed from below every three or four hours, and the kiln kept charged with alternate layers of stone and coke, always keeping the level of the charge one foot below the suction pipe.

The carbonic acid is formed both from the combustion of the coke and the decomposition of the limestone, which under the influence of heat is converted into quicklime and carbonic acid. Calcium carbonate when heated to 1000° C becomes entirely converted into calcium oxide and carbonic acid, provided this latter is allowed to escape; if, on the contrary, the carbonic acid is not removed the decomposition stops at a point depending on the pressure of the carbonic acid. In order to obtain a rapid decomposition of the limestone, it is therefore necessary to carry off the carbonic acid as fast as it is formed, all the more as cold lime absorbs carbonic acid to form calcium carbonate again.

When we consider a lime-kiln in full activity we distinguish in it two different movements in mutually opposite directions—(1) from above to below of fixed materials going very slowly; (2) from below to above of gases flowing very fast.

The weight of the gases is greater than that of the fixed bodies. The space in the lime-kiln may be divided into three zones, which may be called the heating zone, the decomposition zone, and the cooling zone.

The mixture of limestone and coke is heated in the first or heating zone by the current of gas coming from the decomposition zone, this gas thus being itself cooled, and at the same time evaporating the moisture from the coke and the stone.

When the mass sinks deeper it enters the decomposition zone. Here the temperature of the mixture, which has gradually risen, attains its maximum by the combustion of the coke by the oxygen from the air sucked in from below. The limestone is decomposed and loses weight, till, when leaving this zone, it is completely broken up into its two constituents—lime and carbonic acid. The lime sinks into the cooling zone, where the hot pieces give off their heat to the ascending current of gas, while the coke is fully consumed by the oxygen of the air. During its passage through the hot pieces of lime this air will absorb the heat and enter well warmed into the decomposition zone, which will be the more completely achieved the slower and the larger the current of air.

The oxygen combines with the carbon of the coke to form carbonic acid, which mixes with the carbonic acid formed by the decomposition of the limestone, while the nitrogen of the air remains inactive and passes into the gas mixture unchanged. The water vapour in the air acts upon the red-hot coke with the formation of hydrogen and carbon monoxide, which two gases are further burnt to form water and carbonic acid, provided they are not carried off unburnt by too strong a draught, and thus pass into the saturation gas.

By admitting an excess of air so that the saturation gas contains about 1½ per cent. of oxygen, the carbon monoxide is generally oxidized, so that the saturation gas does not usually contain any amount of that gas, and should, on no account, possess a higher content than 0·50 per cent. That portion of the water vapour which does not attack the carbon acts favourably on the decomposition of the limestone, as the temperature at which calcium carbonate decomposes is lowered by the presence of water vapour.

When the temperature rises considerably above 1300° C, the carbonic acid is decomposed into carbon monoxide and oxygen, both of which are then found in the saturation gas.

The hot gases passing upwards through the thick layers of coke and stone are gradually cooled down to a temperature of about 200° C. We saw that the cooling zone serves to heat the air and to cool the burnt lime. The warmer the lime comes out of the kiln the more fuel will be necessary to heat the gas, as the heat in the lime discharged represents mere loss. The cooling zone must therefore have a sufficient capacity and possess a good diameter, this latter feature causing the current to slow down and give ample opportunity for transmitting the heat of the lime to the air. The height of this zone must, however, not be unduly increased, in order to avoid the risk of the lumps of lime being crushed to small pieces, a contingency which would hamper the entry of the air. The combustion in the decomposition zone would then be interfered with, the consequences being deficiently burnt lime and much carbon monoxide in the gas.

The heat necessary for the decomposition of the limestone has to be supplied in the decomposition zone, so that the temperature prevailing there must be sufficiently high. Although calcium carbonate is broken up into its constituents at a temperature of 1000° to 1050° C, the best maximum temperature should be from 1200° to 1300° C, at which point a rapid decomposition may be expected without fear of any fusion of the lime. This temperature prevails only in a comparatively small part of the kiln, so that the stone passes through it in about six hours, which, indeed, is quite sufficient. In order to obtain this the heating zone, where the gases come into contact with the cold stone and coke, must be so spacious that the former can be well cooled and give off as much heat as possible to the said materials, thus saving fuel.

Finally, it is well to leave an empty space in the kiln above the suction pipe in order to serve as an air-box for the pump, and to promote the regularity of the suction.

Experience shows that the capacity of the cooling zone should be as great as the volume of limestone burnt in 24 hours, so that the lime requires 24 hours to pass through it. The heating zone and the decomposition zone are both half the size of the cooling zone, so that the capacity of the kiln must be equal to twice the volume of the limestone to be burnt in 24 hours.

It is evident that in regular work the decomposition zone should occupy the same part of the kiln, *i.e.*, a little more than half-way up the structure. The sight-holes in the wall permit the observation of the interior and of the real position of the decomposition zone. If this sinks—red-hot lime falls out on discharging; and, if it rises—flames escape from the aperture at the top; which, besides placing the workmen in charge of the filling of the funnel in imminent danger, may destroy the iron tubes and the funnel and so render the kiln unfit for use. The right proportions in the kiln may be restored by either exhausting the gases more slowly or more rapidly, and by changing the proportions of fuel and limestone, the operations being always controlled by the analysis of the gas. But, if a kiln once works irregularly it requires days of strenuous effort to get it into proper order again.

The following irregularities may occur:

(*a*) **Sintering.**—This is caused either by impure raw material or too high a temperature or too prolonged a stay in the hot zone by a too tardy withdrawal of the already burnt lime.

In the case of limestone containing too much silica, iron oxide, and alumina, or of coke containing too much ash there is a probability of more or less easily-fusible double silicates being formed. The ash of the coke chiefly consists of infusible aluminium silicate, but in the simultaneous presence of iron oxide and alumina double silicates with lime may be formed, especially if the temperature rises above 1300° C. Besides, the lime may also combine with constituents of the firebricks, forming with them a vitrous fusible combination. This formation of double silicates is the cause of the so-called "scaffolding" of a kiln, while in such a case the lumps of lime become covered with an impermeable layer, which prevents proper slaking.

When these phenomena are observed we have either to employ a purer limestone or else try to reduce the temperature of the kiln by mixing less fuel with the limestone. If, for one or other of these reasons, the lime fuses into a solid mass so that the downward motion is obstructed, no lime falling from the discharge openings (a phenomenon which bears the name of "scaffolding"), it is necessary to try to break the solid mass by poking in it with long irons through the sight-holes and from below.

(*b*) **Unburnt Lime.**—This can arise from too small a proportion of fuel in the charge, that is, insufficient to produce the heat necessary for the total decomposition of the limestone; but it may also be due to the kiln being too small for the amount of work required of it. This can be remedied accordingly: either by mixing more fuel with the limestone or by building a larger kiln; or, if possible, by adopting one of the modifications of the carbonatation methods, in which less lime is employed.

When unburnt lime comes out together with coke on discharging, and especially if this is the case at one particular side of the kiln, this indicates that some part of the kiln has cooled down. Usually this is occasioned by a cold wind blowing against one side, and thus cooling the contents locally to a point below the temperature of decomposition. This can, of course, be remedied by erecting a screen or wall to protect the kiln from the prevailing winds.

(*c*) **Red-hot Lime.**—In this case the decomposition zone has sunk too low and the cooling zone has, accordingly, become too small. By leaving the lime longer in the kiln the decomposition zone rises again, and can further be assisted by accelerating the speed of the pump and thus cooling the lower strata more efficiently.

(*d*) **Over-burnt Lime.**—As already stated lime can be over-burnt by being exposed to too high a temperature, which causes the surface to fuse and to become impermeable to water, or it may become coated with an impermeable layer of impurities which are more or less fusible when combined with lime. We have already learnt to consider silica (both free and in combination with iron oxide or alumina) as a dangerous constituent in this respect; and when a kiln, which has not been overheated, regularly yields over-burnt lime, it is advisable to try whether a purer limestone will not produce better results.

(*e*) **Excess of Carbon Monoxide.**—When the gas passing from the kiln contains more than one per cent. of carbon monoxide it is a proof that the draught is not sufficient, and the speed of the pump must be accelerated in order to promote the combustion of the carbon monoxide in the higher strata by a more abundant supply of oxygen. Sometimes, however, even with a sufficient draught the content of carbon monoxide becomes too high, and this is the case when the coke or the limestone is too moist, *e.g.*, by being stored after having been in the open air and exposed to rainfall, and thrown into the kiln without previous drying. The large amount of water vapour in contact with the red-hot coal gives rise to a kind of water-gas consisting of hydrogen and carbon monoxide and thus yielding an impure saturation gas, contaminated with the poisonous carbon monoxide. In this case a better draught will not improve matters, and the only remedy is to use dry materials.

(*f*) **Excess of Free Oxygen in the Gas.**—The gas from a lime-kiln in good condition must contain a small content of free oxygen which need not exceed $1\frac{1}{2}$ per cent. If the oxygen content is higher than this it may be due to too strong a draught, or to leaks in the kiln or suction pipe. As the pump maintains a vacuum in the kiln, air penetrates through the leaks, and not only dilutes the gas but also causes an undesirable cooling of the contents, thus retarding combustion.

(*g*) **Sulphuretted Hydrogen or Sulphurous Acid in the Gas.**—When the coke contains sulphur as iron sulphide, this element becomes oxidized in the kiln to sulphurous acid, which at the high temperature corrodes the tubes and the pump, especially when the gas has been saturated with water in the washer. On incomplete combustion the gas may sometimes become contaminated with sulphuretted hydrogen, but if the excess of oxygen is a normal one this cannot occur, as the sulphuretted hydrogen is oxidized to sulphur dioxide. Sulphur dioxide and sulphuretted hydrogen can never occur simultaneously as they practically decompose each other with the formation of sulphur and water.

The size and the capacity of the kiln must be adapted to the weight of cane worked up in the factory to obtain the best results. The total height should not exceed 30 ft. otherwise the weight of the column of limestone and coke becomes too great, and the coke may be crushed. As regards capacity, we usually assume a yield of 16 lbs. of lime per 24 hours and per cub. ft. of real capacity of the kiln, though of course this quantity may vary considerably according to the hardness of the stone.

One cub. ft. of limestone weighs 93 lbs. and requires $\frac{1}{3}$ cub. ft. of coke, yielding 51 lbs. of burnt lime. We allow the lime to remain for 2×24 hours in the kiln, so that these 51 lbs. of lime require a capacity of $2 \times 1\frac{1}{3} = 2\frac{2}{3}$ cub. ft., and as the kiln is only filled three-quarters full $2\frac{2}{3} \div \frac{3}{4} = 3\frac{5}{9}$ cub. ft. is the total capacity. One cub. ft. thus yields 14·8 lbs. of lime per 24 hours. When the lime is burnt more quickly, *e.g.*, in $1\frac{1}{2}$ days, the capacity rises considerably, and then every cub. ft. yields $\frac{14{\cdot}8 \times 2}{1\frac{1}{2}} = 19{\cdot}6$ lbs. of lime per 24 hours, showing that the kind of stone and the varying distribution of the materials have a marked influence on the capacity of the kiln.

93 lbs. of limestone yield 51 lbs. of lime and 40 lbs. of carbonic acid (assuming a yield of 55 per cent. of lime and 43 per cent. of carbonic

acid). Taking the carbon content of the coke at 80 per cent., and the proportion of limestone to coke as 100 : 11, then $\frac{93 \times 11}{100} = 10{\cdot}23$ lbs. of coke furnish $10{\cdot}23 \times \frac{80}{100} \times \frac{44}{12} = 30$ lbs. of carbonic acid, bringing the total yield of carbonic acid to $40 + 30 = 70$ lbs. It is evident that for the saturation of the lime obtained only 40 lbs. of carbonic acid are required, but the absorption of the carbonic acid in the saturation-gas is incomplete, amounting in the most unfavourable instance to about 60 per cent. of the total, so that in such a case only $\frac{70 \times 60}{100} = 42$ lbs. of carbonic acid are available where 40 are necessary. This shows that even with the most wasteful process the lime-kiln produces an ample supply of this gas, so that any shortage need not be feared. On the

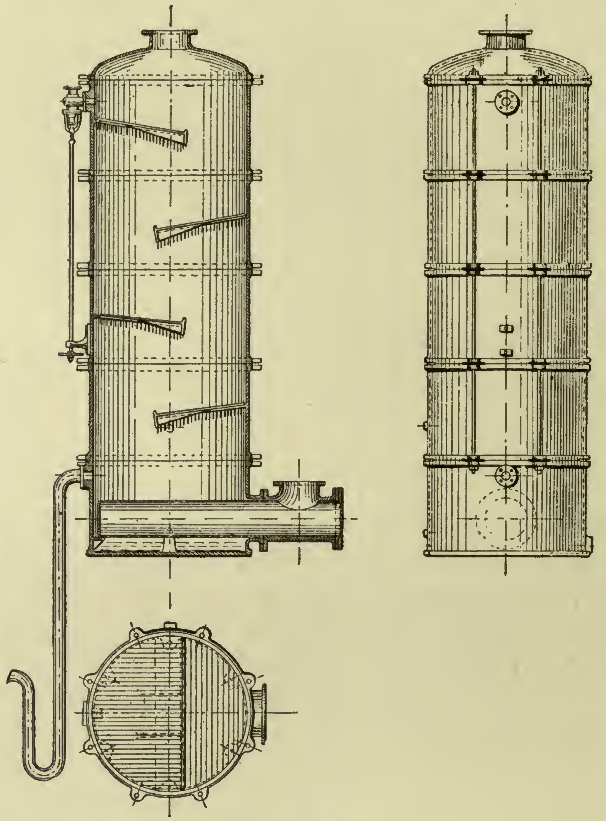

FIG. 23.
Gas-washer.
Inside and outside view.

contrary, it would be possible (in such an emergency, as when the capacity of the lime-kiln is too small for the weight of the canes crushed) to purchase some additional lime from other sources, and yet have sufficient carbonic acid from the kiln to saturate all the lime during carbonatation.

When lime is used in carbonatation in the highest proportion of our table on page 69, *viz.*, 45 tons of limestone to 1,000 tons of cane per 24 hours, or 2½ per cent. of lime on the weight of canes, the capacity of the kiln should be 3,750 cub. ft., according to the calculation given above.

V. CARBONIC ACID.

The mixture of gases pumped out of the lime-kiln is washed and cooled before being sent to the saturation tanks. The current of gases is forced through a gas-washer (Fig. 23) of some design or other, where the tarry substances which may have been formed and remain unconsumed are removed, and where the gas comes out at a temperature of some 40° C.

The composition of a good saturation gas may be represented as follows, calculated on the anhydrous gas:

	Per cent. by Volume.
Carbonic acid	28·5
Nitrogen	69·5
Oxygen	1·5
Carbon monoxide	0·5
	100·0

Complete Analysis of the Saturation Gas.

A complete analysis of the saturation gas is best carried out by the Orsat apparatus. A thin metal tube conducts the saturation gas from the main supply pipe to the laboratory, where it is shut off by a cock. When desiring to analyse the gas, the cock is opened and the current of gas is allowed to escape till it is certain that the air in the pipe has been replaced by the gas of the same composition as that supplied to the carbonatation tanks.

The Orsat apparatus (Fig. 24) consists of a burette *A* of 100 c.c. capacity, divided into fifths of a c.c., and placed in a cylindrical glass vessel filled with water to keep it cool. At its lower end it is connected by means of a long rubber tube with a small bottle containing water acidified with a few drops of hydrochloric acid to inhibit the growth of algae. *B*, *C* and *D* are glass bulbs for the absorption of gases, which are filled with thin, short glass tubes to increase the surface. Each of these bulbs is connected at its lower under end to a similar vessel without glass tubes. The cocks *a*, *b* and *c* serve to connect *A* with *B*, *C* and *D*, while the three-way cock *d* allows the communication of *A* with the gas pipe and with the atmosphere, or also the gas pipe with the atmosphere. The U-shaped tube *e* serves as a filter for the gas and contains a piece of cotton wool to retain dirt.

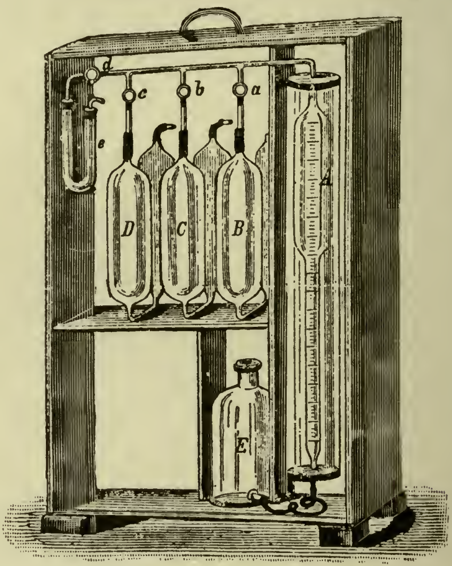

Fig. 24.

Orsat Apparatus.

The vessel *B* is filled with a solution containing 250 grms. of caustic potash in 1 litre of water, and serves for the absorption of carbonic acid.

C is filled with a solution containing 50 grms. of pyrogallol in 1 litre of potash solution of 1·16 specific gravity containing 18 per cent. of potassium hydroxide (KOH). It serves for the absorption of oxygen.

D is filled with a solution of cuprous chloride and in each of the thin glass tubes a spiral of copper wire is placed in order to have always the entire solution in contact with metallic copper. The solution is made by dissolving 200 grms. of cuprous chloride and 250 grms. of ammonium chloride in 750 c.c. of water, and adding previous to use one-third by volume of ammonia of 0·91 specific gravity. If the liquid is not completely clear some more ammonia is added. This solution serves for the absorption of the carbon monoxide.

When analysing the gas the cocks *a*, *b* and *c* are shut, and *A* is brought in communication with the atmosphere. By lifting up the flask *E* the burette *A* is filled to the mark with the acidified water. The cock *d* is shut, and by opening respectively the cocks *a*, *b* and *c* and by raising and lowering alternately *E*, the vessels *B*, *C* and *D* are filled to the marks with their respective liquids.

The tube *e* is connected with the tube supplying the gas to be analysed, and the cock *d* is adjusted so that the gas at first escapes into the atmosphere till it is certain that all the air has been displaced. By moving the cock *d* so that *e* is in communication with *A*, and by lowering *E* the burette is filled with the gas to be analysed; and in order to be sure and have a fine sample it is well to empty the burette again by turning cock *d* to the atmosphere and lifting *E* a couple of times, after which the burette is filled with the sample just to the 100 c.c. mark.[1]

Cock *d* is now shut and *a* is opened, and the gas is transferred into *B*, where it comes in contact with the potash solution, the carbonic acid being absorbed. After sending the gas into the burette again and repeating the transfer a couple of times, all the carbonic acid is certain to be absorbed. The flask *E* is brought near the burette, so that the two levels of the liquid in the burette and in the flask are on the same horizontal line, and then the number of c.c. which the rest of the gas now occupies is read.

The difference is equal to the volume of the carbonic acid absorbed. Cock *a* is shut and *b* opened, and the same operation is repeated to see how much the volume decreases owing to the absorption of oxygen by the alkaline pyrogallol solution, which decrease in volume is equal to the percentage of oxygen. Finally the same operation is repeated with vessel *D*, in which the carbon monoxide is absorbed, the percentage being read on the burette after the residue consisting of nitrogen has been returned.

The succession of absorptions must also be so arranged that first the carbonic acid is absorbed, then the oxygen, and finally the carbon monoxide, as the pyrogallol solution also absorbs carbonic acid and it can only give the real, the oxygen absorption, if all the carbonic acid has first been removed by the potash in the first vessel. For a good absorption it is necessary to introduce the gas into its respective bulk at least twice for the carbonic acid, and three times for each of the other two.

Reliable results can also only be obtained if the acidified water and the solutions are saturated with the gases which are not absorbed by them. Therefore a rapid current of the saturation gas is conducted

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[1] The burette is filled a little over the 100 mark first, after which the level of flask *e* is made just that of the zero mark, and *d* is carefully opened till the gas is just up to the mark. Then one is sure to have 100 c.c. of gas at the pressure of the atmosphere.

through the water before filling it into the burette, while the other solutions are only saturated after they have served a few times for the analysis. The first results obtained with a newly-filled Orsat apparatus are therefore not wholly trustworthy.

The following example shows how the results are recorded :

Reading after absorption by potash 30·8 = 30·8 per cent. carbonic acid
Reading after absorption by pyrogallol,
32·3 − 30·8 = 1·5 per cent. oxygen
Reading after absorption by cuprous
chloride, 32·5 − 32·3 = 0·2 carbon monoxide
Residual nitrogen ... ... 67·5 per cent. nitrogen
100·00

Although the saturation gas is analysed here in a state of complete saturation with water, the results obtained are the same as if they were contained with the quite anhydrous gas, provided the temperature does not change during analysis.

It may be assumed that the tension of the water vapour at the same temperature is identical in the various gases coming into consideration here, and therefore if they are saturated with water at a temperature of 30° C they will contain about 4 per cent. by volume of water vapour. The volume of gas measured is, therefore, 4 per cent. too large, and this is also the case with the remaining gas read every time after the absorptions. All the results are, therefore, 4 per cent. too high, and for this reason the percentage constitution of the gas with its water content is the same as that of the anhydrous gas.

Estimation of the Carbonic Acid Content.

It is not always necessary to analyse the gas completely, since this is only necessary if there is something the matter with the kiln, but in ordinary work the determination of the carbonic acid content is quite sufficient, and can be done in a rapid manner by means of Stammer's tube.

This instrument consists of a burette which is open at the bottom and which bears at the top a small separating funnel connected by a cock. The burette is provided with a scale, the zero commencing at the bottom, while from this point to the cock the contents are just 50 c.c.

The glass cock is a three-way one, which can make communication between the separating funnel and the burette, as well as between the burette and the atmosphere. If the cock is connected with the saturation gas pipe, the gas can be sent immediately into the burette. When analysing the gas the burette is immersed in a wide glass cylinder full of water, the cock is opened and the burette is filled with water. Thereupon connection is made between the burette and the gas pipe; the gas forces out the water, fills the burette, and flows out from the open bottom. The cock is shut, the connection with the gas supply disconnected and the burette raised, so that the water in the cylinder is on the same level as the zero point of the tube, the gas being allowed to rise just to zero by carefully opening the cock, so that just 50 c.c. of the gas are measured.

About 20 c.c. of potash solution of 1·25 – 1·30 specific gravity are poured into the separating funnel and a little run into the burette, which is raised so that its level is somewhat higher than that of the surrounding water.

The caustic potash solution combines with the carbonic acid very rapidly, the water rises in the burette and takes the place of the absorbed gas, and when there is no longer any alteration in the water level, observing the precautions mentioned above, a little more potash solution is added; the lower end of the burette is closed with the thumb; the stopper of the funnel containing the strong potash solution is held fast, and the apparatus shaken vigorously. The carbonic acid is now totally absorbed, so the burette is replaced in the cylinder, and held so that the water is on exactly the same level as that in the cylinder, after which the scale is read. The figure observed multiplied by 2 represents the carbonic acid content of the gas in per cents. by volume.

Detection of Sulphurous Acid and Sulphuretted Hydrogen.

These two gases may occur in the saturation gas when the coke contains sulphur in the form of sulphide. They are never determined quantitatively in the saturation gas, only qualitatively. If the gas contained sulphurous acid, or rather sulphur dioxide, it decolorizes a very dilute starch solution coloured blue with a drop of a solution of iodine in potassium iodide when conducted through it.

Sulphuretted hydrogen is detected by the gas being conducted over a piece of filter-paper, moistened with a solution of lead acetate. A brown colour is the proof of its presence.

VI. SULPHUR.

The sulphur burnt in a special furnace in order to supply the sulphurous acid used in clarification of the cane juices, syrups and molasses is usually the refined article, which, in most cases, is of sufficient purity. The following methods of analysis may be used for testing it.

Methods of Analysis.

Ash.—10 grms. of the finely powdered material are weighed in a tared porcelain crucible, lighted and burnt without heating till the flame is extinguished. The crucible is then heated on a flame, at first gently and later more strongly; and is finally cooled and weighed, the increase in weight representing the ash in 10 grms. of the sulphur.

Sulphur.—Two grms. of the very finely powdered sulphur are treated in a beaker with about 50 c.c. of carbon bisulphide; when most of the sulphur is dissolved, the remainder is allowed to subside and the supernatant liquid is filtered through a dried and tared filter. The residue left in the beaker is again treated with carbon bisulphide, this time with only 25 c.c., and the subsided liquid is again filtered through the same paper, this operation being repeated three times. The undissolved portion is transferred to the filter with carbon bisulphide, the dry filter allowed to stand for half-an-hour, until the solvent has evaporated; after which it is heated for half-an-hour at 100° C in an air-bath. By this heating that part of the sulphur which is insoluble in carbon sulphide is transformed into the soluble modification. After cooling the filter is washed out with carbon bisulphide, and after standing in the air for half-an-hour, it is finally dried in an air-bath at 110° C, and weighed.

The weight of the residue is that of the non-sulphur matter, so that the sulphur is found by subtracting this from the dry matter in the material.

The possible presence of selenium is not taken into consideration here, as this element occurs so infrequently and then only in small quantities.

Moisture.—100 grms. of coarsely powdered sulphur are dried at 100° C to constant weight in an air-bath. As a rule the moisture content is very insignificant in refined sulphur, but since the content of real sulphur is found indirectly, it is mentioned here for the reason that possibly a sample of sulphur might contain an uncommonly large percentage, in which case this would be calculated as sulphur. The water readily evaporates, and therefore the pounding of the sulphur to coarse powder should be done rapidly.

Arsenic.—Arsenic may occur in sulphur in the form of oxide and sulphide, and may be detected qualitatively in the following way: 1 grm. of finely powered sulphur is shaken with 15 drops of ammonia and 2 c.c. of water in a small test-tube, and filtered after half-an-hour. The filtrate is mixed with 30 drops of hydrochloric acid and 15 drops of oxalic acid solution, after which a strip of clean brass foil is dipped into the liquid, which is warmed to 60°–100° C. The presence of arsenic betrays itself by the greyish-black to black colour which the foil assumes.

Arsenic compounds remain in the residue insoluble in carbon sulphide, and can be dissolved therefrom by nitric acid. The quantitative determination is performed in the following way: 25 grms. of the fine sulphur powder are extracted at 70°–80° C with dilute ammonia, after which the liquid is filtered and the sulphur washed out. The filtrate is rendered acid with hydrochloric acid, the arsenic sulphide which had originally been present as such is precipitated, and a current of sulphuretted hydrogen is conducted through the acid liquid in order to precipitate likewise that portion of the arsenic which had been present as oxide. The precipitate is filtered off, washed out, washed into a porcelain dish, evaporated to dryness and dissolved in a few drops of nitric acid. The solution is then transferred to a beaker, rendered alkaline with ammonia, and precipitated with magnesia mixture. The precipitate is filtered 24 hours later through a dried and tared paper, washed with a mixture of three parts of water and one part of ammonia, dried and weighed. The weight of the ammonium magnesium arsenate multiplied by 0·39 represents the weight of arsenic (As) in 25 grms. of sulphur.

A good sulphur should not contain more than 0·5 per cent. of ash and 0·1 per cent. of arsenic.

VII. SULPHUROUS ACID GAS.

Preparation.

The sulphurous acid used in the clarification of cane sugar products is exclusively prepared *in situ* by burning sulphur in a special furnace. The use of compressed liquid sulphur dioxide in cylinders or bombs, which is often met with in the European sugar industry, is not resorted to in the tropics because of the large amount of sulphurous acid employed, and the high expense of the transport of the empty cylinders back to be recharged. Yet if it were possible to use compressed sulphur dioxide in a cane sugar factory, this should be done by preference as it is quite pure, easily handled and very economical; then no sulphur fumes would be wasted which is, unfortunately, the case with sulphur furnaces.

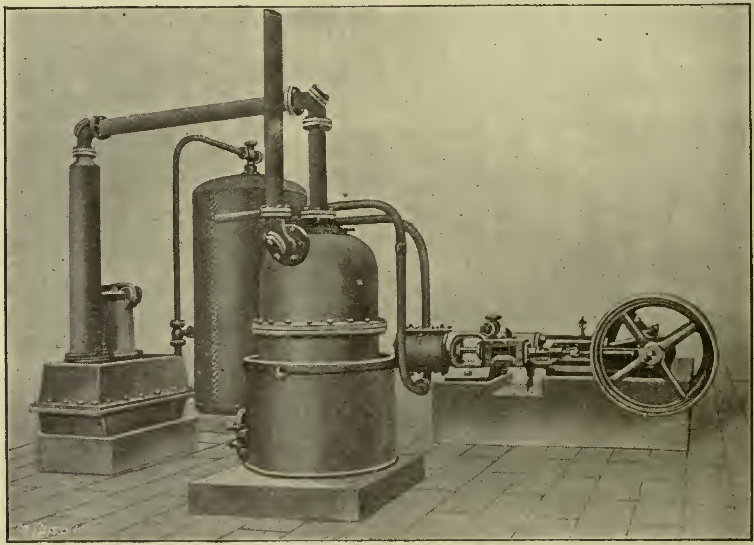

FIG. 25.
Sulphur Furnace.

In most cases the manufacturer makes his own sulphurous acid by burning sulphur in a current of air, which is forced or sucked over the burning sulphur and carried into the sulphitation tanks saturated with the sulphur dioxide gas.

The sulphur furnace (Fig. 25), consists of an iron oven which may be cooled by means of a water-jacket at the top. The pipes through which the gas escapes are also provided with a water-jacket in order to condense the volatilized sulphur, and thus prevent the narrower pipes from getting choked with sublimed sulphur. The sulphur is placed on an iron ladle, which is thrust into the oven after the sulphur is ignited. From time to time the door of the oven is opened to add a fresh supply of sulphur, or a box, shut at the top with a lid and at the bottom with a valve, is placed over the ladle with the burning sulphur. This box is kept full of pieces of sulphur, and now and then the valve is opened to allow a fresh supply to drop into the fire, without admission of too much air. If the sulphur does not burn well this may be remedied by placing a hot iron in it, or by kindling a fire with a fuse, consisting of rope drenched in molten sulphur. Some sulphur ovens are capable of being heated in case the temperature is too low for good and regular burning. In some systems air is forced over the sulphur by an air compressor, while in others, as in the Quarez system, an injector in the discharge pipe sucks off the gases and causes a draught of fresh air for combustion.

It is very important that the air passing over the sulphur be dry, since moist air promotes the formation of sulphuric acid from the sulphurous acid and oxygen of the air, which corrodes the iron oven and the pipes.

The air is dried by passing it through a box provided with trays filled with lime. As soon as the lime is saturated with moisture on one of the trays it is removed and replaced by another one containing fresh lime, so as always to have an abundant supply of hygroscopic material in the current of air. When the air is forced over the sulphur, by means of an air compressor, it is advisable to have this compressor between the dryer and the sulphur oven, for if the dryer is between the compressor and the oven the fire may get extinguished whenever the drying box is opened for changing the trays of lime, the air supply to the oven being then stopped.

When, on the contrary, the sulphur fumes are sucked out of the furnace by an injector, the change of trays does not influence the combustion, for, even if during a few moments the drying of the air is incomplete through the open door allowing undried air to enter, these few moments are not sufficient to cause harm.

The sulphur fumes are not washed before they are conducted into the pipes for the saturators, as thereby oxidation to sulphuric acid might be promoted, but they are conducted through an iron box loosely filled with coke, where, if necessary, some impurities dragged along with the current of gas can be withheld.

The mixture of gases escaping from the sulphur furnace consists of sulphur dioxide, the unused portion of the oxygen, and the nitrogen remaining inactive. Theoretically the sulphur dioxide content can attain 21 per cent. by volume, but in order to ensure a total combustion of the sulphur and prevent entrainment of sulphur vapour, there should be a good excess of air in the furnace, so that in reality the sulphur dioxide content is about 14 per cent. by volume, which may rise to 16 or fall to 12 per cent. without any difficulty being encountered.

Determination of Sulphur Dioxide in the Sulphur Fumes.

The gas from the sulphur furnace is only tested as to its content of sulphur dioxide, which is performed in a way analogous to that of the carbonic acid in the saturation gas.

The sulphurous acid or sulphur dioxide combines with potash to form soluble potash sulphite, so that the decrease in volume represents the amount of sulphurous acid gas in the gas.

The apparatus used in this determination necessarily differs from the one in use in the carbonic acid estimation, because the sulphurous acid is much more soluble in water than the former, and therefore cannot be measured above water.

A very reliable apparatus has been constructed by Léon Landes (Fig. 26). It consists of a burette of just 100 c.c. capacity, which at its narrow end is divided into fifths of a c.c. to a total amount of 34 c.c. The upper end of the burette is provided with a three-way cock and a separating funnel with stopper, while at the bottom a second three-way cock is fitted, which is connected with a flask by means of a rubber tube.

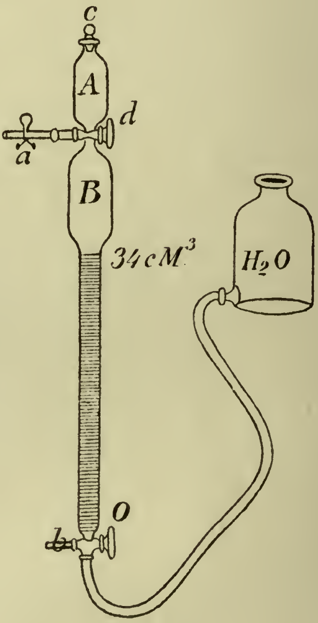

FIG. 26.
Landes' Sulphurous Acid Burette.

Before analysis the burette is washed with alcohol and ether, and fully dried by blowing away the vapours of these volatile liquids with a current of air. The cock *b* is opened in such a way that it is in communication with the flask filled with water. The flask is lifted and the rubber tube is filled with water, which runs out of *b*, the latter being shut as soon as the water comes out of the opening. Next the two three-way cocks are set so that the burette is, on both sides, in communication with the atmosphere; *a* is connected with the pipe supplying the gas to be analysed, which escapes through *b*, until it is certain that the air has been completely replaced by the gas. The cock *d* is now shut and afterwards *b*, the apparatus being disconnected from the gas pipe. Potash solution (a solution of caustic potash of 1·25 specific gravity) is poured into the separating funnel, and some is allowed to run into the burette by carefully opening cock *d*, which at once absorbs the sulphurous acid gas. The burette is removed from its stand and well shaken for a few seconds, after which communication between the burette and the water flask is restored by opening cock *c*. The burette is then replaced in its former vertical position, and after 10 to 15 minutes the level of the water in it is read, taking care to lift the water flask so that both levels are just the same. The decrease of volume in c.c. represents the percentage of sulphurous acid content of the sulphur fumes.

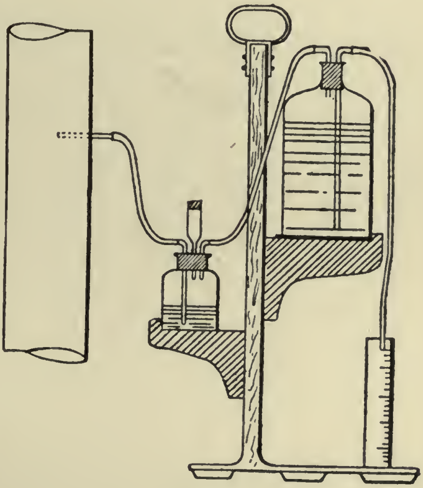

FIG. 27.
Sulphurous acid determination in sulphur fumes.

A second method, which has the merit of being more rapid, is that of Reich improved by Lunge (Fig. 27).

It consists of a wide-mouthed flask, having a content of 250 to 300 c.c., closed by a rubber stopper with three holes, one of which bears a glass tube reaching nearly to the bottom, and through which the gas to be analysed enters. The second hole carries a glass tube, for the exit of the gas, ending near the top; while in the third hole a funnel is inserted, through which the reagent can be added. The gas outlet is connected with an aspirator, while the water flowing out is collected in a measure.

The absorption bottle contains 100 c.c. of boiled and cooled water, to which are added a few drops of starch solution; 10 c.c. of decinormal iodine solution (containing 12·7 grms. of iodine and 20 grms. of potassium iodine per litre) are pipetted into the funnel, and washed with water into the bottle, after which the funnel is closed by a rubber stopper.

The flask is connected securely with the sulphur fumes pipe and with the aspirator, and when it is desired to analyse the gas, a cock in the discharge tube of the aspirator is opened fully and another one in the tube connecting the absorption bottle with the supply tube is opened only a little. The aspirator at once sucks the gas through the iodine solution, while the current is kept at a sufficiently restricted rate that the gas bubbles through slowly enough to see each bubble separately. During the flow of gas the absorption bottle is kept in gentle motion, and the current of gas maintained till the dark blue liquid becomes colourless, owing to the free iodine being combined with the sulphurous acid to hydroiodic acid. As soon as the blue coloration has vanished the cocks are shut, so that the outflow of water is stopped. The quantity of water in the measure is read and the analysis repeated, as one is not always sure that during the first experiment all the air has been replaced by the gas.

From the number of c.c. of water discharged from the aspirator the sulphur dioxide content of the gas is calculated by the formula

$$\frac{1114}{\text{c.c. of water} + 11{\cdot}14} = \text{per cent. } SO_2 \text{ by volume.}$$

This formula is based on the following considerations: 10 c.c. of deci-normal iodine solution oxidize 0·032 grms. of sulphur dioxide, which at 0° C and 760 m.m. pressure occupy a volume of

$$\frac{1000 \times 0{\cdot}032}{2{\cdot}8731} = 11{\cdot}14 \text{ c.c.}$$

This is the quantity of gas absorbed, and the unabsorbed part is equal to the volume of gas that has issued from the aspirator, so that the total volume analysed is $v + 11{\cdot}14$ (v being the number of c.c. which have issued from the aspirator); $v + 11{\cdot}14$ c.c. therefore contain 11·14 c.c. of SO_2, or 100 parts contain

$$\frac{11{\cdot}14 \times 100}{11{\cdot}14 + v} \text{ parts of } SO_2$$

Determination of Acidity in Clarified Juice and in Syrup.

The total acidity of clarified juice and of syrup is determined by the titration of a measured portion of those products with decinormal potassium hydroxide solution.

For the determination of the acid content in clarified juice, 100 c.c. are measured in a porcelain dish, a few drops of phenolphthalein solution added, and deci-normal potash solution added from a burette till the colour of the juice has become pink. For syrup 25 c.c. are diluted to 100 c.c. with water, and treated in the same way.

The acidity is expressed either as acetic acid or as sulphurous acid. In the former case every c.c. of the deci-normal potash is equivalent to 6 mgrms. of acetic acid ($C_2H_4O_2$), and in the latter to 3·2 mgrms. of sulphur dioxide (SO_2).

100 c.c. of clarified juice requiring 10 c.c. for neutralization possess an acidity of 0·06 per cent. expressed as acetic acid or 0·032 as sulphurous acid. The difference in density between juices and water is too small to make an allowance for when analysing juice, while in syrup it may be corrected by dividing the figure by 1·25. As the acidity in the syrup has been determined in 25 c.c. the number of c.c. of deci-normal potash solution must be multiplied by 4 to give the result on 100 c.c.

Instead of such a laboratory titration the acidity can be found by using the Vivien tube. This is a wide glass tube having a narrow neck, which can be conveniently closed with the thumb. It bears the following divisions etched on the glass. Below the zero mark it contains exactly 10 c.c., and above it is divided into 25 degrees, subdivided into tenths of a degree. Every degree occupies a space of 2 c.c., so that the divided capacity measures 50 c.c. Titration is effected by filling the tube with N/100 potassium hydroxide coloured with phenolphthalein to the zero mark and adding the acid juice slowly under the constant movement of the tube, till the pink coloration just disappears. The number of degrees occupied by the juice at that moment is read and recorded.

It is of course feasible, by a very simple calculation, to find the acidity of the juice, but that is seldom done, and only the number of "Vivien degrees" is recorded. Once for all, we state with what degree of acidity in the laboratory the best results are obtained, and after that the number of Vivien degrees which have to be found in the juices and syrups is fixed. Suppose we have found that an acidity of 12 Vivien degrees has proved to yield the best results, then the only thing the attendant of the sulphurous acid saturation of the clarified juice and the syrup has to do is to test occasionally the number of Vivien degrees that his juices possess. When he finds, *e.g.*, the number 10, it means that the juices are too acid, and that he has to shut the valve of the sulphur fumes somewhat; while, if the number exceeds the fixed figure, he has to open it till the juices attain the proper acidity.

Determination of the Sulphurous Acid Content in Juices.

By the determination of the acidity the total amount of free acid is found, and not only that due to the sulphurous acid alone. If it is desired to determine the sulphurous acid alone, the clarified juice and the syrup are titrated with centi-normal iodine solution. 25 c.c. of clarified juice, or 10 c.c. of syrup diluted with its own volume of boiled and cooled water, are pipetted in a porcelain dish, a few drops of starch solution added, and N/100 iodine solution (1·27 grms. of iodine and 5 grms. of potassium iodide to the litre) is added from a burette till the liquid becomes blue. Every c.c. of the iodine solution is equivalent to 0·32 mgrm. of sulphur dioxide, so that the amount of that constituent in grms. per litre may be found by multiplying the number of c.c. employed by 0·0128 in the case of clarified juice, and by 0·032 in the case of syrup. If it is desired to express the amount of sulphur dioxide as a percentage, it suffices to divide the grms. per litre by ten times the specific gravity of the liquid analysed.

As already said, for the determination of the total acid in the juices and syrups it is not always necessary to record the exact amount of sulphur oxide as a percentage, but a conventional degree easily ascertained in the factory may be used, provided in the laboratory it has been decided with what number of degrees the best results are being obtained.

A tube, in shape similar to the Vivien, is employed. The space under the zero mark is 6 c.c., of which the undermost portion of 4·5 c.c. is intended for the juice or the syrup, the second portion of 1·5 c.c. serves for the starch solution, while the tube above the zero mark is divided in 25 degrees, subdivided into fifths of a degree. The value of every degree is 1·4 c.c.

The titration is effected by filling the lowermost portion with the juice or the syrup to be analysed, the second with the starch solution, after which, while gently shaking the tube, centi-normal iodine solution is slowly poured in until the blue coloration remains. The volume of 1 degree being 1·4, the quantity of sulphur dioxide oxidized by the iodine of 1 degree is $1{\cdot}4 \times 0{\cdot}32 = 0{\cdot}45$ mgrm. of SO_2, so that every degree read on the tube is equivalent to 0·1 grm. per litre. As has been said, the calculation is very seldom made, and the instructions to the man-in-charge are to sulphite till the juice or syrup shows a certain number of degrees.

VIII. "BLANKIT" OR SODIUM HYDROSULPHITE.

Sodium hydrosulphite or "Blankit" is a fine white powder, having an acrid odour. When heated on platinum foil it gives off vapours of sulphur dioxide, and leaves behind sodium sulphide. It dissolves easily in water to form an alkaline liquid, which yields white precipitates with barium and calcium chloride, decolorizes ferric chloride by reduction, reduces potassium bichromate to green chromic sulphate; yields a yellow precipitate with lead acetate, a black one with silver and mercury solutions, and decolorizes solutions of iodide in potassium iodide, and of indigo in sulphuric acid. The blue colour of the latter reagent is restored after the hydrosulphite has become oxidized by some reagent or other.

The chemical analysis of the preparation only needs to state whether it still possesses the percentage of real hydrosulphite of 90 per cent., with which it has left the factory, or whether this has diminished in the course of time.

100 mgrms. of dry hydrosulphite are weighed from a weighing bottle into a dry Erlenmeyer flask and a current of hydrogen or nitrogen passed through the flask to displace the air. Next a sufficient quantity of standard ammoniacal copper solution is added to the powder from a burette. The hydrosulphite dissolves, reduces the cupric salt to the cuprous state and thereby decolorizes the copper solution. A few drops of indigo carmine are added, and the titrated copper solution from the burette is poured in till the blue carmine coloration remains.

The copper solution is prepared by dissolving 4·995 grms. of copper sulphate ($CuSO_4$ $5\ H_2O$) in water in a litre flask, rendering alkaline with ammonia and filling up to the mark. The indigo carmine solution is made by dissolving 1 grm. of indigo sodium sulphonate to 1 litre of water.

One c.c. of the ammoniacal copper solution is equivalent to 1·74 mgrms. of $Na_2S_2O_4$, the reaction being represented by the following equation :

$$\underset{499\cdot 5}{2\,CuSO_4\,5\,H_2O} + \underset{174}{Na_2S_2O_4} + 4\,NH_3 = Cu_2SO_4 + Na_2SO_3 + (NH_4)_2SO_3 + (NH_4)_2SO_4 + 3\,H_2O.$$

IX. PHOSPHORIC ACID AND ITS PREPARATIONS.

Constitution.

An analysis of a sample of commercial crude phosphoric acid and two of acid calcium phosphates are as follows :—

Constituents.		Soluble Phosphoric Acid.	Pasty crude Acid Calcium Phosphate.	Pasty crude Acid Calcium Phosphate.
$Ca\,H_4\,P_2\,O_8$	Soluble ...	2·27	28·19	38·18
$Fe_2\,(H_4\,P_2\,O_8)_3$		7·75	12·74	2·63
$H_3\,PO_4$		45·91	18·53	15·89
$Ca\,SO_4$		—	4·93	1·53
$Ca_3\,P_2\,O_8$	Insoluble ...	—	4·44	2·73
$Fe_2\,P_2\,O_8$		—	2·90	6·91
Sand		—	0·73	1·10
Moisture	...	44·07	27·54	31·03
Total	...	100·0	100·0	100·0
Free and half combined $P_2\,O_5$	...	36·95	29·93	27·90

In modern times the rather crude acid calcium phosphate preparations are supplanted by pure mono-calcium phosphate ($Ca\,H_4\,P_2\,O_8$) either in a dry state or as a paste with a varying moisture content.

The newly recommended phosphoric acid preparation, known as "Phospho-gelose", consists of from 25–50 per cent. of kieselguhr and from 75–50 per cent. of technically pure insoluble dicalcium phosphate

($Ca_2 H_2 P_2 O_8$). According to information obtained from the inventor, the proportion of 53 per cent. of either constituent has proved the most favourable for use.

An authentic sample analysed by the author showed this constitution :—

Dicalcium phosphate	44·50
Ferric diphosphate	1·50
Calcium sulphate	2·40
Kieselguhr	44·90
Moisture	6·70
	100·00

An analysis of the phosphoric acid preparations is confined to the determination of the content of free acid, of total phosphoric acid, and of sulphuric acid. The first constituent neutralizes; the second causes a flocculent precipitate; while the sulphuric acid forms gypsum, which is objectionable, and should not be found in any appreciable quantity in the preparations.

Methods of Analysis.

Free Phosphoric Acid.—20 grms. of the well-mixed sample are treated in a litre measuring flask with about 800 c.c. of water, and shaken for half an hour. The liquid is filled up to the mark and then filtered.

100 c.c., equivalent to 2 grms. of the preparation, are transferred to a beaker, and after the addition of a few drops of phenolphthalein solution are titrated with normal sodium hydroxide solution from a burette till a faint pink coloration is produced.

As phenolphthalein show neutrality as soon as the disodium salt is formed, every c.c. of the normal soda solution is equivalent to 0·0355 grm. of phosphorus pentoxide (P_2O_5), or to 0·049 grm. of orthophosphoric acid (H_3PO_4).

Suppose that 20·9 c.c. of normal soda solution were employed, the preparation would contain

$$\frac{20{\cdot}9 \times 0{\cdot}0355}{20} \times 1{,}000 = 37{\cdot}11 \text{ per cent. of } P_2O_5$$

or

$$\frac{20{\cdot}9 \times 0{\cdot}049}{20} \times 1{,}000 = 51{\cdot}20 \text{ per cent. of } H_3PO_4$$

that is as free acid, or possibly as an equivalent quantity of an acid salt.

Total Phosphoric Acid.—25 c.c. of the above mentioned solution of 20 grms. of the preparation in 1 litre are transferred into a beaker, and almost entirely neutralized with ammonia. 50 c.c. of citrate solution[1] are added, and if all turbidity does not disappear at once some more of the solution may be added till a perfectly clear and strongly alkaline liquid is obtained. This is cooled by placing the beaker in cold water, and following this 25 c.c. of magnesia mixture are added slowly with constant stirring. After half an hour about 10 per cent. by volume of ammonia is added and the beaker is left at rest for 12 hours to allow the precipitate of ammonium magnesium phosphate to settle. The addition of the magnesia mixture must, however, take place immediately after the cooling, otherwise calcium phosphate might crystallize out. The precipitate is collected on an ash-free filter, and treated just as the analogous precipitate of ammonium magnesium phosphate in the determination of magnesia in lime (*see* page 116).

The precipitate is finally weighed as magnesium pyrophosphate and by multiplying the weight found by the factor 0·64 that of the quantity of P_2O_5 is calculated.

Sulphuric Acid.—100 c.c. of the solution of 20 grms. of the preparation to 1 litre are precipitated hot with a hot barium chloride solution and the precipitate treated exactly as has been prescribed in the sulphuric acid estimation in the lime. The weight of the barium sulphate multiplied by 0·5828 shows the quantity of gypsum in 2 grms. of the preparation.

Estimation of the Phosphoric Acid Content from the Specific Gravity.

The phosphoric acid content of pure solutions of that acid may be ascertained from their specific gravity, using the table given here. Solutions of acid calcium phosphates cannot be tested in this way, because of their lime-salt content varying so much.

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[1] 150 grms. of citric acid are dissolved in 500 c.c. of water; to this solution 500 c.c. ammonia are added (spec. gravity 0·94) and the mixture diluted to 1500 c.c. The solution may not be used after being kept longer than two months, because it dissolves silica from the glass of the bottle in which it is preserved. As the silica is precipitated with the magnesia, the phosphoric acid content will be found too high if a stale solution has been employed.

Specific gravity of pure solutions of Phosphoric Acid at 28° C. (82·5° F.)

Specific Gravity.	Percentage of the Solution of	
	$H_3 PO_4$.	P_2O_5.
1·0024	1	0·726
1·0079	2	1·452
1·0134	3	2·178
1·0190	4	2·904
1·0246	5	3·630
1·0303	6	4·356
1·0360	7	5·082
1·0419	8	5·808
1·0478	9	6·534
1·0537	10	7·260
1·0597	11	7·986
1·0658	12	8·712
1·0719	13	9·438
1·0781	14	10·164
1·0844	15	10·890
1·0907	16	11·616
1·0970	17	12·342
1·1032	18	13·068
1·1097	19	13·794
1·1163	20	14·520
1·1208	21	15·246
1·1296	22	15·972
1·1364	23	16·698
1·1432	24	17·424
1 1501	25	18·150
1·1571	26	18·876
1·1641	27	19·602
1·1712	28	20 328
1·1784	29	21·054
1·1856	30	21·780
1·1928	31	22·506
1·2000	32	23·232
1 2073	33	23·958
1·2148	34	24·664
1·2224	35	25·410
1·2298	36	26·136
1·2375	37	26·862
1·2453	38	27·588
1·2532	39	28·314
1·2611	40	29·040
1·2698	41	29·766
1·2772	42	30·492
1·2854	43	31·218
1·2936	44	31·944
1·3018	45	32·670
1·3102	46	33·496
1·3186	47	34·222
1·3273	48	34·948
1·3358	49	35·674
1·3445	50	36·400
1·3532	51	37·126
1·3620	52	37·852
1·3718	53	38·578
1·3808	54	39·304
1·3899	55	40·030
1·3980	56	40 756
1·4072	57	41·482
1·4165	58	42·208
1·4259	59	42·034
1·4353	60	43·660

X. SODIUM PHOSPHATE.

This chemical consists of large colourless crystals, withering on contact with the atmosphere owing to loss of water of crystallization, becoming white and dull. Generally the material is of sufficient purity, and the following tests are only given for confirmation. The solution in water of sodium phosphate is alkaline to litmus paper, and gives a yellow precipitate with silver nitrate, which dissolves in nitric acid and also in ammonia.

XI. ANIMAL CHARCOAL OR BONE BLACK.

Constitution.

Animal charcoal or bone black is prepared by carbonizing the large bones of animals out of contact with the atmosphere. In some countries the custom is to carbonize the cleaned bones without their having undergone any preparatory treatment save the removal of flesh and fat, while in others the gelatinous matter is also removed before the carbonization. It is highly probable that the decolorizing power of the char is higher if the gelatine remains, as it appears that the presence of a nitrogenous body plays an important rôle in decolorization.

The composition of a good quality of animal char may be represented by the following analysis :—

Carbon	9·88	per cent.
Calcium phosphate	73·15	,,
Calcium oxide	1·86	,,
Calcium carbonate	7·54	,,
Calcium sulphate	0·08	,,
Magnesium carbonate	0·18	,,
Iron oxide and alumina	0·36	,,
Nitrogen	1·08	,,
Sand	0·25	,,
Moisture	4·88	,,
Undetermined	0·74	,,
	100·00	

Methods of Analysis for Animal Charcoal.

Moisture.—5 or 10 grms. of finely powdered material are dried in an air-bath at 110° C to constant weight and weighed. The difference is moisture. This determination is very important, since new char may absorb great quantities of moisture without any physical alteration.

Carbon and Matter Insoluble in Hydrochloric Acid.—10 grms. of the finely powdered char are boiled for a quarter of an hour with 40 to 50 c.c. of pure concentrated hydrochloric acid in an Erlenmeyer flask covered with a watch-glass. The solution is diluted and filtered through a tared and dried ash-free filter. The whole of the precipitate is collected on the filter, washed till the wash water no longer shows an acid reaction, dried in an air-bath at 110° C and weighed.

The weight of the dry material is that of the carbon and the mineral insoluble matter. The filter and its contents are incinerated in a platinum dish and the weight of the ash previously ascertained subtracted from that of the dry matter.

The difference represents the weight of the carbon in 10 grms. of the char, and the weight of the ash corresponds with the mineral matter insoluble in hydrochloric acid.

Calcium Sulphate.—The filtrate of the former operation together with the wash-waters is heated and precipitated with a hot solution of barium chloride. The precipitate is treated just as has been described under the section *Lime* (page 117), and the weight of the heated precipitate multiplied by the factor 0·4833 represents the weight of the gypsum or calcium sulphate in the 10 grms. of the char employed.

Calcium Sulphite.—10 grms. of the char are treated just as has been described before, with the exception that, together with the hydrochloric acid, a few crystals of potassium chlorate are added to oxidize the sulphite to sulphate. The sulphate is determined again and the difference between the calcium sulphate found before and after oxidation is calculated to calcium sulphite by multiplying the difference by the factor 0·529.

Calcium Carbonate.—The calcium carbonate content is best determined gasometrically by means of Scheibler's calcimeter (Fig. 28). 1·7 grms. of the very finely powdered sample are introduced into the dry flask *A*, together with a hard rubber tube containing hydrochloric acid of 1·12 specific gravity. The level of the water in the burette *C* is brought to the zero mark by the rubber balloon *V* and the cock *P*; the flask *A* is shut with a well-ground glass stopper, which by means of a tube allows communication between the contents of the flask and a thin and flexible rubber bladder placed in flask *B*, this flask in its turn being connected by means of the glass tube *U* with the burette. If by the pressure of the stopper the level of the water in *C* has decreased somewhat, this can be remedied by gently opening cock *q* till the level is again adjusted. By inclining flask *A* so that the hydrochloric acid runs out of the rubber tube and comes in contact with the char, the calcium carbonate is decomposed into calcium chloride and carbon dioxide, which escapes as a gas, causing the bladder to swell and to replace just as much air in the flask *B*, and consequently water in the burette *C*, as the volume evolved. During analysis cock *p* is gently opened to allow water to flow off, taking care that the level in *D* should always remain somewhat higher than in *C*. The flask *A* is well shaken in order to be sure of the total decomposition of the carbonate, and after having waited ten minutes

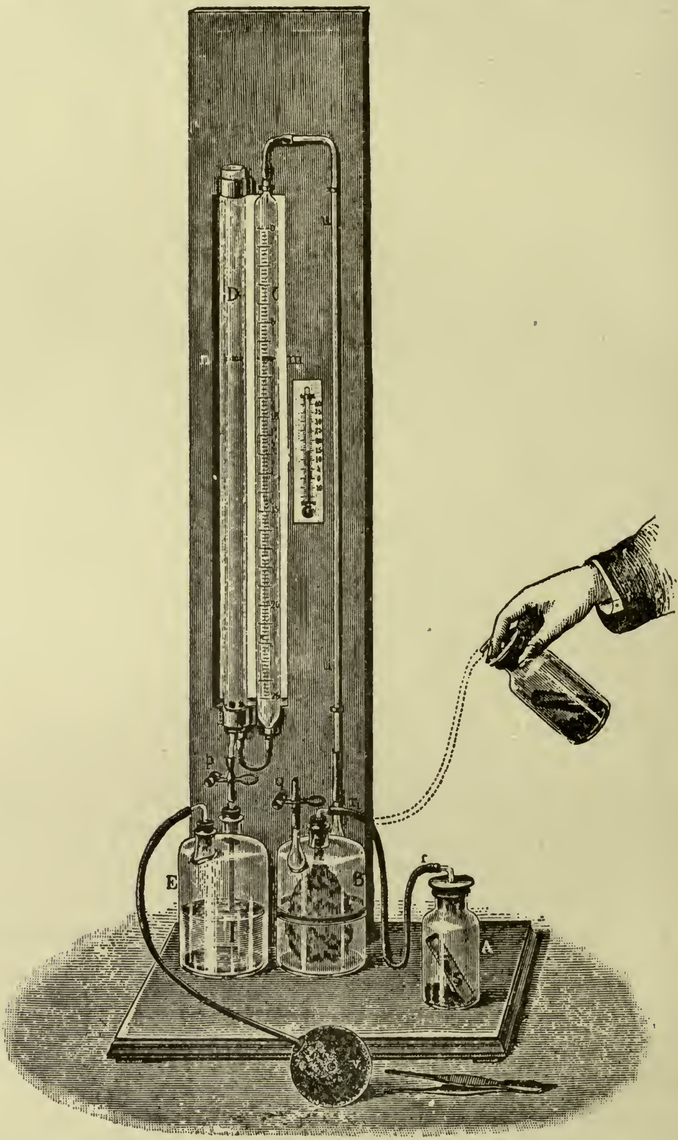

FIG. 28.
Scheibler's Calcimeter.

to allow the temperature, which has risen by the chemical reaction, to fall again, the levels in the tube *C* and *D* are again adjusted to the same height, after which the temperature and the volume are read respectively on the thermometer and the calcimeter. The percentage of calcium carbonate may be obtained from these readings, making use of the table on pages 162 and 163.

Interpretation of the Results of the Chemical Analysis of Animal Charcoal.

The chemical determinations described here have no direct bearing on the quality of the char but chiefly serve for the control of the revivification, as will be shown here.

1. The amount of calcium carbonate in the revivified char must, after being washed with hydrochloric acid and after having been carbonized, be about the same as in new char, *i.e.*, 7 to 8 per cent. The estimation of the amount of calcium carbonate in the spent char will therefore give us a good clue as to the amount of hydrochloric acid to be used.

Suppose that we found at 21° C 8·5° on the calcimeter, then from the readings on the table the calcium carbonate content is found to be:

$$\begin{array}{rcl} 8 & = & 7{\cdot}84 \\ 0{\cdot}5 & = & 0{\cdot}48 \\ \hline 8{\cdot}5 & = & 8{\cdot}32 \text{ per cent.} \end{array}$$

Suppose that we want to reduce that content by 0·5 per cent. by dissolving some of the carbonate in hydrochloric acid; 100 parts of calcium carbonate require 73 parts of pure HCl for solution, so that to lose 0·5 per cent. on 100 parts of the char, 0·365 parts of HCl are required.

In the table on page 169 may be found to how much commercial hydrochloric acid this quantity of HCl is equivalent, so that an easy calculation will show how much of the commercial acid is needed in the factory.

Suppose that the acid contains 33 per cent. of HCl, then for every 100 kg. of the spent char $\frac{0{\cdot}365 \times 100}{33} = 1{\cdot}1$ kg. of the commercial acid are required.

SCHEIBLER'S TABLE FOR THE DETERMINATION OF THE PERCENTAGE A NORMAL WEIGHT OF 1·7 GRMS., AND MEASURING THE CARBONIC

Degrees on the Calcimeter.	Temperature in degrees C.									
	12	13	14	15	16	17	18	19	20	21
1	0·80	0·80	0·79	0·79	0·79	0·78	0·78	0·77	0·77	0·77
2	1·88	1·87	1·86	1·86	1·85	1·84	1·83	1·82	1·81	1·80
3	2·95	2·94	2·92	2·91	2·90	2·89	2·87	2·86	2·85	2·83
4	4·01	4·00	3·98	3·96	3·94	3·93	3·91	3·89	3·87	3·85
5	5·07	5·05	5·03	5·00	4·98	4·96	4·93	4·91	4·89	4·86
6	6·11	6·09	6·06	6·03	6·01	5·98	5·95	5·92	5·89	5·86
7	7·14	7·12	7·09	7·06	7·02	6·99	6·96	6·92	6·89	6·86
8	8·17	8·14	8·11	8·07	8·03	8·00	7·96	7·92	7·88	7·84
9	9·19	9·15	9·12	9·07	9·03	8·99	8·95	8·90	8·86	8·82
10	10·20	10·16	10·12	10·07	10·02	9·98	9·93	9·88	9·83	9·79
11	11·20	11·15	11·10	11·05	11·00	10·95	10·89	10·84	10·79	10·74
12	12·20	12·15	12·09	12·03	11·98	11·92	11·87	11·81	11·75	11·69
13	13·20	13·14	13·08	13·02	12·96	12·90	12·84	12·78	12·72	12·65
14	14·20	14·14	14·07	14·01	13·94	13·88	13·81	13·75	13·68	13·61
15	15·20	15·13	15·06	14·99	14·92	14·85	14·78	14·71	14·64	14·57
16	16·20	16·13	16·05	15·98	15·91	15·83	15·76	15·68	15·61	15·53
17	17·20	17·12	17·04	16·97	16·89	16·81	16·73	16·66	16·57	16·49
18	18·20	18·12	18·03	17·95	17·87	17·79	17·70	17·62	17·53	17·45
19	19·20	19·11	19·03	18·94	18·85	18·76	18·67	18·59	18·50	18·40
20	20·20	20·11	20·02	19·93	19·83	19·74	19·65	19·55	19·46	19·36
21	21·20	21·10	21·01	20·91	20·81	20·72	20·62	20·52	20·42	20·32
22	22·20	22·10	22·00	21·90	21·80	21·70	21·59	21·49	21·39	21·28
23	23·20	23·09	22·99	22·88	22·78	22·67	22·56	22·46	22·35	22·24
24	24·20	24·09	23·98	23·87	23·76	23·65	23·54	23·43	23·31	23·20
25	25·20	25·08	24·97	24·86	24·74	24·63	24·51	24·39	24·28	24·16

OF CALCIUM CARBONATE IN ANIMAL CHARCOAL, USING ACID AT TEMPERATURES BETWEEN 12 AND 30° C.

Temperature in degrees C.									Degrees on the Calcimeter.
22	23	24	25	26	27	28	29	30	
0·76	0·76	0·76	0·75	0·75	0·74	0·74	0·73	0·73	1
1 79	1·79	1·78	1·77	1·76	1·75	1·74	1·73	1·72	2
2·82	2·80	2·79	2·77	2·76	2·74	2·73	2·72	2·71	3
3·83	3·81	3·79	3·77	3·75	3·73	3·71	3·70	3·68	4
4·84	4·81	4·79	4·76	4·74	4·71	4·69	4·67	4·65	5
5·83	5·81	5·78	5·75	5·71	5·68	5·65	5·63	5·61	6
6·82	6·79	6·75	6·72	6·68	6·65	6·61	6·58	6·56	7
7·80	7·76	7·72	7·68	7·64	7·60	7·56	7·53	7·49	8
8·77	8·73	8·68	8·64	8·59	8·55	8·50	8·46	8·42	9
9·73	9·68	9·63	9·58	9·53	9·48	9·43	9·39	9·34	10
10·68	10·63	10·75	10·52	10·46	10·41	10·35	10·30	10·25	11
11·64	11·58	11·52	11·46	11·40	11·33	11·27	11·22	11·16	12
12·59	12·53	12·46	12·40	12·33	12·26	12·20	12·14	12·07	13
13·54	13·48	13·41	13·34	13·26	13·19	13·12	13·05	12·99	14
14·50	14·42	14·35	14·27	14·20	14·12	14·07	13·97	13·90	15
15·45	15·37	15·29	15·21	15·13	15·05	14·97	14·89	14·81	16
16·41	16·32	16·24	16·15	16·07	15·98	15·89	15·81	15·72	17
17·36	17·27	17·18	17·09	17·00	16·91	16·82	16·73	16·63	18
18·31	18·22	18·13	18·03	17·94	17·84	17·74	17·64	17·55	19
19·27	19·17	19·07	18·97	18·87	18·77	18·66	18·56	18·46	20
20·22	20·12	20·01	19·91	19·80	19·70	19·59	19·48	19·37	21
21·17	21·07	20·96	20·85	20·74	20·63	20·51	20·40	20·28	22
22·13	22·02	21·90	21·79	21·67	21·55	21·44	21·31	21·20	23
23·08	22·97	22·85	22·73	22·61	22·48	22·36	22·23	22·11	24
24·04	23·91	23·79	23·67	23·54	23·41	23·28	23·15	23·02	25

As the acid not only acts on the carbonate, but on other bodies as well, it is advisable to take 50 per cent. more than the theoretical quantity found by the analysis.

2. During the filtering process the char absorbs gypsum from the juices and thereby becomes less active, while in addition the sulphates exert an objectionable influence during and after revivification. When carbonizing a spent char containing more than 0·3 per cent. of gypsum, this will act upon the carbon in the retort with the formation of carbon dioxide and calcium sulphide, which latter gives off evil-smelling sulphuretted hydrogen when treated with juices, and may also blacken any metal apparatus with which it comes into contact.

The gypsum can be removed from the char previous to its revivification by heating it with a solution of sodium carbonate, which forms calcium carbonate and soluble sodium sulphate. The latter can be washed away, while the former is removed with hydrochloric acid as stated before.

When revivifying the char the first operation is heating with soda to transform the calcium sulphate into carbonate. The gypsum content is known by analysis, and according to theory 1 part of gypsum requires 0·78 parts of anhydrous sodium carbonate for its treatment, but as an excess is not objectionable it is safe to use twice the calculated value. The char after its treatment with sodium carbonate is washed till the alkaline reaction has disappeared, next it is boiled with the necessary amount of hydrochloric acid for bringing the calcium carbonate content to the original figure, and only then is it dried and carbonized.

Test for the Proper Revivification.—100 grms. of revivified char are boiled for two or three minutes with a solution of sodium hydroxide at 5° Bé., the liquid is filtered, and is examined in a test-tube. If it is nearly colourless the regeneration has been properly carried out, while a dark colour of the filtrate indicates that the revivification has left something to be desired.

Physical Methods for the Determination of the Value of Animal Charcoal.

As has been said already above, the analytical tests to which a char is submitted are very valuable for giving an idea regarding the chemical constitution of the char; but since the action of that material does not depend only on the percentage of constituents it contains, chemical analysis alone is not sufficient. Physical tests have to be resorted to for further information.

Weight of 1 Litre of Char.—The weight of a certain volume of char is to some extent a criterion of its value, since that which weighs most is considered to be the best. A measure just containing 1 litre is filled exactly to the brim with the sample of char from the sacks (without pounding or pulverizing) and weighed. One litre of good char should weigh between 650 and 750 grms., and the best about 725 grms.

It is, of course, not so much the actual weight of the char which decides its quality, but this figure serves as a kind of standard of its hardness, the heaviest char being generally also the hardest and least readily crushed to dust on handling in the cisterns and the washers.

Size of the Pieces of Char.—It is important that the pieces of animal char should be pretty much the same size and also that the char purchased contains none or very little fine dust. These two points are determined by passing the char through a series of sieves of decreasing mesh and noting the percentages of the sample which are retained by the different ones.

Decolorizing Power of the Animal Char.—It is clear that this determination is the most important of all, since the char is only used for decolorizing the sugar juices and its value chiefly depends on its efficiency. Very unfortunately there does not exist a thoroughly reliable method for the determination of that decolorizing power, so that this may be expressed in figures which must be corroborated otherwise.

It has been suggested to mix a weighed portion of char with a solution of some dye-stuff or other to examine how much of the colour is absorbed by the char, but this does not give a standard for the amount of colouring matter which might have been removed from sugar juices by the treatment. Every colouring matter has its peculiar properties, and the absorption by char is by no means identical nor comparable in every case. If it is desired to test the absorptive power of the animal char towards the colouring matter of the juices in a sugar factory, the operation must be done in the laboratory under the same conditions as those which will ultimately obtain in the sugar-house.

A good first molasses, which represents an average of the usual molasses from the factory is selected for the purpose, and a large jarful of it sufficient for the use of a whole campaign is stored.

Next we determine how much of this molasses has to be dissolved in water to 1 litre in order to get a liquid, which after filtration can just be read in a colorimeter of approved design. That figure is noted and if a new batch or a newly revivified char is to be examined a solution of the standard molasses of the coloration fixed once for all is prepared.

100 grms. of the char to be tested are transferred into a 250 c.c. flask, together with 150 c.c. of the molasses solution, and simultaneously a second flask is filled with the same solution. Both flasks are kept immersed for an hour in a water-bath in which the water is on the boil, the flasks being shaken occasionally. After the appointed time the flasks are cooled down and the contents filtered. The liquid from the flask in which the char had been mixed with the solution must be returned to the filter until quite free from black particles.

The two solutions are compared in a colorimeter, and the degree of coloration is expressed by taking that of the molasses without treatment with char as 100. The lower the figure the better the decoloration has been, and the more efficient the animal char.

The same test has once a year to be made with the same molasses and a sample of char which has given good results in the factory, so that the manufacturer can ascertain whether the sample under consideration is a more or less valuable kind. But (as has been said before), the figures obtained by this method are not absolute, and do not allow of any comparison with those found in other laboratories, being useless for comparison with results in other places.

XII. DECOLORIZING CARBONS.

The different high-grade carbons used for decolorizing fats have also found application in the sugar industry, but as the origin and the preparation of these materials are kept secret, nothing can be said of them. We only know of an analysis by Dr. F. Strohmer of "Eponit," which shows the following figures :

Carbon	82·00	per cent.
Hydrogen	1·71	,,
Oxygen	2·64	,,
Nitrogen	0·10	,,
Ash	1·68	,,
Soluble silica and sand	2·46	,,
Moisture	9·41	,,
	100·00	,,

The ash consists of potash 20·58, lime 29·29, and phosphoric acid 2·69, and has an alkaline reaction. On microscopical examination no very striking characteristic could be recognized, the preparation appearing to be a regular amorphous powder. In consideration of this and of the chemical analysis, Dr. Strohmer considers "Eponit" to be a completely carbonized vegetable substance, probably of wood, and comparable to vegetable charcoal.

"Norit" is of an analogous constitution to "Eponit." It contains also a little ash having an alkaline reaction. The carbon content of the preparation is also a high one, being about 90 per cent. in the powder with its common moisture content. It is amorphous and very finely divided, and looks just like powdered vegetable charcoal.

A report upon an analysis of Norit made in Java gave the following particulars: Norit is a very finely powdered, intensely black kind of carbon. On close examination under the microscope, it shows a fibrous structure, as if it had been prepared from sawdust. The fine particles are apt to adhere to each other by mutual attraction, which is especially the case after the material has been moistened.

The chemical composition of a sample was found to be:

Carbon	81·87
Ash	6·03
Moisture	12·10
	100·00

The analysis of the ash is as follows:

Iron oxide	0·70
Calcium oxide	2·11
Magnesium oxide	0·74
Silica	0·67
Phosphoric acid	0·17
Carbonic acid...	0·84
Undetermined matter	0·80
	6·03

Other samples had ash contents of 4·22, 4·29, and 4·25 per cent. respectively, the figure given above therefore being higher than in the other instances.

The carbon content was found to be less owing to the moist tropical Java climate, but the carbon content of the absolutely dry preparation is about 95 per cent.

Chemical analysis fails to give any insight into the decolorizing power of these preparations, so that we do not think it necessary to give methods for the determination of hydrogen or other of the constituents enumerated in Strohmer's analysis.

The best way to test the efficiency of these preparations either before or after their regeneration is to make decolorizing experiments with them on syrups from the factory, just as has been described when treating that point in the section on Animal Charcoal (page 165).

XIII. HYDROCHLORIC ACID.

Hydrochloric acid is used in sugar factories for cleaning the evaporators and for revivifying animal char. The ordinary crude acid (muriatic acid) may be used, but for the latter purpose it should not contain much sulphuric acid, as it would again form gypsum, previously removed by treatment with soda.

The percentage of sulphuric acid is determined in 10 grms. of the hydrochloric acid diluted with 50 c.c. of water with barium chloride, exactly as has been described under the caption *Lime*. The weight of the incinerated barium sulphate multiplied by the factor 0·3432 represents the amount of sulphuric acid (SO_3) in 10 grms. of the hydrochloric acid. That content should not be over 1 per cent.

The percentage of real hydrogen chloride (HCl) in the acid is found by the determination of the specific gravity. The following table contains the specific gravities and the degrees Bé. of the different strengths of the acid with the corresponding percentages. If no great accuracy is desired, the percentage may be derived from the specific gravity without having recourse to the table, by simply dividing the first three decimals of the specific gravity by 5. A hydrochloric acid of 1·166 specific gravity contains, according to the table, 33·2 per cent. of HCl, and on division by 5 = 33·2, or exactly the same figure. With other specific gravities there are small differences between the two, but they are of little consequence.

Degrees Beaumé	Specific gravity at 28° C	100 parts contain HCl.	Degrees Beaumé	Specific gravity at 28° C	100 parts contain HCl.	Degrees Beaumé	Specific gravity at 28° C	100 parts contain HCl.
0	1·000	0·1	11	1·083	16·6	20·5	1·166	33·2
1	1·007	1·5	12	1·091	18·2	21	1·171	34·1
2	1·014	2·9	13	1·100	20·0	21 5	1·175	34·9
3	1·022	4·5	14	1·108	21·7	22	1 180	36·0
4	1·029	5·8	15	1·116	23·3	22·5	1·185	37·1
5	1·036	7·3	16	1·125	25·0	23	1·190	38·2
6	1·044	8·9	17	1·134	26·8	23·5	1·195	39·3
7	1·052	10·5	18	1·143	28·6	24	1·199	40·4
8	1·060	12·1	19	1·152	30·4	24·5	1·205	41·5
9	1·067	13·5	19·5	1·156	31·3	25	1·210	42·7
10	1·075	15·1	20	1·160	32·2			

XIV. CAUSTIC SODA.

Caustic soda is used in sugar factories for cleaning the evaporators and for revivifying animal charcoal or decolorizing carbon, and also for neutralizing an excessive acidity. It arrives in iron drums on which the guaranteed soda content is marked.

This is expressed as "Na_2O per cent." to which likewise is added the equivalent amount of caustic soda properly speaking or sodium hydroxide, the small amount of sodium carbonate which the material sometimes contains being also included.

A guaranteed content of 60 per cent. therefore indicates that the material contains $60 \times \frac{80}{62} = 77{\cdot}4$ per cent. of sodium hydroxide, of which generally a small amount is not hydroxide but carbonate.

The composition of a so-called "65 per cent. caustic soda" may be as follows:

Sodium hydroxide	76·52	per cent.
Sodium carbonate	10·04	,,
Sodium chloride	4·17	,,
Sodium sulphate	2·81	,,
Water	6·46	,,
	100·00	

For the use for which it is intended this material is generally pure enough, but if it is thought necessary to analyse the material the following methods may be used.

Methods of Analysis.

Sampling.—Caustic soda very rapidly attracts moisture and carbonic acid from the atmosphere, for which reason sampling ought to be done very cautiously. The outer layers in the drum may have become damp, and therefore the sample should be taken from the interior. A few pieces are wrapped in paper and beaten to pieces with a hammer, after which some of the smaller ones are selected and put into a well-stoppered bottle.

About 20 grms. of the sample are carefully weighed at once, transferred to a 500 c.c. measuring flask, dissolved in water, filled up to the mark and this solution kept for the determinations. At the same time the amount of the sample for the determination of water is weighed without delay.

Water.—A quantity of the sample weighing between 5 and 10 grms. is carefully weighed in a tared silver or nickel crucible, which is covered with a lid. Heat is gently applied, keeping the crucible covered till the contents are molten, and gradually raised to a dull red for ten minutes. The crucible is left to cool down in a desiccator and weighed. The loss of weight represents the water content in the portion weighed.

Sodium Hydroxide and Carbonate.—25 c.c. of the original solution of about 20 grms. of the caustic soda to 500 c.c. are measured and transferred to a 250 c.c. flask, after which a few drops of phenolphthalein solution are added.

Normal sulphuric acid is added from a burette in such excess that one is quite sure that all the hydroxide and carbonate is saturated. A quantity of 25 c.c. of the normal acid will be quite sufficient. The liquid is boiled during a short time to drive off all carbonic acid which might have been present in the form of carbonate and the excess of acid is titrated back with normal potassium hydroxide solution.

The difference between the c.c. of normal acid and normal alkali is equivalent to the amount of total alkali.

Suppose we had weighed 22·16 grms. of caustic soda and dissolved it in 500 c.c., then the 25 c.c. measured contained $\frac{22{\cdot}16 \times 25}{500} = 1{\cdot}108$ grms. of caustic soda. If for acidification 25 c.c. of acid had been used, and for neutralization 1·7 c.c. of the potash solution, the total alkali in the 1·108 grms. of the caustic soda would have required for the neutralization of the total alkali $25 - 1{\cdot}7 = 23{\cdot}3$ c.c. of normal acid.

1 c.c. of normal acid is equivalent to 0·031 grms of Na_2O, and therefore the 1·108 grms. of the caustic soda contain $23{\cdot}3 \times 0{\cdot}031 = 0{\cdot}721$ grms. of Na_2O or $\frac{0{\cdot}721}{1{\cdot}108} \times 100 = 65{\cdot}1$ per cent of total alkali.

For the separation of the amount present as hydroxide and as carbonate, a second determination is required.

50 c.c. of the original solution are measured into a 200 c.c. flask, diluted with water to about 100 c.c. and heated on a sand-bath. A small quantity of barium chloride solution is added till no further precipitate is formed. The liquid is cooled, filled to the mark, and filtered through a dry filter into a dry cylinder.

The addition of barium chloride decomposes the carbonate, which is precipitated, while the base combines with the chlorine, so that only the hydroxide remains as alkali. The amount thereof is determined by titrating 100 c.c. of the filtrate, equivalent to 1·108 grms. of the caustic soda, with normal sulphuric acid, using phenolphthalein as indicator. Supposing in our example 21·2 c.c. were used, then the volumetric results obtained are as follows:

Neutralization of the total alkali	23·3 c.c.
,, of the hydroxide	21·2 ,,
,, of the carbonate (by difference)	2·1 ,,

1 c.c. of normal acid is equivalent to 0·040 grms. of sodium hydroxide and 0·053 grms. of sodium carbonate. 1·108 grms. of the caustic soda contain:

$21{\cdot}2 \times 0{\cdot}040 = 0{\cdot}848$ grms. of NaOH, or 76·52 per cent.

$2{\cdot}1 \times 0{\cdot}053 = 0{\cdot}111$ grms. of Na_2CO_3, or 10·04 per cent.

Chlorine.—50 c.c. of the original solution are transferred into a flask, rendered faintly acid with nitric acid, after which so much magnesia is added to the liquid, that a small part of it remains undissolved. The liquid is therefore completely neutralized, and is titrated with decinormal silver nitrate with neutral potassium chromate as indicator till the red colour just remains.

1 c.c. of deci-normal silver nitrate is equivalent to 0·00585 grms. of sodium chloride, and if we have used 15·8 c.c. the caustic soda contains

$$\frac{15{\cdot}8 \times 0{\cdot}00585 \times 100}{2{\cdot}216} = 4{\cdot}17 \text{ per cent. of sodium chloride.}$$

Sulphuric Acid.—50 c.c. of the original solution are measured into a beaker, made acid with hydrochloric acid and precipitated hot with a hot solution of barium chloride. The precipitate is treated just as has been described under the section *Lime*, and the weight of the barium sulphate is calculated as sodium sulphate by the factor 0·6089.

As has been said already, it is not necessary to determine all these constituents for use in a sugar factory. If it is required to know the content of a caustic soda solution only approximately, use may be made of the specific gravity table given here.

TABLE SHOWING THE SPECIFIC GRAVITY OF SOLUTIONS OF PURE SODIUM HYDROXIDE AT 28° C. (82·5° F)

Per cent. in the solution.	Specific gravity at 28° C of a content of		Per cent. in the solution.	Specific gravity at 28° C of a content of		Per cent. in the solution.	Specific gravity at 28° C of a content of	
	Na_2O	NaOH		Na_2O	NaOH		Na_2O	NaOH
1	1·012	1·009	21	1·296	1·234	41	1·566	1·443
2	1·027	1·020	22	1·311	1·243	42	1·579	1·453
3	1·040	1·032	23	1·325	1·254	43	1·593	1·464
4	1·055	1·043	24	1·337	1·265	44	1·606	1·474
5	1·071	1·056	25	1·351	1·275	45	1·617	1·484
6	1·086	1·067	26	1·365	1·286	46	1·632	1·495
7	1·101	1·078	27	1·374	1·296	47	1·645	1·503
8	1·116	1·089	28	1·391	1·306	48	1·658	1·514
9	1·129	1·100	29	1·406	1·317	49	1·673	1·524
10	1·142	1·112	30	1·420	1·328	50	1·685	1·535
11	1·158	1·123	31	1·434	1·339	51	1·700	1·545
12	1·172	1·133	32	1·446	1·347	52	1·714	1·555
13	1·187	1·145	33	1·458	1·359	53	1·725	1·565
14	1·200	1·156	34	1·471	1·370	54	1·740	1·575
15	1·215	1·167	35	1·484	1·380	55	1·755	1·586
16	1·230	1·178	36	1·496	1·391	56	1·770	1·596
17	1·241	1·184	37	1·511	1·401	57	1·780	1·606
18	1·254	1·198	38	1·526	1·411	58	1·795	1·617
19	1·266	1·209	39	1 539	1·422	59	1·810	1·628
20	1·281	1·221	40	1·554	1·433	60	1·825	1·638

XV. SODIUM CARBONATE.

Sodium carbonate is used for the revivification of animal char, and also for precipitating lime from juices or syrups.

It is not manufactured in most cane growing countries, and therefore it is exclusively imported in the anhydrous and not in the crystallized state, the freight charges of the crystallized salt with its large water content being much too high.

The analysis of sodium carbonate is carried out on the same lines as that of caustic soda, with this exception, that sampling need not be done with so much precaution, as sodium carbonate attracts neither moisture nor carbonic acid from the atmosphere. It is not necessary to make one solution with which to carry out all the tests, since these can be made with separately weighed portions of the sample.

For the determination of the sodium carbonate content 1 or 2 grms. are weighed out, dissolved in water, boiled with 20 – 40 c.c. of normal acid (using phenolphthalein as indicator), and after boiling titrated back with normal potassium hydroxide, just as has been described in determining the total alkali in caustic soda.

As has been said in that section, 1 c.c. of normal acid is equivalent to 0·053 grms. of sodium carbonate.

Suppose we had boiled 1·5 grm. of the sodium carbonate with 30 c.c. of acid, and 2·2 c.c. of potash solution have been necessary for neutralization, the percentage of anhydrous sodium carbonate would have been $\frac{(30 - 2{\cdot}2) \times 0{\cdot}053}{1{\cdot}5} \times 100 = 98{\cdot}23$.

For the determination of chlorine and sulphuric acid, quantities of 5 grms. are weighed and analysed.

Provided the solutions are pure, the content of the anhydrous and the crystallized sodium carbonate may be derived from the specific gravity of the solutions by making use of the table given here:

Specific Gravity of Solutions of Sodium Carbonate at 28° C (82·5° F)

Specific gravity.	Percentage of the solution of		Specific gravity.	Percentage of the solution of	
	$Na_2CO_3.+10H_2O.$	$Na_2CO_3.$		$Na_2CO_3.+10H_2O.$	$Na_2CO_3.$
1·0038	1	0·370	1·1035	26	9·635
1·0076	2	0·741	1·1076	27	10·005
1·0114	3	1·112	1·1117	28	10·376
1·0153	4	1·482	1·1158	29	10·746
1·0191	5	1·853	1·1200	30	11·118
1·0231	6	2·223	1·1242	31	11·488
1·0270	7	2·594	1·1284	32	11·859
1·0309	8	2·965	1·1326	33	12·230
1·0348	9	3·335	1·1368	34	12·600
1·0388	10	3·706	1·1410	35	12·971
1·0428	11	4·076	1·1452	36	13·341
1·0468	12	4·447	1·1493	37	13·712
1·0508	13	4·817	1·1536	38	14·082
1·0548	14	5·188	1·1578	39	14·453
1·0588	15	5·558	1·1620	40	14·824
1·0628	16	5·929	1·1662	41	15·195
1·0668	17	6·299	1·1704	42	15·566
1·0708	18	6·670	1·1746	43	15·936
1·0748	19	7·041	1·1788	44	16·307
1·0789	20	7·412	1·1830	45	16·677
1·0830	21	7·782	1·1873	46	17·048
1·0871	22	8·153	1·1916	47	17·418
1·0912	23	8·523	1·1959	48	17·789
1·0953	24	8·894	1·2002	49	18·159
1·0994	25	9·264	1·2045	50	18·530

XVI. ULTRAMARINE.

Ultramarine is used for neutralizing any yellow tinge in the colour of white sugar. Its useful effect depends in the first place on the colouring power and fineness of the dye-stuff, while it should not contain matter which gives off sulphuretted hydrogen on being boiled with water.

The tests to which the ultramarine is submitted aim at deciding these points, while the chemical analysis, which is very complicated, owing to the intricate constitution of the ultramarine, although it can of course detect adulterations and admixtures, fails to give information as to the dyeing power and fineness of division. On the other hand, the physical tests are so severe, that an adulterated ultramarine may be rejected as well as a chemically pure product lacking the special qualities required. We shall confine ourselves to the simpler tests, which are quite sufficient for our wants, and omit the very interesting but, for our object, unnecessary methods of chemical analysis.

Degree of Fineness.—10 grms. are mixed in a mortar with 30 to 50 c.c. of water till a homogeneous mixture has been obtained. This is poured into a cylinder of 300 c.c. content and washed completely in till the vessel is quite filled with the mixture. The same thing is done at the same time with a brand of ultramarine blue which has given entire satisfaction in the factory.

After the contents of each have been well shaken, the two cylinders are put side by side and inspected at the end of one hour, two hours, four hours and so on, the rapidity of subsiding, the colour of the supernatant water, and the thickness of the separated subsided layer of dye-stuff being noted at these intervals.

The longer the liquid remains coloured blue, and the less dense the subsided layer, the better is the ultramarine adapted for the blueing of the sugar. This test therefore gives valuable information as to the fineness of the division of the ultramarine.

Colouring power.—0·5 grm. of the ultramarine to be tested is mixed in a mortar with 5 grms. of pure white gypsum, pipe-clay, or barium sulphate, which mixing must continue without pressing too hard, till the mixture is quite homogeneous and does not show either blue or white streaks on rubbing with the pestle. A pinch of the mixture is spread on a piece of paper and rubbed flat with a horn spatula. At the same time a similar mixture is made with a good brand of ultramarine kept as a standard with the same proportion of the white stuff selected and a similar pinch is rubbed flat alongside.

By inspection of the two mixtures, it is possible to see first whether the diluted colour is as intensive as that of the standard and, secondly, whether the tinge is a pure blue one or whether it has a violet or greenish hue. That ultramarine is best which, treated in this way, gives the most intense and the purest blue coloration.

This inspection requires a good but not too strong daylight, and cannot be done by lamplight.

In order to save trouble it is advisable to make, once for all, a series of standards, starting from a good brand of ultramarine and mixing that thoroughly with varying portions of a neutral and inert white material.

100 grms. of calcium carbonate or barium sulphate, or whatever white material is chosen (it being understood that once the choice is made the comparative tests ought always to be made with the same material) are mixed with 10 grms. of the standard blue, and the mixture called 50 per cent. After that a scale is constructed by taking every time 5 grms. of the white material, more or less, and calling the mixtures, *e.g.*, 51, 52, 53, etc., and 49, 48, 47, etc.

53 per cent. = 85 grms. of white material, 10 grms. of ultramarine
52 ,, = 90 ,, ,, ,, ,, ,,
51 ,, = 95 ,, ,, ,, ,, ,,
50 ,, = 100 ,, ,, ,, ,, ,,
49 ,, = 105 ,, ,, ,, ,, ,,
48 ,, = 110 ,, ,, ,, ,, ,,

Mixtures of 1 part of blue to be tested with 10 parts of the same white material used in the construction of the standard may be compared with the standard, and the colouring power of the sample expressed in figures.

Sulphuretted Hydrogen given off by boiling with Water.—50 grms. of ultramarine are treated with water in a distilling flask and boiled. The vapours are conducted through a U-shaped tube with bulbs, in which 25 c.c. of deci-normal iodine solution are measured. In order to prevent loss of iodine the extremity of the tube is closed by a plug of cotton wool soaked in potassium iodide solution. The distillation is continued for a quarter of an hour, after which the iodine solution and the plug of cotton wool are transferred into a beaker and titrated with deci-normal sodium thiosulphate solution, using starch solution as indicator.

1 c.c. of deci-normal thiosulphate solution is equivalent to 1·7 mgrms. of sulphuretted hydrogen.

A good ultramarine should not give off more than 15 mgrms. of sulphuretted hydrogen per 100 grms., therefore the 50 grms. of ultramarine employed should not cause a larger difference than 4·5 c.c. of the deci-normal thiosulphate solution used for the reduction of 25 c.c. of the iodine solution before and after the distillation test.

It is very evident that the water with which the ultramarine is boiled should not contain any trace of acid, and in order to ensure that, water which has just been distilled over a small amount of lime should be used.

Although, as has been said before, the chemical analysis is unable to give the necessary data for the determination of the quality of ultramarine for the purpose for which it is intended here, yet it is necessary to know whether a blue dye, sold as ultramarine, really consists of this matter at all, and for this purpose we give here the following tests of identification.

On being heated on a platinum foil, ultramarine does not change colour nor give off vapours.

When treated with dilute hydrochloric acid, it becomes milk-white and evolves a strong smell of sulphuretted hydrogen.

When heated with a dilute solution of caustic soda, it does not change colour at all.

The presence of adulterations, such as gypsum, barium carbonate, barium sulphate, etc., is only to be detected after a rather difficult analysis, which is best left to expert analysts.

XVII. INDANTHRENE.

Indanthrene is an artificial, synthetic dye-stuff, of which the scientific name is dianthrachinonyl-N-dihydroazin.

On being heated on platinum foil it chars and gives off inflammable vapour, leaving behind a carbonaceous mass, which totally disappears on continued heating.

Neither acids, nor caustic alkalis, even on boiling in high concentration, change its colour.

Indanthrene is insoluble in water, but remains in a suspended state from which it is thrown down as a flocculent precipitate by acids.

When treated with strong reducing agents such as stannous oxide and sodium hydroxide, indanthrene passes over into soluble leuco-indanthrene, which also has a blue colour, and from this solution the original insoluble matter will deposit gradually after oxidation on exposure to the atmosphere.

The dyeing power of indanthrene is best ascertained by shaking a weighed quantity of the sample with a large amount of water, and by comparing the intensity of the colour with one obtained by treating, in the same way, a similar quantity of a sample which has given entire satisfaction.

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