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TABLE OF CONTENTS

Page

INTR	ODUCTION	3
Ι	THE RAW MATERIAL Composition, ratios of components, defects, yeast nutrients in molasses, yields & cane juice vs molasses	4
Π	YEAST SELECTION	13
III	MASHING OPERATIONS Pretreatment of the molasses, partial sterilization, pH, sugar concentration, and quality of the dilution water	39
IV	MITOGENETIC RADIATION Definition, previous work cultures used, observations that indicated the emission of radiation, experiments devised to confirm the mitogenetic effect, and conclusions	50
V	RUM FERMENTATION Alcohol vs. rum manufacture, seed preparation, fermentation time, pure culture technique, yeast nutrients, fermentation temperature, sugar concentration, pH of mash, after-fermentation treatment, heavy rum fermen- tation, Oidium as an auxiliary rum ferment, reduction of the fusel oil fraction and multiple yeast cultures	61
VI	RUM DISTILLATION Constituents of the fermented mash, redistillation method, batch distillation method, continuous distillation method and testing of the distillates	119
VII	RUM CURING AND MATURING	138
VIII	RUM AROMA Influence of raw materials, of the type of yeast and the type of still	171
IX	RUM COMPOSITION AND APPRAISAL Importance of the non-alcohol-number, use of the birectifier, chemical analysis, physical and organoleptic tests, fractional distillation, valuable ratios and appraisal data on six commercial rums	182
Х	BIOLOGICAL AND CHEMICAL CONTROL OF THE RUM DISTILLERY The yeast, molasses, fermenter record, pH control, disinfectants chemical tests, distillery records and calculations	230
XI	SUMMARY IN ENGLISH	258
XII	SUMMARY IN SPANISH	261
XIII	ACKNOWLEDGEMENTS	270
XIV	LITERATURE CITED	271

LIST OF TABLE

Table	e No.	Page
1	Composition of Molasses	6
2	Characteristic Ratios of Molasses	7
3	Results of Fermentation Tests	9
4	Fusel Oil and Non-Alc. No. Produced by Several Yeasts	19
5	Fractional Distillation Results Sample No. 1	24
-	Fractional Distillation Results Sample No. 2	26
	Fractional Distillation Results Sample No. 3	28
	Fractional Distillation Results Sample No. 4	30
	Fractional Distillation Results Sample No. 5	32
	Fractional Distillation Results Sample No. 6	34
6	Fractional Distillation Supplementary Tests	36
7	Comparison of Treated and Untreated Molasses	49
8	Effect of Bacterial Concentration on Yeast Cells	54
9	Yeast Cultures Under Mitogenetic Radiation	55
10	Yeast Cultures Under Mitogenetic Radiation	57
11	Radiation of Bacteria to Yeasts Through Quartz	61
12	Effect of Yeas][t Cell Concentration on Fermentation Results	67
13	Effect of Ammonium Hydroxide and Ammonium Sulphate on Yeast Propagation	69
14	Effect of Temperature on Yeast Propagation	75
15	Effect of Sugar Concentration on Fermentation Results	79
16	Comparative Chemical Analysis of a Genuine Heavy Rum vs a Spurious Rum	92
17	Fermentation Results Using Pure Yeast Cultures vs Cultures in Symbiosis	98
18	Fermentation Results Using Cane Juice and Mixed Culture	106
19	Results of Light Rum Fermentation	
20	Fermentation Data with Variation in Yeast Nutrients	115
21	Results of Tests of Raw Rum Distillates	127
22	Results of Tests of One-Year-Old Rum	128
23	Results of Tests of Fractional Distillation	130
24	Hydrolytic Effect of Diluent on Ester Value	
25	Data on Composition of Molasses Rums During the First Year of Aging	
26	Similar to No. 25 but with Changes in Dilution	162
27	Data on Composition of Sugar Cane Juice Rums While Aging	166
28	Analysis of One, Two and Three Years Old Molasses Rums	168
29	Analysis of One, Two and Three Years Old Sugar Cane Juice Rums	168
30	Average Analysis of Each Class of Rum at Different Aging Periods	169
31	Appraisal Data of a 2 Year Old Jamaica Type of Rum	193
32	Appraisal Data of an 8 Years Old Jamaica Type of Rum	195
33	Appraisal Data of a 2 Years Old Medium Heavy Type of Rum	200
34	Appraisal Data of a 2 Years Old Light Type of Rum	202
35	Appraisal Data of a 2 Years Sugar Cane Juice Light Type of Rum	205

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LIST OF ILLUSTRATION

Fig. N	No.	Page
1	Fractional Distillation Results Sample No. 1	25
2	Fractional Distillation Results Sample No. 2	27
3	Fractional Distillation Results Sample No. 3	29
4	Fractional Distillation Results Sample No. 4	31
5	Fractional Distillation Results Sample No. 5	33
6	Fractional Distillation Results Sample No. 6	35
7	Pure Yeast Fermentation and Mixed Culture Fermentation	52
8	Yeast Cultures Under Mitogenetic Radiation	56
9	Yeast Cultures Under Mitogenetic Radiation	58
10	Alcoholic Fermentation with Auxiliary Bacterial Ferment	100
11	Alcoholic Fermentation with Auxiliary Bacterial Ferment	101
12	Alcoholic Fermentation with Auxiliary Bacterial Ferment	102
13	Alcoholic Fermentation with Auxiliary Bacterial Ferment	103
14	Alcoholic Fermentation with Auxiliary Bacterial Ferment	104
15	Fractional Results of a 2 Years Old Heavy Type of Rum	194
16	Fractional Results of an 8 Years Old Heavy Type of Rum	196
17	Fractional Results of a 2 Years Old Medium Type of Rum	201
18	Fractional Results of a 2 Years Old Molasses Light Type of Rum	203
19	Fractional Results of a 2 Years Old Sugar Cane Juice Light Rum	206
20	Fractional Results of a 2 Years Old Sugar Cane Juice Rum of a Medium Heavy Type	208
21	Fractional Results of Commercial Light Type Rum No. 1	212
22	Fractional Results of Commercial Light Type Rum No. 2	214
23	Fractional Results of Commercial Light Type Rum No. 3	216
24	Fractional Results of Commercial Light Type Rum No. 4	218
25	Fractional Results of Commercial Light Type Rum No. 5	220
26	Fractional Results of Commercial Light Type Rum No. 6	222
27	Effect of Bacterial Contamination on Alc. Production	235
28	Effect of Bacterial Contamination on Acidity	237
29	Sugar Conversion into Alcohol During Normal Fermentation	238
30	Variation in pH and Titratable Acidity During Normal Fermentation	239
31	Sugar Conversion into Alcohol at Constant pH	240
32	pH and Acidity of Fermentation at Constant pH	241
33	Fractionation Results of Molasses Rum Before Storage	248
34	Fractionation Results of Molasses Rum (or Fig. 33 after 6 Months of age)	249
35	Same as Fig. 33, but at 1 Year of Age	250
36	Fractionation Results of a Sugar Cane Juice Rum Before Storage	251
37	Same as Fig. 36 but six months old	252
38	Same as Fig. 36 but One year old	253

LIST OF TABLE

Table	e No.	Page
36	Appraisal Data of a 2 Year Old Sugar Cane Juice Rum Classified as Medium Heavy Type.	207
37	Study of Commercial Rum No. 1	211
38	Study of Commercial Rum No. 2	213
39	Study of Commercial Rum No. 3	215
40	Study of Commercial Rum No. 4	217
41	Study of Commercial Rum No. 5	219
42	Study of Commercial Rum No. 6	221
43	One Year Old Rums	228
44	Two Years Old Rums	229
45	Three Years Old Rums	229
46	Commercial Rums, Age Not Known	229
47	Disinfectants and Their Use	243

STUDIES ON RUM

by

Rafael Arroyo

STUDIES ON RUM

INTRODUCTION

This project was started on January 1936, and was closed on October 1942; so that it had a life of nearly six years. From 1936 to 1937 the work was carried on by Mr. Miguel A. Manzano, as Assistant Chemist, and the writer, as Fermentologist and Chemist. In 1937 Mr. Fernando Marrero was incorporated to the project as Assistant Fermentologist, and in 1938, upon the resignation of Mr. Manzano, the position of Assistant Chemist was occupied by Mr. Leonardo Igaravidez.

We soon discovered that although very old, the rum industry has been almost devoid of assistance from scientific research as shown by the scarce, and in its great part, inaccessible literature on the subject.

It was also found that erroneous ideas and conceptions were prevalent at the time when these investigations were initiated about the elaboration of this beverage, especially regarding the fermentation process, where empirical practices predominated. The nature and necessary characteristics of the fermentation agents employed were utterly unknown or disregarded, and absurd and even perilous curing practices of the raw rums were being followed in many cases due to the ignorance of better and more appropriate methods.

It has been our aim all during this work, to correct in the measure of our ability and power these erroneous methods and ideas, delivering in this way the vigorous young industry from the costly and inefficient empiricism, and leading it into new channels within the realm of modern experimental technique and scientific effort.

To cope successfully with the rapid and vigorous development of the new industry it became necessary to offer to the public and especially to those in the industry, partial publications of the results obtained without waiting for the time when the completed study could be published. This we have lavishly done, as over twenty-two articles dealing with the specific subjects of rum manufacture have been published during the time that these investigations were taking place. Some of these articles appeared in magazines, others in the form of a circular¹ published by this Experiment Station in 1938 in the Spanish language, and of which two editions were made amounting to a total distribution of over four thousand copies, at home and abroad. An exclusive publication devoted entirely to the interests of the

Puerto Rican liquor industry "Spirits Service Institute Bulletin", has also since its foundation cooperated with the writer in the regular publication of his articles on rum subjects – honour for which we are greatly indebted to this important publication.

Our research covered as far as it was possible all of the technical aspects of rum manufacture, more emphasis being given, however, to the fermentation process and related matters such as the very important question of yeast selection, pretreatment of raw materials, mashing operations, and after fermentation treatment of the beers before their actual distillation. For a clearer exposition of methods followed and results obtained, the work has been divided in a series of topics, believing that in this way a better understanding of what was accomplished, in each phase of the research, will be realized.

Ι

The Raw Material

Up to the time when our studies were initiated two types of raw materials were being used from rum manufacture in Puerto Rico: final molasses from the sugar factories and sugar cane juice; the latter formed, however, but a very small fraction of the total output, and besides, the rums thus produced were mainly used for blending purposes in mixtures with molasses rum, rather than put into the market as such. As time passed, the decline of sugar cane juice as raw material was accentuated, till at the time of writing this report it may be properly stated that final, or blackstrap molasses, is the raw material for rum making.

We soon observed that final molasses was being sold and bought without due regard to its chemical composition, excepting in some cases, its Brix density and total sugars content. We decided to start our study with a survey of the chemical composition of final molasses produced by the various sugar factories of the Island in order to investigate the possible influence of the chemical composition of the raw material, other than amount of total sugars, upon the process of rum making and quantity and quality of the end products produced. Through the whole-hearted cooperation and interest taken by the sugar producers of the Island, several samples were obtained from different sugar factories representative of the various sugar cane producing regions of Puerto Rico. These samples were analyzed according to our plans and submitted to fermentation tests. By these means we purposed to find out the influence of the molasses composition on the process of manufacture and on the yields and qualities of the resulting rums.

In table 1 will be found representative analyses of our final molasses from which may be realized the structural differences among the various samples.

These combined chemical analyses and fermentation tests brought into the light important facts that were being overlooked by the rum industry in the use of the molasses. We found out that the chemical composition of the molasses had a direct influence on practically the whole manufacturing process; affecting such factors as yields, distillery capacity, economy of production, fermentation efficiencies, and quality of the products. Moreover, certain ratios were found among the individual constituents of the molasses that were even more important than the magnitude of their individual values.

The chemical analyses of the molasses samples consisted in determinations of: (1) Brix density; (2) true sucrose; (3) reducing sugars; (4) total sugars as invert; (5) ash content; (6) gums; (7) total nitrogen; (8) phosphoric acid as P_2O_5 ; and (9) pH value. A steam distillation test was also performed on each sample of molasses to determine the natural aroma inherent to the material. During the ash determination at the time of acidulating with sulphuric acid for converting to sulphated ash, care was taken to notice the odour produced by the reaction. By these means we could ascertain the immoderate presence of sulphur compounds (as for instance, sulphites) in the material.

From the above tests and the results obtained from the fermentation and distillation trials, we were able in the course of time and with the experience acquired, to catalogue the different molasses as good, fair or poor examples, as to their fitness for rum fermentation. After sometime, there was no necessity of proceeding with the confirmatory fermentation and distillation tests in order to classify a given sample of molasses. Certain ratios were also developed among the different constituents of the raw material that were representative of the quality of the molasses for fermentation purposes. The determination of natural aroma by the steam distillation test was important, as this aroma would naturally exert an influence on the aroma of the distilled spirit. It was also found that the presence of sulphur compounds in appreciable quantities in the molasses was invariably connected with foulness of odour in the raw distillate.

TABLE No. 1

COMPOSITION OF MOLASSES

Sample No.	Brix Density	True Sucrose	Reducing Sugars	Total Sugars, as Invert	Ash	Gums	Total Nitrogen	Total Phosphoric Acid as P2O5	Total pH Sugars Value Ash Ratio	SugarsSugars/AshSucrose	Sugars/ Sucrose	P2O5/T/ Nitrogen Ratio	Gums/T/ Sugars Ratio		Molasses A Distillation	
														Good	Fair	Poor
1	86.40	31.96	21.92	55.56	7.86	3.41	0.94	0.15	5.54	7.07	0.69	0.16	0.06		-	
2	83.20	30.49	17.92	49.93	7.61	3.21	0.66	0.11	5.58	6.56	0.58	0.17	0.06		-	
3	90.80	32.30	23.47	57.47	9.68	2.87	0.48	0.09	6.02	5.93	0.73	0.19	0.05	-		
4	86.60	31.02	24.33	56.98	7.31	2.67	0.62	0.10	5.87	7.79	0.79	0.17	0.05	-		
5	86.80	36.44	19.61	57.97	7.34	2.19	0.72	0.13	6.00	7.89	0.54	0.18	0.04	-		
6	89.60	32.93	23.31	57.97	10.57	3.71	0.45	0.08	6.25	4.48	0.71	0.18	0.06			-
7	92.00	31.53	22.83	56.02	9.86	3.21	0.54	0.11	6.15	5.68	0.72	0.20	0.06		-	
8	85.40	32.85	21.92	56.49	7.34	2.67	0.44	0.09	6.15	7.70	0.67	0.20	0.05	-		
9	90.76	27.61	19.53	48.59	12.90	4.05	0.55	0.14	5.10	3.77	0.71	0.25	0.08			-
10	86.70	34.10	10.70	46.40	10.04	2.90	0.60	0.12	5.50	4.62	0.31	0.10	0.06			-
11	87.70	33.25	17.50	52.50	8.75	2.55	0.72	0.17	5.30	6.00	0.53	0.24	0.05	-		
12	86.00	35.58	19.80	57.25	6.54	2.05	1.15	0.21	5.50	8.62	0.56	0.18	0.04	-		

Contamination of the molasses in transit, or during storage, with undesirable substances, as for instance, fuel oil, will be revealed by the steam distillation test, even when the extent of the contamination be of very slight nature.

TABLE No. 2

SHOWING THE COMPOSITION AND CHARACTERISTICS RATIOS BETWEEN COMPONENTS OF A GOOD, FAIR, AND POOR SAMPLE OF BLACKSTRAP MOLASSES FROM THE STANDPOINT OF RUM FERMENTATION

All percentages are by weight	[1] Good	[2] Fair	[3] Poor
Brix Density	87.60	85.40	88.20
Total Sugars as Invert, Per Cent	57.97	52.91	49.93
True Sucrose, Per Cent	36.44	31.30	34.61
Reducing Sugars, Per Cent	19.61	19.96	13.50
Ash, Per Cent	7.31	9.35	11.57
Nitrogen, Total, Per Cent	1.10	0.60	0.45
Total Phosphoric Acid, as P ₂ O ₅ Per Cent	0.19	0.09	0.21
Gums, Per Cent	2.00	2.55	3.75
pH value	5.50	5.70	6.30
Total Sugars - Ash ratio	7.93	5.65	4.31
Reducing Sugars - Sucrose ratio	0.54	0.64	0.39
P ₂ O ₅ - Total Nitrogen Ratio	0.17	0.15	0.47
Gums - Total Sugars ratio	0.03	0.05	0.08
Natural Aroma by Steam Distillation	Good	Fair	Indifferent

The defects most commonly found during these analyses in some of the samples tested were: (1) low total sugar; (2) high ash; (30) high gums; (4) high total non-sugars; (5) deficiencies in nitrogen and phosphorus contents; and (6) inadequate pH values. In table 2 there are classified three different final molasses that were appraised as good, fair, and poor, respectively, for purposes of rum fermentation. In table 3 will be found comparative fermentation tests conducted with each of the molasses samples given in table 2. Care was taken to use the same amount of total sugars concentrations, the same initial pH and the same variety of yeast for each comparative test. The fermentations were also conducted under the same controlled conditions, so that any differences found in the results could only be attributable to the differences in the chemical structures among the molasses samples other than those of total sugars contents.

A study of the data shown on the three tables will lead to conclusions of great interest to the rum distiller: In the first place will be noticed the structural differences among blackstrap molasses in their chemical aspects. In the 12 typical samples under consideration will be found total sugars values varying from 46.40 to 57.97 per cent; Brix densities from 83.20 to 92.0; ash values from 6.64 to 12.90; gums 2.05 to 4.05; total nitrogen from 0.44 to 1.15 per cent; total phosphoric acid as P_2O_5 from 0.08 to 0.21; and pH values varying from 5.1 to 6.25.

It is obvious that molasses of high sugars content will be better qualified for rum fermentation than those of low sugars content, mostly as to yields. Similarly a high density molasses weighs more per gallon than a low density, and the material being bought and sold on gallon basis, consumers will prefer the higher density molasses.

But what about the non-sugars in the molasses? We found that they too should be taken into consideration by the distiller since they will bear direct influence in the processing, yield, and quality of the finished product. A certain amount of ash yielding minerals is no doubt necessary for yeast growth and development but as the ash content of the molasses rises, it begins to influence adversely the fermenting qualities of the material. The bad effect of abnormally high ash must be considered in its relation to the total sugars in a given molasses is called the Total Sugars-Ash Ratio.

We found that ratios of 6.5:1 or above maybe be considered good; ratios less than 6.5:1, but not below 4.5:1 may be considered fair; and those below 4.5:1 were poor. During fermentation the initial ratio of total sugars to ash narrows considerably and continuously, until towards the finishing stages this ratio may become but a small fraction of its initial value. As this happens, the yeast finds increasing difficulty in obtaining the sugars necessary for its metabolism, while at the same time the increasing alcoholic and saline concentrations in the substrate act in an inhibitory way towards the yeast. This is one, and perhaps the main reason why fermentation proceeds so much slower after the first half of the total period had elapsed. Therefore, high ash ratios will contribute to slow and laborious fermentations, and to the leaving of unusually high amounts of unfermented residual sugars in the beers. Another way in which high ash ratios may contribute to low yields, is through the opportunities for infection offered by a prolonged period of fermentation.

High ash content in the mash will also offer trouble of a different nature, but of importance always to the economy and efficiency of the distillery; for instance, a portion of the original ash of the fermenter where it will mix up with yeast cells accumulated there. This will naturally make more laborious and expensive the recovery of yeast bottoms for their use as feed or fertilizer, due to the labour involved in purifying the yeast residues also foul the plates of the still column in a short time, the consequence

TABLE NO. 3

COMPARATIVE RESULTS OF FERMENTATION TESTS USING THE THREE TYPES OF MOLASSES DESCRIBED ON TABLE 2

Molasses Type No.	Test No.	Initial Brix	Total Sugars grs. per 100 ml	Initial pH value	Fermenta- tion Time, Hours	Final Brix	Final pH Value	Residual Sugars, grs. per 100 ml	Attenu- ation	Alcohol by Volume, Per Cent	Grams alcohol per 100 ml Beer	Alcoholic Yield, on T. Sugars, Per Cent	Fermen- tation Efficiency Per Cent
1 - [Good]	1	15.00	10.87	5.20	25.50	4.30	4.85	0.56	10.70	6.35	5.01	46.09	94.93
	2	18.00	12.76	5.20	30.00	5.50	4.90	0.60	12.50	7.50	5.92	46.45	95.67
	3	20.00	14.78	5.20	30.00	6.20	4.97	0.67	13.80	8.80	6.95	47.00	96.80
	1	17.00	10.87	5.20	30.00	6.80	4.85	1.02	10.20	6.05	4.78	43.97	90.56
2 - [Fair]	2	19.80	12.76	5.20	36.00	7.90	4.80	1.18	11.90	7.20	5.68	44.51	91.67
	3	22.50	14.78	5.20	40.50	9.20	4.90	1.37	13.30	8.15	6.43	43.50	89.59
	1	18.00	10.87	5.20	36.00	8.90	4.80	1.67	9.10	5.60	4.42	40.68	83.78
3 - [Poor]	2	21.00	12.76	5.20	42.50	9.85	4.87	2.33	11.15	6.30	4.97	38.99	80.30
	3	24.50	14.78	5.20	48.00	11.80	4.85	3.00	12.70	7.00	5.53	37.39	77.01

being frequent shutdowns for cleaning purposes with corresponding lost time and extra expenses in labour, fuel and chemicals.

Organic non-sugars, especially gums, also operate adversely towards rum making, much in the same manner as ash does; but in this case the bad effect influences also the quality of the rum produced, the product being high in fusel oil unless highly rectified during distillation. When some of these gums precipitate during fermentation under the influence of the alcohol produced, they form a sort of binding material between yeast cells and precipitated ash thus increasing the difficulty in the purification of the recovered yeast. The gums will also help in facilitating the adherence of ash to the plates of the distilling column and to the surfaces of the feed pipe.

The yeast needs the nutriments of any other plant, most important among which are nitrogen, phosphoric acid and potash. Molasses contains these ingredients in variable quantities, only potash being always present in excess of the requirements. Nitrogen deficiency is quite common, and sometimes phosphoric acid is also wanting. When not naturally present in the molasses in the required amounts, both nitrogen and phosphoric acid must be incorporated to the mash, which means added expense of manufacture. For these reasons it becomes important to determine through chemical analysis, the amounts of these two substances present in the raw material. We found that whenever the total nitrogen in molasses was one per cent or over, there was no necessity of incorporating addition nitrogen. In cases when the nitrogen content ran less than one per cent by weight of molasses, then it was necessary to add from 0.2 to 0.5 per cent of ammonium sulphate on the weight of molasses during mashing.

As to the amount of phosphoric acid, we found an optimum value corresponding to from 0.2 to 0.25 per cent in terms of P_2O_5 on the weight of molasses. It was found that phosphoric acid deficiency was by far less common than nitrogen deficiency in Puerto Rico or two exceptions. Excess of phosphoric acid coupled to nitrogen deficiency was found particularly objectionable during the fermentation time taken for fermentations, as to render the operation a complete failure. Addition of extra nitrogenous nutriments in such cases in those quantities that would establish a ratio of about 1:5 between phosphoric acid and nitrogen, would immediately reinstate normal fermentation time and yields.

Referring to the table 2 we find three different analyses of final molasses representing a good, a fair, and a poor example of this raw material. It will be noticed that in sample (1) we find not only high sugars, low ash, high nitrogen and proper amount P_2O_5 , as well as low gums; but that also the different ratios representative of high molasses quality are also found. Judging entirely from these analytical results, it may be presumed that this particular molasses should prove to be one of exception merit for rum making. And if we look over in table 3 the results of the fermentation tests performed with this molasses as raw material, our presumption will be found fully realized: rapidity of fermentation is found in all three cases, coupled to high yields and fermentation efficiencies, and very low residual sugars.

In table 2, the analytical results of molasses sample 2 show relatively high ash, and rather low nitrogen and phosphoric acid contents; but having a normal percentage of total sugars, the ratios of total sugars to ash and gums are within the range for the obtention of quite fair fermentation results. Such we will find to be the case upon inspection of the fermentation results shown in table 3. The defects of the raw material are responsible, however, for the lower yields, the longer fermentation periods, and the higher residual sugars left over in all corresponding cases.

Turning to sample 3 of table 2 it may be noticed at once its low sugars, high ash, very low nitrogen and unusually high gums. The P_2O_5 content is normal for a high nitrogen molasses, but becomes deleterious (as already explained) in this case due to the low nitrogen content of the molasses. All of these analytical results point out to a laborious fermentation when this molasses is used in actual fermentation work; and such we find to be the case when referring to the corresponding results of table 3. It will be noticed that for the corresponding initial sugar values the Brix densities are much higher than in the other two fermentation series, demonstrating an unusual amount of non-sugars in the raw material. Also the fermentation periods are much longer, the yields of alcohol very appreciably lower, residual sugars in beers very high, and fermentation efficiencies are very low. This becomes more apparent as the total sugars concentrations increase, with corresponding high initial Brix densities.

From the above discussion it becomes clear that no time will be wasted by the rum distiller in effecting a thorough chemical analysis of the raw material he intends using in his daily work.

The advantages thus gained will reflect on the whole course of rum manufacture as well as on the quantity and quality of the final product.

When sugar cane juice is substituted for final molasses as the raw material, there is no doubt that improvements in the chemical structure of the new raw material will be found. But other unfavourable factors enter into consideration which should be carefully studied by the rum producer. Per unit weight of total sugars in the raw materials, final molasses results much cheaper than sugar cane juice. Establishing a comparison between a ton of cane and one of final molasses, we have that the ton of cane will produce about 250 pounds of total sugars as an average; while the ton of final molasses will produce 1,250 pounds of total sugars. In the first place the pound of sugar will cost two cents, and in the second 0.8 cents. Hence, we find that for the same investment 2.5 times more sugars are obtained when buying it in the form of molasses; so that in the matter of first cost final molasses is much cheaper than sugar cane juice.

With reference to yield of rum we found that a ton of cane would produce from 33 to 38 proof gallons of rum, while 100 gallons of final molasses, costing the same as the ton of cane, would yield from 80 to 85 proof gallons.

Other disadvantages of using sugar cane juice as raw material are: (1) additional cost expenses due to the fact that a milling unit and accessories must be bought and operated for juice extraction; (2) lost time due to lack of cane, mechanical troubles, etc.; (3) the scarcity and unsuitability of the raw material after the sugar season is over; (4) greater microbiological contamination of the raw material adds to the labour and difficulties of the processing; (5) lack of storage properties of the raw material; and (6) variations in the quality and characteristics of the rum produced are more frequent than when using molasses as raw material.

If the rums produced from cane juice were of better quality than those obtained from molasses, perhaps it would not be unreasonable to overlook some of the above-mentioned drawbacks; but the fact is that the rums produced from molasses are in no manner inferior in quality to the best produced from sugar cane juice. If any, they are superior in certain respects; for instance, sugar cane juice rum will take a longer aging period to reach maturity, and even then, its taste and aroma will resemble too much that of brandy, to compete with the full rum taste and aroma of the molasses product.

Π

Yeast Selection

The selection of a suitable yeast for rum manufacture should be done prior to the design and erection of the various departments of the distillery, the then it will be easy to design and build in accordance with the natural and inherent characteristics of the selected organism. That these characteristics will in some instances affect the distillery design will be shown presently.

More or less fermentation capacity will be needed either in size or number of fermenters according to whether the yeast in question is a rapid or slow fermenting organism. We found yeasts capable of finishing fermentation in one half the time taken by another variety under equal conditions. The ability of the yeast to stand efficiently rather high temperatures of fermentation will in some cases decide whether or not a cooling system for the fermenting room is to be installed. The fermentation capacity as well as the size of still and boiler for a given production in proof gallons per day will be materially influenced by the ability of the selected yeast strain to stand high alcoholic concentrations in the fermenting mash. For instance, 4 fermenters holding beers with 12.0 per cent alcohol by volume will be equivalent to 8 fermenters of equal capacity filled 6.0 per cent alcohol beer. Naturally, less steam and a smaller column will be needed in the case of the higher alcohol concentration beer to produce the same number of proof gallons. We can add, that even on the design and equipment of an apparently disconnected department, as that of the warehouse used for aging the raw rum, the yeast will exert its influence, for we have found strains capable of producing fast maturing and others producing slow maturing rums. It is easy to see that a fast maturing product will need less barrels and less aging space than a slow maturing one. Then, in a way, the number of aging barrels and the amount of aging space required may be a function of the characteristics of the selected yeast.

According to the above explanation, it seems that the wiser attitude is to study and select the yeast strain to be used and then design the distillery and accessories. Proper yeast selection is perhaps the most important single factor entering in rum manufacture. But we must dwell rather on other aspects of yeast selection than those pertaining to more or less economy of design and erection of buildings, and to the cost of the machinery and equipment for them. The selection of a suitable yeast for rum manufacture has been in the past, and is at present, intermingled with the question of how this selected yeast is to be used in actual rum distillery practice. To many, a selected yeast presupposes its use in pure culture fermentation only, and that otherwise there is no need of going into the trouble of selection. We wish to separate the two ideas from the start, so that no misunderstanding will occur.

It is true that there can be no yeast selection without previous isolation of the microorganism in pure culture; but once the pure culture is obtained, and its suitability for rum production ascertained, it does not follow that in actual industrial use of this yeast, pure culture fermentation is the only possible, or even more desirable way in which it should utilized. In fact, a selected pure yeast culture may be used in rum fermentation in a number of ways, if precautions are taken for keeping the yeast stock culture absolutely pure. For instance, the pure culture maybe be used to build up a pure footing for seeding purposes. This pure seed may then be employed in one of three ways, as inoculum of a: (1) sterilized mash in close, aseptic fermenters; (2) partially sterilized mash; (3) non-sterilized, fresh mash. The fermentation conditions will vary in each of the above cases. In the first case the pitched yeast strain will be the only fermentation agent present in the substrate; in the second case partial sterilization has probably eliminated all types of vegetative microbiological flora from the substrate, leave, however, the organisms with heat resistant spores. According to the length of the fermentation period, resulting pH and alcohol concentration, and other conditions, these spores may or may not pass into the vegetative stage and thus become a factor in the fermentation process. In the third case we are pitching the yeast pure culture into a medium containing probably wild yeasts, besides moulds and bacteria; and in this case we are depending on the vigour and power of the pitched strain to gain predominance of the medium. In all three cases, however, we are using a selected yeast strain.

We thus hope to have made clear that yeast selection in the sense used in this bulletin does not necessarily mean the use of pure culture fermentation in rum manufacture, although that is one of the ways in which the selected yeast may be used. We rather wish to convey the idea that the distiller must first find a *veritable rum yeast* and obtain a pure culture thereof as the initial source of the ferment to be used in his daily work. This is the first prerequisite for successful practice.

There has been a great deal of controversy in the past as well as in the present, on this question of yeast selection in rum manufacture. Extreme and opposite views have been taken by equally distinguished investigators as to the real role of the yeast in the fermentation process. Pairault (²) in a treatise on rum manufacture published in 1903, advocated the use of selected yeast in pure culture only, as the best method of rum fermentation. He stated that the success in rum manufacture depended on the selection of a pure yeast strain *adaptable* to rum fermentation. His use of the phrase "adaptable to rum fermentation" was very important, for others have had the idea that any yeast as long as it was secured in pure culture would answer the purpose as well as another. This unfortunate conception led in many cases to misleading information, since upon experimenting with pure cultures of yeasts unadaptable to rum fermentation, some investigators and practical distillers found out that the fermentations had not produced rum. They have then blamed the pure culture fermentation technique for the disappointments obtained in the results of their experiments, overlooking the real cause, that is, the fact that although in pure cultures, the yeast used by them were *not adapted* to rum fermentation.

On the other hand, Pairault, evidently made sure in his personal experiments that his yeast strains were genuinely and truly rum yeasts. He was so enthusiastic over his pure culture fermentation experiments that he proclaimed in a most emphatic way that bacterial presence in the substrate had no influence in rum taste and aroma, and devoted a whole chapter of his splendid book to explain that bacteria are not necessary in rum fermentation; but that they are really a source of loss, trouble and nuisance to the distiller.

A few years later, 1914, Kayser (³) corroborated the results of Pairault, coming to the conclusion that the use of selected yeast in rum making would bring enormous advantages to the industry, among others, a shortening of the fermentation period, better yields, and products of constant composition. In 1916 Kayser (4) again stated that there was no doubt in his mind that pure, selected yeast would be employed more and more in the rum industry, and that the composition of the rum would be changed at will by the distiller through this practice. He did not express himself in a clear manner on the role of the bacteria in rum fermentation; but since he recommended pure culture technique as the best course to be followed, his objection to bacterial contribution in rum fermentation was tacitly understood.

But an exactly opposite view has been taken more recently by Guillaume (⁵) in his modern treatise published in 1939. He comments on the work of Pairault and Kayser in an adverse manner, stating that the advices of the latter as to the use of pure yeast and sterilized substrate had been followed, and that instead of rum, ethyl alcohol was obtained. Guillaume insists in his excellent treatise that pure yeast culture fermentation will not yield rum, that without bacteria there is no rum.

Among the English investigators we found the same controversial situation on this important matter. Greig (⁶) working in Jamaica, concluded that there were some yeast strains capable of producing a veritable rum aroma and taste without the assistance of intervening bacteria; while Allan (⁷) came to the conclusion that in the question of rum aroma and taste the yeast deserved only a subordinate position, and he though more pertinent to look for flavour and aroma producing bacteria rather than for selected yeast. Allan's successor, Ashby (⁸⁻⁹) criticized his stand, stating that as the yeast must always be the central factor in rum fermentation, it appeared to him quite natural to devote first attention to them, and to observe in particular whether some are really able to engender aroma and flavour of value in rum making.

Our studies on this important subject have proved that the above mentioned investigators were partly right and partly wrong. We must disagree with Pairault that bacteria had nothing to do in the development of rum taste and aroma; but we must equally disagree with Guillaume when he states that without bacteria there is no rum. Our own work has proved that while bacteria are not indispensable for rum production in general, some races of bacteria add in quite perceptible, and sometimes (according to the type of rum) controlling way to the volume and persistence of the rum aroma and taste. Moreover, we found that we need not restrict ourselves to bacteria along in this respect, some moulds also may be used to great advantage.

We firmly believe with Kayser, that the advantages of yeast rums, like the Jamaican export rums and the "Grand Arôme" class of Martinique, the rum yeast is quite capable of carrying on the rum fermentation without the intervention of co-ferments whether bacterium or mould.

We firmly believe with Kayser, that the advantages of yeast selection are of such magnitude as to be capable of results undreamed of today. As the rum industry extends in Puerto Rico, and standardization of rum types begins to be demanded, the yeast selection work will acquire added importance and greater fields of action. We expect in the non-too distant future to see the Puerto Rico rum industry undertaking fundamental work in the breeding of rum yeasts, in the fashion that is being done in Denmark with other races of yeasts by Winge and Lausstsen (¹⁰⁻¹¹⁻¹²) at the Carlsberg Research Laboratory of Copenhagen.

Our work has fully demonstrated the soundness of Professor Kayser's predictions on yeast selection and its effect on rum making. Some of our best rums were produced by pure selected yeast under pure culture fermentation technique. We admit, however, that greater volume and straight in taste and aroma are obtainable through bacterial and other microbiological intervention; but even then, we believe in selecting the intervening ferments and use them under strict control rather than to depend on whatever bacterial flora may, by chance, occur in the raw material used as substrate. At any rate, we wish to assert again that although enhancing the flavour and aroma of certain types of rum, bacteria are not indispensable to successful rum making; in fact, for certain of the light, very delicately scented straight drinking rums that are so much in vogue at present, bacteria are detrimental when present during their fermentation.

We have also found that the Non-Alcohol-Number content of rum is not mainly a product of bacterial activity, as often stated in the literature. This Number, also called Coefficient of Impurities, represents just as well a product of the operating yeast. The yeast obtains nitrogen by splitting off ammonia from the amino acids always present in rum fermentation through the degradation of proteins by the proteolytic enzymes. When this splitting occurs, in every case either an alcohol or an acid is formed with one less carbon atom' for example: R.CH.NH₂, COOH + O yields R. CO. COOH + NH₃. The ketonic acid losses CO_2 forming an aldehyde with still one less carbon atom thus, R. CO. COOH - R. CHO + CO₂. The resulting aldehyde, R. CHO forms an alcohol or an acid by reduction or oxidation, respectively. Thus, R. CHO + O yields R. COOH; and R. CHO + 2 H yields R. CH₂ OH. The R. represents an organic group which differs for each amino acid. The higher alcohols and acids produced are discarded by the yeast, but play an important part in the formation of flavour and aroma, both by themselves as such as through their combination into esters. The esters may also be formed later on, during the distillation of the beer, by chemical reaction of alcohols and acids in their vapour phase.

The higher alcohols (known collectively as fusel oil) form usually a too large percentage of the total Non-Alcohol-Number of the rum; and the present tendency is to eliminate this excessive presence of fusel oil in the commercial rum product. Hence, in our work on yeast selection the more or less abundant production of fusel oil by a given strain was a very important factor to consider.

There exist two general methods of preventing a given rum from possessing an undesirably high fusel oil content; one of which consists in the partial or total elimination of this product during distillation; and the other is rather a preventive measure by which the immoderate formation of fusel oil is impeded during the fermentation stage. Of this latter method we shall treat at length when reaching the subject of fermentation proper; but in so far as the subject of yeast selection is related we must discuss part of it here.

In our experiments a number of rum yeasts were tested on this matter of fusel oil production, and incidentally on their ability to build up a Non-Alcohol-Number when working under pure culture fermentation technique.

A series of mashes made from sugar cane juice were inoculated with different yeast strains and fermented under equal conditions other than the differences of fermenting agents. Temperature of fermentation was maintained within the range 33-35° Centigrade, which is the prevailing fermentation temperature range of most of our distilleries. No additional yeast nutriments other than those naturally found in sugar cane juice were used. At the end of fermentation the different mashes were distilled into rums of the same oil, both being reported in milligrams per hundred millilitres of absolute alcohol.

A variation in the amounts of fusel oil produced by the different yeast strains was observed, as will be found by referring to table 4. The data on this table showed that the selection of a yeast which naturally produces a low content of fusel oil would be a great help in solving the problem of fusel oil in commercial rums. It also demonstrated the soundness of asserting that the yeast alone, without any bacterial help is quite capable of build up a Non-Alcohol-Number. As to ester formation we have already explained how these are formed during fermentation without the intervention of bacteria.

As early as 1907, Ashby (¹³) commenting on the nature of ester formation in rum fermentation stated that according to his results, ester formation did not take place by a mere chemical reaction in the mash, but was intimately related to the actively working yeast cell.

TABLE No. 4

Yeast Strain Number	Fusel Oil Mgs. per 100 ml Absolute Alcohol	Non-Alcohol Number Mgs. per 100 ml Absolute Alcohol	Per Cent Fusel Oil in Non-Alcohol Number
500	157 92 250 85 101 196 188 204 179 184 349	323 350 400 240 281 349 367 440 389 353 556	$\begin{array}{r} 48.60\\ 26.28\\ 62.50\\ 35.41\\ 35.94\\ 49.74\\ 51.22\\ 46.36\\ 46.01\\ 52.12\\ 62.76\end{array}$

SHOWING THE VARIETAL CHARACTERISTIC OF DIFFERENT YEASTS TOWARDS THE PRODUCT OF FUSEL OIL AND NON-ALCOHOL-NUMBER DURING RUM FERMENTATION

Haupt (¹⁴) in his recent work on German rum claims that the nature of the fermenting yeast plays the principal role in the development of rum aroma, even superior to the nature of the raw material used in the composition of the mash. While this is a far reaching assertion, we cite it here in order to demonstrate the paramount importance of selecting the right class of yeast for rum fermentation.

Rum yeasts may be isolated from the rind of the sugar cane itself, or from any of the products and intermediate products obtainable during the process of sugar manufacture. Hence, selection must be exercised even among rum yeasts themselves. Our work has shown that the ideal yeast strain for the manufacture of a given type of rum may be entirely inadequate for another type. In general terms we may define a veritable rum yeast as one fulfilling the following requirements:

1. Producing a fair yield of alcohol based upon the total sugars content of the substrate in a reasonable fermentation period.

2. Producing a raw distillate already possessing the characteristic rum taste, body, and aroma.

3. The raw rum produced should offer a well-balanced chemical composition.

4. The raw rum produced should be capable of acquiring the desired degree of maturity to convert it in a commercial product, in a comparatively short aging period; from one to two years at the most.

5. The yeast should be a good producer of rum oil.

6. In the case of yeasts intended for the production of heavy type rum, they should also be able to stand appreciable concentrations of fatty acids in the substrate.

Other important points to be considered which are more or less related to the economy of the process rather than to the quality of the end product are the ability of the yeast to stand quite high temperature of fermentation, to hold against infecting microorganisms that may gain access to the substrates, and to stand high alcoholic concentrations in the fermenting medium.

Our yeast selection system was therefore based on tests conducted for determining whether a given strain under trial would conform with the above specifications. The procedures followed in each particular case are self-evident and need no further explanation in the majority of the specifications; but we must dwell with some detail upon the method followed for the determination of specifications three and five above.

We soon found out that the chemical analysis of a rum as ordinarily performed was of itself practically useless for the purposes of quality appraisal. Moreover, this routinary analysis by itself gave no orientation on how the chemical composition of the sample influenced its quality as a drink. Rums of similar analysis happened often to be quite different in quality, when judged by the organoleptic tests.

We ameliorated this difficulty by supplementing the ordinary analysis with fractional distillation of the sample under consideration, using for this purpose the birectifier invented by Dr. Curt Luckow, Director of the Division for Distilled Spirits of the Berlin Institute of Fermentology. This birectifier is a small, laboratory distilling head, so constructed as to render it capable of very high rectification in double effect during fractionation. Its dephlegmatory action is also very efficient, and its manipulation very simple.

The fractionation of a rum sample is carried in the birectifier in the following manner: The sample is diluted with distilled water to an alcoholic content of 40.0 per cent by volume. Then 250 ml of this solution are placed in the distilling flask connected to the birectifier, and the liquid is brought to gentle boiling by careful heating with a gas flame of variable intensity. The distillation is so conducted that 25 ml portions are collected at intervals of fifteen

minutes between fractions, so that eight fractions of 25 ml each are collected in a two hours fractionating period. The temperature of distillation at the start and finish of each fraction, as well as the physical appearance of individual fractions are recorded. The different fractions are collected into graduated glass cups (that form an accessory of the birectifier) provided with glass covers. These have the shape of tasting cups used by experienced tasters for alcoholic beverages. During the distillation of the sample some important observations are made: (1) temperature range within which each fraction passed over; (2) characteristic aroma of each fraction; (3) presence of turbidity; (4) presence of oily droplets on the surface of the liquid of individual fractions.

Then the odour and appearance of the residual liquid left in the distilling flask are also observed, and the various fractions are diluted to 100 ml volume with distilled water, and characteristic odour and physical appearance renoted. After this, each fraction is submitted to chemical analysis for the determination of: (1) alcohol by volume; (2) volatile acidity; (3) esters; (4) aldehydes; (5) higher alcohols (fusel oil). The alcohol is calculated in per cent by volume; and the other constituent in terms of milligrams per 100 ml of actual distillate. From the analytical data thus obtained certain important radios are determined among the group constituents of the rum sample. These ratios bear a direct relation to the quality of the fractionated spirit. They also help in determining whether or not the rum under test will classify as a probable quick or slow maturing raw during the aging period. The fractional distillation reveals also the presence or absence of rum oil, and if present, whether in large, moderate, or meagre quantities.

Among the many constituents that enter into the composition of a genuine rum, there is none so important as the essential oil, or mixture of essential oils, to which the name of rum oil has been given. Micko (¹⁶) working with Jamaica rum found that all pure, genuine rums of this class contained this oil in variable quantities, and that the specific odour of Jamaica rum was due principally to this aromatic constituent. Our own work has proved that this, or similar essential oil forms the fundamental aroma of any genuine rum, and that some varieties of yeasts will produce it in variable quantities; but always in very small amounts. We shall treat on this important matter of rum oil more fully in another section of this bulletin.

The use of the birectifier in yeast selection work makes it possible to detect the presence of this most valuable constituent of the compound and complex aroma

of rum. We found that this essential oil usually appeared in the fifth fractions, and in some cases, when present in comparatively large amounts, in the sixth, seventh and eighth fractions in successive decreasing abundance. But it was always more

noticeable in the fifth fraction. Hence in our yeast selection work it became a matter of great interest and importance to find the extent in which a given yeast was able to produce this substance. From the ordinary chemical analysis by official methods the presence or absence of this valuable ingredient could not be ascertained.

The fractional distillation of the raw distillates produced by different yeasts gave us invaluable assistance in the selection of those more suitable for rum making. We found that the first fraction contained most of the low boiling point, low molecular weight, esters and aldehydes; the second and third fractions were practically composed of ethanol, with very little admixture of fusel oil, esters and aldehydes; the fourth fraction could be called the fusel oil fraction, as 65 to 70 per cent of the total amount of the fusel oil contained in the sample appeared here; in the fifth fraction passed over most of the high boiling point, high molecular weight, are the class more desirable in the chemical composition of a veritable rum. Fractions sixth, seventh and eighth, contained sometimes bad odorous products (especially the last two fractions) that we could not identify; but at times these ill-smelling substances distilled over mixed with small amounts of rum oil and very high molecular weight esters and aldehydes. In these instances the disagreeable smell of the products previously mentioned was masked more or less by the presence of these odorous ingredients.

The values of these different constituents of a given distillate varied quite appreciably according to the strain of yeast used in the fermentation of the product. Also, although these individual values were important in themselves, the ratios among constituents and group constituents were of equal, if not greater value. As the ratio of high boiling point, high molecular weight esters and aldehydes approached unity the quality of the rum was considered as improving. Similarly, as the ratio of fusel oil to total Non-Alcohol-Number diminished in value, the quality of the rum was also With the analytical data thus obtained, curves were drawn which offered a graphical representation of the quality of a given distillate, and indirectly of the fitness of the yeast strain involved for rum fermentation.

In table 5 are shown some of the results obtained during the fractional distillation work. In the analytical data all results, excepting that of alcohol content are expressed as milligrams per hundred millilitres of actual distillate. Graphical representations of the results will also be found in each case. These appear in the form of curves drawn with fraction numbers as abscissae and analytical values found as ordinates. A volatile acidity, a fusel oil, an ester, an aldehyde, and an alcohol curve, are found on each graph.

In the study of the results of fractionation, those members of the Non-Alcohol-Number distilling in the first three fractions were considered as low boiling point constituents; and those passing over in the following five fractions were considered as high boiling point constituents.

TABLE No. 5

SOME FRACTIONAL DISTILLATION RESULTS OBTAINED ON RAW RUMS DURING YEAST SELECTION STUDIES

[In the analytical data all results excepting that of alcohol content by volume; are expressed as milligrams per hundred milliliters of distillate]

	Sample No. 1									
Fraction No.	Temp. Range Degs. C.	Appear- ance	Per Cent Alcohol	Mgs. V. Acidity	Mgs. Esters	Mgs. Alde- hydes	Mgs. Fusel Oil	Remarks		
	76 70	d	04.40	4.27	220.40	120.00	11.00			
1	76-78	Clear	91.48	4.27	238.48	129.60	11.82			
2	78-78	Clear	92.60	2.67	42.24	32.40	6.44			
3	78-78	Clear	91.12	2.67	7.04	9.72	7.54	Turbid on Dilution		
4	78-96	Clear	86.78	9.34	35.20	19.44	92.40	Oil Droplets		
5	96-99	Cloudy	11.69	16.01	42.24	22.68	8.60	Oil Droplets		
6	99-99	Clear	1.04	6.23	28.16	12.96	3.22			
7	99-99	Clear	0.80	5.78	28.16	6.48	3.22			
8	99-99	Clear	0.28	3.56	28.16	6.48	3.22			

Sample No. 1

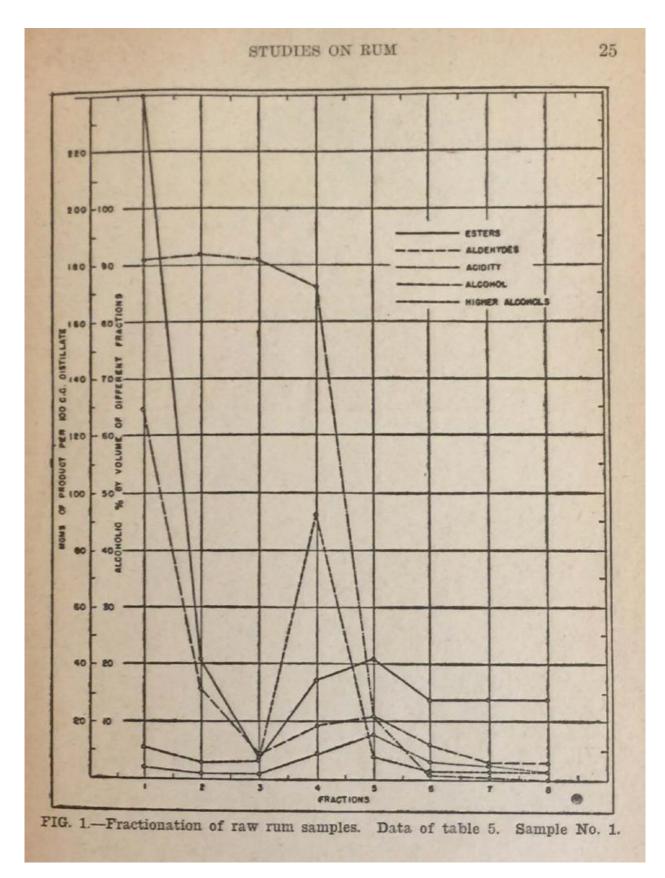


TABLE No. 5

SOME FRACTIONAL DISTILLATION RESULTS OBTAINED ON RAW RUMS DURING YEAST SELECTION STUDIES

[In the analytical data all results excepting that of alcohol content by volume; are expressed as milligrams per hundred millilitres of distillate]

	Sample No. 2									
				Ch						
Fraction No.	Temp. Range Degs. C.	Appear- ance	Per Cent Alcohol	Mgs. V. Acidity	Mgs. Esters	Mgs. Alde- hydes	Mgs. Fusel Oil	Remarks		
1	77-78	Clear	92.84	1.27	232.32	119.88	18.13			
2	78-78	Clear	94.40	1.27	28.16	29.16	9.88			
3	78-79	Clear	95.12	1.27	17.60	25.92	11.56			
4	79-83	Clear	90.04	1.27	28.16	25.92	1.78	Turbid on Dilution		
5	83-99	Cloudy	22.04	25.04	70.40	42.16	13.19	Oil Drops		
6	99-99	Clear	1.84	22.05	28.16	38.88	4.94	Oil Drops		
7	99-99	Clear	1.84	24.38	17.60	35.46	4.94			
8	99-99	Clear	0.52	31.88	17.60	38.88	4.94			

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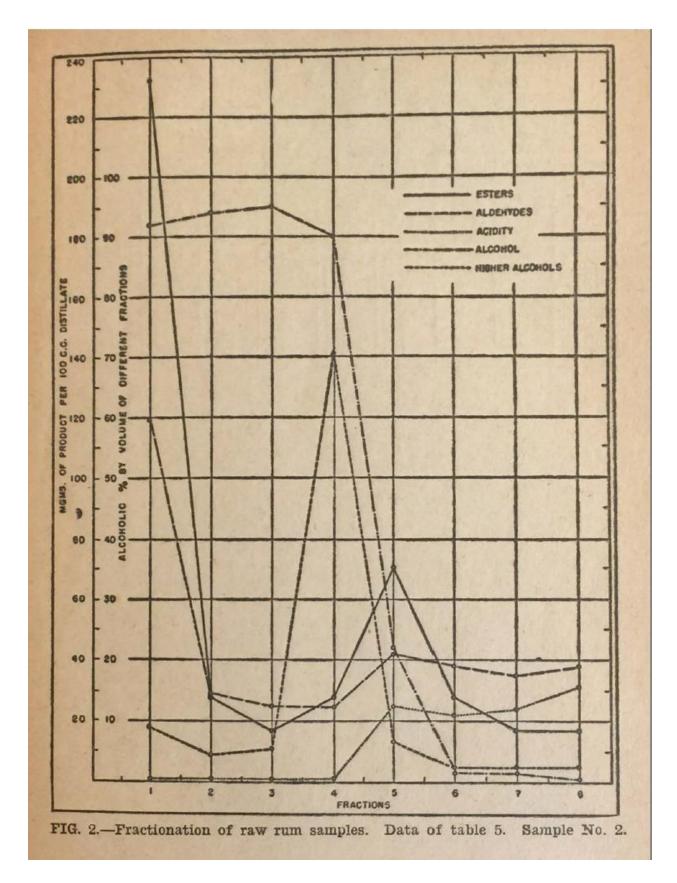


TABLE No. 5

SOME FRACTIONAL DISTILLATION RESULTS OBTAINED ON RAW RUMS DURING YEAST SELECTION STUDIES

[In the analytical data all results excepting that of alcohol content by volume; are expressed as milligrams per hundred millilitres of distillate]

	Sample No. 5								
				Ch					
Fraction No.	Temp. Range Degs. C.	Appear- ance	Per Cent Alcohol	Mgs. V. Acidity	Mgs. Esters	Mgs. Alde- hydes	Mgs. Fusel Oil	Remarks	
1	76-78	Clear	93.68	1.49	84.48	38.88	9.71		
2	78-78	Clear	93.68	1.28	36.50	25.92	5.29		
3	78-78	Clear	93.32	1.06	30.27	16.20	6.19		
4	78-89	Clear	92.99	1.28	24.64	16.20	75.90	Slightly turbid on dilution	
5	89-99	Cloudy	20.52	2.99	18.30	12.96	7.06	Oil droplets	
6	99-99	Clear	2.64	1.28	6.34	12.96	2.65		
7	99-99	Clear	1.60	1.06	6.34	12.96	2.65		
8	99-99	Clear	1.60	1.06	6.34	12.96	2.65		

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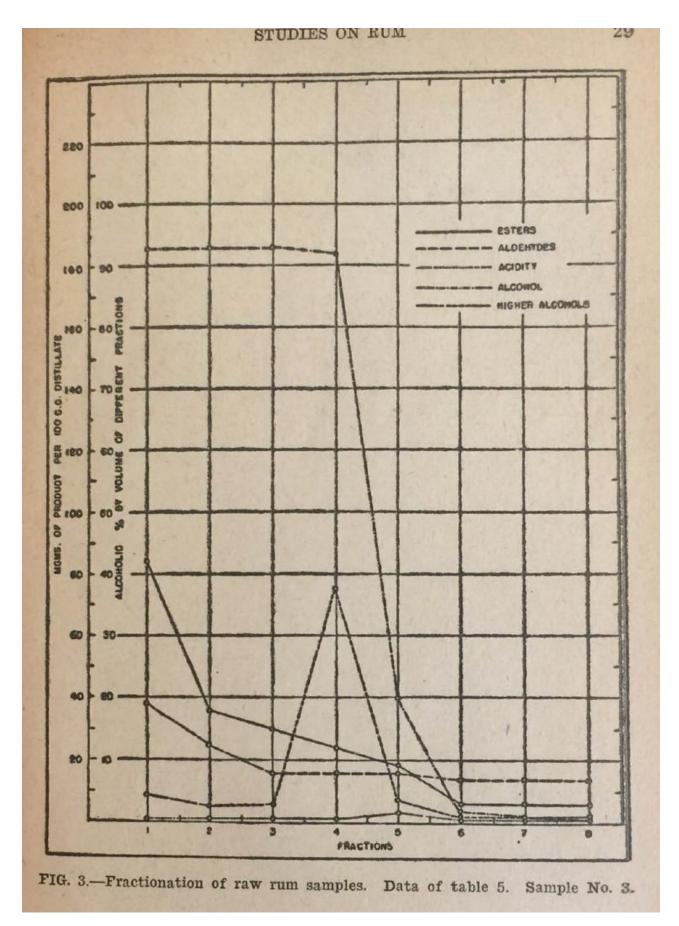


TABLE No. 5

SOME FRACTIONAL DISTILLATION RESULTS OBTAINED ON RAW RUMS DURING YEAST SELECTION STUDIES

[In the analytical data all results excepting that of alcohol content by volume; are expressed as milligrams per hundred millilitres of distillate]

Sample No. 4								
			Chemical Analysis					
Fraction No.	Temp. Range Degs. C.	Appear- ance	Per Cent Alcohol	Mgs. V. Acidity	Mgs. Esters	Mgs. Alde- hydes	Mgs. Fusel Oil	Remarks
1	75-78	Clear	94.88	2.75	143.62	69.86	20.14	
2	78-78	Clear	94.16	2.06	41.54	46.58	10.97	
3	78-78	Clear	93.12	2.75	24.00	40.75	12.84	
4	78-81	Clear	90.96	2.75	21.54	46.58	157.46	Very cloudy on dilution
5	81-99	Cloudy	24.12	15.13	67.87	54.93	14.65	Oil Droplets
6	99-99	Clear	1.60	9.63	28.86	11.64	5.49	Oil Droplets
7	99-99	Clear	1.60	9.63	35.20	11.64	5.49	Oil Droplets
8	99-99	Clear	1.04	8.94	35.20	11.64	5.49	-

Sample No. 4

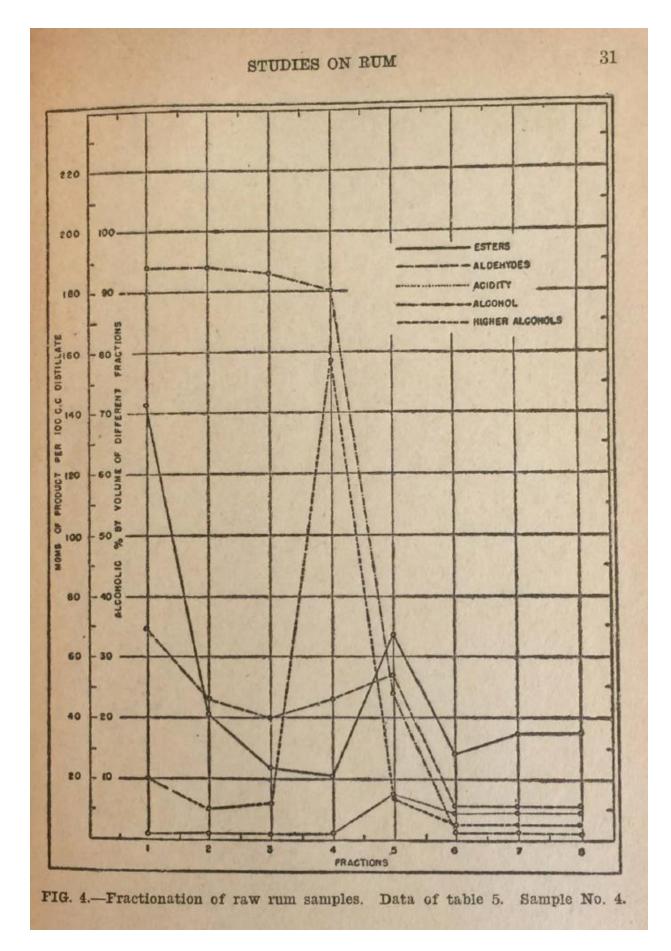


TABLE No. 5

SOME FRACTIONAL DISTILLATION RESULTS OBTAINED ON RAW RUMS DURING YEAST SELECTION STUDIES

[In the analytical data all results excepting that of alcohol content by volume; are expressed as milligrams per hundred millilitres of distillate]

Sample No. 5									
				Ch					
Fraction No.	Temp. Range Degs. C.	Appear- ance	Per Cent Alcohol	Mgs. V. Acidity	Mgs. Esters	Mgs. Alde- hydes	Mgs. Fusel Oil	Remarks	
1	75-78	Clear	93.95	7.50	240.00	95.60	5.90		
2	78-78	Clear	93.12	9.00	46.00	29.90	4.40		
3	78-81	Clear	93.00	9.00	40.50	19.40	4.50		
4	81-88	Clear	87.90	11.30	33.30	27.40	92.70	Slightly turbid on Dilution	
5	88-99	Cloudy	39.30	45.00	153.70	47.50	19.88	Abundant Oil Droplets	
6	99-99	Cloudy	16.14	92.70	90.20	25.80	11.80	Abundant Oil Droplets	
7	99-99	Clear	2.85	111.50	65.90	18.00	11.80	Oil Droplets	
8	99-99	Clear	1.90	147.00	65.90	18.00	11.80	Oil Droplets	

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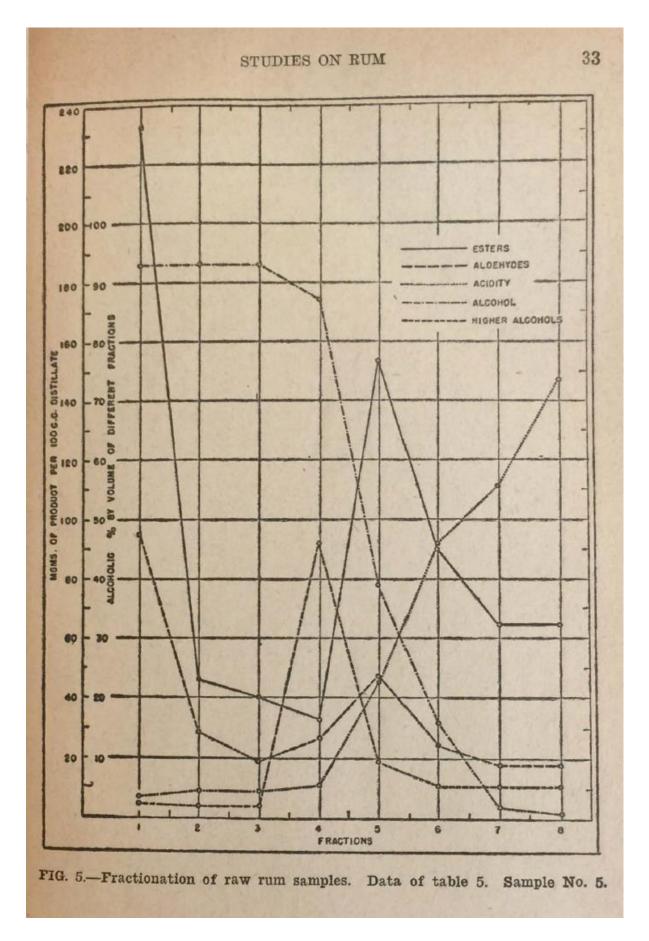


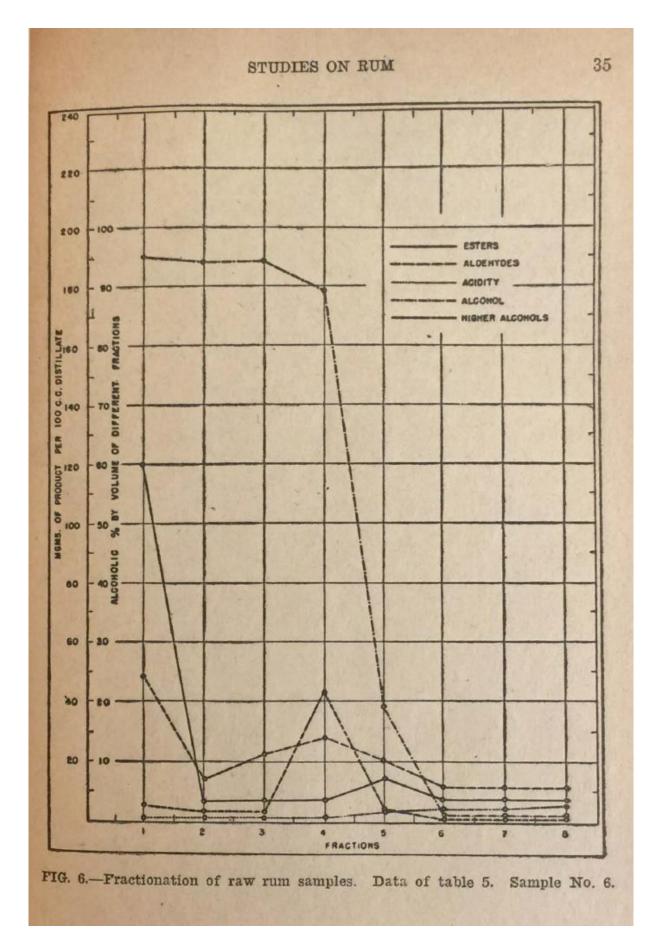
TABLE No. 5

SOME FRACTIONAL DISTILLATION RESULTS OBTAINED ON RAW RUMS DURING YEAST SELECTION STUDIES

[In the analytical data all results excepting that of alcohol content by volume; are expressed as milligrams per hundred millilitres of distillate]

				Chemical Analysis					
Fraction No.	Temp. Range Degs. C.	Appear- ance	Per Cent Alcohol	Mgs. V. Acidity	Mgs. Esters	Mgs. Alde- hydes	Mgs. Fusel Oil	Remarks	
1	76-78	Clear	95.12	1.71	119.68	48.62	5.60		
2	78-78	Clear	94.40	1.50	7.04	14.30	3.05		
3	78-78	Clear	94.76	1.28	7.04	22.88	3.57		
4	78-81	Clear	89.32	1.28	7.04	28.60	43.79	Slightly turbid on Oil	
5	81-99	SlightlyCloudy	19.08	3.41	14.12	20.02	4.07	Few Oil Droplets	
6	99-99	Clear	1.84	4.06	7.04	11.44	1.53		
7	99-99	Clear	1.84	4.70	7.04	11.44	1.53		
8	99-99	Clear	1.32	5.55	7.04	11.44	1.53		

Sample	e No. 6
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Other tests conducted on the rum samples submitted to fractional distillation consisted in determinations of: (1) rum body (2) index of persistence of taste and aroma; (3) sulphuric acid test; and (4) organoleptic tests for taste and aroma. These were of great supplementary value to the fractional distillation results. The results of these supplementary tests may be found in table 6. As some of these tests are not yet of general use in the industry, at least in Puerto Rico, we shall give a succinct account of the methods followed in these determinations:

Rum Body Number:

The sample is lowered to an alcoholic strength of 30 per cent by volume by the addition of redistilled, pure water. A burette is calibrated in such way that it will deliver exactly 10 ml of absolute alcohol in one minute, at the temperature of 25 degrees Centigrade. Then the time taken for the delivery of an equal amount of the rum sample at the same temperature is used as a measure of "Rum Body". The "body" is expressed in hundredths of a minute taken by the rum under test, over the time taken by the standard pure alcohol. Triplicate tests are run, and an average is used as the correct "Body Number". For example, the average reading of a test shows that it takes 1.13 minutes to deliver 10 ml of a given sample. Then the "Body Number" of this particular sample is 13.0; and the higher the number, the intenser the body.

Index of Persistence:

This test shows the extent of the dilution that a rum sample will stand before its aroma and taste can no longer be detected by an experienced rum taster. The diluent used is 40 per cent solution of neutral grain or potato spirits. Specially graduated taster's cups are used for this work. These cups are made of Jena glass, hold about 150 ml, and have loosely fitting glass covers.

TABLE No. 6

Sample No.	mple No. Body No.		Sulphuric Acid Test	Aroma	Taste	
1	9.5	1:16,700	Strong	Fair	Good	
2	9.8	1:16,700	Strong	Good	Good	
3	9.0	1:15,000	Strong	Fair	Fair	
4	10.0	1:25,000	Strong	Very Good	Good	
5	11.5	1:35,000	Very Strong	Excellent	Very Good	
6	8.0	1:5,000	Weak	Fair	Fair	

RESULTS OF SUPPLMENTARY TESTS CONDUCTED ON THE RUM SAMPLES SUBMITTED TO FRACTIONAL DISTILLATION

A stock solution of the rum to be tested is prepared in one of these cups by pouring from a micro-pipette one tenth of a millilitre of the sample. Immediately the cup is filled to the 50 ml mark with the solution of neutral spirits in ordinary water. Distilled water is not used here as diluent for it has been found to interfere with the accuracy of this test by imparting a flat, undesirable taste of the rum solution. In this way is secured a stock solution diluted 500 times. Aliquots of this solution are then used for further dilution tests. That dilution number at which an experienced taster will barely notice the rum taste and aroma, becomes the Index of Persistence Number for the given sample. The indexes thus obtained may be given as such, or may be further classified for raw rums in the following way:

Less than 1:5,0000...... Very Low

1:5,000	Low
1:10,000	Moderate
1:15,000	Normal
1:25,000	High
1:50,000	Very High

This method is a modification of that used by Dr. Luckow already mentioned in connection with the birectifier.

Sulphuric Acid Test:

Ten ml of the sample are run into a 25 ml test tube. Three to five millilitres of strong sulphuric acid are mixed with the liquid in the test tube, and this is left standing overnight. The next morning the mixture is tested organoleptically for residual aroma. Total disappearance of the aroma indicates that the distillate lacked rum oil as an ingredient. A faint survival of aroma indicates a low rum oil content; and strong aroma retention indicates a good product. Sometimes the original aroma changes to a disagreeable odour, and this may be caused by the presence of sulphur compounds in large quantities in the molasses used to produce the rum.

During the fractional distillation, organoleptic tests performed on the various fractions are also of great value. The first fraction will offer a penetrating odour recalling that of a mixture of highly volatile esters and aldehydes. That this is the case may be proved by exposing a portion of this fraction to the atmosphere in a shallow vessel or watch glass. It will then be noticed that the characteristic smell

will disappear in a few hours. Fractions number two and three will offer a more delicate, but greatly diminished aroma, provided the original sample be very rich in esters and aldehydes.

These two fractions will offer an almost neutral spirits odour if the sample is of low ester and aldehydic content. The fourth fraction will offer the characteristic smell of fusel oil, provided the original sample is almost devoid of rum oil; but this fusel oil scent will be greatly masked and subdued in case the sample be rich in rum oil content. The fifth fraction will offer an aroma very similar to the characteristic smell of rum oil. The aroma of the remaining fractions may in some cases resemble that of fraction five, but it will be noticed that especially in the cases of fractions seven and eight this agreeable odour may be contaminated by a characteristic foulness. The aroma of fraction five is very persistent, due to the large content of rum oil and also to the fact that here are found the larger proportion of the high boiling point esters and aldehydes. This aroma has persisted in some instances for a period of fifteen days even when the liquid within the cup has seemingly evaporated out. But if the finger was passed over the bottom surface of this cup it felt oily, showing that a very thin film of rum oil was responsible for the odour still noticed. It also showed that rum oil evaporates out slowly.

It thus appears that yeast selection is not an easy task; the ideal rum yeast strain being hard to find. A yeast may be found giving the right taste, body and aroma but that would take too long for accomplishing fermentation, or that would produce uneconomical yields of the product. At other times fair, or even excellent yields are obtainable in a reasonably short fermentation period; but these good features were not accompanied by the desired organoleptic characteristics of taste and bouquet. In our opinion, quality of distillate and rapid maturing properties are the supreme requirements in rum distillery practice. Of course, if to those may be added high yields and short fermentation periods, so much the better.

The art of the distiller consists in ameliorating or compensating these natural and variable limitations of rum yeast through proper, judicious practices in raw material pretreatment and mashing; as before stated, the proper selection of the yeast strain constitutes the foundation stone of the distillery's success.

III

Mashing Operations

The object of mashing operations is to prepare and condition the raw material in such way that will facilitate to the yeast the carrying out of fermentation under optimal conditions. This presupposes on the part of the distiller an intimate knowledge of at least position; (3) the dilution water; (4) the pretreatment and conditioning that the raw material may require.

We shall begin our discussion with the fourth factor, that is, the matter of pretreatment of the raw material. This will differ with the raw material being used, but as already states in a previous chapter, the most important raw material for rum is final molasses; and in this case the pretreatment has three principal objectives: (1) complete, or partial sterilization of the material; (2) preparation of the material for the development of fine aroma during fermentation; (3) purification of the raw material through precipitation of non-sugars.

The first objective is accomplished through the application of heath a predetermined temperatures. In case of partial sterilization (by which is meant here the destruction of all vegetative microbiological flora) this heating takes place at temperatures not above eighty degrees Centigrade. In case of complete or absolute sterilization (by which is meant here the destruction of high heat resistant spores besides the vegetative cells) recourse must be had to pressure cookers and temperatures between 120 and 130 degrees Centigrade.

In rum manufacture, however, there is rarely the necessity of going into pressure sterilization, it being generally sufficient to destroy the microorganisms in vegetative form existing in the raw material. Heath resistant spores may b e allowed in the substrate, for under proper fermentation conditions by the time they enter into the vegetative stage the peril of their doing any harm has already passed. Pressure sterilization may become necessary in certain cases, as when using, by necessity, a very slowly fermenting yeast for the sake of its other characteristics, or for obtaining a definite sought for aroma and taste in the resulting rum. Most always, the procuring of absolute sterilization of the medium before the rum fermentation, brings into play many adverse factors to the economy of the process, such as: (1) first cost and installation of the pressure equipment; (2) running and maintenance expenses; (3) changes may take place within the material due to the high temperatures involved, that may interfere with its fermentative qualities, for instance undue caramelization of the sugars; (4) more skilful labour must be employed, and (5) there is always the greater risk of manipulation inherent to pressure systems.

We found that in rum fermentation we could secure almost any result with partial sterilization that could be secured by the use of absolute sterilization. We, for this reason, recommend the use of the first to rum producers. Another great advantage in the use of partial sterilization is that the process may be combined in one operation with those intended for the development of fine aroma and for precipitation of non-sugars.

The combined process may be carried on in the following way:

A cylindrical iron tank is equipped with steam heating coils, a motor driven mechanical stirrer and recording thermometer in the Centigrade scale. The molasses is pumped or delivered by gravity into this tank, and is there mixed with a predetermined amount of milk of lime which is calculated to raise the pH of the material by 0.5; the actual amount to be employed being determined experimentally according to the density and original pH of the material, and the amount treated by batch. After introducing the milk of lime, the stirrer is set in motion and hot water is added with vigorous stirring until the resulting mixture attains a density between 45 and 55 degrees Brix. The hot water is shut off at this point, and the temperature of the mixture is adjusted by the heating steam coils so that a temperature between 70 and 80 degrees Centigrade is attained; this temperature being then maintained for at least one half hour.

While still under strong agitation the mass is then passed through a filtering device such as a supercentrifuge, for separation of solid organic and inorganic impurities, such as molasses gums and ash which have been precipitated or separated during the treatment. The clean run off is then delivered into a second tank which is equipped similarly to the first just described, except that its coils are used for water cooling the incoming material instead of for heating purposes. As soon as the coils are covered by the in flowing liquid the cooling is started by circulation of cold water through the coolie coils, agitation being continued during the entire cooling period. When the temperature has dropped to about 35 to 40 degrees Centigrade, there is added to the contents of the tank enough ammonium sulphate and calcium superphosphate to compensate any deficiencies in nitrogen and phosphoric acid inherent to the raw material. Immediately after the incorporation of these nutriments, and while still agitating the liquid, strong sulphuric acid is added until a new pH value of between 5.0 and 5.6 is obtained. The liquid is then filtered through a second supercentrifuge for separation of newly

precipitated impurities. The cleaned, purified, and conditioned material thus resulting, is delivered to a receiving tank from which it is drawn as needed for mashing operations. For very large installations the batch process may be advantageously substituted by a continuous one.

Another valuable modification of this process may be introduced in distilleries situated as adjuncts of raw sugar factories. The modification consists in substituting hot clarified juice from the sugar factory for the hot water previously mentioned. This modification was tried by us in the laboratory, and we found that it was particularly valuable when dealing with very poor grades of final or blackstrap molasses, low in total sugars and high in ash. The total sugars in the material so treated will be increased from 5 to 10 per cent, and this will in turn modify the total sugar-ash ratio in a most favourable manner. The resulting material becomes much fitter for an efficient and quick fermentation. The resulting rum will also have the characteristics sought for by some rectifier when blending molasses and sugar cane juice rums. The total amount of sugar cane juice to be used is not great; about 50 per cent of the weight of raw molasses used daily at the distillery. This means that a rum distillery using 5,000 gallons of final molasses per day would also use about 3,500 gallons of clarified juice.

This pretreating process is very valuable and important, as the molasses is thereby cleaned of foul smelling substances such as dissolved or occluded gases or other volatile substances usually found in the raw material. It is also rid of organic and inorganic impurities always present in the molasses such as excess ash and gums. The use of a supercentrifuge was found the most expeditious way of separating the precipitated or suspended impurities; but other means may be adopted such as pressure filtration through a suitable filtering material; settling and decanting, etc.

The action of heat during this pretreatment renders the material practically free of microbiological contaminants, excepting those in the high heat resistant spores form. The total sugars concentration in the molasses is also increased through the withdrawal of non-sugars.

The addition of the milk of lime during the pretreatment has three main purposes:

a. It prepares the medium for the development during fermentation of the important rum oil.

- b. It neutralizes the free fatty acids which are always present in the molasses, thus eliminating the danger of their volatilization during the heating period which immediately follows; but permitting the reliberation of these fatty acids from their calcium salts upon the addition of sulphuric acid after temperature has been cooled down so that no danger of fatty acid loss may be expected. In this way the fatty acids become available for the formation of valuable esters during fermentation under the catalytic action of the esterese produced by the yeast.
- c. The inner disturbance produced in the material through the alteration in pH value occasioned by the milk of lime causes a copious precipitation of organic and inorganic solid impurities, this precipitation being accelerated and enhanced by the simultaneous application of heat.

In table 7 will be found data on the comparative chemical composition of the raw material before and after the pretreatment, water of dilution being eliminated from the calculation.

It will be noticed that the different Brix densities offer lower values for the treated material, the drops amounting to from 4.0 to 7.0 per cent, calculated upon the original density values. This is of course due to the withdrawal of non-sugars from the material during pretreating operations. This withdrawal of non-sugars brings about an increase in the concentration of total sugars, varying in the different samples presented, from 3.0 to 5.0 per cent calculated upon the original total sugars concentration of the material. Similarly, and for the same reason, the ash and gums drop in amounts equivalent to from 30.0 to 45.0 per cent of their respective original values. The pH values, phosphoric acid, and nitrogen contents have been modified and conditioned so as to approach optimal values.

All of these valuable changes in the composition of the raw material bring about readjustments in the various ratios referred to in our chapter on Raw Materials. In this way ratios considered as poor before the pretreatment of the raw material are transformed to fair, or even good ones. The transformation of the original molasses into a much better and suitable fermentation raw material for rum production is apparent and undeniable.

These beneficial changes in the chemical composition of the raw material are not the only advantages derived from the pretreatment process just described. Physical improvements just as important are also effected, some of which result in bettering fermentation conditions and others in improving the rum quality and the manipulation of the raw material during mashing operations. Among these physical improvements may be mentioned: (1) increased fluidity; (2) elimination of stickiness; (3) amelioration or elimination of foul odours; (4) increased handiness of the material; and (5) decreased surface tension and viscosity.

Biologically, the conditions of the raw material are also greatly enhanced, since the material will be free of contaminating microorganisms in the vegetative stage. This will insure predominance over the substrate to the pitched yeast during the fermentation period.

The beneficial effects of the pretreatment will be felt not only during the succeeding fermentation, but also during distillation, and in the recovery of valuable by-products such as yeast bottoms and potash salts. Let us discuss briefly how this takes place: During distillation, organic and inorganic impurities present in the beer tend to foul the plates of the distilling column. In some instances, this brings about considerable trouble leading to frequent shutdowns for cleaning purposes. During these shutdowns not only is production stopped, but also extra expenditures must be met in the form of labour, chemicals, and steam used for cleaning purposes. Besides, unusual deterioration of the still and other equipment results from too frequent cleaning. Obviously, the degree, or extent of trouble due to this source, will be in direct ratio with the cleanliness of the fermented material entering the still. As one of the most important advantages of the pretreatment is the cleaning effect it has on the material, it follows that distillation is favoured in a great measure by the pretreatment. A saving of steam consumption during distillation is also effected when the beers are clean.

As to the recovery of valuable by-products, it is gaining more and more importance and attention in distillery practice; especially since the entrance of the United States into the World War. Among the valuable by-products we have yeast bottoms and potash salts. The pretreatment of the raw material influences these two recoveries in a marked degree.

During fermentation the yeast accumulates at the bottom of the fermenter, mixed with precipitated solid impurities of organic and inorganic nature. When the raw material has passed through the pretreatment process very little of these impurities are found mixed up with the yeast bottoms. This fact will particularly benefit the by-product by lowering its ash content and increasing the protein value. The by-product is recoverable in a more pure, concentrated form, its value being increased whether as a feed or a fertilizer. With respect to potash recovery, the influence of the pretreating process is yet more pronounced. As we have shown from the comparative ash percentages in treated and untreated molasses, as high as 45.0 per cent of the ash content in the raw molasses is eliminable during pretreatment. But that part of the ash composed of potash salts is not precipitable during the process, this material being left in solution in a much enriched condition. Hence, the concentrated potash salts containing slops, when evaporated and ignited for potash recovery will yield a much purer and concentrated potash product than that usually obtained at present. Actual analyses have proved that the content of K_20 in the material obtained from treated slops is from 10 to 15 per cent high in value than that obtained from ordinary slops. A comparative analysis effected by the writer offered the following figures:

K₂0 percentage in ash obtained from ordinary slops 32.50

K₂0 percentage in ash obtained from treated slops 43.87

The pretreating process may be used by progressive rum distillers as a mean to obtain a raw material for his daily fermentations of practically constant composition. This may be accomplished by careful analysis of each batch of raw molasses bought, and then modifying his pretreatment procedure in such form that the resulting processed molasses will vary very little in chemical composition from day to day. The advantages of a practically constant composition raw material are well realized by the intelligent practical distiller.

Considering now the first of the four factors mentioned above, that pertaining to the peculiar characteristics of the yeast used by the distiller, we have that different strains of rum yeasts require differences in the setting of the mash so that they can perform their work of fermentation under optimal condition. Of special importance in this respect are the initial total sugars concentration, temperature and pH value of the mash. As regards optimum pH value no fixed rule can be given, and every distiller should find out experimentally the most suitable initial pH value for his particular yeast strain. The different values lie however within the range 4.5 to 5.5. A few general remarks will not be out of place here. We have found that the question of pH must be considered in connection with the type of fermentation to which the mash will be subjected later on in the fermentation room. For instance, the optimal pH value for a given strain may be quite different when it is to be used under pure culture fermentation technique than when operating in a non-previously sterilized medium. While a higher pH value, for instance 5.5 to 5.8 may prove optimal under the first condition named, a much lower value, for instance 4.5 to 5.0 may become necessary in the second case, that is, when acting upon a contaminated substrate.

Hence, in the setting of the initial pH at mashing, the distiller must have in mind the conditions under which the succeeding fermentation will take place. The facilities on hand to maintain optimal condition of pH during the entire fermentation period is another factor influencing here the initial pH value of the mash.

While in molasses mashes intended for industrial alcohol fermentation is it customary to set at such pH value so that no further increase in acidity occurs during the subsequent fermentation, this does not always hold for mashes intended for the production of rum, for in this case increase in acidity due to fatty acids formation during fermentation is sometimes very desirable, as they later combine with various alcohols, especially ethanol, to form valuable esters. We have found that rums fermented at relatively high pH values possess more mellowness and delicacy of taste and aroma; but unfortunately the production of such rums is quite more complicated, due principally to the lack of facilities existing in present day distilleries for sterilization, partial or absolute.

Coming to the matter of initial sugars, we have found that their concentration in the mash will depend to a certain extent on the ability of the yeast strain to use them efficiently' but also on the temperature control available during fermentation, on the pH at which this fermentation will take place, the quality of the raw material, and lastly on the type of rum that is to be produced. In general terms, mashes for the production of rum are set at a total sugars concentration within the range 11.0-13.5 grams of total sugars per 100 ml mash, while yeast employed for industrial alcohol production are able, and are required to ferment mashes of from 15-16 grams sugars per 100 ml and even higher. The writer has developed a new process (¹⁵) for fermentation of blackstrap molasses to alcohol or light rums at the distillery of "Borinquen Associates Inc." of Hato Rey, P.R. (of which he is the technical advisor), in which process much higher total values are efficiently fermented than in previous practices. This is accomplished through the incremental addition of sugars in the form of thick mash to the fermenters.

A possible explanation for the difference of behaviour between industrial alcohol and rum yeasts may be found in the fact that the fermentation are conducted generally under much superior biological and chemical control in industrial alcohol distilleries. Also better equipment and more efficient personnel are found in such distilleries.

But apart from the advantages on the part of industrial alcohol establishments, there exists a natural difference between industrial alcohol and rum yeast that would explain the difficulty on the part of the latter to cope successfully with high sugars concentrations in the substrate. This is found in the fact that their respective products of metabolism differ somewhat; the ideal yeast for industrial alcohol being that capable of producing the largest amount of pure alcohol in the least possible time, with practically non-production of a Non-Alcohol-Number. On the other hand, in exactly opposite position, rum production requires the use of yeasts capable of building up an appreciable Non-Alcohol-Number during fermentation, such as organic acids, aldehydes, esters, higher alcohols, essential oils, rum oil, etc.

Now, the toxic effects upon the yeast cell caused by the accumulation of its own metabolic products, will increase in direct ratio to the production of the abovementioned congeneric products of rum fermentation, since it has been proved that the toxicity of some of these congeners towards the yeast is many times greater than that of ethyl alcohol. Hence, the tolerance of rum yeasts to the combined ill effects of its own metabolic products is below that of industrial alcohol yeasts; and consequently fermentation will be arrested sooner than in the case of industrial alcohol production, that is, when a smaller amount of sugars have been utilized by the yeast. Such being the case, setting the mashes at higher concentrations of sugars than the yeast can use efficiently, merely represents a great waste, for the residual sugars will be lost in the discarded slops. The writer has had occasion to analyze distillery slops running up to 3.5 per cent total sugars due to a lack of proper understanding of mashing operations.

The initial sugars concentration that may be used at mashing is also dependent on the temperature at which the fermentation of the mash will take place. This point is of real practical importance, especially in the tropics where high fermentation temperatures are apt to occur unless artificial cooling of the fermenting mash is resorted to. Mashes intended to be fermented at relatively high temperatures, say between 35 and 40 degrees Centigrade, which is a range very commonly found in Puerto Rico, cannot be mashed at initial high sugars concentration; while the lower the temperature at which fermentation will take place (within the range of course of rum yeast) the higher the initial sugars may become. The reasons for this assertion will be given under the chapter on Rum Fermentation. Higher total sugars concentration may also be used when the raw material contains an ample supply of yeast nutriments, or when these may be added artificially in the correct ratios and proper quantities. The second factor, or the knowledge of the chemical composition of the raw material is fully as important as the one just discussed: the yeast, like any other plant is dependent for its growth and propagation on certain elements furnished by the medium in which it lives. Among the elements most essential to the life of the yeast are carbon, nitrogen, phosphorus and potassium; followed by magnesium, iron, manganese, sulphur and calcium. Fortunately, sugar cane final molasses contains most of these elements in variable quantities; but cases of deficiencies as to the amounts are common, especially in nitrogen and phosphorus. These deficiencies, when existing, must be corrected at mashing time.

Dilution of the mash for rum production should be a function of the chemical composition of the raw material as well as of the inherent characteristics of the yeast strain in use. The first point to bear in mind is that of resulting total sugars concentration, for any concentration above what the yeast can handle efficiently under the conditions of fermentation at which it will act, will mean an unnecessary waste. This cannot be done properly without an accurate knowledge of the sugar content of the raw material, treated or untreated. This means that for a given value of initial sugars concentration high test molasses must be diluted more than low sugar test ones; but it must be remembered that when diluting the molasses for right sugars concentration, we are also diluting the concentration of the yeast nutriments in the molasses. Hence, a high test molasses will require more heavy treatment with yeast nutriments than a low test one. Other reason for so doing is the fact that high test molasses is usually more deficient in nitrogenous and mineral constituents. Very low sugars molasses, if of normal Brix densities are usually high in ash and other non-sugars. When dealing with such molasses high initial sugars concentration at mashing is out of the question for this could only obtained by the simultaneous accumulation of initial non-sugars. The result is in such cases that after fermentation has been going on for sometime the ratio of non-sugars to sugars increases so rapidly as to inhibit the zymogenic power of the yeast, with the production of high residual sugars and very poor fermentation efficiencies.

The third factor, or that related to the quality of the dilution water, hardly needs commenting, for the important role played by industrial water in all biochemical processes is well-known. We must mention the fact, however, that in the case of a product like rum, whose price depends on quality, we must use particular precautions with the quality of the water used for mashing especially from a bacteriological standpoint. If greatly contaminated, the water of dilution may render useless and destroy the good effects intended to be produce through the precautions and expense involved in the pretreatment stage already described. Some of the members of the microbiological flora of the dilution water may become responsible for side fermentations during or after the main alcohol fermentation, generating ill-smelling impurities. These will pass in concentrated condition together with the raw distillate, ruining both its taste and aroma. On the other hand, too high mineral content in the water of dilution, especially salts of calcium and magnesium, will raise the ratio of non-sugars to sugars in the mash with detriment to the growth and zymogenic activity of the yeast. Especially during the last stages of fermentation will this inhibitory action be felt, resulting in high residual sugars and unduly prolonged periods of fermentation.

Later on, during the distillation of the fermented mashes, the malefic effect of these non-sugars will be experienced in the form of scale on the plates and feed pipe of the still, lowering its efficiency and giving rise to frequent stoppages for cleaning.

From what has been stated it will be realized how important a matter proper mashing operations become. As a rule, the properly mashed material should offer the following or similar analysis:

Brix density	18.0-21.0
Titratable acidity (in ml of tenth normal alkali per 10 ml mash)	2.0-3.50
pH value	5.0-5.50
Total sugars (grs per 100 ml)	11.5-13.50
Nitrogen (milligrams per 100 ml)	75.0-100.0
Phosphoric acid as P ₂ O ₅ (milligrams per 100 ml)	15.0-20.0

TABLE NO. 7

SHOWING THE COMPARATIVE CHEMICAL COMPOSITION OF BLACKSTRAP MOLASSES BEFORE AND AFTER THE PRETREATMENT OPERATIONS

		SAMPLE NO.										
	-1-		-12-		-3-		-4-		-5-			
	Before	After										
Brix Density Total Sugars (as invert)	90.90 50.17	84.40 52.68	86.40 55.56	83.00 57.23	90.76 48.59	85.06 50.97	86.70 46.40	82.10 48.49	89.60 57.97	84.10 60.72		
Ash (carbonated)	14.20	8.52	7.86	5.50	12.90	7.74	10.04	6.52	10.57	6.60		
Gums Nitrogen (total)	6.19 0.55	3.40 0.95	3.41 0.94	2.04 1.11	4.05 0.55	2.14 0.98	2.90 0.60	1.74 1.01	3.71 0.45	2.11 0.97		
P ₂ O ₅ pH Value	0.21 6.10	0.23 5.20	0.15 5.54	0.20 5.04	0.14 5.10	0.21 4.70	0.12 5.50	0.20 5.00	0.08 6.25	0.19 5.20		
Total Sugars Ash Ratio Gums Total Sugars Ratio	3.53 0.12	6.18 0.06	7.07 0.06	10.40 0.04	3.77 0.08	6.58 0.04	4.62 0.06	7.43 0.04	4.48 0.06	9.20 0.03		
P ₂ O ₅ Nitrogen Ratio	0.38	0.24	0.16	0.18	0.25	0.21	0.20	0.20	0.18	0.20		

49

IV

Mitogenetic Radiation a Factor In Rum Fermentation

This topic could have been discussed as part of the subject matter of the succeeding chapter, but we decided to give it separate attention for two good reasons: (1) some of the subject matter of the following chapter will be easier to understand; (2) mitogenetic radiation and its effect on cell multiplication has been the subject of a great deal of scientific controversy in recent years, and the development of convincing evidence such as appears in this discussion in proof of the existence of this phenomenon becomes a matter of considerable consequence.

By mitogenetic radiation is meant an energy in the form of rays that are given up by some biological materials in certain stages of development. These rays pass through quartz, not through glass; and when they encounter other growing tissues in certain stages of development they may produce effects in these which may be recognized in the form of increased growth or reproductive activity.

Alexander Gurwitsch (¹⁷) first published his discovery of these so-called mitogenetic rays in 1923. Since that time many articles dealing with the subject have appeared, some authors confirming and other failing to corroborate Gurwitsch's work.

We were led to delve into the phenomenon of mitogenetic radiation incidental to these studies on rum manufacture. In our work we followed a modification of the technique first described by Baron (¹⁸), who worked in Gurtwitsch's laboratory. Our results seem to confirm Gurwitsch's claim.

In the course of this study on rum manufacture we used mixed culture fermentation of yeast and bacteria under strict control. The cultures consisted of pure strains of Schizosaccharomyces Pombe (Lindner) and a culture of Clostridium Saccharo Butyricum found and isolated by us. When fermentation was accomplished by the pure yeast strain acting singly in the substrate, it was observed that:

(1) Fermentation, as well as cell multiplication, was very slow, especially during the first 24 hours period.

(2) Most of the cell multiplication and practically all of the alcohol was formed during the period following the first 24 hours, the total fermentation period lasting about 96 hours.

(3) Yeast cell counts conducted immediately after inoculation and at optimum multiplication proved that the original concentration of yeast cells usually increased from 15 to 20 times.

Whenever mixed pure cultures of yeast and bacteria were used under controlled conditions, we invariably obtained altogether different fermentation results:

(1) The duration of the fermentation period was reduced to a very large extent; that is, the former 96 to 100-hour period was reduced by from 40 to 50 per cent.

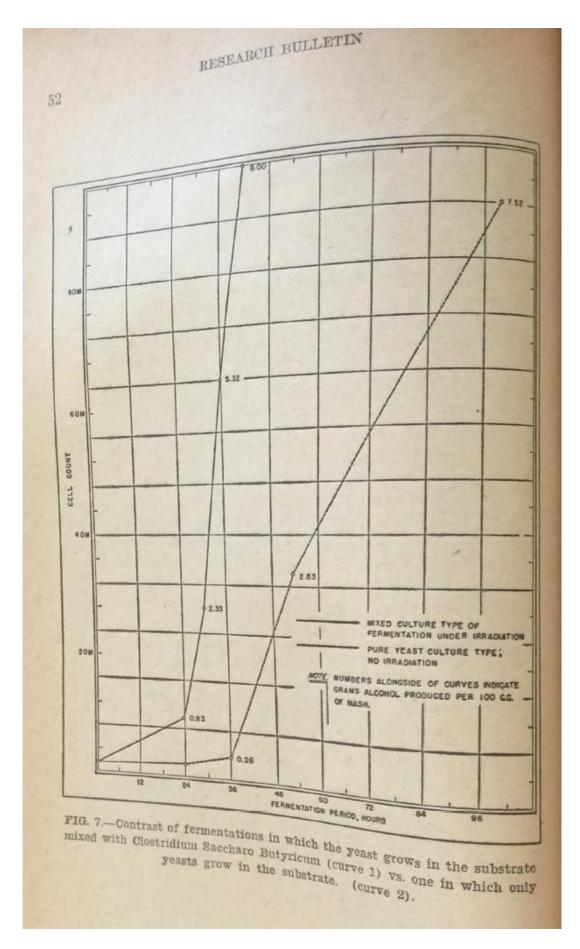
(2) The yeast developed consistently a very high increase in activity as to power of multiplication and zymogenic action. Most of the alcohol was then produced in the first 24 hours of fermentation.

(3) Yeast cell counts conducted immediately after seeding and at optimal development, showed that the increase in cell concentration was no longer in the ratio 1:15 or 1:20; but that ratios of 1:50 and over were obtained.

Fig. 7 shows the results of two fermentations, one in which the yeast acted singly in the substrate and the other in mixed culture with Clostridium Saccharo Butyricum. Curve (1) represents conditions of cell concentration and alcohol formation in the mixed culture fermentation, while curve (2) represents conditions when the yeast acted singly. The time taken for fermentation is given in hours, as abscissae. The ordinates represent cell concentrations in millions per millilitre and grams of alcohol formed per 100 millilitres of fermented mash.

It will be noticed that in the first 36 hours of fermentation the yeast acting alone had produced only 0.264 grams alcohol per 100 ml mash, and the cellular concentration was about 3.2 millions per ml while in the same time the mixed culture fermentation had produced about 5.33 grams of alcohol, at a cell count of 64 millions per millilitre.

This most unexpected and curious phenomenon aroused our interest in searching for its cause. The possibility of mitogenic radiation from bacteria to yeast offered itself as an explanation, although other factors would also have to be considered such as production of growth substances that would have stimulating action upon the yeast culture that revealed itself in the form of the observed phenomenon.



We conducted a careful study of the metabolic products of the bacteria when working in pure culture in a substrate similar to that used for our rum experiments. It was found that apart from the gaseous products, carbon dioxide and hydrogen, the main product of fermentation was a mixture of fatty acids in which predominated normal butyric, acetic and propionic acids. The actual mixed acids content thus found consisted of 93 per cent normal butyric, 4.1 per cent acetic, 1.9 per cent propionic, and one per cent of a mixture of higher acids of the aliphatic series. None of these products, and especially that produced in greater quantity, may be regarded as enhancers of alcohol fermentation or activators of yeast growth and reproduction. However, as experimental confirmation, we studied the effect upon the yeast of these metabolic products *minus* the bacteria themselves.

To this effect, two sets of experiments were devised. In the first set we added to a series of yeast mashes, a different amount of a fermented bacterial mash in which the bacteria had been killed by heat treatment under pounds gauge pressure. In this way, the yeast culture could receive the benefits of the total metabolic products of the bacteria, but the influence of the live bacteria themselves was omitted. The results of this set of experiments indicated no favourable effect on yeast cell activation, on cell multiplication, or on the rate of alcohol production. In fact, adverse results were encountered as the percentage of added bacterial mash was increased, until in those cases where the organic acids concentration in the yeast culture was 0.5 per cent or more (calculated on the total weight of mash) fermentation was greatly retarded or did not take place at all. In table 8 will be found representative results obtained during these experiments.

In the second set of experiments both the effect of the living bacteria and that of the organic acids were eliminated. To the yeast mashes were added different amounts of the slops obtained after driving away by steam distillation practically all of the volatile fatty acids present in the fermented and sterilized butyric mash. By so doing we were allowing for any other products of the bacteria metabolism remaining in the steam distilled mash that could in any any manner stimulate yeast growth and multiplication. But as in the previous set of experiments, negative results were obtained.

These experiments showed that the activation of the yeast observed during our mixed yeast bacteria fermentations depended on the action of the living bacteria, and not on any of its products of metabolism. Two further experiments at intervals of eight days were then performed, using again the mixed culture technique developed during our heavy rum fermentation work. Due to the enlightening results obtained from these experiments, they will be described in some detail.

TABLE NO. 8

Initial Cell	Maximum				Percenta	-	ugars Fe Days	rmentec	1
Count Millions/ml	Cell Count	mash added, in Millilitres	Per cent on total yeast mash weight	1	2	3	4	5	6
		None	None						
5.0	100.0	8.0	0.05	7	30	60	85	100	100
5.0	95.0	16.0	0.10	7	28	56	83	100	100
5.0	88.0	24.0	0.15	6	21	39	78	95	100
5.0	72.0	48.0	0.30	4	15	36	70	87	95
5.0	40.0	64.0	0.40	2	9	20	35	41	50
5.0	17.0	80.0	0.50	1.5	2.5	6	10	15	20
5.0				No Fermentation					

SHOWING THE EFFECT OF INCREASING CONCENTRATIONS OF THE BACTERIAL METABOLIC PRODUCTS UPON THE CULTURE OF SCHIZOSACCHAROMYCES POMBE (LINDNER)

A molasses mash of 15.46 corrected Brix, 5.75 pH value, and total sugars content of 9.88 grams per 100 millilitres, was prepared in 6 one litre Erlenmeyer flasks, each flasks containing 400 ml mash. After thorough sterilization, these flasks were inoculated with 10% seed of a pure culture of Schizosaccharomyces Pombe (Lindner). One of these flasks was used as the control, and the other five were numbered in series 1 to 5 inclusive. The initial cell concentration was then determined. Flash (1) was immediately inoculated with two per cent bacterial seed; and the other four flasks from (2) to (5) inclusive were inoculated in succession, at intervals of seven hours, with the same proportion of bacterial seed. A total fermentation period covering 30 hours was allowed, counting from the time of yeast inoculation. When the 30 hour period was over, small samples were taken from each flash for pH determination, and the Brix readings were determined. Immediately after, the fermentation was stopped in all flasks by the addition of a predetermined amount of 20 per cent sulphuric acid. Determinations of final yeast cell concentration per millilitre mash; total alcohol by volume produced, residual sugars in grams per 100 millilitres mash; weight of dry yeast per 50 millilitres mash, were then performed on the fermented mash of each flask. The results are given in tables 9 and 10 and are graphically represented in figure 8 and 9.

TABLE NO. 9

	Mash Number									
Items Under Consideration	Control	1	2	3	4	5				
Hour and Date of Yeast Inoculation	Oct. 9 9 A.M.	Oct. 9 9 A.M.	Oct. 9 9 A.M.	Oct. 9 9 A.M.	Oct. 9 9 A.M.	Oct. 9 9 A.M.				
Hour and Date when Irradiation Started		Oct. 9 9 A.M.	Oct. 9 4 P.M.	Oct. 9 11 P.M.	Oct. 10 6 A.M.	Oct. 10 1 P.M.				
Initial Cell Count - Millions per c.c.	0.906	0.906	0.906	0.906	0.906	0.906				
Final Cell Count - Millions per c.c	15.625	92.188	150.000	218.750	42.188	20.313				
Final Brix Reading (Missing)										
Final pH value	4.83	4.85	4.82	4.75	4.70	4.75				
Total Alcohol Formed, c.c.	4.48	11.52	16.80	14.32	7.15	5.96				
Residual Sugars, grs. per 100 ml mash	6.64	3.78	1.06	2.36	5.66	6.34				
Weight Dry yeast, in 50 cc aliquot, grams	0.2519	0.4028	0.5009	0.4578	0.2733	0.2977				
Number of hours under bact. irradiation	0:00	30:00	23:00	16:00	9:00	2:00				

BEHAVIOR OF YEAST CULTURES UNDER MITOGENETIC IRRADIATION

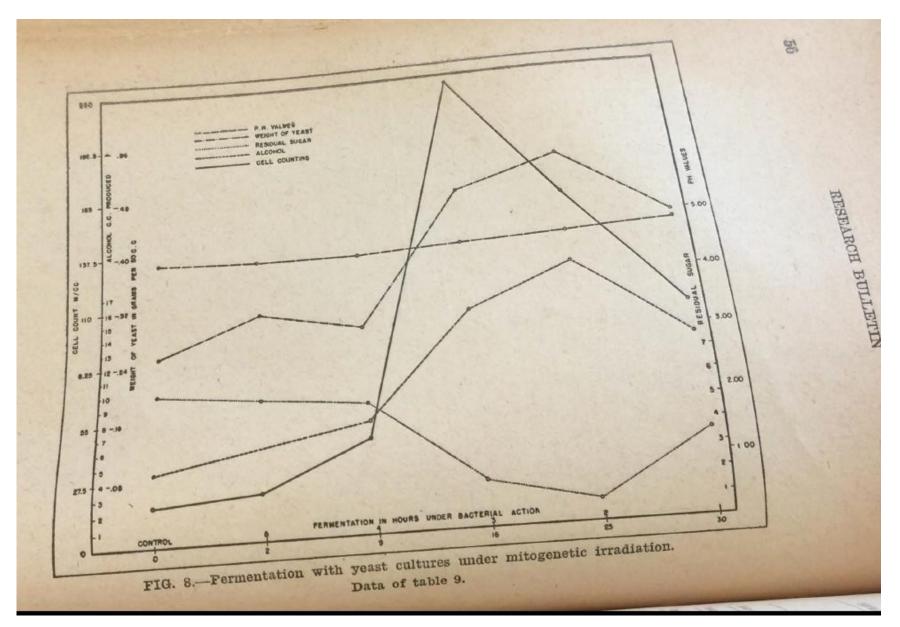
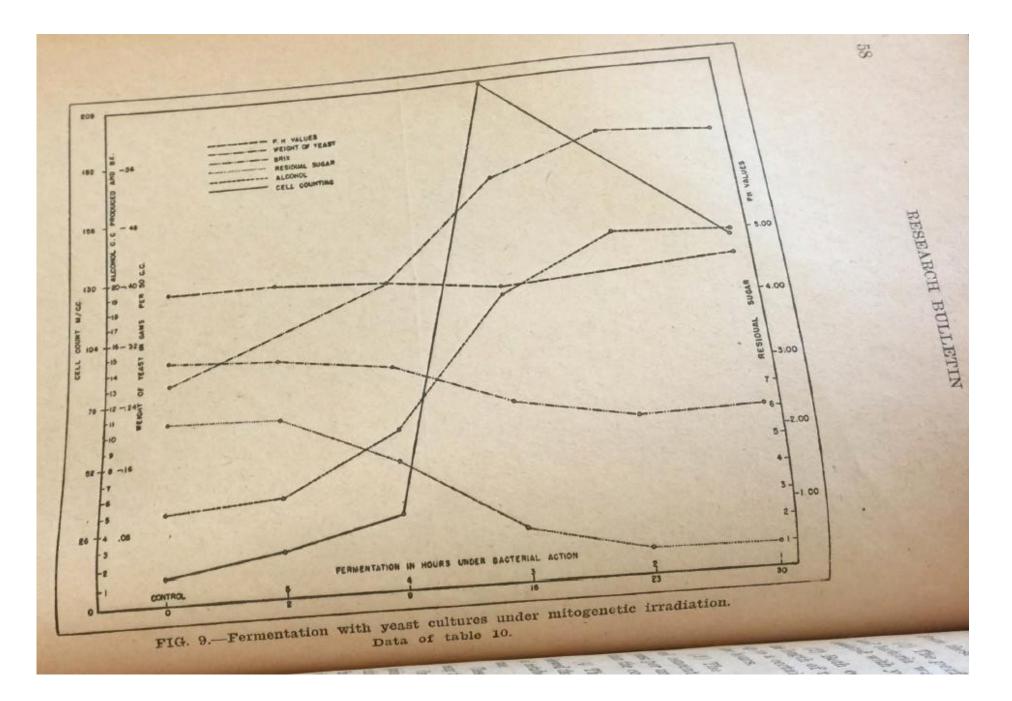


TABLE NO. 10

		Mash Number							
Items Under Consideration	Control	1	2	3	4	5			
Hour and Date of Yeast Inoculation	Oct. 17 9 A.M.	Oct. 17 9 A.M	Oct. 17 9 A.M.	Oct. 17 9 A.M	Oct. 17 9 A.M.	Oct. 17 9 A.M.			
Hour and Date when Irradiation Started		Oct. 17 9 A.M.	Oct. 17 4 P.M.	Oct. 17 1 P.M.	Oct. 18 6 A.M.	Oct. 18 1 P.M.			
Initial Cell Count - Millions per c.c.	1.143	1.143	1.143	1.143	1.143	1.143			
Final Cell Count - Millions per c.c.	9.375	126.563	165.625	206.250	28.125	15.625			
Final Brix Reading	14.47	9.26	9.06	10.36	13.17	14.17			
Final pH value	4.75	4.63	4.53	4.41	4.61	4.78			
Total Alcohol Formed, c.c.	5.01	20.06	20.46	16.94	8.89	5.41			
Residual Sugars, grs. per 100 c.c mash	6.96	1.04	0.98	1.80	4.83	6.25			
Weight Dry yeast in 50 c.c. Mash grams	0.2615	0.5407	0.5513	0.5071	0.3685	0.3106			
Number of hours under bact. irradiation	0:00	30:00	23:00	16:00	9:00	2:00			

BEHAVIOR OF YEAST CULTURES UNDER MITOGENETIC IRRADIATION



From these results it may be concluded that:

(1) The performance of those cultures inoculated with both yeast and bacteria was superior in every case to that of the checks, inoculated with yeast alone.

(2) Both cell multiplication and alcohol formation increased as the length of time of exposure to the effect of the bacteria increased, up to a certain limit, this limit lying between fourteen and twenty-one hours.

(3) The culture under mixed fermentation showed higher final cell concentration; more alcohol formation; more weight of dry yeast per unit volume; less residual sugars; and lower Brix densities than the controls.

(4) The practically constant pH values observed in all cases showed that there was no variation of any consequence in the amounts of metabolic products produced by the bacteria in every individual case.

These results made us quite suspicious of mitogenetic radiation being the real cause of the observed phenomenon.

One more step was felt as necessary in order to change our suspicion into certitude, and this consisted in irradiating the yeast cultures through the walls of a quartz tube, and noticing whether the phenomena observed during the mixed bacteria yeast fermentations would again become manifested. This was done in repeated experiments, over a long period of time, following the technique described below:

The equipment consisted of two glass cylinders of about 400 ml capacity, provided with tightly fitting glass covers. These covers had a central orifice whose diameter was just large enough to allow the insertion of a quartz tube one and five-sixteenth inches in diameter. The cover had also a small glass tube attached to allow for the escape of gases from fermentation taking place within the cylinders during the experiments.

The quartz tubes were surrounded by a quartz washer at a distance of about one inch from their mouths; so that when the tube was inserted into the cylinder, this projecting washer impeded its reaching clear through to the bottom of the cylinder. In this way, after insertion, the ends of the quartz tubes were about two inches over the bottom of the cylinders.

After the two sets of apparatus were assembled, the gas exit tubes and the mouths of the quartz tubes were plugged with non-absorbent cotton and the whole equipment sterilized at 20 pounds gauge pressure during one half hour in an autoclave. Then, working aseptically within a sterile Hansen box, about 250 millilitres of a sterile sugar cane juice mash were poured into each of the two glass cylinders, and one of them was inoculated with a pure culture of Clostridium Saccharo Butyricum. When fermentation was in full vigour (usually after 24 hours) a culture consisting of a recently inoculated defecated cane juice using Schizosaccharomyces Pombe (Lindner) was introduced into each one of the quartz tube. An initial cell count was obtained of this yeas mash previous to its introduction into the quartz tubes. Through this manipulation and technique we had a culture of the yeast within a quartz tube suspended and surrounded by a fermenting culture of the bacteria in the cylinder. As check we had a similar culture of yeast suspended and surrounded by a sterile mash of defecated and clarified cane juice. That is, one of the yeast cultures was submitted to the bacterial radiations through a wall of quartz, while the check received no irradiation, being immersed into a sterile medium.

The whole setup was then incubated at 32 degrees Centigrade during variable periods of time at the end of which cell counts were effected on the irradiated culture and the check. Also losses in weight of both yeast cultures were recorded as a measure of the rate of sugar destruction.

This method was later modified in two manners: (1) the Clostridium Saccharo Butyricum was substituted by Bacillus Tetryl (19) a butyl alcohol and acetone producing bacteria also found and isolated by the writer. In both cases, the phenomenon of accelerated cell multiplication became apparent in the case of the irradiated cultures.

Lastly the quartz tube was substituted by a similar one made of ordinary glass, and then the effect of cell activation did not appear.

In table 11 are found some of the results obtained during these experiments of irradiation through a quartz wall.

Similar results were obtained when the Clostridium Saccharo Butyricum was substituted by the butyl organism, B. Tetryl.

The accumulation of experimental evidence, given here in part only, seems in our opinion, to indicate that the phenomenon encountered in our mixed yeastbacteria culture was due to mitogenetic radiation. Our experiments appear to confirm Gurwitsch's theory. TABLE NO. 11

TO YEAST THROUGH A THIN WALL OF QUARTZ						
Irradiation Time Hours	Cell-Counts per ml N		Mash Weight Losses Grams			
	Irradiated Culture	Check	Irradiated Culture	Check		
0.00 3.00 6.00 12.00 18.00 0.00 15.00	1.30 5.00 18.00 31.25 47.75 2.35 63.60	$ \begin{array}{r} 1.30 \\ 3.00 \\ 11.00 \\ 13.15 \\ 16.85 \\ 2.35 \\ 16.05 \\ \end{array} $	$\begin{array}{c} 0.00\\ 0.80\\ 3.80\\ 12.10\\ 20.80\\ 0.00\\ 10.10 \end{array}$	$\begin{array}{c} 0.00\\ 0.50\\ 2.10\\ 7.70\\ 14.00\\ 0.00\\ 4.10\end{array}$		
24.00 0.00 5.00	105.00 0.12 3.00	37.50 0.12 0.90	16.80 0.00 0.70	9.10 0.00 0.40		
14.00	26.25 90.25 119.80	16.50 36.75 49.95	4.30 12.50 16.20	2.60 5.50 9.80		

SHOWING THE EFFECTS OF MITOGENETIC IRRADIATION FROM BACTERIA TO YEAST THROUGH A THIN WALL OF QUARTZ

V

Rum Fermentation

We believe this to be the most important stage in the process of rum manufacture, and have, therefore, devoted more of our time to this particular phase of the research.

The fermentation method followed at the rum distillery should be determined principally by the characteristics desired in the end product. It then follows that those used when industrial alcohol is the product sought for.

A look at the comparative specifications required for successful practice in the production of rum and industrial alcohol will show at a glance that the aims are altogether different and even antagonistic in some respects; the main resemblance in the two processes being that ethanol is the principal product produced in both cases.

We find that the ideal fermentation for industrial alcohol will require that the largest possible amount of the purest possible ethyl alcohol be formed in the least possible time; while in the ideal rum fermentation, yield of alcohol and rapidity of fermentation must be sacrificed sometimes in behalf of quality, and excellence of taste and aroma in the finished product. But this quality factor depends almost entirely on the quantity and quality of the congeneric products of rum fermentation; the *quality* of these congeners being far more important than the quantity.

The following comparative scheme will bring out these points of difference between industrial alcohol and rum manufacture, as regards fermentation objectives:

Industrial Alcohol Production	Rum Production
(1) A highly efficient yeast capable of utilizing practically all of the sugars in the mash into alcohol.	(1) A fairly efficient yeast as regards sugars utilization and alcohol formation.
(2) The shortest possible fermentation period.	(2) A variable lapse in time of fermentation.
(3) Maximum production of alcohol.	(3) Comparatively large yields of ethyl alcohol.
(4) Least production of congeneric products such as aldehydes, acids, esters, higher alcohols, essential	(4) Adequate production of the right sort of congeneric products especially rum-oil.
(5) A practically standard fermentation technique used.	(5) Variable fermentation technique, according to characteristics sought for in the finished product.

REQUIREMENTS

At the beginning of our consulting work which was carried parallel to these rum studies, we found that in actual practice these points of difference between alcoholic and rum fermentation were being utterly disregarded. Attention was being paid almost exclusively to making industrial alcohol and then transforming it by many obscure and dubious means into a beverage to which the name of rum was given. It became our task to prove that such practices were not conducive to real success in rum making, and to obtain the introduction of sounder methods of fermentation into the industry. The first part was accomplished through our studies and experimental work, and the second through our advices and demonstrations as technical consultant.

According to present trade customs, rums are divided into light and heavy types or classes, the former being subdivided into white and gold according to the color tone at which the bottled rums are marketed. As the fermentation methods that should be followed in the production of each type are, or should be, quite different; we shall treat each class separately. But before doing so it would be well to discuss the subject of rum fermentation in tits broader aspects.

1. Building up the Seed Yeast

The proper building of the seed yeast that will later be used as footing for the fermenters is one of the most important steps of rum fermentation, and unfortunately, one of the most carelessly conducted in most cases.

The seed yeast must be composed of young, active, healthy cells in pure culture. Their number must also be in adequate proportion to the work expected from them during fermentation. It is well known that initiation, and subsequent development of the fermentation process is influenced directly by the number of active cells per unit volume in the mash at pitching time; and therefore, in the cell concentration of the yeast footing utilized for seeding. To illustrate how this initial yeast cell concentration influences the rapidity at which fermentation will be finished, we are giving below a simple experiment:

Five 100 millilitres portions of a sterile molasses mash, containing 5.0 grams of total sugars each, were inoculated with variable quantities of the same pure culture of yeast. The time taken for fermentation to finish in each case was recorded. Below are found the results obtained:

	Number of hours required for
Grams yeast in Seed	fermentation at 30 degrees C.
8.0	
4.0	
2.0	
1.0	
0.5	180.0

This simple experiment proves that under controlled conditions, time of fermentation is an inverse function of yeast cell concentration. The initial yeast cell concentration will not only affect the duration of fermentation, but also the yield and quality of the resulting rum. For purposes of high yields, that yeast cell concentration should be used for seeding as will require no further yeast multiplication and growth to effect the conversion of sugars in the substrate into end products. This is not easy to be obtained in actual distillery practice unless special enriching methods are applied in building the yeast footing. The highly reputed process of Boinot-Melle (2) and also that developed by Hildebrandt F.M. And Erb. Norris M. (2) for the United States Industrial Chemicals Corporation, are excellent examples of successful efforts in obtaining this optimum development in cell concentration for the initiation of the final fermentation. Thus high initial cellular concentration shortens the period of fermentation and insures sufficient yeast cell to complete the fermentation of the sugars present in the mash, without any further cell development becoming necessary and, therefore, without any diversion of carbohydrates to the

development yeast cells. In this way, alcohol yields are greatly enhanced, insuring more economical production.

If industrial alcohol distilleries will always be benefited by the adoption of such fermentation practices, the case is a little different for rum distilleries. In certain cases it is not advisable to have a very rapid fermentation rate at the rum distillery, on account of the characteristics sought and required in the end product. Also the need for refrigeration of the fermenting mash in order to lower the high heat produced by a very rapid fermentation becomes often a restricting factor in the tropics.

The most important factor, however, that should eight in the mind of the rum distiller when inclined to use these enriching processes of yeast seed building, is that of the final quality his finished rum will possess in accordance to the type he wishes to produce. If producing a very light class of rum, he may adopt these procedures with beneficial results provided his distillery is equipped or may be easily equipped for the purpose.

But without going into extremely high cell concentrations, when building the seed yeast, it is always necessary to obtain sufficient concentration. The degree of concentration required will vary, however, with the conditions in which fermentation will take place. Let us discuss this point with some detail, as our consulting practice has shown that at least 75 per cent of the troubles encountered in the fermenting end of the rum distillery are related to lack of comprehension of the paramount role of proper seeding methods.

In the chapter on yeast selection we stated that the selected yeast seed might be employed in one of three ways, as inoculum of: (a) sterilized mash in closed, aseptic fermenters, (b) partially sterilized mash, that is, one free of vegetative forms of infecting organisms; (c) non-sterilized fresh mash, more or less infected.

Now then, the cell concentration in the yeast footing and the total number of cells used per unit volume of inoculated mash, may vary in accordance to the one of the three ways stated above in which the fermentation shall take place. If conditions are such that method (a) is followed, the cell concentration used may be varied at will without risk, and, therefore, the fermentation lapse will be under absolute control of the distiller. By using a very small cell concentration, for instance one half million cells per millilitre of mash, he can prolong the period of fermentation, thus imparting certain characteristics to his end product, or raw rum. By using a very high concentration, for instance, 150 millions cells per millilitre mash, he will insure a

very rapid fermentation and obtain different characteristics in his raw distillate. The great flexibility of operation under method (a) of procedure is clear and apparent.

If method (b) must be followed, flexibility of operation will not be much curtailed provided a fairly fast fermenting, vigorously propagating yeast, is used. The important point in this case is to assure complete control of the medium to the pitched yeast by the time the organisms that may be present in spore form are able to pass into the vegetative stage. When using carefully selected yeast, this may be easily accomplished. But if a very slow fermenting yeast must be used, then it becomes imperative to use the highest possible yeast cell concentration at pitching time.

With method (c) which we found of general use on this Island oat the beginning of our consulting practice, the distiller must exercise much greater care and control in the development of the appropriate amount of yeast seed, for he is to pitch his seed into an uncontrolled medium which may be highly infected. With this method all sense of certainty as to what may develop during fermentation, is lost; the only defence at the moment lies in having ready for inoculation a powerful and highly concentrated pure yeast footing. Flexibility in the use of the inoculum is practically lost, and so is the control over the period of time in which fermentation shall take place. Hence, the control over the quality and especially over the uniformity in chemical composition of the resulting rum is very weak, if any. The statements given above on the influence of amount of yeast footing used at pitching time, and that of the condition of the receiving mashes at the time of inoculation, on the quality and composition of the resulting raw rums, have been proved experimentally by us.

Table 12 data obtained in a series of fermentations conducted with the idea of testing the effect of varying the amount of seed yeast used per unit volume of mash, when fermentations were conducted under conditions described at (a) above; that is, when the yeast was pitched into a closed, aseptic fermenter containing an absolutely sterile molasses mash. Time taken for fermentation, alcoholic yields, analyses of the Non-Alcohol-Number, and organoleptic characteristics of the resulting rums were the variables under consideration.

It will be noticed that as the initial cell concentration increases, the hours for completion of fermentation decrease, until at 32 times more initial concentration, only one eighth of the time is necessary. The respective alcoholic yields do not offer such great variations (as is to be expected); but the higher yields are obtained during the shorter fermentation periods. The Non-Alcohol-Number in the respective tests show a decrease in quantity as the fermentation time shortens, becoming one half as large for the 12 hours period than for the 96 hour period.

Taste and aroma remain good in all tests; but become less perceptible and less complex in nature for the rums produced during the shortest periods of fermentation. General deductions from these tests would indicate that as the initial cellular concentration is increased, fermentation time decreases, alcoholic yields increase, and volume of aroma and tests decreases. The longer the period of fermentation the larger the Non-Alcohol-Number becomes.

If in the above experiment an unsterilized, fresh molasses mash is used, the results obtained are quite different as regards alcoholic yields, but they will not differ in general trend as regards rapidity of fermentation and volume of aroma and taste; with the exception that the differences are of greater magnitude. Also, should the contaminating microorganisms belongs in part to sulphur or to putrefactive bacteria then the resulting raw rum will be fouled in aroma and taste by the products of the contaminants.

The purity of the yeast footing used as inoculum for the fermenter is more important yet than its cellular concentration. Keeping his seed yeast as pure as possible at least until the time when it finally serves as inoculum of the fermenter should be the constant precaution and endeavour of the distiller. The difficulty in obtaining this result is one of the greatest drawbacks of all enriching processes for the seed yeast. It is then very important to know about the most reliable manners in which to develop the yeast footing to an optimum cellular concentration with the risk of running into perilous contaminations.

The idea of using a yeasting machine seems at once to offer the most obvious solution of the problem. Some of these machines, of which the Magné and the Pfaudler, among others, are good representatives, serve their purpose quite satisfactorily if the rum distillery has the adequate technical personnel to direct and supervise the management of such apparatus; otherwise they become themselves a source of infection, all the more perilous on account of being the place of least suspicion.

The method we have always followed and recommended to our clientele consists in using a fresh yeast footing as the start of each day's work. This footing is built up in the distillery laboratory up to that size suitable as inoculum for the first yeast propagating vessel of the distillery. Supposing that the inoculum required for the

TABLE NO. 12

SHOWING THE EFFECT OF VARYING THE INITIAL CELLULAR CONCENTRATION IN MOLASSES FERMENTATION UNDER PURE CULTURE FERMENTATION TECHNIQUE, AS REGARDS DURATION OF FERMENTATION, ALCOHOLIC YIELDS, CHARACTER OF THE NON-ALCOHOL-NUMBER AND ORGANOLEPTIC CHARACTERISTICS OF THE RESULTING RUMS

Initial Cellular Concentration Millions Millilitre	Duration of	Alcoholic Yield	Resultin	g Rum Analysis Milligrams 10	of Non-Alcoho 0 c.c. Abs. Alc.	Resulting Rum Organoleptic Tests		
	Fermentation Hours	on Total Sugars %	Total Acids	Aldehydes	Esters	Higher Alcohols	Aroma	Taste
1.00	96.00 46.57		9.10	11.20	17.50	22.00	Very Good Complex	Very Good
2.00	88.00	46.89	8.90	10.90	17.00	21.10	Very Good Complex	Very Good
4.00	49.00 47.00		7.60	10.00	14.70	16.90	Very Good Complex	Very Good
8.00	36.00	47.77	6.40	8.80	11.40	15.50	Very Good Simple Tone	Very Good
16.00	18.00	48.10	5.90	6.70	9.80	9.80 9.80		Very Good Less perceptible
32.00	12.00	48.72	4.20	5.10	6.90	5.00	Very Delicate Very Good Lower Tone	Still less perceptible but fine

first vessel of the yeast propagating system be five gallons, then the method to be followed is as follows:

From the stock culture, agar slants are inoculated to serve as the starting working cultures. Seven of these agar slants are inoculated at once from the stock culture. These solid slant cultures are kept in a cold incubator at a temperature between 12 and 15 degrees Centigrade; and they should be renewed by a new set of similar cultures every two months.

A portion of the pure slant culture is transferred to 50 ml of sterile molasses mash contained in a 100 ml Erlenmeyer flask; by known methods of bacteriological technique. When this mash has reached vigorous fermentation, it is used as inoculum of a litre of a similar mash in a two litres Erlenmeyer flask; and when again vigorous fermentation is observed, this litre is used as inoculum of a five gallon can. The five gallon can is provided with a sampling device by which small samples of the fermenting medium may be drawn for determination of yeast cell concentration. When that yeast cell concentration, which practice has shown to be the optimum, is reached, this final laboratory seed is taken into the plant to serve as inoculum of the first vessel of the distillery yeasting equipment.

A new slant serves as the origin of this laboratory footing each day for in this manner every slant is re-used only once a week and should it become contaminated during one of the transfers, it lies idle long enough for the development of the contaminant before its new turn for use arrives. This is a safeguard towards the introduction of infection in the distillery via the laboratory footing. A sterile Hansen box should be used during the inoculations from one medium into another.

This method of starting a fresh footing every day at the distillery is much more laborious than using for instance a yeasting machine where one initial laboratory footing may be kept propagating for months; but as before stated, it is much the safer methods. Now, the yeasting machine may be used in the above method of propagation, serving as the first vessel of the distillery yeasting section.

It has been found that practically any amount of yeast cell concentration may be obtained for the final fermenter seed in the method described above, through a judicious feeding of the propagating yeast. In this connection we have found that the use of liquid ammonium hydroxide operates as a very efficient stimulant of yeast growth and propagation. The ammonium hydroxide should be used intermittently during the period of propagation as a solution containing ten per cent ammonia. In this manner derangement of the optimum pH value of the culture is obviated. Liquid ammonia has been found far superior to ammoniacal salts such as the sulphate or tartrate for this purpose. Table 13 offers representative results of experiments conducted in the laboratory on this matter of activating the yeast propagation through liquid ammonia feeding.

TABLE NO. 13

	Cell-C	ounts				
	Millions/	'Milliliter	pH Values			
Hours of Propagation	Ammonium Sulphate Fed Culture	Ammonium Hydroxide Fed Culture	Ammonium Sulphate Fed Culture	Ammonium Hydroxide Fed Culture		
0.00	5.00 [initial seed]	5.00 [initial seed]	5.15	5.15		
6.00	15.00	33.00				
9.00	30.00	75.00				
12.00	65.00	190.00	4.80	5.00		
15.00	120.00	286.00				
18.00	145.00	370.00				
21.00	160.00	430.00	4.65	4.90		

EFFECT OF AMMONIUM HYDROXIDE VS. AMMONIUM SULPHATE FEEDING OF YEAST, DURING PROPAGATION EXPERIMENT

In the experiment whose results are represented in table 13, the ammonium sulphate fed culture received at once four grams of this salt per liter of mash, equivalent to about one gram of pure ammonia. The ammonium hydroxide fed culture for reasons specified above, received this substance at periodical intervals during the test. The total amount of pure ammonium hydroxide fed was one millilitre per litre of mash, or one tenth of one per cent by volume. As already stated this ammonia water was fed in the form of a 10 per cent ammonia solution. This was equivalent to about 0.25 grams pure ammonia per litre of mash.

The results show that liquid ammonia feeding of the yeast during the development of the footing will bring about a very satisfactory yeast cell concentration in a short time. In fact during the first 12 hours of propagation, the liquid ammonia fed culture had developed a stronger cell concentration than that developed in 21 hours by the ammonium sulphate fed culture. Another advantage of

liquid ammonia feeding is the economy effected in terms of pure ammonia used in each case, the liquid ammonia fed culture using only one fourth of the amount required by the ammonium sulphate fed culture. In terms of millions of yeast cells produced per unit weight of pure ammonia, we have that liquid ammonia is about 12 times more efficient than ammonium sulphate. But this is not all; in the case of liquid ammonia feeding we obtain the additional advantage of keeping the optimal conditions of pH practically constant for a given strain. As we proceed with this chapter, additional important results of liquid ammonia feeding during the actual main fermentation in rum making will be presented.

The proper precautions and proper methods to be used in the development of the seed yeast up to the time of its introduction into the first vessel of the yeast propagating system of the distillery, have been discussed at some length. Before this pure laboratory culture reaches the final fermenter, further propagation is necessary at the plant itself.

When the distillery has a yeast propagating machine like the Magné or Pfaudler, the further propagation is greatly simplified as these machines are specially constructed of adequate size to suit the fermentation capacity of given distillery. In such cases these machines may become the recipients of the laboratory-built inoculum, protecting it from contamination and offering all facilities for its further propagation till it reaches its final destination as inoculum of a fermenter or of various fermenters. Otherwise, a series of closed vessels, which are supposably kept under aseptic conditions as practicable, are used for the further propagation of the laboratory footing. Usually from initial culture at the plant to final fermenter, three transfers are required for small and medium-sized distilleries; and four, or even five in the case of very large installations.

It has been the custom to used a five per cent by volume footing as inoculum for the final fermenter. This custom should not be made law; especially in rum fermentation. Unless built under the care of experienced hands, a five per cent by volume footing has no definite meaning. In the first place we have already stated that methods of industrial alcohol fermentation can not blindly be implanted in rum production. A five per cent by volume footing has been used almost generally in alcohol fermentation; but for rum fermentation special considerations exist that will vary the amount of footing to be used; for instance, the influence of percentage footing on the duration of fermentation, and final characteristics of the resulting rum, among others. Again, the initial cell concentration at pitching time is more dependent on the degree of cell concentration of the seed than on the volume used. For instance, a 1 gallon footing with a cell concentration of say 50 million cells per milliliter will be as effective as a 50 gallon footing with a concentration of only one million cell per milliliter. It is better to depend on cell counting than on mere volume of seed.

When conditions for the development of an efficient and sufficient footing are adverse, the distiller has the following recourse: Suppose that conditions are such that even with optimum cell development, he would need a 10 per cent footing for his fermenters but can dispose only of 3.33 per cent. In such case he should first of all deliver his 3.33 per cent footing into the empty fermenter and then fill up with mash till the fermenter is one tenth full. Further filling is then stopped until the inoculated mash in the fermenter shows signs of vigorous fermentation. The fermenter is then continued to be filled with fresh mash to full capacity. In this manner, the needed 10.0 per cent footing has been built in the fermenter itself. As the first portion of inoculated mash really received a 33.33 per cent footing, a very few hours will only be needed before the new footing is ready to receive the rest of the mash. As a matter of fact no time is actually lost with this method, for the filling of the fermenter to full capacity at once upon a 3.33 per cent footing will retard the fermentation much more than the former procedure; with the added disadvantage that a successful fermentation may be altogether prevented on account of the very small initial cell concentration encountered when operating under such conditions.

2. Fermentation Temperature

This most important factor of rum fermentation has not received the careful attention and consideration it deserves in commercial rum making.

It may be stated that an excellent bouquet and aroma are developed in the fermenting rum mashes by a long, gradual fermentation at low temperatures rather than by a short, quick fermentation at higher temperatures. In cool fermentation the rum yeasts produce more esters and other aromatic substances. Reactions unfavorable to the quality are caused, moreover, at high temperatures. A rum fermented at 27 to 30 degrees Centigrade will be mellower, fresher, and with a more desirable bouquet than one fermented at 35 to 40 degrees, as is usual in commercial establishments through ignorance or carelessness. When not operating under aseptic conditions, excessively high temperatures will encourage the growth and propagation of undesirable bacteria, detrimental to yields and quality; and that in extreme cases may bring into the fermenting medium the phenomenon of stickiness" or paralysis of the yeast's activities while considerable amounts of sugars are still present in the beer.

The heat generated during fermentation is very considerable and will naturally tend to cause a rise in temperature of the fermenting mash. According to Rahn (22) the heat evolved from a gram mole of dextrose in the process of conversion to alcohol is 26.0 kilogram-calories; other calculations having reported values varying from 22 to 33. The measurement of Boufford (23) of 23.4 to 23.7 Calories per 180 grams of sugar fermented has been accepted by the majority as a correct figure.

The temperature at which the rum mash will rise will be determined by its initial temperature at pitching time, plus the rise in temperature due to the heat generated by the fermentation reaction, and minus the heat lost during fermentation by radiation and conduction, or through artificial cooling. The higher the temperature of the mash at the moment of inoculation and the higher the total sugars concentration in grams per 100 ml mash the higher the temperature will rise.

In the wine industry, the amount of cooling necessary for the fermenting must have been calculated by Bioletti (²⁴) in the following manner:

Let S = Balling degree of must (approximate sugar content) T = Temperature of contents of vat M = Maximum temperature desired C = Number of degrees F. necessary to remove by coolingThen, $C= 1.17^{\circ} (S + T - M)$

Yeast will cause the alcohol fermentation of the sugars in the mash within an ample temperature range; zero degree Centigrade being considered the lowest, and 50 degrees above zero the highest at which fermentation is possible. But in the practice of rum fermentation this wide range narrows considerably, and although varying with different yeast strains, the generally accepted range is between 25 and 37 degrees C. Within this range, the higher the temperature the stronger and more violent fermentation becomes; so that it may be said that increase of temperature accelerates the rate of fermentation. The formation of grease and glycogen in the

cell is also effected more rapidly as the temperature increases. It is also well-known that yeast is inhibited during fermentation by the formation of alcohol in the fermenting medium, and we find that this inhibitory action is not the same for a given alcoholic concentration at all temperatures; but the inhibitory effect increases directly as the temperature of the mash increases. For example, a given yeast may begin to feel the inhibitory action of its own metabolic products at an alcoholic concentration of 6.0 per cent by volume when the temperature of the substrate is around 35 degrees C., while this same yeast will stand up to 9.0 per cent alcohol by volume without becoming much affected by this alcoholic concentration provided the temperature of the substrate be kept uniformly at about 27 degrees Centigrade.

Also from our previous statement as to the fact that high temperatures accelerate the rate of fermentation, it should not be inferred by this that higher yields of rum on sugars used are to be obtained. On the contrary, the actual final yields will always be lower when fermentation is conducted at very high temperature, leaving an abundance of unused sugars in the mash. Due to high temperature, the alcohol becomes more toxic towards the yeast, and fermentation will be arrested when comparatively low alcoholic concentrations are formed within the fermenting liquid, and long before the exhaustion of the sugars in the medium has taken place. Hence, high temperatures of fermentation are opposed to the obtention of high yields of rum.

But the evil results of high temperature do not stop here; they will also influence adversely the quality of the rum. High fermentation temperatures operate very effectively in producing endoproteolysis and autolysis of the yeast cell. When this happens, a great many products of decomposition are formed, many of which pass over later on during distillation to cause foul odor and bad taste in the end product. Apart from these adverse influences, high fermentation temperatures bring about exhaustion of the yeast cell prematurely, through vital over-excitement.

The adoption in industrial practice of a given temperature of fermentation in the distillery depends upon many factors, among which we may mention:

- (a) Yeast strain used.
- (b) Chemical composition of the raw material.
- (c) Available fermentation capacity.
- (d) Initial total sugars concentration carried at mashing.
- (e) Degree of asepsia available in mashing and fermenting.
- (f) Amount of seed yeast used as footing for the fermenter.

Some of these factors are interrelated as will appear during further discussion:

Some rum yeast strains will naturally resist adverse conditions regarding temperature of fermentation better than others. We have dealt with strains that would succeed in carrying rum fermentation at temperatures between 33.0 and 37.0 degrees Centigrade, while other strains became inhibited at temperatures between 30.0 and 33.0 degrees. It is up to the distiller then to use that strain most suited to the local conditions of his establishment, or to change the conditions to accommodate the particular strain he needs or wants to use. Table 14 presents data regarding fermentation results obtained from two different strains: a Schizosaccharomyces very resistant to high temperatures, and a budding yeast very susceptible to the evil effect of high temperature.

The following points of the data given are worthy of consideration: (a) While fermentation is more rapid in both cases at the higher temperatures, in the case of Strain No. 2 the shortening of the fermentation period coincides with low yields and high residual sugars; while in the case of Strain No. 1 the yields and residual sugars are not affected to such remarkable degrees.

(b) The fermentation efficiency variation between highest and lowest values is only 4.12 per cent in the case of Strain No. 1, while for Strain No. 2 a difference of 18.53 per cent is noticed.

(c) The amounts of higher alcohols produced per 100 ml of absolute alcohol are lower for the Schizosaccharomyces than for the budding yeast.

(d) The percentages of higher alcohol on total Non-Alcohol-Number are also of lower values in the case of the Schizo variety.

(e) Optimum overall conditions of fermentation seemed to take place at 32.0 degrees Centigrade for Strain No. 1 (the Schizosaccharomyces) and at 28.0 degrees Centigrade for Strain No. 2 (the budding yeast)

The chemical composition of the raw material will also influence the choice of fermentation temperatures. For equal initial sugars concentration at setting time higher temperatures of fermentation may be used to lower the non-sugars content of the raw material. The initial total sugars concentration to be had at setting, influences of the fermentation period in that the higher the initial sugars concentration the higher the fermentation temperature to be expected. For this

TABLE NO. 14

SHOWING EFFECT OF RISING TEMPERATURE OF FERMENTATION ON TWO YEAST STRAINS, ONE RESISTANT AND THE OTHER NON-RESISTANT TO HIGH TEMPERATURES OF FERMENTATION, DESIGNATED RESPECTIVELY AS STRAINS Nos. 1 AND 2

Strains	Ferm.	Duration of	Yield Alcohol on	Residual Sugars,	Fermenta- tion	Higher Alcohols	Total Non-Alcohol	Per Cent Higher Alcohols on	Organoleptic Tests on Raw Rum Produced	
No.	Temperature Deg. C.	Fermenta- tion Hours	T. Sugars Per Cent	grams 100 ml Mash	•		No. mgs. 100 cc Abs. Alcohol	Total Non-Alcohol No.	Aroma	Taste
1	28	96.0	46.5	0.70	95.77	40.0	258.0	15.5	Very Good	Very Good
	30	77.0	46.0	0.70	94.74	55.5	304.9	18.2	Very Good	Very Good
	32	60.0	46.7	0.68	96.18	59.8	284.7	21.0	Very Good	Very Good
	34	52.0	45.0	0.85	92.68	76.0	262.9	28.9	Good	Good
	36	56.5	44.5	1.00	91.65	91.7	245.2	37.4	Good	Good
2	28	72.5	47.2	0.55	97.21	60.6	284.5	21.3	Excellent	Excellent
	30	55.0	46.5	0.70	95.77	77.2	257.3	30.0	Very Good	Very Good
	32	60.0	43.9	1.20	90.42	111.4	242.7	45.9	Good	Good
	34	55.5	39.8	2.10	81.97	137.9	266.2	51.8	Fair	Disagreeable
	36	48.0	38.2	2.70	78.68	155.1	282.0	55.0	Fair	Disagreeable

* Fermentation Efficiencies calculated according to Pasteur's Equation.

reason it has been found in actual distillery practice in tropical regions that artificial cooling of the mash in the fermenters must be resorted to if high initial sugars concentrations are desired at mashing. The writer has found in his consulting practice that with wooden fermenters, artificial cooling must be used even at low total sugars concentrations in the mash; for instance, with concentrations above 9.0 grams of total sugars per 100 ml mash. If iron, or steel open fermenters are used, then slightly higher sugars concentrations may be carried; and artificial cooling becomes necessary for concentrations above 11.0 grams total sugars per 100 ml mash. Of course, the site of the distillery has a great deal to do in this matter of artificial cooling, those distilleries situated in the mountainous regions being able to dispense with artificial cooling with less risk than those situated on the coastal region.

Choice of fermentation temperature will also depend on the individual size of the fermenters and the available total fermentation capacity of a given distillery. Where fermenters are of small capacity (those from 2,000 to 5,000 gallons), a greater amount of heat will be radiated into the surrounding atmosphere, and hence, the initial fermentation temperature may be higher than when dealing with large fermenters (capacities between 10,000 and 25,000 gallons). The smaller fermenters will also be much easier to cool down in case artificial cooling must be employed. Hence, higher temperatures of fermentation may be carried in small than in large fermenters.

As to the total fermentation capacity of a given distillery we have cases in which this is so small for the rum production required, that high temperatures of fermentation must be chosen to prevent stoppages in the distillation end of the distillery for lack of available fermented mash to feed the still. In such cases it will pay the distiller to increase at once his fermentation capacity; and if this is not possible (which is the case in these times of war) then he must secure a yeast strain adaptable to rather high temperatures of fermentation, and that is at the same time a fast fermenting organism. With adequate capacity in the fermentation end of his distillery the distiller should choose the lowest fermentation temperature possible without incurring in economic loss.

When operating under aseptic conditions there exists less danger in carrying fermentation at relatively high temperatures; but when dealing with infected substrates, as is usually the case, high temperatures of fermentation may lead to disastrous results. Bacterial contamination is the most common source of infection in the production of rum, and as most bacteria are even more responsive to high temperature conditions than the yeast, it commonly happens that the bacteria in the medium will grow and propagate faster than the yeast and finally overcome the latter, with a resulting failure of the rum fermentation. It is then specially important to keep from excessively high temperature of the fermenting mash when dealing with conditions lacking in asepsia during the fermentation period.

The larger the number of yeast cells present per unit volume in the footing upon which the main fermentation is to be built, and the larger this footing, the quicker will the temperature rise in the fermenting medium and the higher will be the temperature peak developed. The fact that when dealing with non-aseptic mashes a large volume and high cell concentration must be used as footing at pitching time, makes still further difficult the keeping of the proper fermentation temperature in the fermenter. In such cases the existence of means for effecting artificial cooling of the mash is especially important.

3. Initial Sugars Concentration

Of at least equal importance to temperature of fermentation is the subject of initial sugars concentration in rum fermentation. During fermentation, the yeast breaks up the sugar molecule to form ethyl alcohol, carbon dioxide gas and other products of its metabolism. Within certain limitations it could well be said that the concentration of total sugars in the mash determines the alcoholic concentration that will later appear in the beer. Were not the yeast itself affected adversely by this rising alcoholic concentration in the fermenting medium, it could be said that the quantity of alcohol to be expected from a given fermentation would be a direct function of the sugars content in the mash. But the fact is that not only are all yeasts adversely affected by the alcohol built by themselves, but that different races or varieties are affected in different degrees. This phenomenon is also existent in rum yeasts, some strains being more resistant than others to the action of the products of their own metabolism. In general, the most favorable strains as far as production of fine taste and bouquet in the resulting rum, are the least resistant to the deleterious effects of their products of metabolism. The principal cause for this behavior is founded in the character and quantity of the metabolic products other than the ethyl alcohol. Among these, we find higher alcohols, esters, aldehydes, and other congenerics, that produce more toxic effects on the yeast than ethyl alcohol itself; or that tend to increase the toxicity of the latter when in mixtures thereof. We have proved in the course of these studies that as the concentration of these congenerics of alcoholic fermentation increases, the less alcoholic concentration will the yeast be able to resist.

amounts of fermentable sugars remain in the fermenting medium.

Table 15 presents data showing fermentation results obtained with various yeast strains under variable conditions of the initial total sugars concentration, and the optimum sugars concentration for each of the strains used in this test. We must state, however, that these tests were performed under optimal conditions of fermentation temperature, pH yeast nutriments, etc., and that most probably the optimum values for initial total sugars concentrations obtained in the experiment will not be duplicated in actual distillery practice by the ordinary fermentation processes followed at present in most distilleries. The writer (25) has lately patented a new fermentation process, developed at the distillery of Borinquen Associates, Inc. of Hato Rey, Puerto Rico, in which even higher optimum values of sugars concentration are fermented with high yields and fermentation efficiencies, due to the use of a new principle in mashing and fermenting: the incremental addition of a conditioned thick mash to the fermenters. Incidentally, the period of fermentation is also greatly reduced.

Considering the data offered in table 15 it will be noticed that different stains possess variable ability to cope with high initial sugars concentrations in the medium. We have that while strain No. 531 can efficiently ferment up to 15.67 grams of total sugars per 100 ml mash, strain No. 502 can not resist much above 13.04 grams without beginning to feel impaired by the rising concentrations of the metabolic products. It will also be noticed that within a certain range of initial sugars concentration the efficiency of a given yeast does not vary much, but that a sharp decline is initiated as soon as the critical concentration is passed. For example, taking the case of strain No. 502, we have very similar yields and fermentation efficiencies for the sugars values between 10.14 grams and 13.04 grams; but a sharp decline in yields, and rapid rise in residual sugars values are encountered immediately after; that is, within the values 13.77 and 15.94 grams of initial sugars content.

The practical distiller should then be very careful about his optimum total sugars concentration; for any amount of sugars added to his mash in excess of what is strain can handle efficiently, will become wasted sugars, via factory slops; and

his yields will go down in accordance with the quantity of these wasted sugars.

TABLE NO. 15

				TION FOR EAC	CH STRAIN		-
No. 502	Number of Ferm Tests	Gram total Sugars 100 ml mash	Residual Sugars, Grams 100 ml beer	Yield of Absolute Alcohol, Per Cent on Total Sugars in mash	Fermenta- tion Efficiency Per Cent	Total grams Alcohol produced per 100 ml mash	Optimum Total Sugars Concentra- tion grs 100 ml mash
Yeast Strain No. 502	1	10.14 10.87 11.59 12.32 13.04 13.77 14.49 15.22 15.94	$\begin{array}{c} 0.71 \\ 0.89 \\ 0.89 \\ 1.07 \\ 1.09 \\ 1.32 \\ 1.60 \\ 2.05 \\ 2.20 \end{array}$	43.79 43.68 43.44 43.17 43.05 41.91 40.90 38.42 37.14	91.21 89.98 89.49 88.93 88.67 86.34 84.26 79.13 76.51	4.43 4.75 5.03 5.32 5.61 5.77 5.92 5.84 5.92	13.04
Yeast Strain No. 766	1	10.15 11.11 12.06 13.00 13.98 14.94 16.03 16.98 17.04	$ \begin{array}{r} 1.06\\ 1.09\\ 1.16\\ 1.27\\ 1.37\\ 1.88\\ 2.15\\ 2.45\\ 2.69\\ \end{array} $	42.74 42.47 42.44 42.53 42.24 42.15 39.27 37.26 34.86	88.05 87.49 87.43 87.60 87.00 86.83 80.90 76.76 71.80	$\begin{array}{r} 4.34 \\ 4.72 \\ 5.12 \\ 5.53 \\ 5.91 \\ 6.30 \\ 6.29 \\ 6.32 \\ 6.25 \end{array}$	14.94
Strain No. 533	1	9.88 11.52 12.18 13.03 13.88 14.97 15.45 16.29	$\begin{array}{c} 0.55 \\ 0.70 \\ 0.89 \\ 1.05 \\ 1.21 \\ 1.78 \\ 2.35 \\ 2.59 \end{array}$	43.92 43.49 43.26 42.89 42.28 40.68 37.54 35.36	90.46 89.59 89.11 88.34 87.08 83.78 77.32 72.83	4.34 5.01 5.27 5.59 5.87 6.09 5.80 5.76	13.88
Strain No. 531	1	10.12 12.20 14.57 15.12 15.67 16.37 16.92 17.48	$\begin{array}{c} 0.45\\ 0.60\\ 1.02\\ 1.25\\ 1.62\\ 1.90\\ 2.35\\ 2.81\end{array}$	47.28 46.81 46.26 45.21 43.98 41.34 38.94 35.63	97.39 96.41 95.28 93.13 90.58 85.16 80.21 73.40	4.78 5.71 6.74 6.84 6.89 6.77 6.59 6.22	15.67

SHOWING FERMENTATION RESULTS OBTAINED FROM DIFFERENT YEAST STRAINS UNDER VARIABLE TOTAL SUGARS CONCENTRATION; AND OPTIMUM VALUES OF SUGAR CONCENTRATION FOR EACH STRAIN

While it becomes a comparatively easy task to carry such tests as the ones whose results are given in table 15, the distiller will not have in all cases the means to control all intervening factors in his distillery; and hence, if he wants to repeat our experiments in actual commercial practice, he may find that his results vary a great deal from ours. Why? Simply because in our tests we kept under control all intervening factors that might have affected the results we were after, such as temperature, pH, yeast nutriments, infections, etc., etc. But even if unable to repeat our results, it would always pay the practical distiller to determine the optimum value of total sugars concentration he may use at mashing, under the peculiar conditions existing in his down distillery. Only by so doing will he be receiving the maximum returns from his capital investment. This optimum concentration of sugars does not mean necessarily that with which highest alcoholic yields per hundred grams of sugars are obtained, or that giving the highest possible alcoholic concentration in the beer; but that at which the distiller may receive the highest economic reward for his work. This means that extremely high yields and efficiencies, at the expense of curtailment of daily production will not do; or that very high alcoholic concentrations in the beers at the expense of drastic reductions in fermentation efficiency and extravagant loss of valuable sugars in the slops will not do also.

The degree of initial sugars concentration to be used in distillery practice will depend of a number of conditions under which a particular installation is operating. For this reason no specific rule will apply in this matter to all cases. If fermentation temperatures and pH values may be maintained under the will and control of the distiller at all times; and if besides, the distillery has stock or ready access to the acquirement of the necessary yeast nutriments, then he is in position to increase his initial sugars concentration, provided his strain of yeast is one capable of coping with the new conditions.

The nature of the raw material used in the distillery must also be considered in relation to this subject. A higher total sugars concentration may be used when sugar cane juice, or high test molasses is being used as raw material for rum making than when using ordinary blackstrap molasses. However, if a conditioned and purified blackstrap is used with the incremental system of building up the sugars, as described in the patent already mentioned above, we have that as high, or even higher total sugars may be successfully fermented than in the cases when cane sugar juice, or high test molasses are the raw materials used.

Before closing the discussion of this very important factor of total sugars concentration it must be remarked that high total sugars

when properly fermented, will give rise to a high degree of alcoholic concentration in the resulting beer. The ways in which the distillery is benefited by these high alcoholic concentrations may be enumerated as follows:

(a) Less fermenters are necessary for a given daily production of rum. Hence, less labor, floor space, and reduced accessory equipment such as pumps, motors, water and steam lines etc., will be required.

(b) Less steam consumption is required per proof gallon of distilled spirits, since a smaller volume of mash needs be distilled. Incidentally a smaller boiler installation is required.

(c) The high alcoholic concentration serves as a antiseptic for the fermenting mash, during and after fermentation. In this manner infections are prevented, and fermentation failures due to this cause are avoided. Also, in case of breakdowns in the still or other equipment of the distillery, the risk of losing the beers through infection becomes almost negligible.

(d) Ester formation during the fermentation period, and especially later on during distillation is greatly enhanced.

(e) The combined effects of the foregoing advantages, due to high alcoholic concentration in the beers, result in improving the general efficiency and economic status of the distillery.

4. The pH Factor in Rum Fermentation

Rum yeasts vary a great deal in the production of organic acids during the fermentative lapse. The quantities of these acids usually formed vary between the equivalent of 4.7 and 10.0 ml of a decinormal solution of sodium hydroxide for each 10 ml of fermented liquid. Of these totals, the fixed organic acids vary between 2.1 and 5.4 ml; and the volatile acids between 2.1 and 5.8 milliliters of the standard sodium hydroxide solution already mentioned. This increment in the acidity of the substrate during fermentation is measured in distillery practice by readings of pH values.

As in the case of total sugars concentration, and of temperature, the pH of the medium during the fermentation period is of enormous importance in rum fermentation, both from the standpoint of *quantity* and *quality* of the resulting rum. The maintenance of optimal pH value for a given strain during the complete period of fermentation will exercise great influence not only on the yield, but also on the

taste and aroma of the resulting distillate. Its action will be felt specifically as to the influence it has in the production of rum oil and other aromatic bodies, and as to the quantity and class of esters formed during fermentation and distillation of the mash. For every yeast strain, the inhibitory effect produced by the rising alcoholic concentration in the medium depends in direct ratio on the quantity of acids that the yeast produces during fermentation. In this manner, a fermentation may become paralyzed when only from 5.0 to 6.0 per cent of alcohol has been produced, if simultaneously the pH value of the medium has been lowered to readings between 3.0 and 3.5 pH; the had the pH been maintained at optimum value for the yeast employed (say 5.0 or 5.2 for instance), this same fermentation could have produced much higher alcoholic concentration; 8.0 per cent, or more. From these considerations, and those previously presented when discussing fermentation temperature and high alcoholic concentration in the fermenting mash, it may be readily apparent that the conditions of high temperature, low pH value, and high alcoholic concentration in a given fermentation are incompatible and non-attainable. The writer (²⁶) had previously delved on this important question in an article written in the Spanish language for the benefit of local Puerto Rican rum distillers.

If the maintenance of a constant optimal pH for a given rum fermentation results in high alcoholic yields; it also brings about an enhancing effect on the aroma of the resulting rum. By partial and selective neutralization of the organic acids produced by the yeast, two results leading to the formation of the right aroma in the resulting distillate are attained:

(a) Only those esters highly desirable in a rum of quality are mainly formed through partial elimination of formic and acetic acids, present in the fermenting mash in their five state. These acids, being of greater chemical affinity due to the reacting alkali added, will react with the formation of their respective salts. In this way the other free high aliphatic acids also present in the medium will have a better opportunity for ester formation during fermentation and distillation of the product. The esters formed by these higher fatty acids, as explained in the chapter on yeast selection, are the most valuable in the formation of the bouquet of a truly genuine rum. Besides, the great reduction obtained in the lower acids of the series by their conversion into salts, means that less of their esters will be formed, and that their absence in the free state in the resulting distillate, will increase its palatableness and its delicacy of aroma. A raw distillate produced under such controlled conditions of pH will be a fast attainer of maturity during the aging and curing period, and results a fast seller in the market.

(b) In case the yeast in use be a good producer of rum oil, there is nothing that will better contribute to this end than the maintenance of optimal pH during fermentation. It becomes sometimes necessary to carry rather high pH values during fermentation in order to receive to the maximum extent the beneficial effect upon aroma and taste produced by the presence of this most valuable constituent of the compound and complex rum aroma. When speaking of high pH values in rum fermentation we have in mind the range of 5.5 to 6.0 pH.

Before further discussion of the question of pH in the rum distillery it would seem adequate to insert at this point a few helpful conclusions regarding the AlcoholpH-Temperature-Complex, in rum manufacture:

a. During rum fermentation, ethyl alcohol and its congeneric products tend to inhibit the yeast producing them. Due to the higher toxicity in the metabolic products of rum yeast as compared to distillery yeasts, this inhibitory action is stronger than in the case of industrial alcohol fermentation.

b. There exists a great variation among different races and strains of rum yeasts as to the power of resistance towards this inhibitory action. Hence, it becomes necessary that every and each distiller determines the optimum concentration of these products that his particular strain may resist under condition inherent to his particular distillery.

c. As soon as the inhibitory action sets in, the yeast will cease to act efficiently on the sugars that may still be present in the substrate. Therefore, the distiller should exercise great care and control over the total sugars concentration he uses at mashing. Waste of valuable sugars, and poor fermentation yields will be the results of disregarding the careful control of the sugars concentration.

d. High temperatures of fermentation will result in:

- (1) Poor yields of rum.
- (2) Poor quality of rum. However, the expression high temperatures of fermentation does not mean a fixed range for all yeasts. Therefore, it becomes necessary to determine how this expression applies in every particular case. The writer in his consulting practice, has met with yeasts that would ferment best at the range

32-34° Centigrade, while others would be inhibited by this temperature range. For all cases, however, temperatures within the range 35-40° Centigrade become inhibitory.

e. Inadequate pH values, especially those either above 6.0 or below 4.0 pH will bring about inhibition of rum fermentation, although the action of these two extremes are not alike; the higher range acts through the retardation of initiation of fermentation by the yeast; while the lower range acts by the checking of the already started process.

f. Combinations of high temperatures and low pH values will result in more adverse consequences than either restricting factor acting alone. If a distillery must by necessity work under conditions of high temperatures and low pH values, then it becomes necessary to use the lowest possible total sugars concentrations at mashing.

g. Mashes of high total sugars concentrations are recommendable and beneficial as already explained, and they may be successfully fermented, provided that:

- (1) A high alcoholic concentration resistant yeast is available.
- (2) An adequate balancing of nutriments may be built at mashing.
- (3) Temperature of fermentation may be artificially controlled within the range 28-31° Centigrade.
- (4) The pH value of the fermenting mash may be maintained within optimum range, during the entire period of fermentation.

h. Low temperature of fermentation, and adequate pH control will result in the formation of a rum with the following desirable characteristics:

- (1) Suavity and delicacy of aroma
- (2) Agreeable taste and fine body.
- (3) Quick maturing; that is, it will require but little time of aging and curing to become a marketable, first-class rum.

Now that the subject of pH values in the fermentation of rums has been discussed in a general and more or less theoretical way, it would be well to present conditions which affect the handling of the pH question in actual rum distillery practice. Not always may laboratory experiments and results be transplanted to commercial establishments and the same success previously met with in the laboratory duplicated. This is the case with the question of pH, as the author's consulting practice has shown.

In the first place, only few rum distilleries use pH meters; and few pH meters offer reliable readings for a long time when used in a rum distillery, due to mistreatment of instruments and carelessness in their upkeep. The first requisite for duplication of laboratory results is a duplication of the laboratory atmosphere and environment as the commercial plant. This is, and will always be difficult. But leaving aside the question of reliability of instruments and personnel handling them, we still meet with circumstances adverse to the establishment of optimum pH practice in the rum distillery. Questions of design, unharmonious correlation of different stages of manufacture, unhappy disposition and distribution of space and equipment, unbalanced equipment, and many other factors that would take too much space and time to enumerate, all work against the obtention of optimum pH in rum distillery work.

The fact is that tendency in practice is to carry that pH at which the fermentation shall be accomplished the in least possible period of time. The question of whether a better rum could be produced by using a different pH, even if the fermentation period becomes somewhat longer, is not usually considered, and if it happens to be considered, most of the times the reduced fermentation capacity does not allow the trying out of the experiment without incurring in curtailment of production, costly stoppages of the still, etc., etc. This is one of the reasons why we recommend in the chapter on yeast selection that the presumptive distiller should first select the yeast or yeasts he is to employ in distillery practice, and then design his distillery according to the inherent and well studied characteristics of his selected organism. Only in this way could we hope for the possibility of accomplishing in commercial practice the results of a laboratory research.

With these limitations in mind we shall proceed to offer a few suggestions to rum distillers on the management of the pH question.

In the first place, the distiller must feel well assured that when he takes a pH reading in the proper form, his apparatus is not giving misleading information, that is, the pH meter must be in perfect order and condition *all the time*.

The next point for the distiller's consideration should be the question – What type of rum do I wish to produce? And lastly – What equipment have I available for best results?

Other factors remaining constant, the lower the pH maintained, the quicker will the fermentation finish. Also the lighters will the rum produced be. As the pH value is raised, fermentation will take a longer period for its completion and the

heavier will the rum produced be. The maintenance of constant, or practically constant pH during the entire fermentation period, although highly desirable, offers difficulties in actual work. In cases of pure culture fermentation no trouble is met in this respect, due to the fact that only the acidity produced by the yeast has to be neutralized. But in actual distillery practice pure culture technique is very rare. Bacterial contamination is very common, and when neutralizing the acidity of the mash, we are also helping towards the further propagation and development of the bacterial contamination, since bacteria will generally thrive better than yeast at higher pH values. If the bacterial contamination happens to be of the acetic, propionic and butyric groups, they will soon begin to produce acid products and after a few hours of neutralization the acidity will rapidly reappear in the mash in worse form than before the raising of the pH took place. For this reason before practicing constant pH fermentation the distiller must be careful of the amount and type of bacterial contamination existing in his mash. The best neutralizing agents are liquid ammonia and calcium carbonate; the former being superior in that while maintaining the pH at practically a constant value, it will also serve as an excellent nutrient for the yeast cell. Other very important results we have found through the use of liquid ammonia in rum fermentation will be discussed in the same chapter when treating on special methods of rum fermentation.

When a rum rich in volume of aroma and taste is desired, the distiller should try to maintain the highest pH possible without incurring in perilous propagation of contaminants, or in troubles due to lack of proper equipment.

5. The Fermentation Agent.

This topic has been already discussed in our chapter on Yeast Selection. There are yeasts best adapted for the manufacture of heavy types of rum and others best adapted for the manufacture according to the type of rum he wishes to manufacture. When the distillery has not been designed and built with the idea of suiting the characteristics of a given selected strain (as should be the logical way) then the distiller should be careful not to use as his fermentation agent a yeast for which his distillery is not equipped. For instance, suppose the distillery must work on a 36-hours cycle in the fermentation end, and the distiller chooses a slow fermenting

yeast, how will he manage to run the distillery without meeting with trouble? There must exist correlation and harmony between the plant facilities and the characteristics of the fermentation agent.

6. Duration of Fermentation

Duration of fermentation is oftentimes dependent on the fermentative capacity of the distillery. We have already stated that this condition is abnormal and unjustifiable, and that it should not exist in a well designed distillery. Not considering questions of faulty distillery design, the other factors affecting the length of the fermentation lapse are: temperature; pH; yeast nutriments available; total sugars concentration, and inherent characteristics of the yeast; as well as the use or non-use of yeast activators, and the amount of seed yeast used at setting, and method in which this seed yeast is incorporated to the main mash.

Without going into uneconomic extremes, higher temperatures and lower pH values will shorten the fermentation period, the reverse conditions of pH and temperature will lengthen it. A well balanced content of yeast nutriments in the fermenting medium will also materially shorten the fermentative lapse. A range of total sugars concentration may be used with little variation as to the time taken for finishing the process; provided, however, that the optimum value for a given strain is not much exceeded. For instance, and referring again to table 15, strain No. 502 would ferment in about equal periods of time total sugars concentration values from 10.14 to 13.04 grams per 100 ml mash; strain No. 766 will do the same for concentrations ranging between 10.15 and 14.94 grams per 100 ml mash. But if the higher values of these ranges are surpassed, slow and laborious fermentation will set in, materially lengthening the period of fermentation. This is another important reason why every distiller should use a range of sugars concentration suited to his particular strain. The characteristics of the yeast used as fermentation agent, no doubt will be a factor of prime importance, as some strains are naturally slow, and others very fast, in the work of converting sugars into alcohol. Activating chars have been sued as means of inducing a faster rate of fermentation, at the same time protecting somewhat the yeast from the deleterious action of some of its metabolic products. It has also been found that by adding any inert material as very finely divided "bagacillo" or wood dust the fermentation lapse may be shortened.

The cellular concentration per unit volume, total volumes, and manner of applying the seed yeast, or footing, to the fermenter becomes another factor of considerable importance in this matter of fermentation time. The stronger the concentration of yeast cells and the larger the volume of footing, the quicker will fermentation be finished, other factors being under control. The methods of adding the footing to the fermenter is also a matter of importance. When the footing is added upon a practically full fermenter, a sudden dilution of the cell concentration originally present in the inoculum occurs. The cell concentration of the fermenter mash, immediately after seeding, may be but 5 to 10 per cent of that originally in the inoculum. The adverse effect of this sudden dilution will then depend of the amount of original concentration in the footing, and on the power of propagation of the yeast used. Whenever possible it is much safer to add the yeast footing into an empty fermenter and then proceed with its filling with fresh mash. By this method the dilution of the cell concentration in the footing goes on slowly as the fermenter is filled up, and cell multiplication occurs simultaneously with the filling. Of course this method works better in the case of very large fermenters, that take quite a lapse of time in their filling.

Quick fermentation periods offer the advantages of less opportunities of infection with possible loss of a fermenter; less number of vessels to be filled; a quicker liquidation of the fermentation end, if necessary; less chances of having to shut down the still temporarily for lack of fermented material, and in general a quicker transformation of raw material into finished product. These advantages become more important if the end product desired is a light rum; but if a high bodied rum with ample aromatic tone and excellence of taste is desired, then it would be necessary to lose the aforementioned advantages and revert to a longer period of fermentation. So, in the long run, it is quality and special characteristics sought in the finished product that should determine the kind of fermentation to be followed.

7. The Chemical Composition of the Raw Material

The effects of this factor upon the fermentation process have already been taken up in chapters one and three, under the discussion of raw material selection and mashing methods.

8. Afterfermentation Treatment of the Beers

This subject could be equally discussed during the chapter of rum distillation under the heading "Pre-Distillatory Treatment of the Beers"; but on careful consideration we decided to include it here.

A mash under fermentation is supposed to be finished when no further attenuation occurs in its specific gravity, or degree Brix, and when the characteristic ebullition caused by the escaping carbon dioxide can no longer be observed. At this point the mash is converted into a so-called "beer" and may be distilled directly, or submitted to aftertreatment before finally delivering it to the still.

Our studies have proved that the second course is the better, both from the standpoints of rum quality, and conservation and maintenance of the distilling outfit. How this practice of after-treatment of the fermented mash affects the distillation end of the distillery will be discussed fully when dealing with that subject in the next chapter; here we shall limit ourselves to its effect on rum quality, and recovery of a valuable by-product.

The aftertreatment of the beer may be done with three different objectives in view, or for any single one of them. These objectives are:

(*a*) Allowing a period of rest to the fermented mash for purposes of improvement of aroma and taste in the resulting rum.

(b) Recovery of the valuable yeast bottoms for their use either as animal feed or fertilizer.

(c) Delivering a cleaner fermented mash to the still, free of suspended solid impurities.

In our opinion, and whenever possible, the aftertreatment should be conducted for the fulfillment of all three objectives. The treatment with this end in view is conducted in the following manner:

When fermentation is finished, the beer is given a period of rest (which may vary according to local conditions between 12 and 24 hours) either in the fermenter itself, or preferably in closed, aseptic tanks, especially built for the purpose. This period of rest being over, the liquid is then filtered in bulk through a supercentrifuge, by which all suspended solid matter is separated from the fermented mash, including the yeast cells. The perfectly clean fermented mash is then delivery to the still or stills for distillation. The separated yeast and other solid impurities, may be discarded, or if preferred, may be further treated and purified for recovery of the valuable yeast residue.

The advantages of such a procedure will be the obtention of a better rum and the recovery of a very valuable (especially in these war times) by-product.

Special Fermentation Methods

The subject of rum fermentation has been presented in the above discussion in its general and broader aspects, mentioning the most important factors influencing the process. In what follows, we shall present some special fermentation methods developed during our research.

1. Heavy Rum Fermentation, Including the Jamaica Export Types

We started our fermentation studies with the production of the types known in the trade as "heavy rums"; of which the Jamaica export types form a very well known class.

Previous to our investigations the idea prevailed that the types of export rums produced by the Island of Jamaica could not be duplicated elsewhere. The writer thought otherwise, and after obtaining all the information that the meager literature on the subject would afford, he started his own experiments on the fermentation and distillation of such rums. As the work developed, we have been able to produce every one of the heavy rum types manufactured in Jamaica, and samples of such rums have been declared equal excellence to the best Jamaican products by European experts and importers. Incidentally we have proved that the old theory maintained by many rum experts and research workers, that the heavy Jamaica rum was not a genuine product of fermentation and distillation; but that it was artificially concocted by the addition of flavoring and aromatic constituents needs not be taken too seriously, for we certainly have produced these same rums, without having to recur to such expedient. We do not claim, however, that the Jamaica substances, for that we do not know; but we do want to assert the fact that such rums have been produced by us, indistinguishable other than special fermentation and distillation methods.

As explained in the preceding chapter, the part played by mitogenetic radiation in this feature of our heavy rum studies, brought further interest from a scientific point of view to the work on heavy rum fermentation.

The so-called "heavy rums" have usually been differentiated from the more common type known as "light rums" by their corresponding non-alcohol-number coefficient, or coefficient of impurities, as well as by physical and organoleptic differences of body, taste and aroma. A heavy rum is regarded as possessing a higher non-alcohol-number and a richer and more intense taste and aroma than a light rum. A heavy rum is also distinguished in possessing a higher content of rum oil and a very high index of persistence in both aroma and taste; by which is meant that it can endure high dilution with an aqueous solution of neutral spirits before its characteristic aroma and taste can no longer be perceived by an experienced rum taster.

While the heavy type of rum has been manufactured in the past and is being produced at present, most of the methods hitherto followed have been of a haphard or empirical nature, and, therefore, coupled with uncertainty and perils in execution. The final results of these "modus operandi" have been detrimental to the quality of the finished product, to the economy of the process, or to both. These facts have been evidenced in practice by the preference shown by the consuming public toward the light rums. Two main reason exist for this public attitude: (1) The few wholesome, genuine, heavy rums on the market are too expensive for the average purchaser; (2) most of the low priced heavy rums on the market are improperly fermented and distilled, or are artificially concocted. For instance, it has been sought to manufacture heavy rums by merely changing the method of distillation as used in the manufacture of light rums, so that more of the so-called "head products" are allowed to pass over into the main distillate or raw rum. This was done with the idea of increasing the non-alcohol-number, and to add the necessary extra flavor and aroma. These attempts, however, have always failed to produce a first class, genuine, heavy rum, since what is really accomplished by such procedure is the addition to the main distillate of undesirable congenerics of the rum fermentation; being in fact the very same products that are so very carefully and painstakingly eliminated from the distillate when manufacturing light rums. Obviously, a carelessly distilled light rum is not a first class, genuine heavy rum. In table 16 are shown comparative analyses of two heavy rums, (1) a genuinely produced one, and (2) one produced by the method described above.

The pertinent points of difference between these two rum samples are represented especially by their respective indexes of persistence, taste, odor and body number. Also in the different contents of rum oil, and very particularly in the great differences in the ratios between esters and higher alcohols, volatile acidity, and aldehydes, respectively; and in the ratios between high boiling and low boiling points esters and aldehydes for each particular rum.

The comparison also brings out the important fact that although the nonalcohol-numbers are practically identical in amount, yet the indexes of persistence, odor and other characteristics are quite different in the two cases. This confirms our previous assertion that it is not so much the amount of extent of the non-alcoholnumber of a given rum that imprints its characteristics as to a good or bad beverage; but the *nature* of the individual constituents composing this non-alcoholnumber coefficient, and the ratios existing within the group constituents.

TABLE No. 16

	No. 1 Genuine	No. 2 Spurious
Body Number	13.01	10.5
Index of Persistence	1:100,000	1:22,000
Taste	Mellow. fine	Ardent, strong
Odour	Delicate	Irritating
Rum Oil content, amount	Liberal	Slight
Alcohol by Volume, Per Cent	48.84	44.24
Total Acidity (mgs. per 100 ml Abs. Alc.)	204.91	179.80
Fixed Acidity (mgs. per 100 ml Abs. Alc.)	163.70	120.20
Volatile Acidity (mgs. per 100 ml Abs. Alc.)	41.21	59.60
Aldehydes Acidity (mgs. per 100 ml Abs. Alc.)	55.70	89.90
Esters, Acidity (mgs. per 100 ml Abs. Alc.)	198.20	155.15
Higher Alcohols (mgs. per 100 ml Abs. Alc.)	82.94	115.45
Extract (mgs. per 100 ml Rum)	518.00	787.00
Ash	9.20	17.90
Ratio esters: higher-alcohols	238:100	134:100
Ratio esters: volatile acidity	480:100	260:100
Ratio esters: aldehydes	355:100	172:100
Ratio Non Alc. number: extract	105:100	69:100
Ratio Volatile Acidity: total acidity	20:100	33:100
Ratio Ash: extract	1.8:100	2.3:100
Ratio High: Low boiling point esters	97:100	37:100
Ratio High: Low boiling point aldehydes	98:100	77:100
Ratio Higher Alcohols: Non-Alcohol-Number	15:100	21:100
Non-Alcohol-Number	541.75	540.30

COMPARATIVE CHEMICAL ANALYSES OF A SPURIOUS VS A GENUINE HEAVY RUM. IMPORTANT RATIOS AND PHYSICAL ORGANOLEPTIC TESTS ALSO INCLUDED

It has also been observed that the presence of some bacteria in the fermenting alcoholic medium aids in securing flavor and aroma for the resulting rum, and a second practice has been (especially in Jamaica) to carry the rum fermentation forward in a substrate which was purposely badly infected at random with whatever bacterial flora was available. In this way the mash became the abode of all kinds of unidentified and uncultured bacteria which competed with the yeast strain (without check or control) in the fermentation of the sugars present in the rum mash. The success or failure of such a method of heavy rum making depends on the kind and extent of the infection present. Even when successful as to the quality of the product obtained, when by chance or luck the right kind of bacteria, and *only* such, are present as the infecting organisms, this unscientific practice leads to poor results with regard to yields and fermentation efficiencies, thus adversely influencing the economy of the process.

We endeavored from the start of our heavy rum work to improve upon these older and unscientific methods of heavy rum production, and discovered that the problem could well be attacked by a procedure comprising:

(1) the subjection of the raw material to a pretreating operation which fits it for its intended use.

(2) The selection of yeast and bacterial pure cultures adapted for symbiotic fermentation of heavy rum mashes.

(3) The employment of optimum conditions for the production of alcohol, and symbiotic fermentation for the production of aroma and flavor, wherewith to obtain high yields and fermentation efficiencies with a comparatively rapid fermentation, and a high quality of final product.

(4) The employment of a proper distillation method for the resulting beer.

How the first part of this program was carried out has already been treated in chapters one and three of this bulletin and the last part, or that dealing with the subject of proper distillation method will be taken up in our next chapter, where rum distillation in general will be discussed.

Item No. (2) dealing with the proper selection of the symbiotic ferments will be briefly reviewed here. It was found that the type of yeast used must be adapted to the fermentation of heavy rum. All yeasts, the reader will recall, are not suitable for the production of genuine heavy rums, and not even all varieties of rum yeasts will serve. It was, therefor, necessary to carefully select strains of heavy rum yeasts.

The best class of rum yeasts suitable for the fermentation of heavy rums are found among the Schizosaccharomyces or fission yeasts, but a few strains of budding yeasts were also found adapted for the purpose, and employed with success. A characteristic of a proper rum yeast for this purpose is that it should be able to stand moderate, but appreciable concentrations of organic acids during the alcoholic fermentation, particularly saturated aliphatic fatty acids such as acetic, propionic, butyric, valeric and others yet higher in the series. The yeast must also be capable of continuing its fermentation at temperatures between 27 and 33 degrees Centigrade; and cooperate symbiotically with the bacteria utilized as auxiliary fermentation agent. Two of our Schizosaccharomyces, strains Nos. 501 and 502 were found to meet these conditions almost ideally. Two budding yeasts, strains Nos. 531 and 532 were also found adaptable to meet the requirements. Most of the actual experimental work in this connection was effected with strain No. 501.

As to the bacterial auxiliary ferment, a number of bacteria was found adequate and well suited for this purpose; particularly members of the propionic and butyric acid groups. It was found that the bacteria must conform to the following specifications:

(*a*) Their life activities should not be arrested too soon by the yeast metabolic products formed in the fermenting medium, particularly by the ethyl alcohol.

(b) They must not attack or decompose, or materially change, the existing products of the yeast metabolism to such an extent as to materially reduce the yield of rum; that is, they should act upon the sugars and other materials of the molasses itself rather than upon the metabolic products of the yeast.

(c) They should possess the power of acting upon the residual sugars, following the initial alcoholic fermentation, utilizing these sugars in the elaboration of the products of their own metabolism.

(*d*) Their metabolic products should be of such character as of themselves to enhance the flavor and aroma of the resulting rum; or be of such nature that they will readily combine chemically with the metabolic products of yeast fermentation (particularly with ethyl alcohol) to form highly flavored aromatic compounds.

(*e*) They must be of such nature as will readily and fully act in the same class of substrate as required for alcoholic production when fermentation is properly conducted.

Among the bacteria tried in these studies as auxiliary fermentation agents, strains of Clostridium Saccharo Butyricum and of Propionobacterium Technicum were found efficient and well qualified symbiotic agents. Of the two, the Clostridium Saccharo Butyricum produced in more perfection the Jamaica types of rum; but the Propionobacterium Technicum was equally efficient in the production of other types of heavy rums. Besides, a mold of the Imperfecti group, Oidium Suaveolens, was

also found very well adapted for the production of a special type of heavy rum. As our main interest was the preparation of the genuine Jamaica types, Clostridium Saccharo Butyricum was selected as the main auxiliary ferment. As explained in the preceding chapter on mitogenetic radiation, this strain was carefully studies in this connection. The bacillus produced a mixture of valuable aliphatic acids, consisting principally of normal butyric, acetic and propionic acids; but including a small percentage of other higher acids in the fatty series, such as caproic and heptoic acids. The normal butyric acid constituted 90.0 per cent of the mixture. No appreciable amounts of alcohol, aldehydes or ketones were found in the metabolic products of this particular bacillus. Its power to generate mitogenetic energy has been discussed at some length in the preceding chapter. It was also found that the bacteria became inhibited whenever the sugars concentration of the medium was higher than 6/0 grams total sugars per 100 ml mash, or whenever the alcoholic concentration in the fermenting liquid was 8.0 per cent or more by volume, or whenever the pH decreased until it approached the value of 4.0. All of these facts were taken into consideration in the method finally adopted for the fermentation of these rums, which is as follows:

The molasses is firstly pretreated as described in our third chapter on mashing operations. The treated molasses, in the form of a purified thick mash is further diluted by incorporation of additional water to the required density. It is very important here that the density of the thin fermenter mash be kept at such a value that the total sugars concentration per 100 ml shall not exceed a limit between 12.0 and 13.0 grams. The thin mashing control is determined by grams of total sugars per 100 ml of mash rather than by the Brix densities; but for a given plant operating on a substantially standard molasses input, the mashing operator soon learns the Brix density range corresponding to the aforesaid sugars concentrations, and can employ the Brix density as a simple means of controlling the mash dilution. It is preferable to maintain the sugars concentration as high as possible, but a lower concentration than 13.0 grams per 100 ml may be employed if desirable or necessary, with a particular molasses, or yeast, or bacterium.

The maximum initial total sugars concentration of 13.0 grams per 100 ml mash, has been selected to the benefit of the bacteria, as most bacteria of the propionic or butyric groups do not tolerate the sugars at much above 6.0 grams per 100 ml; and are also inhibited by alcoholic concentration of 8 per cent by volume, or more.

96

Since 13.0 grams per 100 ml do not yield over 8.0 per cent by volume (probably yield, 7.3 to 7.7 per cent by volume), a safety factor is provided regardless of the yeast action. Since the total sugars are reduced to about 6.0 grams per 100 ml at bacterial seeding, the consequent alcohol concentration will not exceed 4.0 per cent by volume, and the bacteria are then well able to proceed with their work in symbiosis.

The fermenter first receives an active, vigorous yeast footing and then the thin mash is introduced. This footing should amount to between 5 and 20 per cent, and preferably is about 10 per cent of the total working volume of the fermenter, as this assures a rapid start of the fermentation without involving the difficulties inherent to the preparation of a very large footing ,especially if the fermenter is of very large capacity. The fermenters may be of the closed type, constructed of polished iron or steel, and provided with mash cooling devices and means for agitating the mash either mechanically or by the admission of jets of carbon dioxide gas at the bottom. The carbon dioxide may be obtained from another actively going fermenter, or from compressed carbon dioxide containers. Agitation by means of air or other oxidizing gas is not recommended, on account of the detrimental effect upon the anaerobic bacteria. It is also preferred to provide the fermenter with continuous recorders for temperature and pH.

The thin mash is added upon the yeast footing in the fermenter, with a gentle stirring or agitation of the contents, so as to provide thorough and even distribution of the seed yeast. When all of the mash has been added the pH value is noted, and the contents are corrected to a value between 5.5 and 5.8 by the addition of either sulphuric acid or milk of lime, as the case may require. The setting temperature should be between 30 and 32 degrees Centigrade.

Fermentation is allowed to proceed under temperature control within a range of 30 to 33 degrees C. preferably accomplished by means of cooling coils placed outside of the fermenters and through which the mash may be circulated whenever temperature correction becomes necessary. After the sixth hour of fermentation, tests are made for the determination of total sugars in grams per 100 ml mash. Likewise determinations of alcoholic concentrations in percentage by volume are thereafter effected every two hours. When the percentage of alcohol by volume is about 3.5 to 4.0 per cent, and the grams of total sugars per 100 ml mash have a value below 6.0 grams, the conditions are ready for the incorporation of the bacterial footing to the fermenting mash.

The fermenting mash is first corrected in pH value to a reading of between 5.5 and 5.8, if necessary. The pH value of the bacterial footing is similarly adjusted to essentially the same value, and then while gently stirring or agitating, the bacterial footing is added to the fermenter in an amount equivalent to 1:4 to per cent of the total volume of the fermenting liquid. It is preferred to employ 2 per cent of bacterial footing when a yeast footing of 10 per cent has been used under the above conditions, as the ratio of bacteria to yeast up to 1:5 appears to render optimum result. The higher the ratio of bacteria to yeast up to 1:5, the heavier and more aromatic is the resulting rum; but when the ratio is much higher than 1:5 there is danger of obtaining uneconomically low total yields, since the faster propagation of the bacteria will overcome the yeast.

After the addition of the bacterial inoculum, greater care of the temperature control is necessary, as it is then important that the temperature within the fermenter should not go much above 29 to 30 degrees Centigrade. Correspondingly, the pH value should be so controlled that it will never be below pH 5.0. The fermentation is then allowed to proceed to a finish.

One of the main differences observed during heavy rum fermentation with and without the use of bacteria as auxiliary ferments was the time taken for the finishing of the fermentation period. This phase has been reported in the previous chapter on mitogenetic radiation; but other differences as fully important were also observed, and these will be presented here.

The action of the auxiliary bacterial ferment resulted also in the obtention of a great increase in the ester value of the resulting rum, and in a greater volume of taste and aroma. Usually, the fusel oil content was also smaller in those rums fermented under symbiosis of yeast and bacteria; and the time required for maturing was also less in the case of the symbiotically fermented rums.

In table 17 are presented a few fermentation results obtained through the use of the same yeast stain; with, and without the symbiotic effect. The analytical results obtained in the analyses of the respective raw distillates are also given. Yeast strain No. 501 was used, with Clostridium Saccharo Butyricum as the symbiotic auxiliary ferment.

TABLE No 17

FERMENTATION RESULTS OBTAINED USING (1) YEAST STRAIN NO. 501 SINGLY, AND (2) IN SYMBIOSIS WITH CLOSTRIDIUM SACCHARO BUTYRICUM. ANALYSES OF THE RESPECTIVE RESULTING RAW RUMS AS DISTILLED ARE ALSO GIVEN

	FERMENTATION RESULTS							ANALYSES OF THE RAW RUMS										
		-1-				-2-			-1-				-2-					
Fermentation No	Ferm Time Hours	Residual Sugars, grs per 100mL	Yield Alcohol on T. Sugars, Per Cent	Ferm. Efficiency Per Cent	Ferm. Time Hours	Residual Sugars, grs per 100mL	Yield Alcohol on T. Sugars, Per Cent	Ferm. Efficiency Per Cent	Alcohol by Volume Per Cent	Total Acidity, mgs 100 mL Ab. Alc.	Aldehydes mgs per 100 mL Ab. Alc.	Esters mgs per 100 mL Ab. Alc.	Higher Alcs. mgs per 100 mL Ab. Alc.	Alcohol by Volume Per Cent	Total Acidity, mgs 100 mL Ab. Alc.	Aldehydes mgs per 100 mL Ab. Alc.	Esters mgs per 100 mL Ab. Alc.	Higher Alcs. mgs per 100 mL Ab. Alc.
1	70.0	0.91	41.90	86.30	28.0	0.78	42.11	86.73	74.80	69.10	50.50	89.40	72.30	75.60	73.00	41.90	162.20	33.70
2	82.0	1.16	40.90	84.24	42.0	0.91	40.80	84.03	76.90	31.27	61.80	108.1	90.15	78.30	33.50	65.40	384.40	66.90
3	90.0	0.86	44.00	90.62	36.0	0.94	43.40	89.39	80.00	58.1	51.80	92.50	86.24	79.97	62.4	54.90	176.00	45.60
4	84.0	0.87	41.95	86.40	38.0	0.80	42.40	87.33	77.25	55.0	41.80	96.80	100.2	78.65	57.0	36.40	247.2	80.00
5	96.5	0.89	42.14	86.79	48.0	0.93	42.00	86.50	80.50	29.1	31.70	79.95	91.80	77.90	28.70	34.20	177.1	74.50
6	79.50	0.95	43.68	89.96	36.0	0.92	44.08	90.79	79.80	20.0	27.80	104.8	99.30	81.15	20.70	29.3	178.9	63.50
7	91.00	0.92	41.12	84.69	48.0	0.80	40.86	84.16	77.07	47.8	39.1	71.17	70.28	76.41	51.2	39.5	196.4	42.60
8	95.0	0.97	43.05	88.67	48.0	0.91	43.87	90.36	76.00	49.7	33.2	93.90	101.50	75.23	54.9	31.0	286.5	89.70
9 10	90.25 72.0	0.91 0.81	42.17 43.01	86.85 88.58	41.0 36.0	$\begin{array}{c} 0.85\\ 0.88\end{array}$	41.60 42.90	85.68 88.36	73.57 74.85	25.0 19.70	61.90 33.91	87.80 91.80	73.81 81.18	73.07 73.93	23.40 20.30	65.0 35.70	165.20 183.7	36.50 39.70

Considering the results of table 17 it will be noticed that there are no significant differences in symbiotic and non-symbiotic fermentation as to yields of alcohol per cent on sugars; residual sugars left after completion of fermentation; and fermentation efficiencies in the respective fermentation. But there is a very significant difference as to the total period of fermentation. In this case, with not a single exception, the hours of fermentation are greatly reduced in the cases where the auxiliary ferment was employed.

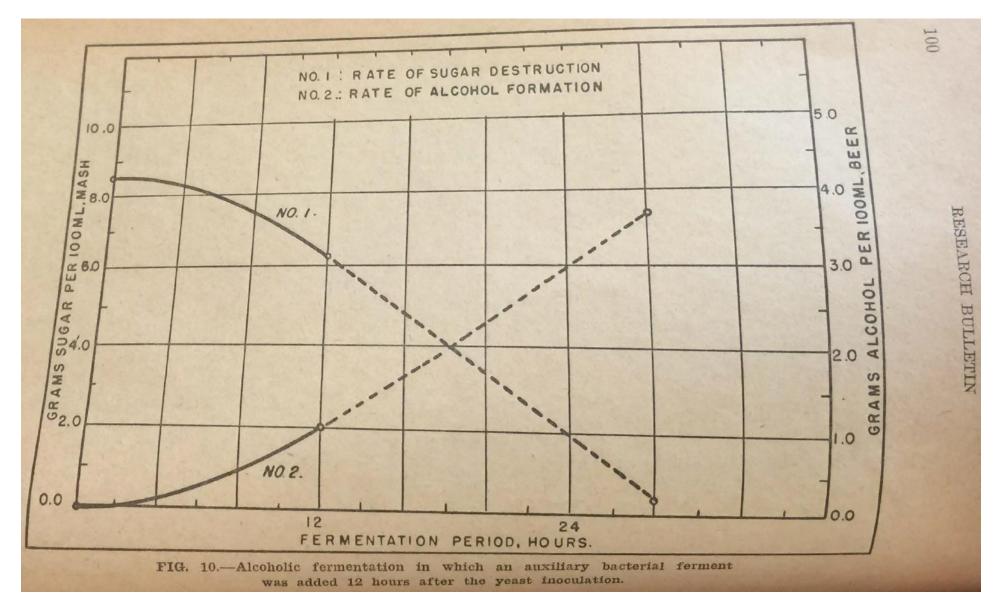
The analyses of the resulting rums, again show no significant differences as to alcoholic strength, total acidities, and aldehydic contents; but they do show consistent and considerable differences in ester and higher alcohol figures, respectively. Hence, the principal results of adding the auxiliary ferment are: (1) diminution of the hours taken for completion of fermentation; (2) increase in ester value of the resulting distillate; (3) decrease in the content of higher alcohols.

Fig. 10 to 13 present curves showing the effects on total fermentation time, sugars destruction, and alcohol formation, when the auxiliary ferment is added at different stages of the alcoholic fermentation. The dashed portion of each curve indicates the moment of bacterial inoculation into the yeast mash, and the total time of symbiotic effect. Fig 14 presents respectively the curves of sugar destruction and alcohol formation under non-symbiotic conditions, that is the yeast working singly. No comments will be given on these figures for they are self explanatory.

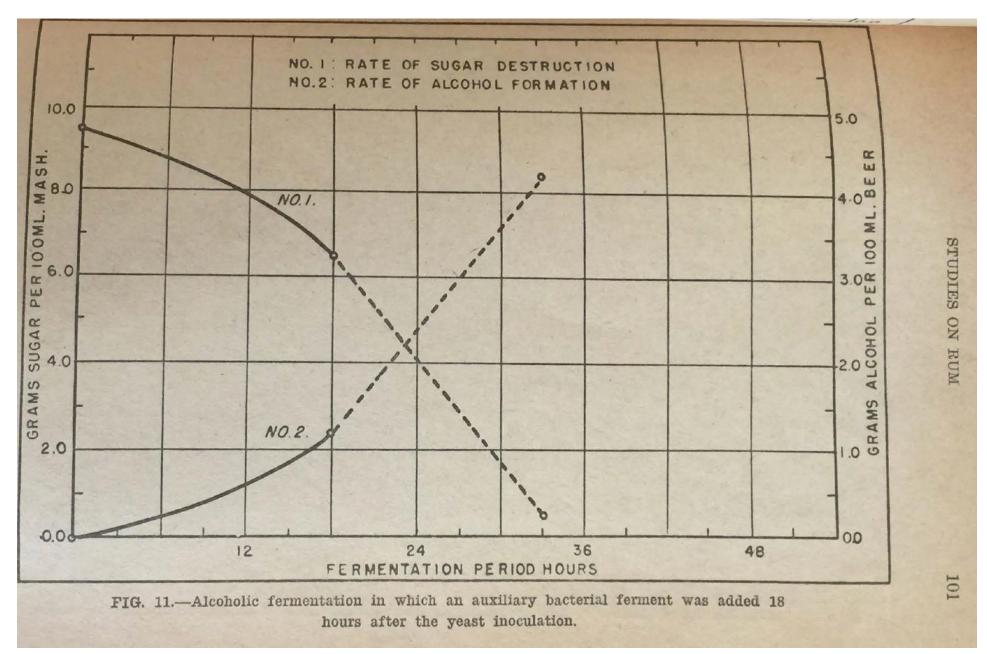
If in the above method of fermentation, Propionobacterium Technicum is substituted for Clostridium Saccharo Butyricum, we shall still have a heavy rum as the end product of fermentation, but this new rum will be quite different to that produced by the former bacterium. It will no longer offer the well-known characteristics of export Jamaica rum.

Another special type of heavy rums was produced during our studies and experiments. This time the raw material used was sugar cane juice. The yeast strain used was No. 764 and the auxiliary ferment was a member of the Fungi Imperfecti, *Oidium Suaveolens*. The *Oidium* was found and isolated by the writer from the sap of a tree much used in Puerto Rico for shading coffee plantations.

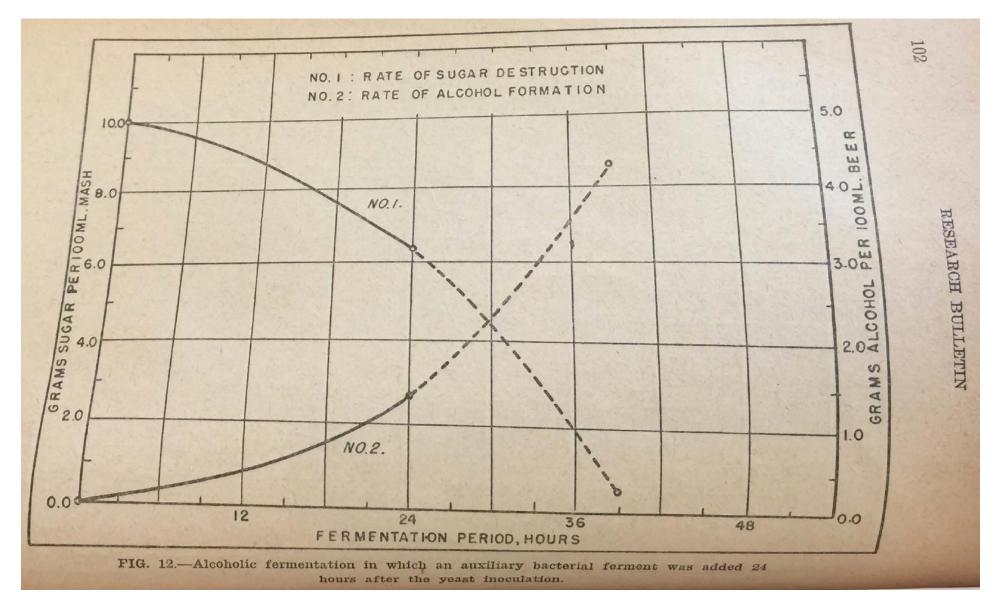
A study of this *Oidium* revealed that it would grow very fast in cane sugar juice medium with the production of a thick film over the surface of the liquid. It was further discovered that it hardly touched the sugars in the medium, but that it was a good producer



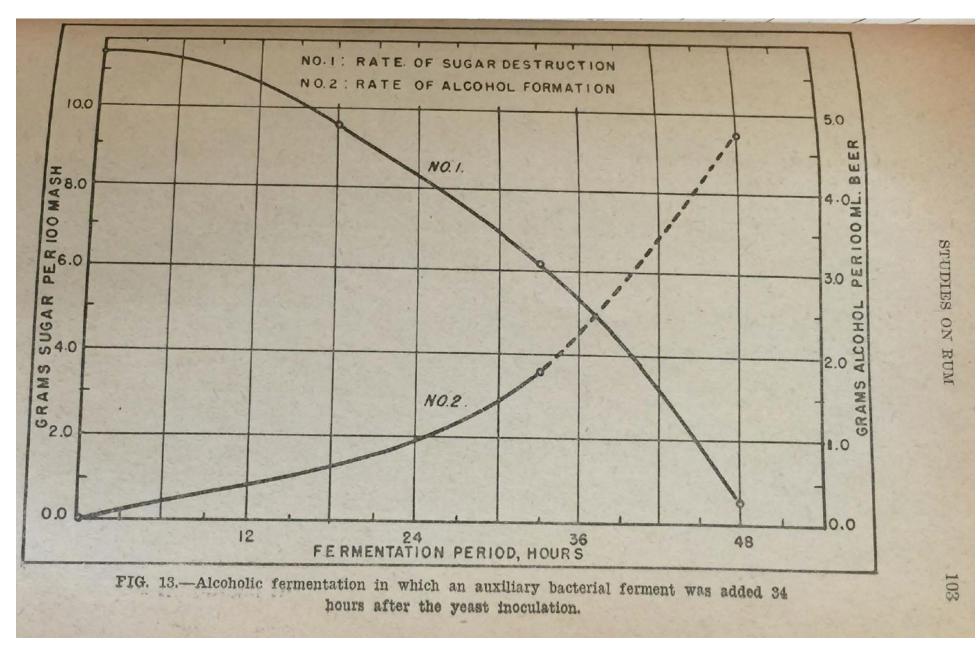
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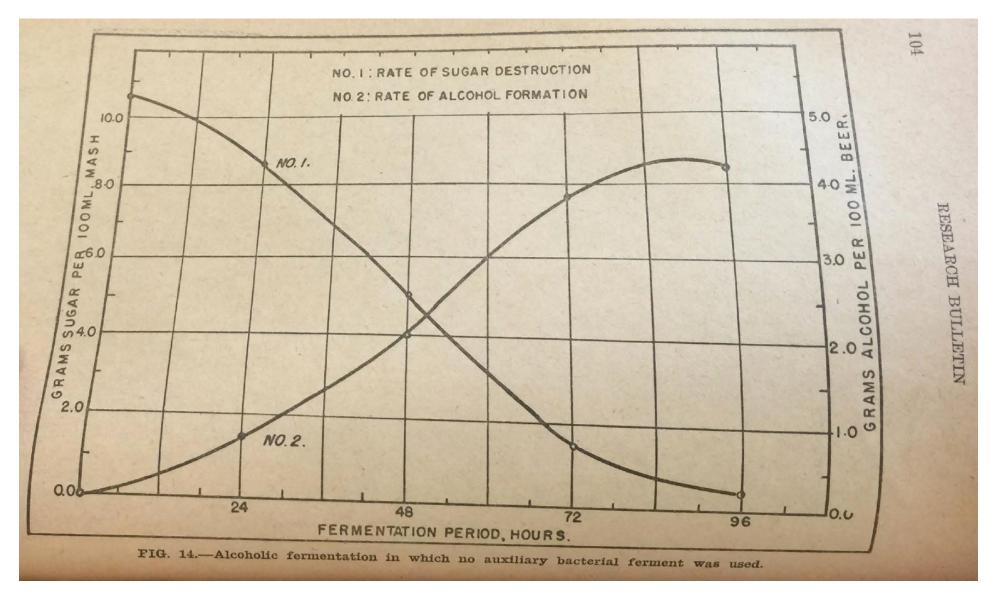
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102



103



of esters and organic acids from the proteins of the raw material. A fragrant odor, very similar to that of ripe apples was the predominant aroma observed.

This *Oidium* was used as an auxiliary ferment for the production of heavy rums from sugar cane juice in two different ways: (*a*) a sterilized sugar cane juice mash of from 12.0 to 15.0 per cent total sugars was inoculated firstly with the *Oidium* culture. After the *Oidium* film was formed on the surface of the medium it was allowed to act upon it for a period that could vary between 24 and 72 hours or more if desired. Then the mash was inoculated with an active footing of yeast No. 764, and fermentation was carried to completion. (*b*) In the second method the yeast was allowed to operate alone in the substrate, and towards the finishing of the alcoholic fermentation the *Oidium* culture was inoculated. The *Oidium* fermentation was allowed to act then for variable number of hours, as desired.

Both methods worked satisfactorily in the creation of a new variety of heavy rum out of sugar cane juice mashes; but the rums obtained differed somewhat in each case, those produced by method (a) being of intenser taste and higher aromatic tone.

Table 18 presents data of fermentation results obtained through the use of the two methods outlined above, and also presents analytical figures on the chemical composition of the respective raw rums produced in each case.

Other types of moderately heavy rums may be prepared either from blackstrap molasses or from *raw* sugar cane juice by selecting a heavy rum yeast strain to effect the fermentation, and using rather high fermentation pH values, low temperature of fermentation and rather high total sugars concentration. If sugar cane juice is used, this must be used in its fresh, raw state, to secure best results.

TABLE NO. 18

FERMENTATION RESULTS OBTAINED WITH CANE JUICE AS SUBSTRATE AND YEAST STRAIN No. 764 AND OIDIUM SUAVEOLENS AS FERMENTATION AGENTS. ALSO ANALYSES OF THE RESULTING RAW RUMS

)0mL	u	ion	e Hrs.)0mL		rs, %	sy %	', %	%	AN	ALYSES	OF THE I	RAW RU	JMS
Oidium fermentation before yeast fermentation	Fermentation No.	Setting Brix	Setting pH	Setting T. Sugars grs 100mL	Hours of Yeast action	Hours of Oidium action	Total Fermentation Time Hrs.	Final Brix	Final pH	Residual Sugars, grs. 100mL Beer	Attenuation	Yield Alcohol on Sugars,	Fermentation Efficiency	Distillation Efficiency, %	Over-All Efficiency,	Alcohol by Volume %	Total Acidity Mgs/100mL Abs. Alc.	Aldehydes, Mgs/100mL Abs. Alc.	Esters, Mgs/100mL Abs. Alc	Higher Alcohols Mgs/100mL Abs. Alc.
	1	15.5	6.00	13.96	42	24.0	86	0.5	4.58	0.46	15.0	41.46	85.39	97.0	82.82	82.88	18.2	83.5	172.0	152.9
	2	16.7	5.75	15.17	78	48.0	126	1.2	4.80	0.91	15.5	37.49	77.22	98.0	75.67	83.33	11.9	67.2	119.3	168.5
	3	15.6	5.78	13.23	48	72.0	120	2.2	4.87	1.16	13.4	37.35	76.93	95.5	75.00	81.32	18.7	53.4	75.8	148.2
	4	16.9	6.09	15.27	96	24.0	120	2.8	4.51	1.36	14.1	38.46	79.22	98.0	77.62	83.31	17.4	69.5	166.7	194.0
	5	16.2	6.00	14.98	72	24.0	96	1.00	4.60	0.77	15.2	40.90	54.28	97.0	81.74	82.39	17.2	63.6	171.6	188.5
	6	14.3	5.37	12.78	72	54	126	0.00	4.26	0.14	14.3	45.82	94.38	97.5	91.91	81.88	9.2	75.6	112.8	298.9
ion	7	12.9	5.46	11.84	26	82.0	108	0.00	4.50	0.60	12.9	43.03	88.63	98.0	86.85	79.80	13.3	100.5	113.5	325.9
	8	12.0	5.53	11.20	46	24.0	70	0.50	4.73	0.60	11.5	44.80	92.28	97.0	89.51	80.74	10.0	81.3	90.4	247.0
	9	12.0	5.46	11.34	45	48.0	93	0.30	4.80	0.60	11.7	44.05	90.73	97.0	88.00	80.84	6.9	94.6	88.2	293.9
	10	12.7	5.34	11.90	36	24.0	60	0.00	4.77	0.18	12.7	45.33	93.37	98.0	91.49	82.35	7.2	65.2	66.3	191.2

106

2. Light Rum Fermentation:

The fermentation of a genuine light rum is not so easy in practice as it seems. This we corroborated during our consulting practice when it was observed that most distillers were trying to manufacture the light types not through adequate fermentation technique, but simply by distilling the beers as is done in the case of industrial alcohol, that is, under high rectification and purification; and at very high proofs (between 183-189 P.). In other words, the idea was the imitation as close as possible of industrial alcohol distillation, and the result was neither rum nor alcohol; but an intermediate product, too well purified to be called rum, and not pure enough to be called alcohol. The artificial conversion of this product into rum was then left in the hands of the various rectifiers.

When the author objected to this "modus operandi" they retorted that there was no other way in which they could manufacture a "light rum". Since our conception of a light rum was not exactly that of an impure industrial alcohol, we resolved to find means of so selecting our yeasts and conducting our fermentations that a "light rums" could be the end product of distillation without resorting to such practices.

From what has been previously commented and explained, it may be recalled that conditions for "light rum" fermentation. Hence, in a general way a "light rum" fermentation should be conducted under the following conditions:

(1) The selected yeast should be a moderate producer of congeneric products of alcoholic fermentation, such as organic acids, aldehydes, esters, higher alcohols, essential oils, etc., etc.

(2) The selected yeast should be a fast fermenting organism.

(3) The substrate in which fermentation takes place should be previously well conditioned and purified.

(4) The building up of the seed yeast should be done under strict aseptic conditions. The footing should be free of all contaminating organisms when pitched into the fermenter.

(5) The footing should have developed the highest possible concentration of active, vigorous yeast cells by the moment it is used; and the largest feasible volume of this footing should be used as inoculum of the fermenter.

(6) The fermenters should preferably be of the closed, aseptic type; and should be provided with efficient cooling device.

(7) The fermented mash should be freed of all suspended solid impurities, included the yeast cells, before delivering it to the distillation end of the distillery. A close-type supercentrifuge is the most adequate filtering device to effect this work.

(8) Moderate temperatures of fermentation should be maintained.

(9) Rather low pH values, in the range 4.5-5.0 pH, should be used.

(10) The total sugars concentration should never exceed the optimum found for the given strain.

(11) Sufficient and well-balanced yeast nutriments should be used so that no retardation of the fermentation lapse may be occasioned by poor conditions of nourishment.

(12) Activators, such as the various carbons and other neutral substances used for the purpose of stimulating the yeast action may be employed.

In the course of our work it was found that a fast fermenting, budding bottom yeast was the most suitable for the production of this type of rum. If possible, complete sterilization of the substrate should be employed during mashing operations. It is very important in the case of "light rum" fermentation that bacterial intervention be excluded from the process in an efficient and reliable way. A contaminated substrate will hardly yield a light type of rum, when the beers are distilled as they should. This was perhaps the main reason why our distillers could not perform rum distillation in its proper sense and meaning and still obtain a type of light rum. Not a single distillery of those visited by the writer was operating under aseptic conditions of fermentation.

Lack of proper supervision, and chemical and biological control during the building up of the seed yeast used as footing was another factor adversely influencing the genuine production of veritable "light rums" Most distilleries worked their footings on the basis of percentage volume on the capacity of the main fermenter without effecting microscopic examination for contaminants, or using cell counts per unit volume as the real inoculating value of a footing. Differentiation between dead or weakened yeast cells and the young, vigorous, active ones was never employed. Most times the seed yeast was already badly infected, and the cell concentration very low by the time the fermenter was reached. Under conditions such as these there is no wonder that recourse had to be taken during the distillation stage to remedy as far as possible all these ills.

Taking great precautions in the obtention of a healthy, pure, vigorous footing, and then pitching it into a badly contaminated fermenter will not help matters to a great extent. Manufacturers of "light rums" should adopt the closed, aseptic fermenter for best results; fermenters able to stand 10 or 15 pounds of steam pressure without risk of injury to themselves or the personnel attending them. In practice, less than those steam pressures above-mentioned are required for sterilization, from five to eight pounds gauge pressure being sufficient in most cases. Open fermenters are hard to keep under aseptic conditions, and if made aseptic, this state will not last for very long, due to the continuous peril of contamination from the environmental factors peculiar to all tropical distilleries.

As soon as the fermentation finishes, the beer should be distilled in this case, since light rums are not supposed to have strong aroma. In order that the beer be delivered as free as possible from suspended solid impurities, filtration in bulk through a supercentrifuge becomes necessary. This will help in a great measure to eliminate rank odor from the distillate; as our experiments have proved that the greater source of rankness in recently distilled spirits comes from the distillate; as our experiments have proved that the greater source of rankness in recently distilled spirits comes from the overheating of these solid impurities in the beer on the sides and plates of the distilling column, or in the kettle of the pot still.

Moderate temperatures of fermentation, rather low pH values, and optimum total sugars concentrations are recommended for reasons that have already been explained before when a short period of fermentation is desired; as light rums should be fermented as rapidly as feasible.

Activating carbons such as Darco-D4 and others are seldom necessary when the precautions regarding yeast selection, fermentation temperature, pH, sugars concentration, and the other factors leading to a rapid fermentation have been attended to. But these activators become necessary sometimes when for one reason or other the fermentation tends to become sluggish. Condensing the requirements for light rum fermentation in a few sentences we have that the conditions needed are:

(a) An appropriate yeast strain, preferably a bottom fermenting budding yeast.

(b) A period of fermentation controlled by the will of the distillers.

(c) Operating as nearly close to pure culture fermentation technique as possible.

Table 19 presents data on the fermentation of light rums, as well as analyses of the resulting rums.

It will be noticed that yields on total sugars are exceptionally good in all cases, but that they grow better as the total sugars concentrations become less, as in samples 9, 11, 13, and 14. The small difference in pH values among mashes and resulting beers, shows that the fermentations were carried under aseptic conditions. The fact that mashes of higher total sugars values were fermented in less time than others of lower total sugars values is explained by the differences in non-sugars among the molasses used for mashing. Samples Nos. 1 to 8 were prepared with a higher sugars, lower non-sugars containing molasses, as compared with samples Nos. 9 to 14.

The analyses of the different raw rums distilled from the fermented mashes show the characteristic qualities of light rums, even when they were not distilled under high rectification nor at very high proofs: low total acidity, aldehydes, esters and fusel oil. Great uniformity in the individual analytical figures will also be noticed.

3. The reduction of the Fusel Oil Fraction during Fermentation:

In the second chapter of this bulletin, when treating of yeast selection it was shown that different strains would produce different amounts of higher alcohols (fusel oil) during fermentation; and then we offered to further treat on the fusel oil question in this chapter on rum fermentation.

Fusel oil is the term by which is commonly designated a mixture of higher alcohols produced during alcoholic fermentation, in which the amyl alcohol predominate, but where other higher alcohols are also found in less amounts.

Apart from the nauseating odor and taste that a high percentage of these bodies will impart to the rums of commerce, it has been thought for a long time that fusel oil also exerts a destructive action on human health. Recent studies on the matter have shown, however, that the deleterious action of fusel oil on the human anatomy is in no way superior to that produced by ethyl alcohol itself; and that since ethanol is the principal constituent of alcoholic beverages, most probably the detrimental results attributed to fusel oil really originate in the ethyl alcohol itself. Be that as it may, the fact stands that the present tendency in the rum trade is towards the elimination of fusel oil, or at least, towards a great restriction of its presence in commercial rums.

TABLE NO. 19

SHOWING RESULTS OBTAINED IN LIGHT RUM FERMENTATION AND ANALYSES OF THE RESULTING RUMS. THE PASTBUR EQUATION FOR ALCOHOLIC FERMENTATION WAS USED IN THE CALCULATION OF FERMENTATION EFFICIENCIES

									L beer			eer	%	, %	ANALYSES OF THE RAW RUMS					
Fermentation No.	Setting Brix	Seeting Temp. Deg. C	Setting pH	Total Sugars Grs/100mL mash	Ferm. Period Hours	Final Brix	Final pH	Ave. Ferm. Temp. C.	Residual Sugars, grs/100mL	Attenuation	Alcohol by Volume %	Grams alcohol 100mL beer	Yield alcohol on sugars,	Fermentation Efficiency,	Alcohol by Volume, %	Total Acidity Mgs/100mL Abs. Alc.	Aldehydes, Mgs/100mL Abs. Alc.	Esters, Mgs/100mL Abs. Alc	Higher Alcohols Mgs/100mL Abs. Alc.	
1 2 3 4 5 6 7 8 9 10 11	23.5 24.0 24.0 23.5 25.0 25.0 25.0 25.5 25.0 25.0 25.0 25	28.5 27.5 29.0 30.0 29.0 28.5 29.0 30.0 26.0 26.5 26.0	4.76 4.78 4.80 4.77 4.9 4.75 4.80 4.82 4.80 4.82 4.80	15.28 15.43 16.43 15.20 16.12 16.40 16.25 13.77 14.40 13.52	30.0 27.0 36.0 27.0 32.0 32.0 32.0 32.0 48.0 45.0 36.0	8.0 8.2 8.5 9.0 8.5 9.0 9.0 9.80 9.50 9.50 9.50	4.59 4.57 4.70 4.65 4.60 4.62 4.59 4.68 4.51 4.43 4.50	30.0 30.5 31.0 32.5 31.5 31.0 32.0 33.0 30.0 29.2 28.5	0.50 0.48 0.52 0.78 0.61 0.60 0.49 0.81 0.42 0.60 0.39	15.5 15.8 15.5 15.0 15.0 16.0 16.0 16.0 15.7 15.5 15.5 15.5	8.72 9.54 9.13 9.48 8.96 9.54 9.96 9.03 8.65 8.32 8.32	6.99 7.53 7.20 7.48 7.08 7.53 7.86 7.12 6.83 6.57 6.55	45.80 48.70 46.60 45.50 46.60 46.7 48.0 43.8 49.60 45.65 48.48	94.50 100.50 96.10 93.80 96.10 96.25 98.80 90.90 102.10 94.00 99.75	80.00 79.45 79.91 80.50 78.41 79.90 81.00 78.25 77.77 80.50 78.10	7.50 6.90 8.10 9.14 7.90 8.25 7.49 10.40 7.80 9.99 8.50	$18.20 \\ 22.90 \\ 21.70 \\ 24.60 \\ 21.12 \\ 22.30 \\ 20.90 \\ 25.10 \\ 21.90 \\ 22.08 \\ 23.10 \\ 21.5$	42.10 45.80 51.12 49.80 52.90 51.30 49.50 48.90 52.00 50.80 45.25	65.80 71.08 64.40 70.90 68.60 72.10 71.10 77.14 71.90 73.90 69.82	
12 13 14	25.5 24.50 24.0	28.0 26.0 26.0	4.72 4.73 4.76	14.19 13.78 13.27	46.0 38.0 33.0	9.00 9.00 8.70	4.49 4.48 4.50	29.0 29.0 30.0	0.41 0.34 0.31	15.5 15.5 15.3	8.55 8.58 8.25	6.75 6.77 6.51	47.70 49.20 49.15	98.30 101.20 101.10	79.80 81.10 78.90	9.10 8.80 7.99	21.50 20.97 19.84	46.70 45.10 42.21	65.20 62.80 61.10	

With this idea in view rum distillers have tried to reduce as much as possible the fusel oil content of their respective products by the easiest way, that is, by effecting this separation during the distillation of the product. But in practice this have proved to be of more difficult accomplishment than originally thought. The difficulties arise from two sources: (*a*) most rum stills have no provisions for fusel oil elimination; (*b*) when the design and mechanical make-up of the distilling unit includes such provision, the difficulty still arises that when trying to eliminate the fusel oil, other valuable constituents of a genuine rum are also simultaneously eliminated. Among these other valuable constituents that the distiller would rather keep in his product are some very important esters and part of the "rum oil".

In view of this situation we endeavored to work out a special fermentation method that would accomplish the desired reduction in the fusel oil fraction of the rums without losing other valuable and desirable constituents of a veritable rum. The method devised was of a preventing its formation, to a great extent, during the fermentation period. By this methods, the distiller has not to preoccupy himself with the problem of eliminating the product during distillation, nor with the losses of other valuable, congeneric products; since the amounts of fusel oil formed will not interfere with the quality of his commercial rum.

Three capital points form the basis of our process:

(*a*) Selection of a yeast which is a natural poor producer of fusel oil. The data presented in the chapter on yeast selection show that this is practicable.

(*b*) Maintenance of low temperatures of fermentation. This is also perfectly feasible, if artificial cooling of the fermenting mash is resorted to.

(c) The use of ammonium hydroxide solution as nitrogenous feed for the yeast during fermentation.

When in the experiment described in the chapter on yeast selection various yeast strains were tested on their fusel oil producing power, we found (table 4) that those strains which resulted the lowest producers, and corresponding amounts produced, where:

	Mgs. fusel oil produced
Yeast Strain No.	per 100 ml Abs. Alc.
500	
501	
503	
504	

It will also be remembered that these amounts of fusel oil were produced at temperatures of fermentation around 35 degrees Centigrade. When a second experiment was effected with these same yeast strains, but using a temperature range within 25-27 degrees C., the following different results were obtained:

	Mgs. fusel oil produced
Yeast Strain No.	per 100 ml Abs. Alc.
500	
501	
503	
504	

When in a third experiment ammonium hydroxide solution in the extent of 0.1 per cent by volume of mash was used as yeast nitrogenous nutrient, besides keeping the same temperature range of fermentation as in the previous experiment, that is 25-27 degrees Centigrade, the results obtained were:

	Mgs. fusel oil produced
Yeast Strain No.	per 100 ml Abs. Alc.
500	
501	
503	
504	
A fourth or animant was then on	dusted This time liquid summaris feeding

A fourth experiment was then conducted. This time liquid ammonia feeding was used, but the temperature of fermentation was kept within the range 33-35 degrees C. The results obtained were as follows:

	Mgs. fusel oil produced
Yeast Strain No.	per 100 ml Abs. Alc.
500	
501	
503	
504	

The data given above shows that during fermentation the fusel oil content of rum may be materially lessened if liquid ammonia feeding of the yeast is practiced, even when the fermentation is carried at the ordinary prevailing temperatures, that is, well above 30 degrees C. But if at the same time the temperature range of fermentation is maintained within 25-27° C. then further reduction of the fusel oil content in rums will be effected.

Table 20 presents other fermentation results obtained when comparing: (1) liquid ammonia feeding vs. natural nutrients found in molasses mash; (2) liquid ammonia feeding vs. natural nutrients found in sterilized sugar cane juice; (3) liquid ammonia feeding vs. feeding with ammonium sulphate, in sugar cane juice and molasses mashes. The analyses of the resulting rums are also given in each case. In this experiment was used a yeast strain which proved to be a heavy producer of fusel

oil when working under uncontrolled conditions. A drastic reduction in the contents of fusel oil will be observed in all cases where liquid ammonia was used as nitrogenous feed for the fermenting yeast. Also a great reduction in the period of fermentation is noticed in favor of the liquid ammonia-fed cultures. Slight increases in ester contents are also found.

4. Constant pH Fermentation for Light Rum

It has been explained in the chapter on yeast selection that the mildness of taste and suavity of aroma in a given rum are dependent firstly on the content of rum oil, and secondly not so much in the quantity, but rather on the quality of the esters it contains. Guided by these principles we have worked the following methods to be used especially for the production of very mildly tasting, delicately scented rums of the light type.

The substrate used may derive from either sugar cane juice or molasses. If sugar cane juice is used, it must first be pasteurized for at least one hour at temperatures between 65 and 80 degrees C. With molasses, the usual pretreatment as described in the chapter on mashing methods is sufficient.

The mash is prepared in the usually way, care being taken that the pH value is carefully adjusted to a reading within the range 5.8-6.0 pH. After the pH value has been accurately determined and adjusted, one gram of sterile calcium carbonate is added per liter of mash, *without* stirring of the liquid, so as to avoid changes in its pH. It has been found that the addition of this amount of sterile calcium carbonate without agitating the contents of the fermenter will not change the preadjusted pH value to any appreciable extent; the carbonate added settles immediately at the bottom of the fermenter. The fermenters are provided with mechanical stirrers to be used only in cases of necessity.

After the addition of the calcium carbonate, the pH of the footing that will serve as inoculum is adjusted to about the same value as that of the main mash, and is added to the fermenter without stirring. Fermentation is then allowed to continue until its finish. During the fermentative lapse samples are drawn for determinations of: (1) Brix; (2) alcohol; (3) total sugars; (4) pH values; and (5) titratable acidity. Graphs are drawn with the analytical values so determined.

TABLE NO. 20

SHOWING FERMENTATION DATA AND ANALYSES OF RESULTING RUMS WHEN COMPARING: (1) LIQUID AMMONIA FEEDING OF THE YEAST vs NATURAL NUTRIENT OF MOLASSES MASH; (2) LIQUID AMMONIA FEEDING vs NATURAL NUTRIENTS IN SUGAR CANE JUICE MASH; (3) LIQUID AMMONIA FEEDING vs. AMMONIUM SULPHATE FEEDING IN MOLASSES AND IN SUGAR JUICE MASHES

		es Mash		e Juice Mash		es Mash	Sugar Cane Juice Mash		
	Check	Liquid Ammonia Feeding	Check	Liquid Ammonia Feeding	Ammonium Sulphate Feeding	Liquid Ammonia Feeding	Ammonium Sulphate Feeding	Liquid Ammonia Feeding	
Fermentation Time, Hrs Initial pH Initial Brix Initial Sugars, grs/100mL Mash Final pH Final Brix Residual Sugars, grs/100mL beer Yield Alcohol on Total Sugars, %	88.0 5.0 21.0 12.5 4.2 6.6 0.7 45.9	42.0 5.0 21.0 12.5 4.8 6.2 0.4 47.0	$102.0 \\ 4.9 \\ 15.7 \\ 14.1 \\ 4.3 \\ 0.0 \\ 0.6 \\ 46.7$	60.0 4.9 15.7 14.1 4.7 0.0 0.2 48.1	72.5 5.0 22.0 13.2 4.5 7.7 0.8 46.9	44.0 5.0 22.0 13.2 4.9 7.1 0.5 47.1	86.0 4.8 16.0 14.5 4.4 0.0 0.48 47.40	58.0 4.8 16.0 14.5 4.7 0.0 0.14 48.50	
ANALYSIS OF THE RUMS Alcohol by Volume, % Aldehydes, mgs/100mL Abs. Alc. Esters, mgs/100mL Abs. Alc. Higher Alcohols, mgs/100mL Abs. Alc.	75.5 22.3 88.8 106.5	- 76.7 19.7 107.9 122.9	73.8 28.5 81.03 313.7	- 75.4 20.9 102.6 107.2	81.65 19.12 113.9 165.1	- 82.46 15.40 137.9 109.7	- 80.08 21.11 68.4 252.9	- 82.28 18.10 102.3 111.2	

FERMENTATION TEMPERATURE RANGE 27-29 DEG. C.

so determined. One of these graphs will represent variations in titratable acidity and pH values during the course of fermentation; and another will represent the rate of sugars destruction and alcohol formation.

It has been found that the added calcium carbonate will begin to react as soon as the pH of the mash is lowered appreciably from the setting value, maintaining in this manner a practically constant pH value during the whole course of fermentation. If necessary (which is seldom the case) the action of the neutralizing agent may be helped by use of the mechanical stirrer.

In this method of fermentation a very fine product is obtained as already stated. This is due to the increased production of rum oil by the yeast at the higher pH value, in the first place. Secondly, we have that the different organic acids already present in the substrate are neutralized by the calcium carbonate in the order of their respective chemical activity and affinity; the same procedure taking place with those other acids produced by the yeast during fermentation. In this guise, the more reactive lower members of the fatty series, such as formic and acetic, will be fixed in the form of their calcium salts far more readily than the less reactive higher members such as propionic, butyric, caproic, heptoic, etc. etc. This gives an opportunity to the higher normal fatty acids to react with the alcohols produced during fermentation, forming very valuable esters. Also, during distillation, the acrid tasting formic and acetic acids will be in a large measure left in the slops of the form of their calcium salts. At this stage those higher acids that remained in the free state during the fermentation period will find another opportunity to react with the alcohol vapors formed in the distilling column. As the temperature is quite high and both organic acids and alcohols are in their vapor phase, conditions of further esterification become ideal, especially if total refluxing is used during a certain period.

We have found the distillate obtained from this special method of fermentation, to be an exclusive product with a seal of excellence very difficult to imitate. Unfortunately, actual distillery tests using this method had to be abandoned due to the difficulty in obtaining aseptic conditions in the fermenting room. As already explained any method in which a pH favorable to bacterial life and reproduction is created, without having the means to protect the yeast from the attack of these bacteria, will fail to produce a light type of rum through purely and exclusively fermentative technique.

5. Multiple Yeast Culture Fermentation:

In our chapter on yeast selection the assertion was made that the ideal rum yeast strain was very difficult to find or obtain. This difficulty may be remedied to some extent by the use of supplementary strains during fermentation, thus obtaining the desired results by the use of multiple yeasts instead of a single individual. One desired characteristic will come from a member, and another from another member of the mixed culture, and by this method results may be obtained that would be unattainable through the use of a single ferment.

In the laboratory we have used combinations of as high as four different strains with excellent results. Usually, however, two strains, well selected for the required end are sufficient. There are two different ways in which this method may be used in actual distillery practice:

(1) The different strains are used to pitch different fermenters and after fermentation is over the respective beers are mixed in the desired proportions before feeding them to the distilling unit. From a biological point of view this would be the easier way out; but from an economic and mechanical aspect, this procedure could hardly be recommended. In the first place duplicate yeasting equipment becomes necessary, both in the laboratory and at the distillery; more labor involved; and more equipment, such as extra tanks, pipe lines, pumps, motors etc. etc., would be required. Complication of equipment, and extra expense in supervision and labor are apparent.

(2) In the second method we find that the economic and mechanical drawbacks of the first procedure are totally obviated; but this time the success will depend on the skill of the fermentologist or bacteriologist in the distillery. In this case the different strains are included in a single mixed culture by the time they leave the laboratory. Hence no modifications or extra equipment are needed at the distillery. The real difficulty with this procedure is to keep a definite proportion of each strain used in the mixed culture. This problem is not easy at all; in fact it is quite difficult. Only through a thorough knowledge of the characteristic of each one of the strains

used, their respective virulence, power of reproduction, rate of multiplication, resistance to adverse conditions, respective individual products of metabolism other than ethyl alcohol, etc. etc. may the fermentologist or bacteriologist in charge, keep a certain ratio of influence on each individual strain in the formation of the taste and bouquet of the resulting rum. At any rate, the keeping of unchanging aroma and taste in the final product becomes a problematical and uncertain matter.

Before closing the subject of rum fermentation it is well to treat of certain phenomena that may occur during this stage of manufacture, especially in small distilleries where no chemical or biological control is practiced. To all outward appearance, fermentation is considered finished when the characteristic agitation coincident with gas generation within the fermenting liquid is no longer apparent; and in most cases this indication is sufficient. But there are cases in which this appearance of the fermenting vat only indicates that fermentative activity has been merely arrested, but it does not necessarily follow that fermentation has been completed in the sense that the optimum breakage of sugar molecules has taken place, with the subsequent formation of the corresponding amount of alcohol and other congeners of fermentation. Fermentation may, in fact, come to an apparent end while considerable available sugars are yet unconverted into alcohol and other products of the metabolism of yeast cells. The causes of this quite common happening are many: premature exhaustion of the elements necessary to yeast nutrition; abnormal rise in temperature, resulting in the inhibition of the fermenting organism by the alcohol-temperature complex; inadequate ratio of non-sugars to sugars; weakening of the zymogenic power of the yeast through the effect of contaminations that existed in, or may be have gained access to, the fermenting liquid, and alterations of the pH of the substrate through the agency of these contaminants or poisoning of the yeast by the products of metabolism of these infecting organisms, etc.

The most familiar and common causes of this phenomenon, however, are exhaustion of yeast nutriments in the fermenting mash, and the effect of the alcoholtemperature complex. In the first of these cases, addition of nutrients, especially ammonium salts of phosphoric acid, will cause fermentation to start anew. In the second case, some means of rapidly lowering the temperature of fermentation will greatly help the situation. The addition of some solid inert matter in a finely divided state will increase the good effect obtained from a lowering of the temperature alone. A more rare phenomenon is sometimes met with: The liquid mass in the fermenter is apparently undergoing fermentation as shown by its agitation, gas generation, and foaming; but it is found that the readings of the Brix scale on samples taken at periodical intervals of time do not show appreciable attenuation. These cases are almost invariably originated by the presence of certain bacteria capable of maintaining the fermentation of the medium after they have inhibited alcoholic fermentation: As generally these contaminants produce chemical substance differing but little in specific gravity from that of water, observations made for drops in density of the fermenting liquid no longer show the inherent and characteristic differences observed when large quantities of alcohol are being formed. In such cases the first thing is to determine the nature, and especially the source, of the contamination for future prevention.

As to the already contaminated fermenter, if the plant equipment allows, it should be sterilized by heating to the required temperature and then reinoculated with a footing from another fermenter undergoing active fermentation. If the plant is not equipped for this procedure the best next recourse is to lower the pH value and the temperature of the mash to the lowest limit at which the yeast may operate efficiently.

This lowering of the temperature and of the pH value of the medium will in some cases check the advances of the infecting bacteria, with the possible saving of the fermenter.

VI

Rum Distillation

In a general way, distillation consists in separating more of less completely, a volatile liquid from fixed or less volatile substances with which it may be associated. In the case of rum, the fermented mash or wort contains decanted and suspended solid impurities, most of which are of nitrogenous nature (yeast cells, yeast gums, albuminoids, pectic substances); fixed soluble substances (organic and inorganic salts, organic acids); a whole series of volatile liquid substances (alcohols, free organic acids, aldehydes, esters, essential oils etc. etc.); and dissolved gases, especially carbon dioxide. In infected mashes, hydrogen, and gaseous compounds of sulfur are frequently found, due to bacterial action and to the presence of sulphate and sulphites in large quantities. Some of the bacterial groups, and especially the butyric acid group exert a powerful reducing action on sulfates and sulphites with the production of hydrogen sulfide and other gases.

All of the above-mentioned constituents of the fermented mash exert, or may exert, their influences during distillation, and some of the volatile ones may show their presence to a smaller or greater extent in the raw distilled liquor. Of these, some will enhance, while others detract, the taste and aroma of the raw distillate. Although from the above description of the constituents of the wort, one may judge of its complex nature, it is mainly composed of water and ethyl alcohol; the numerous other constituents appearing in very small quantities. The volatile ingredients mixed with the ethyl alcohol and water vary considerably in volatility, most of them being less volatile than ethyl alcohol; exceptions to this are some of the esters and aldehydes. They exist in very minute quantities, in some cases mere traces; but in rum making they are of the greatest importance, for the inherent body, aroma and taste that differentiates rum from alcohol are largely attributable to them. The decanted and suspended nitrogenous impurities, as well as some of the fixed substances that are easily decomposed by the action of heat will also exert their influence upon the nature of the raw distillate, and in this case in an adverse manner. The rankiness ("tufo" in Spanish) frequently noticeable in freshly distilled rums owes its origin to a very large extent to the decomposition during distillation of some of these nitrogenous elements of the fermented mash. We shall discuss this important matter further.

Among the volatile substances, we are principally concerned, besides ethyl alcohol, with a series of organic acids (formic, acetic, propionic, butyric, valeric, caproic, heptoic etc.); aldehydes, (especially acetaldehyde, propylaldehyde, butylaldehyde, heptaldehyde etc.); higher alcohols (as propyl, butyl, amyl, hexyl, heptyl, etc.); and esters resulting from the chemical reactions between the alcohols and organic acids. A number of essential oils are also present, of which the one known as rum oil constitutes one of the most valuable and important ingredients in the formation of the bouquet of a truly genuine rum.

Applied then to rum manufacture, the distillation stage will have for its objective the separation from the fermented mash of the ethyl alcohol together with desirable congeneric bodies and some of the water; leaving behind those bodies that may adversely influence the desired characteristic sought in a rum of quality. Rum distillation is thus a process of *selective extraction*, differing a great deal from distillation for the production of industrial alcohol, where total elimination of congeneric products with production of the purest possible ethyl alcohol is the aim in view. The care and art with which this selective process is effected during rum distillation constitutes a factor of paramount importance in the quality of the resulting rum. In this respect, the ideal still for rum production has probably not been designed yet.

The mixture of ethyl alcohol, water, and desirable congeners obtained from this selective distillation of the wort is what is called the raw rum. What class of distilling apparatus is the best for rum extraction from the wort? This question is not always so easily answered without a previous thorough survey of conditions and peculiarities inherent to each particular case, and above all, without an exact knowledge of the nature of the desired end product. In actual practice there are, however, at least three different general ways in which to effect this separation: (1) Through a series of two or more successive simple distillations, that is, using redistillation as a means of rectification. The simplest forms of distillation outfits with some sort of dephlegmatory device, will serve this purpose. (2) By means of a discontinuous, or batch still, provided with a good rectifying column and a total reflux condenser between this column and the final condenser. (3) By means of one of the many models of continuous stills in the market, adapted as far as possible in its general design to the distillation of rum. This is the type most generally employed for large scale production, not so much because of better quality of end product as for compactness, fuel saving, and other economical advantages.

The first method of these three mentioned above is almost obsolete in commercial practice, except for very small scale production of a very high quality product. But we must mention this primitive process for the very fine rums that maybe obtained through its use under adequate technique of operation. There is nothing strange or mysterious in this assertion, if it is remembered that the genuine cognac of France, that exquisite liquor, rarely obtained unadulterated in the trade, which is produced in the region of Charente, is practically all manufactured by this simple method of distillation. We shall never tire of repeating that there exists, by far, more points of similarity between rum manufacture. An expert maker of brandies will in a short time become equally expert in the production of rum. The sooner this basic different between industrial alcohol and rum methods of production is grasped and assimilated by the minds of rum distillers, the sooner they will begin to manufacture products of real quality, and the many cordials now being labeled and sold as genuine rums will then disappear from the market.

Coming back to our methods of rum distillation, we find that the great drawbacks to this first method are the excessive floor space and expense of heat and time necessary to obtain the desired finished product.

The second general method of rum distillation, that employing a batch still with rectifying column, is not used extensively in continental United States, Cuba or Puerto Rico, but it is common in Jamaica and other Islands of the West Indies. The use of this type of discontinuous distillation is much to be recommended for the production of the highest quality product.

In our opinion, and according to our experiments with batch and continuous stills, the use of the batch type of still becomes almost obligatory when the best types of heavy rums, more than ever, rum distillation becomes a selective process, and the batch still is by far more suited to effect this selective action than any other means of distillation. A crude, poor quality of heavy rum may be distilled in a continuous still – this we accept; but when a product of this type, possessing the true marks of excellence, suavity of taste, and delicate yet persistent aroma is required, then recourse must be had to the discontinuous still. We have explained already in the previous chapter how attempts have been made in the past and are being made at present to produce a first class heavy rum in continuous stills and how and why these attempts have met almost invariably with failure. The main cause of these failures has been the lack of provision for selective performance in the rum continuous stills manufactured at present. Of course, like almost any other thing under the sun, the discontinuous method of distillation has both advantages and disadvantages: advantages as regards quality of product, simplicity of construction, easy of manipulation, and flexibility of operation upon the principle of selective distillation; the last-mentioned advantage, and that of quality of product, being the two more important. Its disadvantages are all of an economic nature: as smaller distilling capacity per individual unit, greater space requirements, less compactness and promptness of operation,

and, above all, greater expenditure of both time and fuel for similar production volume as compared to continuous stills.

The third general method of rum distillation, that employing the continuous system, is used almost exclusively in Puerto Rico with perhaps one of two exceptions where a combination of both system, the continuous and the discontinuous, are used. Continuous stills especially constructed for the purpose may be used to advantage in the distillation of the so-called "light rums". It is very desirable, however, that this still be provided with an auxiliary column known as the pasteurizing unit, for the production of the best rums. While the main column of a rum still has practically no control over the product other than its degree of proof, in the pasteurizing column the chemical composition of the rum is controlled quite accurately and conveniently. The pasteurizing column is really a purifying column as well, for the main work done here during distillation consists in the separation of certain constituents of the raw spirits and also in the formation of further aromatic bodies through esterification. For the better performance of this type of rum still, the alcoholic liquid entering the auxiliary pasteurizing column from the condenser of the stripping or main column, should not be of too high proof, for in this case some of the most valuable constituents of a veritable genuine rum will be lost. The rectifying section of the main column should consist of but few plates (from four to five), so as to deliver a distillate to the pasteurizing column with a proof varying between 130 and 160 degrees, under normal working conditions. The degree proof of the distillate delivered by the main distilling column to the pasteurizing column will vary according to the degree of reflux carried during distillation; but the range abovegiven will be found to represent normal working conditions.

The principal cause of failure in most rum continuous stills to produce real high quality, genuine rum, is that this system lacks provisions for attaining that high degree of fractionation necessary to separate the desirable from the undesirable components in the wort under treatment. The usual types found in Puerto Rico (with some exceptions) either must allow both desirable and undesirable components to pass mixed together in the raw distillate, or suppress altogether both of these different types of congeners, the desirable and the undesirable. In other words, selective extraction of the volatile constituents of the fermented mash cannot be efficiently practiced. Herein lies the great advantage of the discontinuous type of rum still, where effective discrimination between these different constituents may be easily and simply effected. Hence, in the case of continuous distillation, the raw rum as it comes from the final condenser or refrigerating unit, will always contain a variable quantity of undesirable products, while at the same time some of the most valuable products, as for instance rum oil, are lost in a great measure. When an attempt is made to eliminate the passing over these undesirable and the undesirable products are equally eliminated, and the resulting raw distillate ceases to be a rum in the strict sense of the word, to become an industrial alcohol of poor quality.

The continuous still has nevertheless many advantages; and no doubt, as the rum industry develops and becomes a really great and important one, the manufacturers of distilling apparatus will endeavor to design a special continuous unit for rum making that will be a decided improvement on what we have at the present time. Already the existence of the pasteurizing column is a conspicuous improvement. The continuous still is admirably constructed for bulk, mass production; and due to this particular advantage it is in great demand among the large rum producers. Its economic advantages in compactness, floor economy, high unit output, and the great saving of time, manipulation, and fuel, are apparent and undeniable. But as to real quality of the finished product, the advantage is at present on the side of the small producer using the discontinuous system, on other equal methods of fermentation and curing of the product.

The inconvenience for large distillers to use the batch still may be partly solved by a combination of the two systems, that is, distilling part of the beer in discontinuous and a greater portion in continuous stills. Judicious mixings of the two distillates will greatly enhance the quality of the resulting rums without losing altogether the economic and mechanical advantages of the continuous system. One of our clients has followed this system with great success. The writer, in his consulting work, has always advocated the use of the discontinuous system to the small distilleries (those producing between 500 and 1,000 proof gallons of spirits in 24 hours), and the combination of both systems mentioned above, to the larger installations.

During our studies in rum distillation we had our own batch still built by the Lummus Company of New York, and fortunately had also access to a continuous still devoid of auxiliary pasteurizing column, installed at the Laboratory of Agricultural Industries of the Department of Agriculture and Commerce of Puerto Rico; and of another continuous still provided with pasteurizing column at the "Borinquen Associates Distillery" of Hato Rey, Puerto Rico, one of the distilleries whose technical direction is under the writer's care. It was a very fortunate coincidence that the three different stills had been designed and built by the same concern, the Lummus Company, so that it became easier to obtain comparative results in our experimental work with the different stills. The conclusions we reached after our distillation experiments and studies, are embodied in the opinions already stated when considering the different systems that may be used in rum distillation. As to the more rudimentary first method already discussed, the experience was gained altogether in small, laboratory scale.

In the perusal of the experimental results that shall presently be brought to the reader's attention, we wish to emphatically state that of all the different factors that in practice are involved in deciding the question of continuous versus discontinuous rum distillation, our interest was restricted to one single point, which was the finding of an answer, through actual experimental evidence, to the follow question – Which of the two methods of distillation will produce the best results as to final quality of the resulting rum?

The work was planned and carried out as follows: The raw material was blackstrap molasses; fermentation of the mashes was carried at the Laboratory of Agricultural Industries in 300-gallon wooden fermenters; and a portion of the fermented mash in each experiment was to be distilled in our own batch still, the rest was to be distilled in the continuous still belonging to the pilot plant connected with the Laboratory above-referred to. Care was taken to have approximately the same degree proof in the respective distillates obtained in each case.

The resulting distillates were tested in the following ways:

- (1) Organoleptic and Physical Tests:
 - (*a*) Aroma.
 - (*b*) Taste.
 - (c) Aroma after sulphuric acid test.
 - (*d*) Rum "body".
 - (e) Index of persistence.

(2) Chemical Analysis:

- (*a*) Total acidity.
- (b) Aldehydes.
- (c) Esters.
- (*d*) Higher alcohols.
- (e) Alcohol by volume.

Three sets of experiments were performed, and the results obtained were consistently in favor of the discontinuously distilled rums. Original aroma, aroma after sulfuric acid treatment, and taste, were found superior in the case of the batch distilled rums. More "body" and higher index of persistence were also found in their case. It was found that the aroma of the batch distilled rums suffered but little by the sulfuric acid test, while the aroma of the rums distilled in the continuous still was greatly injured and weakened by that treatment.

It was also found that the aromatic constituents forming the bouquet of the rums distilled in the continuous still belonged in their great majority to the very volatile (low boiling point) class; so that when samples of the different rums were allowed to stand for some time in shallow watch glasses, the aroma in each case soon became greatly weakened, and after prolonged exposure was practically imperceptible. Just the opposite was true with the rums distilled by the batch still. In this case, the aroma held quite intense and perceptible for a much longer period, and in no case was it entirely lost even after the greater part of the liquid in the glass had evaporated out.

The differences in the chemical composition of the respective raw distillates were just as marked and pronounced as in the case of the organoleptic and physical characteristics. Aldehydes, and especially esters were found consistently higher in value in the case of the batch distilled rums; while the fusel oil content was invariably higher in the case of the rums distilled in the continuous still.

The raw rums thus obtained in each case were set to age in white oak kegs of the same quality and size. After one year of aging the rums were again submitted to the same analytical tests effected previously in the case of the raw distillates. An average sample of each kind of rum was also submitted to fractional distillation in the birectifier.

Tables 21, 22 and 23 respectively, offer the results of these experiments. In the fractional distillation will be noticed the higher values of the ester numbers in the sample representing the batch

TABLE NO. 21

SHOWING CHEMICAL ANALYSIS AND ORGANOLEPTIC AND PHYSICAL TESTS PERFORMED ON RAW RUMS DISTILLED FROM THE SAME BEER, BUT SOME IN BATCH STILL AND OTHERS IN CONTINUOUS STILL

SAMPLES "A" ARE FROM CONTINUOUS, AND "B" FROM DISCONTINUOUS STILL

			Aroma after				(Chemical Analysi	IS	
Sample No.	Taste	Aroma	Sulphuric acid Test	Body No.	Index of Persistence	Alcohol by Volume, %	Total Acidity	Aldehydes	Esters	Higher Alcohols
1-A	Burning, Fair	Fair	Very Slightly Perceptible	8.6	1:5,000	50.82	13.20	70.50	71.00	201.70
1-B	Slightly Burning, Good	Good	Quite Perceptible	9.2	1:10,500	51.76	12.40	105.50	124.0	162.50
2-A	Burning, Poor	Poor	Very Slightly Perceptible	9.0	1:2,000	50.53	44.70	63.70	62.70	260.00
2-В	Slightly Burning, Fair	Fair	Perceptible	9.4	1:7,000	47.55	56.40	109.10	138.80	215.6
3-A	Faintly Burning, Good	Good	Perceptible	9.1	1:9,500	47.70	89.50	39.60	67.20	248.6
3-В	Mellow, Very Good	Good	Strongly Perceptible	9.9	1:17,500	48.35	20.30	85.20	136.50	182.10

NOTE: Acidity, Aldehydes, Esters, Higher Alcohols are Reported in milligrams per 100 milliliters absolute alcohol.

TABLE NO. 22

SHOWING RESULTS OF TESTS PERFORMED ON SAMPLES OF TABLE 21 AFTER THEY HAD BEEN AGEING FOR ONE YEAR IN OAK KEGS

SAMPLES "A" ARE FROM CONTINUOUS, AND "B" FROM DISCONTINUOUS STILL

			Aroma after				(Chemical Analysi	S	
Sample No.	Taste	Aroma	Sulphuric acid Test	Body No.	Index of Persistence	Alcohol by Volume, %	Total Acidity	Aldehydes	Esters	Higher Alcohols
1-A	Slightly Burning, Fair	Good	Slightly Perceptible	9.0	1:12,500	48.90	151.40	95.10	111.10	189.30
1-B	Faintly Burning, Good	Good	Perceptible	9.6	1:20,000	49.50	132.10	98.70	166.80	151.00
2-A	Slightly Burning, Fair	Fair	Slightly Perceptible	9.3	1:5,000	47.86	172.30	81.90	107.40	246.60
2-B	Faintly Burning, Good	Good	Perceptible	10.0	1:12,500	46.90	188.0	89.50	188.00	195.80
3-A	Mellow, Good	Very Good	Strongly Perceptible	9.9	1:25,000	50.86	227.10	56.80	128.00	223.20
3-В	Mellow, Very Good	Very Good	Strongly Perceptible	10.7	1:37,500	47.70	171.20	77.90	178.00	163.80

NOTE: Acidity, Aldehydes, Esters, Higher Alcohols are Reported in milligrams per 100 milliliters absolute alcohol.

distilled rums. Also the much lower values in the different amounts of higher alcohols. All the tests show the superiority of the batch distilled rums.

Whatever the type of equipment employed for distillation of the raw rum the fermentation process and the post fermentation treatment of the wort will notably influence this distillation. When the distillery is prepared to allow for a period of rest to the fermented mash, the distillation stage is thereby greatly benefited. During this period of repose most of the solid matter held in suspension will have an opportunity to settle. Besides, many chemical reactions and inter reactions will have time to come to a finish, greatly enhancing the bouquet, while new aromatic bodies may be formed during this time.

Our experimental evidence has proved beyond all reasonable doubt that at least 75 per cent of the foul odor or rankiness that frequently accompanies freshly distilled rums under present methods of manufacture, originates during the distilling stage, and is due to the chemical decomposition, by the action of heat, of the nitrogenous suspended impurities present in the fermented mash, and to the reducing atmosphere of the hot alcoholic vapors acting on certain gaseous by-products of contaminated beers, and other by-products of alcoholic fermentation. Therefore, as regards the distilling stage, it is of the greatest importance that these organic nitrogenous residues be eliminated as completely as possible from the fermented mash before this is delivered to the still. This organic suspended matter will do harm also in another way during distillation. It will be a source of incrustations on the plates and sides of the continuous stills, or on the bottom, top and walls of the kettles, when batch stills are used; acting also as cementing material for other deposits of mineral nature that are often formed simultaneously. This means a poor rum in the first place, a lowering of the still efficiency, and loss of time and money in frequent shutdowns for cleaning purposes. When perchance, as often happens, the distillation is carelessly conducted, this drawback increases tremendously. Therefore, by feeding the still with a mash cleaned out of all these solid particles, including the yeast itself, we are eliminating all that part of foul odor and bad taste in the raw distillate produced by the overheating and decomposition of these suspended solid impurities, while simultaneously adding to the life and efficiency of the still.

TABLE NO. 23

RESULTS OF FRACTIONAL DISTILLATIONS OF TWO AVERAGE RUM SAMPLES, ONE REPRESENTATIVE OF BATCH DISTILLED, AND THE OTHER OF CONTINUOUS STILL DISTILLED RUMS. FRACTIONATION RESULTS EXPRESSED IN MILLIGRAMS PER 100ML, EXCEPT ALCOHOL BY VOLUME

							FRACTI	ONAL DIST	ILLATION R	RESULTS								
		AVERA	GE SAMPLE	OF BATCH	DISTILLE	D RUMS			AVERAGE SAMPLE OF CONTINUOUS STILL DISTILLED RUMS									
Ensetiers	Temp Range	Appearance Of Fraction		Che	emical Anal	ysis		Remarks	Fraction	Temp Range	Appearance Of							
Fraction No	Of Dist. Deg/C		Alc. By Volume	Volatile Acidity	Esters	Aldehydes	Higher Alcs.	Remarks	No	Of Dist. Deg/C	Fraction	Alc. By Volume	Volatile Acidity	Esters	Aldehydes	Higher Alcs.	Remarks	
1	78-78	Clear	94.04	2.25	228.80	157.02	23.49		1	78-78	Clear	93.32	3.01	220.35	99.17	31.28		
2	78-78	Clear	93.96	1.88	19.01	41.32	12.80		2	78-78	Clear	94.40	2.25	8.45	49.58	17.05		
3	78-78	Clear	94.04	2.25	8.45	37.19	14.97		3	78-78	Clear	92.60	2.63	8.45	41.32	19.44		
4	78-83	Clear	93.32	2.63	12.67	41.32	183.66	Turbid On Dilution	4	78-93	Clear	91.48	5.26	23.23	33.06	244.60	Turbid On Dilution	
5	83-99	Cloudy	56.88	66.87	89.41	66.11	17.09	Oil Drops	5	93-99	Cloudy	21.72	72.13	76.74	53.72	22.76	Oil Drops	
6	99-99	Clear	3.20	87.16	42.94	41.32	6.40	Oil Drops	6	99-99	Clear	1.84	71.00	21.12	33.06	8.52		
7	99-99	Clear	1.84	76.64	12.67	33.06	6.40	Oil Drops	7	99-99	Clear	1.32	66.12	8.45	33.06	8.52		
8	99-99	Clear	1.84	75.14	29.57	33.06	6.40	Oil Drops	8	99-99	Clear	1.60	64.99	8.45	24.79	8.52		

We have found in the course of our investigations that the most effective and practical method to eliminate these solid impurities is by filtration through a supercentrifuge. We have been experimenting with a pilot plant size De Laval Centrifuge of Swedish manufacture, and the results obtained have been very satisfactory. For the light types of Puerto Rican rums, our practice is to centrifuge the fermented mash in bulk, and as soon as fermentation is completed; but for the heavy types, a period of rest is allowed to the fermented liquid before centrifuging. This period of rest will vary in commercial practice according to the nature of aroma desired in the finish product, and with the facilities of equipment for the purpose existing at different establishments. The filtrate obtained from the centrifuge is practically free of all suspended impurities, including the yeast cells. Microscopic cell-counts of the yeast cells before, and after the treatment, have shown that it is efficient up to 99.5 to 100 per cent. The following advantages have been observed from this practice:

(1) During centrifuging of the fermented mash a number of gases which are present are liberated from it. Otherwise these gases would try to interfere with the even, rhythmical development of the distillation process, and part of them might even appear dissolved in the distillate.

(2) If desired, all of the yeast formed during fermentation may be recovered for industrial uses of various kinds, as vitamin production, animal feeds, and fertilizer manufacture. This phase of the treatment is particularly important to Puerto Rican economy, in these days of acute nitrogen scarcity.

(3) Steam economy is effected during distillation due to the clean nature of the liquid fed to the still and its freedom from dissolved obnoxious gases.

(4) The still is kept clean for a longer period of time, thus obtaining economy of operating time, labor and chemicals for its cleaning.

(5) The slops ensuing from the still, if passed through a similar centrifuge, will be free of all suspended impurities, and the problem of its disposal will be greatly simplified.

(6) The problem of crude rum dilution before storage for aging purposes becomes practically solved, for distillation may be conducted at greatly reduced degrees of proof without impairing the quality. of the resulting rum. In the case of cane juice mashes we have been able to distill a very good product at proofs between 110 and 120 degrees.

In the case of blackstrap molasses the proof range should be raised to between 130 and 150 degrees for best results.

(7) No rank odors are detected in these low proof distillates provided the fermentation stage was properly and carefully conducted.

(8) Raw distillates thus produced will be rapid maturing during the aging period, thus a further economy in barrels and storage room is created.

As a practical illustration of the first advantage mentioned above on the supercentrifuging of rum beers before their distillation, reference will be made to a recent commercial rum sample submitted to us for analysis and appraisal of its quality. On opening the bottle, gas ebullition was noticed within the liquid and a peculiar smell reminiscent of sulfur compounds was observed. Part of the rum sample was transferred to a beaker and there strongly agitated by means of an electric stirring apparatus. After a few minutes most of this particular disagreeable odor had disappeared. The ordinary chemical analysis and the fractional distillation test performed on this sample proved it to be of specially fine chemical structure, and very well balanced in its aromatic ingredients. Yet the fact of the presence of dissolved gases in the sample ruined its quality as a beverage due to the repulsive odor felt on opening the bottle. Further investigations on the matter revealed the fact that rum had been manufactured from a very poor class of blackstrap molasses, very rich in sulfur compounds. In fact, the molasses was produced at a sugar refinery using the sulphitation process. Here is a case that could have been prevented, or at least greatly ameliorated, had the beers been centrifuged previous to the rum distillation; as the most of the offensive sulfurous gases in the mash would had been gotten rid of during the supercentrifuging of the beers.

We do not know of any existing rum distillery using this practice of suspended matter and obnoxious gases removal from the fermented mashes before their distillation; but we strongly recommend it, especially for those distilleries using the simplest types of rum stills, devoid of the pasteurizing columns. We further believe that once tried, this procedure will become a permanent practice, since the advantages to be gained will offset the extra labor and expense involved in this operation. Another source of quality contamination of the raw rum during distillation is the quality of the steam used at the still. The steam fed to the still for distillation purposes should be free from objectionable odors, and in fact, from all odors. This is especially so when using exhaust steam at the still. We strongly recommend the purification of the exhaust steam before it is used for rum distillation, for some samples of rums thus distilled, and analyzed by us, have developed a kerosene odor when the sulfuric acid test has been applied, and even on mere dilution of the sample with twice its amount of pure distilled water. Rums having this defect will be particularly disagreeable when used for the confection of cocktails and highballs.

If, as we have asserted, over 75 per cent of the foulness of odor of raw rums originates during distillation; how do we account for the remaining 25 per cent or so? The answer is: that part is fundamentally due to either of two causes, or to a combination of these causes. (1) Defective fermentation in which secondary reactions takes place giving rise to ill smelling liquid products partly soluble in the fermenting liquid. No amount of filtration or decantation will eliminate this source of foulness. (2) Impurities exist in the raw material that are ill smelling as such' or that are so modified and transformed by the action of the yeast and other fermentation agents usually found in rum mashes, as to give rise to substances producing foulness of odor.

These sources of foulness in the raw distillate are completely controllable by proper selection of raw material, fermentation agents, and correct methods of fermentation. For instance, the presence of immoderate amounts of sulphites in molasses is very objectionable from the standpoint of rum manufacture.

The difference as to the origin of foulness in the raw distillate is that about 20 or 25 per cent is formed during fermentation (although this is avoidable); and from 75 to 80 per cent is formed during distillation (which is likewise avoidable). Neither has any reason to exist to the extent at least that they do *exist* under present practices of rum making, if proper precautions are taken. Some of these precautions may be enumerated as follows: (1) careful selection of the raw material; (2) adopting the practice of pretreatment of this raw material; (3) mashing with as pure a water a possible, especially from a bacteriological standpoint; (4) elimination of suspended solid impurities from the fermented mash previous to distillation; (5) using the most proper kind of distillation equipment

in accordance with the type of rum being produced, and (6) using this distillation equipment to best possible advantage, under the supervision and care of skillful personnel.

During the course of these studies special stress was laid on the question of rankiness in recently distilled rums, for this had been a constant source of worry to our distillers and rectifiers. The dread towards "tufo" or rank odor became so intense and general, that very soon any odor in the distillates other than that of pure ethyl alcohol was confused as rankiness or "tufo". Such a state of affairs was not conducive to the following of the best practices in rum making, or to progress in the art of rum distillation. The common trend then was towards the production of a distillate as close as possible to industrial alcohol, and then trying to transform it into rum by many obscure and dubious processes. Of this we shall treat extensively in the chapter on rum curing or maturing. We mention the fact here, for the connection it has with rum distillation. It became our business to demonstrate the fact (then thought of as impossible), that a good rum of the right kind of taste, bouquet, and body could be produced without having recourse to high proofs of raw distillates and to drastic rectification of the product during the distilling stage. At the same time it became our duty to prove that natural good rums could be produced, without recurring to the artificial concoctions so much in vogue in the early days of the renewal of this important industry in Puerto Rico.

With these aims in view the study on the subject of rankiness in raw rums was taken up, and the conclusions reached have already been stated in a brief way. But we feel that a little more explanation is needed as to how the conclusions aforementioned were arrived at.

It was noticed at the start of the investigation that other things remaining constant, and following the old established methods of rum making, the degree of foulness of odor in the raw distillate varied in inverse ratio to the degree proof at which distillation took place. This seemed to offer an explanation why our rum distillers used to distill their raws at degrees proof between 180 and 189. But we soon found out that this practice of rum distillation under high rectification and extremely high alcoholic content eliminated not only the objectionable rankiness, but at the same time the inherent and characteristic rum aroma of the crude distillate; besides becoming the origin of other troubles to be met later on during the further preparation of the commercial product.

These distilling practices eliminated some of the most valuable congeneric products of a genuine rum, such as a great part of the rum oil and some very valuable esters and aldehydes of high molecular weights, high boiling points, that together with the rum oil exercise the greatest influence on the stability and persistence of the rum aroma. On these constituents the adaptability of a rum for the preparation of diluted drinks such as cocktails, punches and highballs is greatly dependent. This practice then opened the way to the *artificial addition* of flavoring and aromatic ingredients to the product before its final presentation in the market. The result was that a poor practice was trying to be corrected by a poorer one.

Another disadvantage of high rectification and high proof distillation was found in the fact that no matter how high may be the proof of the recently distilled rum, this original alcoholic concentration must be greatly diluted before the rum is finally bottled. This apparently insignificant, little matter of rum dilution, becomes a very important one in industrial and commercial practice, as will be shown in our chapter on rum rectification and curing. It will suffice to state here that in whatever stage of the process of rum manufacture this obligatory dilution takes place, it will be a drawback towards rum quality, good taste and aroma; and that the harm done to the quality of the rum shall be greater *the higher the proof at which the rum was originally distilled*. Another good reason for avoiding high proof distillation in rum making.

It was also found in the course of our distillation studies that at a given proof of distillation great differences in final taste and aroma of the raw distillate were still found according to the more or less amount of suspended solid impurities found in the beer being distilled. This fact led to the experiments performed for the elimination of solid impurities in the fermented mash that culminated with the introduction of the practice of supercentrifuging of the beers before submitting them to the distillation process.

As already stated, these studies, and those already mentioned under mashing and fermentation methods, led to the conclusion that true, genuine rums, needless of artificially added ingredients, can be manufactured when proper fermentation and distillation methods are employed. The fallacy that high rectification, high proof rum distillation was unavoidable in rum production has also been exploded.

We asserted that the selection of the still for a given distillery is something that must be considered in connection with many other

factors. Among these, the type of rum to be manufactured, and the amount of aging that it is intended the product should receive before going into the market, are about the two more important. Others are mostly concerned with economic reasons, with which we are not so much concerned in the presentation of these studies.

The batch still is admirably adapted for the production of any type of rum, from the lightest to the heaviest; but becomes a *necessity* when it is desired to manufacture the better class of the so called heavy rums. As to the necessary aging period, the heavy rums distilled discontinuously with proper *selective extraction* of desired constituents will reach maturity in but a fraction of the time necessary for similar products distilled otherwise. The readiness with which the product may be fractionated during the distilling period gives the batch still its more conspicuous advantage for the exercise of selective distillation. Another great advantage is the unique control over the rate of distillation that may be exercised with the batch still, where if desired, the distillation may be carried by drops almost; something quite impossible when operating continuously. Total refluxing for a given time back into the column is another practice of easy accomplishment in the case of the batch still.

The continuous still provided with an auxiliary purifying and pasteurizing column is admirably adapted to the production of the light types of rum with very suave taste and delicate aroma. It is especially adapted for the manufacture of light rums that are to be moderately aged, as the raw distillates obtained with this type of stills are very fast maturing, due to the already existing mellowness of taste and exquisiteness of aroma in the raw distillates. Heavy rums may also be distilled in this type of still, but they will lack the ampleness of taste and bouquet, and above all, the body and index of persistence typical of the best rums of this type. The ordinary simple type of continuous rum stills so often found in the rum distillery have practically no other control over the distillate than determination of its proof. Hence we could hardly expect a good, genuine product from such apparatus. The best that can be done is to distill at the highest possible proof that the column may afford. The final raw distillate will be hard and raw in taste and very variable in the character of its aroma. Most always these types of distillates must be redistilled in batch rectifying columns in order to obtain a fair commercial product.

If to obviate this double distillation, the industrial alcohol four column still is substituted with but minor modifications in operation, then the true nature of rum is destroyed, and an impure industrial alcohol is the product obtained; devoid of rankiness it is true, but also devoid of rum characteristics.

In whatever form or type of still the rum is produced, the distillation should never be overforced; on the contrary, it must be slow, rhythmic, and uniform. Especially are these precautions necessary when working with batch stills towards the beginning and end of distillation, for at the beginning head products are to be separated, and towards the end, the tail products are likewise to be separated from the main product or body of the raw distillate. Much practice and experience are necessary to accurately judge as to the right moment to begin collecting body, and also for beginning the separation of tails. When working with continuous stills of the simpler types the apparent proof should be kept between 170-180; much lower distillation proofs, between 140-150, may be used when a pasteurizing auxiliary column is used in connection with the simple still; and still lower proofs, between 120-130, may be safely used if the batch still is the one under use. Another very important point is the question of proper reflux. With batch stills, the author has found that the practice of total reflux of the alcoholic vapors for a definite period (to be determined in practice) previous to actual distillation is a great help towards obtaining excellent raw distillates. The fact must constantly be borne in mind that in the case of rum, distillation is a process of selective extraction and not of total elimination of all volatile substances with the exception of ethyl alcohol and a little water. There are congeneric products to be eliminated, and others that must be retained. Another important point to be observed in rum distillation is the prevention of sudden cooling and condensing of the alcoholic vapors that will form the raw rum. For this reason the writer favors the use of long, narrow condensers of ample capacity where to zones of temperature demarcation are easily established. The reason for this is that the refrigeration of the mixture of vapors entering the condenser (and refrigeration of the mixture of vapors entering the condenser (and that once condensed will constitute the raw rum) must be so carried as to prevent condensation in an abrupt way. The condensing process must be gradual as the vapors pass from the warmest to the coolest part of the condensing walls; other sharpness of taste and pungency of aroma will be the consequence, through separation of aromatic principles and breakage of the harmonious composition of the mixture.

To resume the salient points of this chapter on rum distillation we will state that:

(1) Rum distillation is a process of *selective extraction*.

(2) The process of fermentation and the predistillation treatment of the fermented mash bear great influence on that of distillation as regards quality of product.

(3) Stills, whether of the continuous or discontinuous type, should have ample capacity so that distillation will never have to be rushed.

(4) Foulness of odor in the raw distillate is unnecessary, undesirable, and may be greatly diminished or entirely eliminated by methods herein outlined.

(5) It is important to distill rum at the *lowest possible* proof compatible with good quality.

(6) The distillation method to be adopted at a particular distillery requires special study in each case; but in a general way, when quality of finished product alone is the issue, then discontinuous or batch stills should be used.

(7) The economic advantages of the continuous system of distillation are apparent and undeniable. For large, bulk production, the continuous still becomes necessary. In this case the simple continuous still provided with an auxiliary digesting and purifying column is, in our opinion, the type to be preferred. Nevertheless, we opine also that even in the case of large mass production a combination of the two systems may advantageously by used.

(8) The fallacy that high rectification, high proof rum distillation was unavoidable, has been exploded.

VII

Rum Curing and Maturing

1

Is the expression "Aged Rum" equivalent to that of "Matured Rum"?

We have observed that a great majority of persons use the two expressions as synonymous, but they are mistaken. When one term is used as equivalent of the other we are merely confusing the end with the means, for really, maturity in the rum is the end sought, and aging is one of the means employed towards the obtention of this end. Now then, although usually an aged rum is also matured, this sequence does not follow necessarily, nor there exists a definite lapse of aging time at which the condition of maturity may be said to have been reached in all cases. On the other hand, a given rum may be matured without necessarily being what is commonly called an old rum. If the quality of maturity depended only, and exclusively, on the amount of time the rum had been kept aging, then perhaps the two expressions could be used indistinctly, but it is not so; aging being only an important factor in the process of maturing. There are others, for instance, the potential capacity and adaptability of the crude rum or raw distillate to acquire the state of maturity. In our opinion, this factor is as important, or more important perhaps, than that of aging.

As an illustration, let us take up an imaginary case of two raw rums and called them "A" and "B" respectively. Both raw rums are set to age in the same kind, size and quality of barrels, and under equal conditions of temperature and relative humidity. At the end of one year the two rums are examined by the usually tests for maturity and it is found that rum "A" has already acquired the quality and general conditions inherent to a matured rum; while rum "B" has not quite reach these conditions. Rum "A" is then bottled, and the aging of "B" is continued, till at the end of another six months we find that it also has reached the state of maturity previously observed in the case of "A". Would it be fair to consider sample "B" as more matured than sample "A" for the mere reason that it has aged for a longer period? Could we be justified in acclaiming rum "B" as superior to rum "A" for the mere fact that it cost more time and money to impart the characteristics of maturity to it? Evidently not. If at all, we could say that "A" was superior to "B" in an economic sense since it acquired maturity in two thirds of the time required by sample "B".

This example has been presented so that the reader may clearly grasp the meaning of ripeness or maturity of product as distinguishable from that of age of product. It is not fair to use solely the time a rum has been in the curing barrel as a criterium of its goodness or of its pretended superiority over a similar rum that has been less time aging in the curing barrel. Hence, any standard of rum quality based solely on the lapse of time the different products have been aged, would be not only unscientific and erroneous, but also decidedly unjust. It is not the age of the rum that is bought and paid for by the public, but the genuine characteristics of body, aroma and taste that on reaching maturity a rum acquires. The time required by different rums to reach this state of maturity during aging will depend, other

conditions being equal, on the type and the quality of the product as a raw distillate.

Our researches on the question have demonstrated that great variation and differences exist in the capacity and adaptability of different raw distillates to acquire maturity. These are some capable of reaching this desirable condition in from one to two years of aging, while others may require twitch, and even thrice this time.

It is really amazing the little importance that is generally conceded to the quality of the raw distillate in most rum distilleries. Instead of trying to produce a raw spirit that would need the least trouble in treatment during rectification before finally bottling the product, producers spend their energies and efforts in finding new, more complicate, and laborious methods of curing the defects of poorly fermented and worse distilled raw spirits.

And yet, to our view, the future of the rum industry is dependent, in its technical aspects at least, on the production of better raw spirits, raw rums that on account of their well-balanced chemical composition and excellence of physical and organoleptic characteristics, will require but little aging time to acquire maturity.

Towards that goal a great part of our efforts have been directed, and we have found that the obtention of maturity is not due to one single cause, as for instance aging; but that this final result is obtained through a happy combination of many factors which being operating with the choice of fermentation agents and raw materials and end with the bottling of the product for public consumption. Every one of the different stages through which the product must pass before reaching the bottle, shall impart to it favorable or unfavorable conditions and characteristics towards the obtention of maturity. Hence, the final success or failure will depend on the manufacturer's ability to employ those methods and technique that better and more efficiently contribute to the rapid acquirement of maturity.

Of some such methods we have been treating in the past chapters, and in this one we shall consider the phase of rum manufacture that supposedly bears the greatest influence on the subject under discussion.

Having thus obtained the rum in the raw state through the process of distillation it becomes necessary to develop to the utmost the inherent characteristics of a good product. This is secured by the process known as curing or maturing. Here we wish to state emphatically that by this process we do not mean converting a *bad product* into a *good, wholesome* one. Not by any means. The rum which is bad in its raw state will continue to remain so, no matter what is done with, or to it. Proper rum curing is not a process to change or transform, but to develop and further enhance the latent qualities already existing in the right kind of raw

distillate. Of course, a poor raw rum may be made to improve, but it will never be converted into a first-class beverage with distinctive seal of excellence through the curing process, whether the natural or slow, or the artificial or rapid curing be employed.

Before starting the curing process, certain preliminary treatment of the raw distillate is almost always necessary, especially under present practices of rum manufacture, and the employment of rapid curing methods. With a high class, genuine raw rum, the treatment should consist only of diluting to a certain proof before storing in oak barrels in the case of natural curing, or before proceeding with accelerated aging in the case of rapid curing. With inferior quality raw distillates further treatment other than mere dilution becomes necessary. This consists in most cases in elimination of foulness of odor from the raw distillate and improvement in taste. There are two principal ways of accomplishing this purpose, one of them essentially chemical in nature, the other essential physical; while intervening between these two are many others, using features of each of the principal two methods. The chemical treatments in the hands of the profane may constitute a source of great danger to the industry itself and to the public health as well. To illustrate this point we wish to present comparative analyses of two rums. Number 1 represents the diluted raw spirit before any further treatments; while number 2 represents the same raw rum after it had passed through chemical treatment for obliteration of foulness of odor and bad taste.

	SAMPLE N	UMBERS
	[1]	[2]
Sp. Gr. at 20/4 deg. C Alcohol by volume, per cent Grs alcohol/100 ml pH value Total acidity, mgs/100 ml Aldehydes, mgs/100 ml Esters, mgs/100 ml Extraneous impurities	0.93056 49.80 39.31 4.80 17.10 53.60 56.60 None	0.94331 42.87 35.86 5.20 182.70 Traces 2.10 Magnanese, potassium, iron, sulphates, chlorides,
Odour Taste	foul bad	ammonium, etc. none very bad

142

A glance at the comparative analytical results will show that: (1) Sample No. 1 that had received no treatment other than mere dilution, was a sample of a poorly fermented, uncarefully distilled rum; hence its bad taste and foulness of odor. Nevertheless it was a rum. (2) Sample No. 2, the same originally as No. 1, received a certain chemical treatment aimed at obliteration of bad odor and improvement of taste. But what did really happen? The writer, who tested these two samples, admits that the "treatment" succeeded in eliminating foul odor; but no good odor appeared to replace the other; the bad taste remained present, worse than before. Besides, the liquid was no longer rum or alcohol, or any other specific substance. Notice that during the "treatment" almost 7.0 per cent alcohol by volume was lost, besides practically complete destruction of esters and aldehydes originally present. An enormous increase in total acidity appears in the treated rum as well as the presence of extraneous constituents formerly absent. Among these constituents we found in greater abundance sulfates, chlorides, ammonium, manganese, potassium, and iron. Only qualitative tests were performed for the presence of these extraneous, newly incorporated substances; but the reactions that occurred showed that appreciable quantities existed in each case. The results of the chemical treatment was disastrous in this particular case.

We shall proceed now to discuss the practice that we consider best and least harmful to the prestige of the vigorous, young industry and to the health of the consuming public. This consists in diluting the raw rum to the optimum proof in relation to the period of time the liquid is supposed to be aging in the barrel. This means that for longer intended aging periods the raws will be diluted to higher proofs than when a shorter aging period is under consideration. The conditions of temperature and relative humidity under which the aging will take place is a factor of great importance in this subject of dilution of the raw spirits. Unfortunately in this regard, the laws and regulations controlling the rum industry consider any proof below one hundred as 100 P. for the purpose of revenue collection. This unfortunate provision makes it prohibitive for the rectifier or distiller to lower the proof of the raws below one hundred for aging purposes. And we call this law or regulation unfortunate because if it did not exist the rectifier or distiller could then really use the most adequate dilution of the raw spirits for aging purposes; and in a great many cases the aging barrels would be filled at proofs varying considerably from one hundred, along the lower scale. It would certainly be a great improvement in the body, taste and aroma of a bottled commercial rum if *no dilution water* had to be incorporated to the rum once it had undergone its aging period. This matter will be

discussed more fully later on in this chapter.

Due then to law and regulations, the raw rums may not be diluted much below 110 degrees proof before they are submitted to the curing process; whether natural, or artificial. When a poor distillate is thus diluted, the foulness of odor becomes much more accentuated and noticeable. At this point the author begs to be excused for a digression in behalf of the average continental American citizen who may read these pages. The American public is well aware of the fact that whisky, as first produced by any of the well-known processes, is a liquid very raw, very unpleasant to the taste, and very disagreeable in aroma. The public also knows it takes many a year of natural aging to change the liquid into a potable product. Even then, blending is oftentimes necessary, in which famous imported European brandies take quite an important place in the development of taste and bouquet. This is not the case with rum. Rum properly fermented and distilled, possesses agreeable taste and fine bouquet as such. In some instances it could be used as a potable beverage outright. This is the reason why we made reference to the natural taste and bouquet of a raw rum. Moreover, rum will mature to its optimum condition in but a fraction of the time necessary in the case of whisky. This fact we have definitely proved in our studies and it has been corroborated by European liquor experts.

When present, this foul odor may be reduced more or less (according to its origin and nature) through the application of activated deodorizing chars. The amounts of the carbon to use will be determined experimentally in every particular case. This is a case of foulness of taste and odor removed through absorption, and the process is to be preferred in all respects to the destructive and polluting, drastic chemical threats with powerful oxidizing agents. After the end has been accomplished, the carbon is removed by filtration. The ideal raw distillate, however, would be one needing no dilution or treatment of any kind before proceeding with its curing, either slow or natural; or accelerated or artificial. These conditions are attainable through proper yeast and raw material selection, appropriate methods of fermentation and post fermentation treatment of the beers; followed by carefully controlled distillation technique based on the principle of selective extraction. Present rum manufacturing methods, with few exceptions, have not come to this high state of development as yet. Hence, mention must be made of the important changes occurring during the diluting, or the diluting and carbon treatment process during rectification.

When a freshly distilled rum is diluted, its chemical composition as well as its physical characteristics are affect. The action of the diluent is felt to a greater or lesser extent, in accordance with: (1) original proof at which distillation took place; (2) chemical composition of the original distillate; (3) nature of the diluent, and manner in which it is applied. The deleterious action of rum dilution is twofold; chemical and physical. The first consists in a dissociation of part of the ester content through the hydrolytic action of the diluent acting in an acid medium; the second operates by salting out or separating certain essential oils through shock, and by the lowering effect on the alcoholic concentration of the raw distillate through the addition of the diluent. Among these oils we have some of the most valuable natural constituents of a genuine rum. When the raw rum has been distilled a very high proof, saw between 170-180 degrees, a very large amount of diluent must be added in order to bring the proof down, say to 110 degrees. Now, the more diluent added the stronger will its hydrolytic effect be on the esters present in the raw distillate, and the stronger will be the tendency towards separation of essential oils. Hence, the caution previously given in the chapter on rum distillation that rum should be distilled at the lowest possible proof compatible with high quality of distillate. When the original chemical composition or the raw rum in its relation to the process of dilution is considered, it will readily be seen that the higher the free acidity and the ester content of the distillate, the faster and the more intense will become the hydrolyzing effect of the diluent on the ester content of the raw spirit. The same holds true on the salting out of valuable essential oils. The nature of, and manner of adding the diluent, will also become a factor of importance during the process of dilution. There are at least six classes of diluent commonly used for this process:

(1) ordinary tap water from the city mains; (2) well water; (3) rain water; (4) distilled water; (5) chemically treated water; (6) alcoholic solutions in distilled or rain water, previously cured by either natural or artificial means. This last-mentioned diluent is the least harmful and best recommended for this purpose, but is the one less used on account of the trouble of preparing and storing under suitable conditions large quantities of these weakly alcoholic solutions. Good practice should restrain the number of diluent to but three of the six mentioned above, and these should include: (1) distilled aerated water, (2) rain water, and (3) alcoholic solutions. Of the two plain water diluent, rain water is to be preferred, as it contains plenty of air and, therefore, lacks the flatness of taste peculiar to distilled water. Thoroughly aerated distilled water has the advantage of being readily available at all times. Diluting with aged mixtures of alcohol and water, or rum and water, will be the best method to follow; but as explained before, this method has its shortcomings especially for the large producer.

Now, whatever the nature of the diluent used, the manner of its application will have considerable influence in the extent of ester hydrolysis, and especially as to the degree of separation of valuable essential oil constituents. Cold diluent, suddenly added in bulk or added in a very short period of time, will prove the most harmful. The opposite conditions of applying the diluent, that is, slightly warm, slowly and in an atomized form, will prove the least harmful. Once the raw rum has been conveniently diluted, when foulness of odor exists, it must be removed in some way, and we have already stated that treatment with deodorizing chars is the best method to use for this purpose. There are however, two risks in this treatment: (1) The carbon will absorb some of the aromatic constituents of the rum together with the ill smelling ones; and by this action the original balance of aromatic constituents will be disturbed, resulting in a modification of original aroma. (2) If a thorough and very efficient filtration is not effected after the carbon treatment, some of the finest particles of the carbon will pass through with the liquid, and will become a source of trouble later on when the rum is finally bottled up for public consumption. It may then appear as a deposit in the bottle, especially when accelerated curing methods are employed.

In the course of these studies on rum we have observed that the harmful effect of the diluent in rum diluting lasts over an appreciable period of time, never less than three months, and extending up to six months. It is really a very important matter that has been lightly, *too lightly*, considered up to the present time.

Table 24 presents data on the ester values of various raw rums as distilled; 24 hours after being diluted for storage and aging; and after three months aging in oak containers.

It will be noticed that strong dissociation of the ester molecules occur in every instance after the raw rums have been diluted, and that this state of original esters hydrolysis is not ameliorated even after three months of curing in an oak keg. The ester loss through hydrolysis is heavier in those rums originally high in ester content. Organoleptic tests on the undiluted and the diluted samples are consistently in favor of the former. Hence, rum dilution is really a complicated problem, and the ill effects of dilution interfere also with the proper and expected progress in quality during the first few months of the curing period. If to obviate the ill effect on the curing of the raw rum during the aging period occasioned by the diluting process, the raw rums are barrelled at the high proofs of distillation, nothing is gained in the long run; for if it true that it will mature quicker during aging in this case, it is also true that its proof must be lowered before it is bottled up. When diluting the cured rum, the effect of the diluent will be more disastrous even than in the case when the beverage was diluted as a raw product. These consideration make the problem of dilution an arduous one. The solution must come during the distilling stage of rum manufacture. Raw distillates must be distilled at low enough proof so that no dilution, or at least very little dilution should be required during rectification and curing. And this must take effect in such manner that the quality of the commercial rum shall remain unimpaired.

After the raw distillate has been prepared for the curing process, the next step is to decide on the method by which this is to be realized. There are two general systems of curing or maturing employed: (1) slow or natural curing, and (2) accelerated or artificial curing. The first offers little variation in present technique; while the latter is very variable; as many methods and combinations of two or more methods exist as there are rectifiers engaged in the business of rum making.

At the re-establishment of the rum industry in Puerto Rico, producers were divided into two different schools regarding rum curing or maturing. One of these groups favored accelerated or artificial aging, the object of which was to impart if possible, the characteristics of a matured rum to the raw distillate in the shortest period of time. Presumably the use of aging barrels was not followed by this group. The other group believed in the classic curing method through natural, slow aging, in the well-known oak barrel.

TABLE NO 24

PRESENTING DATA SHOWING THE HYDROLYTIC EFFECT OF THE DILUENT ON THE ESTER VALUES OF RAW RUMS 24 HOURS AFTER DILUTION AND AFTER AGEING DURING THREE MONTHS. IN THIS CASE THE DILUENT WAS DISTILLED WATER. ESTERS REPORTED IN MGS/100ML ABS. ALC.

	ESTER VALUE CALCULATED ON THE BASIS OF MGS/100ML ABS. ALC. IN RUM								
Sample No.	Undi	luted	Dilt	uted	Aged				
	Proof	Esters	Proof	Esters	Proof	Esters			
1	160	176	110	151	105	138			
2	160	177	110	143	104	132			
3	162	179	110	125	105	105			
4	154	196	108	137	103	127			
5	157	247	105	152	100	148			
6	156	248	108	186	102	161			
7	150	286	105	200	101	186			
8	150	517	108	223	103	206			
9	160	158	110	143	105	134			
10	160	122	110	75	106	63			
11	160	125	108	87	104	75			
12	160	134	110	107	105	73			
13	145	143	108	101	103	93			
14	147	150	110	98	104	89			
15	155	124	108	91	104	81			
16	159	162	108	122	103	105			
17	164	104	105	94	101	89			
18	161	101	108	87	103	83			
19	166	90	110	77	104	56			
20	160	101	108	90	103	85			
21	158	118	110	105	106	102			
22	159	99	108	81	103	86			
23	164	92	110	68	105	66			
24	163	104	108	89	103	75			

We soon discovered in our consulting practice that both schools were incurring in a common error: lack of recognition of the true value of a raw distillate of quality, and of the paramount role it plays in the problem of rum curing. Much thought, ingenuity, time, and money, were spent contriving to impart maturity by rapid methods, on the part of the followers of such processes; while from the other side, the believes in the oak barrel were trying to obtain the best results without considering for a moment the kind of quality of the raw rum with which the barrels were filled.

Our own studies have proved, however, that the real key to successful curing is the quality of the raw distillate, whether artificial or natural curing methods are employed. It would be well to cite here some of the reasons presented to the writer by the followers of rapid curing methods in favor of their practices:

(1) Elimination of the inconveniences, expenses and risks implied in the storage of thousands, even millions of gallons in oak barrels during the curing period.

(2) Rapid processes that eliminate the oak containers are cheaper; hence, returns on capital investments are higher and available in a very short period of time.

(3) Competition being carried mostly on price levels, rums aged for a considerable period of time would have to be sold for practically the same prices being paid for the artificially concocted rums.

(4) The great majority of the consuming public has not been educated to distinguish gradations in goodness of taste and exquisiteness of aroma of different rums. Besides, the main market for Puerto Rican rums seldom uses the straight drink, but rather the rum is used as one of the ingredients of a cocktail, making it harder for the consumer to differentiate about quality among different rums.

(5) Less capital investment is necessary to engage in the business.

(6) Since no official regulations or standards of quality exist for rum appraisal and classification, it results that in commercial practice values are confused, poor rums, fair rums, and really good rums, actually receiving equal prices. Under such conditions they thought that it was better to choose that method of production resulting cheaper and more expedient.

Against these arguments, the followers of the classic curing method offered their own, some of which were:

(1) Rums produced by the slow curing process obtained better characteristics of body, taste, and aroma; and especially less variations in this attributes were found.

(2) Quality increases as the aging progressed, and in this way a permanent clientele is acquired for a certain product.

(3) Superior make-up in the chemical structure of the products.

(4) Stronger guarantees on the building of a permanent, ever increasing industry, safe against changed of legal nature that in the future may interfere with the practice followed by those favoring the artificial curing.

Our own experiments and activities in the field of rum curing have satisfied us that the natural curing in the oak barrel is by far the best method to follow in the process of maturing the raw product. But, it seems to us that rum companies would learn a great deal, and profit still more, by carrying on a little study, observation and research in this question of natural curing. By so doing, means may be found through which to accelerate the curing process without casting aside altogether the classic method using the oak barrel.

To begin with, the aim should be to cut down as much as possible on the factor of aging time, still obtaining the same results as to the degree of maturity imparted. In two words: keep using the old classic method, but try to improve on it so that the state of ripeness in the raw product may be obtained in one half, or perhaps one fourth of the time now required for its accomplishment.

This phase of our research has lacked the time and equipment for a complete study; but the time that we have been able to put into the matter has been rewarded by the acquisition of certain valuable observations and results that will be given to our readers in the next few pages in as concise a form as possible.

The factors affecting maturity of a raw distillate are many and varied, but they may be divided into two main groups:

(1) Those factors that combine and help each other for the production of good quality in the raw distillate.

(2) Those factors that further develop, enhance and extend the intrinsic good qualities already existing in the raw distillate. The first set of factors exert their influences during the manufacture of the raw distillate, and contrive towards the production of the right kind of raw material in the form of the raw rum. Those factors have already been discussed in previous chapters when giving the directions we have found necessary in the manufacture of the raw spirit. The second set of factors are concerned with the actual curing process.

The subject of natural curing shall now engage our attention. The raw rum having been obtained, it becomes necessary to impart to it the condition known as ripeness or maturity by the combined influence of: (1) the barrel, (2) temperature and relative humidity conditions during storage, (3) action of the oxygen in the surrounding atmosphere, and (4) time. Besides, the influences of the four factors above-mentioned will be more or less effective and efficient according to the: (5) percentage of ethyl alcohol concentration by volume existing in the crude distillate when filling the barrel; and (6) by the composition of the Non-Alcohol-Number of the raw product.

We have already discussed the fact that any alteration in the degree proof of the raw distillate brings about a disequilibrium in bouquet and change of taste; but aside from the effect of dilution, other modifications of aroma and taste during aging are dependent on the alcoholic concentration of the raw rum entering the barrel.

The degree of alcoholic concentration in the aging liquid will determine its solvent power for those constituents of the barrel more soluble in alcohol than in water. Some of the extractive substances contained in the staves of the barrel are more soluble in water and others more soluble in alcohol. Hence, the absorptive action of the alcoholic liquid will be conditioned by the respective proportions of alcohol and water in the rum. Hence, the importance of carefully determining the most convenient proof at the time of filling the barrel. Another factor in this matter of proof in the raw rum is connected with the period of aging which is intended to be given to the raw rum. We have already stated that ideally, the rum as it comes already matured from the aging barrel should receive no dilution, or as little as possible. Hence, initial aging proofs, in our opinion, should be kept within the range 105-130, according to different conditions existing at different places. We have tested maturing rums taking the undiluted sample right out of the aging barrel that were far superior in taste and aroma at proof of between 105 and 110 than when these same samples had been diluted with distilled water to the usual commercial proofs of between 85-90. This alone shows the great derangement suffered by a matured, or partially matured, rum when its proof is suddenly altered. We thus advise those distillers or rectifiers that per force must age raws of high proof during curing, not to ever bottle the newly diluted aged rum in haste. In these cases, the diluted matured rums should really be submitted to a second curing period of not less than six months; but the demands of trade not allowing the use of this procedure, then it would be very convenient to keep the diluted aged rum in special oak tanks for at least fifteen days before finally bottling up the product for the market. Aside of the derangement of taste and aroma suffered by the diluted cured rum, it will also be found that if bottled immediately or shortly after diluting, precipitations and sedimentations will later on appear in the bottles, due to the rearrangement of the constituents of the rum in the re-establishment of equilibrium necessary to meet the new conditions obtained by the lowering of the alcoholic concentration. Many extractive substances perfectly soluble at higher alcoholic concentration may become less so under the new diluted conditions, making their appearance as sediment on the bottom of the bottles. This phenomenon will become more apparent the longer the time taken to dispose of the bottle. Hence, hurriedly bottled rums under these circumstances have best chances when quickly consumed by the public, in so far as not showing up the presence of sediments.

There exist considerable differences in the chemical composition of raw rums especially as to the nature and amount of the components of their respective Non-Alcohol-Numbers. During the curing period, the composition of this Non-Alcohol-Number has great influence in the final quality of the matured rum. The products variety; the individual chemical constitution of each one; and their physical properties, constitute factors of great importance in determining the final character of finished, cured product, and the time this will take to obtain maturity.

From a perusal of the literature and own observation and studies we find that a great variety of components enter in the structure of the Non-Alcohol-Number of rums. Not all of these compounds are present, of course, in any one given sample of rum; but they have been found during the study of many different rums, produced under varying conditions in different countries and localities.

The existence of a still higher number of compounds is presumed, since known analytical methods have proved unsuccessful for the detection and identification of components present in almost inappreciable quantities, but that undoubtedly play a part in the complex and compound aroma of rum. The classification of group compounds and of individual members that have been identified in the several groups follows:

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A – Acetals:
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(1) Acetal

B - Alcohols:

(1) Monohydric Saturated Alcohols

(a) Methyl
(b) Propyl, (normal)
(c) Iso-propyl
(d) Butyl, (normal)
(e) Iso-butyl
(f) Amyl, (normal)
(g) Amyl, (secondary)
(h) Hexyl
(i) Heptyl
(j) Octyl
(k) Decyl

(2) Dihydric Alcohols or Glycols Isobutyl glycol

(3) Trihydric Alcohols Glycerine

C – Aldehydes:

- (1) Acetaldehyde
- (2) Propaldehyde
- (3) Isobutaldehyde
- (4) Butaldehyde
- (5) Hexaldehyde
- (6) Heptaldehyde
- (7) Furfural

D – Ketones:

(1) Acetone

- E Organic Acids:
 - (1) Formic
 - (2) Acetic
 - (3) Propionic
 - (4) Butyric
 - (5) Caproic
 - (6) Heptoic
 - (7) Caprylic
 - (8) Capric

F – Esters:

(1) Acetates of:

- (a) Methyl
- (b) Ethyl
- (c) Propyl
- (d) Butyl
- (e) Iso-butyl
- (f) and Amyl alcohols
- (2) Propionates of:
 - (a) Methyl
 - (b) Ethyl
 - (c) Amyl alcohols
- (3) Formates of:
 - (a) Ethyl
 - (b) Propyl
 - (c) Butyl
 - (d) Amyl alcohols
- (4) Butyrates and Isobutyrates of:
 - (a) Ethyl
 - (b) Amyl alcohols
- (5) Oenanthate, caprylate and caproate of: (*a*) Ethyl Alcohol

G - Bases:

- (1) Pyridines
- (2) Amines

H – Essential Oils:

(1) Rum oil, and others

The presence of methyl alcohol in the Non-Alcohol-Number has been accepted by some, and denied by other investigators. Most recent and up-to-date opinion establishes the possibility of this alcohol being present in rums. The acetal is supposed to be formed during the aging period through the reaction of acetaldehyde and ethyl alcohol. The presence of aldehydes and acetals during the curing period probably gives rise to a whole series of condensation products within the liquid in the barrel; and these products enter into the taste and aroma of the matured rum.

Some of the constituents possess lower boiling points than ethyl alcohol. They also possess sharp and irritating odors and tastes. Among these bodies are some of the aldehydes and esters, methyl alcohol, methyl formate, methyl acetate, acetone, acetaldehyde, propaldehyde, etc. Others of these bodies possess higher boiling points than ethyl alcohol; and they also possess qualities of aroma and taste superior to the group first-mentioned. Among these we find ethyl butyrate, amyl formate, butyl acetate, amyl acetate, ethyl oenanthate, caprylate and caproate; butaldehyde, caproic and oenanthic aldehydes, and the essential oils, especially rum oil.

The modifications in aroma and taste suffered by the aging; amount, and class of the curing period will depend a great deal on the amount and class of the different components above-mentioned originally present in the raw spirit. The relative chemical reactive capacity or affinity of these bodies will be an important factor in the oxidation and condensation products that will be formed during the curing period. Their power to combine chemically with some of the extractive products coming from the staves of the barrel becomes a valuable factor in the formation of taste and bouquet.

The physical properties of these constituents of the Non-Alcohol-Number, especially the molecular weights, boiling points, and susceptibleness of forming azeotropic mixtures with other substances, are also of great influence and importance in the acquiring of maturity in a longer or shorter period of time; as well as on the final chemical make-up of the cured product. The pores of the staves forming the barrels offer an exit medium not only to the ethyl alcohol and water molecules in the vapor state within the barrel, but also to these volatile components of the Non-Alcohol-Number. The proportion in which these compounds will pass out of the barrel through its pores, will depend on: (1) size of individual molecule; (2) relative volatility with respect to the ethyl alcohol molecules; (3) amount in existence of a given component; (4) degree of saturation of a given

component already existing in the atmosphere surrounding the barrel; (5) individual tendency of each component towards the formation of azeotropic mixtures with other compounds in the vapor state. The smaller and more volatile molecules will tend to pass out first through the pores of the barrel. This fact seems to explain the condition of suavity in aroma and taste the liquor under curing slowly undergoes; since most always these low-molecular-weight, low-boiling point components of the Non-Alcohol-Number that are more rapidly eliminated from the curing liquid are also the ones that impart sharp taste and pungent aroma to the raw spirit. Some of the light bodies that are not eliminated by evaporation through the pores of the barrel, enter into condensation reactions forming bodies of better aromatic characteristics.

From what has been said it may readily be noticed that in this respect the curing barrel exercises an action very similar to that of the still; that is, separation of undesirable from desirable rum constituents; hence, the less complete this process is carried during distillation must be a process of selective fractionation and extraction. Only by filling the barrel with a *properly selected* raw spirit may we hope for adequate, efficient, and rapid maturing during the curing stage.

Of the various types of aging barrels available, the Puerto Rican rum industry employs mainly those that have already been used in the curing of whisky, among which are found the shaved charred, the charred, and the plain ones. A few distilleries also use new, plain white oak barrels. The main reasons to use the old whisky barrels are that these cost much less than the new barrels, and also because they are already "cured" according to the buyer's opinion.

The quality of aging barrels is a very important question in the process of curing. Unfortunately, the distiller has usually no control over some of the factors affecting this quality; as for instance, character of the soil and climate of the region where the trees were grown; age of these trees when cut; manner in which they were cut; part of the tree from which the staves were derived; variety and amount in the content of resins and other extractive matter existing in the wood; and the period of aging given to these boards previous to the actual construction of the barrel.

One of the factors of greatest importance during the curing process is the degree of porosity existing in the staves forming the

barrel. The exodus of the different compounds already mentioned above from the liquid within the barrel to the surrounding atmosphere, will naturally be conditioned by the porosity of the barrel; the more porous, the quicker and in larger volume will this action take place. The degree of porosity also regulates the admission of air into the barrel, and hence the oxidizing action that takes place during rum maturing. We have then that to greater porosity of barrel corresponds easier and faster elimination of undesirable constituents, greater and faster oxidizing action, and a saving in the aging time necessary to effect the curing. The natural porosity of the barrel may be much improved, if found necessary, during the curing process to which new barrels should be submitted before they are utilized for purposes of rum maturing.

Size of barrel is another important item to consider in the problem of rum maturing. Size is naturally related to porosity; the thicker staves of larger barrels being less porous than those of the smaller ones. Modifications of the degree of porosity are of more difficult accomplishment also in the case of the larger sized barrels. The relation of surface contact to volume of contained liquid is much higher in the smaller barrels. This means that catalytic reactions will be more intense in the latter and that the extractive substances per unit of volume of liquid will be much higher in the case of the small barrel. Maturing will then proceed at a faster rate, other conditions being the same. Very small sized containers have been found objectionable during our studies on account of the woody taste they impart to the aging product when kept in the keg for rather long periods of time. The amount of extract in the aged products are also frequently entirely too high. These drawbacks may be eliminated by a very careful preparation of the keg before it is used; but even then, total insurance against these defects is seldom obtained.

Temperature and relative humidity are factors of paramount important during the curing period. The two factors are interdependent to some extent, and both are of the greatest importance. As the temperature within the liquid in the barrel rises, greater chemical activity among the different constituents in the rum will result. The process of oxidation will also take place at an accelerated rate. The most volatile molecules in the liquid will also try to evaporate out into the atmosphere through the pores of the barrel at a greatly accelerated rate. All of these changes are conducive to the faster curing of the product. There are, however, limits to these increases of temperature over which it would be dangerous to go. If carried too far, considerable alcoholic losses may occur which would be uneconomical. And aside from the loss of alcohol, real harm to the flavor and aroma of the curing product could also be inflicted through the partial loss of valuable rum constituents together with the ethyl alcohol. We consider that different curing temperatures and conditions of relative humidity should be maintained during the aging process, according to the time allowed for completing the curing; trade demands; type of aged product desired, etc. etc.

Relative humidity may be used very effectively during the curing stage as such; and also as a complementary factor to that of temperature. We have found that when a raw rum is aged under conditions of aqueous vapor saturation of the surrounding atmosphere, a rapid lowering of the original degree proof will ensue. This is caused by the rapid exodus of ethyl alcohol molecules *together* with other molecules from the more volatile components of the Non-Alcohol-Number, such as have already been described. Water molecules are held back on account of the saturated water vapor condition in the surrounding atmosphere. On the contrary, if an especially dry atmosphere is prevalent during the aging period, water molecules will also pass out through the pores of the barrel, and in this case the proof of the aging spirit may keep practically constant or even increase. It will all depend on the comparative amounts of alcoholic and water molecules leaving the barrel through evaporation.

It would seem then advisable to keep relatively high humidity in the atmosphere of the aging room during the period necessary for the elimination of the undesirable members of the Non-Alcohol-Number. When analytical evidence shows that these undesirable elements or at least the great majority, have been eliminated, then the relative humidity may be changed so as to check the abnormal losses of alcoholic vapors.

Coming again to the question of used versus new barrels, we believe that a mistake is being made from a technical standpoint as well as from the standpoint of quality of finished product by the preference given to old whiskey barrels. Actual experiments conducted by us have proved that a great difference will be found when one and the same raw rum is aged in a new, and in an old whiskey barrel. The taste and the bouquet of the matured product from the new barrel is in all instances much to be preferred. Besides, the raw stored in the new barrel will age in a fraction of the time necessary in the other case. This will at once expose the fallacy

that used barrels are cheaper. The first cost may be less, much less, sometimes; but in the long run the final cost will be about the same, or less perhaps in the case of the new barrel. This is due to the fact we have just mentioned of the great reduction in aging time in the case of the new barrel. Also we have that the new barrel will give a longer period of effective service, and will impart the desired color to the aging liquor far quicker and more efficiently. But the greatest advantage is the purity and delicacy of taste and aroma not obtainable in the same degree when the old used barrel is employed.

As to the matter of charred vs. plain barrels, we admit that the first mentioned will accelerate the curing and the coloring of the raw spirit; but they will impart a very pronounced "woody" taste to the aging liquor, which may be unimportant in the case of whiskey curing; but which is very objectionable in the high quality rums.

The period of aging necessary for acquiring maturity will of necessity be quite variable according to conditions prevalent at each particular distillery. It is affected primarily by the quality of the raw; and secondarily by the quality, size, and class of container and the conditions of temperature and relative humidity prevalent in the aging room. Also in the facilities obtainable in different places for a careful control over temperature and relative humidity.

In our experiments we worked with both molasses and sugar cane juice rums as well as with rums made from mixed cane juice and molasses mashes in different proportions. Sugar cane juice was used in three different conditions; (1) raw (2) pasteurized and (3) defecated. The raw rums were distilled at proofs varying between 170-180 degrees. With a few exceptions, for purposes of comparison, those raws distilled at higher proof than 100 degrees were diluted to around this degree proof before barreling for curing purposes. Plain and charred barrels were used in the curing work under controlled conditions of temperature and relative humidity. The dilution of the raw spirit was effected in different manners; for instance, in some cases dilution was effected directly after distillation, and 24 hours after, the diluted raw was barreled; in other instances the dilution of the raw was done over a period of a week, adding the dilution water by small increments every day of the week; and still in other cases the raw was stored undiluted, and the dilution water added at definite periods during the aging time.

Records were kept of time of filling, amount of distillate put into the barrel, kind of barrel used etc. etc. Then every three months samples were taken from the different barrels for chemical analysis and for organoleptic tests; a record book being kept for entering this data. After one year of aging, a larger sample was taken for the ordinary analytical work, but this time a fractional distillation test was also performed on the sample, and a set of curves was drawn as graphical representation of the chemical composition of the rum under study. An alcohol, an aldehyde, an ester, a volatile acidity and a higher alcohols curved were drawn. These curves indicated the percentage alcohol by volume of each fraction distilled, and also the contents of the other mentioned components of the rum sample expressed as milligrams per 100 milliliters of the different fractions. Eight fractions were collected during the fractional distillation. A few bottles were filled with this one year old rum, and the remaining rum was allowed to cure in the barrel for another additional year. When a large enough amount of raw rum was available, the curing extended over a period of three years. The sampling of the rums was continued at intervals of three months during the whole curing period of two or three years, and the corresponding analytical work was effected on these samples. Bottles were filled again representative of the two years and three-years-old products. The corresponding fractional distillations were also performed in each case. About 150 rum samples were distilled and aged as above indicated during the period that this investigation lasted.

Table 25 presents data obtained during the first year of aging of molasses rums that were diluted to about 100 degrees proof immediately after distillation.

It may be noticed at once the lowering effect on the ester value of the raw rum produced by the diluting process, particularly in the cases of rums Nos. 4, 5 and 6 that were of fairly high ester contents. As a matter of fact the ester values of rums Nos. 4 and 5 remained lower than in the raw undiluted samples during the entire twelve months period; while the ester value of No. 6 sample was only slightly superior to the original value in the raw undiluted sample after the same aging period. But comparing the final ester values with those of the diluted raw rum samples, we find considerable increase in all cases. We can then say that aging of a raw rum produces increased ester value even in the period of one year. The increase in total acidity is quite noticeable, being in fact quite remarkable. In some cases the total acidity increases from 10 to 20

times the original value in the raw diluted rum. There is not much change in the aldehydic contents during the first year of aging; and in general, the higher alcohols remain constant. Hence, it could well be stated that the main characteristic changes in the chemical constitution of aging molasses rums is a remarkable increase in total acidity, and a much smaller increase in ester value, when the original raw distillate is appreciably diluted immediately after distillation to about 100 degrees proof with distilled water. The increase in the ester value is more apparent when the diluted raw rum is used as basis of comparison; but this is not so apparent if the undiluted raw spirit is used as the basis of comparison. We may add that in general there is a small drop in the alcoholic concentration of the aging rum.

Table 26 presents similar data to that of table 25 with the exception that in this case the rums were not diluted immediately after distillation, but at different stages of the curing process. Two of the samples were kept undiluted during the whole curing period. Samples one and two were kept undiluted throughout the test; sample three was diluted after the first three months aging; samples four, five and six, were diluted after six months aging, and sample seven was diluted after the nine months period of aging.

In a comparative study of the data shown in tables Nos. 25 and 26 respectively, it is noticed at once that the characteristic drop in ester value during the early period of aging is no longer observed. A steady, though not large increase, is noticed in the first two samples that remained undiluted throughout the test. A severe drop is noticed in sample 3 when it was diluted after aging for a period of three months. The ester determination in the six months old sample renders a value much inferior than that obtained after aging for three months. This low ester value increases somewhat during the rest of the curing period but never reaches the original ester content of the raw rum. It may be said that from the time it was diluted this sample acted similarly to those of table 25 as regards changes in ester values. Samples 4, 5 and 6, were diluted after the six months aging period had passed. Here again the same phenomenon of ester hydrolysis takes place; a very comparative low value is obtained in the analysis of the respective non months old samples, which recovers a little by the time of the twelve months old rum analyses. The deduction about the ester question of hydrolysis on dilution, is that the phenomenon will happen not only when the raw rum is originally diluted before filling the aging barrels, but also

RESEARCH BULLETIN - STUDIES ON RUM

TABLE No 25

ALC	OHOLIC ST	RENGHI BY VUI	LUME IMMEDIA	TELY AFTER DIST				
				After Ageing During				
Determinations	Sample	Undiluted	Diluted	3	6	9	12	
	No	Raw Rum	Raw Rum	Months	Months	Months	Months	
	-1-							
(1) Sp. Gr. At 20/4 deg. C	-1-	0.86805	0.92516	0.92641	0.92522	0.92586	0.92428	
(1) Sp. Gri Ri 26/ Paeg. C		76.80	52.50	51.88	52.47	52.15	52.93	
(3) Total Acidity (mgs 100mL Abs. Alc		11.90	8.70	62.40	73.70	150.70	174.40	
(4) Aldehydes (mgs 100mL Abs. Alc		39.60	41.30	66.30	56.50	45.60	40.20	
(5) Esters (mgs 100mL Abs. Alc		61.87	47.10	42.70	60.40	67.50	109.80	
(6) Higher Alcohols (mgs 100 mL Abs. Alc		181.90	177.90	175.40	173.40	174.40	173.00	
(1) So $C_{\rm T}$ At 20/4 day C	-2-	0.86202	0.02060	0.02221	0.02252	0.02299	0.02294	
(1) Sp. Gr. At 20/4 deg. C		0.86393 78.33	0.93069 49.75	0.93321 48.44	0.93352 48.27	0.93388 48.35	0.93384 48.10	
(2) Alcohol by volume, Per Cent(3) Total Acidity (mgs 100mL Abs. Alc		13.60	7.90	48.44 47.90	48.27 67.30	48.55 108.40	153.00	
(4) Aldehydes (mgs 100mL Abs. Alc		46.30	50.80	47.90	30.20	34.10	30.40	
(4) Aldenydes (mgs 100mL Abs. Alc		40.30 51.70	41.11	37.40	40.81	43.70	80.50	
(6) Higher Alcohols (mgs 100 mL Abs. Alc.		299.30	290.40	288.20	288.70	288.80	289.00	
		277.50	290.40	200.20	200.70	200.00	207.00	
	2							
(1) S = C = A + 20/4 + c = C	-3-	0.05026	0.02017	0.02252	0.02200	0.02702	0.02692	
(1) Sp. Gr. At 20/4 deg. C.		0.85836	0.93017	0.93253	0.93309	0.93793	0.93683	
(2) Alcohol by volume, Per Cent		80.35 29.50	50.00 37.10	48.80 121.30	48.50 172.70	45.91 188.20	46.50 211.00	
(3) Total Acidity (mgs 100mL Abs. Alc		29.30 68.30	72.20	52.00	58.70	74.30	82.40	
(4) Aldenydes (higs 100mL Abs. Alc		100.80	92.00	86.50	83.50	128.60	153.30	
		172.10	102.90	170.00	175.00	105.70	107.00	
(1) Sp. $Gr. At 20/4 dag. C$	-4-	0 97792	0.02408	0.02728	0.02655	0.02707	0.02771	
 (6) Higher Alcohols (mgs 100 mL Abs. Alc. (1) Sp. Gr. At 20/4 deg. C. (2) Alcohol by volume, Per Cent	-4-	0.87783 73.07 23.40 65.00 135.20 36.50	0.93498 47.50 19.17 73.00 94.71 38.00	0.93738 46.21 107.20 82.40 87.70 39.50	0.93655 46.55 144.00 48.00 101.20 39.10	0.93707 46.37 169.50 59.30 108.50 39.40	$\begin{array}{r} 0.93771\\ 46.03\\ 227.60\\ 43.70\\ 118.60\\ 39.60\end{array}$	

SHOWING DATA OBTAINED DURING THE FIRST YEAR OF CURING OF MOLASSES RUMS DILUTED TO ABOUT 50 PER CENT ALCOHOLIC STRENGHT BY VOLUME IMMEDIATELY AFTER DISTILLATION

RESEARCH BULLETIN - STUDIES ON RUM								
 (1) Sp. Gr. At 20/4 deg. C. (2) Alcohol by volume, Per Cent	-5-	$\begin{array}{c} 0.87125 \\ 75.60 \\ 73.00 \\ 41.90 \\ 162.20 \\ 33.70 \end{array}$	$\begin{array}{c} 0.93545 \\ 47.25 \\ 67.14 \\ 46.80 \\ 115.40 \\ 34.10 \end{array}$	0.93785 45.95 151.40 75.30 105.40 36.60	$\begin{array}{c} 0.93752 \\ 46.14 \\ 177.10 \\ 108.60 \\ 121.60 \\ 36.80 \end{array}$	0.93806 45.84 220.50 71.80 188.20 36.50	0.93806 45.83 304.90 51.90 142.00 36.80	
 (1) Sp. Gr. At 20/4 deg. C. (2) Alcohol by volume, Per Cent (3) Total Acidity (mgs 100mL Abs. Alc	-6-	0.87561 73.93 20.30 35.70 123.70 118.30	$\begin{array}{c} 0.93385 \\ 48.10 \\ 25.00 \\ 41.80 \\ 91.50 \\ 123.40 \end{array}$	0.93634 46.78 67.80 68.80 80.60 126.00	$\begin{array}{c} 0.93710 \\ 46.36 \\ 123.80 \\ 61.70 \\ 91.00 \\ 127.60 \end{array}$	$\begin{array}{c} 0.93744 \\ 46.18 \\ 216.10 \\ 55.40 \\ 102.80 \\ 128.10 \end{array}$	0.93921 45.20 204.40 48.70 136.80 130.80	

NOTE – The different rums were aged in plain 5-gal kegs. All determinations except that of alcohol by volume, are expressed in milligrams per 100 milliliters absolute alcohol.

RESEARCH BULLETIN - STUDIES ON RUM

TABLE No 26

				After Agei	ng During	
Determinations	Sample No	Raw Rum	3 Months	6 Months	9 Months	12 Months
 (1) Sp. Gr. At 20/4 deg. C. (2) Alcohol by volume, Per Cent (3) Total Acidity (mgs 100mL Abs. Alc	-1-	$\begin{array}{c} 0.86397 \\ 78.30 \\ 33.50 \\ 65.40 \\ 384.40 \\ 76.90 \end{array}$	0.88154 71.63 95.60 64.80 456.20 84.10	0.87948 72.43 130.00 78.70 457.40 83.10	0.88212 71.38 161.50 79.90 493.10 84.30	0.88554 70.02 200.50 94.70 520.80 85.90
 (1) Sp. Gr. At 20/4 deg. C. (2) Alcohol by volume, Per Cent (3) Total Acidity (mgs 100mL Abs. Alc. (4) Aldehydes (mgs 100mL Abs. Alc. (5) Esters (mgs 100mL Abs. Alc. (6) Higher Alcohols (mgs 100 mL Abs. Alc. 	-2-	$\begin{array}{c} 0.87743 \\ 73.23 \\ 24.70 \\ 135.80 \\ 193.40 \\ 285.00 \end{array}$	0.88054 72.00 53.90 106.30 198.00 289.80	0.88688 69.48 59.40 108.90 205.20 279.70	0.88583 69.90 62.70 102.40 211.16	0.88506 70.21 89.40 126.20 223.00
 (1) Sp. Gr. At 20/4 deg. C. (2) Alcohol by volume, Per Cent (3) Total Acidity (mgs 100mL Abs. Alc. (4) Aldehydes (mgs 100mL Abs. Alc. (5) Esters (mgs 100mL Abs. Alc. (6) Higher Alcohols (mgs 100 mL Abs. Alc. 	-3-	0.87217 75.23 54.90 31.00 286.50 89.70	0.88632 69.70 119.00 66.70 306.50 97.00	0.92436 52.89 165.20 84.70 215.20 84.80	0.92278 53.64 206.90 44.30 226.30 83.60	0.92331 53.40 259.30 44.60 263.60 84.00
 (1) Sp. Gr. At 20/4 deg. C. (2) Alcohol by volume, Per Cent (3) Total Acidity (mgs 100mL Abs. Alc. (4) Aldehydes (mgs 100mL Abs. Alc. (5) Esters (mgs 100mL Abs. Alc. (6) Higher Alcohols (mgs 100 mL Abs. Alc. 	-4-	0.85786 80.50 28.70 34.20 127.10 223.90	0.87287 74.98 55.00 65.90 181.50 240.30	$\begin{array}{c} 0.87125 \\ 75.60 \\ 64.90 \\ 49.10 \\ 148.90 \\ 241.60 \end{array}$	0.92718 51.50 91.90 51.90 116.10 234.50	0.92793 51.13 111.10 59.30 122.90 237.10

SHOWING SIMILAR DATA TO THAT OF TABLE 25 EXCEPT THAT IN THIS CASE THE RAW RUMS WERE NOT DILUTED IMMEDIATELY AFTER DISTILLATION, BUT DURING DIFFERENT STAGES OF THE AGEING PERIOD.

RE	SEARCH	I BULLETIN	- STUDIES O	N RUM		
 (1) Sp. Gr. At 20/4 deg. C. (2) Alcohol by volume, Per Cent (3) Total Acidity (mgs 100mL Abs. Alc. (4) Aldehydes (mgs 100mL Abs. Alc. (5) Esters (mgs 100mL Abs. Alc. (6) Higher Alcohols (mgs 100 mL Abs. Alc. 	-5-	0.86568 77.67 87.50 42.30 158.60 229.00	0.88241 71.28 150.60 33.40 179.70 249.50	0.87684 73.45 169.00 31.70 194.10 239.70	0.92152 54.28 203.50 42.70 152.30 209.70	0.92177 54.15 239.90 52.60 188.60
 (1) Sp. Gr. At 20/4 deg. C. (2) Alcohol by volume, Per Cent (3) Total Acidity (mgs 100mL Abs. Alc. (4) Aldehydes (mgs 100mL Abs. Alc. (5) Esters (mgs 100mL Abs. Alc. (6) Higher Alcohols (mgs 100 mL Abs. Alc. 	-6-	0.85670 80.93 17.30 43.00 107.60 211.80	0.86512 77.90 92.90 42.20 123.00 220.00	0.86301 78.65 87.10 31.90 171.70 217.90	0.92366 53.25 116.20 50.10 112.20 194.30	0.92550 52.33 129.90 54.50 134.50 197.90
 (1) Sp. Gr. At 20/4 deg. C. (2) Alcohol by volume, Per Cent (3) Total Acidity (mgs 100mL Abs. Alc	-7-	0.86053 79.55 37.70 34.60 84.10 241.50	0.87998 72.23 89.80 43.00 132.60 255.90	$\begin{array}{c} 0.87810 \\ 72.95 \\ 148.10 \\ 42.30 \\ 134.00 \\ 255.00 \end{array}$	0.87892 72.65 179.20 59.20 159.30	0.93051 49.83 168.20 57.20 117.20

at any other time that dilution may take place afterwards. Rum sample 7 that was diluted after nine months of curing underwent exactly the same ester loss through hydrolysis.

Again the total acidities increase rapidly during the aging period although not so spectacularly as in the case of the samples of table 25. The aldehydes show an average slight increase vs. an average very slight decrease in the case of the samples of table 25. As to the higher alcohols, they again show but little variation if we take into consideration that an error of five to fifteen milligrams in this particular determination is entirely admissible even in simultaneously run determinations of the same sample.

The striking differences between the analytical figures of both tables as regards variations of the products during the aging test, are (1) the fact that the ester values show a consistent increase as the aging progresses as long as no dilution of the samples takes place; (2) the loss of alcoholic concentration of the rum samples during the aging period is much less when the original raw rums are diluted immediately after their distillation.

Table 27 presents similar data to that already reported in the case of molasses rums; but in this instance the rums were distilled from sugar cane juice fermented mashes. The raw material was used after passing through the usual process of defecation and clarification.

It will be noticed that the total acidities in the raw samples of these sugar cane juice rums are generally of much lower values than those of the molasses rums considered in tables 25 and 26; and also that the ester losses due to dilution are not so great. In our view, one fact explains the other, as greater ester hydrolysis would be expected as the medium in which the reaction takes place becomes of a more acidic character. The original ester values are also less in the case of the cane juice rums, which also explains the less pronounced effect upon these rum constituents of the added diluent. As to the aldehydes, they seem to behave in a manner analogous to the case of the molasses rum samples; and the same may be said of the higher alcohols, where very little differences are found between the aged, and the corresponding raw products.

Sample six of table 27 differs from the rest of the list in that this product was distilled at a very low proof, so that no dilution was needed before barreling the raw rum for curing purposes. In this case it will be noticed that the ester value increases steadily during the aging period until at the end of one year it is almost

three times as large as the original value. This does not occur in any of the other samples listed. It could be stated then, that sugar cane juice rums are not so rapid nor so conspicuous as in the case of molasses rums. We have called attention before to the fact that molasses rums attain maturity faster than sugar cane juice rums. Tables 28 and 29 respectively, present the analyses of some one year, two years, and three years old rums of molasses and sugar cane juice origins; and table 30 shows the average analysis for each class of rum at different aging periods.

A study of the data presented on these tables shows that the acidity continues to increase during the second year of aging at a very appreciable rate although not quite as fast as it does during the first year. This is the case both for molasses and for sugar cane juice rums. During the third year of aging the increase in acidity is not very significant in either case. While during the first year of aging the aldehydes behave in a variable and inconsistent way, we find that during the second and third year of aging the aldehyde value shows a steady moderate increase in both types of rums. The esters remain always of larger values in the case of the molasses rums; the one year old molasses rum comparing in some instances in this respect with the three years old sugar juice rums. A slight decrease in the contents of higher alcohols is noticeable, especially after the third year of aging. This may be due to the effect of esterification of some of the higher alcohols during the aging period.

After the first year of curing, the average molasses rum shows a far better balanced chemical constitution than that of the average sugar cane juice rum, which fact demonstrates faster maturing qualities on the part of molasses rums. This superior chemical structure of the molasses rums becomes more apparent when a fractional distillation test is effect as complement to the regular chemical analysis. We shall take up this matter again in a future chapter of this bulletin. We have already explained the importance of the relations or ratios among the group constituents in the constitution of a rum, and how these ratios affect the appraisal of the quality of a rum, and how these ratios affect the appraisal of the beverage. If we examine some of these ratios in relation to the data of table 30 we have that in the case of the respective one year old rums, the ester to acidity; and that of Non-Alcohol-Number

RESEARCH BULLETIN - STUDIES ON RUM

TABLE No 27

DEFECTED AND CLARIFI					After Agei		
Determinations	Sample No	Undiluted Raw Rum	Diluted Raw Rum	3 Months	6 Months	9 Months	12 Months
 (1) Sp. Gr. At 20/4 deg. C. (2) Alcohol by volume, Per Cent	-1-	0.85338 82.09 12.70 74.20 82.60 144.60	0.93355 48.26 17.60 66.70 72.90	0.93516 47.40 81.00 51.30 51.90	0.93471 47.64 125.14 44.00 62.80	0.93284 48.63 187.90 59.80 97.70	0.93359 48.24 222.20 54.30 113.20 138.90
 (1) Sp. Gr. At 20/4 deg. C. (2) Alcohol by volume, Per Cent	-2-	0.84916 83.55 11.70 42.90 63.20 158.10	0.93303 48.53 6.40 51.70 54.40	0.93805 45.84 35.60 46.90 46.00	0.93725 46.28 63.30 40.20 76.10	0.93564 47.15 109.40 46.20 87.00 -	0.93669 46.58 133.10 48.70 119.40 145.10
 (1) Sp. Gr. At 20/4 deg. C. (2) Alcohol by volume, Per Cent	-3-	$\begin{array}{c} 0.85882 \\ 80.17 \\ 5.30 \\ 46.90 \\ 41.60 \\ 114.40 \end{array}$	0.93205 49.04 4.90 40.20 39.60	0.93516 47.40 42.60 39.20 33.30	0.93675 46.55 58.70 36.86 71.83	0.93712 46.35 107.20 47.00 75.90	0.93605 46.92 125.90 57.30 95.00 117.28
 (1) Sp. Gr. At 20/4 deg. C. (2) Alcohol by volume, Per Cent (3) Total Acidity (mgs 100mL Abs. Alc	-4-	$\begin{array}{c} 0.85913 \\ 80.05 \\ 8.40 \\ 56.00 \\ 49.50 \\ 98.60 \end{array}$	0.93356 48.25 9.50 44.60 43.70	0.93605 46.93 33.00 36.70 56.30	0.93695 46.44 50.40 37.00 59.80	0.93561 47.16 92.60 39.60 60.70	$\begin{array}{c} 0.93606 \\ 46.92 \\ 108.00 \\ 43.20 \\ 63.70 \\ 101.50 \end{array}$

DATA OBTAINED DURING THE CURING OF SUGAR CANE JUICE RUMS FOR A PERIOD OF ONE YEAR DEFECTED AND CLARIFIED SUGAR CANE JUICE WAS USED DURING THE FERMENTATION OF THESE RUM SAMPLES

RESEARCH BULLETIN - STUDIES ON RUM								
 (1) Sp. Gr. At 20/4 deg. C (2) Alcohol by volume, Per Cent (3) Total Acidity (mgs 100mL Abs. Alc	-5-	0.85422 81.80 6.00 56.90 59.00 111.00	0.92739 51.40 4.70 52.30 56.40	0.93425 47.89 39.30 37.20 55.10	0.93524 47.36 39.10 42.90 59.50	0.93492 47.53 83.50 45.40 62.40	0.93416 47.94 99.90 53.80 88.80 95.90	
 (1) Sp. Gr. At 20/4 deg. C. (2) Alcohol by volume, Per Cent	-6-	$\begin{array}{c} 0.92977 \\ 50.20 \\ 13.30 \\ 34.30 \\ 36.90 \\ 142.80 \end{array}$	- - - - - -	0.93622 46.84 75.02 29.02 48.85	0.93662 47.15 143.20 55.60 67.20	0.93565 47.14 168.40 55.90 69.50 -	0.93387 48.09 169.90 61.70 97.10 132.90	

NOTE – The rum samples were aged in 5 gals plain, white oak kegs. All determinations except that of alcohol by volume are expressed in milligrams per 100 milliliters of absolute alcohol.

RESEARCH BULLETIN - STUDIES ON RUM

				CHEMICAL	ANALYSIS		
Sample No.	Time Aged (Years)	Sp. Gr. At 20/4 Deg. C	Alcohol by Volume Per Cent	Total Acidity	Aldehydes	Esters	Higher Alcohols
1	ONE	0.93791	45.90	250.00	27.80	149.40	76.10
2	ONE	0.93421	47.90	234.40	30.50	139.60	164.00
3	ONE	0.92985	50.15	179.30	25.50	136.80	84.00
4	ONE	0.93702	46.40	217.90	51.30	151.70	130.00
5	ONE	0.93606	46.93	303.20	49.40	187.50	114.00
6	ONE	0.92647	51.85	158.70	49.00	91.60	233.90
7	ONE	0.93236	48.88	129.90	60.70	75.70	181.00
8	ONE	0.92747	51.35	190.80	35.80	119.90	121.80
9	ONE	0.93045	49.85	255.80	78.00	172.90	215.00
10	ONE	0.93143	49.35	164.11	28.77	89.14	181.80
11	TWO	0.93900	45.32	367.20	79.20	275.80	75.00
12	TWO	0.93868	45.49	488.40	102.40	278.50	148.00
13	TWO	0.93125	49.43	271.30	70.00	227.80	80.20
14	TWO	0.93796	45.89	305.70	91.30	253.20	120.90
15	TWO	0.93601	46.96	462.20	83.90	247.40	112.50
16	TWO	0.92726	51.47	270.30	94.80	157.40	232.99
17	TWO	0.93100	49.58	193.80	87.50	152.70	161.00
18	TWO	0.92500	52.58	253.20	82.50	177.50	101.50
19	TWO	0.92954	50.31	281.30	113.90	223.80	206.10
20	TWO	0.92796	51.11	231.50	84.20	182.66	173.60
21	THREE	0.93526	47.35	327.21	128.48	317.36	168.10
22	THREE	0.93366	48.20	395.10	107.50	286.70	145.10
23	THREE	0.93376	48.15	301.52	103.02	230.30	71.80
24	THREE	0.93589	47.01	275.40	105.50	299.70	82.60
25	THREE	0.93600	46.95	318.89	100.31	225.57	115.20

TABLE NO. 28 PRESENTING THE ANALYSIS OF ONE, TWO, AND THREE-YEARS-OLD MOLASSES RUMS. MEMBERS OF THE NON-ALCOHOL-NUMBER REPORTED AS MGS, PER 100ML ABS. ALC.

TABLE NO. 29

PRESENTING THE ANALYSIS OF ONE, TWO, AND THREE-YEARS-OLD DEFECATED SUGAR CANE JUICE RUMS. MEMBERS OF THE NON-ALCOHOL-NUMBER REPORTED AS MGS, PER 100ML ABS ALC.

			CHEMICAL ANALYSIS							
Sample No.	Time Aged (Years)	Sp. Gr. At 20/4 Deg. C	Alcohol by Volume Per Cent	Total Acidity	Aldehydes	Esters	Higher Alcohols			
1	ONE	0.93606	46.92	108.00	39.60	56.30	147.90			
2	ONE	0.93416	47.94	99.90	53.80	55.10	198.90			
3	ONE	0.93172	49.12	108.70	52.40	57.30	216.50			
4	ONE	0.93223	48.95	132.40	50.70	53.90	131.80			
5	ONE	0.93169	49.23	108.70	57.70	53.60	205.15			
6	ONE	0.94070	44.36	44.40	51.20	38.60	97.40			
7	ONE	0.93187	49.14	100.30	54.70	53.70	98.81			
8	ONE	0.93387	48.09	169.90	55.90	59.50	142.80			
9	ONE	0.93786	45.94	214.60	58.50	61.30	151.40			
10	ONE	0.93103	49.56	127.90	87.60	124.30	147.30			
11	TWO	0.93964	44.96	187.71	70.54	58.71	141.34			
12	TWO	0.93887	45.39	189.94	64.50	104.69	192.87			
13	TWO	0.93738	46.21	193.74	63.36	114.26	202.62			
14	TWO	0.93872	45.47	255.22	75.12	123.85	123.01			
15	TWO	0.93923	45.20	183.37	70.16	128.47	190.98			
16	TWO	0.93949	45.05	95.70	65.00	66.42	80.99			
17	TWO	0.94081	44.31	202.07	77.10	119.17	98.81			
18	TWO	0.94175	43.78	272.66	61.30	112.56	122.20			
19	TWO	0.93874	45.46	313.70	75.15	131.65	147.14			
20	TWO	0.93762	46.08	208.67	63.54	148.95	127.80			
21	THREE	0.93931	45.15	224.34	93.38	132.55	121.11			
22	THREE	0.94016	44.67	223.43	90.99	189.15	130.11			
23	THREE	0.94055	44.45	199.58	68.58	166.32	141.20			
24	THREE	0.94052	44.47	299.25	73.11	201.87	98.50			

TABLE NO. 30

Determinations	AVERAGE CHEMICAL ANALYSIS OF:								
	ONE-YEA	AR OLD	TWO-YE	ARS OLD	THREE-YEARS OLD				
	Molasses Rums	Cane Juice Rums	Molasses Rums	Cane Juice Rums	Molasses Rums	Cane Juice Rums			
Sp. Gr. At 20/4 Deg C. Alcohol by Volume, % Total Acidity, (mg/100mL Ab. Alc.) Aldehydes, (mg/100mL Ab. Alc.) Esters, (mg/100mL Ab. Alc.)	0.93241 48.85 208.41 43.67 131.42	0.93418 47.92 121.49 56.31 62.36	0.93251 48.80 312.49 88.97 217.67	0.93921 46.19 212.71 68.57 110.87	0.93493 47.52 323.62 108.96 271.90	0.94016 44.68 236.65 81.52 172.50			
Higher Alcohols, (mg/100mL Ab. Alc.)	150.16	149.80	143.18	142.20	117.56	122.73			

SHOWING AVERAGE ANALYSES FOR EACH CLASS OF RUM AT DIFFERENT AGEING PERIODS. MEMBERS OF THE NON-ALCOHOL-NUMBER ARE REPORTED IN MGS/100ML ABS. ALC.

to higher alcohols, are superior in every case in the molasses than in the sugar cane juice rum. It will also be noticed that the chemical structure and the ratios among the group constituents improve conspicuously in the case of the sugar cane juice rums, while in the case of the molasses rums the improvement is merely as to increase in aromatic compounds, and not in the relations among the different group constituents. As a matter of fact, the one year old average rum sample is as perfectly balanced as the three year old, in so far as the different ratios and relations among the chemical constituents in each rum. This comparison would not hold true in the case of the sugar cane juice rums, where it may be readily observed that the three year old sample is by far superior in chemical structure to the one year old. All of these facts go to prove that molasses rums will mature in a fraction of the time required for the maturing of sugar cane juice rums; a great advantage on the part of natural curing of molasses rums.

We shall now close this chapter on the maturing of raw rums by touching lightly on the matter of accelerated curing of the raw distillate. Not all, or rather few, of the rums in the market have passed through a curing process such as we have outlined above. Our era of acceleration and impatience in all affairs of human endeavor would not allow of an exemption in the case of rum making. On the other hand, the ever increasing demands of the trade, the lack of adequate working capital, the anxiety for immediate returns, immoderate and unfair competition, and many other influences of business, compel the manufacturers to place their products on the market in the shortest possible time. As a direct result of the above-mentioned conditions, accelerated or quick aging processes have been developed, and are being developed all the time. There exist practically as many "secret methods" of artificial curing of rums as rectifiers are engaged in the business. Judging from what has been accomplished thus far, and from the nature and quality of the "rums" thus produced, the writer's opinion is that the results obtained are very mediocre and unsatisfying; leaving the problem of artificial rum curing an open question.

Processes for rapid curing may be divided into two general classes: (1) Those merely tending to accelerate the reactions and changes occurring during natural aging, and in this way accomplishing maturity of raw product in a short time; but without the addition to the raw of extraneous substances, the so-called carriers of taste, aroma, and body. (2) Those intended to accomplish the results mentioned under (1); but using besides these extraneous matters, imparters of taste, aroma and body. The methods used under (1) will fall into four main divisions: (a) moderate heat treatment or intense cold treatment; or alternate treatment of heat and cold; (b) treatment with compressed air; oxygen, hydrogen peroxide or ozone; (c) exposure to actinic rays; (d) electrolytic treatment and use of catalysis. Methods under (2) above, may include all of the methods under (1); besides the addition of flavoring and aromatic substances for development of taste and bouquet. Among these added substances we may mention: (a) various types of sweet wines, among which the various "Moscateles" and "Málagas" from Spain; and prune wines from Scotland are much in vogue; (b) infusions of herbs, leaves, barks of trees, roots, etc. etc.; (c) alcoholic and aged fruit extracts, among which peaches, prunes, figs and apricots are much used; (d) artificial essences of rum or brandy; \in various natural and synthetic essential oils, and flavoring extracts as cassia oil, oil of cloves, artificial or natural vanilla flavor, oil of bitter almonds (free of hydrocyanic acid); and various sugars, as sucrose, dextrose, sugar cane syrup, maple syrup, and bee honey.

In this guise, beverages are made that although more deserving of the name of cordials or liqueurs, are labeled with the name of rum. We believe that all of this is avoidable and unjustified, should more and better attention be bestowed on the different stages of rum manufacture, and especially on rum yeast selection. Governmental regulations and inspection of the rums produced and sold in the local and United States markets would be a great help towards fostering the interests of the industry, and securing the genuine article for public consumption.

VIII

Rum Aroma

The writer wishes that it be clearly understood that when treating upon the subject of the aroma or bouquet of rum, reference is made exclusively to the natural aroma of a genuine rum, matured through natural means in the traditional oak barrel. Artificially imparted aromas are entirely beyond the scope of this chapter.

The aroma of rum is the result of two sets of factors: (1) the natural; and (2) those depending on the technique of manufacture. Among the natural factors we have the raw material, the yeast strain, and bacterial intervention. Among the factors depending on the technique of manufacture we have the influence of conditioning and purifying the raw material previous to its actual use in mashing operations; mashing and fermentation methods employed; type of still and technique used during distillation, and care and control exercised during the curing process.

The nature of the raw material used, no doubt exercises a profound influence on the aroma of rum. The best of rum yeasts employed in the fermentation of a beet molasses mash will not yield a product to which the name of rum could be applied. On the other hand, the best quality of sugar cane molasses will not produce rum, if fermented with a beer yeast strain. The chemical composition of the proper raw material, as for instance cane molasses, will have a great deal to do with that of the rum produced, and hence with nature of its aroma. For instance, the relative amounts of proteins contained in different molasses, will determine to a great extent the degrees in the contents of higher alcohols, free fatty acids, and esters found later in the raw spirit. The aroma of the raw rum will then be influence by both the quantity, and quality of each group of aromatic constituents. According to the above statement, final sugar cane molasses, the so-called blackstrap, will yield a rum with different aroma than one produced from sugar cane syrup or first molasses, due to its higher content in protein matter. Also, and for the same reason, a raw sugar cane juice will produce a raw rum quite different in aromatic tone to that of defecated and clarified juice. The rum produced by the raw sugar cane juice will be much richer in the makeup of its Non-Alcohol-Number than that obtained from the defecated and clarified juice. Hence, raw cane juice will tend to produce the "heavy" type of rum, while defecated and clarified cane juice will tend to produce a very light type of rum. Our experiments dealing with sugar cane juice rums have demonstrated that:

(1) The rums produced from raw cane juice are more aromatic, but the aroma lacks the finesse of those produced from defecated and clarified sugar cane juice. These rums also will take a longer period of aging to reach maturity. When the organoleptic tests for aroma are applied to a sample of this type of rum, it gives the impression that a blend of various rums rather than a single one is being tested; in a word, the rum aroma lacks stability and uniformity, being also rather pungent.

(2) The rums obtained from pasteurized sugar cane juices, but not clarified, occupy an intermediate position with respect to the quality of the aromatic gama. Their aroma is more uniform, stable, suave and delicate. It possesses a more harmonious blend of the different components. Complexity of aroma may be present, but it is stable; and presents a single aromatic effect.

(3) The rums produced from the defecated and clarified sugar cane juice have the same general characteristics as those produced in the case of pasteurized cane juice; but they possess, besides, greater amplitude and penetration of aroma. Certain tinge of the peculiar odor of matured rums may also be observed in the bouquet of these particular products.

The natural aromatic constituents present in the raw materials used will become another important factor in the bouquet of the resulting rum. In this particular we have that different varieties of sugar canes yield juices with different classes and amounts of aroma. Also in a given cane variety, the aromatic tone will differ according to state of maturity, time elapsed between cutting and grinding the cane, whether burned or fresh cane is cut and ground, etc. etc.

As to the sugar cane final molasses we have that they also have different natural aromas, and in some cases the odor of the raw material may be indifferent or even offensive. Whether good and agreeable, or bad and offensive, the natural odor of the molasses shall undoubtedly influence the aroma of the rums produced from them. A steam distillation test should always be made on a sample of the molasses to be purchased. The first 50 or 100 ml of distillate obtained, will show the character of the natural aroma of the sample. This test for natural aroma of the raw material is very important in another respect, as in some cases the natural aroma of the molasses itself may be acceptable, but during

transit to the distillery the molasses may, through carelessness, become badly contaminated with substances that would render it unfit for rum making; as for instance, fuel oil.

The nature of the yeast used to ferment the raw material has as much influence at least, as the raw material itself. As already explained in the chapter on yeast selection, every rum yeast strain will produce a different aroma in the rum made through its agency. The characteristics in the different strains that have more to do in this development of exclusive aromas, conform to the extent and ability of each variety to produce certain enzymes, and the selective action exercised by these enzymes in their attack upon the chemical constituents of the substrate. For instance, a certain strain may produce proteolytic enzymes in greater amount and variety than another. In such case, the strain in question would break up the protein molecules more readily than the other, and through the deamination of the resulting amino acids give rise to a series of higher alcohols and fatty acids in the substrate. The aroma of the rums produced by each of the above-mentioned yeasts would naturally be quite different.

But there is still another difference of behavior among rum yeasts that exercise a far greater influence in aroma differentiation and strength than the factor we have just discussed. This is the ability of different yeasts to produce essential oils, especially the so-called rum oil.

We have already mentioned in our chapter on yeast selection the paramount importance of rum oil production by the selected yeast. In fact, the ability of a given yeast strain to produce fair quantities of this aromatic constituent during rum fermentation entirely overshadows any other favorable qualification; for this aromatic constituent is mainly responsible for the peculiar and characteristic rum aroma. This aroma is not to be found in any other distilled spirit, and is, besides, of very difficult imitation.

For our part, we firmly believe rum oil to be the most influential and controlling factor in originating rum taste and aroma; and all veritable rum yeast should be a good, or at least, a fair producer of this essential oil. Prior to the researches of Micko, already alluded to in the course of this writing, rum oil did not even figure as one of the constituents in the Non-Alcohol-Number of genuine rums. Later on, Guillaume (³³) mentioned the important and interesting fact that the specific aroma of the Martinique molasses rums seemed to be produced by an essential oil resulting from the fermentative

action of the yeasts employed in their manufacture. And such, we here in Puerto Rico, have found to be the case in the production of our genuine rums. Rum oil is not an ester, aldehyde, ketone or acetal; yet it is the all-essential element in the development of genuine rum aroma. The other aromatic constituents of the Non-Alcohol-Number of a genuine rum help, most surely, in the formation of the fully developed and finished aroma; but the rum oil forms the foundation of this unique scent.

The question naturally arises as to how is this essential oil produced. Is it an exclusive product of the yeast's metabolism? Is it a constituent of the raw material existing in some other form, which is transformed or converted into this particular essential oil during fermentation through the action of the yeast? A definite answer we can not give; but the conclusions we have arrived at from our observations and experiments are that:

(1) The formation of the oil during fermentation seems to be a function of both the yeast and the substrate.

(2) All of the yeast strains used in our experiments did not produce the oil in the same quantity, although working the same substrate under the same conditions.

(3) Milk of lime treatment of the raw material as explained in our chapter on mashing and pretreatment, resulted in the obtention of greater amounts of rum oil during the fermentation.

(4) Differences in the aromas of the resulting oils could be noticed when using quite different yeast strains' for instance, the rum oil produced by fermenting the substrate with one of our Schizosaccharomyces was different in aroma to that produced by one of our budding rum yeasts. This difference was of course small, and the general character of the aroma was quite similar; but the difference was readily recognizable after a little experience.

(5) Discontinuously distilled rums proved to be of higher rum oil content than those produced in continuous stills.

In order to prove in a more objective way the supreme importance of rum oil in the compound and complex bouquet of rum, we endeavored to produce synthetic rums in our laboratory. Natural rums were analyzed and then the various analyses were copied and followed in the composition of our synthetic products, keeping the same proportion found in the genuine natural rums as to the structures of the Non-Alcohol-Numbers. But in the synthetically prepared rums no rum oil was used. The results were entirely unsatisfactory, not a single one of the synthetic formulas produced a beverage that could be recognized as a rum.

When as little as one hundred parts per million of a previously prepared one per cent solution of rum oil in absolute alcohol, were added to each of the synthetic rums, their respective aromas suffered a wonderful transformation, the characteristic rum odor appearing in every case. The result of these experiments established further with added conviction and force that fact that without rum oil there is no true rum, irrespective of the variety and amounts of other aromatic ingredients that may be present. On the other hand, rum oil and ethyl alcohol alone will not produce a rum, proving that while the oil is the essential characteristic of a veritable rum, it must be recognized and accepted that other aromatic constituents in the form of aldehydes, esters, higher alcohols, fatty acids, etc. etc., are also necessary to complete and enhance the aromatic gama.

Another way in which the inherent characteristics of the yeast strain influence the aroma of rum is the lapse of time necessary for different strains to complete fermentation of the sugars in the substrate. We have met with yeasts requiring 24 or 36 hours to complete the fermentation that another yeast under the same circumstances and conditions would complete in 72 or 96 hours. The bouquets of rums produced in each case will be quite different, provided the distillation is carried under identical conditions. The strain taking the longer period of time for completion of fermentation will almost invariably produce the more highly scented rum.

Lastly, the ability of different strains to produce and stand high degrees of alcoholic concentration in the fermenting medium will also be a measure of the intensity and purity of the resulting rum aroma. And this for two different reasons: (1) because with higher alcoholic concentration in the fermenting medium, the esterification reaction during, and after fermentation takes place more readily; (2) at the higher alcoholic concentration there exists less opportunity for infecting organisms to develop in the medium producing volatile substances of their own metabolism that might interfere with the nature and purity of the rum aroma upon distillation. Having thus discussed the set of factors influencing the aroma of rum which were classified as the natural factors, let us now discuss briefly those factors of the second set, or those depending on the technique of manufacture followed in the obtention of the finished product.

Modification and improvement of the aroma of the finished rum begins when the progressive distiller exercises a certain degree of

selection when acquiring his raw material. We have already explained that natural differences in the various raw materials used in rum making will be responsible for differences in the final bouquet of the finished product; and that even within the same raw material' as for instance, final sugar factory molasses, enough differences of chemical structure exist to influence the aroma of the resulting rums. Hence, careful selection on the part of the distiller will be very valuable in this respect. For instance, if found of good quality for his purposes, the distiller should keep using the same molasses from the same sugar factory all the time, instead of buying his raw material indiscriminately here and there; sometimes for the sake of a very small saving in first cost. Our advice is to keep a uniform and constant supply of the same raw material, even when the first cost may be a little higher. In this respect those sugar factories having a rum distillery as an adjunct, and using only their own molasses have a decided advantage. Not only have they a uniform supply of practically constant composition, but they also have the further advantages of being able to use freshly produced molasses; and of changing the quality of these molasses at will, if that should become necessary, by merely introducing modifications in the process of sugar manufacture.

A further modification of raw material may be exercises after its selection, through the process already described in our third chapter, or through the process already described in our third chapter, or through similar methods as therein described. The conditioning and purification of the raw material will affect in a very large degree the quality of the rum aroma. Many objectionable impurities in the material are eliminated at this stage of manufacture, that if left in the molasses, would greatly interfere with the quality of the aroma in the finished product. As this has been already explained in an earlier chapter, it is unnecessary to dwell longer on this matter.

The type of still, individual features within a given type, and technique employed during the distillation stage are necessarily factors of the utmost importance in the condition and improvement of the rum aroma. At this stage the intelligent rum distiller has the opportunity to correct those errors that may have occurred during the previous stages of rum manufacture, and that might prove detrimental to the final aroma of the finished product.

Continuous stills provided with a pasteurizing and purifying column will undoubtedly produce improvements in the aroma impossible to obtain when using the ordinary, common type of rum still. They also possess the advantage that lower proofs of distillation may be carried without impairing the quality of aroma and taste of final distillate. A certain degree of that selective action between desirable and undesirable constituents of the aroma of rum may also be exercised.

Discontinuous stills become the greatest asset a rum distillery may possess towards the production of the right aroma in the final distillate. The ease with which the rate of distillation may be modified at will, the great degree of fractionation that is easily accomplished, and the low proof of distillation that may be secured without damaging the quality of the raw rum, are qualities unique of this type of rum distilling apparatus. Another, and perhaps the greatest advantage as regards production of the right aroma, inherent to this type of still, consists in the greater amounts of the invaluable rum oil that is extracted from the fermented mash during its distillation.

With any type of still used, the predistillation treatment of the beers offer one of the greatest safeguards against undesirable odors that would interfere with the aromatic tone of the crude rum.

As to individual features in the design of either class of distilling outfits that influence the final aroma of the distillate, we may mention the use of as large as possible wine heaters; and long, narrow condensers for the condensation of the vapors forming the raw distillate.

As explained in the chapter on curing and maturing of the raw rum, during the curing period the aroma of rum undergoes its greatest and most fundamental modification. It is really at this stage that the beverage becomes a potable drink. The factors influencing the rum aroma during curing have been explained in the previous chapter; so, it would be unnecessary to repeat them here. We may add, however, that according to the degree of care, science, and art displayed in all and every feature affecting its curing, the raw rum becomes a great commercial success or an utter failure. We have seen many an excellent raw rum ruined during its curing; and also many mediocre ones that were greatly improved. It all depends on the degree of knowledge of the basic principles involved in the art of curing that is put into play during this final, and most important phase of rum manufacture.

The final aroma of the already matured rum has been formed and developed by two groups of factors:

(1) The aggregate of aromatic bodies existing originally in the raw rum.

(2) Modifications and transformations suffered by some of these bodies, and the formation of new aromatic constituents during the curing period.

In our opinion, the first group is fundamentally the most important, as the one exercising the greatest influence in the complex and compound aroma of the commercial rum. But the second group of factors is very important also for the enhancing effect it bestows on the original rum aromatic constituents, and also for its mellowing and refining influence on the whole product. We hold no doubts also that new aromatic bodies are produced within the liquid aging in the barrel, about some of which we know nothing, due to lack of suitable methods for their detection; as shall be presently explained.

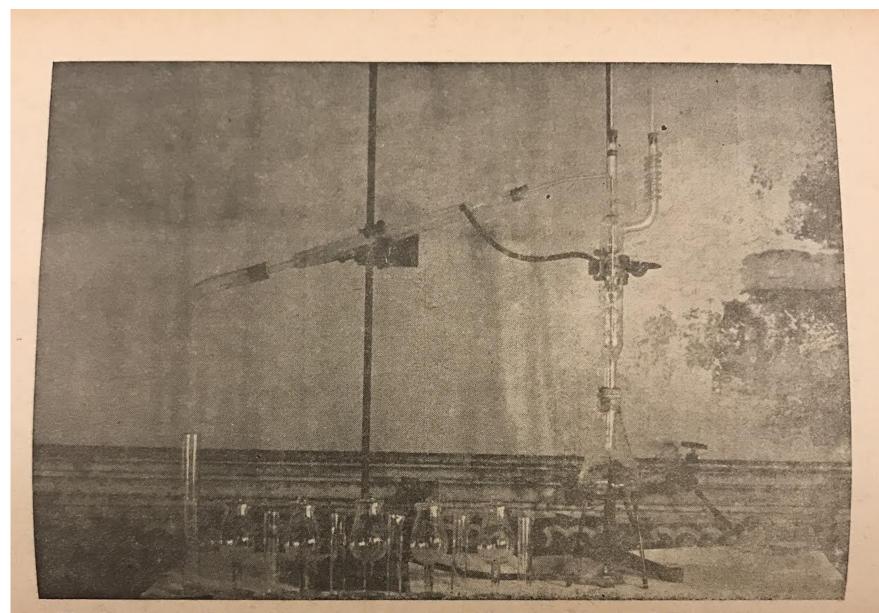
The aggregate of aromatic bodies forming the first group above-mentioned are much better known than those of the second, with the exceptions perhaps of rum oil, which is mainly known by research workers and rum investigators. This is due to the fact that raw distillates may be analyzed as such, without previous preparation or modification of the sample for analysis. The different aromatic compounds are, therefore, determined as *actually existing* in the sample. This is *not* the case when a sample of matured rum is to be analyzed. In this case the sample must be redistilled previous to its analysis, and the results as reported, represent conditions existing in the distillate obtained from the sample, rather than in the sample itself. How may we be certain that no decomposition or variations of the original chemical structure of the matured spirit have taken place during the heating and subsequent distillation of the original sample? In our opinion oxidation and degradation processes take place during this preparation of the sample for analysis, resulting in the destruction of aromatic bodies present in the original sample; and formation of other compounds not present originally as such in the sample. More illuminating methods for rum analysis, in general, are desirable; but in the case of matured rums they become a necessity. Right here there exists a wide field for study and research in the line of analytical chemistry. We lacked the knowledge, and ability, and tome, to undertake this subresearch within our main studies; but feel that by raising the question some one's curiosity and interest may be aroused who is better prepared than the writer to attack the problem. Of one thing we are absolutely certain, and this we have actually proved in the course of these studies; if a sample of matured rum is distilled with great care to avoid losses of any kind, as is usually done during the preparation of the sample for analysis, and once the distillation is finished the distillate is again incorporated to the residue left in the distilling flask, we would say that having lost nothing, we should again our original sample. But such is not the case, for a very pronounced and

remarkable difference in organoleptic characteristics will be found invariably between another undistilled sample of the rum in question and the one that underwent the distillation.

The aromatic bodies of the raw rum are formed during fermentation and distillation, or are already existing in the raw material. They consist especially of esters, aldehydes, free fatty acids, higher alcohols, and essential oils, including rum oil. The aromatic constituents in the matured spirit consist of all of those mentioned above in the case of the raw distillate plus those formed during the aging period; some of which are acetals and other condensation products. Also new compounds are formed by inter reaction among the constituents of the raw rum and those extracted from the staves of the aging barrel.

But the ordinary methods for rum analysis only teaches us the amounts of each aromatic group constituent found in the rum under observation, giving no information as to the kind of individual group constituents and the nature and quality of their contribution towards the formation of the bouquet. We must keep constantly in mind that some of the members composing the Non-Alcohol-Number, as for instance, fatty acids and high alcohols, are not specially noted for the fragrance or finesse of their respective aromas; and that even not all aldehydes and esters have acceptable aromas. Hence, two different rum samples may have almost the same chemical analysis (as performed) and yet be altogether different in organoleptic characteristics.

This proves that chemical analysis alone, as usually practiced, is almost entirely devoid of use and importance in the appraisal of the rum aroma, nor does it give any illumination as to what particular constituents of the Non-Alcohol-Number of rums are mainly responsible for the nature of their aromas. Had it not been for our fractional distillation work with the help of the Birectifier of Dr. Luckow, we would have never discovered the presence of the invaluable rum oil in Puerto Rican rums; nor found out the almost controlling influence this aromatic essential oil has on the composition of the rum aroma.



The Birectifier-An indispensable apparatus for rum research and quality appraisal of the finished product.



View of Hansen type of sterile transfer box in biological laboratory.

IX

Rum Composition, Differentiation and Appraisal. The Role of

The Birectifier in Rum Investigations

French scientists and investigators have given more of their time and attention to the first part of the subject matter of this chapter than those of any other nation. According to Guillaume (27) quite a number of brilliant French investigators have contributed in the past and present, to the study of rum composition, classification, differentiation and appraisal. Researches on the finished product far surpass those dealing with the subject of its manufacture in all French literature. Guillaume makes special mention of the names of Blarez, Simon, Bonis, Zizine, Rocques, Annotel, Sanarens, and Kervegant; proceeding to mention their most important contributions and to abstract the salient points of each.

We have thought it adequate as a sort of introduction to this chapter to refer to the most pertinent conclusions and opinions from these distinguished investigators, in a review of chapter IV of the work of Guillaume, already alluded to above.

Blarez defines the Non-Alcohol-Number. It is to be observed here that while the analytical practice in continental United States and Puerto Rico is to report the higher alcohol as amyl alcohol, the French custom is to report them as isobutyl alcohol. Aldehydes and esters are expressed respectively as acetaldehyde and ethyl acetate, same as the American custom. He considers the esters as the most important group constituent of the Non-Alcohol-Number. The importance of the advice of the experienced rum taster as complement of the chemist's analytical report is greatly emphasize when discriminating between genuine and adulterated samples.

Professor Simon tends to demonstrate that the classification of rums based upon their Non-Alcohol-Number offers the great inconvenience of granting too much importance to certain members of this Number whose roles in rum aroma and taste are quite contestable.

Bonis reaches the conclusions that: (1) It would be illusory to differentiate rums with the use of present analytical methods. (2) The Non-Alcohol-Number should amount to no less than 250 milligrams per 100 milliliters of absolute alcohol in a commercial rum.

Zizine, in his doctoral thesis on rum studies, comes to a conclusion that we consider of very great interest and far-reaching importance in the light of our own researches. This author concludes that from the analytical standpoint, the present methods of interpreting the analytical results furnished by the rums, are quite imperfect, since a given product possessing the required Non-Alcohol-Number *is not necessarily a rum*. The expert must depend a great deal on the organoleptic tests, exercising himself in the tasting of genuine products of excellence.

A. Bonis, Director of the Central Laboratory for the Repression of Fraud, arrives at the same conclusions of Professor Simon, that is, the classification of rums on the basis of their total Coefficient of Impurities, or Non-Alcohol-Number, is not rational. He prefers to adopt the ratio esters volatile acidity. To him it is the harmony existing among the various constituents, rather than the quantities of these constituents what counts.

Rocques explains that up to the present time rum appraisal and differentiation must be made attending to their organoleptic qualities and Non-Alcohol-Number.

Dr. Sanarens indicates the perils in the lowering of the Non-Alcohol-Number, that according to his idea, invites to fraud in rum making. According to him, there should be an average Non-Alcohol-Number of:

(1) 200 to 300 for those rums obtaining a ratio,

$$(2) \frac{Higher Alcohols}{Esters} < 1.0$$

(2) 250 to 500 for those rums obtaining a ratio,

$$(1) \frac{Higher Alcohols}{Esters} > 1.0$$

Both Kervegant and Bettinger deplore the lack of analytical methods which would facilitate the expert appraisal of rums.

It will be inferred from these reports that the question of rum classification and appraisal is a very intricate and difficult one. So is the determination of fraud in the composition of commercial rums.

Our studies upon this phase of rum making have not yielded a definite and conclusive method by which we could ascertain in all cases whether or not a given rum is a genuine or spurious product; neither have they given rise to the possible establishment of a system of grading the quality of rum, as has been done for instance with milk and other products. But they have at least added another feature in this important phase of the study of rum, with the introduction of the birectifier and fractional distillation into the appraisal of commercial rums, as well as into the general study of rum manufacture. The general contribution of the birectifier in rum studies is very great indeed, and it becomes especially important when entering the phase of rum differentiation and analysis, as well as of rum appraisal and fraud detection.

We shall, therefore, review the contribution of this simple, yet highly efficient piece of laboratory equipment, to our investigations in rum manufacture. It would be proper to start with a description of the apparatus with the aid of its photograph, shown in this chapter. As to the manner in which it is operated, this has been already described in our second chapter when treating on yeast selection.

The Birectifier is a small glass-made still consisting of a distilling flask and a very efficient still head for the work that is required of the apparatus. It will be noticed that the still head is so constructed that the alcoholic vapors rising from the boiling liquid in the flask must do so in the space between the inner and outer condensers; and must then pass through a set of air cooled coils before finally entering the still head outlet into the attached water condenser. As the hot vapors ascend the outer surface of the interior condenser, and the inner surface of the outer condenser, they begin to cause reflux of the higher boiling point constituents back into the distilling flask. When the rest of the vapors enter the glass coils shown in the picture, a still intenser condensation of the less volatile components takes place. These condensed products pass down through the inner condenser in the liquid form and are delivered back into the distilling flask through a glass siphon acting as a water seal to the inner condenser. The inner condenser is supposed to carry down the condensate from the glass coils back into the boiling liquid in the flask; but no vapors should ever pass up through this inner condenser. The presence of the liquid seal at the siphon forming the lower end of the inner condenser helps to so regulate the pressure at the distilling flask that no vapors will pass up through this inner condenser. In this manner a very efficient separation of the constituents forming the Non-Alcohol-Number may be effected during the fractionation of a given sample. For further details in the manipulation and "modus operandi" of this apparatus, the reader is referred to chapter two of this bulletin.

The very important role played by the Birectifier in the work on yeast selection has been thoroughly discussed, so we need not repeat it here. But the Birectifier is very useful in many other ways. For instance, during the control of the process of curing; and for the purpose of rum classification and appraisal according to its chemical composition.

During the curing of our experimental rums we had occasion to effect fractional distillations of different rums at different periods of aging. Had it been possible, we would have effected a fractional distillation for each one of our experimental rums at intervals of not over six months; but this was not altogether feasible with the available technical help. So it was necessary to pick certain samples for more intensive testing along this line and perform the fractional distillation test on all samples after two years of aging, and prior to their bottling. In some cases, however, one year and three years old rums were also fractionated. But all rums were fractionated previous to their bottling after two years of aging. Two years was selected as the bottling age for most of the rums on account that by this time of curing they showed maturing qualities, except in the case of sugar cane juice rums, that as already stated, took a longer time.

The birectifier becomes again useful in the classification and appraisal of commercial or experimental rums; and in the case of the former ones it also becomes a valuable and convenient tool in the detection of artificially added aromatic and flavoring substances. We feel certain that if ever governmental regulation and inspection of rum purity and quality is established by law in Puerto Rico, the birectifier will prove an invaluable asset in the successful operation of whatever measures are enacted to protect genuine, unpolluted products, from the artificially concocted ones; in this way also protecting the public health.

As in the case of the French investigators, whose conclusions have been outlined in previous pages, our own work has demonstrated that there does not exist as yet a purely chemical method for rum classification or appraisal. To this date, the opinion of the expert rum taster must enter as perhaps the most important factor in the work of differentiation and appraisal.

To begin with, the routinary rum analysis helps but little by itself, but becomes more interesting and illuminating if considered only as one factor (and this the least important), in the process of classification and appraisal. Standing alone, as the source of evidence from which origin or quality of a given rum is to be judged, then the ordinary chemical analysis is practically almost useless for as already stated, two rums of identical chemical analysis (as shown from this ordinary, routine analysis) may be really as different in quality and general potable properties as night is from day. The average reader may rightly ask however – but why? Why is it that two rums with identical chemical composition may be altogether different in taste and aroma? But the initiated will soon realize that this supposed identity may b e really an illusion; the two rums actually being of quite different chemical constitutions; except for the respective amounts of ethyl alcohol, perhaps, but even this may not be actually identical.

It all comes from the conventional method of reporting the results of a rum analysis. Omitting for the present the content of ethyl alcohol in the rum sample from our discussion, we have that the members of the Non-Alcohol-Number, which really are the ones that characterize and differentiate one rum from another, are reported in groups under a single chemical constitution; for instance all the aldehydes present in the sample are classified as acetaldehyde, all esters as ethyl acetate, all acids as acetic acid and all higher alcohols as either isobutyl or as amyl alcohol.

But it is known that at least six different aldehydes may be present in a rum, and that the chemical structure, and aromatic characteristics of each one differs from the others, and that besides, their degrees of volatility and indexes of persistence are also quite different. We have found, for instance, that the index of aromatic persistence of acetaldehyde is only 1:10,000; while that of hexaldehyde is 1:200,000 and that of heptaldehyde 1:500,000.

Similarly, it is known that a great variety of esters are found in different rums, among which are some of very valuable aromas and others of indifferent, or even repellent ones. Here also the individual indexes of aromatic persistence are very variable. For instance, we have found that while the index for ethyl acetate is only 1:20,000, that for the propionate is 1:50,000; and those for ethyl butyrate and caproate, respectively, are 1:200,000 and 1:500,000.

What has been stated in the case of aldehydes and esters, also applies in the cases of the other members of the Non-Alcohol-Number. As to rum oil, this most valuable constituent in the natural aroma of Puerto Rican rums, the ordinary analysis offers no means for its detection.

The ordinary analysis by itself cannot tell much about the probable state of maturity in the rum, nor of the probable time it has been aging. It will not help either to establish definite differences between rums made from different raw materials as for instance molasses and sugar cane juice. Nor does it give a definite means of pronouncing a certain rum as adulterated; or whether it was produced through natural or artificial curing. What the ordinary analysis really indicates is the ascendancy of a certain group constituent upon another. That is, we may say that it contains more aldehydes than esters or vice versa, or high or low acidity, or fusel oil content. The determination of proof before and after the sample is distilled, and the examination and analysis of its extract may render more information about the nature of the rum under study than the group determination of its Non-Alcohol-Number constituents.

When to this chemical analysis is added the information obtained from a consideration of the organoleptic qualities of the rum in question, after examination by an expert rum taster, then our knowledge of the possible rum quality characteristics and history, becomes somewhat enlarged. And when these two methods of attack are complemented by fraction distillation work with the help of the birectifier, then our horizon greatly widens and it becomes a simpler task to differentiate one rum from another or to appraise the individual characteristics of quality. This is the method we have followed in the appraisal and differentiation of our own experimental rums, and some imported and domestic commercial brands.

While it is true that the fractionation work could have been improved by effecting a more thorough chemical analysis tending to classify each of the individual aromatic constituents found in a given fraction; our method of procedure proved quite successful in the accomplishment of our purpose. The individual determination of group constituents in each fraction meant such an enormous amount of work that even with twice the available technical staff we doubt if it could have been accomplished. We admit that in this respect as in the work of curing and maturing there is much more to do; and that what has been accomplished has only served to prepare us for better and more exhaustive work along these lines of in a future time.

Coming then to the method developed by us, we have that the following course was followed:

(1) The usual chemical analysis including:
Sp. Gr. at 20/4 degrees Centigrade
Direct Proof
Proof after distillation of the sample
Alcohol by volume in distilled sample
Total acidity (in mgs. per 100 ml Ab. Alc.)

Fixed acidity (in mgs. per 100 ml Ab. Alc.) Volatile Acidity (in mgs. per 100 ml Ab. Alc.) Esters (in mgs. per 100 ml Ab. Alc.) Aldehydes (in mgs. per 100 ml Ab. Alc.) Higher Alcohols (in mgs. per 100 ml Ab. Alc.) Extract, per 100 ml rum sample Ash, per 100 ml rum sample

(2) Physical and Organoleptic Tests including:

Aroma Taste Body Index of Persistence Sulfuric Acid Test

(3) Fractional Distillation Using the Birectifier.

In this test the sample was first diluted to 40.0 per cent alcoholic strength by volume, and then fractionated into 8 fractions, that were consequently tested organoleptically and chemically. For details of technique followed during the fractionation of the sample and subsequent work done on each fraction the reader is referred to the latter part of the second chapter, under yeast selection.

(4) Valuable Ratios.

From the data thus accumulated certain ratios were formed, indicative of either quality or nature of the rum being tested. Among these ratios, the most important are:

Esters/Higher Alcohols Esters/Aldehydes Esters/Volatile Acidity High B.P. esters/Low B. P. esters High B. P. Aldehydes/Low B. P. aldehydes Higher Alcohols/Total Non-Alcohol-Number

(5) Curves were drawn using the results of the fractional distillation which formed a graphical representation of the chemical structure of each rum. An alcohol, an aldehyde, a volatile acidity, an ester, and a higher alcohols curves were drawn.

From the data offered by the usual chemical analysis, we construct the ratios: esters:volatile acidity; esters:higher alcohols; esters:aldehydes; build up the total Non-Alcohol-Number, and construct the ratio of higher alcohols to this Non-Alcohol-Number. The determinations of extract and ash also help a great deal in judging the probably curing time the rum has received or whether extraneous substances have been incorporated to the rum in question for purposes of adding flavor and aroma. These two determinations are of great value in the appraisal of a given rum sample if other features brought in by the other tests are also taken into consideration. When the kind of, and treatment given to the barrel in which the rum has been aged are known, then the determinations of extract and ash are a good guide towards judging the age of the given sample.

The physical and organoleptic tests throw invaluable light upon the origin of, and degree of maturity of a given sample, when performed by an expert on the matter. The determination of body is of special help in judging whether the sample is a molasses or a cane juice rum, and also in determining if the distillation was carried in continuous or discontinuous type of still. Molasses rums, pot distilled, have the highest body values, then in succeeding lower values stand the continuously distilled molasses rum, the pot distilled cane juice rum, and the continuously distilled cane juice rum. The index of persistence for aroma and taste tells much about the sample under consideration. It will give an idea upon the class of rum; its probable chemical structure regarding the class of esters and other aromatic compounds of the Non-Alcohol-Number, and will especially indicate the degree or extent in which rum oil will be found present during fractionation. It will also help in distinguishing between molasses and cane juice rums; and in the class of cane juice rums, whether raw juice or clarified juice was used as the raw material. This test will also give further evidence on the genuine or artificial production of the sample under consideration. Whether produced from cane juice or from molasses, this test will help in differentiating "heavy: from "light rums. It is sometimes very helpful to effect the index of persistence test in the rum sample as such, and also after it has gone through the sulphuric acid test. Fraudulent samples that may obtain a satisfactory index of persistence before the sulphuric acid treatment will usually utterly fail during the second test.

The fractional distillation test serves as a complement to both the organoleptic and the regular chemical analysis, extending their respective fields of information and throwing added light in the problem of rum differentiation and appraisal. The components of the Non-Alcohol-Number are by these means separated into either different fractions, and every fraction receives essentially different classes of aromatic compounds. That this is the case is proved both by individual organoleptic

tests on each fraction, and by carrying also individual organoleptic tests on each fraction, and by carrying also the ordinary chemical analysis for every separation fraction. It is found that fractional separation of the aromatic constituents takes place in the following order: The most volatile aldehydes and esters together with a small amount of the lower higher alcohols are found in the first fraction. The second and third fractions are composed mainly of pure ethyl alcohol with varying, but always very small amounts of higher alcohols, esters and aldehydes. The fourth fraction retains most of the higher alcohols, up to 70 per cent of the total amount present in the sample, sometimes; but never less than 60 to 65 per cent of the total. We might call this, the higher alcohol fraction. Some of the less volatile aldehydes and esters also begin to pass over with this fraction; and if the given sample of rum is rich in rum oil, this constituent will also be found in this fraction. This might be also called the transition fraction where the last bodies of higher volatility and the first bodies of lesser volatility are found. The fifth fraction contains most of the high molecular weight, high boiling point esters and aldehydes, mixed with the greater part of the rum oil (when this is present in the sample), and a very small amount of the higher in the series, superior alcohols. The free acidity begins to rise sharply at this fraction, especially when dealing with molasses rums, and its presence remains in quite appreciable amounts in all the succeeding fractions. This fifth fraction could well be designated as the higher esters, and rum oil fraction.

In fractions six to eight inclusive, there are found small amounts of very high boiling point esters and aldehydes, together with some fusel oil and liberal amounts of fatty acids. Also, when the rum under fractionation is rich in rum oil, this will show its presence in these fractions.

We have given above the description of what happens when a genuine molasses rum is fractionated using the birectifier. Great variations from this stand will be met in the practice of rum fractionation, especially when dealing with rums of unknown manufacture and curing. Artificially concocted ones, will act quite differently during fractionation, as will be shown later on in this chapter. Cane juice rums will not show the characteristic rise in esters during their fractionation for those fractions from fifth to eighth inclusive, in the same degree as heavy molasses rums. An idea on the degree of maturity reached by a given sample may be obtained by the organoleptic test of the first fraction. The less matured spirits will give rise to pungency of odor and irritating taste in the liquid collected. As the product gains in maturity both the aroma and flavor of this first fraction becomes less sharp, till in fully matured samples veritable suavity of taste and delicacy of aroma are sometimes obtained.

If the first fraction thus becomes an index of the state of maturity reached by the rum under examination, the fifth fraction likewise becomes the main guide in ascertaining the distinctive quality of the taste and aroma possessed by the rum sample. This is easily proved organoleptically by performing the following simple experiment. If after fractionation is finished, all the different fractions with the exception of the fifth are again mixed together and the mixture incorporated to the residue left in the distilling flask, there will be found a remarkable disparity and strangeness in taste and aroma between the reconstructed and the original rum sample. In fact the two liquors would seem totally unrelated. But when the fifth fraction is then incorporated to the mixture, the new taste and aroma which is developed will be found to be similar enough to those of the original sample as to plainly show the relationship existing between them. Moreover, any combination of the different fractions into which will enter the fifth, even when one or two other fractions be omitted, will still produce taste and aroma similar to those of the original sample. It is mainly the happy combination of the highly aromatic constituents of this fifth fraction what bestows the distinctive characteristics of aroma and taste to truly genuine molasses rums.

In order to find the relative contribution of the different aromatic bodies found in each of the eight fractions towards the index of aromatic persistence of the original rum sample, we determined in a few cases the individual indexes of persistence of each fraction. In order to have the same alcoholic strength in each fraction before proceeding to the determination of the different indexes, fractions one to four were diluted with water, and fractions five to eight inclusive were strengthened by addition of neutral spirits so that all of the fractions contained 40 per cent alcohol by volume before their respective indexes of aromatic persistence were determined. The average results obtained in the case of six trials with molasses rums are given below:

Fraction	Index of Aromatic
Number	Persistence
1	1: 100,000
2	1: 7,500
3	1: 10,000
4	1: 100,000
5	1: 500,000
6	1: 150,000
7	1: 50,000
8	1: 10,000

The index of aromatic persistence found in an average composite sample of the six rum samples was: 1:125,000.

The fractional distillation test is valuable again in another direction: When extraneous aromatic flavoring substance have been added to the rum, such as oil of bitter almonds, vanilla, oil of cloves, cassia oil, etc. etc., these will usually pass in a concentrated form in one or two of the eight fractions, or remain in the residual liquid left in the distilling flask. The concentrated condition in which the added substance is obtained, simplifies its detection through purely organoleptic inspection.

Let us now discuss some of the work on appraisal and differentiation of rums.

Table 31 presents the data obtained in the examination of a genuine, two years old, heavy type of molasses rum, produced during our experimental work. Fig. 15 offers a representation of the results of fractionation.

The results in table 31 will be considered in relation to those of similar nature presented in table 32, which shows the figures and descriptive data obtained in the examination of an eight years old, heavy type rum guaranteed by the government of Jamaica. Fig. 16 represents the results of the fractional distillation.

A study of the results shown in table 31 and 32 respectively, will offer points illuminating some of our assertions in previous pages. We have often repeated that the state of maturity in a rum is not always a direct consequence of the number of years it has been aging; and that a younger rum may show less, the same, or more signs of maturity than an older one. Well, a study of these two rums forms a splendid illustration of our assertion. It is well to keep in mind that this is really an extreme case, since we are comparing a two years old with an eight years old product.

If we look into the results obtained from the organoleptic and physical tests, we hardly find any differences, except a very slight on in Body Number; and the fact that suavity of aroma is greater in the case of the eight years old. The greater suavity of aroma is really the only factor favoring the longer aged rum.

TABLE 31

SHOWING DATA OBTAINED IN THE EXAMINATION OF A TWO-YEARS-OLD HEAVY TYPE OF MOLASSES RUM OBTAINED DURING OUR WORK ON THE JAMAICA TYPE

- ORGANOLEPTIC AND PHYSICAL TESTS: Aroma: Excellent, conforms in tone and quality to the original Jamaican; rich and powerful. Taste: Very good, very similar to original Jamaican. Body: No. 13; characteristic of heavy rums. Index of Persistence: 1:200,000. Sulphuric Acid Test: Positive; strong residual odor.
- (2) CHEMICAL ANALYSIS: Specific Gravity at 20/4 deg. C. Direct Proof, Per cent Proof after distillation, Per cent Alcohol by Volume Per Cent (in distillate) Total Acidity (mgs in 100mL Abs Alc) Fixed Acidity (mgs in 100mL Abs Alc) Volatile Acidity (mgs in 100mL Abs Alc) Esters (mgs in 100mL Abs Alc) Aldehydes (mgs in 100mL Abs Alc) Higher Alcohols (mgs in 100mL Abs Alc) Extract (mgs in 100mL rum) Ash (mgs in 100mL rum) Non-Alcohol-Number

(3) FRACTIONAL DISTILLATION RESULTS:

 0		а. п		CHEMICAL ANALYSIS					
Fraction No		Appearance of Fraction	Alcohol by Volume	Volatile Acidity	Esters	Aldehydes	Higher Alcohols	Remarks	
			Per Cent						
1	78-78	Clear	93.32	1.76	605.44	194.48	10.87		
2	78-78	Clear	95.12	0.86	176.00	40.04	5.93		
3	78-78	Clear	95.80	0.86	140.80	34.32	6.93		
4	78-83	Clear	90.76	0.86	183.04	34.32	85.03	Cloudy on dilution	
5	83-98	Cloudy	44.76	65.74	246.40	80.08	7.91	Oil Drops	
6	99-99	Clear	2.64	83.67	176.00	57.20	2.96	Oil Drops	
7	99-99	Clear	1.32	79.41	183.04	74.36	2.96	Oil Drops	
8	99-99	Clear	1.32	82.39	140.80	45.76	2.96	Oil Drops	

NOTE: Members of Non-Alcohol-Number reported in mgs/100mL of fraction.

(4)	VALUABLE RATIOS:	
	Esters: Higher Alcohols	3,11:1,00
	Esters: Aldehydes	2,61 : 1,00
	Esters: Volatile Acidity	107 : 1,00
	High BP : Low BP Esters	101 : 1,00
	High BP : Low BP Aldehydes	109 : 1,00
	Higher Alcohols: Non-AlcNumber	0.10 : 1,00

0.93410

95.96

96.64

48.32

360.00

132.53

227.47

244.10

93.70

78.50

329.60

776.30

8.00

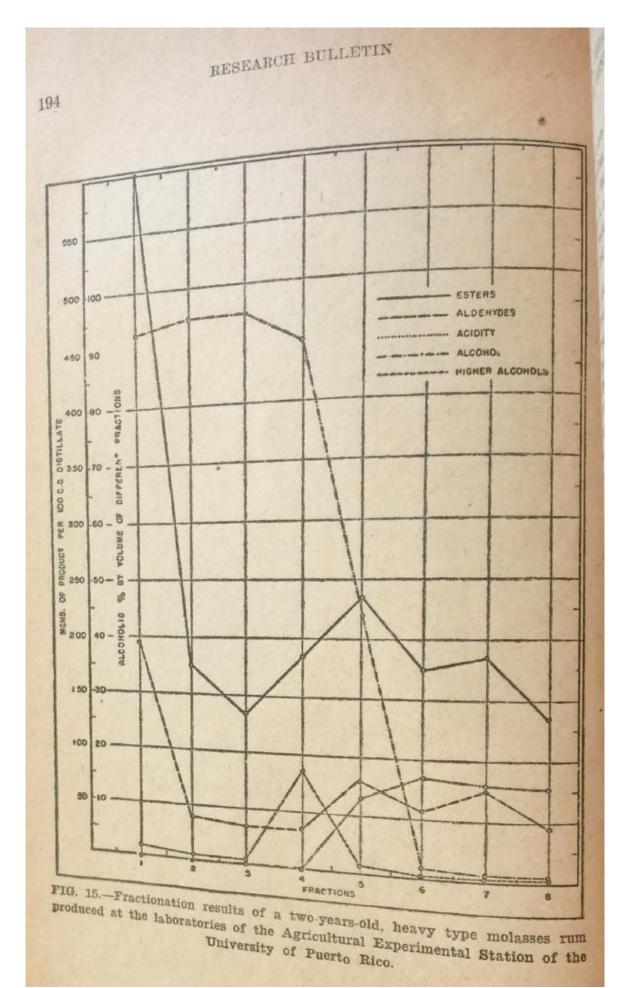


TABLE 32

SHOWING DATA OBTAINED IN THE EXAMINATION OF AN EIGHT-YEARS-OLD, GENUINE JAMAICAN RUM, IMPORTED FROM JAMAICA

- ORGANOLEPTIC AND PHYSICAL TESTS: Aroma: Excellent, very suave. Taste: Good, characteristic of its class. Body: No. 14; also characteristic of the type. Index of Persistence: 1:200,000. Sulphuric Acid Test: Positive; strong residual odor.
- (2) CHEMICAL ANALYSIS: Specific Gravity at 20/4 deg. C. Direct Proof, Per cent Proof after distillation, Per cent Alcohol by Volume Per Cent (in distillate) Total Acidity (mgs in 100mL Abs Alc) Fixed Acidity (mgs in 100mL Abs Alc) Volatile Acidity (mgs in 100mL Abs Alc) Esters (mgs in 100mL Abs Alc) Aldehydes (mgs in 100mL Abs Alc) Higher Alcohols (mgs in 100mL Abs Alc) Extract (mgs in 100mL rum) Ash (mgs in 100mL rum) Non-Alcohol-Number

(3) FRACTIONAL DISTILLATION RESULTS:

	Fraction No. Temperature Range of Distillation Deg. C.		CHEMICAL ANALYSIS					
Fraction N		Appearance of C. C. Distillatio of G. C. Distillatio of G. C. Distillatio of G. C. Distriction	Alcohol by Volume	Volatile Acidity	Esters	Aldehydes	Higher Alcohols	Remarks
			Per Cent					
1	78-78	Clear	93.68	1.28	439.50	80.08	11.49	
2	78-78	Clear	92.24	1.28	96.45	14.30	6.26	
3	78-78	Clear	92.24	1.49	11.97	22.88	7.32	
4	78-80	Clear	91.12	2.13	23.94	17.16	89.85	Turbid on dilution
5	80-99	Cloudy	29.40	18.79	344.96	40.04	8.36	Oil Drops
6	99-99	Clear	1.04	25.62	87.30	22.83	3.13	Oil Drops
7	99-99	Clear	0.00	28.18	11.97	17.16	3.13	Oil Drops
8	99-99	Clear	0.00	35.87	11.97	17.16	3.13	Oil Drops

NOTE: Members of Non-Alcohol-Number reported in mgs/100mL of fraction.

(4)	VALUABLE RATIOS:	
	Esters: Higher Alcohols	2,39:1,00
	Esters: Aldehydes	3,56 : 1,00
	Esters: Volatile Acidity	4,81 : 1,00
	High BP : Low BP Esters	0.80:1,00
	High BP : Low BP Aldehydes	0.98:1,00
	Higher Alcohols: Non-AlcNumber	0.13 : 1,00

0.93243

97.66

97.88

48.94

264.38

141.01

123.37

198.20

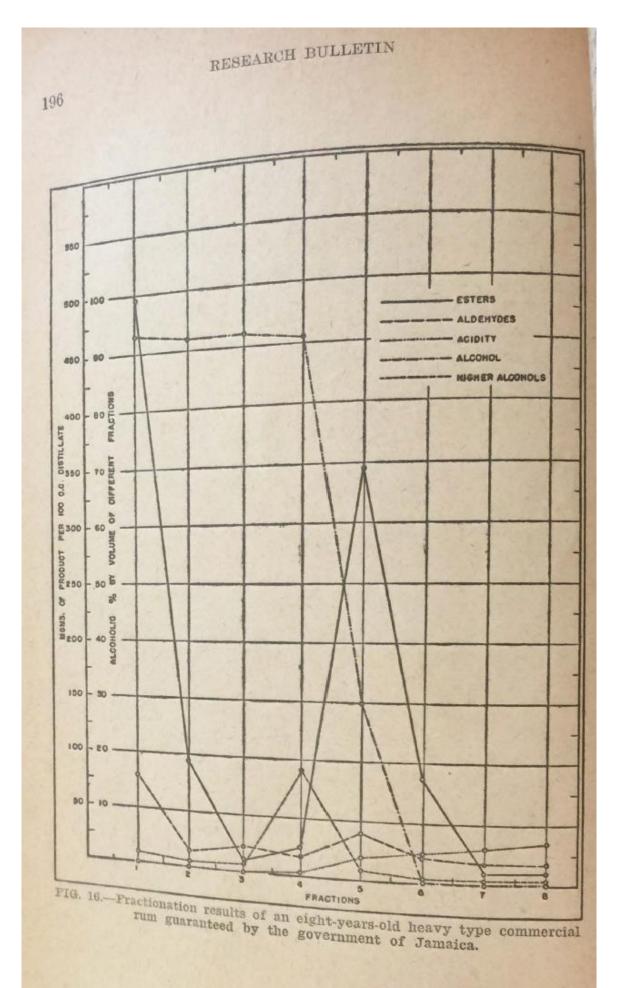
55.70

82.94

20.20

601.22

1,390.10



The ordinary chemical analysis results, begin to explain the greater suavity of aroma in the case of the older rum from a consideration of the respective volatile acidities, and aldehydic contents. It will be noticed that the volatile acidity of the younger rum is almost twice as great as that of the older rum; and a similar situation is observed in the case of the respective aldehydic contents. The observation of these relations, as stated before, begins to explain the cause for the greater suavity of aroma in the case of the older rum. We know that the volatile organic acids found in rums possess no properties of odor that would in any way enhance the aroma of a given rum, although they may more readily influence its taste. Hence, to less volatile acidity we may expect greater suavity of aroma. In the case of the aldehydes the same result would be expected, excepting when only high boiling point aldehydes are at issue.

When considering the results of the fractional distillation and of the analytical work performed with each fraction we may observe that:

(1) The first fraction of the older rum shows less than one half the amount of very volatile, irritating odor producing aldehydes, of those present in the corresponding fraction of the younger product. This again throws light upon the fact of greater suavity of odor encountered in the organoleptic tests in the case of the older rum.

(2) The volatile acidity, which really begins to pass over in appreciable quantities from the fifth fraction on, is much greater in the case of the younger rum in every fraction after the fifth and in the fifth inclusive.

(3) The general distribution of esters and aldehydes having high boiling points favors the chemical structure of the younger rum.

It will be noticed that appreciable or even high quantities of valuable esters and aldehydes may be found well distributed throughout all the fractions from the fourth to the eighth inclusive, in the case of this rum; while in the case of the older sample, those fractions below the fifth, and also the fourth, are almost destitute of these most valuable aromatic products. There are also less higher alcohols present in every respective fraction in the case of the younger product.

The inference is reached that in the original raw state, the younger rum was a superior product. Its general chemical structure was superior and more varied and abundant in those aromatic bodies that count so much during the process of curing. This is proven by the fact that in one fourth the curing time it has become a strong competitor of the much older rum for quality. If we then look into the valuable ratios we find that the older rum shows superiority in the ratios of esters aldehydes and esters volatile acidity; but inferiority in all the rest of these ratios.

Hence, in a final judgment we would decide that the only great advantage of the older over the younger rum is that of greater suavity of aroma; for as to the taste, the younger rum was much to be preferred. The richness and tone of the aroma was also superior in the younger sample. The two rums would be declared as of equal ranking in classification and appraisal, since the delicious, delicate suavity of aroma in the case of the older rum counterbalances the advantages of the younger product.

Another point of interest derived from the study of these two rums is that rums acquire maturity and goodness during curing not only through gaining in new aromatic compounds; but also through the losing out of some already possessed by the raw spirit. Very volatile aldehydes and esters are among those bodies which are lost out through the pores of the barrel during the curing period, together with very volatile organic free acids. The fixed acidity in genuine rums offer a higher value the longer the time of curing and also according to the kind and type of barrel used. The fixed acidity is all originated from the wood of which the barrel is made (when dealing with genuine rums). It increases at first and then remains practically constant; but its ratio to volatile acidity is very variable at any given time. In newly distilled spirits this ratio becomes almost zero, and in very old rums the ratio narrows a great deal, even when the fixed acidity reaches an almost constant value, due to the continued loss of volatile acidity through the pores of the curing barrel. The volatile acidity increases rapidly in value for the first two or three years of aging, and then begins to decrease, due to the fact that more than what is produced within the barrel is lost.

Table 33 offers the study of a molasses rum classified as "medium heavy" type; and table 34 the study of another molasses rum classified as of the "light type". Both are 2 years old rums. Figs. 17 and 18 represent the results of the respective fractional distillation tests.

If these results are compared with those obtained in the study of the rums classified as heavy, some interesting differences will be observed. The body number value becomes less as we pass from the heavy to the light type, giving the ratios 13:11:9, respectively. Hence, body number value serves to differentiate among heavy, medium heavy and light rums. The same results are obtained with the indexes of persistence, which are highest for the heavy and lowest for light types.

The ratios of esters to higher alcohols, esters to aldehydes, and high to low B. P. esters are also higher in the case of the heavy rums.

If we look into the fractional distillation results in each case, we find that the spectacular rise of the ester value in fraction five, is not so apparent in the rums classified as medium heavy, and very especially in those classified as light. The light type is also characterized by a higher ratio of higher alcohols to Non-Alcohol-Number. The Non-Alcohol-Number itself shows a decreasing value from the heavy towards the lighter rums. It will be noticed that rum oil is present in all fractions below the fourth in the case of the heavy type, while its presence is observed in the fifth and sixth fractions of the medium heavy and only in the fifth fraction of the light type. This, and the fact that there are less amounts of high boiling point esters in the lighter types of rums, account for the decreasing values observed in their respective indexes of persistence.

TABLE 33

SHOWING THE STUDY OF A TWO-YEARS-OLD MOLASSES RUM CLASSIFIED AS MEDIUM HEAVY TYPE

(1) ORGANOLEPTIC AND PHYSICAL TESTS:

Aroma: Very Good. Taste: Good. Body: No. 11 Index of Persistence: 1:125,000. Sulphuric Acid Test: Positive; strong residual odor.

(2) CHEMICAL ANALYSIS:

Specific Gravity at 20/4 deg. C.	0.93946
Direct Proof, Per cent	90.12
Proof after distillation, Per cent	90.86
Alcohol by Volume Per Cent (in distillate)	45.43
Total Acidity (mgs in 100mL Abs Alc)	198.80
Fixed Acidity (mgs in 100mL Abs Alc)	48.90
Volatile Acidity (mgs in 100mL Abs Alc)	149.90
Esters (mgs in 100mL Abs Alc)	183.10
Aldehydes (mgs in 100mL Abs Alc)	105.40
Higher Alcohols (mgs in 100mL Abs Alc)	82.68
Extract (mgs in 100mL rum)	202.30
Ash (mgs in 100mL rum)	12.70
Non-Alcohol-Number	569.98

(3) FRACTIONAL DISTILLATION RESULTS:

0.	n re		CHEMICAL ANALYSIS					
Fraction No.	Temperature Range of Distillation Deg. C.	Appearance of Fraction	Alcohol by Volume	Volatile Acidity	Esters	Aldehydes	Higher Alcohols	Remarks
			Per Cent					
1	78-78	Clear	97.24	1.71	386.50	171.60	11.46	
2	78-78	Clear	96.52	1.71	36.60	48.62	6.24	
3	78-78	Clear	95.52	1.28	12.60	48.62	7.30	
4	78-84	Clear	94.76	1.28	23.94	62.94	89.58	
5	84-98	Cloudy	27.84	62.98	135.35	102.96	8.33	Oil Drops
6	98-99	Clear	1.84	60.21	36.00	68.64	3.12	Oil Drops
7	99-99	Clear	2.12	55.51	12.60	74.36	3.12	1
8	99-99	Clear	2.12	54.23	23.94	74.36	3.12	

NOTE: Members of Non-Alcohol-Number reported in mgs/100mL of fraction.

(4)	VALUABLE RATIOS:	
	Esters: Higher Alcohols	2,21:1,00
	Esters: Aldehydes	1,74 : 1,00
	Esters: Volatile Acidity	1,22 : 1,00
	High BP : Low BP Esters	0.53 : 1,00
	High BP : Low BP Aldehydes	1,43 : 1,00
	Higher Alcohols: Non-AlcNumber	0.15 : 1,00

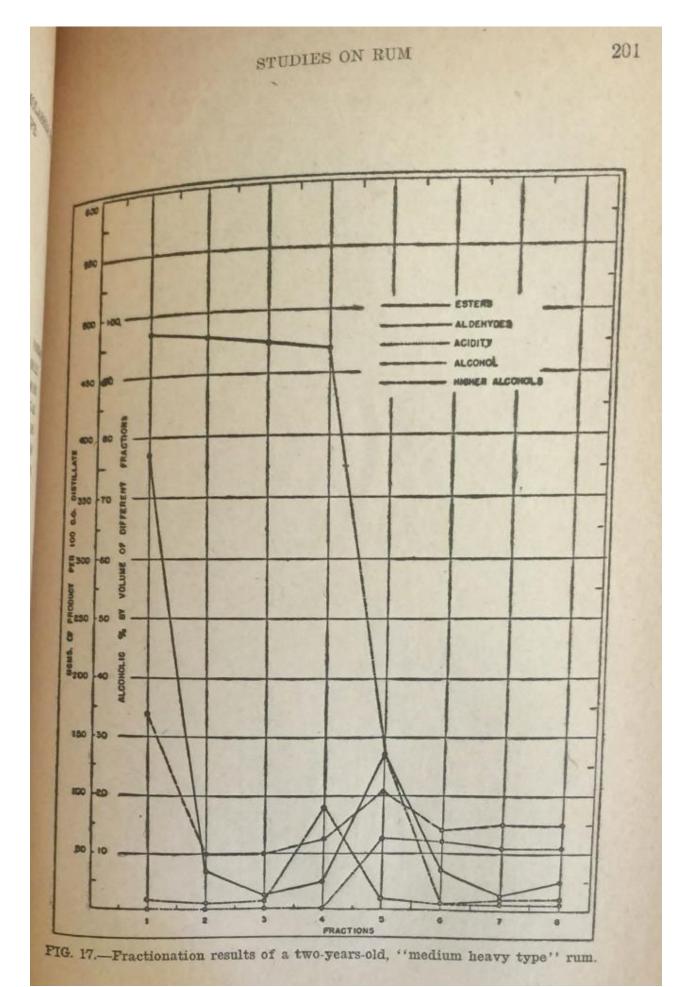


TABLE 34

SHOWING THE STUDY OF A TWO-YEARS-OLD MOASSES RUM CLASSIFIED AS OF THE LIGHT TYPE

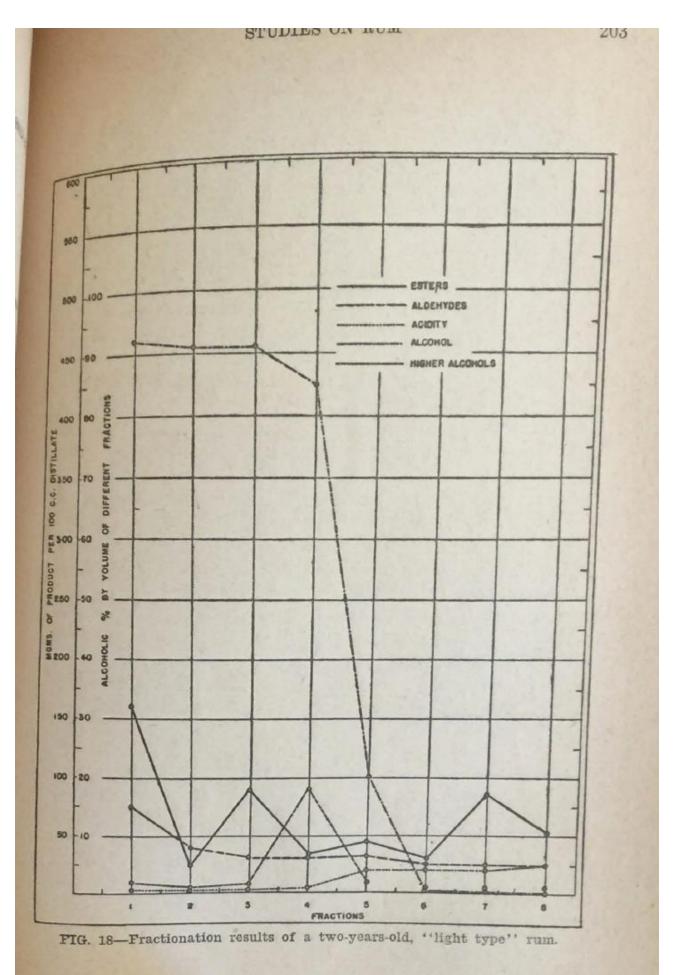
- ORGANOLEPTIC AND PHYSICAL TESTS: Aroma: Good. Taste: Very Good Body: No. 9 Index of Persistence: 1:50,000. Sulphuric Acid Test: Positive; with development of a poor extraneous odor.
- (2)CHEMICAL ANALYSIS: Specific Gravity at 20/4 deg. C. 0.92893 Direct Proof, Per cent 101.26 Proof after distillation, Per cent 101.26 Alcohol by Volume Per Cent (in distillate) 50.63 Total Acidity (mgs in 100mL Abs Alc) 54.74 Fixed Acidity (mgs in 100mL Abs Alc) 10.94 Volatile Acidity (mgs in 100mL Abs Alc) 43.80 Esters (mgs in 100mL Abs Alc) 76.47 Aldehydes (mgs in 100mL Abs Alc) 38.12 Higher Alcohols (mgs in 100mL Abs Alc) 83.27 Extract (mgs in 100mL rum) 529.20 Ash (mgs in 100mL rum) 12.40 Non-Alcohol-Number 252.60

(3) FRACTIONAL DISTILLATION RESULTS:

0.	n re		CHEMICAL ANALYSIS					
Fraction N	Fraction No. Temperature Range of Distillation Deg. C.	Appearance of	Alcohol by Volume	Volatile Acidity	Esters	Aldehydes	Higher Alcohols	Remarks
			Per Cent					
1	78-78	Clear	92.24	2.08	161.43	73.40	11.54	
2	78-78	Clear	91.84	2.38	26.88	40.78	6.29	
3	78-78	Clear	91.84	2.38	90.61	32.62	7.35	
4	78-83	Clear	83.80	6.54	35.34	32.62	90.22	
5	83-99	Cloudy	21.44	21.40	45.69	32.62	8.39	Oil Drops
6	99-99	Clear	0.52	19.02	28.44	24.47	3.14	1
7	99-99	Clear	0.00	19.62	83.71	24.47	3.14	
8	99-99	Clear	0.00	24.96	52.59	24.47	3.14	

NOTE: Members of Non-Alcohol-Number reported in mgs/100mL of fraction.

(4)	VALUABLE RATIOS:	
	Esters: Higher Alcohols	0,92 : 1,00
	Esters: Aldehydes	2,01 : 1,00
	Esters: Volatile Acidity	1,75 : 1,00
	High BP : Low BP Esters	0.88 : 1,00
	High BP : Low BP Aldehydes	0.94 : 1,00
	Higher Alcohols: Non-AlcNumber	0.33 : 1,00



Tables 35 and 36 respectively, show the studies of two years old defecated and clarified sugar cane juice rums, the first classified as light and the second as medium heavy. Figs. 19 and 20 respectively, show the results of the fractionations.

Comparing the studies of these rums with those produced from molasses, differences are at once apparent. The body numbers and indexes of persistence are much lower for the sugar cane juice rums when compared with their corresponding classification in the molasses class. The ester values are also lower for the juice rums, especially when in the respective results of the fractional distillation work the high boiling point class of esters are compared. In the samples under discussion the differences in the valuable ratios of both kinds of rums are quite apparent. In the molasses rums, even in the lightest type, the ratios Esters/Higher Alcohols; Esters/Aldehydes, Esters/Volatile Acidity are either over unity or very close to unity; while in the sugar cane juice class these ratios are generally under unity. This is a very decisive and striking differentiation between the two classes of rums. The difference is much more striking, however, when the respective ratios of high boiling point to low boiling point esters are considered.

If reference is made to the graphical representations of the results of fractionation in each class of rums, the differences among the respective curves makes easily distinguishable a graph of a molasses rum from one pertaining to a cane juice rum.

	TYPE OF RUM							
Valuable Ratios		Molasses	5	Sugar Cane Juice				
	Raw	One Year	Two Years	Raw	One Year	Two Years		
Esters: Higher Alcohols Esters: Aldehydes Esters: Volatile Acidity Higher Alcohols: Non-Alcohol-Number	0.66:1 1.90:1 4.90:1 0.47:1	0.86:1 2.10:1 1.05:1 0.30:1	1.28:1 2.22:1 1.03:1 0.22:1	0.36:1 1.41:1 5.83:1 0.60:1	0.46:1 1.43:1 0.86:1 0.41:1	0.72:1 1.74:1 0.76:1 0.29:1		

From average values obtained in the study of a great number of molasses and sugar cane juice rums, we have found the following general ratios among the group components of the Non-Alcohol-Number in each class:

TABLE 35

SHOWING THE STUDY OF A TWO-YEARS-OLD DEFECATED AND CLARIFIED SUGAR CANE JUICE RUM OF THE LIGHT TYPE

 ORGANOLEPTIC AND PHYSICAL TESTS: Aroma: Excellent, very suave. Taste: Good, characteristic of its class. Body: No. 8.0 Index of Persistence: 1:25,000. Sulphuric Acid Test: Positive; quite perceptible.

(2) CHEMICAL ANALYSIS: Specific Gravity at 20/4 deg. C. Direct Proof, Per cent Proof after distillation, Per cent Alcohol by Volume Per Cent (in distillate) Total Acidity (mgs in 100mL Abs Alc) Fixed Acidity (mgs in 100mL Abs Alc) Volatile Acidity (mgs in 100mL Abs Alc) Esters (mgs in 100mL Abs Alc) Aldehydes (mgs in 100mL Abs Alc) Higher Alcohols (mgs in 100mL Abs Alc) Extract (mgs in 100mL rum) Ash (mgs in 100mL rum) Non-Alcohol-Number

(3) FRACTIONAL DISTILLATION RESULTS:

 0			CHEMICAL ANALYSIS					
Fraction No.		Appearance of C U I I I I I I I I I I I I I I I I I I	Alcohol by Volume	Volatile Acidity	Esters	Aldehydes	Higher Alcohols	Remarks
			Per Cent					
1	78-78	Clear	90.40	2.25	184.45	58.56	19.86	Slight Turbidity
2	78-78	Clear	91.12	2.25	2.11	29.28	10.83	
3	78-78	Clear	88.96	2.25	2.11	29.28	12.66	
4	78-83	Clear	87.32	2.25	2.11	29.28	155.31	Turbid on dilution
5	83-99	Cloudy	23.52	50.34	23.23	29.28	14.45	Oil Drops
6	99-99	Clear	0.28	57.10	2.11	19.52	5.41	Oil Drops
7	99-99	Clear	0.00	60.86	2.11	14.64	5.41	
8	99-99	Clear	0.00	72.13	2.11	14.64	5.41	

NOTE: Members of Non-Alcohol-Number reported in mgs/100mL of fraction.

(4)	VALUABLE RATIOS:	
	Esters: Higher Alcohols	0,41 : 1,00
	Esters: Aldehydes	0,85 : 1,00
	Esters: Volatile Acidity	0,34 : 1,00
	High BP : Low BP Esters	0.17:1,00
	High BP : Low BP Aldehydes	0.92:1,00
	Higher Alcohols: Non-AlcNumber	0.31 : 1,00

0.93964

89.92

90.10

45.05

187.71

15.01

172.70

58.71

68.80

143.34

146.40

458.56

6.40

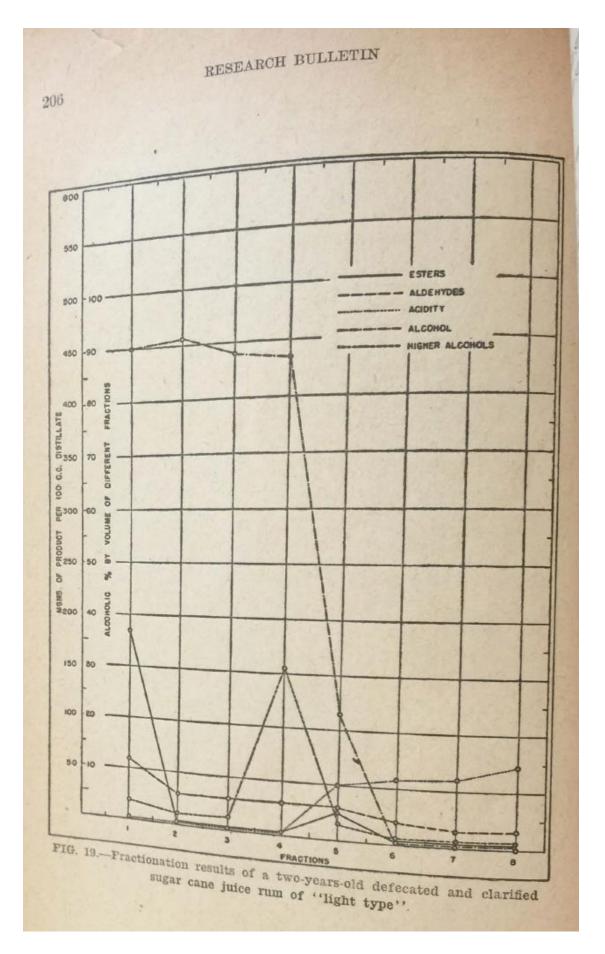


TABLE 36

PRESENTING THE STUDY OF A TWO-YEARS-OLD DEFECATED AND CLARIFIED SUGAR CANE JUICE RUM CLASSIFIED AS MEDIUM HEAVY

(1) ORGANOLEPTIC AND PHYSICAL TESTS:

Aroma: Good. Taste: Good. Body: No. 9.0 Index of Persistence: 1:75,000. Sulphuric Acid Test: Positive; strong residual odor.

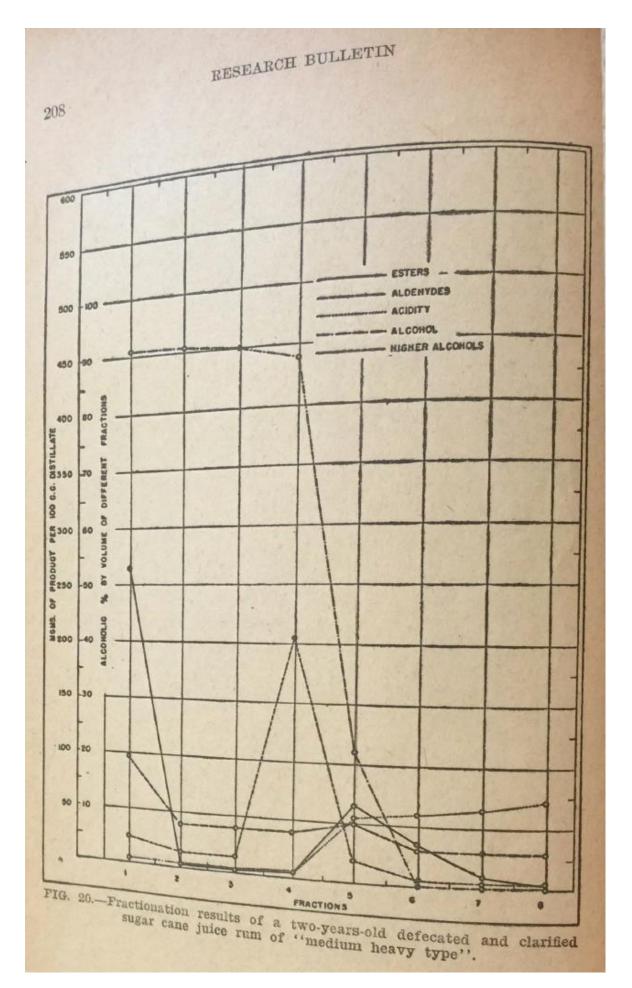
CHEMICAL ANALYSIS: (2) Specific Gravity at 20/4 deg. C.

Specific Gravity at 20/4 deg. C.	0.93923
Direct Proof, Per cent	90.40
Proof after distillation, Per cent	91.36
Alcohol by Volume Per Cent (in distillate)	45.68
Total Acidity (mgs in 100mL Abs Alc)	183.37
Fixed Acidity (mgs in 100mL Abs Alc)	29.33
Volatile Acidity (mgs in 100mL Abs Alc)	154.04
Esters (mgs in 100mL Abs Alc)	128.47
Aldehydes (mgs in 100mL Abs Alc)	70.16
Higher Alcohols (mgs in 100mL Abs Alc)	190.98
Extract (mgs in 100mL rum)	212.00
Ash (mgs in 100mL rum)	3.60
Non-Alcohol-Number	572.98

FRACTIONAL DISTILLATION RESULTS: (3)

Fraction No.	Temperature Range of Distillation Deg. C.	Appearance of Fraction	CHEMICAL ANALYSIS					
			Alcohol by Volume	Volatile Acidity	Esters	Aldehydes	Higher Alcohols	Remarks
			Per Cent					
1	78-78	Clear	91.12	3.38	265.41	97.60	26.46	Turbid on dilution
2	78-78	Clear	91.84	2.25	2.11	39.04	14.42	
3	78-78	Clear	90.40	2.25	2.11	39.04	16.87	
4	78-92	Clear	88.96	4.88	4.22	39.04	206.93	Turbid on dilution
5	92-99	Cloudy	22.64	55.60	66.18	48.80	19.25	Oil Drops
6	99-99	Clear	0.00	60.11	34.50	29.28	7.21	Oil Drops
7	99-99	Clear	0.00	63.87	8.45	29.28	7.21	Oil Drops
8	99-99	Clear	0.00	72.88	2.11	29.28	7.21	

(4)	VALUABLE RATIOS:	
	Esters: Higher Alcohols	0,67:1,00
	Esters: Aldehydes	1,83 : 1,00
	Esters: Volatile Acidity	0,83 : 1,00
	High BP : Low BP Esters	0.43:1,00
	High BP : Low BP Aldehydes	1.00:1,00
	Higher Alcohols: Non-AlcNumber	0.33:1,00



The two years aging record of over seventy rums in each class was used as basis of the above figures. It must be borne in mind, however, that these rums were aged in 5 gallons oak kegs, and that this fact may influence the rapidity found in the changes of the different ratios at different curing periods. But, at any rate, the results have comparative value in the differentiation of rums within a class, and within a different classes. They serve also as a guide in the appraisal of commercial brands, sometimes advertised as of very prolonged curing; but whose study in the fashion already described shows different.

It will be noticed in the above ratios that for molasses rums the ratio of esters to higher alcohols shows a steady increase from the raw to the two years old product. The rate of increase is much more pronounced in the period of aging between the first and second year. In the case of the sugar cane juice rums, although the values of the ratios are much lower - about one half - than the corresponding in the molasses rums, they show, however, the same characteristics of steady increase, and a more striking increase during the second year of aging. One of the reasons for this greater increase in this ratio during the second year of aging is due to the ill effect of dilution upon the ester content of the rums during the first year of curing. If we consider now the ratio of esters to aldehydes in both kinds of rums, we find that this also steadily increases as the curing progresses, although not in the same extent as the esters:higher alcohols ratio. Here again the corresponding comparative ratios are higher in the case of the molasses rums. Taking up now the ratio of esters to volatile acidity we find a sharp decline in both cases from the raw to the one year old products. This should not be surprising when we consider the extremely low value of volatile acidity in the raw rums, and the spectacular way in which this acidity increases even in a few months of curing. The ratio becomes stabilized after the first year of curing, being greater than unity for the molasses rums and less than unity for the sugar cane juice rums. The ratio of higher alcohols to Non-Alcohol-Number is lower in the case of the molasses rums, especially in the raw products. In both classes of rums this ratio diminishes with aging, and after two years it becomes about one half that of the raw rums values. This should not be attributed so much to loss of higher alcohols, but rather to the great increase in the Non-Alcohol-Number throughout the curing period. There are, however, slight losses of higher alcohol through conversion to esters.

A study of these general ratios and their behavior during the curing period offer a very valuable help in the work of differentiation and appraisal of a given rum sample, when used in conjunction with the other tests to which the sample is submitted as already described. We shall proceed with the presentation of the results obtained in the study of a few commercial rums picked out of the many we have examined during the course of these studies. The samples selected for this work were obtained in the local rum market; but obviously, the names of brands and their respective manufacturers are purposely omitted. The rums will be distinguished from each other merely through numbering. The series will include six rums, all of which belong to the light type, and the respective studies are found in tables 37 to 42 inclusive. Figs. 21 to 26 inclusive correspond respectively to the fractional distillation results of tables 37 to 42 inclusive.

The study of these six samples of commercial rums shows definite differences from those previously presented as examples of rums of known manufacture and history. The difficulty encountered in the classification and appraisal of commercial rums is very great indeed, for the tester merely receives a sample of a rum whose history as regards source of raw material, strain of yeast, methods of fermentation and distillation followed, kind of curing methods used, etc. etc., are almost always entirely unknown to him. Besides, the rum may be a blend of molasses and sugar cane juice rums; or of a very matured rum cut with raw rum or even neutral spirits. Then, we have the possibility of rums made by altogether artificial means; or mixtures of artificial and natural rums, etc. etc. All of these possibilities greatly interfere and complicate commercial rums appraisal and classification. Up to the present time government regulation of the rum industry takes care of but one single thing: prevention of fraud in the matter of tax collections, but nothing is being done for the prestige of the industry or the safeguarding of public health through inspection and regulation of manufacturing, rectifying, and curing methods of production.

Coming back to the data presented on the commercial rums, we shall interpret in each case the results of the study, and reach certain conclusion, always within the limitations imposed by the factors already explained above.

SHOWING THE STUDY OF COMMERCIAL RUM NO. 1

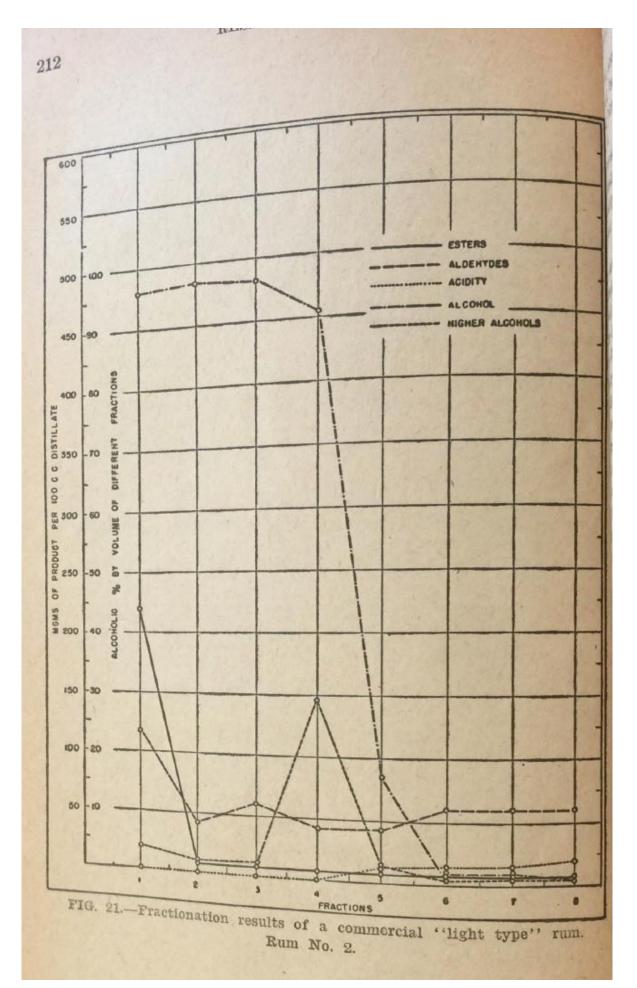
(1)	ORGANOLEPTIC AND PHYSICAL TESTS:	
	Aroma: Good.	
	Taste: Fair, aroma much superior to taste, lack of mellowness.	
	Body: No. 11	
	Index of Persistence: 1:25,000.	
	Sulphuric Acid Test: Positive; quite perceptible.	
(2)	CHEMICAL ANALYSIS:	
	Specific Gravity at 20/4 deg. C.	0.94313
	Direct Proof, Per cent	85.94
	Proof after distillation, Per cent	87.78
	Alcohol by Volume Per Cent (in distillate)	43.89
	Total Acidity (mgs in 100mL Abs Alc)	52.24
	Fixed Acidity (mgs in 100mL Abs Alc)	4.65
	Volatile Acidity (mgs in 100mL Abs Alc)	47.59
	Esters (mgs in 100mL Abs Alc)	69.58
	Aldehydes (mgs in 100mL Abs Alc)	43.98
	Higher Alcohols (mgs in 100mL Abs Alc)	131.94
	Extract (mgs in 100mL rum)	106.40
	Ash (mgs in 100mL rum)	15.40
	Non-Alcohol-Number	297.74

(3) FRACTIONAL DISTILLATION RESULTS:

0.	a. u		CHEMICAL ANALYSIS					
Fraction No.	Temperature Range of Distillation Deg. C.	Appearance of Fraction	Alcohol by Volume	Volatile Acidity	Esters	Aldehydes	Higher Alcohols	Remarks
			Per Cent					
1	78-78	Clear	96.16	1.28	218.24	117.60	18.29	
2	78-78	Clear	96.88	1.28	7.04	42.00	9.97	
3	78-78	Clear	96.52	1.28	7.04	58.80	11.66	
4	78-92	Clear	91.12	1.71	7.04	42.00	143.00	
5	92-99	Turbid	17.32	12.39	7.04	42.00	13.30	Oil Drops
6	99-99	Clear	2.64	14.95	7.04	58.80	4.98	Oil Drops
7	99-99	Clear	2.40	16.87	7.04	58.80	4.98	Oil Drops
8	99-99	Clear	0.80	20.08	7.04	58.80	4.98	Oil Drops

NOTE: Members of Non-Alcohol-Number reported in mgs/100mL of fraction.

(4)	VALUABLE RATIOS:	
	Esters: Higher Alcohols	0,52 : 1,00
	Esters: Aldehydes	1,58 : 1,00
	Esters: Volatile Acidity	1,46 : 1,00
	High BP : Low BP Esters	0.15 : 1,00
	High BP : Low BP Aldehydes	1,19:1,00
	Higher Alcohols: Non-AlcNumber	0,44 : 1,00



SHOWING THE STUDY OF COMMERCIAL RUM NO. 2

 ORGANOLEPTIC AND PHYSICAL TESTS: Aroma: Good, but not that of a genuine rum. Taste: Bad, bitter and sharp. Body: No. 9 Index of Persistence: 1:25,000. Sulphuric Acid Test: Residual odor scarcely persistence.

Sulphuric Acid Test: Residual odor scarcely perceptible; high index of persistence probably due to added aromatic bodies destroyed by the action of the sulphuric acid.

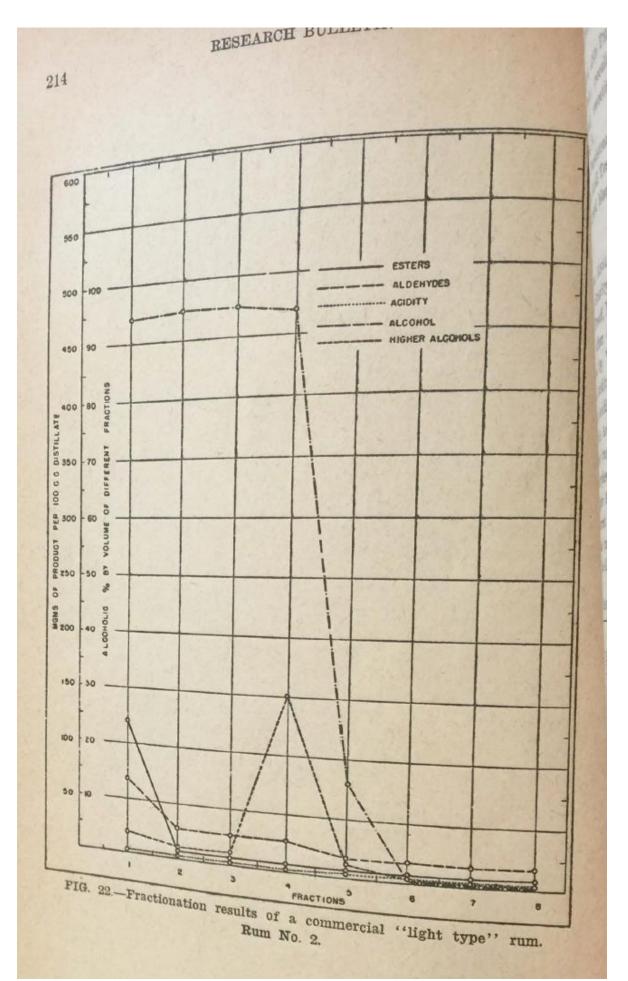
(2)	CHEMICAL ANALYSIS:	
	Specific Gravity at 20/4 deg. C.	0.94638
	Direct Proof, Per cent	82.08
	Proof after distillation, Per cent	84.72
	Alcohol by Volume Per Cent (in distillate)	42.36
	Total Acidity (mgs in 100mL Abs Alc)	23.88
	Fixed Acidity (mgs in 100mL Abs Alc)	0.00
	Volatile Acidity (mgs in 100mL Abs Alc)	23.88
	Esters (mgs in 100mL Abs Alc)	51.47
	Aldehydes (mgs in 100mL Abs Alc)	33.12
	Higher Alcohols (mgs in 100mL Abs Alc)	137.67
	Extract (mgs in 100mL rum)	92.40
	Ash (mgs in 100mL rum)	21.60
	Non-Alcohol-Number	246.14

(3) FRACTIONAL DISTILLATION RESULTS:

0.	n re		CHEMICAL ANALYSIS					
Fraction No.	Temperature Range of Distillation Deg. C.	Appearance of Fraction	Alcohol by Volume	Volatile Acidity	Esters	Aldehydes	Higher Alcohols	Remarks
			Per Cent					
1	78-78	Clear	94.40	1.49	119.68	68.64	19.08	
2	78-78	Clear	95.12	1.49	7.04	28.60	10.40	
3	78-78	Clear	95.48	1.49	7.04	28.60	12.16	
4	78-83	Clear	94.40	1.49	7.04	28.60	149.17	Turbid on Dilution
5	83-99	Slightly Turbid	16.44	4.49	7.04	17.16	13.88	Few Oil Drops
6	99-99	Clear	1.84	5.55	7.04	17.16	5.20	
7	99-99	Clear	1.60	5.55	7.04	17.16	5.20	
8	99-99	Clear	1.04	6.84	7.04	17.16	5.20	

NOTE: Members of Non-Alcohol-Number reported in mgs/100mL of fraction: Strong odor of Cassia Oil in sixth and seventh fractions.

(4) VALUABLE RATIOS:	
Esters: Higher Alcohols	0,37:1,00
Esters: Aldehydes	1,55 : 1,00
Esters: Volatile Acidity	2,16:1,00
High BP : Low BP Esters	0.26 : 1,00
High BP : Low BP Aldehydes	0,77 : 1,00
High BP : Low BP Esters	0.26 : 1,00



SHOWING THE STUDY OF COMMERCIAL RUM NO. 3

 ORGANOLEPTIC AND PHYSICAL TESTS: Aroma: Fair; peculiar odor noticed, as of burnt sugar cane. Taste: Bad; sweetish, and sharp. Body: No. 9 Index of Persistence: 1:12,500. Sulphuric Acid Test: Slightly perceptible: development of or

Sulphuric Acid Test: Slightly perceptible; development of odor of kerosene oil; liquid turned black, showing carbonization of some organic substance, probably sugar..

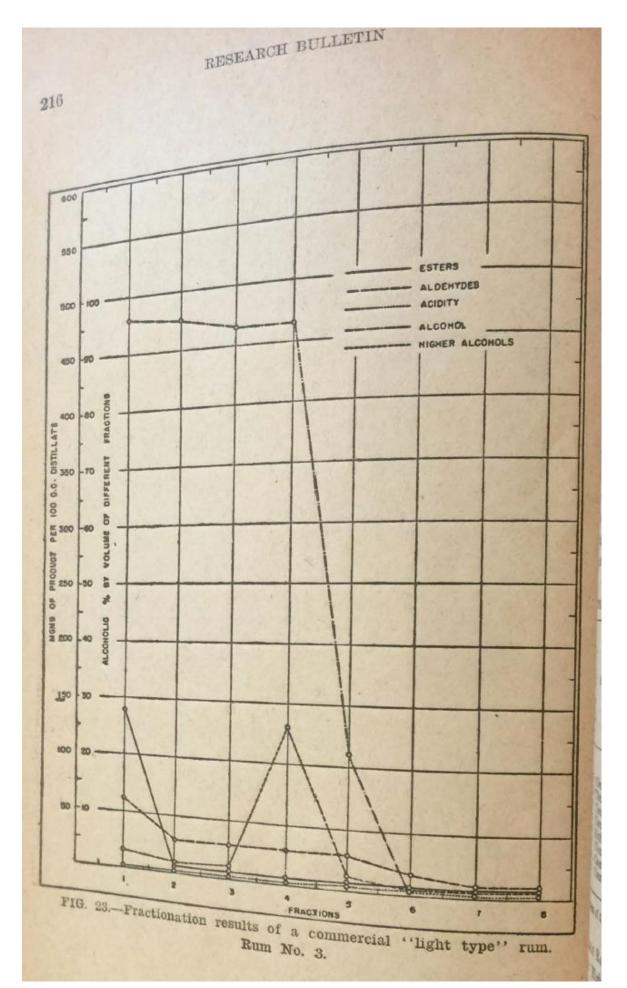
(2)	CHEMICAL ANALYSIS:	
	Specific Gravity at 20/4 deg. C.	0.94401
	Direct Proof, Per cent	84.90
	Proof after distillation, Per cent	87.80
	Alcohol by Volume Per Cent (in distillate)	43.90
	Total Acidity (mgs in 100mL Abs Alc)	18.40
	Fixed Acidity (mgs in 100mL Abs Alc)	6.90
	Volatile Acidity (mgs in 100mL Abs Alc)	11.50
	Esters (mgs in 100mL Abs Alc)	58.00
	Aldehydes (mgs in 100mL Abs Alc)	42.20
	Higher Alcohols (mgs in 100mL Abs Alc)	119.61
	Extract (mgs in 100mL rum)	503.00
	Ash (mgs in 100mL rum)	24.80
	Non-Alcohol-Number	238.21

(3) FRACTIONAL DISTILLATION RESULTS:

	n		CHEMICAL ANALYSIS					
Fraction No.	Temperature Range of Distillation Deg. C.	Appearance of Fraction	Alcohol by Volume	Volatile Acidity	Esters	Aldehydes	Higher Alcohols	Remarks
			Per Cent					
1	78-78	Clear	96.88	1.71	140.80	62.92	16.57	
2	78-78	Clear	95.80	1.71	7.04	28.60	9.03	
3	78-78	Clear	93.68	1.71	7.04	28.60	11.56	
4	78-80	Clear	93.20	1.71	7.04	28.60	129.60	
5	80-99	Slightly Turbid	22.92	4.27	7.04	28.60	12.06	Few Oil Drops
6	99-99	Clear	1.32	5.13	7.04	17.16	4.52	
7	99-99	Clear	1.32	5.13	7.04	11.44	4.52	
8	99-99	Clear	1.04	5.98	7.04	11.44	4.52	

NOTE: Members of Non-Alcohol-Number reported in mgs/100mL of fraction.

(4)	VALUABLE RATIOS:	
	Esters: Higher Alcohols	0,48 : 1,00
	Esters: Aldehydes	1,37 : 1,00
	Esters: Volatile Acidity	5,04 : 1,00
	High BP : Low BP Esters	0.23:1,00
	High BP : Low BP Aldehydes	0,81 : 1,00
	Higher Alcohols: Non-AlcNumber	0,50 : 1,00



SHOWING THE STUDY OF COMMERCIAL RUM NO. 4

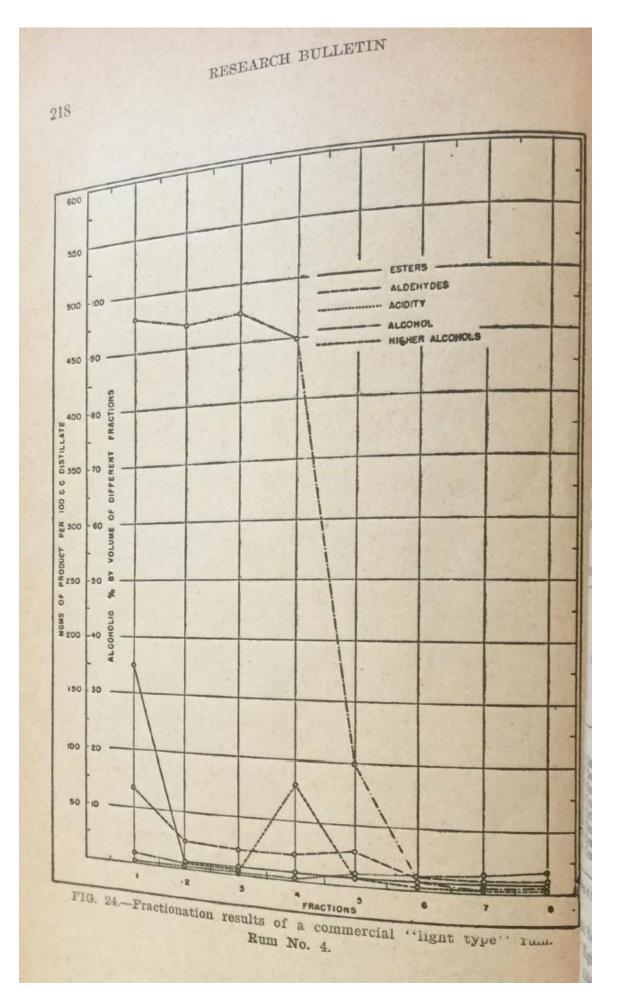
- ORGANOLEPTIC AND PHYSICAL TESTS: Aroma: Good; characteristic of a mixture of cured and fresh rum, plus added aroma. Taste: Good; but rawness indicated; added flavor. Body: No. 11 Index of Persistence: 1:16,700. Sulphuric Acid Test: Positive; quite perceptible genuine after-odor.
- (2)CHEMICAL ANALYSIS: Specific Gravity at 20/4 deg. C. 0.94373 Direct Proof, Per cent 85.24 Proof after distillation, Per cent 89.10 Alcohol by Volume Per Cent (in distillate) 44.55 Total Acidity (mgs in 100mL Abs Alc) 59.54 Fixed Acidity (mgs in 100mL Abs Alc) 27.47 Volatile Acidity (mgs in 100mL Abs Alc) 32.07 Esters (mgs in 100mL Abs Alc) 70.19 Aldehydes (mgs in 100mL Abs Alc) 57.03 Higher Alcohols (mgs in 100mL Abs Alc) 75.49 Extract (mgs in 100mL rum) 727.20 Ash (mgs in 100mL rum) 8.00 Non-Alcohol-Number 262.25

(3) FRACTIONAL DISTILLATION RESULTS:

0.	n re	<u>ਬ</u> . ਰ		CHEMICAL ANALYSIS					
Fraction No.	Temperature Range of Distillation Deg. C.	Appearance of Fraction	Alcohol by Volume	Volatile Acidity	Esters	Aldehydes	Higher Alcohols	Remarks	
			Per Cent						
1	78-78	Clear	96.52	1.71	176.00	68.64	10.46		
2	78-78	Clear	94.04	1.28	7.04	25.76	5.70		
3	78-78	Clear	95.12	1.28	7.04	22.88	6.67		
4	78-82	Clear	90.76	1.71	7.04	22.88	81.79		
5	82-99	Slightly Turbid	20.24	10.25	7.04	28.60	7.61	Oil Drops	
6	99-99	Clear	2.12	12.18	7.04	11.44	2.85	1	
7	99-99	Clear	1.32	13.67	7.04	11.44	2.85		
8	99-99	Clear	1.32	16.66	7.04	11.44	2.85		

NOTE: Members of Non-Alcohol-Number reported in mgs/100mL of fraction.

(4)	VALUABLE RATIOS:	
	Esters: Higher Alcohols	0,93 : 1,00
	Esters: Aldehydes	1,23 : 1,00
	Esters: Volatile Acidity	2,10:1,00
	High BP : Low BP Esters	0.19:1,00
	High BP : Low BP Aldehydes	0,73:1,00
	Higher Alcohols: Non-AlcNumber	0,29 : 1,00



SHOWING THE STUDY OF COMMERCIAL RUM NO. 5

- (1)ORGANOLEPTIC AND PHYSICAL TESTS: Aroma: Very Good; though lacking in purity of tone. Taste: Very Good; though artificially modified. Body: No. 11 Index of Persistence: 1:25,000. Sulphuric Acid Test: Positive; quite perceptible after aroma.
- CHEMICAL ANALYSIS: (2)Specific Gravity at 20/4 deg. C. 0.94325 Direct Proof, Per cent Proof after distillation, Per cent Alcohol by Volume Per Cent (in distillate) Total Acidity (mgs in 100mL Abs Alc) Fixed Acidity (mgs in 100mL Abs Alc) Volatile Acidity (mgs in 100mL Abs Alc) Esters (mgs in 100mL Abs Alc) Aldehydes (mgs in 100mL Abs Alc) Higher Alcohols (mgs in 100mL Abs Alc) Extract (mgs in 100mL rum) Ash (mgs in 100mL rum) Non-Alcohol-Number

	n re	e e	CHEMICAL ANALYSIS					
Fraction No.	Temperature Range of Distillation Deg. C.	Appearance of Fraction	Alcohol by Volume	Volatile Acidity	Esters	Aldehydes	Higher Alcohols	Remarks
			Per Cent					
1	78-78	Clear	95.48	1.07	176.00	64.80	12.01	
2	78-78	Clear	94.36	1.50	7.04	25.92	6.55	
3	78-78	Clear	94.40	1.28	7.04	32.40	7.65	
4	78-83	Clear	92.96	1.28	7.04	32.40	93.91	Turbid on Dilution
5	83-99	Turbid	20.52	10.68	21.12	32.40	8.74	Oil Drops
6	99-99	Clear	1.84	12.82	7.04	19.44	3.27	Oil Drops
7	99-99	Clear	1.84	14.53	7.04	12.96	3.27	Oil Drops
8	99-99	Clear	1.32	17.94	7.04	12.96	3.27	Oil Drops

FRACTIONAL DISTILLATION RESULTS: (3)

NOTE: Members of Non-Alcohol-Number reported in mgs/100mL of fraction.

(4)	VALUABLE RATIOS:	
	Esters: Higher Alcohols	0,66 : 1,00
	Esters: Aldehydes	1,90 : 1,00
	Esters: Volatile Acidity	1,86 : 1,00
	High BP : Low BP Esters	0.30:1,00
	High BP : Low BP Aldehydes	0,89:1,00
	Higher Alcohols: Non-AlcNumber	0,35 : 1,00

85.80

87.22

43.61

72.05

41.17

30.88

57.44

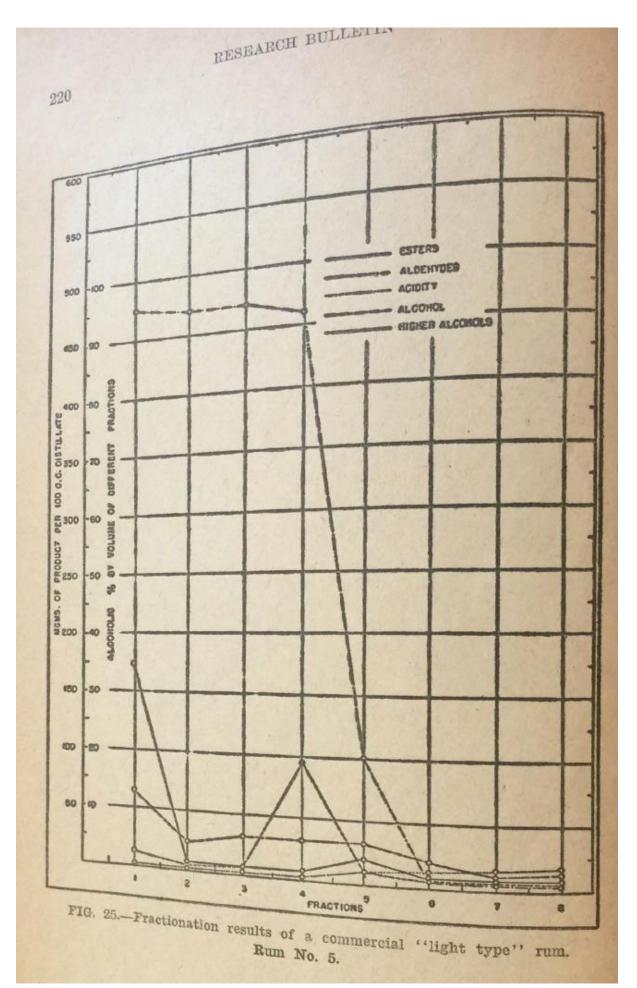
30.21

86.67

626.00

246.37

9.00



SHOWING THE STUDY OF COMMERCIAL RUM NO. 6

- ORGANOLEPTIC AND PHYSICAL TESTS: Aroma: Good; artificial ingredients present. Taste: Fair; sweetish as of added sugars. Aroma superior to taste. Body: No. 7.5 Index of Persistence: 1:5,000. Sulphuric Acid Test: Part of the added aroma persisted; black color developped.
- (2) CHEMICAL ANALYSIS: Specific Gravity at 20/4 deg. C. Direct Proof, Per cent Proof after distillation, Per cent Alcohol by Volume Per Cent (in distillate) Total Acidity (mgs in 100mL Abs Alc) Fixed Acidity (mgs in 100mL Abs Alc) Volatile Acidity (mgs in 100mL Abs Alc) Esters (mgs in 100mL Abs Alc) Aldehydes (mgs in 100mL Abs Alc) Higher Alcohols (mgs in 100mL Abs Alc) Extract (mgs in 100mL rum) Ash (mgs in 100mL rum) Non-Alcohol-Number

0.	e u		CHEMICAL ANALYSIS						
Fraction No.	Temperature Range of Distillation Deg. C.	Appearance of Fraction	Alcohol by Volume	Volatile Acidity	Esters	Aldehydes	Higher Alcohols	Remarks	
			Per Cent						
1	78-78	Clear	91.12	3.86	118.27	89.72	5.66	Turbid on Dilution	
2	78-78	Clear	92.96	1.78	24.99	40.78	3.08	Turbid on Dilution	
3	78-78	Clear	91.48	1.78	21.54	48.94	3.61	Turbid on Dilution	
4	78-83	Clear	90.76	2.38	18.09	53.01	44.26	Turbid on Dilution	
5	83-99	Clear	35.16	5.65	38.79	57.09	4.12	No Oil Drops	
6	99-99	Clear	0.00	11.89	22.60	32.62	1.54	-	
7	99-99	Clear	0.00	13.08	21.26	32.62	1.54		
8	99-99	Clear	0.00	14.27	14.57	32.62	1.54		

(3) FRACTIONAL DISTILLATION RESULTS:

NOTE: Members of Non-Alcohol-Number reported in mgs/100mL of fraction.

(4)	VALUABLE RATIOS:	
	Esters: Higher Alcohols	1,52 : 1,00
	Esters: Aldehydes	0,76:1,00
	Esters: Volatile Acidity	1,78:1,00
	High BP : Low BP Esters	0.70:1,00
	High BP : Low BP Aldehydes	1,16:1,00
	Higher Alcohols: Non-AlcNumber	0,18 : 1,00

0.94837

79.60

86.00

43.00

41.82

6.96

61.92

61.92

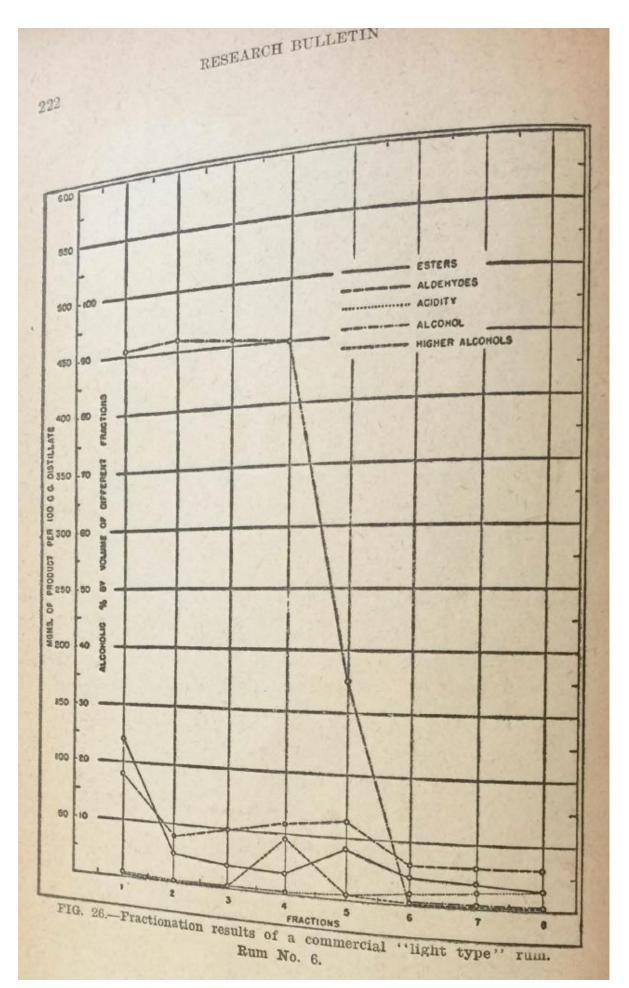
81.72

40.85

12.80

226.31

1,208.80



Rum No. 1 of table 37 is probably a genuine product of very little aging, perhaps not more than three or four months. It was distilled under high proof, in a still lacking the pasteurizing column. Hence, its very low ratio of high to low B. P. esters. Head products are abundant in this rum and are responsible for the lack of mellowness. Its low Non-Alcohol-Number indicates the light type of molasses or blend of molasses and sugar cane juice rum. The rather high ratio of higher alcohols to Non-Alcohol-Number also supports the inference about the rum being really a blend of the above-mentioned different kinds of rum. Its high index of persistence is due to mainly to the presence of rum oil in quite generous amounts. This rum could become a fairly good product under prolonged aging in a well-conditioned and quite porous barrel.

Rum No. 2 of table 38 is a product quite different to rum No. 1 just discussed. In this case the rum is altogether a raw distillate to which has been added artificial coloring, aroma, and flavor. The presence of the extract in the sample is due to the caramel used for imparting color and the flavoring substances used in its concoction. Its high index of persistence was due to added aroma, which is shown by the fact that after the sulphuric acid treatment its aromatic tone was scarcely perceptible. There was but little presence of rum oil in this sample as shown in the fractional distillation test, and further supported by the sulphuric acid test. Aroma due to rum oil in the sample does not disappear after the sulphuric acid test. The causes of its bad, bitter and sharp taste are its great rawness and the artificial addition of aromatic substances of probably bitter taste. The complete absence of fixed acidity in the sample is also a very eloquent sign of its rawness. Here again may be noticed the poor ratio of high to low B. P. esters and of higher alcohols to Non-Alcohol-Number, but the amount of total acidity for a raw product points more to a straight molasses than to a mixture of molasses and sugar cane juice rum. This is a poor representative of a commercial product.

Rum No. 3 of table 39 had a sweetish, bad, sharp taste. The sweetness was due to added sugar during the processing. Its index of persistence is low as would happen to a raw product with little rum oil, and very few highly aromatic esters and aldehydes; as shown by the results of its fractional distillation test, and the low ratios of high to low B. P. esters and high to low B. P. aldehydes. The rum was distilled under considerable high proof, probably between 185 and 189 degrees. The large amount of extract is the result of the added caramel and sugar. Its high ash content may be the result

of chemical treatment for obliteration of foulness of odor and taste. Again we have a very poor ratio of higher alcohols to Non-Alcohol-Number. This is another poor specimen of a commercial product. A mixture of molasses and sugar cane juice rum is here indicated.

On examination of the ordinary chemical analysis of rum No. 4, table 40, two features are found conspicuous at once: (1) the great difference in degree proof between the rum sample as such, and its distillate; (2) the very high extract characteristic of rums of long time aging in barrel. But in this case the rum sample was comparatively raw (not over 6 months aging) and, therefore, the cause of the high extract was the same responsible for the great difference in alcoholic concentration between the sample and its distillate. Added solid substances, especially sugars, are almost always responsible for these anomalies in the composition of commercial rums.

This particular sample was probably representative of a cut of an old rum with fresh distillate. As in the rest of the commercial rums whose studies have been discussed, we find poor ratios of high B. P. to low B. P. esters, which indicate distillation at very high proof by which head products predominate in the members of the Non-Alcohol-Number, if high rectification is not used simultaneously during the distillation. The high body number, the medium index of persistence, and the good ratio of higher alcohols to Non-Alcohol-Number indicates that this rum or rum blend was produced from straight molasses rum.

The organoleptic tests indicate the presence of added aroma and flavor.

Rum sample No. 5 of table 41 is probably a straight molasses rum of perhaps a year or so of aging in barrel. But before bottling, the product was modified by addition of extraneous flavoring substances. The high index of persistence in aroma and taste indicates the presence of fair amounts of rum oil, which was shown during the fractional distillation. Although the amount of extract is rather high for a rum of its probable age, the difference in proof between the sample and its distillate is not so high as in the previously discussed rum No. 4. This would indicate that at least a considerable part of the extract had a natural source. The comparatively large amount of fixed acidity would indicate that the rum had been aged for a considerable length of time, but this is sometimes a misleading factor, as often the manufacturer adds liquid ammonium hydroxide to the rum before bottling with the idea of fixing some of the volatile acidity of the rum in the form of ammonium salts. It will be noticed that in this sample there is a small rise in the ratio of high B.P.:low B.P. esters; and a lowering in the ratio of higher alcohols to Non-Alcohol-Number. Generally speaking, this sample proved superior in quality to all the others so far discussed among the commercial products.

Rum sample No. 6 of table 42 is a characteristic artificially built up rum, using industrial alcohol, aromatic esters, and body and flavor giving substances in the concoction of the commercial rum. However, all this was done with true knowledge and art, so the resulting beverage was agreeable in taste and aroma and if it lacked the genuine rum odor, it at least was a fairly agreeable cordial.

The organoleptic and physical tests began to show the fact that sugars and aromatic and flavoring constituents had been used. Then, the very low body number indicated that the rum was produced under very high rectification or was made artificially from practically pure ethyl alcohol. The sulphuric acid test obliterated that part of the aroma produced by the added esters and aldehydes; but part of the aroma passed successfully this test, showing that other aromatic bodies not affected by the action of the sulphuric acid were also added. That this aroma was not due to the rum oil was evident from the fact that during the fractional distillation this oil proved to be absent from the sample.

Turning to the ordinary analysis of this rum, we find a tremendous difference in the degrees proofs before, and after distillation of the sample; which again shows the apparent lowering of alcoholic strength effected in the rum through the addition of extraneous ingredients. This is again confirmed by the extraordinary amounts of extract found in this sample.

Referring to the fractional distillation data, we find two features not noticed yet in any of the other fractionations; which are the absolute absence of rum oil and the presence of turbidity on dilution in the second and third fractions. These two fractions which usually are very clear upon dilution and almost devoid of aroma, were found to become turbid on dilution and also possessing a quite perceptible peculiar aroma which could not be classified. This shows the presence of some strange aromatic substance less soluble in water than in alcohol. This was probably the aromatic constituent that passed successfully the sulphuric acid test. The sugar free extract was determined in this case, and it was found that the 1,208.80 milligrams of extract, 1,112.00 were due to added sugars, leaving a sugar free extract of only 96.80 milligrams.

All of these facts indicate that this was an spurious sample of rum concocted by a skillful person in the art, for it will be noticed that the aromatic bodies added were selected to offer a fairly good chemical structure in the artificial rum, and good enough ratios among the group constituents, as may be readily seen by referring to them in table 42.

This is why it becomes almost impossible to judge the source, quality, and genuineness of a rum, from one single test. A number of tests interrelated among them are necessary, most times, to allow of even an approximate assertion.

Now, the most important single factor involved in the work of classification on degree of genuineness and purity in the product, unquestionably is the determination of the presence or absence of rum oil in the sample. But even this is not an infallible guide, for the rum may be a genuine product in the sense of being unadulterated, and still lack the presence of rum oil in its chemical composition. The absence of rum oil may originate from two different causes: (1) the yeast strain used may not produce it. (2) The method of manufacture may practically eliminate the rum oil from the commercial product, even when the yeast used be a good producer of the oil. The test could, however, gain added significance, at least for locally produced rums, if only those yeast which are good producers of rum oil are used at the different distilleries; and if manufacturing methods (especially during distillation) are conducted in such way as to preserve the valuable, distinguishing, characteristic rum oil in the commercial product.

Before closing this chapter on rum differentiation the writer wishes to inform on a rapid, simple method he has developed, which although incapable of ascertaining in a definite, unmistakable way, the genuineness or spuriousness of a given rum, will, however, establish a very sound reason for further investigation in suspicious cases. There are a great many instances, however, in which this simple method will be sufficient to settle the question of genuineness or spuriousness without any further investigation. In all cases, this simple technique should be the starting point from which to pass judgment on the authenticity of commercial rums.

The method is based upon three simple determinations, viz.:

- (1) Extract in 100 milliliters of the rum sample.
- (2) Direct proof of sample.
- (3) Proof of sample after its distillation.

The writer has had occasion to observe the relation existing in different rum samples, among the content of extract and the proof of the commercial product before, and after distillation. It has been observed that genuine rums, whose extract content has entirely been derived from the staves of the aging barrel, will show but little difference in their degrees of proof before and after distillation of the sample. Moreover, it has also been found that the differences in degree proof between the undistilled and distilled samples are not directly proportional to the amount of extract found. This fact shows that the effect exercised by the content of extract on the difference of proof between that of the sample as such, and its distillate, is influenced not only by the amount, but also by the nature and chemical composition of this extractive matter. In this guide, we find genuine rums of less content of extract showing sometimes more difference than other genuine rums of higher extract content, in their respective direct proofs and proofs after distillation. But these differences are *never* of great magnitude.

Therefore, it seems that natural extractive matter obtained during the aging process has little power to cause great variations in the degrees proofs before and after distillation of the given sample' and that the kind and nature of the extractive matter dissolved in the rum has a greater effect than its quantity.

In the case of genuine rums, the degree proof at which the rum is barrelled for aging, the kind and size of barrel, the treatment and curing to which these barrels were submitted previous to their use for aging purposes, and the period of aging time allowable in different cases, will have great influence on the relative amounts and nature of the extractive matter to be found later in the cured product. But we have found that in all cases, the differences between direct proof and proof after distillation of the sample will be of small degree when dealing with the genuine article.

The picture is altogether different when dealing with spurious rums; that is, rums whose aroma and flavor, and the whole, or great part of the extract content, have been artificially imparted through the addition of ingredients extraneous to genuine rums. In these cases of artificially concocted commercial rums, almost without exception, the differences between the direct proof and the proof after distillation of the samples, are of quite perceptible magnitude; amounting in some cases to several degrees. Also, these differences are almost invariably directly proportional to the amount of extract found in the different samples analyzed. This opposite behavior between the two classes of rums mentioned above, gave rise to the development of our method of differentiation under discussion; which consists in running duplicate determinations of the amount of extract found in 100 milliliters of the sample, expressing the average result in terms of milligrams per 100 milliliters of spirit; then taking the degree proof directly on the sample as received, and on its distillate, by the pyknometric method. Then, from a consideration of the degree of difference between the respective degrees proof, and of the amount of extract found, the probable genuineness or spuriousness of the given sample under consideration may be ascertained.

In cases of doubt after performing these simple tests, recourse must be had to other more complicated methods of differentiation, as outlined in the subject matter of this chapter.

Tables 43 to 46 inclusive, show the relations among amounts of extract and the degrees proof before and after distillation of the samples, in the cases of: (1) One year old, (2) Two years old, (3) Three years old, genuine rums; and (4) A set of commercial rums picked up at random in the local market, and which included both native and foreign brands, with no guarantee as to time of aging.

The genuine, one to three years old rums, whose analyses are given in table 43 to 45 inclusive, were aged in 5 gallons oak kegs. The kegs were cured before they were filled up with the respective spirits for aging and maturing.

TABLE NO. 43

	Extract	Degre	Amount of variation deg. proof	
Sample No.	No. (Mgs. Per 100 ml) Dir			
1	100.20	94.20	94.84	0.64
2	139.20	95.00	95.60	0.60
3	145.70	89.15	89.50	0.35
4	161.10	91.60	91.76	0.15
5	162.00	90.14	90.32	0.18
6	168.80	87.00	87.16	0.16
7	104.40	94.24	94.40	0.16
8	151.90	92.72	93.20	0.48
9	207.70	93.22	93.30	0.08
10	241.00	137.06	137.36	0.30
Averages	158.20	96.44	96.74	0.30

ONE-YEAR-OLD RUMS

	Extract	Degr	Amount of		
Sample No.	(Mgs. Per 100 ml)	After Direct Distillation		variation deg. proof	
1	169.00	89.50	90.40	0.90	
2	185.60	89.14	90.04	0.94	
3	200.40	92.42	93.00	0.58	
4	204.00	92.08	92.60	0.52	
5	212.00	90.40	91.36	0.96	
6	266.80	90.78	91.78	1.00	
7	226.80	90.78	91.78	1.00	
8	269.60	88.62	89.34	0.72	
9	265.20	85.36	86.26	0.90	
10	282.00	90.92	91.68	0.76	
Averages	228.14	90.00	90.82	0.82	

TABLE NO. 44 TWO-YEARS-OLD RUMS

TABLE NO. 45 THREE-YEARS-OLD RUMS

	Extract	Degree	Amount of	
Sample No.	(Mgs. Per 100 ml)	Direct	After Distillation	variation deg. proof
1	209.00	91.90	92.56	0.66
2	216.80	87.00	89.00	2.00
3	262.80	89.52	90.65	1.13
4	312.40	94.30	95.82	1.52
5	432.00	96.30	97.40	1.10
6	472.20	88.88	90.48	1.60
Averages	317.53	91.32	92.65	1.33
-				

TABLE NO. 46 COMMERCIAL RUMS, WITH NO GUARANTEE AS TO AGE

	Extract	Extract Degree Proof			
Sample No.	(Mgs. Per 100 ml)	Direct	After Direct Distillation		
1	503.00	84.90	87.80	2.90	
2	518.00	88.50	91.30	2.80	
3	555.50	85.10	88.30	3.20	
4	626.00	84.50	87.80	3.30	
5	639.60	98.00	100.00	2.00	
6	692.00	84.90	88.50	3.60	
7	927.20	85.00	89.10	4.10	
8	1,208.80	79.60	86.00	6.40	
9	1,451.20	77.84	86.00	8.16	
10	2,075.00	71.20	86.00	14.80	
Averages	919.63	83.95	89.08	5.13	

Biological and Chemical Control of the Rum Distillery, Calculations and Reports

At the time when our studies were initiated this phase of rum manufacture was utterly ignored by our producers with the exception of but one large distillery. But even in this more up-to-date distillery, the biological and chemical control lacked the necessary effectiveness, being really in its nascent stage. The rum industry occupied the position held by the sugar cane industry some 50 years ago, as to methods of manufacture and process control. These conditions have been greatly improved during the last two or three years, and at the time of writing there exists a strong current for the introduction of chemical and biological control, more of less complete, in practically all our more important distilleries. Modern manufacturing processes tend to increase the productive capacity of old distilleries, cheapen the cost of installing new ones, and increase yields and quality of finished products.

As to the necessity of such chemical and biological control in the rum distillery there is not the slightest doubt in our mind. How could a distiller work in an efficient manner ignoring the chemical composition of his raw material, its physical characteristics, its microbiological contamination, etc. etc.? Or how could he be certain as to the nature of his final product without a thorough knowledge of the biological characteristics and products of metabolism of the ferment, of ferments, that were to operate upon his raw material converting it into the desired end product? And how is the distiller going to follow the course of manufacture and know of its condition at any given moment, if he lacks the necessary supervision and technical control that would indicate the obstacles to overcome and offer clues for future improvement and new developments? How are we to elude or overcome contaminations whose origins are unknown? How are we to obtain characteristics in the end product incompatible with the nature of our ferments or the environment in which these must operate? What shall serve as guide and orientation for changes or innovations that would benefit the distillery? How shall we know the yields, with certainty and accuracy; the losses and their causes; the relative efficiencies of the fermentation and distillation ends of the works and costs and profits without an adequate supervision and control?

Admitting then, that such technical supervision and control is absolutely essential - where does each phase of this control begin

and end? We may indicate that both the chemical and biological phases of the control begin with selection of ferments and raw materials' the biological phase of the control ending with the delivery of the fermented mashes to the distilling end of the distillery, and the chemical phase with the bottling of the commercial rum.

Let us then see how this control should be exercised during the various stages of manufacture, and the services it will render in each one.

In the selection of a suitable rum yeast strain, biological and chemical factors must be considered. We have choose a strain, top of bottom fermenting; rapid, moderate, or slow fermenting; high or low acidity resisting; adaptable or not to high alcoholic concentrations; resistant or not to high temperature of fermentation, or to infections from extraneous microorganisms, etc. etc. All of these characteristics will be determined by the biological control. On the other hand, we may select a yeast for the quantity or quality of its Non-Alcohol-Number, or become of its low production of a particular member of this Number; or on account of its high yields of ethyl alcohol, etc. etc. In this second case the chemical control becomes the most important factor.

It has been explained in the chapter on yeast selection how this work was carried on. It was also shown how the "birectifier" was highly instrumental in obtaining necessary data and information regarding the metabolic products of fermentation entering into the chemical composition of the Non-Alcohol-Number produced by a given yeast strain; and more especially in determining whether or not the yeast in question was a good, fair, or poor producer of the most valuable rum oil.

The molasses entering the distillery should also be subjected to both a chemical and a biological control. The chemist will inform the distiller about the chemical composition of his raw material and the different ratios existing among its constituents. From this data the distiller will calculate the merits of his raw material and the probable yields that may be expected from it. It will also serve as a guide in the selective purchasing of his molasses. The determinations of most importance and practical value are: total sugars as invert, total nitrogen, phosphoric acid as P_2O_5 , molasses gums, and ash. Of great value also are the determinations of pH value and natural aroma. The methods of analysis followed by us were obtained from the Official Methods of the Association of Agricultural Chemists of the U.S.; Cane Sugar Handbook by Spencer and Meade; and Food

Analysis, by Wood; and the reader is referred to these various sources of information for detailed descriptions of the pertinent analytical procedures.

The second, or biological control, offers to the distiller an idea of the nature and extent of the microbiological flora already present in the raw material. With this knowledge at hand, he may plan on how best to carry the subsequent stages of mashing and fermenting so as to check or contravene to the maximum extent within his means the deleterious action of the existing infection.

A very simple method of procedure is to use small portions of the molasses in the confection of liquid media (using sterile water) suitable respectively for the growth of yeasts, molds, and bacteria. These different mashes are placed in sterile Erlenmeyer flasks and allowed to stand at suitable temperatures. Observations are then effected at different intervals of time for the development of fermentation or growths indicative of the contaminations present. This simple control may be effected at any distillery, even when no bacteriologist, or other highly trained person is employed; and it is particularly beneficial when sterilization of the raw material is not executed at the distillery.

Let us, as an example, suppose that such a test has been carried on the molasses stock for a one week's run of a given distillery, and that it shows the material to be highly contaminated with bacteria. The distiller not having any means of sterilization, or available antiseptics at his command, may yet contravene the rapid development of the infection in the fermenters by: (1) using a low pH at setting, that is, between 4.0 and 4.5; (2) using low temperature of fermentation, that is, between 26 and 28 degrees Centigrade; (3) using rather high Brix densities, between 22 and 23 degrees; and (4) pitching a yeast footing of the highest possible cellular concentration and volume into the fermenter.

When pretreatment of the raw material is performed previous to mashing operations as already explained in the third chapter, the biological and chemical control of this process is also very necessary. Microscopical examination of the material before, and after the treatment, will indicate its degree of effectiveness in destroying microbiological life. Comparative chemical examination of the treated, versus the untreated raw material, will demonstrate to what extent and degree it has been benefited from a chemical and fermentative standpoint by the given treatment. Modifications in the *modus operandi* of this pretreatment may then be introduced in accordance with the results obtained from this control.

Also from the chemical analysis of the raw or the treated material, as the case may be, we may calculate the existing deficiencies in yeast nutriments and add them accordingly. Economy in added nutrients is in this way effected.

The weight of raw material entering the distillery for mashing operations multiplied by the percentage of total sugars in the material by weight, will give the total amount of sugars entering fabrication. When this raw material is in the form of heavy, undiluted molasses, the obtention of an average sample that would accurately represent the total amount of molasses mashed for the day's work is indeed very difficult. Such is not, however, the case when the pretreated, diluted material forms the basis of calculation for the total amount of incoming sugars. In this case, the very greatly increased fluidity and mixing qualities of the material makes it an easy task to obtain a continuous sample, much in the same fashion that the diluted juice is sampled at the cane sugar factories. A very accurate account of the incoming sugars may then be effected, whose importance will be at once realized by the control chemist or plant superintendent.

The most precise biological control must be exercised during the building up of the yeast seed footing for the fermenters, whenever a selected yeast is used at the distillery. A description of the method developed and followed by us for this purpose has already been presented to the reader's attention in the introductory paragraphs of the fifth chapter.

During setting and subsequent fermentation of the mashes, the process should be under strict technical control. To begin with, a record should be kept for each fermenter in the following, or similar form, according to individual distillery equipment and conditions, and to available technical personnel:

Individual Fermenter Record Date Fermenter number Hour inoculated Cellular concentration and volume of footing Type and amount of infection, if any Initial cellular concentration in fermenter Time of maximum cellular concentration Cellular concentration at end of fermentation Appearance of contamination, if any Duration of fermentation, hours Mash Analysis and Observations:

(a) Degree Brix

(b) pH value

(c) Setting temperature degrees C.

(d) Total Sugars, grams/100 ml mash

(e) Titratable acidity

Beer Analysis and Observations:

(a) Maximum temperature attained

(b) Average temperature degrees C.

(c) Final temperature degrees C.

(d) Final degree Brix

(e) Degree of attenuation

(f) Average pH value

(g) Final pH value

(*h*) *Final titratable acidity*

(i) Residual sugars, grams/100 ml beer

(*j*) Alcohol by volume, per cent

(k) Grams alcohol/100 ml beer

Calculations:

(a) Sugar fermented, per cent total sugars

(b) Yield of alcohol on weight of sugars

(c) Fermentation efficiency, per cent

(d) Gallons of beer in fermenter

(e) Gallons of alcohol in fermenter

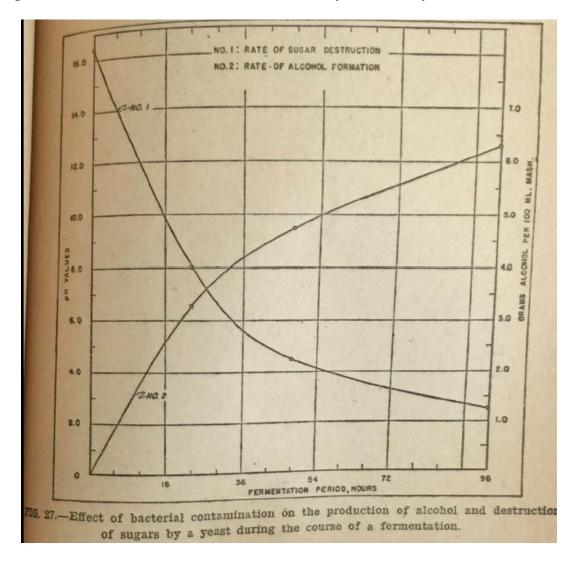
Remarks:

From the analytical work thus performed and observations made, valuable knowledge and experience is gained concerning this most important phase of rum production, and calculations may be effected that show the good or poor results obtained from each individual fermenter. Also the behavior of different fermenters may be compared and reasons found out for better results in one case than in another.

At periodic intervals during the lapse of fermentation, observations on temperature and degree Brix of the fermenting liquid should be effected. Also determinations of pH values and titratable acidities; and of grams alcohol and total sugars per 100 ml substrate.

With the latter data graphs may be constructed showing in one case the variations in pH values and titratable acidities; and in the other case, the rate of sugar destruction and alcohol formation during the period of fermentation. Figs. 27 to 32 show some of these curves drawn during our experimental work.

Figs. 27 and 28 represent a case of bacterial contamination. Notice the prolonged period of fermentation (96 hours), the slow rate of sugars destruction and of alcohol formation. We find from inspection of Fig. 27 that about 6.31 grams of alcohol per 100 ml beer were formed in 96 hours of fermentation out of 16.5 grams of sugar available for conversion. This means a yield of only a little



over 38 per cent on total sugars. Fig. 28, which represents conditions of pH and titratable acidity during the period of this fermentation, clearly shows the state of infection that occurred, since the pH value and titratable acidity at setting were 6.1

and 3.3 ml of the alkali, respectively, and at the end of fermentation these values had changed to pH 3.5 and titratable acidity of 9.6 ml of tenth normal alkali.

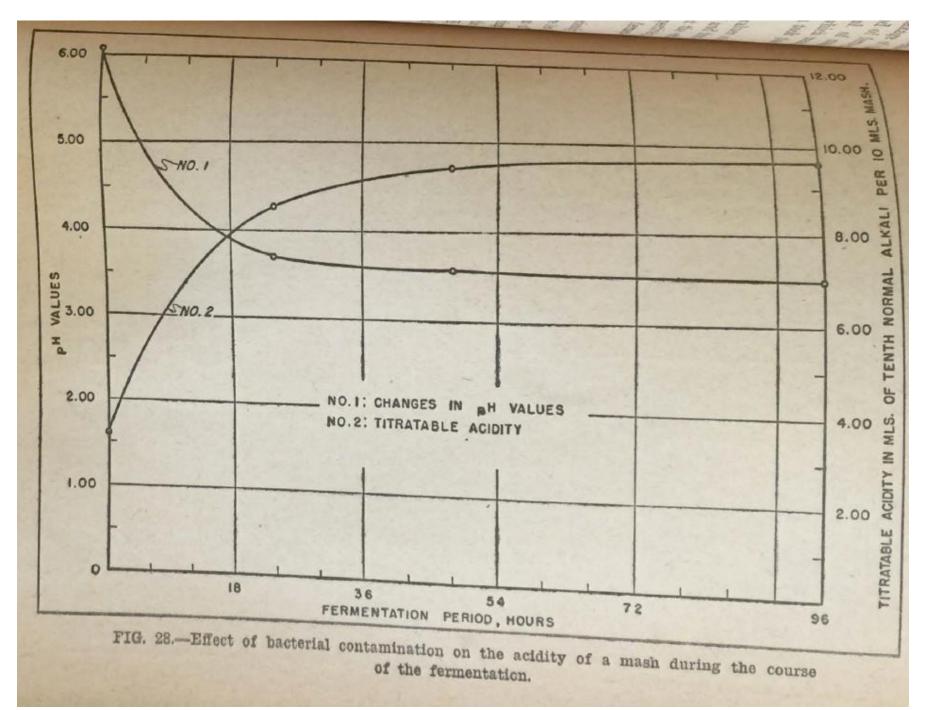
Figs. 29 and 30 represent a normal, efficient fermentation. Referring to Fig. 29 which represents conditions of sugars conversion into alcohol during fermentation, we find a steady breakage of the sugars and conversion into alcohol from beginning to end of fermentation. Out of 12.5 grams of total sugars per 100 ml of mash, we find that 6.0 grams of alcohol have been produced, which means a yield of 48.0 per cent on sugars. And the conversion took place in the short period of 36 hours.

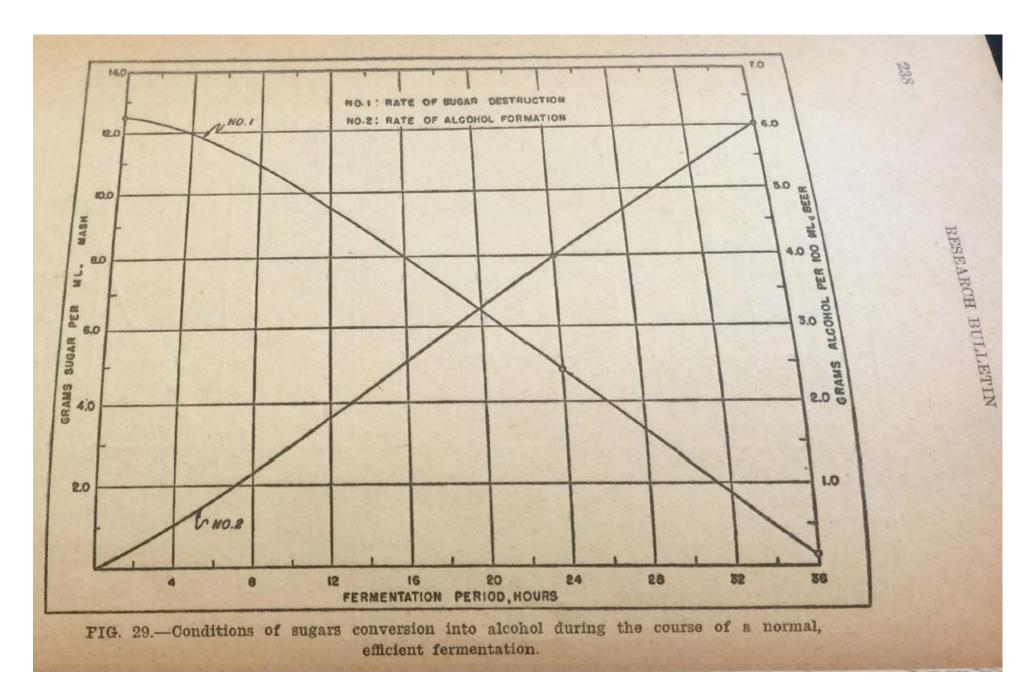
Considering Fig. 30 which shows conditions of pH values and titratable acidities during fermentation, we have initial pH and titratable acidity values of 4.55 and 1.38, respectively, which at the end of the fermentation period had changed to 3.34 and 3.78, respectively, which means normal acid formation during the period of fermentation.

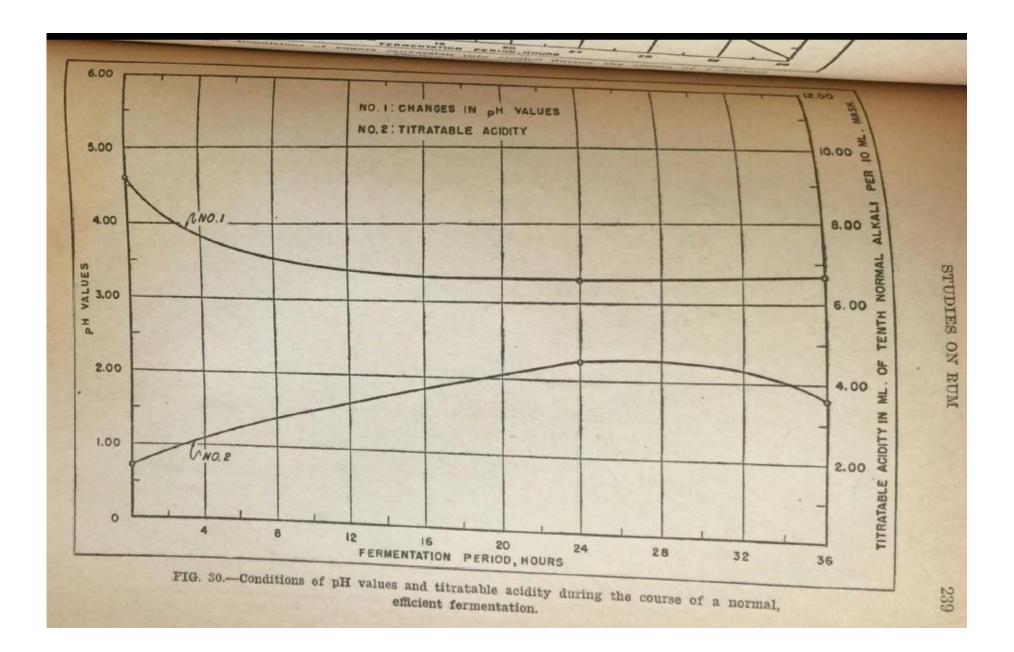
Figs. 31 and 32 represent a fermentation carried under practically constant pH value and titratable acidity throughout the entire fermentation period. Notice the short time taken for completion of the fermentation, and how nearly parallel run the curves representing respective pH values and titratable acidities. In the case of the pH values, the variation from initial to final value is only of 0.15 pH; and in the case of the titratable acidity the increase at the end of fermentation is of 0.24 ml of tenth normal alkali per 10 ml mash.

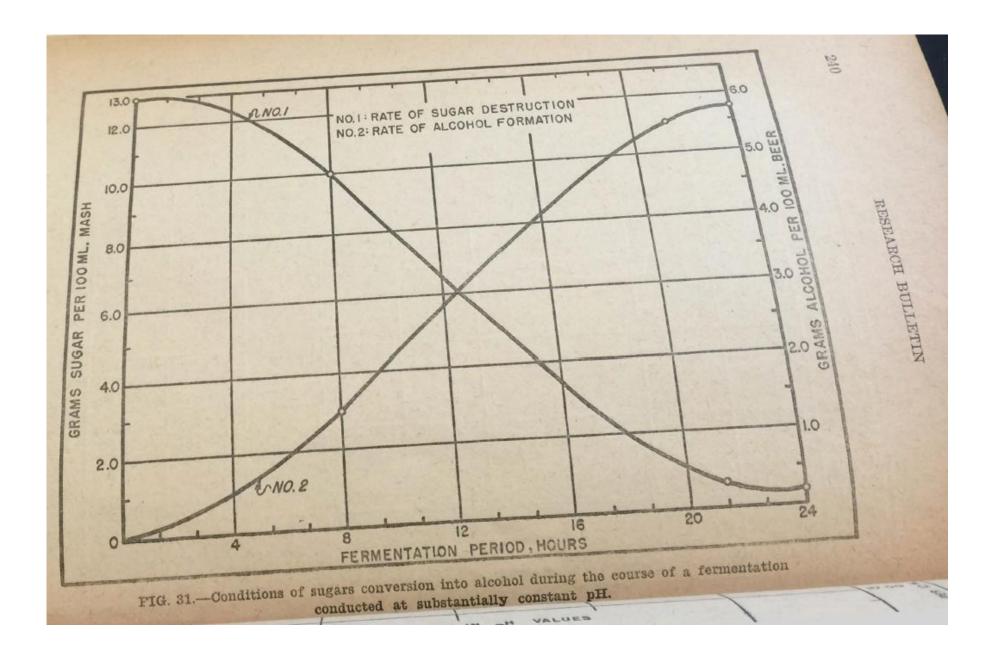
Fig. 31 shows a very rapid conversion of sugars into alcohol, for in 24 hours, out of 13.0 grams of total sugars per 100 ml mash, there have been formed 6.2 grams of alcohol, thus obtaining a yield of over 47.5 per cent on the weight of sugars. As stated before in the chapter on Rum Fermentation, the rums produced under this constant pH technique are of exception mildness of flavor and delicacy of aroma.

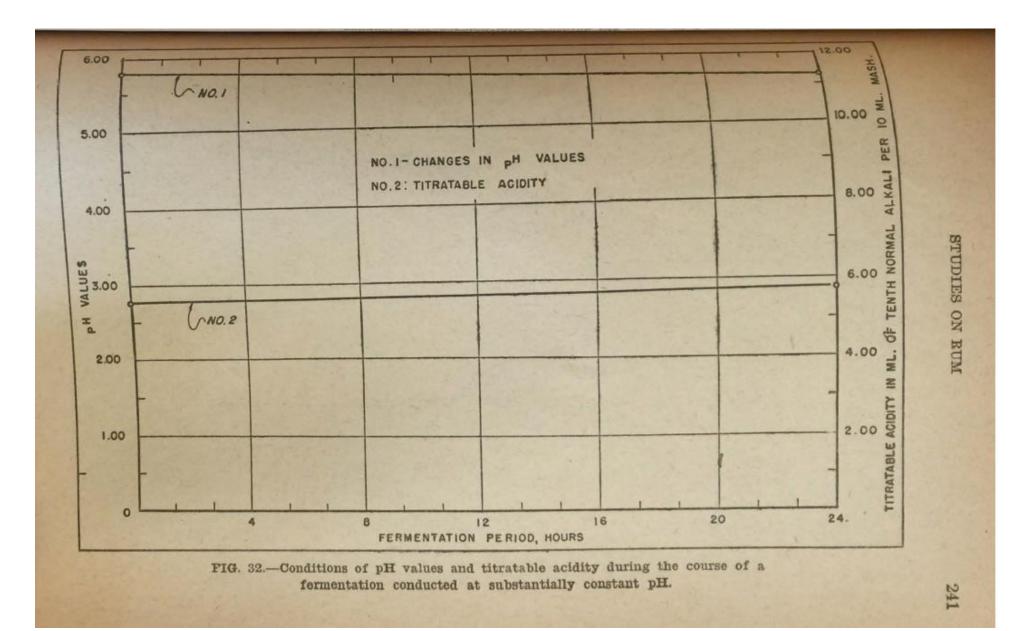
The determinations of pH values and titratable acidities at periodic intervals during the fermentative lapse, become a ready and easy means of detecting bacterial infection. A sudden drop in the pH value, accompanied by a similar rise in titratable acidity are sure signs of infection by acids-producing bacteria; especially acetic acid or butyric acid ones. In this connection it may be pertinent to observe that it is the titratable acidity which must be more closely controlled during the course of fermentation, especially when dealing with molasses mashes, since due to their usually well buffered conditions, the titratable acidity may attain dangerous proportions without the pH value showing a proportional alteration. **RESEARCH BULLETIN - STUDIES ON RUM**











While the presence of vegetative forms of the common acid producing bacteria is easily detected through simple microscopic observations of the substrate, the case becomes more difficult when attempting to detect the presence of wild yeast. Unless the morphological characteristics of the adventitious yeast infecting our mash be quite different to those of our cultured strain, simple microscopical observation may lose much of its valor and efficiency as a control measure. This is due to the fact that morphological differences among quite similar strains of yeast are not of easy establishment, even for the experienced experimenter and observer. This difficulty is aggravated by the fact that the same yeast strain will produce different cellular forms and shapes, according to age, nutrition, access to oxygen, and reaction of the medium. The sporulation test will be a great help in the detection of infection by wild yeast; but it has the inconvenience of taking too long a time.

When the distillery has the custom of giving a period of repose to the fermented mashes before distilling them, the biological control should be extended to this phase of the manufacture. Infection, especially by mold fungi, is very likely to occur once the alcoholic fermentation is accomplished. When incipient signs of infection of this or other nature are detected, the best procedure is to stop the resting period and distill the beers at once. Thorough disinfection of the fermenter, or special resting tank, should be effected as soon as the beer has been distilled.

Table 47 presents data on various disinfectants and ways of using them.

It may be said that with the delivery of the fermented mash to the distillation department, the biological control ends at the rum distillery. But not so as regards to chemical control.

As will be shown later in this chapter, when treating on the subject of distillery calculations and reports, by means of the technical control exercised up to, and including the fermentation and post-fermentation stages of rum manufacture, we shall know the number of gallons of ethyl alcohol delivered to the stills for distillation. Once the distillation is performed, we may easily determine what percentage of the total alcohol in the beer has been actually recovered by the still. Low yields may have their origin in the fermentation phase, the distillation, or in both. It is the chemical control that determines this with precision. Without control we are utterly incapable of determining where the weak spots exist in our process.

RESEARCH BULLETIN - STUDIES ON RUM

TABLE NO. 47

DISINFECTANTS AND THEIR USE

Name of Substance	Physical State	Active Element	Cleaning of Wooden Containers	Cleaning of Metal Containers	Cleaning of Rubber Tubing	Cleaning of Flasks	Cleaning of Floors, Walls, etc	Remarks
Water Vapor		Heat	Used, but not for lacquered or pitched vats	Used here	Spoils the rubber	Used, but hot water is more often employed		
Chalk	Solid white blocks	CaO					Well suited for white-washing	Active and cheap valued after CaO content
Caustic Soda	Solid; white hygroscopic solution used	NaOH	Used, together with soda ash	One to three per cent solutions used	Used in 3 to 5 per cent solutions	2 to 3 per cent solution after acid treatment		Wood is attacked by concentrated solutions
Soda Ash	Crystals or white powder	Na ₂ CO ₂	Dilute solution together with caustic soda	Five to 10 per cent solutions used	Three to 5.0 per cent solutions	One to 5.0 per cent solution. Not very good when used alone.		When crystals are used, 60% water of crystallization must be taken into consideration
Calcium Bisulphite	Solution containing 5.0 to 8.0 per cent SO ₂	H ₂ SO ₂	Stock solution used diluted 5 times	Stock solution diluted 5 times	May be used diluted 5 times		May be mixed with milk of lime for white- washing	Bad keeping properties evaluated after content of SO ₂
Formalin	Polymerized form is solid; 40 per cent solution	НСНО	One to 2.0 per cent solution. Specially active with steam	Two per cent solution			0.5 per cent solution for moistening of floors	Strong after washing with water to get rid of remains
Carbolic Acid	Crystals or solution	C ₆ H ₅ OH						Not to be used in distillery or brewery

RESEARCH BULLETIN - STUDIES ON RUM

TABLE NO. 47 - Continued

DISINFECTANTS AND THEIR USE

Name of Substance	Physical State	Active Element	Cleaning of Wooden Containers	Cleaning of Metal Containers	Cleaning of Rubber Tubing	Cleaning of Flasks	Cleaning of Floors, Walls, etc	Remarks
Fluorides	Small crystals	HF	0.5 per cent solution with all types of containers	0.5 per cent solution	0.5 per cent solution			Attacks glass and metals. Keep in wooden or ebonite containers
Hypochlorites	White powder	Cl	3 ½ kilograms per 100 liters of hot water	No good			3 ¹ / ₂ kilograms per 100 liters of hot water. Brushing needed.	
Permanganates	Violet brown crystals	KMnO4	0.5 to 0.5 per cent solution		5.0 per cent solution			Oxydizing deodorizing
Oil of Vitriol	Thick liquid	H ₂ SO ₄	To very dirty containers 10.0 per cent solution	Diluted 20 times for polishing				Dangerous in mixing with water; use stoneware
Antiformin	Concentrated solution	Cl & OH	Five per cent solution	Five per cent solution	Five per cent solution	Two and ½ per cent solution	One to 2 kgs mixed with milk of lime in 100 liters of water	Good disinfectant; dissolves fat
Montanin	Liquid	H_2SiF_6	Five per cent solution	Fiver per cent solution	Fiver per cent solution		Twenty per cent solution	

During distillation, continuous samples should be taken for each shift of all waters used for condensation or refrigeration, as well as of the discharged slops. At the end of each shift these samples should be analyzed for determination of alcohol. Troubles of a mechanical nature in the still's condensers, refrigerating system for the product, etc. etc. may be check through the performance of these tests. Also immoderate losses of alcohol in the slops are detected separately for each shift. High loss of alcohol in the discarded slops may become of serious consequences to the economy of the process. These losses may be due to various different causes; as for instance, accidents during distillation, unsteady or very variable pressure of the incoming steam, poor regulation of the feed of beer, mechanical troubles within the stripping column, bad design of the still, or through carelessness of the still operator. For well-designed stills, under normal conditions of performance, the loss of alcohol in the slops should not go beyond the range 0.05-0.15 per cent by volume. During our consulting experience we have seen cases in which 10 to 15 per cent of all the alcohol produced during fermentation was being lost with the slops. Here we had a case where poor yields were being obtained consistently, and due to lack of technical control, the loss had never been detected. The use of a control chemist at this particular rum distillery would have saved the loss of over 100,000 thousand proof gallons of alcohol per year or about \$40,000. And yet the management thought that the company could not go into the extravagance of hiring a control chemist at about \$3,000 a year.

The raw distillate produced daily should be carefully analyzed for determination of: total acidity, aldehydes, esters, higher alcohols, and alcohol by volume. The determination of total acidity is of paramount importance as a means of process control. Whenever this titratable acidity offers a sudden rise in value without the distillation proof having been lowered, we may better get ready for trouble in the fermentation department. This is a sure sign of infection as the fermenting end of the distillery, and the sooner the focus is found and steps are taken for disinfection, the better off will everyone concerned be.

Besides these chemical tests, the regular physical and organoleptic tests already alluded to during the writing of this bulletin should also be performed; and when the quantity and quality of the technical staff so permits, the above tests should be supplemented by a fractional distillation conducted with the help of the "birectifier" or other similar and equally efficient fractionating unit.

The data of all these different tests are recorded together with the serial numbers of the first and last barrel filled with this particular distillate for further use and comparison during the aging period.

During the maturing of the rum in the aging room, the changes both organoleptic and chemical that take place should be followed and recorded. This may be done with particular success by drawing a composite sample from the barrels and after performing the customary chemical analysis and physical and organoleptic tests; conducting a fractional distillation test as already described. This should be done at least twice during the year, that is, at periods of six months; but whenever possible, four of these tests should be performed during the year, at intervals of 3 months.

As stated before, in our own studies we were unable to execute this work with every sample of rum we manufactured in our pilot plant; but we did so in a few cases.

Figs. 33, 34 and 35 show the successive fractional distillations performed on a molasses rum during the first year of aging. Fig. 33 represents the fractionation of the raw rum before it was stored in the barrel; fig. 34 represents the same rum after having aged for six months; and fig. 35 represents the one year old product.

Figs. 36, 37 and 38, represent the same conditions as described above; but in the case of a defecated and clarified sugar cane juice rum.

Let us now consider the matter of calculations and reports for the rum distillery. The calculations that will follow will not vary much in different distilleries, but as to methods of reporting the results of manufacture and process control, there will be found great variations. We are enclosing report forms that may be used to advantage either as such, or modified to suit specific conditions at different distilleries.

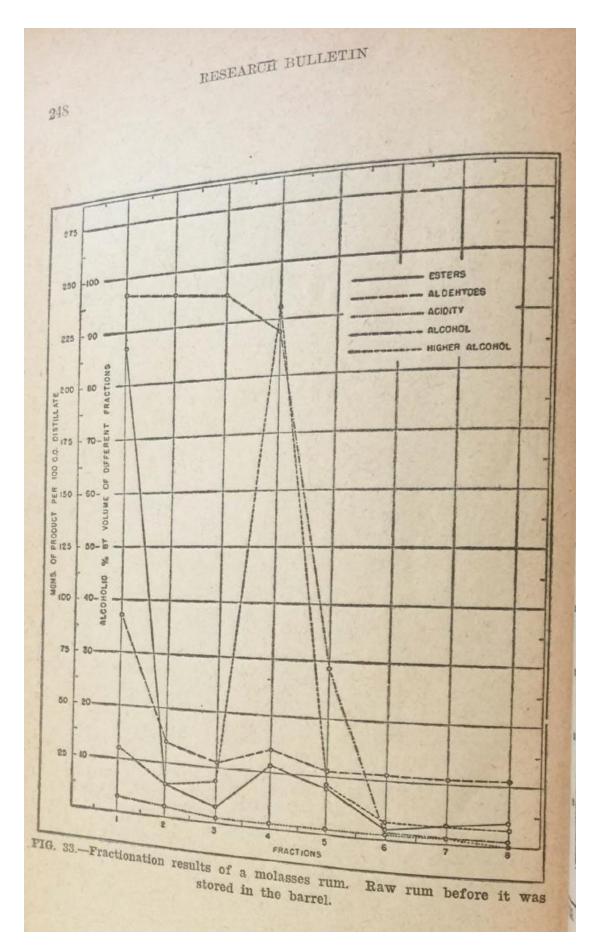
Certain old established methods used by managers and owners of distilleries bring confusion when trying to compare the work, methods, yields and general manufacturing results of different distilleries. Definitions of concepts of common understanding and acceptance, and some standard practice in reporting results are imperative if relative progress in manufacturing methods is to be judged from the reports of these distilleries. For instance, the common term used in expressing yields related to proof gallons of rum per gallon of molasses. From a scientific standpoint this definition of yields means nothing. The gallon of molasses varies in composition within very wide margins. For instance, and to mention only the most important component, the pounds of total sugars in a gallon of blackstrap molasses may vary between 5.50 and 6.50, depending on the density and percentage of total sugars in the material. The alcohol being produced by the sugars, we may readily see that the gallon of molasses containing more pounds of sugar must yield more rum than the one containing less, under otherwise equal conditions of manufacture. But the distillery using the inferior quality molasses may be actually utilizing the sugars in this material to greater advantage and efficiency than the other, and yet, in the expression for yield it will appear as doing inferior work.

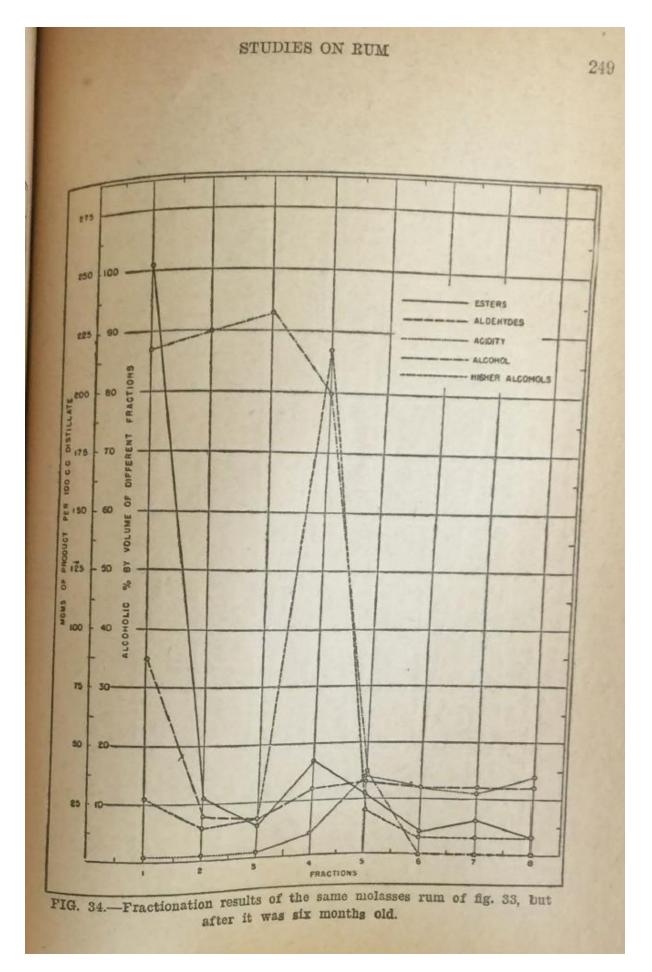
Two expressions for yields may be used, and should be used exclusively when trying to do comparative inspection or study among the work carried at different distilleries. There are: (1) pounds of alcohol produced per pound of sugar entering the process; or (2) proof gallons of alcohol or rum, per pound of total sugars entering the process. Expressed in either of these two forms, the term acquires real and valuable significance in comparative work. The old expression of yields in terms of proof gallons of rum per gallon of molasses was the natural outcome of the lack of technical control at the distillery. The only records available were those of gallons of molasses used, and proof gallons of rum produced.

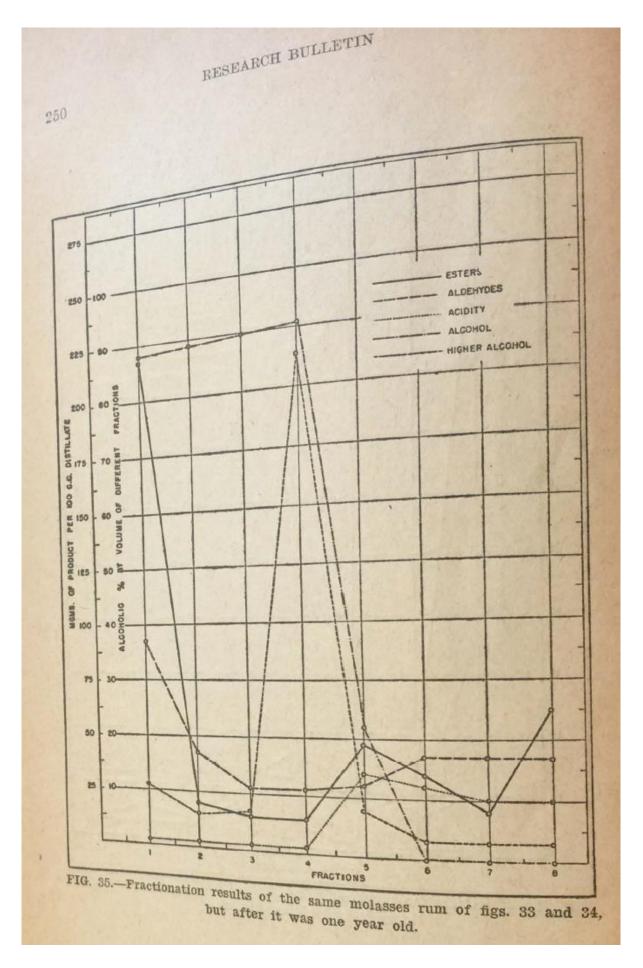
The present method of buying and selling molasses for distillery use, also has its disturbing influence on comparative distillery work, and difficulties of standardization of reports. Moreover, it seriously affect the economy of the distillery and the true value of the reported gains or losses in some instances. It is high time that blackstrap molasses be bought and sold for its content of total sugars and the ratio existing between these total sugars and total non-sugars. Our studies have shown this to be the only fair and equitable basis both for the seller and the purchaser.

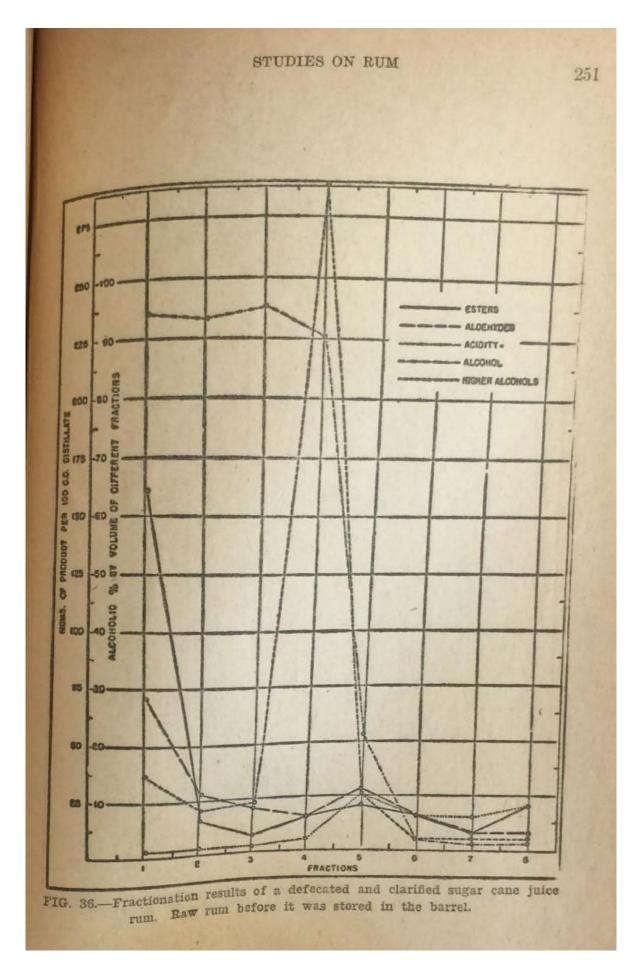
Calculations begin with the analysis and weight of the raw material entering the distillery for the day's work. From its weight and percentage of total sugars by weight, the number of pounds of sugars entering the process becomes known.

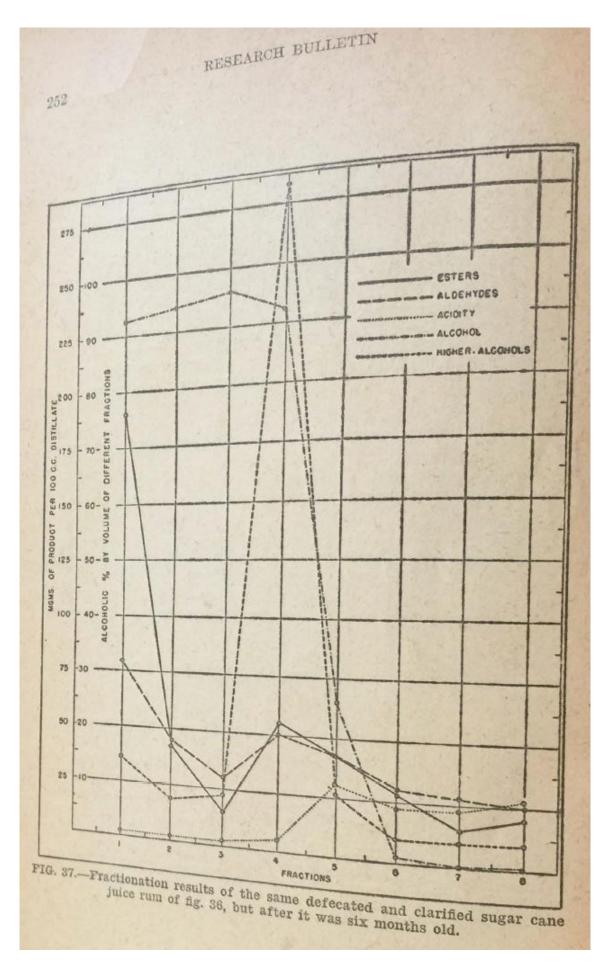
From the mash analysis are found the grams of total sugars per 100 ml, the setting degree Brix, pH value, and titratable acidity. For purposes of calculations the degree Brix and the grams of total sugars per 100 ml mash are useful for later on, after the fermentation

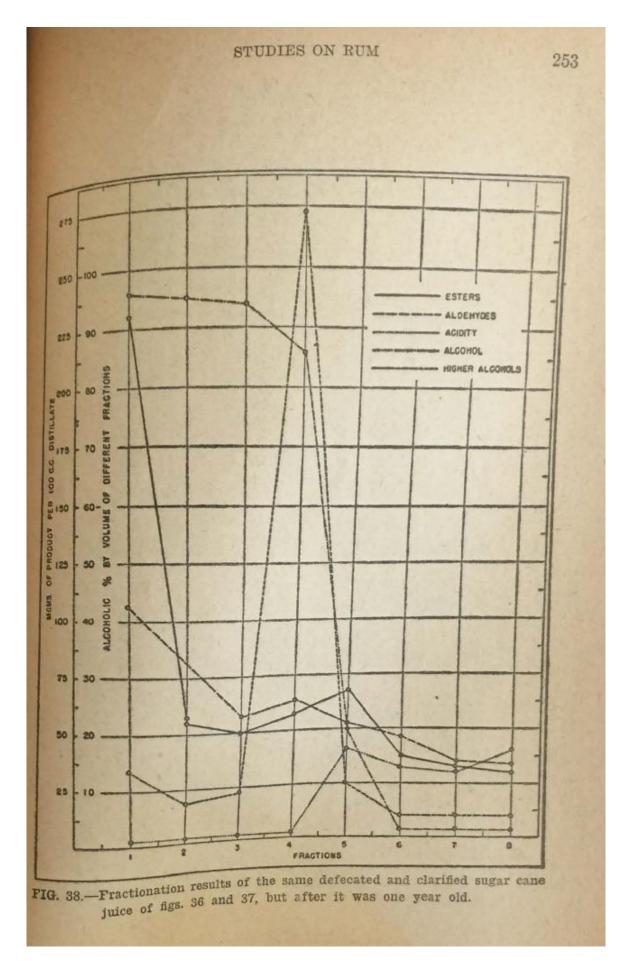












DAILY DISTILLERY RECORD

DATE:

- 1. MOLASSES:
 - (1) Gallons
 - (2) Brix
 - (3) Total Sugars, % by Weight
 - (4) Total Weight, Lbs.
 - (5) Weight per Gallon, Lbs.
 - (6) Total Sugars, Lbs
 - (7) Quality Coefficient
- II. MATERIALS:
 - (1) Ammonium Sulphate, Lbs.
 - (2) Calcium Superphosphate, Lbs
 - (3) Sulphuric Acid, Gallons
- III. MASHING:
 - (1) Fermenters used, Numbers
 - (2) Total Volume, Gallons
 - (3) Setting Brix
 - (4) Total Sugars, grams/100mL
 - (5) Total Sugars, Lbs.
 - (6) Total Sugars, Lbs/Gallon
 - (7) pH Value
- IV. BEER:
 - (1) Final Brix
 - (2) Residual Sugars, grams/100mL
 - (3) Residual Sugars, Lbs.
 - (4) Alcohol by Volume, %
 - (5) Alcohol, grams/100mL
 - (6) Total Alcohol, Proof-Gallons
 - (7) Total Alcohol, Lbs.

V. FERMENTATION RESULTS:

REPORT NO:

- (1) Attenuation
- (2) Sugars fermented, % Total Sugars
- (3) Alcohol yield, % Total Sugars
- (4) Fermentation Efficiency, %
- (5) Proof-Gallons, per 100 gal beer

VI. DISTILLATION:

- (1) Hours
- (2) Type of Distillate
- (3) Total Wine Gallons
- (4) Average Proof
- (5) Total Proof Gallons
- (6) Alcohol, Gallons
- (7) Alcohol, Lbs
- (8) Distillation Efficiency, %

VII. YIELDS:

- (1) Lbs Alcohol/100 Lbs. Sugars
- (2) Proof Gallons Alcohol/Lb. Sugar
- (3) Proof Gallons Alcohol per day
- (4) Proof Gallons Alcohol per hour
- (5) Proof Gallons per gal fuel oil used
- (6) Over all Distillery Efficiency

VIII. LOSSES OF ALCOHOL:

- (1) During Fermentation, Lbs.
- (2) During Distillation, Lbs

DATE		
BEFORT No.	For the Week	To Date
I MOLASSES: (1) Gallons.		*******
(1) Galication (2) Brix (3) Weight, Ibs		
Total Sugars, per cent by Weight.		
(5) Total Sugars, Ibs	*** ****************	*******
II. MASH:		
(1) Gallons (2) Brix		******************
(3) Total Sugars, grams /100 ml.		
(4) Total Sugar, Ibs		
III. BRER:	1.58	1
(1) Alcohol, per cent by volume. (2) Total Alcohol, Gallons		
(3) Total Alcohol, lbs		
(4) Residual Sugars, Ibs		
IV. FERMENTATION RESULTS:		
(1) Lbs. Alcohol per 100 lbs. Total Sugars.		
 (2) Proof Gals. Alcohol per lb. Total Sugars		******************
(4) Sugars Fermented per cent Total Sugars		
(5) Fermentation Efficiency, per cent		
V. DISTILLATION:	the state of the	
(1) Effective Hours. (2) Wine Gallons distilled		
(3) Total Proof Gallons made and estimated		
(4) Gallons 200 Proof Alcohol. (5) Lbs. 200 Proof Alcohol.		
(6) Hourly rate of production, Proof Gallons.(7) Rate of beer feed, Gallons per hour.		
(8) Distillation Efficiency, per cent.		
I. YIELDS:		
(1) Production per dev Proof Gallons		
(2) Production per effective day, Proof Gallons		
Caller		
 (4) Lbs. Alcohol /100 lbs. Total Sugars. (5) Proof Gallons. Alcohol /100 lbs. Total Sugars. 		
(6) Over all Distillery Efficiency, per cent		
II LOSSES:	19 Artes	
(1) Lbs. of sugars unconverted to alconol.		
 (3) Lbs. of alcohol lost during distingtion (4) Total lbs. alcohol lost. (5) Total Proof Gallons. alcohol lost. 		
(5) Total Floor Canons MARKS:		
RANAS MINING		

is finished. The analysis of the beer, or fermented mash, provides us with additional data used in calculation of results, such as final degree Brix, residual sugars in grams per 100 ml beer, and alcohol percentage by volume. The alcohol percentage by volume is easily reduced to its equivalent in grams per 100 ml beer by consulting the alcohol tables.

With the data mentioned above, we proceed to calculate the degree of attenuation; the percentage sugars used on total sugars during fermentation; the yield of alcohol on total sugars by weight; the yield of alcohol in beer in total proof gallons, and in proof gallons per pounds of sugar; and the percentage of fermentation efficiency. It is customary to express the fermentation efficiency in absolute, or in relative terms. When expressed in absolute terms the equation of Gay Lussac is used as basis for the maximum possible yield that may be expected, or 51.11 per cent alcohol on weight of sugars; but most commonly, the Pasteur's equation is used in which only 95.0 per cent of 51.11 is considered as the highest possible yield to be obtained in distillery practice, or 48.55 per cent. In the calculations used in this chapter we refer to the Pasteur's fermentation efficiencies, and use, therefore, the factor 48.55.

- (1) Attenuation: Degree Brix at setting final degree Brix.
- (2) Percentage total sugars fermented, on total sugars used: (Grams total sugars in 100 ml mash grams residual sugars in 100 ml of beer) divided by grams total sugars in 100 ml mash, and multiplied by 100.
 Example: Grams total sugars in 100 ml mash = 16.50 Grams residual sugars in 100 ml beer = 1.25

Then, percentage total sugars fermented on total sugars used: (16.50-1.25) where α and α

$$\left(\frac{10.50^{-1.25}}{16.50}\right)X\ 100 = 93.84$$

(3) Yield of absolute alcohol on weight of total sugars: (Grams alcohol in 100 ml beer divided by grams sugars in 100 ml mash) X 100 *Example:* Grams alcohol in 100 ml beer = 8.00

Grams total sugars in 100 ml mash = 17.00 Then, % yield = $\left(\frac{8.00}{17.00}\right)X$ 100, or 47.06

(4) Fermentation Efficiency: (Obtained yield divided by 48.55) X 100 *Example:* Obtained yield = 47.06%; then % Fermentation Efficiency = $\left(\frac{96.93 \times 95.00}{100}\right) = 92.08\%$ (5) Total proof gallons of alcohol in beer: (Number of gallons of beer X % Alcohol by volume in beer X 2) divided by 100. *Example:* Number of gallons of beer = 50,000 % alcohol by volume in beer = 10.00 Then, total proof gallons of alcohol = $\left(\frac{50,000 \times 10.0}{100}\right) \times 2$, or 10,000.

Knowing the amount of proof gallons of rum or alcohol in the beer, we may find what percentage of these are actually recovered after the beer has been distilled. The amount recovered is actually weighted and measured. The distillation efficiency is then calculated, thus:

(6) Distillation Efficiency: (Proof gallons of alcohol actually recovered divided by proof gallons alcohol in beer) X 100 *Example:* Proof gallons alcohol recovered = 9,500 Proof gallons alcohol in beer = 10,000 Then, % distillation efficiency = $\left(\frac{9,500}{10,000}\right) X$ 100, or 95.00%

From the fermentation and distillation efficiencies, the "overall" distillery efficiency is calculated thus:

(7) Over All Distillery Efficiency: (% Fermentation Efficiency X % Distillation Efficiency) divided by 100
 Example: % Fermentation Efficiency = 96.93

w Permentation Efficiency = 96.93
% Distillation Efficiency = 95.00
Then, Over All Efficiency =
$$\left(\frac{96.93 \times 95.00}{100}\right) = 92.08\%$$

Other calculation required are those pertaining to yields. As stated before, these should be reported in terms of pounds of alcohol per pound of sugar used, or in proof gallons per pound of sugar. Since the number of pounds of sugars entering the process are known from the analysis and weight of raw material, and also known from the analysis and weight of raw material, and also known from the in their weight are known, it becomes a very simple matter to calculate the yields.

Before closing this chapter of our bulletin on Rum Studies, it would be well to say a few words in regard to research in the rum distillery. Those rum companies that can afford the extra expense could not make a better investment than in this most important phase of rum production. Most of my readers will smile at the idea of asking for investment in research in rum production when even the control laboratory is lacking, or is quite inadequate, in most distilleries. We are nevertheless making a plea for the introduction of research of the most intensive kind in our rum industry. It will be only by and through research, that the hold we have acquired on the American market may be maintained and extended. If there is an industry in need of research, it is the rum industry; and none is in better, or more favorable conditions to endow its workers with richest rewards. The field is practically virgin; what little has been accomplished by these studies has served, more than anything else, to open our eyes to the hidden treasures and magnificent rewards the industry keeps for those who will in the future investigate the many possibilities existing. Research that will cheapen or expedite production: but above all, research that will improve the quality, so that both producers and consumers may be proud of our Puerto Rican rums.

The plea is placed on both before our government, and before the Association of Distillers and Rectifiers, for they are of all Puerto Ricans, those receiving, the greatest benefits from the industry. It is important that in the future our rums be ready to compete in the United States markets on the basis of sheer quality and not because of tariff protection. It is necessary that even after paying higher wages than those prevailing at present in the industry, we shall be able to compete with rums produced in countries where labor is not equally rewarded, by greater efficiency and rapidity in production. We must be able and ready to reduce costs of production through means of higher technique and more scientific methods; not by a reduction of wages to our laborers and skill workers.

For all of this to be accomplished with the minimum derangement and trouble for the industry, there is only one way, and that way is *research*.

SUMMARY

These studies on rum and its manufacture were initiated some 9 years ago, foreseeing the marvelous development that has actually occurred in the rum industry of Puerto Rico.

Among the pertinent conclusions and results derived from this study, the following may be mentioned:

(1) The selection of the adequate yeast for the production of the desired type of rum is of vital importance to the success of the distillery.

(2) The most important raw material used in rum making, viz., final, or blackstrap molasses, usually presents defects of chemical constitution that interfere either with the economy and yields of the process or with the quality of the finished product. Methods were developed to obviate wholly, or in part, these inherent shortcomings of the raw material.

(3) In the course of rum fermentation studies, phenomena were observed that could only be explained on the basis of mitogenetic radiation. We were thus led to delve into the subject of Gurwitsch's assertion; and in the course of the study, developed added proof sustaining his claim of mitogenetic radiation.

(4) Important changes and methods of rum fermentation were developed that are a great improvement over the empirical, obsolete methods, generally followed previous to our researches. New types of rums were created; and the famous types of Jamaica export heavy rums were duplicated in such way that no distinction was apparent between the Puerto Rican and the Jamaican products. European experts on Jamaica rums of these types, who tested the products of our research, have declared them comparable to the best produced in Jamaica.

The development of these types of rum, so popular in the European markets, may mean much in an economic way to our Island; since that market, the greatest in the world, has not even been touched by our rum industry. The fact that we can produce faster, with better yields, and superior efficiency, should weigh heavily in the acquisition of a place in the above-mentioned markets.

(5) The results obtained during our distillation studies have greatly improved the commercial practice of rum distillation, and the raw distilled at present are of a far better quality than those produced seven or eight years ago. Our distillers had fallen into the fallacy of trying to transform industrial alcohol into rum of quality. We have shown that industrial alcohol, and rum distillation, are as different as day from night.

(6) What we have done on the subject of rum curing and maturing has served mostly to impress upon ourselves the vastness of what may be done along these lines in the future. We have merely scratched the surface of the subject, and even so, the rewards of our labor have been abundant.

We sincerely hope that intensive work along this phase of rum manufacture will be reopened at once either by this or by similar governmental institutions, or possibly better, by the Association of Rum Producers of Puerto Rico.

The outstanding fact learned in our work was that natural rum curing in oak barrels could be reduced to but a fraction of the time now taken for its accomplishment if more pains and greater care were taken by producers in the:

(a) Quality of the raw distillate with which the aging barrel is filled.

(*b*) Quality of the barrel itself.

(c) Design of curing room.

(*d*) Proper control of temperature and relative humidity conditions during the aging period.

Of all these different factors entering into the question of rum curing and maturing, the predominant one is the first mentioned.

The methods of fermentation and distillation used at the distillery will exert a very great influence upon the curing stage.

(7) The aroma of genuine rums will depend entirely on the amount and quality of their Non-Alcohol-Number. Our studies have demonstrated, however, that the Non-Alcohol-Numbers of different rums are quite different in amounts as well as in the individual classifications of its components. We have found also, that quality of individual component is far more important than quantity; and, that the relations existing among different group components create a sort of harmonious estate or condition mainly responsible for the compound and complex effect of rum aroma. It was also found that of all the constituents entering into the Non-Alcohol-Number, a certain oil or mixture of essential oils, to which we have given the generic name of "rum oil", forms the basis on the genuine rum aroma. The production of this rum oil during fermentation, and its preservation during distillation of the product should be very carefully attended to by the wide-awake producer. Practically all rum yeasts produce this oil during fermentation but the extent in which it is produced varies from yeast to yeast; and is influenced, besides, by the conditions of the substrate.

(8) A system of rum classification, differentiation, and appraisal was developed during our research. The system is far from perfect; but it represents a worthy attempt towards standardization of products and detection of fraud.

(9) Two features of outstanding value have resulted from these studies that are intimately connected with each other:

(1) The recognition of the paramount influence of "rum oil" in rum manufacture and quality; and (2) the introduction of the "birectifier" in rum research and process control in Puerto Rico. We could not have detected, or at least might have taken a much longer time in detecting, the presence of the invaluable "rum oil" as a member of the Non-Alcohol-Number of our experimental rums had it not been for the fact that the "birectifier" was acquired for this work.

As a matter of fact, the "birectifier" of Dr. Luckow became the most important and valuable tool in our research. Later, we realized its equal, or greater value, for distillery control work. This inexpensive fractional distillation unit was instrumental to our success in the detection of "rum oil"; in the work on yeast selection; and above all, during the curing period; and in the attempt to establish a system for rum differentiation and appraisal. Our only regret is that we lacked the time and personnel to apply in a more intensive and extensive way this unique tool in our studies. We believe this little laboratory apparatus will acquire ever increasing use not only in research, but in control laboratories, as well.

(10) The lack of technical control in our rum distilleries at the initiation of our studies was simply appalling. We have endeavored to imbue into the minds of local distillers the necessity of such control, for the benefit of all concerned; but *very especially* for their own. Instructions are given in the last chapter of this bulletin on how this control should be carried. This does not mean that our directions should be followed to the letter in all cases but the ideas and methods presented may serve as the basis for the preparation of specific programs of control at the different distilleries now lacking them, according to individual ideas, necessities and conditions of work.

Resumen

De acuerdo con los reglamentos de la Institución a que pertenemos, se hizo necesaria la publicación de este boletín en el idioma inglés. Hubiéramos deseado hacer una doble publicación en los respectivos idiomas: español e inglés; mas debido a la duplicación de gastos editoriales no nos fue posible cristalizar esa idea.

Sin embargo, para beneficio de los muchos países productores de ron, en que predomina nuestra hermosa lengua castellana, y a donde es probable llegue este boletín, hemos decidido hacer este resumen lo más extenso posible dentro de las limitaciones ya expresadas. Creemos firmemente que al hacer esto, al mismo tiempo estamos extendiendo el radio de acción y uso de este boletín en nuestro propio país, donde indudablemente también predomina el hablar hispano.

Se iniciaron estos estudios poco después de la derogación de la ley de prohibición en Estados Unidos y Puerto Rico, y cuando la nueva industria había dado incipientes pasos que habrían de traer muy rápidamente el enorme y deslumbrante desarrollo de todo conocido. Ese desenvolvimiento fue previsto por nosotros y nos complacemos en habler contribuido con nuestro humilde óbolo a su éxito.

Durante el total transcurso de este trabajo, ha sido nuestro principal objeto corregir en cuanto posible fuera, ideas y métodos erróneos existentes en la manufactura de esta bebida, librándola de este modo del dispendioso empirismo, y dirigiéndola por nuevos cauces basados en la moderna técnica experimental y el esfuerzo científico.

Los resultados y conclusiones más importantes nacidos de estos estudios los presentamos a continuación.

(1) En Puerto Rico la materia prima predominante en manufactura de ron la constituye las mieles finales de nuestros ingenios azucareros. Pronto pudimos observar que esta materia prima adolecía de defectos en su estructura química y física, los cuales redundaban en detrimento de la economía del proceso de manufactura, o de la calidad del producto. Conseguimos aminorar grandemente estos defectos inherentes a la materia prima mediante el desarrollo de un proceso de purificación nada costoso y de sencilla aplicación. Por medio de este proceso se convertía una miel francamente de inferior calidad, en una de mediana aceptación; y otra de calidad media en una de primera clase. Encontramos las relaciones que deben existir entre los azucares y los no azucares constituyentes de la miel para su mejor y más eficiente empleo en la industria del ron. De este modo, se aumentaba grandemente el rendimiento posible y nos acercábamos más a la eficiencia teórica en fermentación alcohólica. Todo lo cual venía a redundar en el provecho económico y bienestar destilerías nos facilitaba los medios para ejecutar nuestras ideas en gran escala y hacer las modificaciones pertinentes, y casi siempre necesarias, al operar en escala comercial. Esto nos ha permitido vaciar en este boletín no solamente resultados y teorías de laboratorio experimental, sino también deducciones y métodos que han pasado por el crisol de la práctica comercial y de la técnica aplicada en gran escala. Hemos podido comprobar también la falacia de la supuesta superioridad del ron de guarapo o jugo de cana, sobre el que se origina de mieles finales. No solamente es infundada la tal superioridad del jugo de la caña sobre las

mieles, sino que los rones de los derivados son de más difícil confección y de mucha más lenta maduración durante el periodo de añejamiento. Entre los rones de guarapo de cana, aquellos fabricados del guarapo crudo tardan mucho más en llegar al estado de madurez óptima que los fabricados de guarapo defecado y clarificado. Estos últimos son también de más fino "bouquet" y mejor gusto. Ahora, para la venta en su estado crudo - sin añejamiento - son preferibles los rones fabricados de guarapo crudo, por su particular degustación en ese estado.

(2) La selección de la estirpe de levadura apropiada para producir la clase de ron que deseamos fabricar es uno de los puntos más trascendentales para el destilador. En este sentido hemos tenido gran trabajo en conseguir la aceptación en la práctica de este concepto. Comúnmente existía la creencia que cualquier levadura productora de alcohol en cantidades aceptables era adecuada para fermentación de ron. Todas las levaduras no son adecuadas para la manufactura de ron. Aun entre aquellas que se usan apropiadamente, es necesario establecer distinciones y selección. La levadura ha de ser seleccionada de acuerdo con la clase de ron que nos propongamos manufacturar. Por ejemplo, la estirpe más adecuada para la producción del tipo de ron jamaiquino de exportación, estaría complemente mal elegida para la producción de los tipos livianos de Cuba y Puerto Rico. Hay que atender, además, a otros factores en la selección de la levadura; tales como el tiempo que emplea determinada estirpe en terminar la fermentación, su resistencia a altas concentraciones alcohólicas en el medio en que se emplea; los productos metabólicos que genera, además del alcohol etílico; su resistencia a altas temperaturas y variaciones de pH en el medio, resistencia a ciertas infecciones bacterianas muy corrientes en la sala de fermentación, etc. etc. Todas estas características deben ser estudiadas en el transcurso del tiempo dedicado a seleccionar nuestro fermento. Por otro lado, las características del fermento influyen en el diseño y equipo de la destilería, y esta debe ser construida y equipada después que la levadura haya sido seleccionada, pues de este modo la construcción y equipo quedara en consonancia harmónica con las cualidades y defectos inherentes al fermento, consiguiéndose de esto modo economía en diseño y construcción en unos casos, y siempre, mayor facilidad de producción y mejor calidad en el producto comercial. Es preferible adaptar el equipo al fermento que el fermento al equipo; pues si lo primero es siempre factible, no así lo segundo.

Sobre todo, se hace necesario al seleccionar nuestra levadura, estar seguros que esta es una buena productora del llamado "Aceite de ron". Esta es una substancia oleaginosa, producto del medio y la levadura, cuyo aroma forma la base del "bouquet" característico de los rones genuinos. Hemos encontrado que todas, o casi todas las levaduras de ron, producen este aceite o mezcla de aceites esenciales; pero en muy distintas cantidades, y de acuerdo con la clase de substrato en que se emplean, y métodos seguidos en su preparación.

(3) Es necesario que ciertos factores sean atendidos y ciertas necesidades llenadas en el momento de confeccionar la batición, o amasijo, donde la levadura ha de llevar a cabo las operaciones fermentativas. Por ejemplo, el grado de concentración de azucares totales expresado en gramos por cada 100 mililitros de batición, debe ser cuidadosamente ajustado de modo que no sobrepase los limites dentro de los cuales puede desenvolverse eficientemente nuestro fermento. De lo contrario sería dejada una gran cantidad de azucares sin conversión a alcohol; constituyendo dichos azucares una perdida innecesaria en los mostos de desperdicio. Es asimismo necesario tener la seguridad que el fermento ha de encontrar en el medio los alimentos necesario para suda vida, desarrollo y reproducción. El valor pH óptimo para la levadura en uso debe existir en la batición al ser está confeccionada, y debe, además, mantenerse durante todo el transcurso de la fermentación. El agua empleada en la dilución de la materia prima debe ser razonablemente pura desde el punto de vista de su composición química; y mucho más pura todavía desde el punto de vista de su flora microbiológica.

(4) En el transcurso de nuestros trabajos en fermentación del tipo de rones jamaiquinos, hubimos de observar ciertos fenómenos los cuales eran difíciles de explicar al principio. Por ejemplo, durante el trabajo teníamos ocasión de fermentar los medios con levadura pura o con cultivos de levadura y de bacterias bajo condiciones controladas. Notamos que en las fermentaciones simbióticas de levadura y bacteria los siguientes fenómenos se sucedían:

(1) El tiempo de fermentación disminuía notablemente llegando a ser menos de la mitad del tomado por la levadura cuando trabajaba sola en el medio.

(2) La multiplicación celular de la levadura aumentada siempre en grades proporciones, llegando a ser diez y veinte veces mayor que cuando la levadura trabajada en cultivo puro. (3) La concentración alcohólica del medio subía con gran rapidez, produciéndose casi todo el alcohol durante las primeras 24 horas de fermentación. Sin embargo, cuando la levadura trabajaba por si sola en un medio similar, la concentración alcohólica subía muy lentamente durante las primeras 24 horas.

(4) Aun cuando los dos organismos (levadura y bacteria) eran separadas por una pared de cuarzo, estos fenómenos persistían.

Todo esto nos hizo entrar en un estudio de la teoría de irradiación mitogenetica expuesta por Gurwitsch por primera vez en 1923. Este estudio confirmo nuestras sospechas que los fenómenos observados durante nuestras fermentaciones simbióticas tenían su origen en el fenómeno de Gurwitsch. Al mismo tiempo nuestros estudios aportaron pruebas adicionales confirmando la teoría de irradiación mitogenetica. Este ha sido uno de los problemas más discutidos, en pro y en contra, por el mundo científico desde que Alejandro Gurwitsch dio a la luz sus primeros experimentos y proclamo su teoría.

(5) Gran parte de nuestros estudios fue dedicada a la fase fermentativa de la manufactura de ron. En ella hemos conseguido algunos de los mejores frutos de nuestra labor y creemos haber ensanchado el horizonte futuro de nuestra gran industria licorera. Los métodos empíricos de fermentación, usados con anterioridad a estos estudios, han sido substituidos por lo menos parcialmente por métodos adelantados dentro de las normas de la técnica fermentativa moderna. Hemos podido señalar con anticipación la mayoría de los obstáculos que pueden presentarse al destilador durante esta importantísima fase de fabricación de ron y expuesto con claridad la diferencias que deben existir en las fermentaciones de ron, de aquellas destinadas a la producción de alcohol industrial; analizando uno por uno todos los factores conducentes a la obtención de resultados óptimos durante la fermentación del producto.

Nuevos métodos fermentativos nacidos de nuestros estudios y experimentación han culminado en la creación de tipos de rones nuevos, los cuales podrán abrirse paso en el futuro, en los hasta el presente inexplorados mercados europeos. Los rones de tipos de exportación jamaiquinos, que tanta aceptación y fama han ganado en esos mercados, han sido duplicados con éxito y aun mejorados en el transcurso de nuestros experimentos. En cuanto a la técnica seguida en la producción de estos tipos de ron, supera grandemente los métodos empíricos seguidos en la gran mayoría de los casos en la Isla vecina; resultando de todo esto que los rones de tipo jamaiquinos pueden ser manufacturados por nuestros métodos con:

- (1) Mayores rendimientos.
- (2) En una fracción del tiempo empleado en Jamaica.
- (3) Con igual, si no superior calidad.
- (4) A un coste mucho más económico.

(6) En la etapa destilatoria de la manufactura de ron hemos podido demostrar las diferencias en método a seguirse de acuerdo con el tipo y calidad de ron que desee manufacturarse. Hemos demostrado la íntima relación que existe entre esta fase del proceso y aquellas que le anteceden, muy especialmente en cuanto al tratamiento predestilatorio de los mostos fermentados. El alambique debe ser seleccionado de acuerdo con la clase de ron que se desee fabricar. Por ejemplo, para rones de los llamados del tipo liviano, los de destilación continua son superiores, siempre que cuenten en su diseño con los ajustes necesarios para ser usados específicamente en la producción de ron. Entre esta clase de aparatos destilatorios, aquellos que cuentan con la columna llamada de pasteurización son los más eficientes de los hasta ahora usados en Puerto Rico.

En cambio, si se desea trabajar el tipo de ron "pesado" como los de exportación de Jamaica, no hay duda que el aparato de destilación discontinua es muy superior. Esto no significa, sin embargo, que no sea posible obtener rones inmejorables del tipo "liviano" en un alambique de destilación discontinua. En todos los casos han de influir grandemente los conocimientos, experiencia y pericia del destilador encargado del proceso.

Si las etapas de manufactura de ron, previas a aquella de la destilación del productor, influyen grandemente en esta última, es no menos verdad que la influencia de la fase destilatoria está íntimamente relacionada con los resultados que hemos de obtener luego durante la rectificación y maduración del producto crudo. Y aun con cuestiones de capacidad y equipo del almacén de curación, o sala de "añejamiento", como suele llamársele.

Hemos expuesto los peligros inherentes a la destilación de ron bajo técnica exagerada de alta prueba y rectificación; explicando cómo y de qué manera se desvirtúa la misma naturaleza del producto que corre la calidad del ron crudo en el proceso de dilución para rebajar su concentración alcohólica antes de ser envasado en las pipas de añejamiento. Insistimos que todo método o técnica destilatoria que tienda a inhibir la presencia de cantidades adecuadas de "aceite de ron" en el destilado crudo, ya lleva consigo la razón de su fracaso.

De la calidad del crudo depende, como factor principal, el tiempo que ha de ser necesario durante el periodo de curación para impartir a este las características deseables de un ron madura. Y con esto están relacionados, desde luego, tamaño de los almacenes de curación, numero de pipas necesarias, gastos de personal, etc etc. Vemos, pues, la gran importancia de la fase destilatoria en fabricación de ron.

(7) Aunque hubiésemos dedicado todo el tiempo prestado a estas investigaciones, exclusivamente a la fase de la maduración del producto crudo, no hubiésemos ni remotamente agotado las grandes posibilidades en ella existentes. Pero lo poquísimo efectuado ha servido para abrir nuestro entendimiento, con mayor conocimiento de causa, a la mucho que se puede, y se debe hacer, en esta etapa de la elaboración del ron. Empezamos, pues, por admitir que si hemos hecho *muy poco* en este sentido, en cambio hemos *aprendido* como hacer *mucho* si en el futuro se nos ofreciese una nueva oportunidad. Vamos más lejos, para pedir al señor Director de esta Institución que abogue con las autoridades pertinentes para reabrir este capito de nuestras investigaciones en un futuro inmediato o cercano, pues tenemos nuestro plan de ataque bien pensado, y entusiasmo para llevar la obra a un completo éxito no nos falta.

Durante el trabajo ya efectuado hemos podido darnos cuenta de la importancia de ciertos factores en la curación del curdo, y de los efectos beneficiosos a la industria que podrían derivarse de la aplicación de ostros que hasta la fecha no han sido debidamente estudiados, o permanecen huérfanos de aplicación experimental. Como varias veces hemos repetido, la idea, el plan a desarrollar, esta lucido en nuestra mente; pero hemos carecido de tiempo, personal, y equipo necesarios.

De los datos, ya comprobados durante el trabajo efectuado hemos sacado las siguientes deducciones:

(1) No es necesario recurrir a los métodos de curación artificial para obtener los resultados en economía de tiempo y gastos: únicos incentivos en la práctica de tales métodos.

(2) El factor primordial en la aceleración natural de la madurez en un destilado crudo, está representado en la calidad del mismo. Con esto queremos decir que el ron que es malo al nacer, esto es, como crudo; seguirá siéndolo siempre, en menor

intensidad tal vez; pero malo siempre, a pesar de los anos de añejamiento. Por el contrario, aquel que posee cualidades de estructura química y de características físicas adecuadas, adquirirá rápidamente las condiciones de degustación y aroma que diferencian un ron maduro de uno crudo. Por lo tanto, no es cuanto tiempo, sino como es el ron fresco capaz de aprovechar ese tiempo de curación, lo que cuenta en mayor grado.

(3) Factores accesorios a la adquisición de este desarrollo natural de madurez en el crudo los constituyen:

(a) Calidad, clase y tamaño de la pipa de curación.

(b) Tratamiento dado a esas pipas antes de efectuar el envase del ron crudo.

(c) Diseño y construcción del almacén para envejecimiento.

(d) Equipo de ese almacén para efectuar cambios necesarios en temperatura y en el grado de humedad relativa de su ambiente interno, así como renovación y circulación de aire fresco exterior.

(e) Uso eficiente y científico de los factores arriba mencionados.

(f) Establecimiento de métodos de control del periodo curativo, capaces de avisar el momento preciso en que el ron bajo curación ha adquirido completo estado de madurez.

(8) El aroma de un ron genuino depende enteramente de la cantidad y calidad de su coeficiente No-Alcohol. Por este coeficiente se entiende el conjunto de cuerpos aromáticos existentes en todo ron genuino que son derivados de los compuestos congenéricos formados durante la fermentación y destilación del producto; y de otros que se forman durante el proceso de curación. La calidad aromática de los constituyentes de este Coeficiente No-Alcohol es mucho más importante que la cantidad de los mismos. Por esto se hace necesario tratar de desarrollar durante la fermentación y destilación del producto, aquellos compuestos químicos capaces de prestar esa nota de calidad al "bouquet" del ron.

Encontramos que entre todos los constituyentes del aroma del ron, el aceite o mescla de aceites esenciales a que hemos dado el nombre genérico de "Aceite de Ron", se destaca como principal ingrediente y base fundamental del "bouquet" de un ron genuino. Se hace, por lo tanto, necesario tratar de obtener levaduras que al reaccionar con el medio en que se desenvuelven sus actividades fermentivas, produzcan en la mayor cantidad posible este valioso aceite.

El aroma del crudo será modificado grandemente durante el periodo curativo. La pericia y habilidad desplegada en el uso de los diferentes factores que entra en juego durante esta fase de la producción del ron comercial, decidirán el carácter, sello distintivo, y calidad general del aroma de este.

(9) En el transcurso de estos estudios hemos formulado un sistema para la clasificación, diferenciación y justipreciación de los rones, basado principalmente en la destilación fraccionada de los mismos, en combinación con pruebas físicas, químicas y organolépticas. Aunque no reclamamos una eficiencia absoluta, ni infalibilidad en nuestro método, creemos, sin embargo, que representa un esfuerzo de valor, y genuino, en la justipreciación y clasificación de los rones comerciales. La introducción del birectificador del Dr. Luckow, de la División de Aguardientes y Licores del Instituto de Fermentologia de Berlín, en estos estudios ha sido valiosísima; pues luego hemos podido persuadirnos de la importancia tremenda que este sencillo aparato tiene para el investigador en manufactura de ron y también para ser usado por el destilador practico en el control técnico de la destilería. A nosotros este aparatito nos ha sido de incalculable valor en el trabajo de selección de levaduras apropiadas para fermentación de ron; durante el periodo de curación de los destilados crudos, y muy principalmente en el establecimiento de un sistema de justipreciación y clasificación de los rones comerciales. Opinamos que el birectificador será usado cada vez más extensamente y con mayor provecho en nuestra industria licorera.

(10) Al tiempo de dar comienzo a nuestras investigaciones, el estado de la industria era verdaderamente lamentable en cuanto a la falta de supervisión y control técnico en la destilería. La industria presentaba en ese sentido el mismo aspecto que el de la industria de la caña de azúcar hace 50 anos.

Tratamos desde el principio de establecer normas de control técnico, tanto químico como biológico, que pusiesen de manifiesto la gran necesidad de estas actividades en la rutina de las destilerías. Fuimos más lejos aún, tratando de iniciar núcleos para trabajos de investigación dentro de los mayores y más ricos productores. Aunque tuvimos un éxito parcial en cuanto a hacer comprender la necesidad de la primero, no resulto lo mismo en cuanto a encauzar los trabajos de investigación.

En el último capítulo de esta obra damos instrucciones detalladas en cuanto a cómo ha de llevarse el control químico y biológico en la destilería de ron; no con el objeto ni con la pretensión de que esas instrucciones deban ser ejecutadas de ese modo al pie de la letra, sino para que sirvan más bien de ejemplo o modelo para que cada destilería confeccione su propio programa de control de acuerdo con sus medio y necesidades. Señalamos los puntos esenciales a que hay que atender para que el sistema de control sea eficiente y efectivo en mejorar la marcha de la destilería y la calidad de sus productos.

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NOTE:

Patents: Beoniot-Melle F. United States Patents 2,054,736 and 2,063,223, 1936. Hildebrandt, F.M. and M. Erb. United States Patent 2,169,244. 1939.