

THE J.A.S.T. JOURNAL.

# Modern Trends in Distillery Practice.

# J. R. MCFARLANE, B.Sc., F.R.I.C.

Chief Chemist Gray's Inn (Jamaica) Central Factory Ltd.

(Mr. J. G. Davies in the Chair).

Although Jamaica has undoubtedly made considerable progress in distillery practice during the past ten years, this has been devoted almost entirely to the establishment of a rational system of chemical control, and to the improvement of existing methods. Little or no attempt has been made to adopt either the principles or practices of techniques used in other countries, although it is probable that many of the advances made could be employed with advantage in this Island. One reason for the apparent lack of enterprise in this direction must be the fact that Jamaica has always been blessed with a superabundance of molasses, so that it has probably been felt that there was no need to strive for greater efficiency in our distiller-This, it is held, is a fallacy since improvement ies in efficiency and reduction in capital expenditure for plant must go hand in hand with reduced cost of production and subsequent increased profits. Now that the quota has been substantially cut, this will be more evident and, for distilleries to play their proper part in the economics of the Industry, it will be all important to bring production costs to as low a figure as possible. In other parts of the world, where molasses is imported and where it comprises the most expensive item in spirit production, conservation of this material is regarded as essential, and an increase in overall efficiency of even one half of one percent is regarded as an important achievement which may mean a considerable increase in the margin of profit. For this reasoon many of the newer methods introduced elsewhere, regarded here as giving only minor improvements, have proved highly successful and have, in certain cases, made the difference between profit and loss for the Company concerned.

As it is difficult to keep abreast with progress in the outside world here in Jamaica, a review of modern trends in distillery practice has been prepared in an attempt to present the more important innovations which have been introduced elsewhere, and in the hope that some of the ideas may prove of value in our distilleries. This survey, covering such a large field, must of necessity be lacking in detail but, for those who are interested, reference to the literature are appended.

Progress in distillery tenchique has been along several well defined lines which may be summarised as:—

- (1) The pre-treatment of molasses.
- (2) The preparation of seed yeast in pure culture.
- (3) Incremental fermentation and the use of high density mashes.
- (4) Continuous fermentations.

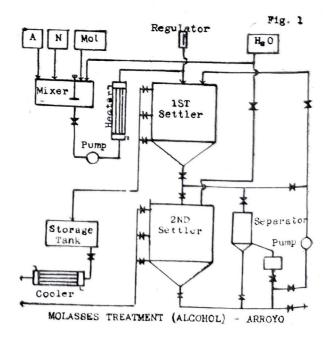
- (5) Fermentation processes involving the re-cycling of yeast.
- (6) Special methods of fermentation for rum production.
- (7) Innovations in distillation practice.
- (8) Disposal of effluent

and each of these sections will be dealt with briefly.

#### 1. THE PRE-TREATMENT OF MOLASSES.

Molasses contains undesirable constituents and substances not readily available for alcoholic fermentation as well as those required for the production of alcohol or rum. In addition, the yeast nutrient requirements are seldom fulfilled by the molasses alone. Several processes have been evolved for the treatment of molasses prior to fermentation in order to improve its value as a substrate and to remove those substances which would interfere at one stage or another with the process of fermentation.

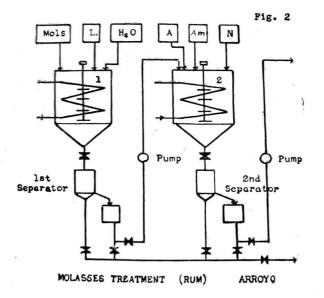
Arroyo has evolved two processes, one for alcohol production (1) and the other for the manufacture of rum (3). In the 1st. method (Fig. 1) the





molasses is diluted to about 60° Bx., nutrients being added, and treated with concentrated sulphuric acid in sufficient quantity to lower the pH by 0.5 unit. (This usually requires approximately 0.5% acid on the weight of molasses.) The treated thick The treated thick molasses is heated to  $80^{\circ}$ C. and maintained at this temperature for six hours in a settling tank. The clear "thick mash" is drawn off and, (after cooling to 40°C.), used to mix "thin mash" for fermentation, while the sediment is either diluted with water and re-settled or separated by means of a centrifugal separator, the dilute clear liquor being returned to process and the sludge discarded. This treatment leads to improved recovery, and several claims in this respect are made by the patentee. Owen has studied the process, and shown that the Owen (2) fermentation efficiency is undoubtedly improved. The removal of calcium ions and of other impurities causes a substantial reduction in scaling of the still, while the material is freed from vegetative forms of micro-organisms though heat resisting spores will not be destroyed. This treatment is not entirely suitable for rum production, and should be limited to alcohol distilleries and those producing a "light" rum.

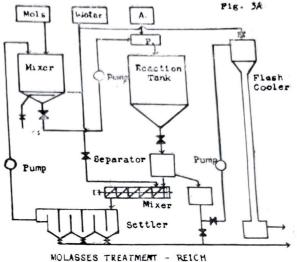
A special treatment (Fig. 2) is advocated for use in the production of heavier rums (3) differing some-



what from the above. Molasses is treated with sufficient milk of lime to raise the pH by 0.5 unit, diluted with hot water to about 60° Bx. and maintained at a temperature between 75° and 80° C. for at least half an hour. The whole of this thick mash is fed to a centrifugal separator and the clear liquor cooled to 40°C. and treated with sufficient sulphuric acid to lower the pH to between 5.0 and 5.2. After one hour, the pH is raised to 5.4 - 5.6 by means of strong ammonia solution and nutrients are added, the whole of the material then being passed through a second separator. The clear thick mash is run to a storage tank, and the sludge to waste. Here, again, Arroyo claims improvement in efficiency and in quality accompanied by considerable reduction in scaling and this treat-

ment is advocated in his "heavy rum" technique mentioned later.

A third method of treatment (Fig. 3A) has been



devised by Reich (4) and, while this was originally introduced for the express purpose of improving the quality of the yeast obtained from the dead wash, it also improves the molasses and prevents scale formation.

Molasses is diluted to  $45^{\circ}$  Bx. with hot water and heated to  $95^{\circ}$  C. by steam injection. Sulphuric acid is added by means of a "Proportioner" at a rate of one gallon per 500 gallons of 1:1 molasses. The treated thick molasses is maintained at  $90^{\circ}$  C. for one hour to allow the completion of the reaction, and then centrifuged. The clear liquor colled to  $25^{\circ}$  C. by means of a "flash cooler," which apparently resembles a barometric condenser, and diluted to form thin mash, while the sludge is discharged into a scroll conveyor, diluted with water, and allowed to flow through a settler fitted with baffles. The dilute clear liquor is returned to process at the mixing tank.

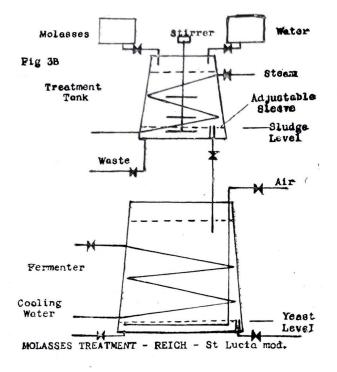
In all these processes pasteurisation of the molasses is achieved, but the method of Standard Brands Inc. (5) involves heat treatment of the molasses at a higher temperature ensuring complete sterilisation. It is doubtful, however, if sterilisation is essential in most alcoholic fermentations since the spores remaining after pasteurisation cannot develop into vegetative forms sufficiently rapidly to affect the fermentation during its relatively short cycle. Moreover the increased cost of production of sterile molasses would not justify such a procedure in normal practice (6). The main advantages of these processes would appear to be

- (1) Improvement in fermentation efficiency and therefore in yield.
- (2) The removal of scale-forming constituents.



# (3) Production of yeast of low ash content for fodder purposes.

It is of interest to note that several B.W.I. distilleries have adopted modifications of the Reich process with some success, Bradshaw (7) that it had proved very useful in St. Lucia (Fig. 3B)



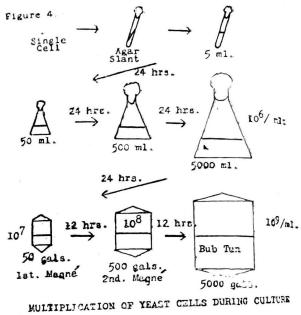
particularly with regard to scale prevention, the continuous still showing no vestige of scale after three months operation, but there are no reports of the application of Arroyo's methods though his treatment for rum production would appear applicable to Jamaican distilleries.

#### THE PRODUCTION OF SEED YEAST IN PURE 2. CULTURE.

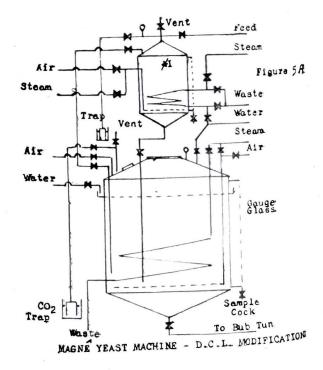
The advantages of "pure culture" yeast seeding do not need any emphasis since most modern distilleries have adopted this procedure. For the benefit of these who are unacquainted with the technique an outline of the method is appended to this paper. This outline has been kindly provided by Dr. E. V. Bell of Distillers Company Limited, the foremost authority on alcoholic fermentation in Great Britain and it would not be out of place here to express my appreciation of the assistance rendered by Dr. Bell, who was a colleague of mine in England, in supplying much of the information which has made this paper possible.

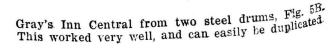
The initial stages of yeast selection are both tedious and exacting, individual yeast cells being isolated by micro-technique and cultivated. The whole of the yeast population used in seeding fermenters throughout the season are usually derived The magnitude and rate of from a single cell.

growth up to the seeding stage is somewhat inadequately illustrated by Fig. 4.

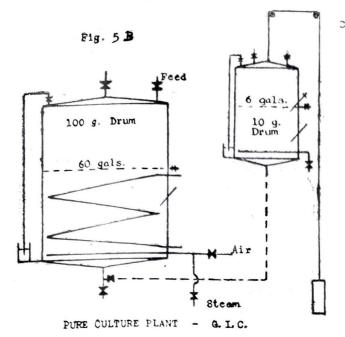


The "Yeast Machine" as standarised by Magne and by Pfaulder is well known, and many modifica-A British modification is shown in tions exist. Fig. 5A together with a simple plant fabricated at





THE J.A.S.T. JOURNAL.

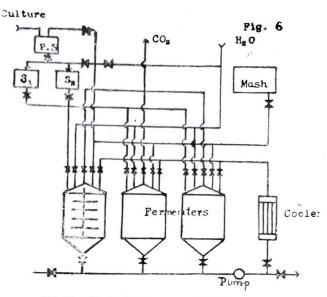


The usual Magne machine is somewhat complicated in any earance, but is not unduly difficult to operate although the laboratory stages of yeast culture demand scrupulous cleanliness and considerable skill; however, by the use of dried yeast it is possible to avoid all but the last laboratory stages. The main advantage of the yeast machine is that it can be operated for lengthy periods without the introduction of fresh culture, provided that the wash used in it is always sterilised before use. In most distilleries other than those in the British West Indies, pasteurised molasses is used in mixing the wash but even here, where no such precautions are taken, the fact that pure culture seeding gives a predominance of active, healthy cells of a desirable yeast strain should be sufficient to ensure vastly improved results.

# 3. INCREMENTAL FERMENTATION AND THE USE OF HIGH DENSITY MOLASSES.

In general, it is not found possible to increase the alcohol content of dead wash above some 8% in normal fermentation procedure but it is obvious that could this figure be raised to, say, 11-12% al-ohol by volume, the saving in time and labour and the increased throughput would be well worthwhile. Since the strains of yeast usually employed in distilleries will not effect complete fermentation of mashes of higher density than about 22° Bx. the increase in final alcohol content cannot be brought about by the use of heavier mashes under normal fermentation conditions, although it is possible to acclimatise the yeast to slightly increased concentrations of molasses in mash. In order to overcome this difficulty the principle of incremental fermentation has been introduced and this is well illustrated by Arroyo's technique for production of alcohol.

Arroyo's method (1) incorporated his molasses treatment and made use of closed fermenters with an external heat exchanger for cooling the fermenting mash. Stirring mechanism is also fitted (Fig. 6). The whole of the water required for the fermentation is placed in the fermenter and 50% of



23 NOV 1950

**FERMENTATION** SYSTEM - ARROYO

the total molasses to be used added as thick mash yielding a thin mash of  $20 - 24^{\circ}$  Bx. and pH 5.0 - 5.2. Seeding with an active pure yeast culture is at the rate of 10-15% on total volume and the seed is added with gentle agitation. After twelve hours a further 30% of the total thick mash is added with stirring and the pH adjusted if necessary. Fermentation is allowed to proceed for a further six hours when the remainder of the molasses is added (20%), again adjusting the pH, and the fermentation carried to completion. Temperature is con-trolled within the limits of  $28 - 30^{\circ}$  C. throughout the fermentation by means of the external cooler. It will be seen that the molasses is added over a period of eighteen hours, while the entire period of fermentation is from 45-60 hours, not a great increase over the normal, particularly when the concentration of alcohol in final wash (up to 12%) is taken into account. It would not be possible to ferment a mash containing 22-23% T.S. as I.S. directly by the usual methods without a substantial drop in alcohol yield and it is claimed that the incremental method gives a very high concentration of alcohol in the fermented wash in a reasonable fermenting time, and an increased throughput for any given size of plant, resulting in considerable reduction in the cost of production.

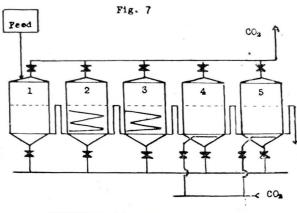
A modified procedure has been adopted in Queensland, the mash being mixed to  $24-26^{\circ}$  Bx., and added to the fermenter in eight equal increments, spaced over an arbitrary time range found by experiment but averaging rather less than two hours between additions. Seeding is at the usual rate of 6% on total volume, a specially acclimatised yeast culture being employed, molasses additions are completed in 12-13 hours, and the fermentation finished in 30 -- 36 hours. Although the initial temperature is not allowed to exceed 32°C. no attempt is made to control the temperature of the fermenting mash and this may rise as high as 40° C., apparently without ill effect. The alcohol concentration in final wash is between 8-10%bv volume, not as high as in the Arroyo method, but the time of fermentation is considerably lower and a greater throughput with less fermenter capacity is claimed.

 $\mathbf{43}$ 

In considering the fermentation of high density mashes it is interesting to note Arroyo claims (8) that the addition of Turkey Red Oil (1: 6000 by volume on mash) gives an increase in efficiency and prevents any tendency to foam. There would seem to be some credence for the latter claim as it has been the practice in Great Britain for many years to add small proportions of antifoam oil to the fermenters. Improvement in distillation properties, is also claimed, scaling being reduced and foaming, characteristic of high density washes, almost entirely absent.

## 4. CONTINUOUS FERMENTATIONS.

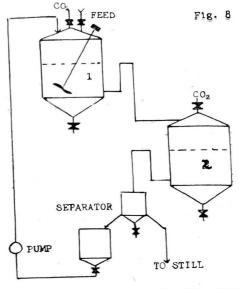
It has long been thought that it should be possible to operate the process of fermentation in a



CONTINUOUS FERMENTATION - ALZOLA

continuous manner and several patents have been taken out, or suggestions put forward, to make this possible. However, there would seem to be some doubt as to the success of these methods which are, in many cases, both complicated and costly. The concept of fermenting mash overflowing from one fermenter to the next in a series of vessels was made use of by Alzola (9) (Fig. 7). A pure yeast culture is developed in the first fermenter where it is fed continuously with sterile mash. The first fermenter being filled, the fermenting mash flows through suitable connections to the second and thence to the third, fourth and fifth, the feed to the first being controlled according to the stage of the fermentation, the sugar content of the mash decreasing and the alcohol content increasing in each succeeding fermenter. All fermenters are worked full, and cooling coils are fitted to the second and third, while agitation of the mash in the last two fermenters is effected by passing carbon dioxide through the liquid. The process need only be discontinued when the yeast has either degenerated or become infected and it is then possible to employ the fermenters in reverse order and start a new fermentation in the opposite It is claimed that considerable economy direction. of labour is effected by this process, and that the fermentation cycle is shorter (due to the elimination of the cleaning period). The greatest objection would appear to be that yeast is being withdrawn continuously from the system and fresh yeast must be grown, at the expense of alcohol production, to replace it or the rate of fermentation would otherwise fall away.

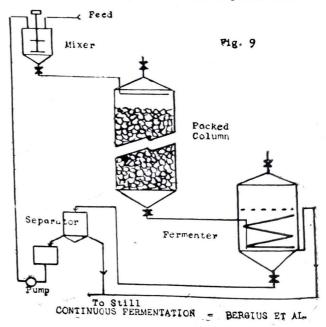
Karsch (10) (Fig. 8) attempted to overcome this by recycling the yeast. In his process the entire



CONTINUOUS FERMENTATION - KARSCH

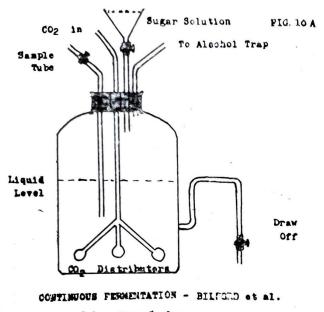
quantity of yeast used is fed continuously into the first fermenter together with the mash to be fermented, thence to a second fermenter, and to a centrifugal separator from which the yeast, as a thick cream, is returned to process, the fermented wash, free from yeast, being sent to the still. If complete fermentation could be effected in the two fermenters, it would appear that this process could work continuously since the yeast is rapidly removed from contact with the fermentation products and returned together with fresh mash in the first fermenter.

In the process of Bergius, Koch and Zimmerman (11) (rig. 9) a packed column is placed between



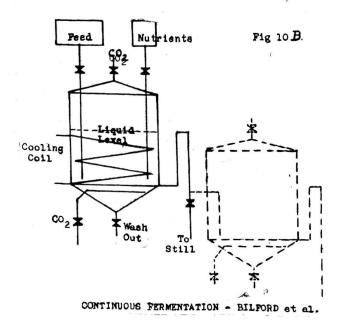
the two fermenters (of Karsch) and the mash from the second fermenter is either passed through a continuous separator, the yeast being returned to the first fermenter where mash is added continuously or part is withdrawn continuously and the remainder recirculated. The latter method was the original proposal but, since it incurs the withdrawal of yeast with the wash, it would seem that the use of a separator would be preferable.

Bilford et al (12) have developed a laboratory

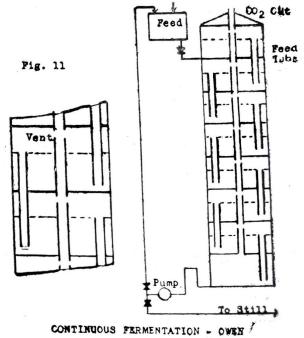


Laboratory Scale

process involving the use of a single fermentation vessel but this method could easily be adopted to large scale use if thought feasible (Figs. 10A and 10B.)

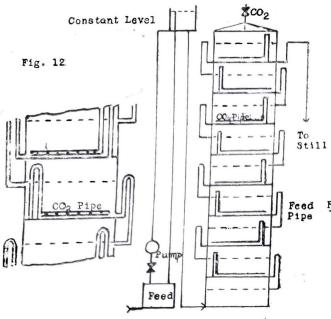


Feed and nutrients (as required) are fed continuously into the fermenter which is fitted with a cooling device and means of injection of carbon dioxide for agitation. A very high yeast concentration is employed and the fermented wash is withdrawn continuously by means of a constant level device once the fermentation has proceeded sufficiently. As the reported densities of the fermented washes are high, it would appear that fermentation cannot be completed in one vessel, and the use of several fermenters in series would be advisable. In order to keep the yeast population at a sufficiently high figure a high rate of feed will be required to maintain sufficient sugar and nutrients in the solution,



and it might be preferable to make addition of fresh mash and withdrawals of fermented wash alternatively, when it is suggested that the process might become feasible in one fermenter.

The use of a column down which fresh mash trickles has been proposed by Owen (13) (Fig. 11) but this process has only been employed on a laboratory scale. The mash and yeast are introduced at the top of the column, and the yeast tends to stay on the upper plates. The fermented wash is withdrawn at the base and may be recirculated if necessary. The reported efficiencies are rather low, even after recirculation, but it is possible that some improvement might be observed on a larger scale. Victorero (14) (Fig. 12) makes use of a special type of column, consisting of a series of superimposed fermenting vessels. The fresh mash is fed into the lowest compartment and moves upwards through each section, being completely fermented by the time it is withdrawn at the The carbon dioxide evolved in each comtop.



CONTINUOUS FERMENTATION - VICTORERO

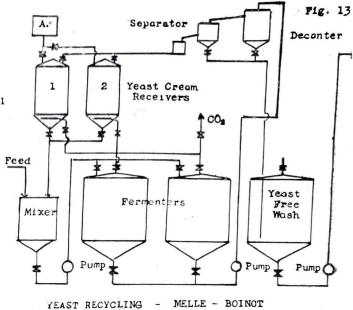
partment is used to agitate the fermenting liquid in the compartment above promoting rapid fermentation, since the yeast is prevented from settling out. It would appear that this column is complicated in design, the feed and carbon dioxide pipes, in particular, being rather involved while, as in several other designs, yeast is withdrawn with the fermented wash. A further patent has been taken out on the same lines by De Mattos (14A).

The advantages of continuous fermentation would seem to be a reduction in the size of plant required for fermentation, saving in labour and a more rapid fermentation, but these may well be offset by the necessity for replacement of yeast and the lack of complete fermentation.

## 5. FERMENTATION PROCESSES INVOLVING THE RE-CYCLING OF YEAST.

The possibility of re-using the yeast crop from a fermentation has already been mentioned and the process has been developed very successfully. In the Dansk Gaerings — industri process (15) a very rapid fermentation is secured by seeding the mash at the very high rate of 25 - 500 Kg. (at 75% water content) per 1000 litres of mash. In this manner the duration of fermentation is reduced to about 1/20th. of that required for an ordinary batch fermentation seeded at the usual low rate of about 0.4 Kg./1000 litres. When fermentation is complete, the yeast is recovered by centrifuging and is re-used for another similar fermentation. It is said that this process can be continued for several months, but no claim for increased alcohol production is made, the purpose of the method being to reduce the time of fermentation very considerably.

The Boinot process of the Usines de Melle (16) also involves the recovery of the yeast at the end of the fermentation and its re-use in subsequent fermentations. The proportion of yeast to mash is about 10 Kg. per 1000 litres, this being regarded as the optimum for maximum alcohol production. The fermentation is carried out in the normal (batch) manner and all the yeast separated by centrifuge. The yeast is washed with acid and re-used for the succeeding fermentation (Fig. 13). In ordinary fer-



mentation, yeast growth always accompanies alcoholic fermentation, and from 3 - 6% of the total sugars are utilized in this manner. The suppression of this comsumption of sugar is effected by the Boinot process and a substantial increase in yield thus made possible. This is achieved by maintain-ing the "specific cellular concentration" at a constant figure so that reproduction of yeast cells takes place only to replace the very small proportion of cells which die in each fermentation procedure rather than on the strain of yeast employed as is indicated by the fact that different distillers employ different strains of yeast. It is claimed that, since the yeast, as separated, is almost free from infecting organisms, it is unneccessary to sterilise the molasses or fermentation plant so that there should be considerable saving in this respect in addition to the increase in yield said to be obtain-Since the throughput with this process for a ed. given fermentation plant is considerably greater than the normal method, (2 to 3 fold), capital expenditure in installation should be reduced substantially. Judging by the number of distilleries which have adopted the Boinot process and which express satisfaction with the results, it would appear that the claims are justified and it is possible that, without any undue additional outlay, the process might in a modified form be applied to our alcohol distilleries.

## 6. SPECIAL RUM FERMENTATION.

Little has been said so far about progress in the production of rum but a considerable amount of work has been done by Arroyo (17), some of which is thought worthy of comment. All of Arroyo's methods involve the use of specially prepared molasses and of pure culture yeast footings. In the manufacture of light rums which have, up to the present, usually been made by the blending of industrial alcohol with heavier rums, Arroyo (3) used purified molasses mash diluted to contain the optimum sugar concentration for the yeast employed. The yeast, which is specially selected for the purpose, being one that yields sufficient proportions of the desired "congenerics" and which will withstand high alcohol concentration, is grown in pure culture, and seedings made at the rate of 10% on total volume. Closed type aseptic fermenters of polished steel or iron (inside surfaces) are used and these are fitted with stirrers and an external cooler. The yeast concentration in the mash (Fig. 6). should be of the order of 5 x 107 cells per ml., and the mash is adjusted to a pH of 4.5 - 4.7, nutrients being added as required. The temperature is controlled within narrow limits  $(27 - 30^{\circ}C.)$  and the fermentation is rapid, being complete in thirty hours. Stirring is employed towards the end of the fermentation to keep the yeast cells in contact with the solution, and the wash is centrifuged after leaving the fermenter. This method, it is claimed. gives a straight light rum in good yield, free from undesirable impurities.

Fermentation at constant pH has also been suggested by Arroyo (18), being carried out in the presence of 0.1% w.v. sterile calcium carbonate at a pH of 5.8 - 6.0. No agitation is employed and it is assumed that internal cooling would be preferable in this case.

For the manufacture of intermediate types of rums, the use of mixed yeast culture is proposed (19), two or more strains of yeast being employed. While it is possible to carry out separate fermentations with each strain and to combine the fermented washes in the requisite proportions, the use of mixed cultures is preferred although the growing of these to the seeding stage and the maintainance of the correct proportions of each strain is very intricate and demands considerable skill in technique.

In the production of heavy rums the use of bacterial cultures is advocated (20). Molasses treated by the Arroyo process for rum production (3) is diluted to contain 12 - 13% T.S. as I.S. and fermented with a pure culture yeast, preferably of the fission type (Schizosaccharomyces); a 10% v.v. seeding of active yeast being employed. External cooling is employed and the temperature of the fermenting wash must be kept within the range of  $30 - 33^{\circ}$ C. The initial pH should be adjusted to 5.5 - 5.8.

After six hours, the total sugar and alcohol concentrations are tested every two hours, and when the alcohol concentration lies between 3.5 - 4% by volume, and the total sugars fall below 6% w.v. the pH of the liquid is again adjusted to its initial value, a 2% v.v. footing of a pure culture of Clostridium saccharobutyricum at pH 5.5-5.8 is added with gentle stirring, and the fermentation allowed to proceed to completion at a temperature of  $29 - 30^{\circ}$ C.

The sugar and alcohol concentrations are chosen to give optimum conditions of fermentation for both yeast and bacteria which, it is claimed, exhibit symbiosis. The ratio of bacterial footing to yeast footing should not exceed 1:4 and 1:5 has been shown to be the optimum value for Jamaican type heavy rums. Other types of bacteria such as Propionobacterium Technicum or moulds (Oidium suaveolens) yield rums with different characteristics. It is claimed that an exact control of the quality of the rum can be obtained in this manner, that the yield is higher, and that ageing is very rapid.

# 7. CONTINUOUS AND FRACTIONAL DISTILLA-TION IN RUM PRODUCTION.

The pioneer work of Arroyo in rum production has now been generally recognised and it is no longer customary to pass over his ideas with a cursory glance. Hence, his work on the distillation of rum, while not, perhaps, directly applicable in Jamaica, should be given careful consideration as it opens up a new field in distillery practice. The fermentation process for the production of straight light rums has been mentioned; the distillation procedure is also somewhat unusual (21.) The fermented wash, freed from solid matter by centrifuging, is distilled in a continuous still of special design. While the description of this still is rather vague, it would seem to be a two column apparatus, the second being designed as a "purifying column" which not only separates the more volatile of the undesirable constituents as heads but also acts, to a certain extent, as a reflux. The product, which is drawn off from the base of the purifying column, is kept within the range 85 - 90% alcohol by volume, a somewhat higher strength than is customary in this Island. A later paper (22) suggests the use of fractionation as a means of producing rums of varying characteristics from one and the same fermentation. The fermented wash is first distilled in a continuous still giving a main fraction of "neutral" spirit, comprising some 85% of the total distillate, and a secondary fraction, presumably comprised of heads and fusel oils combined. containing those constituents which are needed to constitute rum together with alcohol and water. An alternative method is also suggested where no separation is effected in the first (or beer) distillation. This smaller fraction is collected until sufficient has been obtained for further distillation, when it is fractionated after dilution to 45 - 50% alcohol by volume. A specially designed still is employed and five fractions are collected. The first discarded as containing undesirable constituents, the third, which is practically pure alcohol and water, is added to the neutral spirit, while the second, third and fifth fractions are combined in varying proportions with neutral spirit to yield rums of differing characteristics. It is claimed that the rums produced in this manner are of constant quality, that the ageing period is greatly reduced due to prior removal of undesirable constituents, and that the character of the product can be varied at will to suit the immediate market. Cost of production is not, it is said, unduly increased due to the greater overall efficiency of the process.

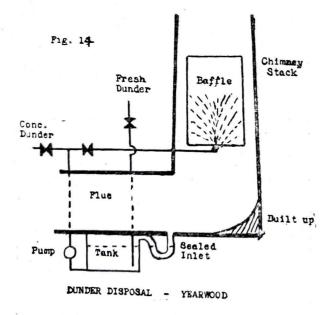
# 8. EFFLUENT DISPOSAL.

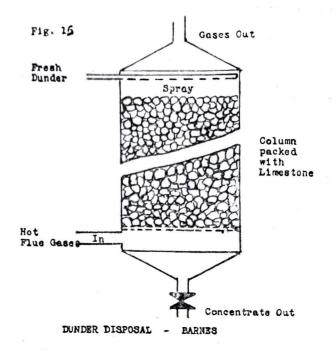
The problem of efficient and adequate means of dunder disposal has been before the Jamaican Technologist for some years now but the solution does not seem appreciably nearer. It is, perhaps, comforting to think that a similar situation exists elsewhere in the world though this does not prove of any assistance in providing a solution.

An exhaustive bibliography of the methods of dunder disposal was prepared by Davies (23) and this gives some idea of the vast amount of work and time which has been devoted to this problem. The micro-biological processes suggested by various workers are well known and they have been adequately described by Southgate (24). While they would seem too extensive and costly to instal at individual distilleries in Jamaica and while their efficacy is still open to some doubt, it is of interest to note that the production of methane, for use as a fuel, by anaerobic digestion (25) would seem to be an economic proposition. Unfortunately this would not solve the dunder problem since the B. O. D. value, while substantially reduced, is still relatively high.

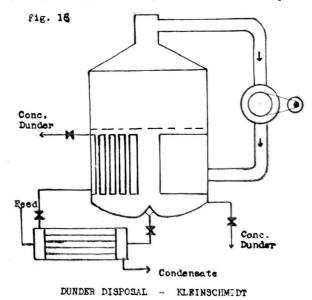
Disposal and simultaneous application as a fertilizer offers a partial solution in some cases, particularly where the dunder may be mixed with irrigation at or near to the distillery or where the points of direct application in undiluted form are reasonably close. Innes (26) has inevstigated the value of dunder as a fertilizer and advocated its use in this manner.

The recovery of valuable by-products might make direct evaporation and/or incineration an economic proposition and, in this respect, the processes of the Societie des Sucreries et Distilleries du Soissonais, involving the production of a product containing nitrogen, potash and organic matter, and the recovery of glycerol, and of Walmesly (27) for recovery of glycerol by liquid-liquid extraction where slopping-back is practiced, are of some interest.





If direct concentration is to be considered as the most logical propostion in spite of the initial expenditure, a compression distillation system based on Kleinschmidt's heat pump principle might prove economical, since the power equivalent required per lb. of water evaporated in a single vessel of this type is approximately one half of that required



Concentration of dunder by normal means would prove expensive and the capital outlay high. Yearwood's proposal (28) to evaporate the dunder by spraying into the hot gases at the base of the factory chimney (Fig. 14) has not met with general approval but the use of a column packed with limestone and heated with flue gases, as suggested by Barnes (29), may hold out greater possibilities, (Fig. 15). in a normal quadruple effect. This principle was used during the last war for the production of potable water from sea water (30) and the plant was some 15 - 20 times as efficient as a single effect evaporator. Fig. 16 illustrates the apparatus in a simple form but it is not necessarily to be assumed that it could be adopted in this form since it has not been tried out on the large scale. However, for dunder concentration, the initial and operational costs should both be substantially lower than for the conventional type of evaporator. Author's Note:

In the foregoing paper the term mash is used for all media' prior to completion of fermentation. The terms thin mash, wort and live wash are therefore more or less synonymous. Fermented wash is of course dead wash, while dunder is also termed slop or lees. Total sugars are expressed as invert sugar (T.S. as I.S.).

#### BIBLIOGRAPHY.

- 1. R. Arroyo U. S. P. 2275150 I. S. J. 45, 219, 250, (1943) Sugar 38, 2, 18, (1943) Sugar J. 11, 8, 5, (1949)
- 2. W. L. Owen Sugar 43, 4, 37, (1948)
- 3. R. Arroyo U. S. P. 2386924 I. S. J. 44, 107, 163, (1942) 45, 53, 135, (1943) 48, 101, 250, (1946)
- 4. G. Reich Trans. Am. Inst. Chem. Eng., 38, 1049, (1942) I. S. J. 47, 218, 306, (1945) Sugar 40, 3, 26, (1945)
- 5. W. H. Buhrig & H. M. Harff, (Standard Brands Inc.) U.S.P. 2337641
- 6. R. Arroyo Studies in Rum, 1945 p. 39.
- 7. Bradshaw Proc. Conf. B.W.I.S.T., Antigua, 1949
- 8. R. Arroyo Sugar 43, 1, 31, (1948)
- 9. F. Alzola U. S. P. 2371208 Proc. 14th. Conf. Assoc. S.T. Cuba, p. 317 I S. J. 44, 274, (1942)
- 10. W. Karsch U. S. P. 2155134 I. S. J. 41, 322, (1939)

11. F. Bergius et al., U. S. P. 2146326 I. S. J. 41, 405, (1939)

- 1? H. R. Bilford et al., Ind. Eng. Chem. 34, 1406, (1942) I. S. J. 45, 81, 124, (1943)
- 13. W. L. Owen Sugar 43. 2, 36, (1948)
- 14. F. A. Victorero U. S. P. 2450218
- 14a. R. de Mattos U. S. P. 2451156,

I. S. J. 51, 316, (1949)

- 15. Aktiesels Kabat Gaerings-industri, B. P. 401495
- 16. Usines des Melles & F. Boinot, B. P. 393082 420878 455339

				483363
I. S. J.		37,	241,	(1935)
				(1936)
	39.	112.	200,	(1937)
				(1939)

17. R. Arroyo See "Studies in Rum," Puerto Rico, 1945, and numerous papers in I.S.J. and other journals.

- 18. R. Arroyo Studies in Rum, 1945, p. 114.
  - 19. R. Arroyo Ibid., p. 117.
  - 20.
     R. Arroyo
     Sugar
     40,
     11,
     34,
     (1945)
     I.
     S. J.
     48,
     101,
     (1946)
     101,
     (1946)
     101,
     (1946)
     101,
     (1946)
     101,
     (1946)
     101,
     (1946)
     101,
     (1946)
     101,
     (1946)
     101,
     (1946)
     101,
     (1946)
     101,
     (1946)
     101,
     (1946)
     101,
     (1946)
     101,
     (1946)
     101,
     (1946)
     101,
     (1946)
     101,
     (1946)
     101,
     (1946)
     101,
     (1946)
     101,
     (1946)
     101,
     (1946)
     101,
     (1946)
     101,
     (1946)
     101,
     (1946)
     101,
     (1946)
     101,
     (1946)
     101,
     (1946)
     101,
     (1946)
     101,
     (1946)
     101,
     (1946)
     101,
     (1946)
     101,
     (1946)
     101,
     (1946)
     101,
     (1946)
     101,
     (1946)
     101,
     (1946)
     101,
     (1946)
     101,
     (1946)
     101,
     (1946)
     101,</t
  - 21. R. Arroyo, I. S. J. 51, 150, (1949)
  - 22. R. Arroyo Sugar 44, 7, 34, (1949) I S. J. 50, 289, (1948)
  - 23. J. G. Davies J.A.S.T. 8, 3, 17, (1945) I. S. J. 48, 266, (1946)
  - B. A. Southgate "Treatment & Disposal of Industrial Wastes." H.M. Stationery Office, London, 1948.
  - 25. A. M. Buswell Ind. Eng. Chem. 31, 1349, (1939)
  - 26. R. F. Innes Proc. Conf. B.W.I.S.T. Antigua,
  - 27. Walmesley (I.C.I., Ltd.), B.P. 578259, 578260
  - R. D. Yearwood & C. Fitzwilliam I.C.T.A. (Trinidad, Circ. 55, May 1946
    I. S. J. 48, 321, (1946)
  - 29. A. C. Barnes Proc. Conf. B.W.I.S.T. Antigua, 1949, (See Ref. 25)
  - 30. A. Latham Mech. Eng., (1946), p. 221 Sugar 43, 6, 46, (1948)

#### APPENDIX.

# THE BATCH FERMENTATION PROCESS AS PRACTISED IN GREAT BRITAIN. DR. E. V. BELL DISTILLERS COMPANY LIMITED.

The production of industrial alcohol from mclasses in Great Britain today is largely carried out as a batch process starting in the distillery laboratory with a pure culture of a specially selected yeast strain. A molasses solution containing 10--11 per cent T.S. as I.S. is prepared in the laboratory and is suitably fortified with yeast nutrients such as ammonium sulphate or phosphate or a mixture of the two and is acidified with sulphuric acid to pH 4.6-4.8. The initial seed stage is a few ml. of wort contained in a test tube and this is inoculated from an agar slant culture of the particular yeast. It is incubated at about 84°F. for twenty four hours and then transferred to the next larger seed stage which is about ten times the volume. By further stages, each increasing about ten times in volume, an actively fermenting liquid seed yeast culture of about four litres is produced in the laboratory. All the laboratory scale seed stages are pressure sterilised 30 that the yeast remains a pure culture, uncontaminated by other organisms.

The laboratory culture is used for inoculating the first stage of a pure culture yeast machine and in Great Britain, the type of culture plant invented by Magne is largely employed. This apparatus is made of tinned copper and has two stages of approximately 40 and 400 gallons capacity, the first stage being situated above the second. Both stages

1949.



are charged with molasses of a similar composition to that used for the laboratory seed stages, and they are pressure sterilised. The Magne machines are rather complicated in appearance and are fitted with means for maintaining the desired temperature during fermentation by either heating or cooling. They also have connections for supplies of steam and sterile compressed air. The outlet pipe for the carbon dioxide produced during fermentation is trapped so that the gas has to pass through an antiseptic solution such as formaldehyde to prevent the ingress of infection. After inoculation of the 40 gallon stage with the four litre laboratory culture. a temperature of 86°F, is maintained and moderate aeration may be employed to assist the growth of the yeast culture, which takes about twentyfour hours. The 40-gallon stage is then transferred by gravity to the 400-gallon stage which is similarly fermented. Immediately the 400-gallon stage has been inoculated, 40 gallons of the inoculated wort are transferred back to the empty first stage by means of air pressure and allowed to ferment there, serving as an inoculum for the next 400-gallon charge of wort in the second stage. In this way, once the yeast culture machine has been inoculated with a laboratory culture, it is possible to carry on the development of yeast cultures for a long time. In practice, in this country, it is customary to carry the culture on for one week only and then to reinoculate the first stage with a fresh culture from the laboratory. With a battery of these yeast culture machines, the second stages of each machine are sometimes connected by cross-transfer pipe lines so that once one machine has been inoculated with a laboratory culture, each of the others can be started up from this initial fermentation. For the successful operation of the Magne culture plant it is essential that all valves, cocks and joints are maintained in good condition, otherwise infection is liable to obtain an entrance. Air filters must be kept efficient and careful supervision of the plant is essential. The internal tin coating of the two copper vessels must be kept intact, otherwise the yeast culture is liable to become enfeebled or to die off, due to the toxic effect of copper contamination.

The next and last step in the development of the seed yeast is the "bub" stage which is carried out in a closed vessel, usually made of steel, called a These vessels are of approximately 4000 bub tun. gallons capacity (ten times as large as the 2nd. stage yeast culture vessel). The wort is prepared and pasteurised in the bub tun. Sufficient molasses to give a wort containing about 12 percent T.S. as I.S. is run into the bub tun and rather less than an equal volume of water is added together with ammonium sulphate as yeast nutrient (0.1% w.v. on the wort) and sufficient sulphuric acid to give a wort pH value of 4.6 to 4.8. Live steam is used to bring the solution to the boil and the mash is pasteurised by holding it there for a short time. It is then cooled by means of internal cooling coils and diluted to volume with cold water, the temperature being adjusted, usually to 86°F., before seeding with the 400 gallons of culture from the yeast machine. During the bub stage, temperature is controlled by the internal cooling coils and is not allowed to rise above 90°F. Mild aeration may be employed. The bub fermentation is allowed to proceed until about 2/3 of the sugar in the wort has been fermented before using it as seed in the final fermentation stage. The bub fermentation is the first stage which is not absolutely sterile. The pasteurisation of the wort destroys vegetative forms of infecting organisms present in the molasses and any infection from spores, which take longer to develop, is kept in check by the actively fermenting wort with its appreciable alcohol content and anaerobic conditions due to the carbon dioxide evolved.

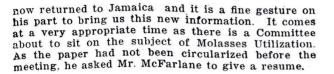
The size of the final fermenter stage corresponding to a 4000-gallon bub is about 70,000 gallons, so that the rate of seeding is between 5 and 6 percent bv volume. The sizes of the fermenters at the various Distilleries in this country vary from 35,000 to 100,000 gallons. Of recent years there has been a tendency to employ large fermenters of 70,000 to 100,000 gallons capacity. These are made of mild steel, rivetted or welded, or of wood, and are closed vessels so that the by-product carbon dioxide can They are fitted with attemporating be collected. coils. Wooden fermenters are less easy to keep clean bacteriologically than steel ones since they will not stand steam sterilisation. They probably have a longer life since they do not suffer like steel from the corrosive effect of the slightly acid molasses solutions. Owing to the better thermal insulation of the wooden vessels, a greater area of cooling coils is required than in steel fermenters to control the fermentation temperature.

Usually the fermenter worts are pasteurised and they are prepared in a separate molasses dissolving vessel. Into this vessel, molasses for one or two fermenters is run, the quantity being measured by means of a pneumercator gauge fitted to the molasses storage tank. It is diluted with a little water and sulphuric acid sufficient to give a wort of pH 4.6-4.8is added. The mash is heated and dissolved with live steam and raised to a temperature varying from about  $180^{\circ}$  to  $212^{\circ}$ F. The desired temperature is held for a sufficient time to pasteurise the mash which is then cooled as it is pumped to the fermenter, where it is diluted with cold water and ammonium sulphate added as yeast nutrient (usually 0.1% w.v.).

The temperature is adjusted to a suitable degree for starting the fermentation and may vary from 80° to 86°F. depending on the season of the year, whether the fermenters are of wood or steel and whether they are situated inside a fermenter building or are outside and unprotected from the weather. The bub seed stage is run into the fermenter and mixed by slight aeration after which no further air is used. The fermentation rapidly gets underway and the temperature rises and is controlled by the attemperators and held at a maximum of about 95° to 98°F. until the fermentation slackens when the cooling is stopped and the temperature allowed to fall naturally. Fermentation usually takes about forty-eight hours to complete. The worts in the fermenters usually contain from 12.5 to 17% T.S. as I. S. and give washes containing from 7 to 9% alcohol by volume. The alcohol carried off with carbon dioxide may be recovered by water washing the gas or by means of activated carbon and amounts to approximately 1 per cent of the alcohol produced.

#### DISCUSSION.

The Chairman said he was sure that all members were glad to see Mr. McFarlane again present a paper before this Section. They would remember the high standard of his papers prior to his departure for England. During his stay in England he took up an appointment with the Distillers Company. He has



Mr. McFarlane then gave a resume of his paper with illustrations. In the discussion which followed Mr. Floro referred to Bradshaw's modification of the Reich process and inquired as to the Brix to which the molasses was diluted.

Mr. McFarlane replied that it was about 30 - 35Brix (400 gallons molasses diluted to 1,000) and the mixture settled for 3 hours.

Mr. Floro expressed appreciation for the interesting paper which had been presented, but wished to know whether it was not usual to aerate in growing the seed, whether the particular yeast for any of these modern processes would have to be specially chosen as they might be overcome by the more virile wild yeasts, what was the generally accepted optimum level of yeast population of fermentation and the reason in continuous processes for the recirculation of the  $CO_2$ ?

To these questions Mr. McFarlane replied that the aeration was usual in all seed plants. As regards the type of yeast, opinions differed; in the Boinot process it was claimed that there would be no difference while in the Australian process a special yeast was used. Fleischmann's dried yeast as commonly used in local distilleries is suspected to be a mixed culture or at least not a pure culture. With regard to the yeast cell concentration the average in batch fermenters is fifteen to twenty million per ml., while in the incremental process a concentration of 500 x 106 cells per ml., was used, and sometimes even higher, even 500 kg. (at 75% H<sub>2</sub>O) per 1,000 litres as in the Danish recycling process. The CO., was recirculated only to agitate the wash.

Hon. F. M. Kerr-Jarrett inquired whether a fission yeast was used in these processes. It was his impression that only a budding yeast was used for light rum production. Furthermore with the restriction of the rum crop would it be possible to use the same yeasts for rum and alcohol production? Finally he felt that by a more efficient process there would be savings in fuel since extra efficiency must give better economy.

Mr. McFarlane replied that Arroyo alone used a fission yeast in his process for heavy rum. The normal yeast of rum production was the budding yeast. Further, rum and alcohol could be made by the same yeast provided a pure culture was maintained. As regards the manufacture of Absolute Alcohol he sounded a warning that the process was both skilled and difficult as was the manufacture of dry ice, unless the operator was fully conversant with the techniques.

Mr. Barnes observed that in the past the local department of Agriculture had had a Microbiologist on its staff, one of whose duties was the production of pure culture yeasts for the distilleries. However, for the past 17 to 20 years the work had been discontinued. He felt that the post might have to be re-instituted if they were to achieve and maintain the highest efficiency.

Mr. Floro felt that Mr. McFarlane was unduly pessimistic over the difficulties of Absolute Alcohol manufacture. There were no less than 15 plants operating in Cuba while in the United States every city had its almost automatic dry ice plant. What was important was proper equipment and a good start.

Mr. McFarlane agreed with Mr. Floro and stated that he had meant that a qualified man was necessary at the start but that once we had been shown how, we surely had enough intelligence to carry on.

The Chairman in closing the discussion thanked Mr. McFarlane for his stimulating paper. For those who tried to keep abreast of the fermentation literature, it was difficult to see how it all fitted and Mr. McFarlane had made a fine job of tying it up into a neat parcel. This paper was probably the most important one to be presented to the Meeting and he knew that all would join him in thanking Mr. McFarlane for it. He believed that Mr. McFarlane had further comments to offer on the dunder disposal problem and these would no doubt be heard later.

The President in closing the paper reiterated the Chairman's sentiments and thanked those who had taken part in the discussion. It will be noted that the value of the minor elements in the dunder is not included.

In addition it is felt that the bacterial action started by the dunder is most important in improving the fertility of the soil.

# COSTS OF DUNDER APPLICATION.

	Cost	s per day
Labour — Driver $10/-$ + boy	5/3	15/3
Gas — 10 trips at 2 miles		10/-
@ 6 m/g @ 3/- O:l & Grease — 10% of above		10/=

Tyres — £120 @ 8	8,000	miles			6/	-	
Repairs £60 for 6	,000 1	miles			4/	-	
Insurance & Licen	ce —	£30 for	6,00	00	2/	_	
Depreciation £700	for 55	,000 m.			5/	_	
Miscellaneous over supervision et	c.	cost per	day		- <mark>6/</mark>		•
Cost per acre	••••		=	£	1	4	8
Total saving per	day		=		5	14	11
Total saving per c	rop		=		600	0	0

# Further Experiments on Dunder.

A. C. BARNES, C.M.G., B.Sc., F.R.I.C., A.M.I., Chem. E., Director, T. A. F. SEXTON, B.Sc., F.R.I.C., Analyst. H. S. IVE, M.S.A., Agronomist, Research Department, The Sugar Manufacturers' Association (of Jamaica) Ltd.

-----:0:-----

#### INTRODUCTION.

The subject of dunder disposal and utilization has engaged the attention of the Association for several years, and has been studied by numerous workers in the B.W.I. and elsewhere, including the staff of the Research Department of the Sugar Manufacturers' Association (of Jamaica) Ltd. The problem of treating distillery slop, which we call dunder, in such a manner as to render it innocuous came under prominent notice in Jamaica with the enactment of the Wild Life Protection Law in 1944, although objections to the practice of discharging this material into streams and other places where a nuisance could be caused, date back for a great number of years. Within the experience of the senior author of this paper, attempts were made to discourage the practice of polluting streams sixteen years ago, and more recently, in 1939, legal action was taken against the leading Company manufacturing rum for the alleged pollution of a public stream by discharging dunder into it. The case was ultimately decided in favour of the Company concerned on the grounds that they had acquired a prescriptive right to pollute the stream in question. Such a position could not be sustained under the present Law.

The industry has long realised the necessity for intensive inquiry into this important matter and efforts have been made over a number of years by research workers and manufacturers of run to overcome the objections to former methods of disposal of distillery effluents.

In the earlier stages attention was focussed on possible methods of treating the effluent in such a manner as to render it unobjectionable and suitable for direct discharge into water courses or into the sea. In Jamaica experimental work was undertaken by J. G. Davies (1) (2), in co-operation with J. Munro (3) of the United Fruit Company at Bernard Lodge Dr. A. C. Thaysen (4), Director of the Imperial Microbiological Institute, Trinidad, has more recently made inquiries and carried out investigations with the same object. The detailed work of these investigations is on record, and conclusions may be summed up by a statement that rum distillery slop cannot be rendered innocuous except by a large and costly plant and the application of expensive methods of treatment. Experimental work in Jamaica indicated that the dunder from pot stills is more difficult to treat than the slop from grain distilleries. The Ohio River pollution survey car-ried out a few years ago shows that each 5,000 gallons of proof spirit produced requires the same facilities for disposal of distillery slop in an innocuous form as a sewered town having a population of 60,000 persons. J. G. Davies (5) concluding in 1945 that if all distilleries had to dispose of their dunder in such a manner, and assuming that the Ohio River pollution survey figures were applicable to Jamaica dunder, the aggregate size of the disposal plants required for the effluent for the whole rum crop would be equivalent to that serving a sewered town of 350,000. He emphasised the fact that plants would have to be situated near to distilleries with attendant increased costs of installation and operation.

If therefore the question is to be confined to one of treatment and disposal in an innocuous form, the magnitude of the problem will readily be appreciated, and it is safe to assume that such methods if put into practice would render the manufacture of rum entirely uneconomical, if indeed they did not destroy the Industry. An attendant difficulty which should be mentioned, is the seasonal character of rum manufacture which introduces further complications.

It has been demonstrated as a result of investigations of sugar cane soils carried out during recent years in Jamaica that many areas on which cane is grown are deficient in Potash, and that the extensive use of potassic manures is necessary for the economic production of sugar. This combined with the attention given to the dunder disposal problem has concentrated attention on the possibilities of using the material as a useful source of potash for use as a fertilizer, either by direct application or after some method of treatment. J. R. MeFarlane (10) published a paper in 1940 in which he summarised methods of obtaining potassic fertilizers from sugar factory and rum distillery residues including dunder. A meeting of the Chemical and Engineer-

ing Sections of this Association discussed the matter at their first Meeting in 1943 (6), and at the annual Conference held in December of the same year a paper (3) was presented on dunder disposal in which reference was made to the usefulness of the effluent when applied to soils deficient in potash. A further paper by J. G. Davies (1) in 1945 again made brief reference to this, though it mainly dealt with disposal methods, and contained a valuable bibliography. At the Ninth Annual Conference, December, 1945, M. B. Floro (7), dealt with the evaporation of distillery slop after having observed the operation of a new slop concentration plant in New Orleans. In a discussion at a meeting of the Chemical and Engineering Sections in December 1946 (8), attention was again called to the considerable manurial value of dunder applied to the land.

It is known that some estates have for many years made use of dunder in this manner by various means, but no experimental results were available until a paper by R. F. Innes (9) was presented to the 1949 meeting of B.W.I. Sugar Technologists in Antigua, in which significant response to the application of liquid dunder on potash deficient soils were recorded.

Prior to this the same author in a private communication pointed out that the aggregate quantity of potash removed from the soil by an island sugar cane crop of one million eight hundred thousand tons was equivalent to six thousand one hundred and fifty tons of Muriate of Potash worth approximately one hundred and twenty thousand pounds, and that the greater part of the potash in the reaped cane is accounted for in the distillery dunder. He referred to the composting with dunder with filter mud and excess hagasse at Frome, in regard to which it is expected that experimental results will be available during 1950 crop, suggesting that similar methods might be applied at distilleries situated on estates where the soil is deficient in netash.

The senior author has publicly discussed on many occasions the desirability of investigating methods of concentrating dunder and treating the concentrate in such a manner as to render it easily transportable, so that estates where potash deficiencies do not occur can more readily dispose of the dunder to deficient areas.

Although from such information as is available it appears that the cost of making a suitable concentrate would exceed its commercial value as a fertilizer, the net result would be a far less expensive method of disposing of dunder than chemical and biological treatment with a costly plant designed to render the material innocuous, without making use of any of its valuable manurial constituents. There is nothing inherently difficult in evaporating dunder to a syrupy consistency, but in this form it would be difficult to transport and apply to the land. In order to obtain some information regarding suitable metals for the construction of vessels for a concentration process, experiments were initiated in 1947 with the object of determining the differences in corrosion and behaviour of certain metals when immersed in hot and cold dunder. Corrosion tests were therefore conducted by J. G. Davies (5) with cast iron, copper, aluminium, brass, steel and stainless steel. In his report, which described the methods used, he pointed out that dunder has both a Of the six metals corrosive and erosive action.

tested, stainless steel, brass, and copper, in that order, were the only ones to show satisfactory resistance to both effects, stainless steel being practically unaffected either by hot or cold dunder after a period of ten weeks.

Although these results cannot be taken as couclusive because the actual analytical composition of the metals was not known, it is clear that any vessels for the concentration of dunder must be constructed of resistant material and that local corrosion at various points must be provided against. It is clear that further investigation is required before a satisfactory type of vessel can be recommended.

M. B. Floro's description of the equipment used in New Orleans is a useful starting point, and J. G. Davies' observation (5) that evaporation of the dunder should take place in a multiple effect with the first vessel under pressure should be noted.

The experimental work now to be described has been commenced with the object of ascertaining whether concentrated dunder can be converted into a solid form suitable for storage, easy transportation and ready and controlled application to the land. The inquiries have not been concerned with the methods of concentration which would have to be applied under economical systems of operation, but deal rather with any changes which occur during evaporation and later treatment, and of methods for making a solid product.

The subject has become of greater importance with the increasing costs of potassic manures and the difficulty of obtaining them in adequate quantities.

# EXPERIMENTAL.

The experiments which have been so far carried out in the course of this investigation have been of a simple nature as the time available has been insufficient for extensive work. The data obtained are however of interest, and indicate the desirability of pursuing further and more exhaustive inquiries.

Four lines of approach have been followed. The first deals with the concentration of dunder with and without the use of vacuum. The second demonstrates the manner in which the plant foods. viz., nitrogen, phosphoric anhydride and potash present in the original dunder are concentrated as moisture is lost. The third is concerned with loss of acidity during concentration whilst the fourth indicates possible methods of converting concentrated dunder into a dry powdered form.

#### THE ORIGINAL MATERIAL.

Ten gallons of dunder from a pot still was received from the Monymusk distillery of the West Indies Sugar Co. Ltd. As was normal, a layer of insoluble material was found on the bottom of the container, and was dispersed evenly throughout the liquid by agitation. After thorough mixture it was transferred to Winchester bottles to permit of easier subsequent handling.

#### CONCENTRATION.

(1) Without Vacuum. The original density of the dunder was 1.055. A kilogram of the wellshaken dunder was introduced into each of four evaporating basins which were then heated on a water bath and the contents concentrated to various extents. The temperature of the material reached about 87°C in this process. Table 1 sets out the data obtained

Sample	Approximate Degree of Concentration	Initial Weight Grms.	Final Weight Grms.	Residue %	Specific Gravity	Time
1	50% of original weight	1002.3	484.3	48.32	1.117	4 hours
2	25% of original weight	1003.7	252.6	25.17	1,233	6 hours
3	(maximum extent (possible under	1033.6	170.0	16.94	—	16 hours
4	(these conditions	1013.9	173.7	17.13	—	16 hours

TABLE 1.

(2) With Vacuum. A kilogram of well shaken dunder was concentrated for 16 hours on a water bath and then transferred to a vacuum oven. The weights of the residues obtained under various conditions of temperature and pressure were noted and are contained in Table 2.

TABLE 2.

Original weight of dunder taken .... In 1000 grms. Weight of Residue after concentration on water bath .... 166 grms.

Stage	Time	Temperature	Vacuum	Residue grms.	Residue %
A	45 mins.	98°C	16 ins.	145	14.5
B Further	105 "	98°C	16 "	137	13.7
С "	120 "	105°C	27.5 "	86.7	8.67

The final residue obtained from stage C was much too viscous to permit a gravity reading to be taker by means of a hydrometer spindle, and it set to a solid state when cool. This final residue however was very hygroscopic and appeared to be quite unsuitable to be dealt with on a commercial scale. The Plant Food Constituents:

The nitrogen, phosphoric anhydride and potash were determined in the original dunder and also in the concentrates 1, 2, 3, and 4, each analysis being carried out in triplicate.

Material	Nitrogen %	$P_{2}O_{5}\%$	Potash % as K <sub>2</sub> O
Original dunder Concentrate 1 "2 "3	0.17 0.34 0.67 0.97	0.05 0.11 0.20 0.28	1.63 3.66 5.20 8.51
., 4	0.97	0.28	8.51

TABLE 3.

It is obvious that the increase in concentration of plant foods present is in inverse proportion to the degree to which the dunder is concentrated. It will be noted that the nitrogen content of this particular sample of dunder was rather low, and that there was no loss of nitrogen in the evaporation process.

#### The loss of acidity from evaporation:

As there was no reason to determine the acidity in the dunder with a high degree of precision, a small volume was diluted and titrated with standard caustic soda solution using phenolphthalein as an indicator, expressing the acidity as percentage of acetic acid. When this procedure was followed it was found extremely difficult to judge the end point due to the highly buffered condition of dunder, and the darkening of the material in alkaline solution. Electrometric titrations were therefore carried out using the pH meter with a glass electrode and a calomel half cell with an efficient stirring device in the reaction beaker. Since phenolphthalein has a useful indicator range beginning at pH 8.3, the volumes of caustic soda corresponding to this value were read off from titration curves. The results obtained on the original dunder and the four concentrates are given in Table 4.

TABLE 4.

Material	Weight taken	Vol. N. 14 NaOH	% Acetic Acid
Original dunder Concentrate 1 ,, 2 ,, 3 ,, 4	10.44 grms. 5.54 ,, 5.92 ,, 1.64 ,, 1.98 ,,	33.0 mil. 32 59 59 50 50 50 50 50 50 50 50 50 50 50 50 50	1.48 2.47 4.27 5.36 5.30

The acidity figures show clearly that during evaporation there is a loss of acid material. Had there been no loss the acidity in concentrates 3 and 4 would have been approximately 8.3.

# Commercial Application:

As pointed out in the introduction, it would be highly useful if the dunder could be used in a dry form. Several experiments were therefore made wherein various quantities of plaster of Paris were added to the dunder concentrate No. 2. Quantities such as 1:1; 1:1.5; dunder to plaster of Paris were tried with varying degrees of success. It was noted that the time of setting occupied about 48 hours. Calculations showed that this is not economic because of the cost of plaster of Paris.

The possibility of quick lime being a suitable material for producing a dried powder suggested itself. Accordingly a fresh supply of dunder concentrate was prepared which had lost 84.3% of its original weight by evaporation. Of this material three 20-grm. lots were taken and the following mixtures prepared:

I. Dunder Concentrate Quicklime	Result 20 Dry powder 5	
II. Dunder concentrate Quicklime	20 Dampish product 2.5 Not very sticky	
III. Dunder concentrate Plaster of Paris Quicklime	20 Plastic product, 5 easily moulded which 2.5 set hard after 24 hrs	

The action of the quicklime was to dehydrate the dunder concentrate with the formation of a dry product.

As was expected ammoniacal smells were observed when the quicklime was added. The odour however was rather different from ammonia in that it had a fishy smell due possibly to the evolution of ethyl or methyl amines.

Whilst the loss of ammonia would not by any means invalidate the process from an economic standpoint, it was felt that it was desirable to avoid it if possible. It was thought that dead yeast cells might have been responsible for the bulk of the nitrogenous products present. The solids in the dunder were therefore removed in four ways —

- 1. By straight filtration through paper.
- 2 By first mixing with coral sand and then filtering through paper.
- 3 By first mixing with finely ground coral sand and then filtering through paper.

4 By centrifuging. The filtrate from (1) was found to contain 0.11% nitrogen. Hence it was concluded that the nitrogenous products were in colloidal suspension or in true solution. The sand did not speed up the rate of filtration to any marked extent.

The said treatments greatly reduced the acidity, the finely ground product being more effective than the coarse variety (see Table 5). Moreover the colour of the dunder was appreciably darkened.

TABLE 5.

Treatment	Acidity/100 ml.	Vol. NoOH/N/14
1. Straight filtrat	ion 1.56	36.5
2. Coarse Sand	0.66	15.5
3. Fine Sand	0.34	8.0

It was found that the filtrates which had been passed over sand had a decidedly increased pH value due to the acid removal. Normal pH was 4.0 while after treatment with fine sand it rose to 5.9. These filtrates from the sand treated dunder moreover immediately started to grow microflora in profusion on their surfaces.

The limiting factor of time as already mentioned has not enabled us to go into the chemical side of this problem very deeply. Some empirical method of simply assessing the degree of concentration will have to be worked out on as many samples of dunder as possible. If the quicklime treatment is adopted field experimentation will have to be instituted to ascertain whether there is going to be antagonism between the lime used and the potash present in the product so far as the uptake of potash by cane plants is concerned. Furthermore the addition of quicklime on a commercial scale may be hazardous and special precautions may be necessary, which appear not difficult to devise.

The method for preparing a dry free flowing powder rich in potash should be investigated on a pilot plant scale, and costs of production determined. Fertilizer trials on potash deficient soils should be conducted with the material.

#### REFERENCES.

- Methods of Dunder Disposal A Bibliography— J. G. Davies, J.A.S.T. Quarterly, VIII, 3 p. 17.
- Investigations into Dunder Disposal in Jamaica, J. G. Davies, J.A.S.T. Quarterly, VIII, 3, p.22.
- Dunder Disposal, J. Munro, J.A.S.T. Quarterly, VII, 3, p.4. VIII, 1, p.5.



- 4 Disposal of Rum Distillery Waste Waters, A.C. Thaysen, Proc. B.W.I. Sugar Technologists, 1948, p.21.
- 5. Departmental Reports, S.M.A.J. Research Dept. (not published).
- 6. J.A.S.T. Quarterly VII. 1, p.8.
- 7. Observations on the Evaporation of Alcohol Distillery Slop, M.B. Floro, J.A.S.T. Quarterly IX. 3, p.52.
- 8. Dunder Disposal, G.W.P. Stretton, J.A.S.T. Quarterly X. 3, p.45.
- 9. Some Preliminary Observations on The Agricultural Utilization of Dunder. R. F. Innes, Proc. B.W.I. Sugar Technologists, 1941.
- Some observations on the possibility of replacing imported fertilizers by local products — J. R. McFarlane, J.A.S.T. Quarterly IV. 2, p.2, and particularly pp. 10-13.

#### DISCUSSION

Mr. Floro said that the important question was to find a practical method of concentrating dunder. As far as he knew this could not be done economically. He proceeded to say that if it were concentrated to 75% solids, there would still be 25% moisture. Calcium oxide would absorb only 18% by weight of moisture, so that the proportion of lime necessary would be very great and in order to make it a paying proposition, lime would have to be bought very cheaply.

Mr. Barnes said that he had tried to make it clear in his closing sentence that there was no question of the solidifying of dunder being an economic proposition. What mattered was that it offered some method of using the material.

Mr. Floro stated that he hoped Mr. Barnes had not misunderstood him, but if they were expected to derive any benefit from the process, some definite idea should be offered as to how the solution should be achieved and at what cost.

Mr. MacFarlane said that it was usual to consider the use of quadruple effect in the concentration of dunder. He had seen where a single effect compression distillation plant designed by Klein-Schmidt appeared to be twice as effective as the quadruple effect for the production of distilled water and cheaper to install. Mr. Innes said that he had the opportunity of visiting a large distillery in North America in which this problem arose. Their method consisted of separating the yeast through centrifugals, drying it, and selling the product as cattle feed; then they concentrated the dunder to 25% solids and spray dried it. They estimated that at \$70 per ton it was still an uneconomic proposition. The capital involved for spray drying was between \$500,000 and \$1,000,-000. He also added that the capacity of the plant was 6,000 gallons per hour.

Mr. Douglas Aitken wanted to know whether trickling filters had been tried. He had been in contact with a firm who was designing and installing equipment of this type for treating distillery wastes from whiskey distilleries for 60 years. They had designed a pilot plant for treating dunder and it did not appear that the cost of such a plant would be prohibitive.

Mr. Barnes said that trickling filters had been tried by Mr. J. G. Davies. He stated that the waste liquor from sugar cane was far more difficult to handle than the effluent from a grain distillery.

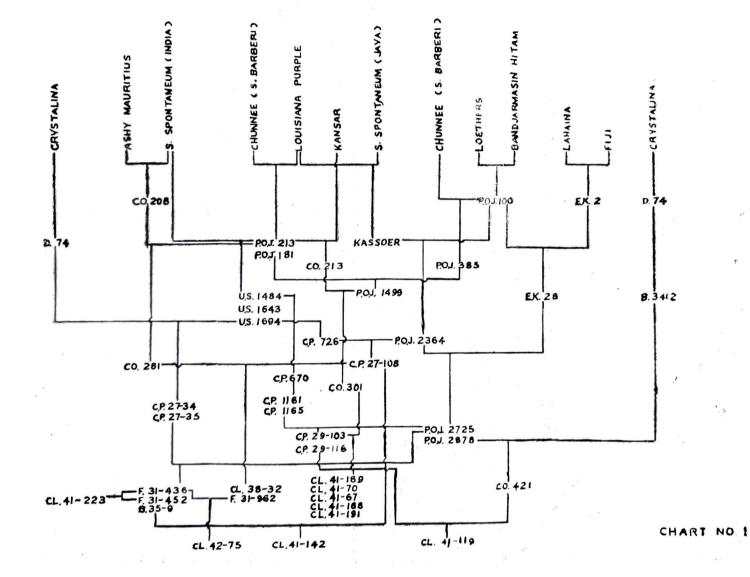
Mr. J. G. Davies stated that as far as he remembered trickling filters were considered and rejected.

**Mr. Innes** said that Dr. Southgate had suggested that aerobic fermentation might be an answer to the problem. Dr. Thaysen had tried this method, but he had heard that it had been a failure.

Mr. J. G. Davies re-iterated that Jamaica rum effluent was a totally different product to effluent from a grain distillery.

The Chairman said that he was sorry to have to bring this discussion to a close after such a short time. He said that from his experience in Trelawny, and he was sure from the experience of many others present, there used to be a dunder pond on every estate. He regretted that Mr. Clarke did not have available the figures he had prepared on yield per acre so that the members could obtain an idea of the benefits to be derived. He remembered that as far back as 1940, Mr. Barnes and his colleagues suggested the concentrating of dunder, but the idea had been abandoned. At Frome they utilized 11 million gallons of dunder in the fields last year. From his observations he was sure that the benefit was very definite on the Shrewsbury section of his estate. In conclusion he wished to congratulate Mr. Clarke and his colleagues on their practical approach to the problem, and to thank them for the able manner in which they had explained their methods.

GENEOLOGICAL DIAGRAM SHOWING LINES OF DESCENT OF IMPORTANT DISEASE RESISTANT SUGAR CANES CULTIVATED COMMERCIALLY BY UNITED STATES SUGAR CORPORATION



THE J.A.S.T. JOURNAL.

1