clearly defined for us by the Lords of the Treasury. The first duty of Government is to repudiate at once and for ever such a fatal doctrine, and to realize while there is yet time that all our main industries are in imminent peril. The attack will be sharp and sudden. Woe to this country if our Government is not prepared to meet and repel it. The military authorities gave them fair warning, which they disregarded and are now paying the penalty. A similar warning now comes from those who know in the industrial sphere. If this is also disregarded we may look for the undoing of Imperial Britain.

G. M.

The Scientific Control of a Rum Distillery.

By F. I. SCARD, F.I.C.

The scientific control of a rum distillery differs in many respects from that of the sugar factory, of which the distillery is usually an adjunct. In the sugar factory the main object of the control is to ascertain whether the recovery of the sucrose of the cane as a merchantable sugar is as high as it should be, and if not to indicate the precise spot where the manufacturing fault is situated. The question of flavour, with the exception of fancy grocery sugars such as "Demerara" is a matter of no moment. The obtaining of as much of the sucrose as possible in a suitable form is the great desideratum, and no account is taken of the taste or smell of the sugar if unobjectionable so long as, in the case of whites, it is pleasing to the eye, and in that of refining sugars, there is the requisite polarization. The microscope practically plays no part in the control, unless it is to confirm the presence of *Leuconostoc mesenteriodes* or kindred organisms which sometimes occur with the unheated juice and the indication of which is readily noticeable without the aid of this instrument.

In the case of a rum distillery the position is very different. It is not the sucrose alone which has to be accounted for in the course of manufacture, but all the fermentable sugars, glucose, and invert sugar, as well as sucrose, which find their way to the distillery. The object of the operations of a distillery is not to separate and obtain these sugars as such, but as a product formed from them by biological means before its actual separation by distillation, a product in which the flavour is a vital point in its value. The microscope thus plays an important part in the control of a distillery.

For the control of a rum distillery what is wanted is:

1. The knowledge of the amount of sweets entering the distillery.

2. The record of the attenuation of the wash, so that the amount of spirit made may be determined.

3. The knowledge of the amount of spirit separated from the wash by the still, so that the relation of the amount of spirit made to the sweets entering the distillery may be determined.

4. The examination of the lees, or exhausted wash, for spirit, as a check upon the working of the still.

5. The estimation of the amount of "esters" or flavouring ethers of the spirit; the determination of the obscuration, or amount of spirit put out of action to the ordinary hydrometer test, an important item when the rum is shipped to the United Kingdom; and the examination of the rum for faults.

I. The customary method of working in a distillery is to set up the wash with measured quantities of whatever is going to be utilized in the factory for fer-

mentation purposes—molasses, "skimmings," the "bottoms" of scum tanks, and in some cases even raw juice. These are mixed, either in a mixing cistern or in the vat itself, with water to give the required gravity, in the former case being transferred to the fermenting vat as the mixing progresses. In either of these procedures, the recording of the quantity of sweets entering the distillery is much simplified, as the composition of the wash in this respect and the measured content of the vat at once give the required data for calculation.

As regards the method of analysis required, in this case the polariscope is best omitted. In molasses, double polarization by the Clerget method is necessary to give accurate results, and as all the sugar has to be expressed in terms of glucose, it is better to invert the wash and estimate its reducing power by means of the copper test, thus obtaining the expression of the "sweets" at once as glucose.

In order to do this successfully, the wash must be clarified with acetate of lead, and not with the basic acetate, which would carry down any levulose of invert sugar that chances to be present, and the excess of lead precipitated with sulphate of soda. 100 c.c. of wash can be taken, 10 c.c. of a saturated solution of acetate of lead added, and the mixture well shaken, 10 c.c. of a saturated solution of sulphate of soda is then added, and the 120 c.c. thus obtained filtered. Of the filtrate 60 c.c. are taken, inverted with hydrochloric acid in the usual way, and made up to 500 c.c. This represents a dilution of 10 times. Titration is now effected either with the ordinary Fehling's solution or else with Pavy's modification of it. The latter is to be preferred, as there is, with its use, no error from the production of ammonia through decomposition of nitrogenous bodies by the excess of alkali, and also because the absence of precipitate renders it much more easy to ascertain the precise point of decoloration. 100 c.c. of Pavy has to be used; this quantity has the same glucose value as 10 c.c. of Fehling, but with greater depth of colour, which makes the whole operation more delicate.

It sometimes happens that the wash is not set up all at once, but that fermentation is allowed, purposely, to start before the set is completed, being gradually fed with "sweets" until the desired charge is obtained. In this case the constituents of the wash must be measured separately, and the sweets determined separately too.

It is, of course, absolutely necessary, for the records to be of value, that the measurements should be accurate, and if the vat charges are taken on the basis of calculation, the entire charge should be reckoned and no allowance made for "bottoms" which are, of course, cleaned out before the next charge is made.

If the above directions are followed, the quantity of sweets, expressed as glucose, going to the distillery is obtained, and this quantity has to be accounted for in the subsequent stages of the process.

2. The next stage is to ascertain, from the degree of fermentation of the wash, the quantity of spirit that has been formed.

To this end the specific gravity of the wash is carefully observed directly the setting of the vat charge is complete, and daily records of it kept until the reduction in gravity has ceased, the sampled wash being cooled to the original temperature in each instance before the specific gravity is taken. The ultimate specific gravity is then compared with that of the wash when set up, and the difference constitutes the degrees of attenuation of the wash. Thus, if the wash was set up at 1·060, and fermented until the specific gravity had sunk to 1·010, the attenuation would be 50 degrees.

The attenuation having been observed, the amount of alcohol found is at once indicated. Every 5 degrees of attenuation represents for practical purposes 0·60

gallon of absolute alcohol per 100 gallons of wash. In the above instance, therefore, the amount of alcohol produced per 100 gallons of wash would be $\frac{50}{5} \times 0{\cdot}60 = 6{\cdot}0$ gallons.

This figure is, of course, subject to slight variation according to the extent to which other bodies than alcohol are produced from the sugars during fermentation, but it may be taken as a good working basis for not only determining the quantity of the various fermentations, but also for checking the subsequent work of the still.

Of course, if the wash is set up intermittently, the charge being gradually completed while fermentation goes on, this portion of the control cannot be carried out. But this practice is rare, and bad in itself, and need not be considered.

Means are now afforded of comparing the amount of alcohol formed with the quantity of "sweets" used to set up the wash. The theoretical quantity of glucose required to make a gallon of absolute alcohol at 60° F. is 15·52 lbs., or 15·28 lbs. per gallon at 80° F. The attenuation gives the amount of alcohol formed, which can thus be compared with the quantity the sweets in the wash are capable of producing. Thus, if the wash contained 11 grms. of sweets in 100 c.c., 100 gallons would contain 110 lbs., which, at 15·52 lbs. to the gallon of alcohol would produce 7·7 gallons. The attenuation shows, say, 6·5 gallons of alcohol per 100 gallons of wash, the recovery as alcohol in the fermenting loft has, therefore, been $\frac{6{\cdot}5 \times 100}{7{\cdot}7} = 84{\cdot}4$ per cent.

As already mentioned, the microscope plays an important part in the control of the fermenting loft. The great enemy to fermentation is the putrefactive, bacillus and the wash requires to be constantly examined for the presence of these organism. A few are invariably present, but, if the condition of the wash is favourable to their development, the yeast plant is soon smothered, and there is nothing else to be done but to clean up the distillery in every detail. It is as well also to keep a microscopic eye on the yeast plant, to see if it is developing properly, and at the same time to look out for moulds or other organisms inimical to the yield of alcohol.

(*c*) The figures showing the amount of alcohol produced in the fermentation of the wash can now be utilized for the purpose of checking the working of the still.

The number of gallons going to the still in the form of wash during the week is recorded, together with the amount of alcohol received from it. These should agree within 5 per cent. with a pot still and 1 per cent. with a continuous still. The lees, or spent wash, should also be examined for alcohol by distillation, daily in the case of a continuous still, and from every distillation with a pot still, to see if any alcohol is escaping in this way. 250 c.c. should be taken and 50 c.c. distilled off, the gravity of which is taken with a specific gravity bottle, and corrected for temperature, when any loss of alcohol will be at once discovered.

An additional check on the working of a still is afforded in the case of pot stills by a curve of running strengths being obtained from time to time. The strength of the spirit is taken every five or ten gallons according to the power of the still, and a curve plotted out from these data, which will be characteristic of the work of the still, and any departure from which will at once indicate some fault in the still itself. It may be remarked incidentally that the better the work of a still the steeper will be the curve of strengths.

By the above methods the following information has been obtained :—

1. The amount of sweets, as glucose, entering the distillery.
2. The amount of alcohol produced in the fermentation.

3. The amount of alcohol, as rum, actually obtained.

Thus a satisfactory check is obtained upon the quantitative performances of the distillery.

(*d*) The rum having been made, coloured and reduced, if necessary, to the proper strength, there now remain three operations by which indications are obtained of its quality, the information from which is essential if a level of quality is to be maintained, viz., the determination of the amount of "esters" present, the determination of the degree of obscuration, and the examination for faults.

The "esters," or ethereal salts, which impart the flavour and especially stimulating properties to rum are estimated as follows:—

100 c.c. of the rum is distilled until 70 c.c. have passed over. In the distillation the acidity is carefully neutralised with soda. 20 c.c. of decinormal soda are then added, and the mixture kept on the gentle boil in a flask fitted with a reflux condenser for one hour, using small fragments of pumice stone to prevent bumping. The excess of soda is then titrated back with decinormal acid, and the difference between the 20 c.c. of soda used and the number of c.c. of decinormal acid registered will give the quantity of "esters" present. These are expressed in terms of ethyl acetate for 100,000 parts of alcohol in the rum.

The obscuration, or the extent to which the strength of the spirit as taken by the hydrometer is veiled by the colour used to colour the rum for the market is an important item, inasmuch as the Customs duty on rum in the United Kingdom is assessed on the actual, and not apparent, spirit present, which the retailers' limit of dilution is checked by the hydrometer.

In order to ascertain the amount of spirit obscured, the following is a reliable and simple method, and preferable to the distillation method in the case of strong spirits like rum. The specific gravity of the coloured spirit is taken in a specific gravity bottle, or by Sikes' tables, if the Sikes' hydrometer is used. 100 c.c. are then taken and evaporated until all the spirit has been driven off, i.e., when the residue has reached a syrupy consistency. The residue is now dissolved in water, and made up accurately to 100 c.c. at the same temperature at which the gravity of the coloured spirit was obtained. The specific gravity is now taken. The decimal part of the gravity is then subtracted from the gravity of the coloured spirit, the remainder giving the gravity of the spirit without the colour. From this gravity the quantity of alcohol present can be obtained by reference to tables. If Sikes' tables are used, the process is much simplified, but, if not, the gravity thus obtained can be corrected for temperature, so that some of the recognized tables may be used, by adding or subtracting as the case may be, 0·0005 for each degree Fahrenheit. Thus, if the original gravity was 0·857 and the gravity of the non-alcoholic portion 1·004, the gravity of the alcoholic portion will be 0·853. If this is at 80° F., the addition of 20 × 0·0005 or 0·010 gives the gravity of the alcoholic portion at 60°. The original gravity is corrected in the same manner, and the proportion of alcohol in the coloured and uncoloured rum is thus obtained. With Sikes' tables, of course, the matter is much simplified.

The obscuration is properly expressed in terms of proof spirit by volume obscured. Thus, if 100 parts of the coloured spirit were found to contain 140 parts of proof spirit, and the test showed an actual presence of 143 parts of proof spirit, the obscuration would be $\frac{3 \times 100}{143} = 2{\cdot}1$ per cent.

Faults in rum are found by the following test. A portion of the coloured rum taken from the cask before shipment is diluted with twice its volume of distilled water if it is strong rum of the Demerara description, or with an equal

volume if of the weaker Jamaica kind. It is then placed in a small cylinder covered over with a glass plate, and allowed to stand for 24 hours. If at the end of this period there is no appearance of cloudiness the rum is free from "faults." If a cloudiness appears it may be due to:—

(1) Resinous matter from the wood of the cask;

(2) A precipitate from too-highly burnt colour;

(3) The presence of low bodies of the fusel oil class which should have been kept back in the low wines.

The foregoing forms a reliable method for the scientific control of a rum distillery. From the nature of the operations of a distillery, involving as they do a complex subject like fermentation, the control does not admit of the comparative exactitude of sugar factory control. The two great objects of the rum making are to get as much alcohol from the sweets used, and to secure that the rum is of good flavour and free from faults, and if the chemist in charge of the distillery follows the preceding directions carefully, and brings to bear brains and intelligence on the subject on the lines laid down, so that the facts thus obtained are properly utilized, there should be no difficulty in the satisfactory technical administration of the distillery under his control.

The Clarification of Cane Juice in the Manufacture of Raw Sugar.

By NOËL DEERR.

In the following contribution some attempt is made to put the process of the clarification[1] of cane juice, as carried out in the manufacture of raw sugar, on a quantitative basis and to remove it from the position of an art or craft. As carried out in the many factories that the writer has visited, the quantity of lime added seems to depend on the caprice of the operator in charge combined with his "clinical" observations of the colour of the juice, the rate of settling, the quality of the filter-cake formed. The writer has never seen the operation carried out under such definite chemical control as is followed in the carbonatation of juices, and GEERLIGS[2] indeed hints at the impossibility of such careful work due to the constantly changing nature of the raw material. The capricious and theoretical nature of the operation as generally carried out is indicated by PECK[3] who writes: "For example in Hawaii, it is the opinion of some of the sugar boilers that the Yellow Caledonia cane should always be boiled slightly under-limed or acid, while Rose Bamboo, to give good work, must be neutral or even slightly alkaline." In Demerara where the writer believes that the art of sugar making is highly developed, the sugar boilers in the mills in which he was formerly engaged used to vary the lime according to how the juice "cut" on being allowed to settle in glass containers. If however any of these craftsmen were asked to write a detailed account of what they meant by the juice "cutting" they would probably fail just as much as the most skilful smith would be unable to describe exactly what he meant by a "welding heat."

[1] Under the term "clarification," GEERLIGS includes (*a*) defecation with smaller quantities of lime and (*b*) the carbonatation process with large quantities of lime. The term "clarification" as used here is synonymous with defecation as used by GEERLIGS.

[2] "Cane sugar and its manufacture," page 131. [3] Haw. Exp. Rep. 18.