Some nitrogen experiments were conducted on one of them. But there were no indications that the use of nitrogen produced any profitable increase of yield.

The two other plantations were concerned with phosphoric acid and potash experiments. In the case in which 100 lbs. phosphate was applied as basic slag, an increase of 3½ tons of cane and 1100 lbs. sucrose was noted in the yield. In the other case the application of 50 lbs. of potash as sulphate led to an increased yield of 500 lbs. sucrose, and the application of 100 lbs. led to an increased yield of 750 lbs. sucrose per acre.

RESEARCHES ON JAMAICA AND ARTIFICIAL RUM.

By Dr. Karl Micko,

Director der Staatlichen Untersuchungsanstalt für Lebensmittel in Graz.

I.—Examination of Jamaica and Artificial Rum.

Jamaica rum is one of the most valuable and highly esteemed spirits of commerce,—a distinction which it owes to its characteristic and inimitable flavour. As it is produced in the tropics it is evident that duty and transport charges raise its original cost to a high figure on the European market. The concentrated rum, known as "original rum," is too dear for the ordinary consumer; it is therefore general for the retailing trade to break down the original rum, and by so doing the price falls in proportion to the degree of dilution effected. With many well-established firms it is the usual custom to express the content of the broken spirit in degrees of the dilution and to fix the price accordingly. Generally, the original rum is broken down to different dilutions for the convenience of the smaller dealers. It is, however, preferable for the merchant to import the original rum and break it down himself. In this way he is able to control the dilution, and to be certain of the true content of the diluted spirit in original rum; moreover, economy is thus effected, for charges for dilution and other expenses are saved.

In the case of many kinds of rum it is necessary to break down with spirit, for when in the concentrated condition their flavour is not always apparent. It is a peculiarity of certain rums that the fine aroma is only developed after breaking down; and this was instanced quite recently in our laboratory in the case of a sample of Jamaica rum for which a high price had been paid, but which when added to tea was stated to have a disagreeable taste. We were, however, soon convinced that this was such an example, and that the fine flavour was only hidden, for on breaking the sample down with 60 per cent. spirit the rich aroma and taste of genuine Jamaica rum was developed.

Since genuine Jamaica rum is a costly spirit, it is only to be expected that many attempts are made to imitate it. There are

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numerous receipts for the cheap manufacture of rum in Europe from molasses; but although it is known that the esters of formic, acetic, butyric, capric, and other acids occur in Jamaica rum, it has been impossible up to the present to make a spirit even approaching the genuine article. How far such attempts have been from successful may be judged by the fact that a rum expert has no difficulty at all in identifying an artificial rum by its flavour and aroma. There are upon the market many spirits which have been imitated much more successfully than Jamaica rum. Brandy, for example, is now imitated with such skill that the figures obtained by chemical analysis do not always indicate the fictitious article. Indeed, a brandy can be prepared to give analytical values the same as those found from a genuine sample; and brandy experts are now in no way so certain of judgment as formerly. The rum taster, on the contrary, has a much easier task; the analyst, moreover, can not only readily differentiate the genuine from the artificial product, but is in the position to be able to detect what might easily escape the expert, namely, the admixture of even small amounts of genuine Jamaica rum with an artificial spirit.

The reply to the question as to why it has not been found possible after so many attempts to even approximately imitate the peculiarly fine aroma and taste of Jamaica rum is that it is characterized by a special flavouring constituent, which is not to be found in the best rums made in Europe, nor in the artificially made product. The flavour and aroma of potable spirits is not derived from one but from a number of different bodies; this is true of Jamaica rum, but the basis of its characteristic flavour is an aromatic constituent which is peculiar to it alone.

This constituent can readily be separated by fractional distillation, even when present in small amounts. If Jamaica rum is distilled, a simple tubular condenser being used, and the distillate collected in eight fractions, the first four are not in any way specific of the genuine rum. The peculiar flavouring constituent comes over mostly in the fifth and sixth fractions; towards the end of the distillation the amount of this body gradually decreases so that in the eighth fraction little or none is present. Generally most of it comes over in the sixth, but the alcohol content and method of distillation have, of course, an influence in determining the particular fraction. Concentration to a definite fraction can obviously be more readily effected by using a still-head.

Together with the flavouring constituent a characteristic body of a terpene-like odour also comes over. That terpenes occur in brandy has been pointed out by K. Windisch in his well-known work on the subject,* and he has expressed the opinion that a certain terpene or terpene hydrate is also present in rum and may contribute

* Arbeiten aus dem Kaiserl. Gesundheitsamte, 1893, 8, 279.

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largely to its characteristic flavour. Whether this particular terpene body is peculiar only to Jamaica rum we are unable to say with certainty, but we can at any rate assert that it is not the principal distinctive substance of Jamaica rum. It is always present in Jamaica rum together with the other flavouring constituents, and we have never found it in the artificial product.

Besides the flavouring bodies, other substances which are less volatile come over; amongst these are certain resinous bodies which partly dissolve in sodium hydroxide, from which they can afterwards be precipitated by the addition of acids.

As in the case of other potable spirits, aldehydes and volatile acids are found in Jamaica rum. The content in these bodies is subject to very large variations, and here the sophisticator has an opportunity of adulterating Jamaica rum with artificial spirit without surpassing the limits generally found by analysis. The mixing of artificial rum with original rum cannot be practised to any great extent, because by so doing the true flavour is decreased, and the value of the spirit consequently diminished. The adulteration of broken Jamaica rum with artificial rum, to the contrary, is often done. Of eleven samples which we tested for aldehydes, all were found to give distinct reactions. As we shall see further on, artificial rum can have as high a volatile acidity as Jamaica rum.

The difficulty of judging rum on the ground of its analytical figures is generally caused by the fact that these figures are incapable of expressing the distinctive feature of Jamaica rum, namely the presence of its peculiar flavouring substance. There are, however, certain qualitative tests for Jamaica rum, and what is not shown by a chemical analysis can without difficulty be detected by a trained sense of smell. The usual analytical figures can, nevertheless, corroborate the judgment of a rum, and information of much value can be learnt from them.

The samples which we have examined were: Original Jamaica rum; Jamaica rum broken down with dilute alcohol; Jamaica rum mixed with artificial rum; and artificial rum.

Original Jamaica rum was not often examined, because the sale of this article is confined to special firms and it is only occasionally that customers require it tested. The spirit marked "Jamaica Rum" is as a rule broken down with dilute alcohol, and is sold in this form to the customer by all firms dealing with it; it is only to be expected that here it is necessary to exert a careful chemical control.

Since genuine Jamaica is the most expensive rum of commerce it is not surprising that the sophisticator marks his product "Jamaica Rum"; it is, moreover, not beneath him to apply this title to a product which is nothing more than artificial spirit. This is borne out by the table of analytical results which is given below.

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	Mark.	Sp. gr. at 15·5°C.	Alcohol per cent. by vol.	Volatile Acids(as acetic acid) grms. per 100 cc. rum.	Esters (as ethyl acetate) grms. per 100 c.c. rum.	Flavouring Constituent of genuine Jamaica Rum.	Flavouring Substances, foreign to Jamaica Rum.	Coal Tar Dye Stuffs.	Taste and Aroma.	Remarks.
1	Finest Old Jamaica Rum	0·9440	44·9	0·0036	0·070	None.	Present (vanillin)	Absent.	Of artificial rum	An artificial rum
2	Rhum vieux de la Jamaique	0·9287	53·1	0·0036	0·099	Traces.	,, ,,	Present.	,, ,,	An artificial rum, with small amount of Jamaica rum
3	Rhum de la Jamaique..	0·9005	65·8	0·0036	0·123	Distinct traces	Present.	,,	Not of pure Jamaica rum	Artificial rum and Jamaica rum
4	Finest Old Jamaica Rum	0·9448	44·5	0·0110	0·058	Traces.	Present (vanillin).	Absent.	Of artificial rum	Artificial rum, with small proportion of Jamaica rum
5	Finest Old Jamaica Rum	0·9202	57·1	0·0110	0·065	,,	,, ,,	,,	,, ,,	ditto
6	Old Jamaica Rum, Kingston	0·9100	61·7	0·0110	0·090	,,	Present (cassia oil)	,,	,, ,,	ditto
7	Finest Old Jamaica Rum	0·9452	44·2	0·0280	0·071	None.	Present (vanillin).	,,	,, ,,	Artificial rum
8	Old Jamaica Rum, Kingston..	0·9212	56 6	0·0096	0·062	,,	,, ,,	Present.	,, ,,	ditto
9	Finest Old Jamaica Rum	0·9361	49·3	0·0024	0·040	,,	,, ,,	Absent.	,, ,,	ditto
10	Jamaica Rum	0·9160	59·1	0·0144	0·151	,,	Present (vanillin and cassia oil).	,,	,, ,,	ditto
11	Jamaica Rum..	0·9273	53·7	0·0015	0·141	Small amount.	Present (vanillin).	Present.	Not of pure Jamaica rum	Artificial rum, with some Jamaica rum
12	Jamaica Rum	0·9243	55·2	0·0200	0·198	Distinct traces	,, ,,	,,	ditto	Artificial rum and Jamaica rum
13	Genuine Jamaica Rum	0·9337	50·2	0·0195	0·092	None.	,, ,,	Absent.	Of artificial rum	Artificial rum
14	Old Jamaica Rum .. .	0·9284	53·2	0·0180	0·077	Small amount.	,, ,,	Present.	Not of pure Jamaica rum	Artificial rum, with some Jamaica rum
15	Old Jamaica Tea Rum.	0·9221	56·2	0·0054	0·162	Traces.	,, ,,	,,	Of artificial rum	Artificial rum, with small amounts of Jamaica rum

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16	Superfine and concentrated Jamaica Rum	0·9618	65·3	0·0052	0·052	Small amount	..	..	Absent	Not of pure Jamaica rum	Artificial rum, with some Jamaica rum
17	Black Head Rum, Kingston, Jamaica	0·9347	50·0	0·0051	0·018	Present.	..	..	..	ditto	Artificial rum, with Jamaica rum
18	Finest Old Jamaica Rum	0·9341	50·3	0·0063	0·019	,,	,,	,,	,,	ditto	ditto
19	Genuine Jamaica Rum.	0·9350	49·9	0·0064	0·019	,,	,,	,,	,,	ditto	ditto
20	Black Head Rum, Kingston, Jamaica	0·9526	39·7	0·0144	0·117	None.	,,	,,	,,	Of artificial rum	Artificial rum
21	Original Jamaica Rum.	0·9083	62·5	0·0110	0·077	Very distinct.	Absent.		Present.	Of broken Jamaica rum	Broken Jamaica rum
22	Finest Old Jamaica Rum	0·9428	45·6	0·0110	0·021	None.	Present (vanillin).		Absent.	Not of pure Jamaica rum	Artificial rum
23	Fine Old Jamaica Rum.	0·9202	57·1	0·0060	0·020	Traces.	———		,,	Of artificial rum	ditto
24	Jamaica Rum..	0·9037	64·5	0·0144	0·063	Distinct.	Absent.		,,	Of broken Jamaica rum	Broken Jamaica rum
25	Porto Rico Rum .. .	0·9039	64·4	0·0024	0·208	None.	Present (cassia oil)		Present.	Of artificial rum	Artificial rum
26	Cuba Rum	0·9117	61·0	0·0024	0·106	None.	———		Absent.	ditto	ditto
27	Finest Jamaica Rum ..	0·8915	69·6	0·0096	0·227	Very distinct.	Absent.		,,	Of broken Jamaica rum	Broken Jamaica rum
28	Jamaica Rum..	0·9224	56·1	0·0024	0·042	Distinct.	,,		Present.	ditto	ditto
29	Finest Jamaica Rum .	0·9225	56·1	0·0120	0·123	,,	,,		Absent.	ditto	ditto
30	Finest Jamaica Rum...	0·9437	45·1	0·0144	0·109	None.	———		Present.	Of artificial rum	Artificial rum
31	Old Jamaica Rum .. .	0·9261	54·3	0·0060	0·042	Distinct.	Absent.		Absent.	Of broken Jamaica rum	Broken Jamaica rum
32	Jamaica Rum..	0·9179	58·2	0·0336	0·173	Very distinct.	,,		,,	ditto	ditto
33	Finest Jamaica Rum ..	0·9367	49·0	0·0180	0·113	,,	Present.		,,	ditto	ditto
34	Rum vero, Jamaica	0·9318	51·5	0·0036	0·014	Present.	Absent.		Present.	ditto	ditto
35	Jamaica Rum	0·8891	70·5	0·0370	0·378	Large amount.	..		Absent.	Of Jamaica rum	Jamaica rum
36	Original Jamaica Rum..	0·8793	74·4	0·0360	0·378	,,	,,		,,	,, ,,	ditto
37	Original Jamaica Rum.	0·8813	73·7	0·0430	0·799	,,	,,		,,	,, ,,	ditto
38	Original Jamaica Rum..	0·8886	71·2	0·0642	0·396	,,	,,		,,	,, ,,	ditto

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Artificial rum has obviously only the value of the alcohol contained in it. For its production artificial rum essences are used, the cost of which is comparatively low. These substances must be added in small amounts: a certain limit must not be surpassed, for the taste of the product would otherwise be rendered unpleasant. It is for this reason that the ester content of artificial rum is, as a rule, low. The adulteration of broken Jamaica rum with artificial rum is enticing in view of the fact that such a mixture has more or less the flavour of the genuine product and in consideration of the large profits to be gained from such a procedure. It is only fair, however, to the majority of the trade to point that genuine Jamaica rum is sharply differentiated from the artificial spirit. In Austria artificial rum goes under the names of "Cuba Rum," "Façon Rum," "Wirtschafts Rum," and "Inlander Rum." The name "Cuba Rum" for an artificial rum is indeed not a strictly proper one, but it is now so firmly established in the trade that it can scarcely cause confusion. Cuba rum, moreover, always indicates a spirit of inferior value to Jamaica rum, and on this account the cost of this artificial rum is considerably lower than that of even the very highly broken genuine product.

The results of our examination of 38 samples of various rums are summarized on the table given on pages 228-229.

Method of Examination.—As the table shows, the following determinations were carried out: Specific gravity, free acids in the distillate from 100 c.c., and ethers; the characteristic flavouring constituent of Jamaica rum, and foreign flavouring and colouring bodies were also examined.

The alcohol content was calculated from the specific gravity of the sample. As rum contains soluble substances this method is not strictly correct, but it was sufficiently accurate to approximately indicate the strength of the spirit. For an original rum it would be advisable to establish a standard of not less than 70 per cent. by volume of alcohol. In Austria there are no regulations at all as to the alcohol content of rums.

To determine the free acids in the distillate, 100 c.c. of the sample were rinsed into a distillation flask with 15 c.c. of water and the liquid distilled down to about 10 c.c. The distillate was then neutralized with N/10 sodium hydroxide and the result expressed in terms of acetic acid. Jamaica rum has as a rule a greater volatile acidity than artificial rum. The determination of the free acidity in the distillate bears this out, and it is generally found that this value is with Jamaica rum well above those given by artificial rums. The acid value of artificial rum is often strikingly low; it, however, sometimes happens that this value is as high as that of a Jamaica rum when broken down, and for this reason the acid value cannot be regarded as a certain criterion for the judgment of rum.

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The ethers were determined by the cold saponification method. To the neutralized distillate, 30 c.c. of N/10 sodium hydroxide were added, and the liquid allowed to remain in a closed flask for at least 24 hours. In the case of concentrated rums N/2 alkali was used.

With artificial rums it was observed that the ester aroma had completely disappeared the next day; but with concentrated Jamaica rum, and even with broken Jamaica rums having a low ester content, the characteristic aroma could be detected after 24 hours, although to a somewhat less extent. By using, however, stronger alkali, viz., a N/2 solution, this aroma more readily disappeared, and gave place to one resembling the terpenes of coniferae.

From the greater power of resistance against dilute alkali of the flavouring constituent, and from the fact that after the disappearance of the ester aroma scarcely any more alkali is absorbed, we have come to the conclusion that the characteristic flavour of Jamaica rum is hardly to be ascribed to the esters.

The ester content of artifical rum is, as we have already mentioned, only small, and cannot be appreciably raised without imparting to the product a bad flavour. In the case of broken Jamaica rum the ester content can be considerably diminished by the dilution; still, as we show further on, such a spirit can nevertheless be recognized as a genuine one. Although the ester content of original Jamaica rum is subject to very great variations we would have no hesitation in stating that sample 21 in the table marked "Original Jamaica Rum" is not an original rum, and this from the ester content alone without judging from other defects which are indicated by the analytical figures.

The samples 36, 37, and 38 marked "Original Rum" came from reliable sources and may be taken as genuine; their ester content varied between 0·378 and 0·799, which are about the same values as those given in Konig's "Chemie der menschlichen Nahrungs- und Genussmittel."

It may be asked whether the ester content may be considered a criterion for the quality of a Jamaica rum. The ester content can indicate whether the rum is concentrated or dilute. But the quality of the spirit cannot be judged on the ground of the ester determination, for obviously the quality of the rum depends not upon the amount of esters but upon their nature and relative proportions, as well as upon the other flavouring substances present. The strength of the aroma of Jamaica rum is indeed dependent upon the peculiar flavouring constituent; but the flavouring constituent is not saponifiable, and therefore is not indicated by the ester determination.

It is to be remarked that during the estimation of the esters in Jamaica rum the liquid assumes a more or less yellow colour according to the extent to which the rum has been broken; but that with artificial rum the liquid often remains colourless, or is but very

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slightly coloured. This yellow coloration is due to the greater content of the Jamaica rum in aldehydes, including furfural, than in the case of the artificial product.

We have, however, met with samples of artificial rum which, on saponification, assumed a fine yellow colour, which was not surpassed by even original Jamaica rum. In such cases it is probable that the artificial rum manufacturer had added aldehydes to his product in the hope of more nearly imitating the genuine article.

(*To be continued.*)

THE NEW YORK SUGAR TRADE LABORATORY.

The first annual Report of the New York Sugar Trade Laboratory shows that this institution has been successfully supplying a long felt want in New York sugar circles. Mr. C. A. Browne, the chemist-in-charge, reports as follows to the trustees of the Laboratory:—

Gentlemen,—Your chemist has the honour to submit herewith a report of the work of the New York Sugar Trade Laboratory for the first thirteen months ending December, 1908.

In accordance with the agreement entered into between yourselves and the undersigned, the work of equipping and organizing the laboratory was begun October 15th, 1907. Laboratory and office quarters were leased on the fifth floor of the Mallory Building, No. 80, South Street, and these were opened for work on Monday, December 2nd.

As assistant chemists of the laboratory, Mr. M. H. Wiley, formerly of the Sugar Laboratory, U.S. Bureau of Chemistry, Washington, D.C., and Mr. J. A. Hall, Jr., of the Louisiana Sugar Experiment Station, New Orleans, La., were engaged. The rapid increase in the number of samples to be tested made it soon necessary, however, to enlist the services of another chemist. Accordingly in March, Mr. G. H. Hardin, formerly of the Louisiana Sugar Experiment Station, was engaged. All three chemists had served previously under my charge either in Washington or New Orleans, and they have fully justified my selection in the care and accuracy of their work. A typewriter and boy were also engaged to assist in the work of office and laboratory.

The polariscopes ordered for the laboratory from Germany were delayed two months in shipment and until their arrival reliable instruments were rented from Messrs. Eimer & Amend, New York. The two instruments purchased from abroad are Schmidt & Haensch half shadow polariscopes with double wedge compensation, and Lippich polarizer, mounted on trestle supports. The polariscopes were standardized by the manufacturers for use at 20° C. (68° F.) and were

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RESEARCHES OF JAMAICA AND ARTIFICIAL RUM.

By Dr. Karl Micko,
Director der Staatlichen Untersuchungsanstalt für Lebensmittel in Graz.

(Continued from page 232.)

Fractional Distillation.—Fractional distillation forms the most important method of gauging a rum, for it is thereby possible to concentrate the ethers, ethereal oils, and other aromatic constituents to definite proportions, and to identify them by their smell. For this purpose 200 c.c. of rum were mixed with 30 c.c. of water, and the mixture fractionally distilled. Eight fractions were collected, of which seven consisted of 25 c.c. and the eighth comprised the balance of the distillate. As much of the liquid was distilled off as could be removed without burning the concentrated residue. To carry out the smelling test, glass beakers were employed which were filled each with one of the fractions. According as the liquids adhering to the walls evaporated, the different smells arising from the ethers and other volatile bodies passed off. The first two or three fractions contained besides alcohol a very light volatile ether, also formic and acetic acid ethers. The subsequent fractions gave off smells peculiar to artificial rums and not to Jamaica rums. The typical aromas of Jamaica rums are found as a rule in the fifth or sixth fractions; *which* depends chiefly on the alcohol content of the original sample, being later in a rich alcoholic rum and earlier in a poor one. In the case of original or concentrated rum these aromas are divided amongst two or three fractions, whereas in diluted rum they are only noticeable in one fraction. As already said, the typical aroma of Jamaica rum is accompanied by a body rich in terpenes. Both bodies are entirely wanting in artificial rum. This terpene body has, however, a less pronounced odour and is less characteristic a proof of Jamaica rum, as in other high class spirits similar bodies rich in terpenes are to be found.

Artificial rum often gives off odours which are practically wanting in Jamaica rum, *e.g.*, of strawberries, cassia or vanillin. These will establish the mixing of artificial with Jamaica rum.

It is to be observed that the aroma test must take precedence over the tasting, as otherwise the sensitiveness of the former will be practically destroyed. If the tasting is however undertaken first, then the mouth must be well rinsed out with water before inhaling the aromas. The smelling tests should preferably be carried out in the morning hours, as then the sense of smell is stronger than in the afternoon, as smokers can testify.

The last, or else the penultimate, fraction appears turbid in the case of Jamaica rum, providing it has not been diluted too much.

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This turbidity disappears on the addition of sodium hydroxide, only to reappear more strongly on acidifying. But if the sample be from an artificial rum the last fraction is as a rule clear. The partial solubility of the heavier volatile constituents of rum in sodium hydroxide is however no proof of a Jamaica rum, as it is a feature of other high class spirits also. Hence it happens that spirits derived from wines sometimes contain relatively large amounts of the heavy volatile bodies insoluble in water, which however consist only in a small degree of the higher alcohols such as amyl alcohol. A part of these bodies dissolves in sodium hydroxide and is reprecipitated on the addition of a mineral acid; another part dissolves only when heated in sodium hydroxide, but remains in solution when cooled, and only when acidified assumes a flocculent or oily turbidity. The smell of the sample is altered by the latter treatment. One is dealing here, it should be observed, with bodies of clearly complex composition somewhat akin to ethereal oils, and which are decomposed or otherwise altered when heated.

The heavy volatile bodies from the last fraction of rum can be separated by the aid of chloroform. Here we find before all others vanillin, which is often added to commercial rum essences and so is found in most artificial rums. If the rums be distilled to as great a concentration as feasible, the vanillin carried over in the vapour appears equally in the distillate. It is most prevalent in the eighth fraction; in the case of strong hydrated rum even the seventh fraction may contain it. In rums which contain over 70 per cent. alcohol it is advisable to mix the highly concentrated distillate with 20 to 30 c.c. of water and then to continue the distillation further.

The three last fractions were shaken up in a separating funnel with about 5 c.c. of chloroform. Since the sixth fraction of a rum rich in alcohol can still contain so much spirit that any separation of the fluids is not possible, it is necessary in such a case to add enough water to the fraction to enable the chloroform to separate from the remaining fluids. The chloroform is run into a beaker, and the beaker placed on a hot water bath. The chloroform must not boil however but only slowly evaporate. It is best to expedite the evaporation of the chloroform by frequently rotating the glass and as soon as the last trace of chloroform has disappeared the beaker should be covered with a watch-glass and laid aside to cool. Thereupon the smell of the residue can be tested.

The sixth fraction of an artificial rum often reveals a smell of cassia oil and other bodies all foreign to Jamaica rum. The seventh, and the eighth especially, contain vanillin providing this was present in the original sample. The smell of vanillin generally does not develop at once but only after an interval of hours or even days. It is therefore necessary when this smell is not immediately forthcoming

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to cover the last two samples with a watch-glass, leave them for two or three days and test them from time to time for the smell. It may happen when only a trace of vanillin is present that its smell is hidden by the scent of the other aromatic bodies, which however eventually volatilize or lose their smell owing to some influence such as oxidation, while the more stable and heavier volatile vanillin remains behind and then is gradually detected by its characteristic smell.

In the case of Jamaica rums the chloroform solution produces aromatic oleaginous or resinous residues; but the author has so far failed to detect vanillin in them with any certainty.

For the detection of vanillin in rum he employed the following test:—150 to 200 c.c. of rum were made distinctly but not excessively alkaline, and while still alkaline were heated on a water bath to volatilize the alcohol, then acidified with HCl, separated with chloroform, and the chloroform solution evaporated at as low a temperature as possible. The small residue was often resinous and gave off smells which hid that of the vanillin, and sometimes hardly let it reveal itself at all, so that the author had to treat the residue with warm water, filter off the liquid from the undissolved portion and again shake up with chloroform. After evaporating the solution and allowing the residue to stand, the smell of vanillin if it was at all present was as a rule easily detected.

Chloroform is better than carbon bisulphide for the detection of small quantities of vanillin, for the latter has to be freshly prepared for the test, since otherwise it will give off an odour which would affect that of the vanillin. Apart from that, carbon bisulphide on account of its inflammability is not conducible to pleasant working.

This experiment has the disadvantage as compared with the distillation method that on shaking up the spirituous rums with chloroform an emulsion is easily formed, and it needs a longer interval before the chloroform will separate from the aqueous liquid. This disadvantage is absent from the distillation test, as in the latter immediately after shaking up of the aqueous distillate with chloroform the two liquids separate sharply, and there is no need for any further cleansing of the residue from the chloroform solution. Finally, the search for vanillin can be combined in one operation with searches for other aromatic essences.

Foreign colouring bodies are frequently present in concentrated rum. Even if it does not happen that the quality of a rum is judged by its colour, the presence of these colours reveals a case of intentional manipulation and as a matter of fact they are often found in imitation Jamaica rums or in mixtures of Jamaica with false rums, also in broken Jamaica rums that are sold as original Jamaica.

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The testing of rums for foreign colouring bodies may, however, prevent any further identification of rum samples, such as may be demanded in legal cases. This concerns in particular the estimation of the ethers and the volatile acids in a distillate, but the alcohol content can be ascertained definitely by means of a hydrometer. As an instance one may cite the samples Nos. 17, 18, and 19, in the tables which were obtained from the same dealer, were produced to all appearance from the same recipe, but had been furnished with labels of different origin and with different specifications.

The tests made on the sample of commercial rums that were submitted to the *Untersuchungsanstalt* for analysis are to be found in the tables (see pages 228 and 229) numbered from 1 to 34. The author deems it unnecessary to give particulars of more samples than these, as it would only lead to needless reiteration of figures. It is, however, clear enough from the instances cited that there is no difficulty in distinguishing artificial rum from Jamaica rum. One is able, even in the case of strongly broken rums and, within reasonable limits, also in the case of a low content in ethers as in sample 28, to identify the typical aroma of Jamaica rums. In many samples said to be Jamaican, but which were artificial rums, traces of this aroma of Jamaica rum were detected. The writer ascribed this to their being mixed with small amounts of Jamaica rum and in order to confirm the theory of his supposition he interrogated the spirit merchants and their answer was invariably that they added some Jamaica rum to their " Wirtschafts," " Cuba," and artificial rums in order to improve their flavour. It must, therefore, not be overlooked that for similar reasons some Jamaica rum may be added to artificial rum essences whereby the peculiar taste will be imparted to them.

Additions of artificial rums to Jamaica rums yield aromatic bodies not found in the latter. But a more difficult task awaits one when only a small addition is under test, for then the adulteration may be hidden by the aroma of the Jamaica rum. In such a case it is advisable to fractionally distil a larger quantity than 200 c.c., and then to separate the individual fractions by further distillation. The artificial rum is less visible in the first fractions than in later ones, for both in Jamaica and in artificial rums the most volatile constituents consist mainly of the esters of formic and acetic acids and of alcohol. The first distillates of artificial rums have however a more obtrusive smell than have Jamaica rums, and this smell is also of a kind not found in Jamaicas, so that a case of adulteration is easily proved. Besides there are differences in taste and smell in the case of false rums. Many of them are at once detected by their strong smell of artificial ethers and vanillin. Others have a more finished aroma and taste, according as the distiller has the greater skill in the

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preparation of the artificial rums. In the tables we find No. 25 described as a Porto Rico rum; this was marketed with considerable advertisement. It contained a striking amount of cassia oil, and had also every indication of being a false rum. In the column, "Flavouring Substances foreign to Jamaica Rum," the only adulterants mentioned are vanillin and cassia oil. But that does not imply that only these and no other foreign matters were present. They have only been cited because they are more easily identified by smell, and, especially vanillin, are very commonly found in false rums, while other aromatic bodies observed, if they are not found in Jamaica rums too, are much more difficult to identify.

The difficulty of imitating the aroma of rum does not lie so much in the accurate selection of ethers as in the circumstance that the typical smell of Jamaica rums arises from bodies which either belong to the class of ethereal oils, or stand in close relation to them, but have no definite formula. Windisch was right in speaking of "ethereal rum oils," which he had obtained by the separation of rums with chloroform.

E. Sell, from his own investigations into the composition of rums, comes to the following conclusion. "The opinion expressed at the conclusion of some investigations on cognac, to the effect that it was impossible to distinguish genuine from fictitious liquors by mere chemical tests, is not a bit less apposite in the case of valuing rums. Here also the preference must be given to such expert opinion as bases its decision on the taste and the smell of the sample."

It is thus the case that the figures obtained through chemical analysis are not by themselves reliable for distinguishing artificial from Jamaica rums, but must be supplemented by investigations into the taste and smell. Any analyst who has a sensitive nose and palate can not only distinguish Jamaica rum from artificial rum, but also a large proportion of the cases where the two have been mixed. In virtue of his wider knowledge of chemical bodies and through suitable experimentation in the laboratory, the analyst is in a better position to detect the foreign bodies not found in Jamaica rum than is the practical expert. But estimations of price and quality fall necessarily within the latter's sphere.

(*To be continued.*)

The Anglo-Ceylon sugar estates in Mauritius did well in 1908, 120,400 tons of cane yielding 13,360 tons of sugar or 11 per cent. on weight of cane. This sugar realized a net profit of £25,000.

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RESEARCHES ON JAMAICA AND ARTIFICIAL RUM.

By Dr. Karl Micko,
Director der Staatlichen Untersuchungsanstalt für Lebensmittel in Graz.

(Continued from page 414.)

II.—The Identification of the typical Flavouring Body of Jamaica Rum.

For the identification and nearer characterization of the peculiar flavouring constituent of Jamaica rum, I proceeded in the following manner:—

1900 c.c. "Original Jamaica Rum" (No. 38, page 229) was fractionally distilled, eight fractions (I.-VIII.) being collected. Fractions I. and II. possessed a very distinct smell of formic and acetic esters, Fraction IV. a smell of butyric ester. The specific aroma which characterizes Jamaica rum appeared in Fraction V. but only feebly. It was quite distinct, however, in Fraction VI., and strongest in Fraction VII. Fraction VIII. had an acid and at the same time aromatic smell; it was cloudy and oily drops floated on its surface.

Fractions V. and VI. were mixed together and the mixture fractionally distilled, four fractions (I.-IV.) being collected. The first two of these fractions contained no typical aroma, and in the third fraction it appeared only to a very small extent. It existed strongly in the fourth fraction together with a body of a terpene-like odour. This fourth fraction was mixed with Fractions VII. and VIII. of the original distillation, and the whole fractionally distilled into five fractions (A-F).

Fraction A had only a little rum aroma; it was much stronger in Fraction B, but Fraction C contained the chief quantity of the peculiar flavouring constituent. In all three fractions was present the above mentioned terpene-like body (reminding one perhaps of juniper oil). Dilution of these three fractions produced turbidity.

Fraction D was turbid and watery, and did not possess the characteristic rum aroma, its smell being rather of other aromatic bodies. It was shaken up with chloroform, the chloroform solution separated, and the chloroform carefully evaporated away. It left behind a yellowish, resinous substance of an aromatic smell, which dissolved in caustic soda, and on acidification was reprecipitated. It is questionable whether this resinous substance is an original product of the fermentation. On the other hand many aldehydes incline to condensation, forming resinous substances which behave in the same way as the above with alkalis and acids. It is quite probable, therefore, that aldehydes are concerned in the formation of the aroma of brandy.

At all events, aldehydes react readily with alkalis. Grey says the specific rum aroma is produced by the action of lime on sugar

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solutions during its manufacture, and it is not impossible, therefore, that aldehydes are concerned in the production of the peculiar rum aroma. By the following experiments it will be seen that the peculiar flavouring constituent of Jamaica rum assumes another smell by the action of caustic soda. The formation of the rum aroma would be best studied by investigations during the different phases of manufacture.

Fraction E had only a slight smell; it was turbid and was shaken up with chloroform. The chloroform solution left on evaporation a substance resembling that obtained from Fraction D. Fraction F had hardly any smell. Fractions A, B, and C all gave the aldehyde reaction on additions of Schiff's reagent, and the furfurol reaction with aniline acetate. The aldehyde reaction was strongest in Fraction A, and weakest in C, but Fraction C behaved in quite the opposite manner with the two reagents.

The following experiments were undertaken with distillates B and C:—

Experiment 1.

Since all signs tend towards the fact that the peculiar flavouring body of Jamaica rum does not belong to the esters, it remained to be proved whether it was not due to an aldehyde or ketone group which may be present. To decide this question I made use of Fractions B and C, treated 5 c.c. therefrom separately with phenylhydrazine, hydroxylamine and semicarbazide. Fraction B was closely observed for change of smell, because it did not contain so much of the typical flavouring body, and any change would, therefore, be noticed more quickly than in Fraction C. But even after one week the typical aroma was recognizable, the phenylhydrazine test only having less smell, yet still quite recognizable, and, therefore, no reaction with either of the three reagents had taken place.

By the negative result of this experiment, it is not probable, therefore, that the typical flavouring body of Jamaica rum belongs either to the aldehydes or ketones.

Experiment 2.

The chief quantity of Fraction B was first treated with a saturated solution of sodium bisulphite, whereby any traces of aldehydes were removed, and the mixture shaken up with ether, whereby the typical flavouring constituent passed into the ether. The separated bisulphite solution give out no aroma on treatment with dilute sulphuric acid.

The ethereal solution was shaken up with sodium carbonate in order to remove any sulphurous acid present. The typical aroma remained unchanged. The ethereal solution was separated and subjected to careful distillation on a water bath, the ether passed over first containing no typical aroma, then followed the alcohol together

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with the typical flavouring constituent. The aroma was not very pure or strong since Fraction B contained only a small quantity of the typical flavouring body.

Only a part of the alcohol was distilled off. The alcoholic distillation residue exhibited no distinctive smell of the typical flavouring body of the rum; it was distinctly alkaline. Probably a slight trace of sodium carbonate remained in the ethereal solution thus causing the alkaline reaction of the residue. The latter was treated with excess of ether, the ethereal fluid separated the next day and the ether carefully distilled off on the water bath. The small quantity of alcoholic residue remaining from the distillation had a pronounced terpene-like odour reminding one of juniper oil, such as I have continually noticed to be present with the peculiar flavouring constituent. The neutral reacting fluid gave only a feeble furfurol reaction, but a distinct though not strong reaction with Schiff's reagent. It still contained, therefore, a trace of aldehyde but the principal amount was at all events removed. The alcoholic residue was mixed with 10 c.c. N/2 NaOH, which produced a turbidity and also a yellow coloration on standing. The smell of terpene was preserved for some days. By titration with acid it was found that only 0·10 c.c. of NaOH had been used.

The titrated fluid was again made alkaline and shaken up with ether. The ethereal solution left behind after the evaporation of the ether a yellowish, aromatic terpene smelling like oil. The other part of the liquid turned cloudy on acidification with dilute hydrochloric acid. On shaking up this cloudy solution with ether and separating them, evaporating off the ether, only a trifling quantity of a brownish yellow oil was left whose aroma was not aromatic.

From this experiment it follows that the typical flavouring constituent does not enter into combination with sodium bisulphite as the aldehydes do to form an oxysulphonic acid. The terpene-like body which is present with the typical flavouring constituent is not soluble in dilute sodium hydroxide, and suffers no loss of smell through prolonged contact with the same. This body belongs at all events neither to the esters nor to the aldehydes.

Experiment 3.

This was conducted on Fraction C, which contained the chief quantity of the typical aroma. It was submitted to distillation until a little alcohol passed over. The distillation residue is called (*a*), the distillate (*b*). Both (*a*) and (*b*) showed strongly the typical aroma, but the aroma from the distillate was much purer than that arising from the residue which smelt besides of the before mentioned terpene-like body and also of other aromatic bodies.

The residue (*a*) was cloudy and on the surface thereof swam drops of oil. It was very carefully neutralized with barium hydrate and

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diluted with water, shaken up with ether, separated and the ethereal fluid carefully distilled, so that nearly all the ether and only traces of the typical flavouring body distilled over.

The residue left amounted to only a few c.c.; it reacted feebly acid, and a strong but not pure smell of the typical flavouring constituent. The trifling quantity of acid was neutralized with very dilute sodium hydrate and then 30 c.c. N/2 caustic soda was added. The alkaline solution with the oil drops after a time turned strongly yellow. After four days the smell of the typical flavouring constituent disappeared, and a peculiar aromatic smell took its place. The terpene smell was, however, very distinct. On titration with acid the consumption of N/2 caustic soda was found to be 0·9 c.c. The titrated fluid was again made alkaline and shaken up with ether. On separation and evaporation of the ethereal layer an oil was left behind, similar in smell to that obtained in Experiment 2. The other layer of liquid was submitted to distillation in a current of steam in order to see whether it contained any volatile acids.

About 400 c.c. of distillate was collected which was slightly cloudy and reacted almost neutral. Evidently it contained volatile acids in only small quantities. It was made alkaline with barium hydrate, evaporated to dryness, the residue extracted with hot water, then filtered and the filtrate evaporated to dryness. The residue left was very small, and on the addition of two drops of dilute sulphuric acid a smell resembling that of butyric ester was formed.

The quantity of this substance was so small that it at all events did not correspond to the residue which required 0·9 c.c. N/2 caustic soda for saponification. The residue left from the extraction with hot water did not dissolve completely in hydrochloric acid. A white turbidity of the hydrochloric acid solution was caused through a fine flocculent substance, which did not dissolve in ether, but in alcohol. The quantity was so small, however, that it could not be put to further test.

The distillate (*b*) contained the typical flavouring body in its purest form. For the purification and isolation of the typical flavouring constituent the distillate (*b*) was strongly diluted with water, then shaken up with ether, and the ethereal solution further shaken with water. The typical flavouring body always remained in the ether which was distilled off very carefully at a temperature of 50°C., so that ether and practically none of the flavouring constituent distilled over. The alcoholic residue amounting to about 50 c.c. was diluted with water several times in order to remove the alcohol, then mixed with ether, the ethereal solution washed several times more with water and separated. It amounted finally to approximately 30 c.c.

5 c.c. of this left behind, after evaporation of the ether at room temperature, colourless drops of a fluid which possessed the typical aroma of Jamaica rum very intensely, and which in one hour

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completely volatilized and filled the laboratory with the characteristic aroma of Jamaica rum.

The aroma is more characteristic in the dilute than in the concentrated condition, and its boiling point certainly is higher than that of ethyl alcohol, yet it evaporated at ordinary temperature fairly quickly. When rum is rubbed on the palm of the hand the typical aroma can be detected for a fairly long time, and it seems, therefore, that Jamaica rum contains more difficult volatile bodies than the typical one, this being held in solution longer, thus preventing quicker volatilization. When the rum is fractionally distilled the typical constituent is concentrated in one or two fractions, and the aroma from these fractions is much stronger than in original rum, also evaporating more quickly out of the palm of the hand than in the case of the original rum.

A further 5 c.c. of the same ethereal solution was taken and shaken with 5 c.c. of N/10 sodium hydrate for some minutes, the alkaline fluid separated off, and the remainder washed with water until neutral. The typical flavouring body remained unaltered and is, therefore, not soluble in sodium hydrate. On treating this ethereal solution with 10 c.c. N/10 sodium hydrate and leaving for four days in a lightly corked flask with periodical shaking up, the smell at the end of this time was decisively altered, being certainly aromatic, but not corresponding with the typical flavouring body. The consumption of N/10 NaOH was found to be only 0·15 c.c. on titration with acid.

The same experiment was repeated only with this difference that the saponification was carried out by heating for half hour on the water-bath under reflux condenser. Only 0·1 c.c. of N/10 NaOH was used in this case, and the smell was altered as in the preceding case.

The titrated fluid was again made alkaline, shaken up with ether, the ethereal layer separated, and after gently warming the remaining fluid on the water-bath to expel traces of ether it was acidified with dilute H_2SO_4. From both tests a scarcely perceptible turbidity was produced, and no smell was apparent.

For the third experiment 10 c.c. of alcohol (previously distilled over caustic soda), 10 c.c. N/10 caustic soda and 5 c.c. of the ethereal solution which had been shaken with dilute caustic soda, were mixed together. After half an hour's heating on the water bath under reflux condenser, the titre of the fluid remained almost the same. The smell was, however, altered as by the first two tests. From all investigations with distillate (*b*) no yellow coloration took place by the action of alkali, as was the case with the residue (*a*). The yellow coloration produced in (*a*) would probably be due to aldehydes or furfurol in small quantities, but the amount of alkali used was so small that it could hardly be attributed to aldehydes. When an alkaline solution of furfurol is allowed to stand, however, a yellow colour is produced at first, afterwards turning cloudy. By the

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acidification of this turbid alkaline solution, a reddish brown flocculent precipitate is thrown out.

As the investigation with Fraction B proves, the typical flavouring body does not combine with alkali. The absence of a yellow colour points to the fact that it cannot belong to the aldehydes. We have from the earlier experiments seen that no combination is effected with ether phenylhydrazin, hydroxylamine and semicarbazide. Neither does it combine with sodium bisulphite. On the other hand, caustic soda alters the smell of the typical flavouring constituent slowly in the cold, but more quickly in the warm, but no saponification takes place however.

From the above investigation with distillate (*b*) it is seen that the typical aroma is not due to the esters.

Summary of Results.

From the above investigations, the following conclusions may be drawn :—

1. Jamaica rum contains an aromatic constituent peculiar to it alone, which is the basis of its characteristic flavour. This constituent is found neither in high class European spirits nor in artificial rum.

2. This typical flavouring body of Jamaica rum is a colourless not difficultly volatile fluid of a delicate aromatic smell and its boiling point lies higher than that of ethyl alcohol.

3. This typical body belongs neither to the esters, ketones, or aldehydes. It has the general characteristics of an ethereal oil, and it is not improbable that it stands in nearer relation to the terpenes.

4. The typical flavouring body does not dissolve in caustic soda, but on prolonged contact with it, it assumes an aromatic but more resinous smell.

5. In Jamaica rum as in other high class spirits is a body possessing a terpene-like aroma which is entirely absent from artificial rum. But it is less characteristically a proof of Jamaica rum as in other high class spirits similar bodies rich in terpenes are found.

6. In Jamaica rum there occurs in the last distillation fraction an aromatic smelling, resinous substance, which dissolves in caustic soda and is precipitated by the addition of acids. It is questionable whether this substance is a primary fermentation product. For we can produce such substance easily from aldehydes.

7. The analyst with sensitive nose and palate can easily distinguish artificial from Jamaica rum. He is also in the position to be able to detect mixtures of Jamaica with artificial rum.

8. From chemical analysis alone, however, no thorough conclusion is possible but when used in conjunction with the smelling test it is extremely valuable. The ester number is of especial value for determining whether the given sample is of a concentrated or a diluted rum.

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