registered, is one more testimonial to the efficacy of wise scientific organization. The policies developed during the last four years of the Society's history are working out even better than the most sanguine advocate anticipated. Of those policies the organization of Divisions and the publication of special journals devoted to particular branches of chemistry are the most prominent and important, although the earlier organization of Local Sections and the creation of corporation membership must not be overlooked. There are several independent chemical organizations and independent chemical journals of a scientific type in America which have not yet joined issues with the American Chemical Society. It is unnecessary to state that these societies and publications are doing excellent work. Nevertheless the thought must arise again and again in the mind of an unprejudiced observer as to the possibilities, if complete organization of the chemical forces in America could be brought about.

THE DISTILLATION OF WHISKEY.[1]

By A. B. Adams, Chemist, Bureau of Internal Revenue.

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Until recently but little attention has been paid to the chemistry of whiskey, and practically none to the changes taking place in its distillation. As to what occurred in the different parts of the process, what was the composition of the different portions of the distillate and residues, but little was actually known. The work of Schidrowitz[2] has been about the only work published on this part of the subject, and as this is upon the methods as used in the British Isles, where the processes of making whiskey differ somewhat from those used in this country, it was thought desirable to obtain some data upon the processes used in this country. It is with the view of shedding some additional light on the subject that this paper is offered.

In the United States nearly every distiller uses one of two kinds of stills for the first distillation. The three-chambered beer still, either wooden or copper, is used in Maryland and Pennsylvania; in Tennessee and Kentucky the small ten- or twelve-chambered continuous beer still is used. It was, therefore, decided to obtain samples representative of a day's run from a distillery in Pennsylvania using the three-chambered beer still, and one in Kentucky using the continuous beer still.

The Pennsylvania distillery selected, uses a three-chamber charge wooden beer still: the vapor pipe passes out of the center of the top of the still, then downward into the bottom of the doubler—a copper cylinder about 30 inches in diameter, and as high as the beer still. Into this doubler are placed at the beginning of each charge the combined heads and tails of the previous charge. The vapors from the beer still enter this liquid, boil it, and pass out at the top of the doubler in a vapor pipe connected to a worm where they are condensed.

The object of cutting out and returning the heads and tails to the doubler appears to be to cut out of the whiskey those portions of the distillate which contain undesirable products, such as an excess of aldehydes, and certain indeterminable bodies occurring in the tail of the run, called by many fusel oil, which, however, are proven by these analyses not to be "fusel oil" or the higher alcohols. By reboiling, the alcohol is saved and the "undesirables" gradually disappear, partly by chemical change and partly by elimination.

Starting with the still in operation and a charge just run—it being charged every 28 minutes—the slop or spent beer in the lowest chamber is drawn off, and the partly dealcoholized beer in the second chamber dropped into the bottom chamber; the contents of the first or top chamber are dropped into the second chamber, and new beer (about 1,000 gallons) from the charging tank is emptied into the first chamber. The residue from the doubler of the beer still is emptied into the second chamber of the still, and the doubler recharged with the "heads" and "tails" of the previous run of the beer still, consisting of about 172 wine gallons, the composition of which can be seen by averaging the heads and tails in the proper proportion.

	Grams per 100 liters.						
	Proof.	Acids.	Esters.	Higher alcohol.	Aldehydes.	Furfurols.	Quantity gallons.
Average of heads....	111.4	7.2	126.7	80.0	164.0	0.0	12
Average of tails.....	61.2	9.6	15.8	37.0	7.0	0.4	160
Average.....	64.6	9.4	23.5	40.0	17.9	0.38	172 total
Contents of doubler at end of charge.....	....	27.6	7.0	6.1	trace	0.0	172 (about)

Live steam is then turned into the bottom chamber, a pressure of about 4–5 pounds being used. In about eight minutes the distillate begins to come over. The first runnings called "heads" are very turbid and are collected in the "low wine" tank as soon as the distillate clears, which takes about two minutes; a sufficient quantity is considered cut out as "heads;" the distillate is then turned into what is called the "high wine" tank, the contents of which are redistilled the following day. This "high wine," or "middle run," is continued for about five minutes or until the proof has dropped to 112 when the flow is again turned into the "low wine" tank where it is mingled with the "heads." The distillation is discontinued when a certain number of inches of low wines has been run and the proof has reached about 8°. The contents of the lowest chamber are now exhausted of alcohol

[1] Published by permission of the Commissioner of Internal Revenue. Read at the summer meeting of the American Chemical Society, Detroit, Mich., 1909.

[2] See Schidrowitz in *Royal Commission on Whiskey*, Vol. 1, Great Britain. Schidrowitz in *The Journal of the Institute of Brewing*, 1906.

and are drawn off. This is one cycle of the beer still and is repeated eighteen times each full day. By this method of operation the beer is finally separated into only two products, slop and "high wines," the "heads" and "tails" being again distilled in the next run of the still.

Four hundred bushels of grain are mashed in the proportion of forty-five gallons of water to the bushel, producing 18,000 gallons of beer each day. The "high wines" are collected from each of the eighteen charges forming one day's production, thoroughly mixed, reduced to 100° proof and the next day redistilled in a pot still, which is a large copper still about 8 ft. in diameter, 6 ft. high with a small dome, or boiling head, about $2^1/_2$ ft. in diameter on top. The vapor pipe, about 7 inches in diameter, rising from this dome to a height of 15 ft., runs horizontally for about 15 ft. then down about 15 ft. to the worm. There is no return pipe of any kind on this still which would carry back any of the condensed vapors. The still holds 1900 wine gallons, and a closed steam coil or scroll is the source of heat.

The distillate in this case, as in the primary distillation, is separated into "heads," "middle run" and "tails." It takes about thirty minutes to run the "heads," about five hours for the "middle run," and one and a quarter hours for the "tails." The "heads" and "tails" are sent to the beer well in equal proportions for each 1000 gallons of beer in order that they may be redistilled in the beer still with the beer and keep the "middle run" free of off products. The "middle run" is reduced to proof and the succeeding day is bonded as whiskey. In this distillation as in the first, there is no final separation except into the "middle run," which is the finished product of the still, and the "lees water" which is emptied into the sewer (the "heads" and "tails" being mixed with the beer).

On February 2, 1908, samples were taken as follows:

21699	Unfermented mash	Represents	18,000 gals.
21700	Beer ready to be distilled	"	" "
21701	Slop or spent beer as emptied from lowest chamber of beer still	"	" "
Beer still:		Quantity.	Time of running.
21702	Sample aver. of heads	12 gals.	2 minutes.
21703	" 1st of middle run	80 "	5 "
21704	" middle of middle run		
21705	" end of middle run		
21706	" aver. of tails	160 "	12 "
21708	" contents of high wine doubler at end of charge.	172	
21709	" aver. high wines of day...	1900 gals.	at proof.
Redistillation still:			
21691	Sample as distillation started	40 gals.	30 minutes.
21692	" of heads just before cutting off		
21693	" middle run immediately after heads cut off	1200 "	5 hours.
21694	" 2nd middle run, 2 hours after 21693		
21695	" 3rd middle run, $1^1/_2$ hours after 21694		
21696	" 4th middle run, $1^1/_2$ hours after 21695		
21697	" just as soon as tails started	250 "	$1^1/_4$ hours.
21698	" tails just before cutting off tails.		
21707	" lees or residue in still	400 "	
21710	An average sample of the middle run or whiskey before being diluted to proof.		
21711	An average sample of the previous day's run diluted to proof.		

The methods used were those in Bulletin 107 revised, Bureau of Chemistry, using the Allen Marquardt method for "fusel oil." The "fusel oil" on "slops" and "lees" was determined by saponifying 500 cc. and distilling as usual.

The analysis of these samples is as follows:

		Grams per 100 liters.					
		Proof.	Acids as acetic.	Esters as acetic.	Aldehydes as acetic.	Furfurol.	Higher alcohols.
21699	Unfermented mash	0.0	93.6	...	trace	0.0	2.0
21700	Beer ready for distillation	10.4	381.6	...	"	0.0	19.1
21701	Slop or exhausted beer	0.6	290.4	...	"	0.0	2.6
Beer still:							
21702	Heads, average of one charge	111.4	7.2	126.7	164.0	0.0	80.0
21703	1st sample from middle run	144.3	8.4	110.9	126.0	1.4	173.0
21704	2nd sample from middle run	133.6	7.2	26.4	18.0	2.6	174.7
21705	3rd sample from middle run	117.3	7.2	22.9	7.0	2.4	137.3
21706	Tails, average of one charge	61.2	9.6	15.8	7.0	0.4	37.0
21709	Average of high wines or singlings, day's production	138.3	4.8	38.7	36.0	1.6	170.0
Redistillation still:							
21691	1st heads, very first run	94.1	2.0	95.0	78.0	0.0	50.0
21692	2nd heads, just before heads cut off, $^1/_2$ hour later	163.2	2.4	70.4	94.0	0.80	160.0
21693	Middle run immediately after heads cut off	163.7	4.8	56.3	96.0	0.85	160.4
21694	2nd middle run, 2 hours after 21693	157.5	6.0	15.8	5.7	1.55	265.4
21695	3rd middle run, $1^1/_2$ hours after 21694	153.4	4.8	7.0	2.1	1.7	349.4
21696	4th middle run, $1^1/_2$ hours after 21695	114.7	3.0	14.1	1.5	6.0	153.6
21697	Tails immediately after middle run cut off	95.2	4.2	15.8	1.2	7.5	74.4
21698	Tails just before finishing of tails	15.4	3.6	8.8	0.0	2.6	10.2
21707	Lees water	...	2.4	8.8	trace	0.0	2.0
21710	Finished product before dilution	150.0	3.0	21.1	14.9	1.55	285.2
21711	Finished product after diluting for bonding (product of previous day)	100.9	1.2	15.8	6.5	1.0	191.9

NOTE.—When the acids were first determined the results in some of the cases were higher than is here reported, due, it was found, to the presence of CO_2. Also, wherever aldehydes were present in excess, the higher alcohol content was increased; the aldehydes, therefore, had to be destroyed by means of metaphenylendiamine hydrochloride.

Proof.—In both the first and second distillation it will be noticed that the first runnings are lower in proof than in sample taken in the course of the next few minutes. This is explained in part by the fact that there are depressions in the worms, which hold some of the last runnings of the previous distillation which are low in proof, and until this low-proof material is displaced, the proof of the distillate shows lower than it should be.[1] It takes but a minute for the still to run true as will be seen from the analysis.

Taking first the curve calculated from the proofs of the distillates of the beer still, we see that the first proof is low as previously explained.

The still starts at a proof of 10°.

	Degrees.	
Heads:		
1 minute later.....	92	Proof.
2 "	122	
Middle run:		
Middle run starts at.....	122	
½ minute later.....	144	
1 " "	144	
2 " "	144	
3 " "	132	
4 " "	122	
5 " "	112	Cut off middle run.
Tails:		
Beginning of tails at.....	112	
2 minutes later.....	92	
4 " "	72	
6 " "	52	
8 " "	32	
10 " "	20	
12 " "	8	Cut off tails and recharge still.

These proofs were taken during the running of one charge.

Excluding the first running, the proof curve is nearly a straight oblique line descending. Taking the proof curve of the results from the redistillation, we find practically the same results with the exception that the high proof holds for a longer time, some three and a half hours by reason of the larger volume of spirits.

Higher Alcohols.—We find exactly the same result in the "fusel oil," or rather the higher alcohols, namely that the content varies directly as the proof, and the curve is a straight oblique descending line for the beer still distillation. In the curve from the redistillations, almost the same results are shown except that the highest point is not reached until just as the proof begins to descend, proving that the "fusel oil" follows the alcoholic distillation.

It has been thought by many that the higher alcohols, or most of them, would be found in the "slop" and "lees water," but this is not a fact, the larger amount being in the high-proof portion of the distillate. Calculating the results to per cents., we find that in the slop from the beer still there are practically no higher alcohols, at most a trace, there is 4.7 per cent. separated in the heads, and 28.9 per cent. in the tails, leaving 66.4 per cent. of the higher alcohols which are distilled in one charge of the beer still, left in the high wine portion of the distillate. It should be borne in mind that the 33.7 per cent. of higher alcohols, which are in the heads and tails, are not thrown out but are placed in the beer still doubler and are redistilled with the next charge; the only higher alcohols in the beer which do not find their way into the high wines at the end of the day are the traces which remain in the slop.[1]

In the redistillation by averaging the samples of heads we find them to contain 1.2 per cent. of the higher alcohols, while the tails contain 3.00 per cent., and the lees water 0.2 per cent., making a total of 4.4 per cent. of the higher alcohols which are in the "spirits" in the redistillation still when it is started, but which do not appear in the finished whiskey. Here, again, it should be noted that the "heads" and "tails" are returned to the beer well for redistillation, leaving a total elimination of higher alcohols of only 0.2 per cent. It is, therefore, evident there is practically no fractionation of the spirit whereby any appreciable quantity of higher alcohols is eliminated, as practically all of the "fusel oil," or higher alcohols produced during fermentation are found in the finished distillate when bonded.

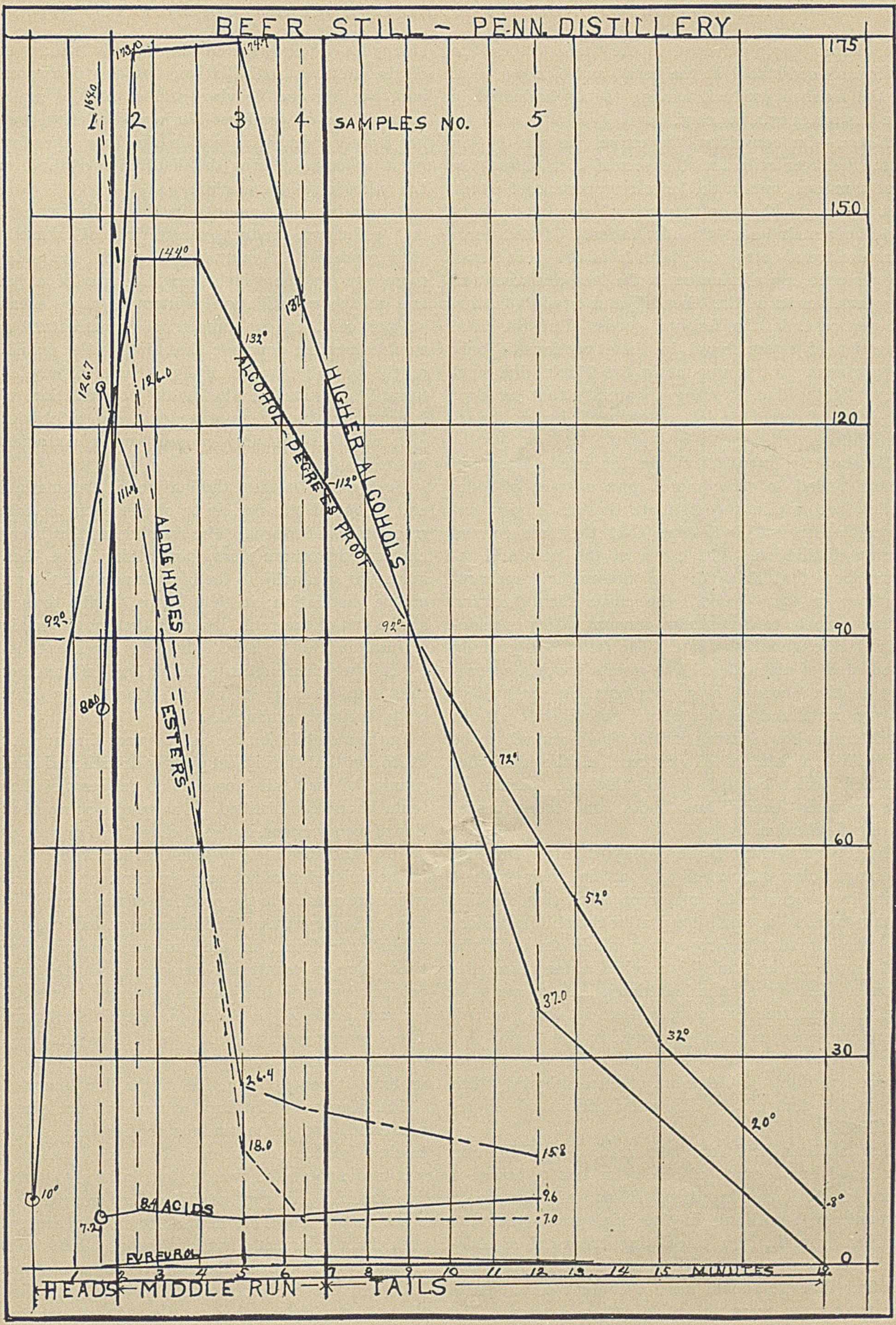
Acids.—It is found in considering the acids, that while a considerable amount is formed in the beer, only about one-fourth is distilled over, about three-fourths remaining in the "slop." While no attempt was made to determine their proportionate content quantitatively it is known that almost all of the acids remaining in the exhausted beer are lactic, succinic and phosphoric acids, which are not volatile at the temperature of boiling water.

The curve for the distillation of the acids would be a straight (nearly) horizontal line, beginning at 7.2 and ending at 9.6. In the redistillation the same point holds good, the content of acid being almost a constant throughout the distillation. It will be noted that the amount of acids found in the beer still distillates does not check with the difference between the amounts in the slop and in the beer. Only a very small proportion of the acids distilled, appear in the total "high wines." (The acidity of the beer is 381.6; that of the spent beer 290.4—a difference of 91.2 grams per 100 liters; the acidity of the "high wine" for the day is 4.8).

In the redistillation the total acidity found in the different fractions is 85.0 per cent. of that in the "singlings," showing that a slight loss takes place during the redistillation. Based upon the acidity found in the fractions 1.5 per cent. are separated in

[1] This is one reason why the "heads" are cut out; namely, to flush the worm before collecting the "whiskey run."

[1] The results of analysis give a figure of 2.6 grams per 100 liters. It would be very difficult to prove this to be due solely to the oxidation of the higher alcohols, as the method is not adapted to determine traces. It will be noticed that practically the same result was obtained for the unfermented mash (2.0) as for the spent beer (2.6) yet no one will dispute the fact that no higher alcohols are present in the unfermented mash. On these grounds it is felt justifiable to say that only a trace of higher alcohols is found in the spent beer.


BEER STILL - PENN. DISTILLERY
SAMPLES NO.
1
2
3
4
5
HIGHER ALCOHOLS
ALCOHOL - DEGREES PROOF
ALDEHYDES
ESTERS
ACIDS
FURFUROL
MINUTES
HEADS
MIDDLE RUN
TAILS
175
150
120
90
60
30
0
1
2
3
4
5
6
7
8
9
10
11
12
13
14
15
19
184.0
173.0
174.9
144°
132°
137
126.7
126.0
112°
111.0
92°
92°
88.0
72°
52°
37.0
32°
26.4
20°
18.0
15.8
10°
9.6
8.4
8°
7.2
7.0

the "heads," 17.3 per cent. in the "tails" (these are afterwards redistilled in the beer), and 64.0 per cent. in the finished product, leaving to be eliminated in the lees water 17.2 per cent.

Esters.—On consulting the curves we see that in both the beer still and the second distillation, the first runnings contain the largest proportion of esters, but rather quickly drop to a normal amount throughout the remainder of the distillation. Of the esters that are found in the distillates from the second still, 10 per cent. are eliminated in the lees, and about 18 per cent. found in the "heads" and "tails," leaving 72 per cent. in the finished goods. But the total quantity of esters found in these portions is only 66 per cent. of the amount in the "high wine singlings" of the day, showing apparently that some 34 per cent. of esters are lost or changed during the redistillation.

Aldehydes.—Careful work on the mash, beer and "slop" failed to show more than a trace of aldehydes, and yet in the distillate there is a very appreciable amount, indicating that they are formed during distillation. The curve of the beer still results show that aldehydes are present in large proportions in the "heads," the curve then dropping rather quickly to a minimum amount which is maintained throughout the run. The "slop" is entirely exhausted of aldehydes. The results prove a separation in the "heads" of 33 per cent., in the "tails" 19 per cent., and in the "slop" nothing but a trace (indeterminable), leaving in the "high wines," to be redistilled, a total of 48 per cent. of the aldehydes distilled, which consists of the aldehydes present in the previous "heads" and "tails" and those formed during distillation.

In the redistillation, the aldehydes, after the first runnings, almost immediately attain their highest point in the curve, the major portion being distilled within two and one-half hours after the still is started; the curve gradually drops to a minimum until the last sample of the "tails" is reached where the results show no aldehyde. A calculation of the amounts in the different fractions proves that in the "heads" there are separated 16.0 per cent., in the "tails" 0.7 per cent., and in the "lees" none, leaving in the finished product 83.3 per cent. of the aldehydes which are in the sum of the different fractions of the redistillation.

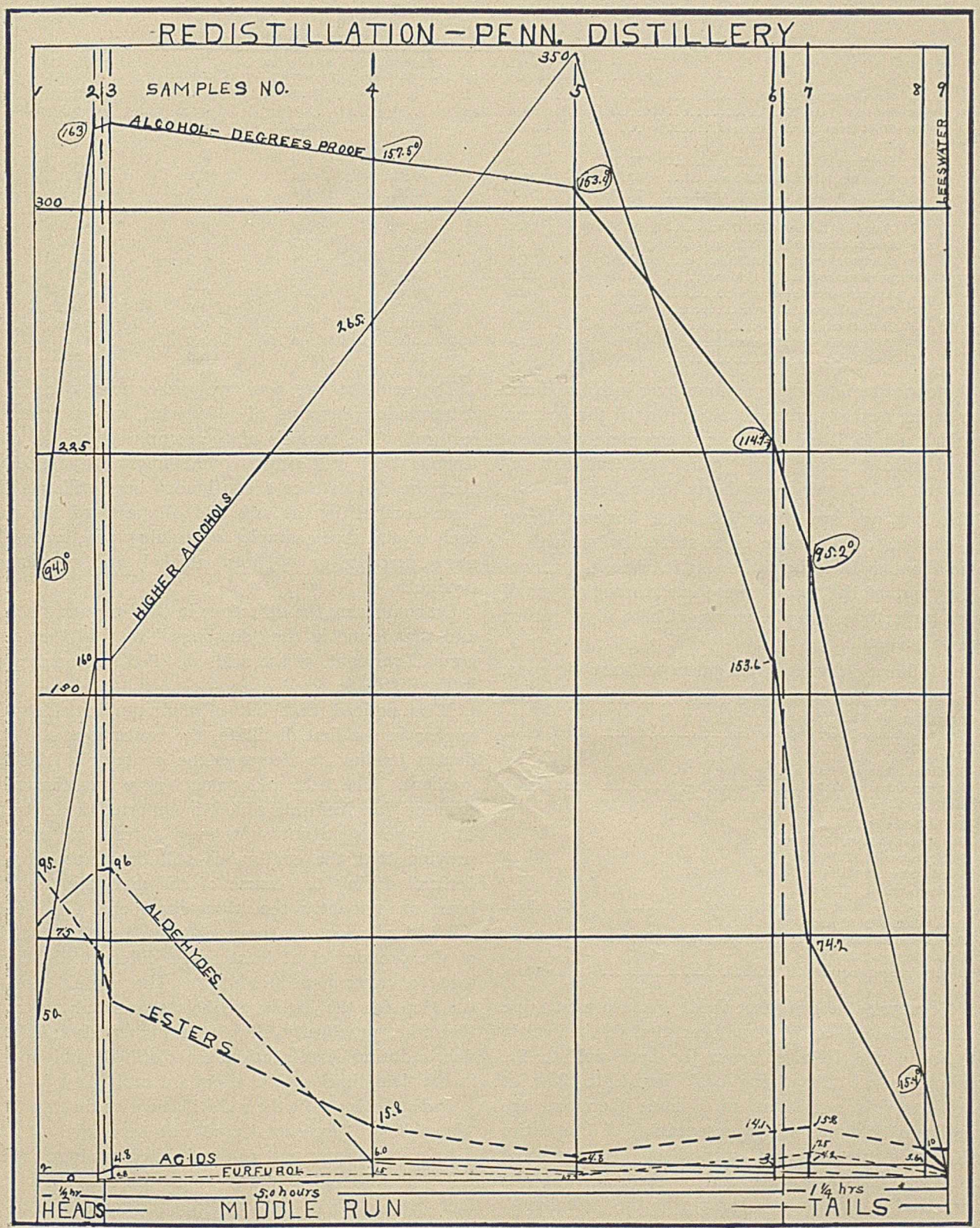
As has been said, the beer ready for distillation contained but a trace of aldehydes but of course a trace of aldehydes in 18,000 gallons of beer would amount to something in the high-proof "spirits" distilled from it. On calculating the total of aldehydes present in the "high wine" back to the beer, we find that we should have a content of 2.7 parts per 100,000, whereas we actually have a trace. The aldehydes in the "high wine" represent also, the aldehydes which were in the "heads" and "tails" of the previous redistillation, but this figures to 0.2 part per 100,000, leaving still a total of 2.5 parts in the beer, or produced during distillation, but, as has been shown, it is not in the beer, therefore, it must be formed during the distillation of the beer by the oxidation of the alcohol.

Comparing the amounts in the "high wine" and in the finished product, recognizing, of course, that they represent different days' goods, we find that there was in the "high wine" 0.4940 of a gallon, and in the redistillation products 0.2147, a loss of 56 per cent. in the process of redistillation. One would therefore conclude that during the distillation of the beer by means of live steam, aldehydes are formed by oxidation; during the redistillation, which is conducted by means of a steam coil, aldehydes are lost, which is probably caused by further oxidation into acids.

Furfurols.—In the examination of the mash, beer and "slop," I was unable to obtain even the faintest reaction for furfurol. The results of the beer still expressed in a curve start from nothing in the "heads," increasing gradually to the highest point at the middle of the distillation; it then drops slowly to nothing at the end of the run. There is then no separation of furfurol in the "heads," about 35 per cent. in the "tails" (which are returned to the doubler), and none in the "slop," leaving in the high-wine run 65 per cent. of the furfurols produced.

In the redistillation the curve is very similar with the exception that the highest point is not reached until the running of the "tails" or until 70 per cent. of the contents have been distilled. The results show a separation of 0.79 per cent. in the "heads," 6.3 per cent. in the "tails," and absolutely none in the "lees water," leaving in the finished product 92.9 per cent. of the furfurols which are present in the products of the second distillation, or 89 per cent. of the furfurols present in the "high wines" are found in the finished product, proving that there is practically no change in the furfurol content by redistillation, and further, that all the furfurol produced is found in the finished product. These results seem to prove that furfurol is produced at this distillery during the distillation of the beer, probably through the action of the live steam.

The Kentucky distillery, whose product was next examined, uses a continuous copper beer still, containing fourteen chambers. The still is about four feet in diameter and twenty feet in height, heated by live steam. This type of still differs from the charge chamber still in that, in the latter, the contents of each chamber are let down into the next lower chamber by the beer runner at the end of each charge period. While in the continuous still, as the name implies, there is no interruption to the process, the beer is pumped in at the top of the still (heated nearly to the


REDISTILLATION — PENN. DISTILLERY
SAMPLES NO.
1
2
3
4
5
6
7
8
9
ALCOHOL — DEGREES PROOF
163
157.5°
153.9
114.7
95.2°
94.1°
350
265
300
225
190
160
153.6
HIGHER ALCOHOLS
LEESWATER
95
96
95
75
50
ALDEHYDES
ESTERS
74.2
15.4
15.8
14.1
15.8
4.8
6.0
4.8
AC IDS
FURFUROL
½ hr
5.0 hours
1¼ hrs
HEADS
MIDDLE RUN
TAILS

boiling point) in a continuous stream, flows through the down pipe into the next plate or chamber, flows across this plate (which is perforated with holes through which live steam is continually ascending) to the opposite side where it flows over into the down pipe of the next chamber, and so on until the bottom of still is reached and the beer is completely dealcoholized. No "heads" and "tails" are cut out because the flow of spirit is uniform in quality.

The singlings are redistilled in a horizontal pot still shaped very similar to a steam boiler with a dome about 12 ft. in height, and 3 ft. wide, the vapors passing out of the top of the dome, through a small vapor pipe, then into the worm. There is no return pipe on this still.

The following samples were taken on March 8, 1909.

1	Beer from tub No. 9, representing	8370 gallons.
2	Beer from tub No. 9 (duplicate)	
3	Slop or exhausted beer, representing	9300 gallons.
	Beer still:	
4	Singlings taken at 11.07 A.M.	
5	Singlings taken at 11.30 A.M., representing	1236 gallons.
6	Singlings taken at 11.53 A.M.	
	Redistillation still:	
7	Foreshots taken at 12.27 P.M.	
8	Spirits taken at 12.29 P.M.	
9	Spirits taken at 12.55 P.M.	
10	Spirits taken at 1.25 P.M.	
11	Average (of 7–10) of whiskey as bonded	875 gallons.
12	Tails taken at 1.30 P.M.	
13	Tails taken at 1.33 P.M.	175 gallons.
14	Tails taken at 1.38 P.M.	
15	Backings or "leeswater"	361 gallons.

The samples above described are representative of the distillation of the same beer, that is, the beer in tub No. 9 was followed from the beer well to the bonding room. The "singlings" began to flow at 10.45 A.M. and ceased at 12.12 P.M.; therefore, the "singlings" samples are representative of the entire run, as this still produces "spirits" of the same proof from the beginning to the end of the run. (It was impracticable to obtain a sample of the "singlings" after they had been mixed.)

The redistillation started at 12.26 P.M.; the first sample was taken one minute after the flow started, and the second sample three minutes after. Sample No. 10 was taken just before the flow was turned into the "tails." The "tails" are mixed with the beer to be distilled the next day; the "backings" are emptied into the sewer.

The following are the results of analysis:

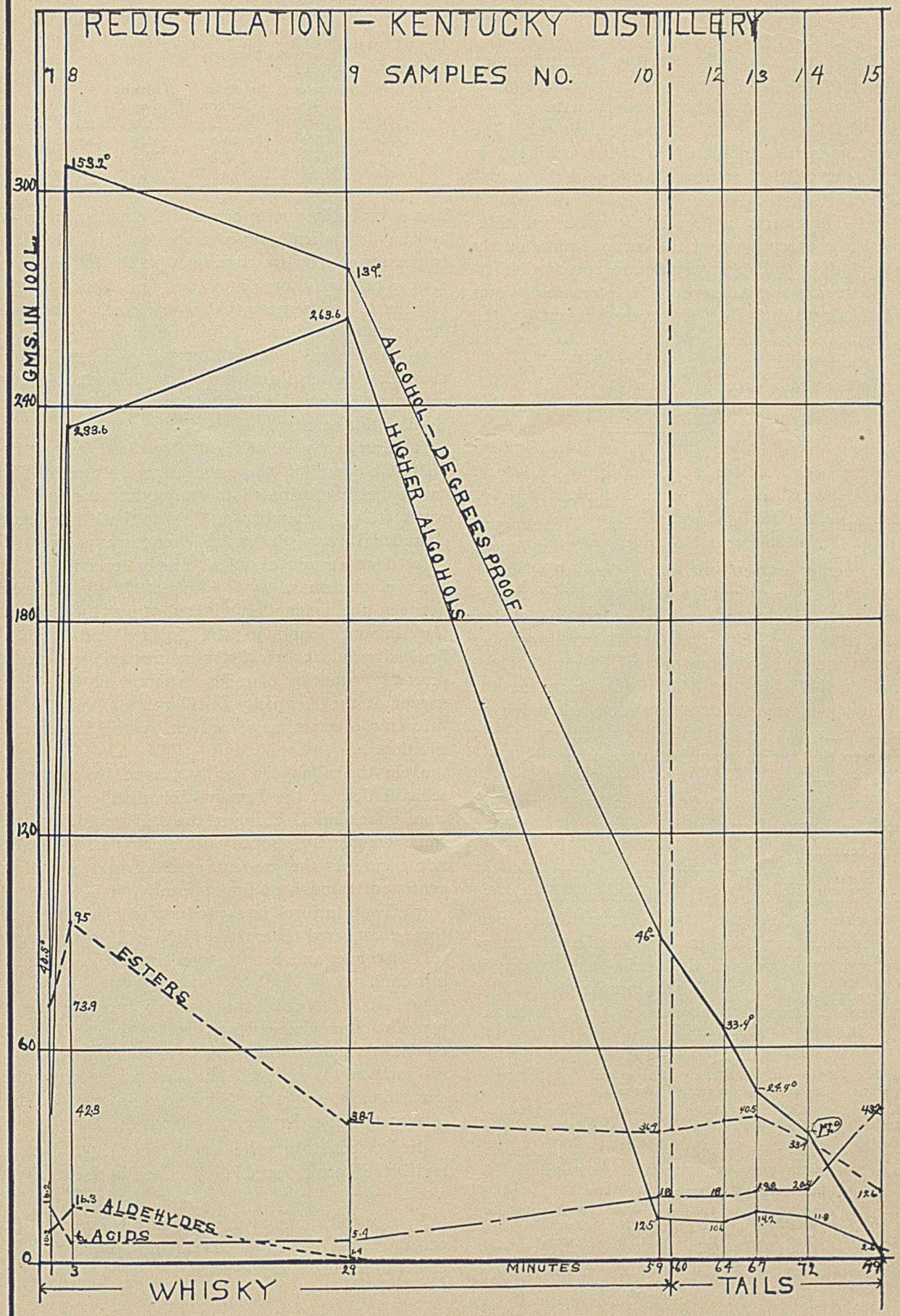
		Grams per 100 liters.				
	Proof.	Acids as acetic.	Esters as acetic.	Aldehydes as acetic.	Furfurol.	Higher alcohols.
1 Beer from tub No. 9	...	54.0	..	0.54	0.05	18.9
2 Beer from tub No. 9	...	54.6	..	0.74	trace	18.5
3 Slops from 1st distillations	...	39.3	..	0.36	trace	1.3
Beer still:						
4 Singlings taken at 11.07 A.M.	71.7	21.0	42.4	1.0	0.0	101.1
5 Singlings taken at 11.30 A.M.	67.7	20.4	49.3	0.9	0.0	99.9
6 Singlings taken at 11.53 A.M.	71.7	21.0	45.8	0.8	0.0	103.4
Redistillation still:						
7 Foreshots taken at 12.27 P.M.	40.5	16.2	73.9	10.3	0.0	42.3
8 Spirits taken at 12.29 P.M.	153.2	6.0	95.0	16.3	0.0	233.6
9 Spirits taken at 12.55 P.M.	139.0	5.4	38.7	1.4	0.0	263.6
10 Spirits taken at 1.25 P.M.	46.0	18.0	36.9	0.0	0.0	12.5
11 Average sample—bonded	102.1	6.6	40.6	2.6	0.0	161.5
12 Tails taken at 1.30 P.M.	33.4	18.0	38.7	0.0	0.0	10.6
13 Tails taken at 1.33 P.M.	24.4	19.2	40.5	0.0	0.0	14.2
14 Tails taken at 1.38 P.M.	17.1	20.4	33.4	0.0	0.0	11.8
15 Backings	0.0	43.2	17.6	0.0	0.0	2.6

The acids of the beer are rather low, there is a determinable amount of aldehydes and a trace of furfurol. The analysis of the samples from the continuous beer still proves what might be expected, that the distillate of a continuous beer still has the same composition at any and all parts of the run, each of the three samples containing practically the same percentages of both alcohol and congeneric products.

On comparing the difference in the beer and "slop," and that found in the "singlings," we find that only a small per cent. of the acids distilled appear as such in the distillate.

It is noticed that but a minimum of aldehydes appear in the first distillate, the continuous still evidently tending to decrease the content of this ingredient. The beer and "slop" gave an affirmative reaction for furfurol, which is reported as a trace. The singlings do not show even a trace of furfurol, proving that the continuous still does not produce furfurol during the course of the distillation of the beer, as evidently the three-chambered still does. (This conclusion has been checked by the analysis of the product of at least seven different distilleries using a continuous beer still.) The examination of the slop for the higher alcohols shows only a trace; therefore, practically all of the higher alcohols produced during fermentation are distilled, and appear in the "singlings" to be afterward redistilled.

Redistillation.—In the redistillation we find that the "heads" or "foreshots" are not separated at this distillery but form part of the finished product. (This policy is followed at many houses on account of the fact that much of the flavoring matter seems to be present in the first runnings of the distillation.) The "spirits" are distilled much faster here than in the Pennsylvania distillery. The same remarks made upon the previous redistillation apply here; the proof starts low for reasons previously given, but in two


REDISTILLATION — KENTUCKY DISTILLERY
SAMPLES NO.
7
8
9
10
12
13
14
15
GMS. IN 100 L.
300
240
180
120
60
0
159.2°
139°
263.6
233.6
ALCOHOL — DEGREES PROOF
HIGHER ALCOHOLS
95
ESTERS
73.9
42.3
38.7
16.3
ALDEHYDES & ACIDS
5.4
6.4
46°
33.4°
24.9°
34.7
40.5
33.7
43.4
12.6
19.8
20.4
18
18
12.5
10.4
14.2
11.8
3
29
59
60
64
67
72
79
MINUTES
WHISKY
TAILS

minutes runs true, and the curve is then a straight descending line.

The acids drop gradually, starting at 16.2, but at the last sample of the "middle run" increase, leaving in the "backings" a higher proportionate acidity (acetic) than was present in the "singlings." The per cent. of acids eliminated in the "backings" is about 63 per cent. of the total acidity of the "singlings."

Esters.—The esters distil as in the other redistillation with the exception that the curve is more nearly horizontal, due, probably, to the more speedy distillation. Reducing the proportion of esters to a comparable basis we find that there are present in the "singlings" 0.5661 gallon,

In the whiskey.....	0.3553	gallon,
In the "tails".....	0.0656	"
Backings.....	0.0634	"
	0.4843	"

or in the whiskey, 73.4 per cent. in the "tails," 13.5 per cent. in the "backings," 13.1 per cent. of the esters resulting from the redistillation. This amount is only 85.5 per cent. of the esters in the "singlings," a loss of nearly 15 per cent. during redistillation.

Aldehydes.—The examination for aldehydes proves the greatest amount to come over in the first few minutes of the distillation, while all has come over before the end of the whiskey or "middle run." There are no aldehydes in the "tails," and none in the "backings." There is about twice as much aldehyde in the whiskey as in the "singlings;" this is directly opposite to that which occurred in the other distillery.

Furfurols.—There was no furfurol in the "singlings," and there is none in the redistillation (but furfurol is found after this whiskey has been in a charred barrel).

Higher Alcohols.—The results are the same as in the other redistillation, a very small amount appears in the "backings." A calculation of the higher alcohols shows 1.253 gallons in the "singlings," 1.4131 in the whiskey, 0.0214 in the "tails" and 0.0094 in the "backings," showing slightly more higher alcohol in the redistillation product than in the "singlings."

RECAPITULATION.

Pennsylvania Distillery.—*Singlings.*

Heads. Per cent.	Middle run. Per cent.	Tails. Per cent.	Slop or lees. Per cent.	
4.7	66.4	28.9	Trace	Higher alcohols.
3.8	27.3	68.9	..	Acids.
21.3	43.3	35.4	..	Esters.
33.0	48.3	18.7	..	Aldehydes.
0.0	65.0	35.0	..	Furfurol.

Pennsylvania Distillery.—*Redistillation.*

1.2	95.6	3.0	0.2	Higher alcohols.
1.5	64.0	17.3	17.2	Acids.
9.4	71.9	8.7	10.0	Esters.
16.0	83.3	0.7	...	Aldehydes.
0.8	92.9	6.3	...	Furfurol.

Kentucky Distillery.—*Singlings.*

No separation to heads and tails.

Kentucky Distillery.—*Redistillation.*

No heads.	Whiskey or middle run. Per cent.	Tails. Per cent.	Backings. Per cent.	
...	97.8	1.5	0.7	Higher alcohols.
...	23.4	13.6	63.0	Acids.
...	73.4	13.5	13.1	Esters.
...	100.0	..	..	Aldehydes.
...	...	..	..	Furfurol.

This is probably due to errors of the method increased by the multiplication necessary for this calculation. In per cents. 0.07 are eliminated in the backings, and 1.5 per cent. pass over into the "tails" and are redistilled next day, proving that 99 per cent. of the higher alcohols produced are in the finished product.

In conclusion, I would state that, in my opinion, the following facts are proven regarding the changes occurring during the distillation of whiskey as practiced in this country.

(1) Practically all of the higher alcohols (or "fusel oil") are found in the finished product, only slight traces being eliminated in the spent beer and lees-water. This statement applies to both the charge chambered beer still and the continuous beer still.

(2) That certain chemical changes occur:

a. In the acids, as is proven by the difference between the amount eliminated from the beer and the amount found in the finished product, the finished product being almost neutral, yet no appreciable quantity of acids is found in the "lees," except at the Kentucky distillery.

b. In the esters, as is proven by the loss in redistillation.

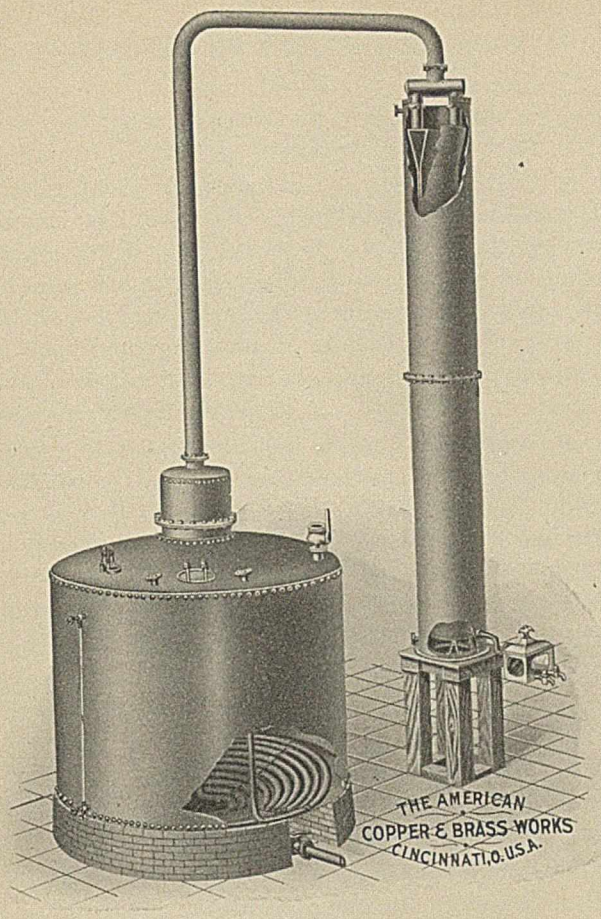
c. In the aldehydes, as is proven by the loss in redistillation at the Pennsylvania distillery. If there were no chemical changes, the still would finally become completely choked with aldehydes. This does not occur, as the content of aldehydes runs fairly consistent throughout the season.

(3) That furfurol is a product of distillation in a three-chambered beer still, or a still operating on this principle, and is not a product of the continuous beer still.

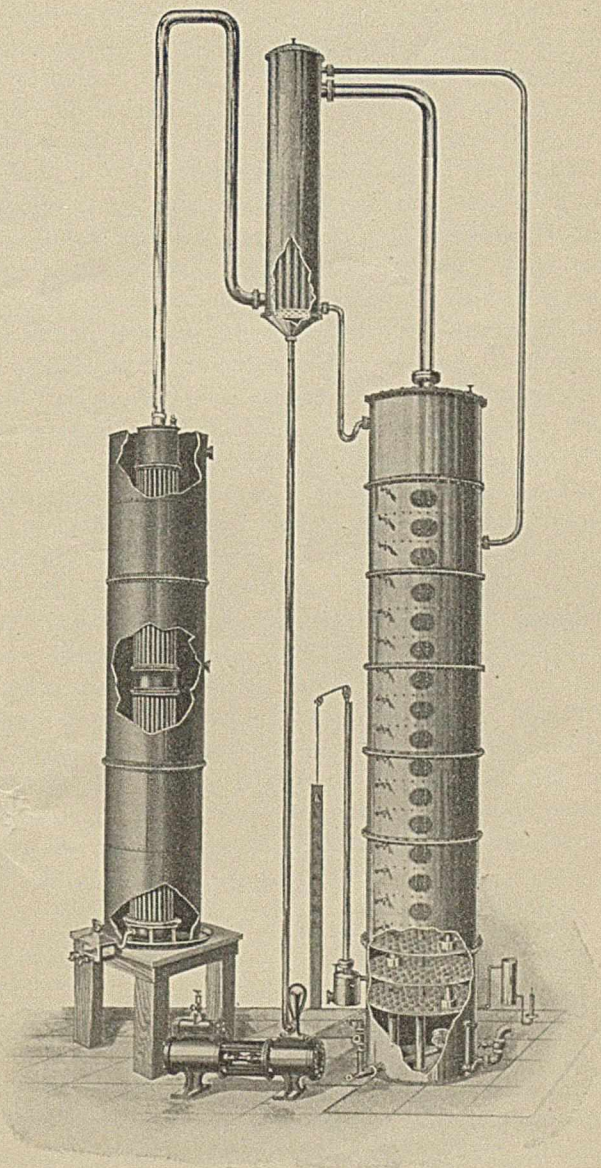
(4) That the substances which produce the unpleasant odors, frequently found in the "tail" of the distillation and in the "lees," are not "fusel oil" as is commonly supposed, but factors at present indeterminable, probably being water-soluble products of fermentation distilled over from the beer.

Credit should be given to Messrs. A. W. Schorger and Peter Valaerr for able assistance in the analytical work.

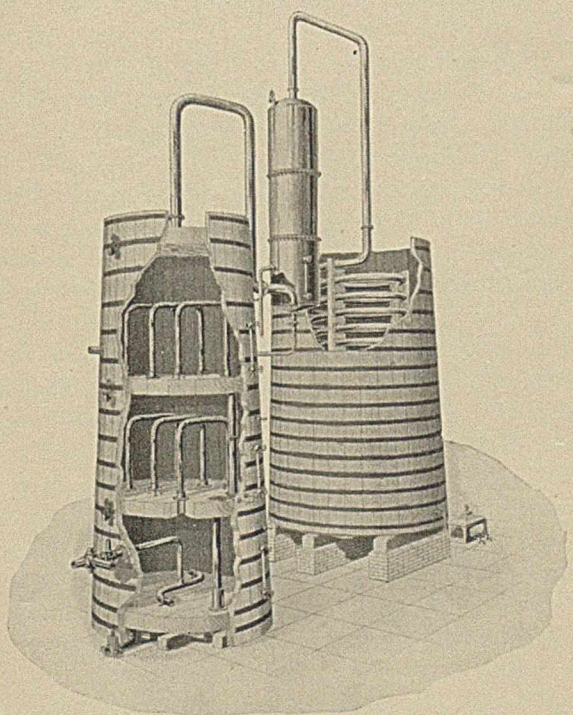
I desire to express my thanks to the American Copper and Brass Works, of Cincinnati, Ohio, for the loan of the illustrations of the stills.



Redistillation still.



Continuous beer still.



Three-chambered charge beer still.