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# A CRITICAL STUDY OF THE SUITABILITY OF ION-EXCHANGE PROCESSES

## APPENDIX.

### Costs of Sodium Chloride for De-Liming Operation.

CaO per cent. juice (average factory) = 0.0200 per cent.

CaO per cent. juice "break-through" level = 0.0075 per cent.

CaO per cent. juice removed = 0.0125 per cent.

Assume clarified juice 90 per cent. on cane :

CaO removed per ton cane =

$$\frac{2240 \times 90 \times 0.0125}{100 \times 100} \text{ lb.} = 0.252 \text{ lb.}$$

NaCl required =  $6.25 \times 0.252 \text{ lb.} = 1.57 \text{ lb.}$

Cost of NaCl at 2 cents per lb. = 3.14 cents per ton cane ground.

Total expenditure on labour and chemicals for cleaning evaporator in typical Trinidad factory = 2.4 to 3.0 cents per ton cane ground.<sup>30</sup>

The cost of the chemical alone for the ion exchange process is more than the total cost of cleaning evaporators by present methods.

- 1 U.K. Patent No. 557,690 of 1942; *I.S.J.*, 1944, p. 165.
- 2 U.S. Patent No. 2,366,630 of 1945.
- 3 U.S. Patent No. 2,388,195 of 1945.
- 4 U.S. Patent No. 2,359,902 of 1944; *I.S.J.*, 1946, p. 80.
- 5 U.S. Patent No. 2,388,222 of 1945; *I.S.J.*, 1946, p. 166.
- 6 HAAGENSEN. *I.S.J.*, 1946, p. 240.
- 7 WEITZ. *I.S.J.*, 1943, p. 100.
- 8 WAY. *Journ. Roy. Agric. Soc. Engl.* 1850, p. 313.
- 9 EICHORN. *Landw. Jahrb.* 1875 (see 11 below).
- 10 GANS. German Patent No. 197,111 of 1906.
- 11 MYERS *et al.* *Journ. Ind. Eng. Chem.*, 1941, p. 697.
- 12 SPENCER. U.S. Patent No. 1,455,363 of 1923.
- 13 BRUCE and RILEY. U.S. Patent No. 2,139,299 of 1938.
- 14 RUDOLF. U.S. Patent No. 1,304,206 of 1919.
- 15 U.K. Patent No. 487,675 of 1938.
- 16 ADAMS and HOLMES. *Journ. Soc. Chem. Ind.*, 1935, p. 1-6T.
- 17 Abstract, *I.S.J.*, 1935, p. 407.
- 18 RAWLINGS and SHAFOR. *I.S.J.*, 1942, p. 19.
- 19 GUTLEBEN and HARVEY. *I.S.J.*, 1945, p. 11.
- 20 Abstract, *I.S.J.*, 1945, p. 251.
- 21 STEVENS. *I.S.J.*, 1945, p. 250.
- 22 BLANN. *The Sugar Bulletin*, 1945, No. 22, p. 198.
- 23 GAYLE. *La. Univ. Eng. Expt. Station News*, 1945, 1, 3, p. 11-15.
- 24 BOYD and SCHMIDT. *I.S.J.*, 1942, p. 100.
- 25 SPENGLER and TÖDT. *I.S.J.*, 1944, p. 160.
- 26 Anon. *Australian Sugar Journal*, 1946, p. 127.
- 27 The Permutit Co., 330 West 42nd St., New York, U.S.A.
- 28 Betz Handbook of Industrial Water Conditioning (1945), p. 28.
- 29 Sugar Research Foundation; *I.S.J.*, 1946, p. 302.
- 30 Private communications from Chemists of Trinidad factories.

## The Chemistry of Rum Production.<sup>1</sup>

By J. R. McFARLANE, B.Sc., A.R.I.C.,  
Chief Chemist, Caymanas Estates Ltd.

Since rum production in Jamaica dates far back into the dim and distant past, it is not surprising that the methods employed have come to be regarded as axiomatic or that empirical formulae have been the rule rather than the exception. It must seem a little strange that little or no attention has been paid to the scientific aspect of rum production in the Island. At the beginning of the century a foundation was laid by the excellent work of COUSINS,<sup>2</sup> ASHBY,<sup>3</sup> ALLEN<sup>4</sup> and GREG,<sup>5</sup> but since that time any attempt to build on their structure has been very limited. ARROYO, in Puerto Rico, has carried out extensive researches<sup>6</sup> and has added considerably to our knowledge, but his suggestions do not seem to have met with wide approval here. So far as can be ascertained, no serious attempt has been made to correlate the proportions of the various constituents present in the wash with the quality of the rum produced, or to determine the conditions under which the desired proportions of those constituents can be produced at will. In an attempt to show that rum production is indeed a scientific industry, it has been thought that a brief outline of the chemistry of rum fermentation and of the reactions which are involved in the production of those substances which go to make up what is known as Jamaica rum will serve to demonstrate the fact.

Jamaica rum can be considered as an aqueous mixture of ethyl alcohol, aliphatic acids, esters, higher aliphatic alcohols, aldehydes, furfurals, and traces of certain unidentified substances which appear to be responsible for the character of the product as

distinct from other spirits. Rum is produced by the alcoholic fermentation of aqueous solutions of molasses (or cane juice) and dunder by means of yeasts in the presence of bacteria and other micro-organisms which promote subsidiary reactions leading to the production of substances other than alcohol. It has long been claimed that environment plays an essential part in the production of specific marks of rum and, for this reason, great care has been taken not to destroy the character of the fermentation, manufacturers having gone to such extremes in this direction in the past that some of the distilleries were definitely insanitary. Under the conditions of adventitious fermentation as practised in Jamaica, there is little doubt that each distillery produces a distinct rum; nevertheless FLORO, at Frome, has demonstrated the possibility of manufacturing several marks, which were previously made on individual estates, at one central distillery.

It has also been regarded as essential to employ pot stills, but, while this type of still will normally produce rum with a greater proportion of higher boiling constituents than the continuous type, good rum has recently been made in a continuous experimental still at Frome, and there is no reason why, with careful setting of the plates, all but the heaviest types of Jamaica rum should not be distilled in this manner. ARROYO has succeeded in producing a rum in Puerto Rico, under very different conditions to those obtaining in Jamaica, which, he claims, is "indistinguishable from Jamaica rum in taste, bouquet and chemical analysis;" it can be manufac-

tured "in a fraction of the time required for the confection of rums of similar quality under Jamaican methods and conditions;" and it ages so rapidly that a 3-month-old rum offers the chemical analysis of a 2-year-old Jamaica rum, while a 6-month-old product is comparable with 5-year-old Jamaica rum.<sup>7</sup> This would seem to indicate that the control of conditions of fermentation is the most important factor in the production of rum.

*Type of Yeast.*—In general, top-fermenting yeasts are employed in rum distilleries in spite of the fact that most other distilleries employ bottom-fermenting yeasts. In rum production pure cultures are not used, the fermentation being spontaneous, and it is obvious that undesirable strains will be present. This affects the efficiency and it is probable that a fermentation efficiency of 85 per cent. is exceptional. This fermentation efficiency must not be confused with the overall efficiency reported by a number of our distilleries, but is the percentage of the theoretical yield of alcohol which would be produced in the absence of all reactions other than alcoholic fermentation. Although the adoption of a pure culture system seems remote there is no apparent reason why cultures which would ensure a preponderance of desirable strains should not be employed in an attempt to raise the efficiency.

It is of interest and possible importance to note that, whereas budding yeasts now appear to predominate in Jamaican distilleries, the presence of a fission yeast (*Schizosaccharomyces mellacei*) was reported by GREG<sup>5</sup> some 50 years ago, and was alleged to be partially responsible for the characteristic flavour of Jamaica rums. There is little evidence of this type of yeast being present in most distilleries in any appreciable quantities at the present time. ARROYO has suggested that a top-fermenting fission yeast will produce a more desirable quality of rum than the usual budding type, and has investigated the problem, employing Pombe yeast (*Sch. pombe*) which was discovered by LINDNER in 1887.<sup>8</sup> Re-introduction of a fission yeast into our distilleries in sufficiently pure culture to ensure its being the predominant organism might lead to improvement of both efficiency and quality, since it is reported that *Sch. mellacei* yields 6.6–7.6 per cent. alcohol.

*Temperature.*—While there appears to be no definite optimum temperature at which a yeast thrives best, there is a fairly narrow range over which reasonably good results can be obtained. For the production of rum it is not usual to allow the maximum temperature of the wash to exceed 35°C. and temperatures above 37.5°C. should most certainly be avoided if reasonable yields are to be expected. Actually, the lower the temperature, the slower will be the fermentation, but the better will be the quality of the rum produced, and it is generally accepted that distilleries at higher elevations produce better rum for this reason. ARROYO claims that, for good

quality rum, the temperature should be maintained at about 27°C., but without elaborate cooling devices, this would be extremely difficult to achieve in most Jamaican distilleries.

*pH of Substrate.*—In general, low pH values favour reactions involving yeasts, and higher values those involving bacteria, although it is possible to acclimatize micro-organisms to conditions very different to those under which they normally thrive best; however the products of metabolism may, and most probably will, be different in these cases. In the production of alcohol, as distinct from rum, the optimum pH value is in the vicinity of 5.0, but for rum production a pH of 5.8 is claimed by ARROYO to yield the best results. This degree of acidity would seem to allow of sufficient bacterial activity to ensure the production of those amounts of secondary constituents required to maintain the quality of the rum at the desired level, whereas at a pH of 5.0 the bacterial activity is almost completely suppressed. The maintenance of a constant pH during fermentation is advocated by ARROYO, who states that by this means "rums of great cleanliness of aroma, which aged rapidly were produced, while the mature rum had mellowness, smoothness and delicacy of flavour." No progress in this direction appears to have been made in Jamaica though the buffering effect of dunder must have a limited influence on the pH of the wash.

*Yeast Nutrients.*—With regard to the actual nutrients required, with the exception of carbon, hydrogen and oxygen, which are supplied by the sugars, the main requirements appear to be nitrogen, phosphorus and an alkali metal, though there is little doubt that trace elements may play an important part in yeast metabolism. In rum manufacture, nitrogen is supplied in the molasses and in dunder, where the precursors of amino-acids are produced as the result of breaking down of yeast proteins. It has been claimed that potassium plays an important part in the metabolism of yeast. This element is one of the main elemental constituents of molasses ash. It is probable that sodium, which is also present in fair quantity in molasses, can replace potassium in the metabolism. Yeast cells appear to derive energy rather than food from the decomposition of sugars. The process under anaerobic conditions may be regarded as respiration without any marked growth, whereas under aerobic conditions propagation occurs with very considerable cell growth and multiplication.

*Effect of Metabolic Products.*—The products of metabolism of yeasts have the effect of retarding the metabolism, and may also cause the formation of involution forms which are usually associated with old cultures where there are accumulations of waste products. The inhibitory effect of alcohol is well known and has a direct bearing on rum and alcohol production though it is by no means certain that the

concentration of alcohol in the wash is the limiting factor in the inhibition of yeast growth. The degree of inhibition of the higher alcohols is more marked than that of ethyl alcohol and appears to depend on the position of the alcohol in the homologous series. SEGAL<sup>9</sup> gives the following percentages of alcohols as completely inhibiting alcoholic fermentation (and also yeast growth): ethyl alcohol, 12.0; propyl alcohol, 3.4; butyl alcohol, 1.5; iso-amyl alcohol, 0.6; and fusel oils, 0.8 per cent. The degree of inhibition of other metabolic products does not seem to have been investigated at any length, but there can be little doubt that this phenomenon does occur. Yeasts, however, can be acclimatized to greater concentrations of various substances as is instanced by the fermentation of washes set at high Brix which, with the ordinary strains of yeasts, would only be partially fermented. Actually, the process of acclimatization consists of the selection of the very small proportion of cells which have the desired properties and their propagation at the expense of the remainder.

*Acids.*—Next to alcohol, the organic acids constitute the most important components of rum and they are found in the final product, not only in the free state but also combined with alcohols as esters. Of the free acids, some 97–98 per cent. is acetic acid, the remainder being about 1 per cent. butyric acid together with traces of formic, caproic, pelargonic, capric and other aliphatic acids. There are three methods in which the acids may be produced; first, by the direct fermentation of sugars by means of bacteria; second, by the bacterial oxidation of the products of alcoholic fermentation; and third, by the de-amination of amino acids, probably brought about by enzymatic action.

In the case of acetic acid, it is probable that the greater part is formed by the oxidation of alcohol by acetic acid bacteria, although organisms are known which can produce acetic acid directly from

sugars, as is evidenced by the souring of cane juice under anaerobic conditions.

In adventitious fermentations such as are general in Jamaica, sufficient acetic acid bacteria are usually present in the wash for the production of the acid required in the production of the lighter rums, but it appears customary to prepare acid externally in the case of the heavier rums, though there would seem to be no fixed practice for this. It seems likely that butyric acid is formed by the direct anaerobic fermentation of sugars by bacteria, the *Clostridia*, of which these bacteria form an important group, being part of the normal flora of molasses, entering the factory on the soil about the cane roots. ARROYO has investigated the formation of butyric acid and has isolated an organism which can yield some 45 per cent. of butyric acid on sugar converted together with hydrogen and carbon dioxide.

Butyric acid is a yeast poison, but, in the small quantities present, it is not likely to have more than a slight retarding action on fermentation. Moreover, ARROYO reports that certain butyric acid bacteria (*Clostridium saccharo butyricum* Arroyo in particular) can live symbiotically with yeasts, especially those of the fission type. The effect of the bacteria is to accelerate the rate of fermentation and to improve the quality and ageing properties of the rum produced. Since the beneficial effect can be exerted through quartz, it is considered probable that the bacteria produce radiant energy capable of stimulating the yeast cells to a very rapid rate of multiplication. The majority of these acids are to be found in traces in rum, and in view of their high boiling points as compared with alcohol (78°C.) are probably distilled in steam towards the end of the distillation. They should, therefore, be more concentrated in the retort lees, a fact which is borne out by the use of these lees for the preparation of "lime salts" used in the preparation of "high ether" rums.

(To be concluded).

## Sugar-House Practice.

**Proper Illumination for Refineries.** I. P. CREVI.<sup>1</sup>  
*Sugar Industry Engineers, New York, 1945 Meeting.*

Lighting surveys, conducted on a nation-wide basis, have shown that an average intensity of less than two foot candles exists in most plants; and that direct glare, reflected glare, insufficient illumination, non-uniformity of illumination, and harsh shadows are prevalent everywhere. Eyes are being forced to perform visual tasks at only a fraction of the lighting intensity for which nature moulded them. Inadequate lighting causes fatigue, but even with lighting of

adequate intensity, there can be no comfort if there is glare. Lighting must be uniformly distributed over the entire working area.

When conditions are such that surroundings are dark and gloomy, production suffers, costs increase, and accidents increase. Modern illumination in the modern industrial plant is an investment that pays big dividends, for better lighting means better plant operation. The following procedure is a simple and accurate method whereby lighting equipment can be easily determined for any installation:—

<sup>1</sup> Of the Westinghouse Electrical Corporation, New York.

# The Chemistry of Rum Production.<sup>1</sup>

By J. R. McFARLANE, B.Sc., A.R.I.C.,  
Chief Chemist, Caymanas Estates Ltd.

(Continued from page 75).

## PART II.

*Esters.*—Esters, or “ethers” as they are usually termed in Jamaica, are formed by combination with alcohols with the elimination of water. The ester content has come to be regarded as the criterion of quality in a rum, and, although it is not the essential factor in determining its characteristics, there is a definite trend showing increase in other constituents with increase in esters, so that there is a real basis for a system of classification of rum types according to their ester content. With regard to the four main types, the ester content is usually given as:—

Common Clean	80 — 150	parts per 100,000 alcohol.
Plummer . . . .	150 — 200	“ ”
Wedderburn	200 — 300	“ ”
Flavoured . .	700 — 1600	“ ”

Ethyl acetate comprises more than 98 per cent. of the total esters present in rum, ethyl butyrate about 1 per cent., and a mixture of higher esters the remainder. It is probable that the greater part of the esters in lighter rums is formed by direct esterification of the alcohol produced by fermentation, but micro-organisms are known which are capable of the production of esters directly from sugars. One of these organisms was isolated by PECK and DEERR<sup>10</sup> and produced over 7000 parts of esters per 100,000 parts of alcohol, while an even more active organism was isolated by ASHBY<sup>3</sup> in Jamaica. These organisms are apparently non-sporing *Torulae* and play an important part in the production of high ester rums.

*Lime Salt Process.*—The production of high esters by the “lime salt” process is an example of how the equilibrium can be shifted by the removal of one product of reaction. In this process, calcium salts of the acids from retort lees are treated with sulphuric acid in the presence of alcohol (high wines) and the mixture of acids, after precipitation of calcium sulphate, used in the high wine retort. The concentrations of alcohol and acid as compared with that of water will be enhanced by the removal of this latter constituent by the excess sulphuric acid and the degree of esterification will be greatly increased.

When strong rum is broken down with water, the equilibrium is disturbed, and the reaction from right to left, that is, the saponification of the ester, is accelerated. Hence the ester content of freshly-diluted rum is invariably lower than that of the original strong rum, and ARROYO states that a period of from 6–12 months may elapse before equilibrium is re-established. He also suggested the use of pre-treated aqueous alcohol for breaking down rum.

It has long been recognised that certain rums on ageing will decrease in ester content, while others exhibit the opposite phenomenon. Analyses of these

rums will show that these changes are due, for the most part, to the gradual establishment of equilibrium, the rise or fall of ester content depending on whether there is an excess or deficit of ester compared with the amount demanded by the equilibrium equation.

In addition to ethyl alcohol, rum contains varying amounts of higher aliphatic alcohols, including *n*-propyl, *n*-butyl, iso-butyl, active and inactive amyl and traces of higher members of the series. These are generally considered to add body to the rum, and the amounts present increase with the degree of heaviness of the rum. The toxicity of these alcohols is such that it has been stated that the after-effects of over-indulgence in rum are due to them, their rate of decomposition or expulsion from the system being very much slower than in the case of ethyl alcohol, but it has been suggested that the esters are equally responsible for such effects.

The production of acetaldehyde as an intermediate in alcoholic fermentation is probably the source of this substance in rum, although it might be produced by the oxidation of alcohol. Traces of formaldehyde and of formic acid are probably formed by oxidation of hexoses. The small amounts of furfural always found in rum are produced from pentoses which either enter with the molasses or are produced by degradation of the yeast nucleic acids, of which *d*-ribose is a component part.

In addition to the identified constituents there are always present certain unidentified substances which, although they are to be found in minute traces, serve to characterize the rum. GREG<sup>5</sup> isolated a substance from dunder by extraction with petroleum ether which appears to be responsible, in part at least, for the characteristic odour of rum. This substance was also investigated by MICKO<sup>11</sup> who suggested that it might be related to the terpenes. It is probably identical with the “rum oil” of ARROYO, who considers that it is the essence of all aroma in rum; although its constitution is unknown, it would appear that the amount present depends on the type of yeast employed for the fermentation, fission yeasts being particularly effective in this respect. Since the rum oil has a high boiling point, the higher temperatures attained in a pot still towards the end of the distillation are more favourable to its distilling over than the steady lower temperatures existing in a continuous still, and unless a very effective yeast is employed it is possible that the prospects of making Jamaica rum in a continuous still may be remote.

*Aroma.*—With regard to aroma, there can be little doubt that the esters, more particularly ethyl butyrate and the higher esters, play an important part. It has been suggested that the more volatile ethyl

acetate serves as a vehicle to convey them to the nose, but COUSINS<sup>2</sup> claims that the accentuation of aroma on dilution is due to a reduction of the volatility of the acetate, making the effect of the higher esters more pronounced. The development of those substances responsible for the aroma and flavour of rum has been attributed to numerous causes. GREG<sup>5</sup> was of the opinion that the type of yeast used was the main factor, whereas ALLEN<sup>4</sup> considered that a combination of fission yeasts and butyric acid bacteria played a very important part.

This latter view is supported by ARROYO, and in his opinion 75 per cent. of the aroma is controlled during fermentation by the type of yeast and bacteria used, together with correct temperature and *pH* control. The remaining 25 per cent. may be attributed to proper distillation and ageing. He stresses the desirability of centrifuging the wash prior to distillation, the removal of pectins, albuminoids, etc. preventing the formation of an unpleasant aroma. The type of fermentation appears to be of importance, ASHBY<sup>3</sup> observing that a slow fermentation with a top-fermenting fission yeast accentuated the production of high esters and good flavour. KAYSER states that with spontaneous uncontrolled fermentation the esters and volatile acids are higher but the higher alcohols lower than when using sterilized material and pure cultures of fission yeasts. In the case of flavoured rums the *Torulae* probably play an important part by the direct production of esters from sugars.

It will be seen from the foregoing survey that it is not possible to produce rum without the utilization of a proportion of the sugars for the production of constituents other than alcohol. Hence any increase in these constituents will mean a decreased yield of rum. Some idea of the losses inherent to the production of Jamaica rum is given by FLORO<sup>12</sup> who considers that an efficiency of 70 per cent. of the theoretical yield of alcohol would be very good in a Jamaican distillery. He points out that the losses will be least for the lightest rums and will increase with increasing heaviness.

*Possibility of Pure Culture.*—In view of the much better yields which are possible with pure culture fermentation, it might be thought that some progress in this direction would have been made, but it must be remembered that various marks are standardized with regard to quality and the manufacturer is very chary of doing anything which, while it would increase his yield, might cause deterioration in quality. There is little doubt that it would be possible to employ modern methods of fermentation with pure yeast and bacteria cultures and temperature and *pH* control, for the production of Jamaica rum and to manufacture all but the heaviest types of rum in a continuous still, but a considerable amount of research and experimental work would be necessary

before such methods could be applied in Jamaica on a commercial scale.

Under the present haphazard system of spontaneous fermentation as practised here the type of rum must depend very largely on the conditions obtaining in the distillery and this has given rise to the belief that any particular mark of rum can be produced in one particular distillery only. As already mentioned, this has been shown to be a fallacy, several distinct marks having been made in one distillery, but, so long as conditions in the distilleries remain unchanged, environment will be an essential factor in the production of Jamaica rum. The pronouncements of the Sugar Industry Commission<sup>13</sup> are particularly illuminating in this respect, and there can be no doubt that, if progress is to be made in the industry, some attempt must be made to set it on a scientific basis.

A start has been made in the right direction with the institution of chemical control in the distilleries and this will bring home the extremely low efficiency of production, but it will be necessary to go much further and to set up a research scheme, preferably under the aegis of the Sugar Manufacturers' Association, whereby all knowledge may be pooled, before any real progress can be made towards the realization of a really efficient rum industry in Jamaica. The logical step would be the acquisition of a first-class bio-chemist with experience in fermentation processes so that any scheme which might be evolved would be certain to start along the right lines, for under present conditions the chemists in the Island have neither the time nor (as they will be the first to acknowledge) the necessary experience to carry any such scheme to a successful conclusion, and this proposition should, therefore, receive the earnest consideration of all who are interested in the further progress of the rum industry in Jamaica.

1 *J.A.S.T. Quarterly*, 1946, 9, Nos. 3 and 4, pp. 54-66 (here abridged).

2 *W.I. Bull.*, 1906, 7, p. 120.

3 *Ibid.*, 1910, 12, p. 302.

4 *Ibid.*, 1906, 7, p. 141.

5 *Bull. Bot. Dept. Jamaica*, 1895 and 1896.

6 Consult *I.S.J.* names indexes under "Arroyo" from 1940.

7 Circular 21/40, S.M.A., Rum Section.

8 *Woch. Brauerei*, 1887, p. 44.

9 *Microbiol. (U.S.S.R.)*, 1938, 7, p. 93.

10 *H.S.P.A. Bull.*, 28; *I.S.J.*, 1909, pp. 394, 454.

11 *I.S.J.*, 1909, pp. 225, 410, 446; 1910, p. 411.

12 *J.A.S.T.*, 1944, 8, p. 14.

13 *I.S.J.*, 1946, pp. 32-36.

**PHASE MICROSCOPY.**—Phase microscopy requires the use of two additional pieces of equipment. An annular diaphragm in connexion with the substage condenser illuminates the specimen with a hollow cone of light; a special diffraction plate is inserted within the microscope objective. Depending on the kind of diffraction plate used, regions within the specimen of different optical path can be made bright on a dark background, or dark on a light background. The study of plant and animal life, parasites, emulsions, replicas of surfaces, glass and other transparent substances, minerals, crystals, synthetic fibres and other materials will be greatly aided by the use of this instrument.