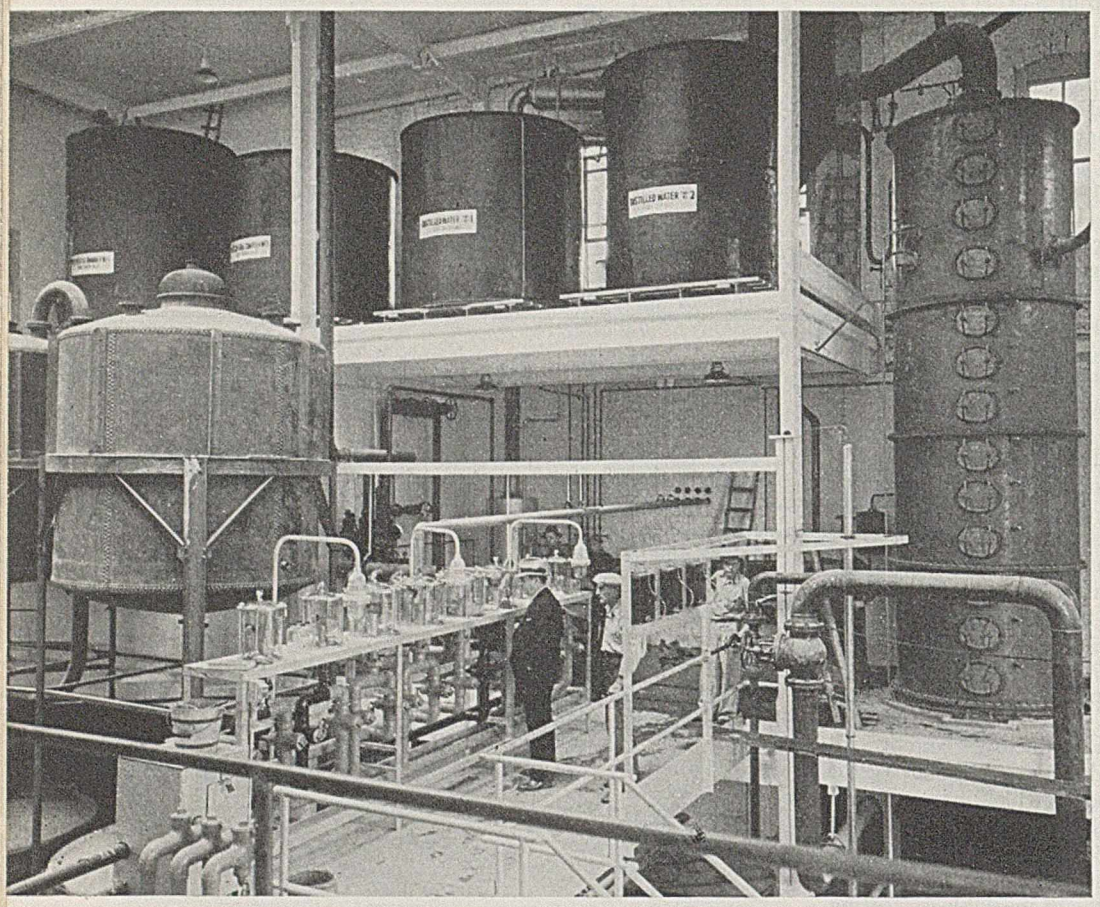


INTERIOR OF DISTILLING ROOM SHOWING CONTINUOUS STILLS, POT STILLS, AND GOVERNMENT WEIR BOXES

Courtesy, National Distillers Products Corporation

Changes in Whisky Stored for Four Years ..

PETER VALAER AND W. H. FRAZIER
Bureau of Internal Revenue Laboratory, Washington, D. C.

AN INVESTIGATION was begun in the fall of 1929 to determine the character of whisky and the changes taking place during its aging in wood. The whisky so tested was the product of twelve distilleries which at that time had been given permits to operate in order that the rapidly dwindling stocks of whiskies withdrawn for medicinal purposes might be replenished. The quantity and variety of the whisky was limited, and the selection of the distillers was made from the numerous applications that had been filed. This selection was based on the distiller's previous experience, reputation, equipment, location, and other factors. In conducting these experiments the twelve distilleries offered complete coöperation.

All of the legitimate whisky manufactured during the prohibition period is represented by the whisky referred to in this article and is straight whisky distilled at or below 160° proof. With one possible exception, no whisky of the lighter body type was made during this period.

Since repeal many distilleries have been in operation. Some of them are distilling at a proof greater than 160° and manufacturing what is known to the trade as a light-bodied whisky containing less congenerics, particularly fusel oil.

Since repeal, at the request of the Government, more than 100 barrels of whisky have been set aside in various bonded warehouses in different parts of the United States for observation and experiment. Some of these distilleries are manufacturing whisky of a lighter body; as soon as the 2-year storage period is ended, it is the intention of the Treasury Department to publish the results of analyses of these barrels.

Methods of Sampling

The most comprehensive treatise on this subject previously published (1908) was that of Crampton and Tolman (*2*) based

The largest increase of acids, esters, solids, and color is during the first 6 months of storage. The acids and esters did not reach a ratio of 1 : 1, and the analyses showed no fixed relationship between these two compounds as found by Crampton and Tolman, at the end of 4 years.

There is an actual as well as an apparent gain in acids over the 4-year period using the data calculated to original volume. The actual acid gains were from 24.9 to 56 grams per 100 liters (average 40.1). There is an actual as well as an apparent gain in esters over the 4-year period when calculated to original volume. The esters actually gained from 7.4 to 21.3 grams per 100 liters (average 15.5). There is an actual loss of fusel oil during the aging period, which is from 6.9 to 58.4 grams per 100 liters (average loss 28.6), when calculated to original volume.

Quick-aging increases the color, solids, and acids.

Whisky changes while standing in glass. There is usually a decrease in acids and a tendency for esters to increase. There is often an increase in color. A definite change took place in the newly distilled whisky that stood 4 years in glass; it lost practically all of its slop taste and odor.

The constituents are continually undergoing changes as the aging process progresses, as found by Crampton and Tolman.

The increase in acids in charred barrels is due partly to fixed acids extracted from the wood but mostly to the formation of volatile acids during the aging process.

upon work done in this laboratory. The purpose of the present article is to study the medicinal whiskies as to variation in composition of the different types, changes effected during storage, and changes effected by quick-aging, to the end that a basis for differentiating between the products of individual distilleries may be available. In the course of this work a large number of samples was obtained which will further facilitate comparative studies of present production.

Medicinal whisky as defined by the U. S. Pharmacopeia X is as follows: "Whisky is an alcoholic liquid obtained by the distillation of the fermented mash of wholly or partly malted cereal grains, and containing not less than 47 per cent and not more than 53 per cent by volume of C_2H_5OH, at 15.56° C. It must have been stored in charred wood containers for a period of not less than 4 years."

Accordingly samples were taken on or near the date of entry of the barrels in the warehouses, and every 6 months for the ensuing 4 years so that 9 quart samples in all were drawn from each barrel, 198 samples from eleven distilleries. Because of conditions beyond the laboratory's control, it was impossible to obtain the samples from the twelfth distillery in time to include them in this investigation. In compiling the data for this paper, 1584 original determinations were made and approximately half as many second determinations. The samples were analyzed on or near the day of their arrival in the laboratory for proof, acids, esters, total solids, and color. Determinations for fusel oil, aldehydes, and furfural were made at the end of the 4-year period. The methods of analyses were substantially those of the A. O. A. C. (*1*).

The samples taken for this investigation represented a variety of whiskies: two sweet-mash ryes, two sour-mash ryes, two sweet-mash bourbons, and five sour-mash bourbons. Four of the samples had received quick-aging treatment. The whisky had been stored in warehouses of which seven were brick, three wood, and one metal construction.

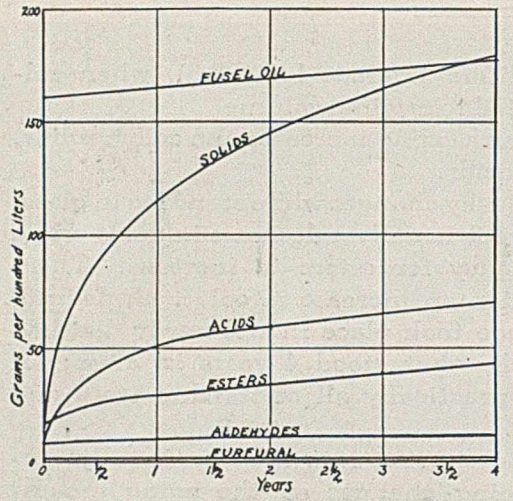
Although most of the original laboratory work was performed by one individual, W. H. Frazier (now deceased), the influence of the personal equation, in so far as the determination of acids, esters, solids, and fusel oil were concerned, was of little significance. For instance, in the titration of acids on a 25-cc. portion, the difference between the first light red of the titration end point and the full red end point was found to be only 10.05 cc. or equivalent to from 1 to 2 grams of acids per 100 liters. The personal equation factor, however, is greater in color determinations—for example, in the estimation of aldehydes and furfural and in the color of the whisky itself.

Two tests included in the Crampton and Tolman article but seldom employed in practical work (determination of color removed by ether and the paraldehyde tests) were omitted.

The grams of congeneric substances per 100 liters of whisky calculated to original volume were given only for the final

Courtesy, Glenmore Distilleries Company

INTERIOR OF WAREHOUSE SHOWING BARRELS IN RICKS



CHANGES IN COMPOSITION OF WHISKY OVER 4-YEAR PERIOD, USING FIGURES CALCULATED TO PROOF

4-year-old whiskies at the end of each section of Table II, where complete analysis of each barrel is shown. The gallonage content of the barrels is given twice—when the whisky was placed in the warehouse and again when the last sample, 4 years old, was taken.

In taking samples from the barrels semi-annually instead of annually, as was done by Crampton and Tolman, data were obtained at shorter intervals. This permits the checking of whiskies in the trade somewhat more closely than was obtained in the Crampton and Tolman work. They stated that practically no change had taken place in glass. On June 22, 1935, all of the samples were reanalyzed for their acid content, and a decrease was found in some instances of as much as 11 parts per 100,000, the average reduction being about 5 parts. Only those whiskies which were taken when new showed no, or practically no reduction in acid content. Eleven of the samples which showed the greatest reduction of acids while stored in glass were also reanalyzed for esters, aldehydes, and furfural (Table VIII). The conclusions obtained from these determinations were that there is a tendency for acids, aldehydes, and furfural to decrease slightly and for the esters to increase slightly during storage in glass bottles.

In order to determine what changes take place in newly distilled whisky that has been stored in clear-glass quart bottles for over 5 years, ten samples, taken at the time of production, received and analyzed in the fall of 1929 and in the spring of 1930, were reanalyzed in July, 1935. The results of this analysis showed that there was practically no change in the composition of these whiskies; that is, the acid, ester, aldehyde, furfural, and solid content was approximately the same as when first analyzed. There was, however, a surprising change in that the whisky had lost all of its "slop" taste and odor so characteristic of newly distilled whisky, licit or illicit. Many distillers, anxious to find an early market for their wares, often find this "slop" odor difficult to remove. Removal has been thoroughly accomplished in the bottle, although it may have required a good portion of the glass storage period to obtain this result. Experiments are now being conducted to determine just when, or how long it takes for this "slop" odor and taste to disappear from newly distilled whisky when stored in ordinary glass bottles.

Although the A. O. A. C. methods of analyses used here have been revised several times since 1908, they do not vary essentially from the methods used by Tolman and Crampton (*2*).

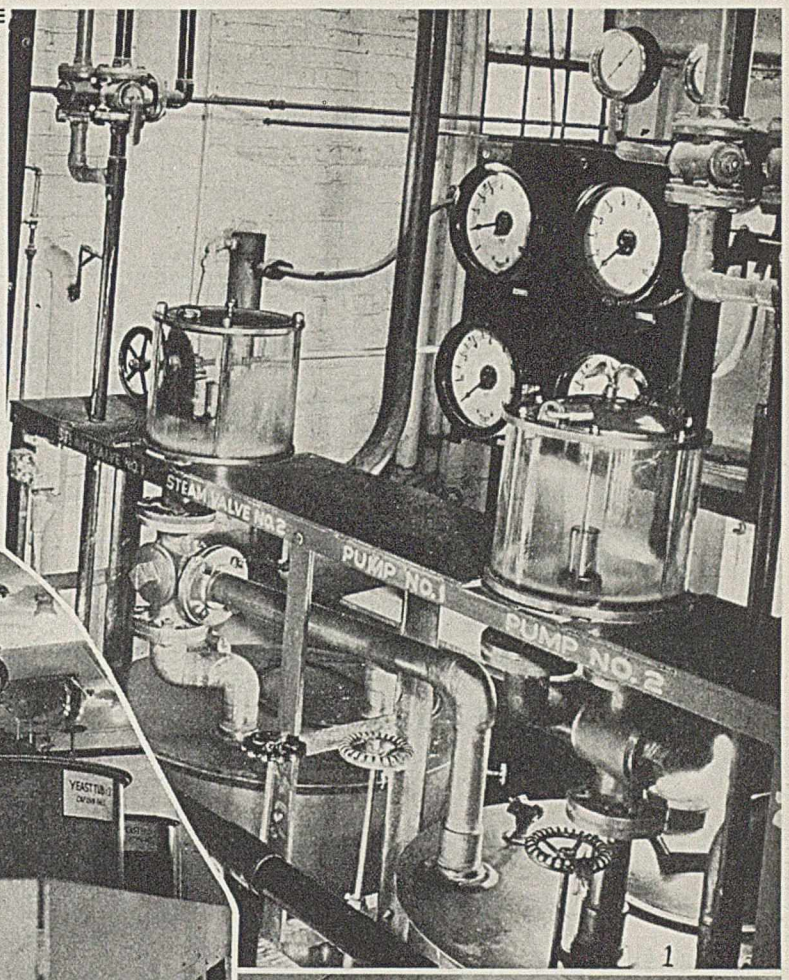
The color readings were made in a 0.5-inch (1.27-cm.) standard cell and by the use of the Lovibond tintometer, using the brewer's scale, brown series 52 (brown slides). This instrument is ideally suited for this work because the brown series 52 matches as nearly the color of whisky naturally aged in wood as has been found. The brown shade of caramel color is slightly off-shade when compared to the standard slides, series 52. Coal-tar color combinations introduced for the purpose of imitating whisky color, charred chip color and uncharred chip color, can be detected by those experienced in using this instrument, since these coal-tar color combinations are also off-shade.

The Marsh test for artificial color is still the most universally used test in the examination of whisky for the detection of caramel and some other artificial color. All of the 198 samples tested gave a negative reaction with Marsh's reagent. Amyl alcohol alone as a color reagent is less useful, giving in many

TABLE I. STORAGE CONDITIONS[a]

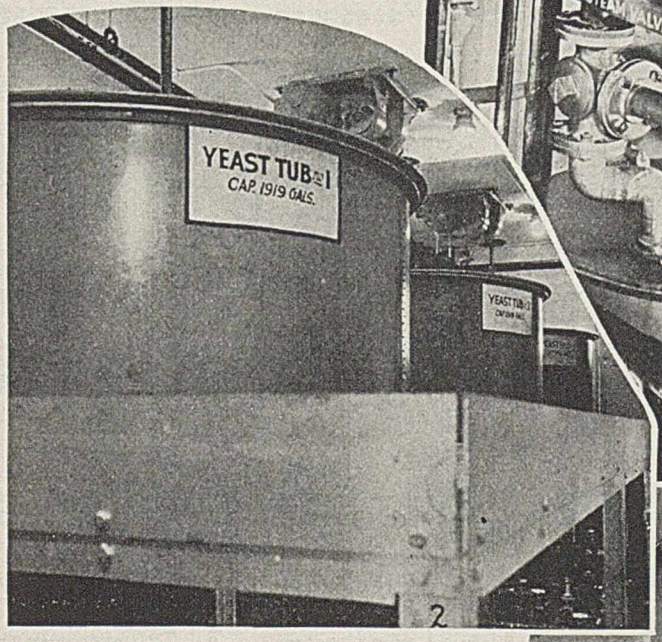
Distillery	Quick-Aging	Mash	Mash Compn.	Warehouse Temp.	Type of Warehouse	Still
			Bushels	° *F.*		
1	No	Sweet, bourbon	Corn 556, rye 11, malt 66	No heat	Frame, ironclad	Continuous copper, with doubler
2	No	Sour, bourbon	Corn 758, rye 120, malt 52	Hot-air heated, 2 mo.	Brick	Continuous copper, with doubler
3	No	Sweet, rye	Rye 689, malt 172	Steam heated; 75 winter, 80 summer	Brick	3-chambered copper, with doubler
4	No	Sour, bourbon	Corn 301, rye 64, malt 65	Steam heated; about 95	Concrete and brick	Continuous copper, with doubler
5	Yes	Sweet, rye	Rye 415, malt 117	Steam heated, Nov. to May, 75–80	Brick (numerous glass windows)	3-chambered, with doubler
6	Yes	Sour, rye	Rye 1188, malt 312	Hot-air heated; av. 72	Metal	3-chambered, with doubler
7	Yes	Sour, bourbon	Corn 1155, rye 279, malt 198	Heated (fan system); 65 in winter	Wood, metal-clad	3-chambered, with doubler
8	No	Sweet, bourbon	Rye 808, malt 142.5	Steam heated; 75–80	Brick	3-chambered copper, with doubler
9	No	Sweet, bourbon	Corn 556, rye 11, malt 66	No heat	Brick	Continuous copper, with doubler
10	Yes	Sour, bourbon	Corn 420, rye 90, malt 90	Heated at beginning	Brick	Continuous, with doubler
11	No	Sour, bourbon	Corn 301, rye 68, malt 61	No heat	Wood, ironclad	Continuous, with doubler

[a] All cooperage new white-oak charred barrels.

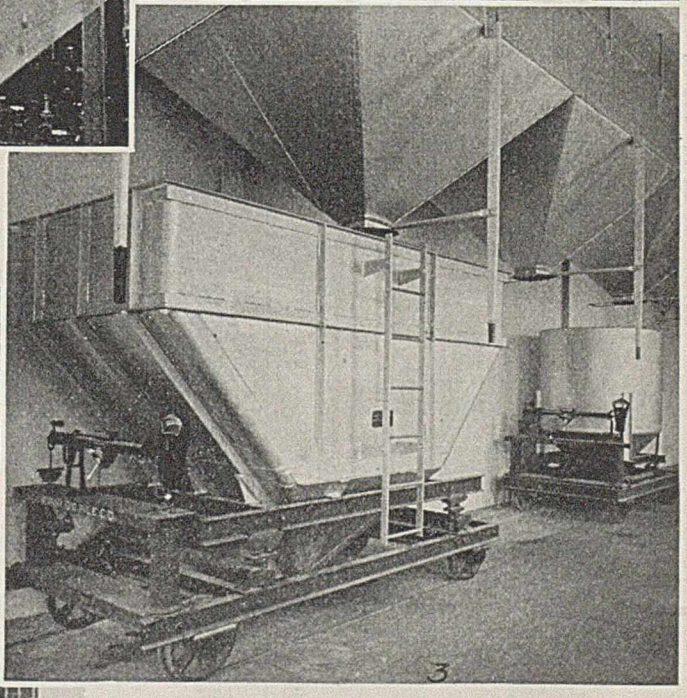


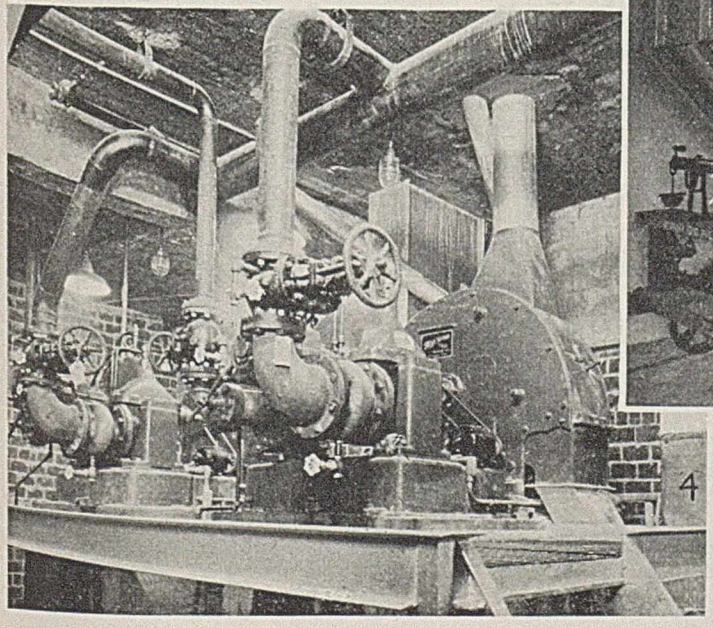
1. Section of Control Room Showing Control Board and Tail Boxes

Courtesy, Shenley Products Company, Inc.



2. Yeast Tubs with Agitators





3. Large and Small Grain Meal Hoppers with Weighing Scales

4. Modern Grain-Grinding Equipment Driven by Steam Turbine

Nos. 2, 3, and 4 are reproduced through the courtesy of the Old Crow Distillery of the W. A. Gaines & Company Division, National Distillers Corporation.

Table II. Analyses of Whisky Samples

1a. Serial No. barrel, 105. Date of inspection, Dec. 23, 1929. Sweet mash bourbon, no quick-ager. Composition of mash: 556 bu. corn, 11 bu. rye, 66 bu. malt. Yield: 2598 proof gal., 4.1 gal. per bu. Distilled in continuous copper still and doubler. Standard white-oak charred cooperage. Stored in warehouse B, 5th floor, rick 14, bottom tier. Same location as when entered. Frame, ironclad warehouse. No heat. Proof of distillation, av. 140°.

Lab. No.	Approx. Age, Years	Date Sampled	Proof	Analysis, Grams per 100 Liters: Acids	Esters	Fusel oil	Solids	Alde-hydes	Fur-fural	Color, 0.5-In. Cell
83492	17 days	1/9/30	102.2	5.3	13.8	230.7	13.1	3.9	Trace	0.5
93689	0.5	7/9/30	101.6	31.9	18.2	244.3	61.6	5.9	0.9	4.4
2793	1	1/9/31	102.9	38.5	23.1	245.4	89.8	6.8	0.9	6.3
10205	1.5	7/14/31	103.3	44.7	24.7	240.4	106.0	7.7	0.9	7.3
17869	2	12/26/31	104.6	48.2	25.2	229.6	117.4	9.6	0.8	8.1
25780	2.5	6/28/32	105.1	51.4	28.4	237.7	125	10.3	0.9	9.0
32745	3	12/22/32	106.0	52.1	29.9	249.9	129.8	10.3	0.9	9.4
38344	3.5	6/26/33	107.6	54.1	34.4	241.3	132.5	10.7	0.9	9.8
43140	4	1/2/34	108.3	58.7	37.6	260.8	147.2	11.1	0.9	11.1
Calculated to original volume:										
43140	4	1/2/34	100.0	41.88	26.8	186				

Original gage 50.39 wine gal. (51.90 proof gal.); final gage 35.94 wine gallons (38.75 proof gal.).

1b. Serial No. barrel, 251. Date of inspection, Dec. 26, 1929. Composition of mash: 556 bu. corn, 11 bu. rye, 66 bu. malt. Yield: 1763 proof gal., 2.8 gal. per bu. Sweet mash bourbon, no quick-ager. Continuous copper still and doubler. Standard white-oak charred cooperage. Frame ironclad warehouse with no heat. Proof of distillation, av. 140°.

Lab. No.	Approx. Age, Years	Date Sampled	Proof	Analysis, Grams per 100 Liters: Acids	Esters	Fusel oil	Solids	Alde-hydes	Fur-fural	Color, 0.5-In. Cell
83493	14 days	1/9/30	102.2	17.6	14.7	145.5	14.8	3.8	Trace	0.5
93690	0.5	7/9/30	102.1	41.1	22.7	149	64.8	4.7	1.9	4.9
2794	1	1/9/31	103.0	52.4	29.9	154.6	100.7	5.4	1.9	7.3
10206	1.5	7/14/31	103.7	56.7	30.6	146.0	113.8	5.4	1.9	8.7
17870	2	12/26/31	105.4	60.4	33.4	141.1	121.7	5.3	1.9	10.4
25781	2.5	6/28/32	105.8	61.3	33.8	153.9	139.2	6.1	1.9	11.8
32746	3	12/22/32	106.5	68.2	35.5	157.8	142.3	6.1	1.9	12.2
38345	3.5	6/26/33	107.9	70.1	38.4	151.8	153.9	5.9	1.9	12.5
43141	4	1/2/34	109.2	73.1	43.5	162.1	164.3	7.3	2.2	13.3
Calculated to original volume:										
43141	4	1/2/34	100.0	51.5	30.67	104.1				

Original gage 51.87 wine gal. (53.42 proof gal.); final gage 36.56 wine gal. (39.78 proof gal.).
(Very low yield. Cooling pipe froze and burst; water seeped into mash causing poor fermentation.)

2a. Serial No. barrel, 3630. Date of inspection, April 4, 1930. Sour mash bourbon. Composition of mash: 258 bu. corn, 120 bu. rye, 52 bu. malt. Yield: 2012 proof gal. spirits, 468 proof gal. per bu. No quick-ager. Charred white-oak cooperage. Copper continuous still and doubler. Brick warehouse heated with hot air for about 2 months to 80° F. Proof of distillation, av. run 118°.

Lab. No.	Approx. Age, Years	Date Sampled	Proof	Analysis, Grams per 100 Liters: Acids	Esters	Fusel oil	Solids	Alde-hydes	Fur-fural	Color, 0.5-In. Cell
88291	New	4/4/30	100.8	8.3	17.5	158.8	11.3	2.4	None	0.5
99308	0.5	11/14/30	101.1	45.0	27.0	159.3	111.6	4.8	0.6	6.9
8208	1	5/26/31	101.3	48.6	29.5	156.3	122.6	4.7	0.6	7.9
14824	1.5	10/19/31	102.9	54.2	35.1	160.7	141.1	4.7	0.8	9.2
22467	2	4/4/32	103.4	56.9	35.7	159.2	138.5	4.6	0.8	9.2
30136	2.5	10/6/32	104.6	58.5	37.9	164.1	163.1	4.6	0.8	10.5
36252	3	4/13/33	105.3	59.3	36.8	175.5	164.3	4.6	0.8	10.4
40880	3.5	10/4/33	106.9	66.2	42.0	174.5	180.9	5.2	0.8	12.2
45320	4	4/13/34	107.1	67.2	44.4	175.9	188.5	6.0	0.8	13.1
Calculated to original volume:										
45320	4	4/13/34	100.0	51.0	33.67	133.5				

Original gage 47.32 wine gal. (47.79 proof gal.); final gage 35.92 wine gal. (38.37 proof gal.).

2b. Serial No. barrel, 3642. Date of inspection, April 4, 1930. Composition of mash: 258 bu. corn, 120 bu. rye, 52 bu. malt. Yield: 2012 gal. proof spirits, 4.68 gal. per bu. Sour mash bourbon. Copper continuous still and doubler. Standard white-oak cooperage. Brick warehouse, hot-air heated 2 months, 80° F. Proof of distillation, av. run 118°.

Lab. No.	Approx. Age, Years	Date Sampled	Proof	Analysis, Grams per 100 Liters: Acids	Esters	Fusel oil	Solids	Alde-hydes	Fur-fural	Color, 0.5-In. Cell
88292	New	4/4/30	100.5	9.6	17.5	155.8	13.9	2.4	None	0.5
99307	0.5	11/14/30	100.7	47.7	31.5	153.0	117.2	4.8	0.8	7.5
8209	1	5/26/30	101.2	50.4	28.7	160.8	122.5	5.5	0.8	8.9
14825	1.5	10/19/31	102.5	55	34.3	154.5	144.7	6.2	0.8	10.2
22468	2	4/4/32	103.1	59.4	38.4	159.6	144.9	6.9	0.8	10.2
30137	2.5	10/6/32	104.3	64.4	39.6	170.5	164.2	6.1	0.8	11.9
36253	3	4/13/33	104.9	64.3	40.3	163.6	173.5	6.1	0.8	11.9
40881	3.5	10/4/33	106.6	68.7	43.8	166.8	188.9	6.6	0.8	14.3
45321	4	4/3/34	106.8	70.3	47.4	189.1	193.3	6.7	0.8	14.5
Calculated to original volume:										
45321	4	4/3/34	100.0	52.27	35.27	145.25				

Original gage 47.71 wine gal. (48.18 proof gal.); final gage 35.5 wine gal. (37.57 proof gal.).

3. Serial No. barrel, 2691. Date of inspection, May 3, 1930. Composition of mash: 689 bu. rye, 172 bu. malt. Yield: 3740 proof gal., 4.34 gal. per bu. Sweet mash rye, no ager. Copper 3-chambered still with charge tank. New white-oak charred barrels. Brick steam-heated warehouse, av. temp., 75–80° F. Proof of distillation, 130° to 140°.

3a. Serial No. barrel, 2691

Lab. No.	Approx. Age, Years	Date Sampled	Proof	Analysis, Grams per 100 Liters: Acids	Esters	Fusel oil	Solids	Alde-hydes	Fur-fural	Color, 0.5-In. Cell
89997	3 days	5/6/30	100.2	9.5	15.8	83.2	7.6	20.8	0.6	0.5
170	0.5	11/13/30	101.4	43.8	26.0	89.4	99.2	22.7	1.0	7.7
7869	1	5/19/31	101.4	53.3	29.5	92.0	125.6	20.5	1.6	9.9
16896	1.5	11/3/31	102.8	58.4	31.7	96.7	146.1	21.8	1.4	10.2
24286	2	5/17/32	104.6	60.8	35.3	97.6	160.4	24.8	1.5	11.7
31397	2.5	11/3/32	105.6	65.9	37.1	96.6	174.2	22.7	2.1	12.3
36953	3	5/3/33	106.4	72.2	42.6	98.4	197.9	22.6	2.1	13.6
41870	3.5	11/2/33	108.6	74.0	46.2	95.6	196.6	22.1	2.0	15.2
46039	4	5/2/34	110.7	72.6	54.8	99.3	213.8	21.7	1.8	17.2
Calculated to original volume:										
46039	4	5/2/34	100.0	48.24	36.44	66.01				

Original gage 48.41 wine gal. (48.9 proof gal.); final gage 32.17 wine gal. (35.58 proof gal.).

3b. Serial No. barrel, 2751

Lab. No.	Approx. Age, Years	Date Sampled	Proof	Analysis, Grams per 100 Liters: Acids	Esters	Fusel oil	Solids	Alde-hydes	Fur-fural	Color, 0.5-In. Cell
89998	3 days	5/6/30	101.7	8.8	14.7	78.7	7.6	20.5	0.6	0.4
171	0.5	11/13/30	101.2	45.1	27.0	87.0	92.1	21.7	1.1	7.4
7870	1	5/19/31	101.3	49.8	30.4	93.0	114.7	20.5	1.4	8.9
16897	1.5	11/3/31	102.2	56.4	32.7	99.0	131.7	20.4	1.6	9.3
24287	2	5/17/32	104.2	58.7	36.3	90.4	145.3	21.5	1.5	10.1
31398	2.5	11/3/32	105.1	62.8	37.7	97.1	154.5	21.3	1.5	10.5
36954	3	5/3/33	107.0	63.2	41.1	92.2	182.9	19.4	1.7	11.7
41871	3.5	11/2/33	107.6	71.4	45.0	96.5	179.9	19.4	1.7	13.0
46040	4	5/2/34	109.1	71.5	47.6	96.0	194.5	20.5	1.7	14.9
Calculated to original volume:										
46040	4	5/2/34	100	53.53	35.63	71.86				

Original gage 47.96 wine gal. (48.44 proof gal.); final gage 35.9 wine gal. (39.39 proof gal.).

4. Date of inspection, Jan. 28, 1930. Composition of mash: 301 bu. corn, 64 bu. rye, 65 bu. malt. Sour mash bourbon, no ager. Continuous copper still and doubler. Charred white-oak cooperage. Yield: 1981 proof gal., 4.6 gal. per bu. Concrete and brick warehouse, steam-heated. Av. temp., 95° F. Proof of distillation, av. run 118°.

4a. Serial No. barrel, 1490

Lab. No.	Approx. Age, Years	Date Sampled	Proof	Analysis, Grams per 100 Liters: Acids	Esters	Fusel oil	Solids	Alde-hydes	Fur-fural	Color, 0.5-In. Cell
84639	3 days	1/31/30	100.5	9.0	21.6	189.2	8.8	10.3	None	0.5
94289	0.5	7/30/30	100.3	41.9	32.5	199.2	92.2	14.8	1.4	6.5
2792	1	1/30/31	101.2	49.8	32.6	199.2	111.9	12.7	1.2	8.0
10207	1.5	7/14/31	101.6	56.5	35.5	201.2	131.7	13.4	1.6	9.4
19462	2	1/29/32	102.3	58.6	37.2	206.3	146.7	14.1	2.0	10.8
27356	2.5	8/1/32	103.0	62.9	39.3	209.2	160.7	12.4	2.3	11.6
34020	3	1/28/32	103.8	64.2	39.4	204.3	161.5	13.9	2.7	11.6
39372	3.5	7/25/33	104.1	67.5	37.2	208	174.1	13.1	2.9	12.5
43820	4	1/30/34	106.0	71.3	39.0	219.2	186.2	13.2	3.0	14.1
Calculated to original volume:										
43820	4	1/30/34	100.0	52.72	28.84	162.0				

Original gage 48.54 wine gal. (49.03 proof gal.); final gage 35.88 wine gal. (37.97 proof gal.).

4b. Serial No. barrel, 1491

Lab. No.	Approx. Age, Years	Date Sampled	Proof	Analysis, Grams per 100 Liters: Acids	Esters	Fusel oil	Solids	Alde-hydes	Fur-fural	Color, 0.5-In. Cell
84640	3 days	1/31/30	100.0	9.0	21.6	194.4	8.0	7.2	0	0.5
94290	0.5	7/30/30	99.8	42.1	32.7	203.9	94.2	12.8	2.2	6.5
2791	1	1/30/31	100.5	48.9	33.3	195.2	119.8	15.1	2.0	8.0
10208	1.5	7/14/31	101.1	56.4	36.5	190.6	131.1	13.8	1.6	9.4
19463	2	1/29/32	102.0	60.5	38.7	214.1	151.5	14.1	1.6	10.9
27357	2.5	8/1/32	102.6	63.2	39.9	196.5	162.8	13.3	2.0	12.2
34021	3	1/28/33	103.1	65.8	43.1	199.8	163.8	13.2	1.9	12.1
39273	3.5	7/25/33	103.4	68.5	40.8	204.3	181.6	14.7	2.3	13.2
43821	4	1/30/34	105.0	72.6	41.5	222.1	193.5	14.5	2.3	14.3
Calculated to original volume:										
43821	4	1/30/34	100.0	52.8	30.2	161.6				

Original gage 48.74 wine gal. (49.22 proof gal.); final gage 35.46 wine gal. (37.18 proof gal.).

5a. Serial No. barrel, 3570. Date of inspection, April 26, 1930. Composition of mash: 465 bu. rye, 109 bu. rye malt, 10 bu. rye in yeast, 8.4 bu. rye malt in yeast, 1.2 bu. barley malt in dona. Sweet mash rye, quick-aged. Yield: 2531 proof gal., 4.08 gal. per bu. Three-chambered still with doubler. White-oak charred cooperage. Brick warehouse. Temp. in still, 204° F. Proof of distillation, 115°.

91672	New	4/22/30	101.6	8.3	23.3	124.7	2.8	3.2	1.1	0
91673	Same, quick-aged	4/22/30	101.4	15.4	20.8	118.1	26.7	3.9	1.4	3.5
98607	0.5	10/22/30	101.5	52.0	31.2	126.6	116.7	7.1	3.0	9.9
7211	1	4/22/31	102.1	63.5	37.9	131.0	133.3	7.8	3.7	12.2
16785	1.5	11/10/31	102.8	71.2	41.1	138.7	160.7	7.4	3.8	13.6
23787	2	4/22/32	104.0	73.8	42.3	143.8	166.7	6.9	2.5	13.9
31307	2.5	10/22/32	104.8	79.0	50.4	120.1	180.6	6.9	3.4	15.3
36603	3	4/22/33	106.2	81.3	53.8	126.7	186.8	6.8	3.6	15.3
41655	3.5	10/22/33	107.0	80.7	55.5	125.8	189.5	6.7	3.4	15.9
45984	4	5/10/34	108.6	84.0	58.3	128.8	195.2	7.0	3.3	16.8
Calculated to original volume:										
45984	4	5/10/34	100.0	64.25	44.63	98.57				

Original gage 48.22 wine gal. (48.7 proof gal.); final gage 36.9 wine gal. (40.13 proof gal.).

5b. Serial No. barrel, 3831. Date of inspection, April 26, 1930. Composition of mash: 465 bu. rye, 109 bu. rye malt, 10 bu. rye in yeast, 8.4 bu. rye malt in yeast, 1.2 bu. barley malt in dona. Sweet mash rye, quick-aged. Yield: 2570 proof gal., 4.15 proof gal. per bu. Three-chambered still with doubler. White-oak charred cooperage. Brick warehouse. Av. temp., 70–79° F. throughout year. Temp. in still, 204° F. Proof of distillation, 115°.

91674	New	4/26/30	101.5	5.9	20.8	119.6	4.3	2.4	1.2	0
91675	New, quick-aged	4/26/30	101.2	16.6	18.3	113.0	27.9	3.2	1.8	3.5
98608	0.5	10/26/30	101.9	52.4	26.8	120.9	106.3	3.9	2.4	9.3
7212	1	4/26/31	102.7	60.8	35.2	127.7	121.7	4.3	2.1	11.7
15174	1.5	10/26/31	103.6	67.2	35.7	130.8	147.7	7.7	2.0	12.1
23788	2	4/26/32	104.6	67.7	37.0	132.9	156.8	7.1	1.9	12.9
31308	2.5	10/22/32	105.0	70.8	42.7	112.3	166.2	7.3	2.1	13.8
36604	3	4/26/33	106.8	73.6	47.8	114.5	173.8	7.1	3.0	14.5
41656	3.5	10/26/33	108.1	74.6	44.8	111.5	179.2	7.0	2.8	15.3
45985	4	5/10/34	108.2	76.5	48.8	135	187.1	7.0	3.1	16.2
Calculated to original volume:										
45985	4	5/10/34	100	56.6	36.1	99.9				

Original gage 48.09 wine gal. (48.57 proof gal.); final gage 35.58 wine gal. (39.02 proof gal.).

6. Date of inspection, Dec. 14, 1929. Composition of mash: 1188 bu. rye, 312 bu. malt. Yield: 6528 proof gal., 4.35 gal. per bu. Sour mash rye, quick-aged by steam coils with expansion tanks. Continuous copper still and doubler. White-oak charred cooperage. Seven-story metal, hot-air-heated warehouse. Av. temp., 72° F.

6a. Serial No. barrel, 2797

83218	25 days	1/8/35	101.9	28.3	15.5	126.2	54.9	7.9	0.4	6.9
93681	0.5	7/8/30	101.8	47.2	25.2	129.7	105.0	10.2	0.8	8.8
2073	1	1/8/31	102.5	58.5	30.9	121.9	129.4	10.1	1.6	10.2
9907	1.5	7/7/31	103.2	62.8	31.5	127.5	147.5	10.5	1.7	11.6
17442	2	12/14/31	104.2	65.6	33.8	134.1	156.9	10.7	2.1	13.4
25354	2.5	6/15/32	104.1	72.6	35.9	132.3	176.9	11.5	1.9	13.9
32585	3	12/17/32	105.2	74.1	41.4	139.3	181.3	11.8	1.9	15.7
38034	3.5	6/13/33	105.8	74.8	41.6	144.4	183.7	11.3	2.1	16.1
42615	4	12/11/33	106.9	78.6	48.3	146.4	193.4	11.8	1.8	17.3
Calculated to original volume:										
42615	4	12/11/33	100.0	58.11	35.7	96.05				

Original gage 47.9 wine gal. (48.38 proof gal.); final gage 35.43 wine gal. (38.14 proof gal.).

6b. Serial No. barrel, 2798

83219	25 days	1/8/30	101.7	28.3	17.2	121.5	59.4	8.7	1.2	6.4
93682	0.5	7/8/30	101.6	47.2	26.8	123.0	112.0	10.2	1.4	9.4
2074	1	1/8/31	102.5	56.2	31.8	121.8	135.1	10.1	1.4	10.2
9908	1.5	7/7/31	103.4	60.9	33.2	124.7	145.4	10.8	1.6	11.6
17443	2	12/14/31	103.8	64.7	34.3	133.9	160.8	11.0	1.7	13.5
25355	2.5	6/15/32	104.8	68.7	36.1	138.6	171.0	11.5	1.9	13.8
32586	3	12/17/32	105.9	71.4	42.4	137.4	180.2	11.7	1.9	14.6
38035	3.5	6/16/33	106.8	71.4	41.2	139.8	184.3	11.5	1.8	15.0
42614	4	12/11/33	107.6	74.7	48.2	132.6	186.7	11.1	2.2	15.8
Calculated to original volume:										
42614	4	12/11/33	100.0	55.5	35.83	96.49				

Original gage 46.81 wine gal. (47.27 proof gal.); final gage 34.78 wine gal. (37.43 proof gal.).

7. Date of inspection, Jan. 7, 1930. Composition of mash: 1155 bu. corn, 297 bu. rye, 198 bu. malt. Sour mash bourbon, quick-aged by heating to certain temp. for 2.5 hours. Yield: 7715 proof gal., 4.68 proof gal. per bu. White-oak charred barrels. Continuous still and doubler. Wooden-frame, metal-clad warehouse heated by fan system. Temp. about 65° F. during winter months.

7a. Serial No. barrel, 5967

83217	1 day	1/8/30	101.2	11.9	14.4	124.4	33.6	5.53	1.0	3.5
93683	0.5	7/8/30	101.7	36.6	21.2	135.0	94.4	7.9	2.0	7.4
2071	1	1/8/31	102.7	40.9	21.9	141.4	113.3	8.6	1.5	9.7
9905	1.5	7/8/31	104	47.3	22.4	130.3	126.0	9.2	1.5	10.6
18512	2	1/8/32	104.7	51.6	27.7	128.6	135.2	10.7	1.5	11.5
26027	2.5	7/6/32	105.8	53.3	28.3	139.0	145.6	11.3	1.8	12.8
33277	3	1/6/33	106.5	54.1	32.6	142.9	146.2	9.8	2.2	13.1
38690	3.5	7/6/33	106.9	58.4	32.9	148.1	159.0	9.7	1.9	13.1
43144	4	1/9/34	108.4	59.8	39.4	144.5	158.5	9.2	2.2	13.4
Calculated to original volume:										
43144	4	1/9/34	100.0	47.62	31.36	115.09				

Original gage 47.74 wine gal. (49.22 proof gal.); final gage 38.81 wine gal. (41.12 proof gal.).

7b. Serial No. barrel, 5968

83216	1 day	1/8/30	101.3	5.9	15.6	127.7	11.3	2.4	0	0
93684	0.5	7/8/30	101.3	39.1	22.6	138.1	88.6	7.1	0.9	5.9
2072	1	1/8/31	102.2	47.0	26.3	132.8	101.2	8.6	1.0	6.4
9906	1.5	7/8/31	103.2	51.2	29.0	129.0	113.0	7.7	1.2	7.3
18511	2	1/8/32	103.8	54.9	29.7	127.1	124.4	9.6	1.2	7.7
26026	2.5	7/6/32	105.2	57.0	32.6	138.9	133.5	9.9	1.2	8.6
33278	3	1/6/33	105.9	60.1	37.2	140.5	134.9	10.6	1.3	9.5
38691	3.5	7/6/33	106.2	62.1	34.8	155.8	146.8	10.6	1.1	9.9
43145	4	1/9/34	107.6	63.6	43.3	145.6	141.8	9.7	1.3	10.2
Calculated to original volume:										
43145	4	1/9/34	100.0	47.79	32.58	109.43				

Original gage 48.86 wine gal. (49.35 proof gal.); final gage 36.73 wine gal. (39.6 proof gal.).

8. Date of inspection, May 5, 1930. Composition of mash, 808 bu. rye, 142 bu. malt, sweet mash rye, no quick-ager. Yield: 4160 gal. proof spirit, 4.27 gal. per bu. Copper still, 3 chambers with charge tub (with doubler); capacity, 24,000 gal. New white-oak charred cooperage. Brick warehouse, heated by steam. Av. temp. winter and summer, 75–80° F. Proof of distillation, av. 138°.

8a. Serial No. barrel, 424153

89999	1 day	5/6/30	100.0	6.0	16.7	205.1	3.2	11.3	3.2	Colorless
169	0.5	11/5/30	100.7	41.7	27.5	228.0	94.1	12.7	3.0	7.9
7867	1	5/19/31	101.2	53.4	34.8	201.0	120.2	13.4	4.4	8.4
16894	1.5	11/5/31	102.3	56.3	36.6	215.9	131.4	14.6	3.9	9.3
24284	2	5/5/32	104.4	62.1	37.1	217.5	150.8	13.6	4.2	10.5
31395	2.5	11/5/32	106.1	65.1	43.1	218.9	150.7	13.8	4.0	12.2
36951	3	5/7/33	107.4	67.6	43.9	212.3	172.3	13.8	3.7	12.6
41868	3.5	11/4/33	109.0	69.3	42.8	213.9	170.4	13.9	3.9	13.3
46037	4	5/4/34	110.6	69.4	48.5	223.6	171.8	14.1	3.8	13.6
Calculated to original volume:										
46037	4	5/4/34	100.0	50.4	35.2	162.3				

Original gage 45.84 wine gal. (46.3 proof gal.); final gage 33.28 wine gal. (36.39 proof gal.).

8b. Serial No. barrel, 424154

90000	1 day	5/6/30	100.0	6.6	16.7	222.7	3.2	12.0	4.8	Colorless
168	0.5	11/5/30	100.3	44.3	29.0	224.6	91.7	14.4	4.8	7.5
7868	1	5/19/31	100.7	47.6	33.2	225.3	116.5	14.3	5.2	8.4
16895	1.5	11/5/31	102.5	58.5	38.2	218.1	140.1	15.6	5.1	9.8
24285	2	5/5/32	103.5	59.1	38.7	226.9	143.1	16.2	5.0	10.8
31396	2.5	11/5/32	105.2	62.7	41.0	220.0	154.0	16.7	4.9	11.9
36952	3	5/7/33	105.9	64.6	43.2	220.1	162.9	16.6	4.9	11.8
41869	3.5	11/4/33	107.3	67.1	45.9	217.3	166.8	17.1	4.5	13.0
46038	4	5/4/34	108.9	67.2	47.7	225.0	172.1	17.6	4.4	13.0
Calculated to original volume:										
46038	4	5/4/34	100.0	49.1	34.8	164.3				

Original gage 45.78 wine gal. (46.24 proof gal.); final gage 33.44 wine gal. (33.66 proof gal.).

(Table II continued on next page)

Table II (*Continued*)

Lab. No.	Approx. Age, Years	Date Sampled	Proof	Acids	Esters	Fusel oil	Solids	Aldehydes	Furfural	Color, 0.5-In. Cell
9. Date of inspection, Jan. 8, 1930. Composition of mash, 556 bu. corn, 11 bu. rye, 66 bu. malt. Sweet mash bourbon, no quick-ager. Yield: 2372 gal. proof spirit, 3.74 gal. per bu. Continuous copper still and doubler. White-oak charred cooperage. Brick warehouse. No heat.										
9a. Serial No. barrel, 662										
83494	5 days	1/13/30	102.6	9.35	15.4	176.7	19.3	4.9	Trace	0.5
93687	0.5	7/14/30	102.9	35.0	20.5	167.6	62.4	5.8	1.3	4.4
2075	1	1/10/31	103.3	46.5	23.9	177.2	89.9	7.7	1.0	6.3
9909	1.5	7/2/31	103.9	49.7	25.4	174.5	98.4	5.8	1.0	7.2
18513	2	1/6/32	104.5	56.3	29.1	176.8	113.7	5.7	1.4	8.1
26028	2.5	7/8/32	105.3	55.9	29.3	179.7	132.6	5.7	1.4	9.5
33229	3	1/9/33	106.1	59.4	30.3	214.0	140.7	5.7	1.4	9.9
38686	3.5	7/5/33	106.7	60.2	30.5	191.3	155.0	5.6	1.4	9.8
43146	4	1/8/34	108.1	65.5	39.9	188.0	159.4	9.3	1.4	11.1
Calculated to original volume:										
43146	4	1/8/34	100	50.1	30.5	143.8				
Original gage 50.26 wine gal. (51.7 proof gal.); final gage 38.46 wine gal. (41.24 proof gal.).										
9b. Serial No. barrel, 663										
83495	5 days	1/13/30	102.5	9.4	13.7	185.4	20.1	4.9	Trace	0.5
93688	0.5	7/14/30	102.8	35.0	20.5	169.3	65.7	5.8	1.2	4.9
2076	1	1/10/31	103.2	46.5	23.9	178.1	96.9	5.8	1.3	6.8
9910	1.5	7/2/31	103.4	49.9	25.5	170.2	100.0	5.8	1.3	7.7
18514	2	1/6/32	104.2	56.5	32.1	168.9	117.3	5.8	1.6	8.6
26029	2.5	7/8/32	105.1	54.8	30.2	190.1	135.7	5.7	1.4	9.9
33280	3	1/9/33	106.1	58.8	28.5	206.5	141.0	5.6	1.4	9.9
38687	3.5	7/15/33	106.7	58.9	29.1	187.6	158.1	7.5	1.4	10.4
43147	4	1/8/34	107.9	64.0	39.2	187.6	159.7	9.6	1.4	11.6
Calculated to original volume:										
43147	4	1/8/34	100.0	49.47	30.29	145.14				
Original gage 50.06 wine gal. (51.57 proof gal.); final gage 38.72 wine gal. (42.11 proof gal.).										
10. Date of inspection, May 2, 1930. Composition of mash, 420 bu. corn, 90 bu. rye, 90 bu. malt. Yield: 2745 gal. proof spirit, 5.49 gal. per bu. Sour mash bourbon, quick-aged in hothouse for several days at 100° F. Continuous-type still; capacity, 600 bu. with doubler of 4825 gal. White-oak charred cooperage. Brick warehouse, unheated. Proof of distillation, 130°.										
10a. Serial No. barrel, 1434										
93692	25 days	5/27/30	101.3	33.2	26.9	172.0	70.6	7.9	2.4	5.9
2789	0.5	1/24/31	102.0	49.4	32.8	177.8	121.9	9.4	2.9	9.3
8206	1	5/27/31	102.1	50.5	35.3	182.7	129.8	9.4	2.7	10.8
14826	1.5	10/16/31	103.6	61.4	36.5	195.4	159.9	10.0	3.1	13.5
23615	2	5/2/32	103.8	62.4	37.3	185.7	166.1	11.6	3.0	14.5
31309	2.5	11/8/32	105.5	69.4	40.9	183.2	183.2	12.9	3.0	15.6
36866	3	5/1/33	105.7	68.1	37.9	174.9	191.8	12.9	2.8	15.6
41720	3.5	11/2/33	106.9	71.3	40.3	174.5	207.4	12.0	3.0	16.4
45884	4	4/30/34	107.1	74.9	45.2	208.3	206.7	13.4	2.4	17.7
Calculated to original volume:										
45884	4	4/30/34	100.0	57.9	34.9	160.9				
Original gage 48.79 wine gal. (49.76 proof gal.); final gage 37.69 wine gal. (40.91 proof gal.).										
10b. Serial No. barrel, 1435										
93691	25 days	5/27/30	101.0	31.2	28.1	179.8	78.4	12.7	2.8	6.9
2790	0.5	1/24/31	102.1	44.7	30.2	182.8	103.2	13.3	2.9	8.3
8207	1	5/27/31	103.0	46.4	33.6	184.5	111.6	13.6	2.5	9.3
14827	1.5	10/16/31	103.8	53.2	35.6	183.9	137.9	13.4	3.0	11.1
23616	2	5/2/32	104.8	58.4	37.8	177.2	144.7	13.0	2.9	12.4
31310	2.5	1/8/32	105.4	59.9	39.3	182.1	160.8	13.6	3.0	13.3
36867	3	5/1/33	105.7	63.6	35.4	188.2	163.9	14.0	2.7	13.3
41721	3.5	11/2/33	107.2	71.6	41.9	183.9	181.3	13.4	2.6	14.0
45885	4	4/30/34	107.3	73.8	39.4	213.2	182.4	13.4	2.4	15.4
Calculated to original volume:										
45885	4	4/30/34	100.0	57.1	30.4	164.8				
Original gage 47.5 wine gal. (48.45 proof gal.); final gage 36.72 wine gal. (38.88 proof gal.).										
11. Date of inspection, Jan. 3, 1930. Composition of mash, 301 bu. corn, 68 bu. rye, 61 bu. malt. Sour mash bourbon, no quick-ager. Continuous copper still and boubler. White-oak charred cooperage. Wooden, ironclad warehouse. No heat. Proof of distillation, 125°.										
11a. Serial No. barrel, 649										
83220	4 days	1/7/30	100.5	6.6	16.6	199.6	15.7	5.6	Trace	1.0
93685	0.5	7/7/30	99.7	35.9	25.1	221.5	73.4	8.8	1.4	5.5
2069	1	1/6/31	100.5	49.0	26.3	212.8	107.3	11.1	2.2	7.5
10203	1.5	7/14/31	100.7	54.8	30.6	226.3	120.5	11.1	2.2	8.4
18416	2	11/4/32	101.3	63.4	33.9	232.0	150.3	11.4	2.2	9.9
26024	2.5	7/6/32	101.9	64.8	37.1	212.4	152.5	11.8	2.3	10.8
33275	3	1/6/33	102.4	68.0	38.2	215.7	154.5	12.1	2.5	11.7
38688	3.5	7/6/33	102.9	69.4	39.6	209.6	171.9	12.1	2.1	13.3
43142	4	1/4/34	104	74.4	44.0	213.3	170.4	11.2	2.7	14.1
Calculated to original volume:										
43142	4	1/4/34	100.0	57.3	33.9	164.1				
Original gage 47.32 wine gal. (47.79 proof gal.); final gage 36.42 wine gal. (38.47 proof gal.).										
11b. Serial No. barrel, 650										
83221	4 days	1/7/30	100.6	6.6	16.7	213.5	19.9	6.0	Trace	1.0
93686	0.5	7/7/30	100.3	31.7	25.4	219.4	67.6	8.0	1.8	5.0
2070	1	1/6/31	101.0	44.0	27.0	210.0	103.4	8.7	1.6	6.9
10204	1.5	7/14/31	102	48.2	29.3	218.3	113.6	9.4	1.6	7.8
18417	2	1/4/32	102.9	52.5	31.6	219.8	132.2	9.3	1.6	8.8
26025	2.5	7/6/32	103.3	55.8	34.0	212.1	141.0	10.1	1.7	9.7
33276	3	1/6/33	104.1	57.6	33.8	210.4	139.8	10.8	2.1	9.6
38689	3.5	7/6/33	104.3	61.0	32.1	225.3	159.9	11.5	2.3	10.5
43143	4	1/4/34	106.1	65.0	43.1	220.6	164.2	10.9	2.8	12.3
Calculated to original volume:										
43143	4	1/4/34	100.0	50.6	33.5	171.6				
Original gage 48.03 wine gal. (48.51 proof gal.); final gage 37.35 wine gal. (39.53 proof gal.).										

(Analysis columns: Grams per 100 Liters.)

instances strong positive reactions in the lower layer with these entirely genuine samples of naturally colored whisky. Williams' reagent (a mixture of amyl alcohol, toluene, and tartaric acid solution used in conjunction with the Williams' test, *5*) gives a slight color in the lower layer with the 3-year-old and older whiskies; hence, it is considered inferior to the Marsh reagent for the detection of artificial color. A genuine naturally aged whisky should give a negative or practically negative test with Marsh's reagent. Experience in this laboratory has shown that a positive Marsh reaction can be either due to caramel, to some few coal-tar dye colors, or to uncharred or imperfectly charred white-oak chips. On the other hand, a negative Marsh reaction does not indicate the absence of artificial color because certain coal-tar dyes in whisky react negatively to the Marsh reagent. However, the shade of the color of the whisky, as examined in the Lovibond tintometer, is likely to appear off-shade to the experienced whisky analyst; hence it may be detected and confirmed by the double dying test of the A. O. A. C. or other simple means of identification. A positive Marsh reaction, due to uncharred white-oak chips, should be submitted to the modified Marsh test (*3*) in order to distinguish between the color produced by caramel and uncharred white-oak chips.

History of Samples

Eleven distilleries were represented and 2 barrels of whisky were set aside at each distillery for these experiments. The distilleries were numbered 1 to 11 to distinguish one from the other, and the barrels were lettered *a* and *b* to distinguish between the two barrels from the same distillery. The 22 barrels can be further distinguished from each other by the serial number which was assigned to each by the U. S. storekeeper-gager in charge.

The following information was obtained concerning each sample, and the data precede each series of analyses (Table

TABLE III. LOSS OF WINE AND PROOF GALLONS IN 4-YEAR STORAGE

Distillery No.	Pkg. Serial No.	Original Volume W. G.[a]	Original Volume P. G.[b]	Net Contents Left W. G.	Net Contents Left P. G.	Lost by Leakage and Evapn. W. G.	Lost by Leakage and Evapn. P. G.	Per Cent Loss by Leakage and Evapn. W. G.	Per Cent Loss by Leakage and Evapn. P. G.	Samples Taken W. G.	Samples Taken P. G.
1a	105	50.39	51.9	35.94	38.75	14.45	13.14	28.67	25.32	2.25	2.37
1b	251	51.87	53.42	36.56	39.78	15.31	13.64	29.52	25.53	2.25	2.38
2a	3630	47.32	47.79	35.92	38.37	11.4	9.42	24.10	19.71	2.25	2.34
2b	3642	47.71	48.18	35.50	37.57	12.21	10.61	25.59	22.04	2.25	2.33
3a	2691	48.41	48.9	32.17	35.58	16.24	13.32	33.55	27.24	2.25	2.37
3b	2751	47.96	48.44	35.90	39.39	12.06	9.05	25.15	18.68	2.25	2.37
4a	1490	48.54	49.03	35.88	37.97	12.66	11.06	26.08	22.56	2.25	2.32
4b	1491	48.74	49.22	35.46	37.18	13.28	12.04	27.25	24.46	2.25	2.31
5a	3570	48.22	48.7	36.90	40.13	11.32	8.57	23.48	17.6	2.25	2.36
5b	3831	48.09	48.57	35.58	39.02	12.51	9.55	26.02	19.67	2.25	2.36
6a	2797	47.90	48.38	35.43	38.14	12.47	10.24	26.03	21.16	2.5	2.57
6b	2798	46.81	47.27	34.78	37.43	12.03	9.84	25.69	20.82	2.50	2.57
7a	5967	48.74	49.22	38.81	41.12	9.93	8.10	20.37	16.46	2.25	2.36
7b	5968	48.86	49.35	36.73	39.60	12.13	9.75	24.83	19.76	2.25	2.35
8a	424153	45.84	46.30	33.28	36.39	12.56	9.91	27.40	21.41	2.25	2.37
8b	424154	45.78	46.24	33.44	33.66	12.34	9.58	26.96	20.72	2.25	2.35
9a	662	50.26	51.7	38.46	41.84	11.8	9.86	23.48	19.07	2.25	2.37
9b	663	50.06	51.57	38.72	42.11	11.39	9.46	22.65	18.34	2.25	2.36
10a	1434	48.79	49.76	37.69	40.91	11.10	8.85	22.75	17.79	2.25	2.34
10b	1435	47.50	48.45	36.72	38.88	10.78	9.57	22.70	19.76	2.25	2.34
11a	649	47.32	47.79	36.42	38.47	10.9	9.32	23.04	19.51	2.25	2.31
11b	650	48.03	48.51	37.35	39.53	10.18	9.98	22.24	18.52	2.25	2.32

[a] Wine gallons. [b] Proof gallons.

TABLE IV. GAIN OVER 4-YEAR PERIOD USING CALCULATED-TO-PROOF DATA

Mash	Distillery No.	Serial No.	Analysis, Grams/100 Liters — Proof	Acids	Esters	Fusel oil	Solids	Color
Sweet, bourbon	1a	105	6.1	53.4	23.8	30.2	134	10.6
	1b	251	7.0	55.5	28.8	16.5	150	12.4
Sour, bourbon	2a	3630	6.3	58.9	26.9	17.1	177.2	12.6
	2b	3642	6.2	60.7	29.9	39.4	179.4	14.0
Sweet, rye	3a	2691	10.5	63.1	39	6.14	206.2	16.7
	3b	2751	7.4	62.7	32.9	17.3	187	13.5
Sour, bourbon	4a	1490	5.5	62.4	17.6	31.0	177.5	13.7
	4b	1491	5.0	63.6	19.9	27.7	185.5	13.8
Sweet, rye	5a	3570	7.0	75.6	35.0	4.1	192.3	16.7
	5b	3831	6.7	70.6	28.0	15.4	182.7	16.2
Sour, rye	6a	2797	5.7	49.9	32.7	3.7	138.5	10.4
	6b	2798	5.3	46.4	31.0	0.7	127.3	9.4
Sour, bourbon	7a	5967	7.2	47.9	25.0	20.2	124.9	9.9
	7b	5968	6.3	57.6	27.7	17.9	130.7	10.2
Sweet, rye	8a	424153	10.6	63.4	31.8	18.5	168.6	13.6
	8b	424154	8.9	60.6	30.9	2.3	168.9	12.8
Sweet, bourbon	9a	662	5.5	56.1	24.5	11.3	140.1	10.6
	9b	663	5.4	54.6	25.4	2.3	139.5	11.1
Sour, bourbon	10a	1434	6.0	41.8	18.3	26.3	136.1	11.8
	10b	1435	6.2	42.6	11.3	33.4	104	8.5
Sour, bourbon	11a	649	3.5	67.8	27.4	13.7	154.7	13.1
	11b	650	3.5	58.5	26.5	7.1	144.3	11.2
Average			6.53	57.89	27.01	16.83	156.77	12.4

II): composition of mash; gallons of proof spirits produced; yield of gallons of proof spirits for each bushel of grain; sweet or sour mash; kind of spirits, rye or bourbon; description of still in which spirits were manufactured, as type, capacity, doubler, etc.; if quick-aging was applied to the spirits before warehousing; description of the cooperage used, such as new, re-used, white oak, charred, etc.; whether the spirits were changed from the original place of storage and the conditions of the new warehouse; whether the warehouse was of brick, wood, or metal construction, giving humidity if possible, and the average temperature, summer and winter; whether the warehouse was heated and by what means; the original gage of barrels, and the regage at the expiration of 4 years using Gager's Form 1520.

Quick-Aging

On account of the extensive practice of quick-aging whisky before it goes on the market at the present time, the significance of the usual analytical figures has been changed to some extent. The most significant and at the same time the most easily ascertained chemical figure was at one time the acids, but owing to the prevalence of quick-aging, the acid content has become less important than the esters in indicating age. The esters show little change or increase by these processes. Cheaper whisky that is to be sold at once, or soon after manufacture, is at present almost invariably quick-aged by one of the several processes used. All of the treatments using heat with chips or charred barrels increase the acids, solids, color, and furfural. The same proportion of uncharred chips produces a higher solid and acid content than the charred or toasted chips, but there is less color and ash. The acids that are added to whisky by these processes are usually nonvolatile, but some volatile acids are obtained. These treatments have no effect on the content of fusel oil, and the aldehydes are but slightly increased. The color shows the most pronounced change. Almost any depth of color can be easily produced, but as mentioned before it is off-shade.

QUICK-AGING EXPERIMENT. Two barrels of bourbon whisky made from mash of the same composition and consisting of part of the same distillation were set aside in the warehouse and aged together under the same storage conditions. The two whiskies were identical and were handled throughout in the same manner, with the exception that barrel 5967 was quick-aged by heating it at a certain temperature for 2.5 hours, and after it became cool it was placed beside the untreated barrel. Although this treatment would be considered mild in comparison with the usual quick-aging processes employed, Table VI shows that there was some effect upon the whisky. This quick-aging process increased the acids, solids, color, and furfural at the start. The color, solids, and furfural maintained the ratio of increase until the end of the 4-year period. There was no difference in the proof, which was 101 at the start and 108 at the end of the 4 years for each barrel. The fusel oil in both of these samples was approximately the same, and they both showed about the same gradual increase at the end of the 4-year period.

Distillery 5 had samples taken of their two serial numbers before they were quick-aged and immediately afterwards. Table VII shows the analysis for comparison. Samples 91672 and 91674 were taken before the quick-aging treatment, and samples 91673 and 91675 were treated by immersing a steam coil in the package for 3 hours at a temperature not over 160° F. Table VII shows that this form of quick-aging increases the solids, acids, and color, and that there is apparently a decrease in fusel oil and esters.

TOTAL ACIDS. The acid determination is one of the sim-

TABLE V. LOSS OVER 4-YEAR PERIOD

Serial No. of Barrel	Wine Gallons (and Proof)[a] Original	Wine Gallons (and Proof)[a] Final	In Barrel: Grams fusel oil Before	In Barrel: Grams fusel oil After	In Barrel: Wine gal. pure alcohol Before	In Barrel: Wine gal. pure alcohol After	Loss: Total wine gal. liquid	Loss: Grams fusel oil	Loss: Wine gal. H_2O	Loss: Wine gal. alcohol
(Corn) 1a, 105	(100.2) 50.39	(108.3) 35.94	449.8	384.34	25.75	19.46	14.45	65.46	8.16	6.29
(Corn) 1b, 251	(102.2) 51.87	(109.2) 36.56	292.0	244.8	26.51	19.96	15.31	47.2	8.76	6.55
(Corn) 2a, 3630	(100.8) 47.32	(107.1) 35.92	287.0	256.3	23.85	19.24	11.40	30.7	6.79	4.61
(Corn) 2b, 3642	(100.5) 47.71	(106.8) 35.50	282.8	280.1	23.97	18.96	12.21	2.7	7.2	5.01
(Rye) 3a, 2691	(100.2) 48.41	(110.7) 32.17	152.8	135.2	24.25	16.2	16.24	17.6	8.19	8.05
(Rye) 3b, 2751	(101.7) 47.96	(109.1) 35.90	145.2	142.3	24.39	19.56	12.06	2.9	7.23	4.83
(Corn) 4a, 1490	(100.5) 48.54	(106.0) 35.88	347.6	315.5	24.39	19.02	12.66	32.1	7.29	5.37
(Corn) 4b, 1491	(100.0) 48.74	(105) 35.46	358.9	313.0	24.37	18.62	13.28	45.9	7.53	5.75
(Rye) 5a, 3570	(101.6) 48.22	(108.6) 36.90	218.5	195.4	24.50	20.04	11.32	23.1	6.86	4.46
(Rye) 5b, 3831	(101.5) 48.09	(108.2) 35.58	208.3	196.8	24.61	19.25	12.51	17.5	7.15	5.36
(Rye) 6a, 2797	(101.9) 47.9	(106.9) 35.43	233.2	186.0	24.40	18.94	12.47	47.2	7.01	5.46
(Rye) 6b, 2798	(101.1) 46.81	(107.6) 34.78	233.7	174.6	23.81	18.71	12.03	59.1	6.93	5.10
(Corn) 7a, 5967	(101.2) 48.74	(108.4) 38.81	232.1	230.1	24.66	21.03	9.93	2.0	6.30	3.63
(Corn) 7b, 5968	(101.3) 48.86	(107.6) 36.73	239.3	217.7	24.75	19.76	12.13	21.6	7.14	4.99
(Rye) 8a, 424153	(100.0) 45.84	(110.6) 33.28	355.7	311.6	22.92	18.4	12.56	44.1	8.04	4.52
(Rye) 8b, 424154	(100.0) 45.78	(108.9) 33.44	385.8	310.1	22.89	18.21	12.34	75.7	7.67	4.67
(Corn) 9a, 662	(102.6) 50.26	(108.1) 38.46	344.9	296	25.78	20.79	11.8	48.9	6.81	4.99
(Corn) 9b, 663	(102.5) 50.06	(107.9) 38.72	360.1	296.1	25.66	20.89	11.34	64.0	6.57	4.77
(Corn) 10a, 1434	(101.3) 48.79	(107.1) 37.69	321.7	318.9	24.71	20.28	11.1	2.8	6.67	4.43
(Corn) 10b, 1435	(101.1) 47.50	(107.3) 36.72	329.1	318	24.01	19.7	10.78	11.1	6.47	4.31
(Corn) 11a, 649	(100.5) 47.32	(104.0) 36.42	359.3	305.8	23.78	18.94	10.9	53.54	6.06	4.84
(Corn) 11b, 650	(100.6) 48.03	(106.0) 37.35	390.4	331	24.16	19.79	10.68	59.37	6.31	4.37

[a] The figures in parentheses are proofs.

employed on the whisky from these distilleries.

ESTERS. Since esters are not produced by the ordinary quick-aging processes, these congenerics present a reliable index to the age of the whisky. The method of analysis requires much care, and the details must be followed implicitly if the results are to be considered of utmost value. The principal ester present is ethyl acetate (acetic ether), but no doubt esters of the higher alcohols are also present, since it is impossible to obtain the characteristic aroma or bouquet of aged whisky with ethyl acetate alone.

In the chemical examination here involved, the A. O. A. C. official method was used, except that 10 cc. of the distillate were taken for aldehyde and furfural determinations.

SOLIDS. Solids (extract) are derived from the wood owing to contact of the whisky in storage, and they are usually of a definite character in authentic, regularly aged whisky. This is largely due to the uniform character of the standard charred white-oak barrels. During the aging period the whisky steadily increases in solids (extract), which will be quite regular under normal conditions. The largest amount of extract is obtained during the first 6 months of storage. During these months an average of 70 grams per 100 liters was extracted. During the next 6 months an average of 22 grams per 100 liters was extracted. During the 6-month periods thereafter the amounts extracted were progressively smaller and smaller, until between the 3.5- and 4-year period only 4 grams per 100 liters of solids were apparently extracted.

FUSEL OIL. The determination of fusel oil, or the higher alcohols, is considered one of the most

TABLE VI. DIFFERENCE IN SAME WHISKY WITH AND WITHOUT QUICK-AGING

Age	Total Acids 5967, Q. A.	Total Acids 5968	Esters 5967, Q. A.	Esters 5968	Fusel Oil 5967, Q. A.	Fusel Oil 5968	Solids 5967, Q. A.	Solids 5968	Color 5967, Q. A.	Color 5968	Furfural 5967, Q. A.	Furfural 5968
Years	*Grams per 100 liters calculated to proof*											
New	11.9	5.9	14.3	15.6	124.3	127.6	33.6	11.2	3.5	0	1.0	0
0.5	36.6	39.1	21.2	22.6	135	138.1	94.4	88.6	7.4	5.9	2.0	0.9
1	40.9	47.0	21.8	26.3	135.6	132.8	113.3	101.2	9.7	6.4	1.6	1.0
1.5	47.3	51.2	22.4	29	130.3	129.7	126	113	10.6	7.3	1.5	1.2
2	51.6	54.9	27.7	29.7	128.6	127.1	135.2	134.4	11.5	7.7	1.5	1.2
2.5	53.3	57.0	28.3	32.6	138.9	138.9	145.6	133.5	12.8	8.5	1.8	1.1
3	54.1	60.1	32.6	37.3	142.9	140.5	146.2	134.9	13.1	9.4	2.3	1.3
3.5	58.3	62.2	32.9	34.8	148.1	155.8	158.9	146.8	13.1	9.9	1.9	1.1
4	59.8	63.6	39.4	43.3	144.5	145.6	158.5	141.8	13.4	10.2	2.2	1.3

TABLE VII. EFFECT OF QUICK-AGING WITH STEAM COIL

Lab. No.	Treatment	Proof	Acids	Esters	Fusel Oil	Solids	Color
			Grams per 100 liters				
	Serial No. 3570						
91672	Not quick-aged	101.6	8.3	23.3	124.7	2.9	0
91673	Quick-aged	101.4	15.4	20.8	118.1	26.7	3.5
45984	(91673) after 4-yr. aging	108.6	84	58.3	128.8	195.2	16.8
	Serial No. 3831						
91674	Not quick-aged	101.5	5.9	20.8	119.6	4.3	0
91675	Quick-aged	101.2	16.6	18.3	113.0	27.9	3.5
45985	(91675) after 4-yr. aging	108.2	76.5	48.8	135.0	187.1	16.2

TABLE VIII. ACID AND ESTER RELATIONS

(In grams per 100 liters, calculated to 100° proof alcohol)

At 1-Year Period: Lab. No.	Acids	Esters	Difference	At 4-Year Period: Lab. No.	Acids	Esters	Difference
2793	38.47	23.12	15.35	43140	58.71	37.57	21.14
2794	52.41	29.89	22.52	43141	73.1	43.51	29.6
8208	48.55	29.52	19.0	45320	67.2	44.36	22.94
8209	50.37	28.68	21.69	45321	70.25	47.40	22.85
7869	54.0	29.9	24.1	46039	72.6	54.8	19.7
7870	49.75	30.4	19.35	46040	71.51	47.6	24.9
2792	49.81	32.62	17.19	43820	71.32	39.02	32.3
2791	48.94	33.26	15.68	43821	72.57	41.48	31.09
7211	63.47	37.9	25.57	45984	83.96	58.33	25.63
7212	60.75	35.15	25.61	45985	76.52	48.8	27.72
2073	58.51	30.89	27.62	42615	78.55	48.25	30.3
2074	56.2	31.77	24.43	42614	74.69	48.22	26.47
2071	40.89	21.85	19.04	43144	59.8	39.38	20.42
2072	46.97	26.26	20.71	43145	63.57	43.34	20.23
7867	53.38	34.8	18.58	46037	69.43	48.53	20.9
7868	47.64	33.19	14.45	46038	67.22	47.68	19.54
2075	46.47	23.85	22.62	43146	65.47	39.87	26.6
2076	46.49	23.87	22.62	43147	63.97	39.16	24.81
8206	50.54	35.34	15.20	45884	74.93	45.19	29.74
8207	46.43	33.62	12.81	45885	73.80	39.36	34.44
2069	48.96	26.27	22.69	43142	74.42	44	30.42
2070	43.97	27.01	16.96	43143	65.03	43.12	21.91
Av.	50.14	29.96	20.17	Av.	70.39	44.95	25.62

plest operations made in the analysis of spirits and perhaps may be considered the most important when dealing with whisky that has not been manipulated by quick-aging processes. Reference to samples taken from distilleries 5, 6, and 10 (Table II) shows that the acids have been lifted from 8.3 parts (average of the new whisky not quick-aged) to 33 parts or more by the comparatively mild quick-aging processes

TABLE IX. PERCENTAGE OF WATER-INSOLUBLE COLOR IN SAMPLES

Age	Distillery 1		Distillery 2		Distillery 3		Distillery 4		Distillery 5		Distillery 6		Distillery 7		Distillery 8		Distillery 9		Distillery 10		Distillery 11	
Years	*a*	*b*	*a*	*b*	*a*	*b*	*a*	*b*	*a*	*b*	*a*	*b*	*a*	*b*	*a*	*b*	*a*	*b*	*a*	*b*	*a*	*b*
New	..	..	3	7	0	0	0	0	57.7[a]	58.7[a]	56.9[a]	49.1[a]	41.2[a]	0	0	0	0	0	56.0[a]	60.0[a]	0	11.2
0.5	52.7	52.9	48.1	45.5	64.4	65.7	61.4	58.8	67.0	57.1	65.4	64.3	71.3	57.3	66.4	67.6	30.2	35.0	63.6	58.5	49.1	44.5
1	58.1	69.7	58	51.2	65.7	70.1	63.3	62.5	67.3	66.2	68.3	68.3	66.7	58.1	63.4	62.7	39.6	40.9	67.5	65.0	55.8	52.5
1.5	53.1	67.1	54.3	55.4	64.2	70.4	65.5	63.4	68.3	65.0	69.5	70.9	69.6	60.6	65.2	68.7	51.1	56.3	68.9	64.6	55.6	50.9
2	54.7	69.5	63.4	60	63.4	70.8	67.3	61.1	70.3	64.3	67.3	64	74.8	58.0	68.8	68.6	55.5	60.4	69.4	64.1	60.7	61.6
2.5	51.9	66.7	58.3	64.5	72.7	67.2	68	61.9	72.7	68.8	71.4	69.6	72.0	65.2	69.4	67.5	56.7	59.0	74.4	69.0	67.9	65.4
3	64.5	74.3	69	60.1	70.3	72.6	67.3	61.9	73.6	73.1	75.8	73.3	72.2	64.3	72.7	67.8	61.5	65.4	73.7	72.1	66.6	71.5
3.5	66.7	74.6	68.3	66.7	77.3	76.7	69.2	68.3	73.3	73.5	75.9	72.2	75.0	68.2	72.0	73.6	61.5	65.5	76	70.0	71.2	71.5
4	64.9	..	68.5	67.7	76.7	..	73.3	70.7	75	73.9	75.7	77.3	76.5	70.1	70.1	70.6	63.9	67.0	74.6	73.8	71.9	73.6

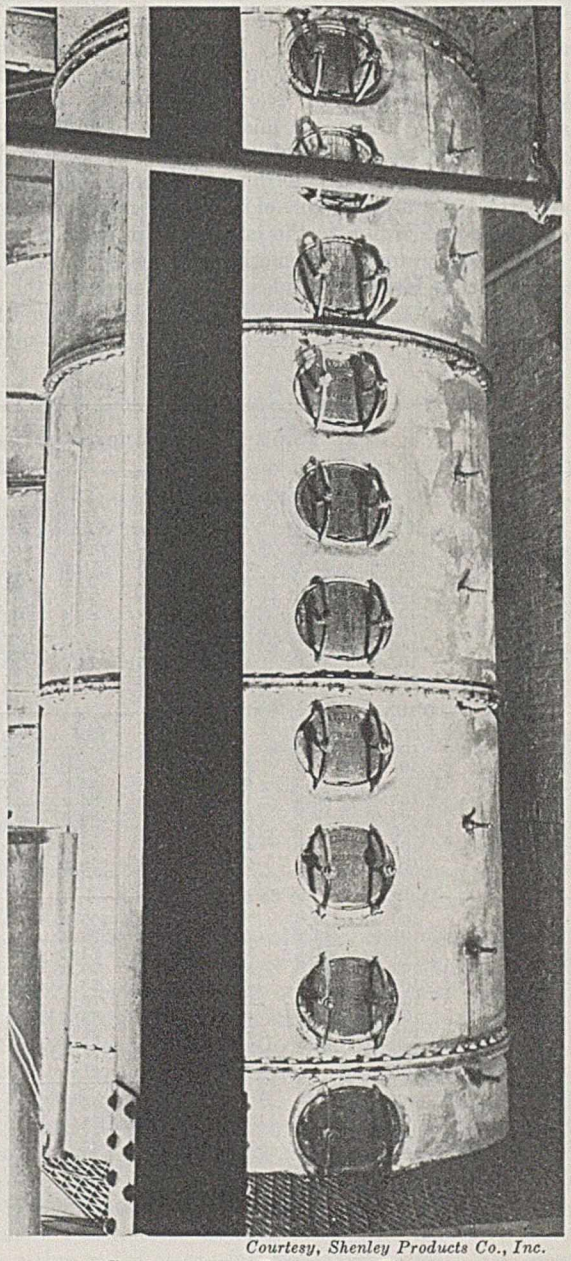
[a] Quick-aged.

important determinations made in the analysis of distilled spirits, giving more information as to the method of distillation in the manufacture of spirits than any other one factor. Table II shows that during the storage period there was an increase in the fusel oil content which was due to concentration of the whisky, but when calculated back to the original volume there was actually a loss of this compound.

ALDEHYDES. Aldehydes were present in all the whiskies examined. This determination is difficult to make accurately. Table II shows that the aldehydes gradually increased in the whisky, but, like the fusel oil, when calculated back to the original volume there was an actual loss. When bottled there appears to be a gradual reduction in the amount of aldehydes while standing in glass.

FURFURAL. Furfural is much easier to check than aldehydes because during this analysis it is less sensitive to temperature conditions. The most important origin of furfural is evidently the charred barrel. The new whiskies show only a trace of furfural, and it is probable that this trace was extracted during its brief contact with the new charred barrel. Extensive work with numerous samples of white-oak chips extracted before and after charring shows that the uncharred white-oak wood does not contain furfural. Most of the furfural is obtained during the first 6 months in contact with charred oak barrel and there is practically no increase after this period.

In referring to average acid and ester relation, Crampton and Tolman (*2A*) stated: "The fourth year the acids are 62 and the esters 61 and they remain practically the same during the next 4 years. This shows that these two substances gradually approach an equilibrium, which they reach about the fourth year and which does not change afterward." The results of the present work do not bear out this statement. Table VIII shows that after 6 months the esters always lag behind the acids, and as the period of storage increases the lag becomes greater rather than less. For instance, often there is a greater difference between the acids and esters of the 4-year-old whiskies than between the 1-year-old whiskies.



Courtesy, Shenley Products Co., Inc.

SECTION OF A DISTILLING COLUMN

Water-Insoluble Color

The A. O. A. C. tentative method for water-insoluble color was used. The principle of the method is to make an aqueous extract of the solids of the whisky, filter, then make to original volume with alcohol, and compare the colored solution produced with the whisky itself. In this way the water-soluble color is read, and by subtraction the percentage of water-insoluble color is determined.

The determination is of considerable value in checking artificial colorations. These percentages of water-insoluble color are fairly uniform, and any great deviation from the average is cause for suspicion as to the genuineness of the whisky or possibly as to rectification.

The figures of water-insoluble color obtained from these experiments show approximately 10 per cent higher results than those obtained by Crampton and Tolman (*2*). In fact, these figures show as high average results for 4-year-old whisky as those of Crampton and Tolman did on 8-year-old whisky. The Crampton and Tolman chemists used the Walker and Schrieber method for determining water-insoluble color, whereas the 198 samples involved in the present investigation were analyzed according to the later Tolman method which has been incorporated in the methods of the A. O. A. C. as tentative, for the last two decades. The difference in the two methods lies in the fact that the Walker and Schrieber method used a small amount of hot water to extract the water-soluble portion of the whisky residue, whereas the present A. O. A. C. method specifies cold water for the extraction. It is naturally presumed that the hot water extracted more water-soluble material, leaving less water-insoluble material to be reported.

Table IX shows the percentage of water-insoluble color on the samples involved in this investigation. The water-soluble color can be obtained by subtracting this figure from 100.

Volatile Acids

In addition to the method for determination of the volatile acids in whisky used in this laboratory, attention should be called to a method that has been made use of in other labora-

TABLE X. COMPARISON OF TOTAL, VOLATILE, AND FIXED ACIDS IN WHISKY

Lab. No.	Age, Years	Proof	Acids, Grams/100 Liters, Not Calcd. to Proof: Total	Volatile	Fixed	Acids, Per Cent: Volatile	Fixed
Distillery No. 1*a*; Serial No. 105							
83492	17 days	102.2	4.8	4.8	0	100	0
93689	0.5	101.6	27.6	21.6	6.0	78.3	21.7
2793	1	102.9	37.2	29.4	7.8	79.0	21.0
10205	1.5	103.3	38.4	31.8	6.6	82.8	17.2
17869	2	104.6	43.2	38.4	4.8	88.9	11.1
25780	2.5	105.1	43.2	37.8	5.4	87.5	12.5
32745	3	106.0	49.2	37.8	11.4	76.8	23.2
38344	3.5	107.6	52.8	40.2	12.6	76.1	23.9
43140	4	108.3	62.4	40.2	22.2	64.4	35.6
Distillery No. 2*a*; Serial No. 3630							
88291	New	100.8	7.2	7.2	0	100	0
99308	0.5	101.1	38.4	36.0	2.4	93.8	6.2
8208	1	101.3	43.2	37.8	5.4	87.5	12.5
14824	1.5	102.9	49.2	40.8	8.4	82.9	17.1
22467	2	103.4	50.4	44.4	6.0	88.1	11.9
30136	2.5	104.6	52.8	45.6	7.2	86.4	13.6
36252	3	105.3	60.0	46.8	13.2	78.0	22.0
40880	3.5	106.9	63.6	48.6	15.0	76.4	23.6
45320	4	107.1	67.2	53.4	13.8	79.5	20.5
Distillery No. 3*a*; Serial No. 2691							
89997	New	100.2	9.6	9.6	0	100.0	0
170	0.5	101.4	43.2	31.8	11.4	73.6	26.4
7869	1	101.4	51.6	37.2	14.4	72.1	27.9
16896	1.5	102.8	55.2	43.8	11.4	79.3	20.7
24286	2	104.6	61.2	45.0	16.2	73.5	26.5
31397	2.5	105.6	60.0	45.6	14.4	76.0	24.0
36953	3	106.4	72.0	49.2	22.2	68.3	31.7
41870	3.5	108.6	72.0	51.6	20.4	71.7	28.3
46039	4	110.7	81.6	58.2	23.4	71.3	28.7
Distillery No. 4*a*; Serial No. 1490							
84639	New	100.5	9.6	7.2	2.4	75.0	25.0
94289	0.5	100.3	42.0	31.2	10.8	74.3	25.7
2792	1	101.2	45.6	37.2	8.4	81.6	18.4
10207	1.5	101.6	52.8	39.6	13.2	75.0	25.0
19462	2	102.3	55.2	43.8	11.4	79.3	20.7
27356	2.5	103.0	58.8	46.2	12.6	78.6	21.4
34020	3	103.8	63.6	49.2	14.4	77.4	22.6
39272	3.5	104.1	67.2	49.8	17.4	74.1	25.9
43820	4	106.1	70.8	58.2	12.6	82.2	17.8
Distillery No. 5*a*; Serial No. 3570							
91672	New	101.6	8.4	7.2	1.2	85.7	14.3
91673	New, Q. A.	101.4	15.6	13.2	2.4	84.6	15.4
98607	0.5	101.5	52.8	40.8	12.0	77.3	22.7
7211	1	102.1	64.8	46.2	18.6	71.3	28.7
16785	1.5	102.8	73.2	53.4	19.8	73.0	27.0
23787	2	104.0	76.8	57.6	19.2	75.0	25.0
31307	2.5	104.8	82.8	60.0	22.8	72.5	27.5
36603	3	106.2	86.4	61.8	24.6	71.5	29.5
41655	3.5	107.0	86.4	66.0	20.4	76.4	23.6
45984	4	108.6	91.2	67.2	24.0	73.7	26.3
Distillery No. 6*a*; Serial No. 2797							
83218	25 days	101.9	26.4	21.6	4.8	81.8	18.2
93681	0.5	101.8	46.8	36.0	10.8	76.9	23.1
2073	1	102.5	54	43.8	10.2	81.1	18.9
9907	1.5	103.2	60.6	48.6	12.0	80.2	19.8
17442	2	104.2	63.6	52.2	11.4	82.1	17.9
25354	2.5	104.1	67.2	51.0	16.2	75.9	24.1
32585	3	105.2	70.8	54.6	16.2	77.1	22.9
38034	3.5	105.8	75.6	58.8	16.8	77.8	22.2
42614	4	106.9	75.6	58.2	17.4	77.0	23.0
Distillery No. 7*a*; Serial No. 5967							
83217	1 day	101.2	14.4	10.2	4.2	70.8	29.2
93683	0.5	101.7	34.8	26.4	8.4	75.9	24.1
2071	1	102.7	43.2	31.8	11.4	73.6	26.4
9905	1.5	104	43.2	33.6	9.6	77.8	22.2
18511	2	104.7	48.0	35.4	17.4	73.8	26.2
26026	2.5	105.2	Not enough sample left			..	..
33277	3	106.5	55.2	38.4	16.8	69.6	30.4
38690	3.5	106.9	56.4	42.6	13.8	75.5	24.5
43144	4	108.4	61.2	44.4	16.8	72.5	27.5
Distillery No. 1*b*; Serial No. 251							
83493	14 days	102.2	18.0	15.0	3.0	83.3	16.7
93690	0.5	102.1	38.4	32.4	6.0	84.4	15.6
2794	1	103.0	50.4	38.4	12.0	76.2	23.8
10206	1.5	103.7	51.6	42.0	9.6	81.4	18.6
17870	2	105.4	55.2	43.2	12.0	78.3	21.7
25781	2.5	105.8	57.6	45.0	12.6	78.1	21.9
32746	3	106.5	63.6	49.2	14.4	77.4	22.6
38345	3.5	107.9	66.0	54.0	12.0	81.8	18.2
43141	4	109.2	70.8	54	16.8	76.3	23.7
Distillery No. 2*b*; Serial No. 3642							
88292	New	100.5	9.6	9.6	0	100	0
99307	0.5	100.7	42.0	33.0	9.0	78.6	21.4
8209	1	101.2	45.6	37.8	7.8	82.9	17.1
14285	1.5	102.5	52.8	45.0	7.8	85.2	14.8
22468	2	103.1	54.0	43.8	10.2	81.1	18.9
30137	2.5	104.3	57.6	48.6	9.0	84.4	15.6
36253	3	104.9	61.2	49.8	11.4	81.4	18.6
40881	3.5	106.6	69.6	51.6	18.0	74.1	25.9
45321	4	106.8	70.8	56.4	14.4	79.7	20.3
Distillery No. 3*b*; Serial No. 2751							
89998	3 days	101.7	8.4	8.4	0	100.0	0
171	0.5	101.2	40.8	34.8	6.0	85.3	14.7
7870	1	101.3	48.0	40.8	7.2	85.0	15.0
16897	1.5	102.2	54.0	44.4	9.6	82.2	17.8
24827	2	104.2	56.4	46.8	9.6	83.0	17.0
31398	2.5	105.1	62.4	48.0	14.4	76.9	23.1
36954	3	107	62.4	49.2	13.2	78.8	21.1
41871	3.5	107.6	68.4	54.0	14.4	78.9	21.1
46040	4	109.1	73.4	58.8	14.6	80.1	19.9
Distillery No. 4*b*; Serial No. 1491							
84640	New	100	9.6	7.2	2.4	75.0	25.0
94290	0.5	99.8	40.8	33.0	7.8	80.0	20.0
2791	1	100.5	48.0	37.2	10.8	77.5	22.5
10208	1.5	101.1	51.6	40.2	11.4	77.9	22.1
19463	2	102	57.6	43.2	14.4	75.0	25.0
27357	2.5	102.6	62.4	48.6	13.8	77.9	22.1
34021	3	103.1	62.4	49.2	13.2	78.8	21.2
39273	3.5	103.4	69.6	51.0	18.6	73.3	26.7
43821	4	105.0	74.4	55.8	18.6	75.0	25.0
Distillery No. 5*b*; Serial No. 3831							
91674	New	101.5	4.8	3.0	1.8	62.5	37.5
91675	New, Q. A.	101.2	..	..	..	..	..
98608	0.5	101.9	49.2	39.6	9.6	80.5	19.5
7212	1	102.7	58.8	45.6	13.2	77.6	22.4
15174	1.5	103.6	62.4	48.6	13.8	77.9	22.1
23788	2	104.6	66.0	51.6	14.4	78.2	21.8
31308	2.5	105	70.8	53.4	17.4	75.4	24.6
36604	3	106.8	73.2	54.6	18.6	74.6	25.4
41656	3.5	108.1	76.2	57.6	18.6	75.6	24.4
45985	4	108.2	82.8	58.8	34.0	71.0	29.0
Distillery No. 6*b*; Serial No. 2798							
83219	25 days	101.7	27.6	21.6	6.0	78.3	21.7
93682	0.5	101.6	45.6	33.6	12.0	73.7	26.3
2074	1	102.5	54.0	42.0	12.0	77.8	22.2
9908	1.5	103.4	60.0	45.6	14.4	76.0	24.0
17443	2	103.8	62.4	49.2	13.2	78.8	21.2
25355	2.5	104.8	66.0	49.2	16.8	74.5	25.5
32586	3	105.9	69.6	49.8	19.8	71.6	28.4
38035	3.5	106.8	69.6	54.0	15.6	77.6	22.4
42615	4	106.9	79.6	58.8	20.8	73.9	26.1
Distillery No. 7*b*; Serial No. 5968							
83216	1 day	101.3	6.0	5.4	0.6	90.0	10.0
93684	0.5	101.3	36.0	27.6	8.4	76.7	23.3
2072	1	102.2	44.4	36.6	7.8	82.4	17.6
9906	1.5	103.2	46.8	40.8	6.0	87.2	12.8
18512	2	103.8	48.0	37.8	10.2	78.8	21.2
26027	2.5	105.2	50.4	39.0	11.4	77.4	22.6
33278	3	105.9	58.8	45.6	13.2	77.6	22.4
38691	3.5	106.2	62.4	49.2	13.2	78.8	21.2
43145	4	107.6	62.4	49.8	13.6	79.8	20.2

TABLE X (*Continued*)

Lab. No.	Age, Years	Proof	Acids, Grams/100 Liters, Not Calcd. to Proof: Total	Volatile	Fixed	Acids, Per Cent: Volatile	Fixed
Distillery No. 8*a*; Serial No. 424153							
89999	1 day	100.0	4.8	4.8	0	100.0	0
169	0.5	100.7	42.0	31.8	10.2	75.7	24.3
7867	1	101.2	51.6	38.4	13.2	74.4	25.6
16894	1.5	102.3	52.8	42.0	10.8	79.5	20.5
24284	2	104.4	60.0	47.4	12.6	79.0	21.0
31395	2.5	106.1	68.4	49.8	18.6	72.8	27.2
36951	3	107.4	67.2	50.4	16.8	75.0	25.0
41868	3.5	109.0	72.0	54.0	18.0	75.0	25.0
46037	4	110.6	74.4	57.0	17.4	76.6	23.4
Distillery No. 9*a*; Serial No. 662							
83494	5 days	102.6	7.2	4.8	2.4	66.7	33.3
93687	0.5	102.9	31.2	27.6	3.6	88.5	11.5
2075	1	103.3	42.0	36.6	5.4	87.1	12.9
9909	1.5	103.9	46.8	37.2	9.6	79.5	20.5
18513	2	104.5	50.4	41.4	9.0	82.1	17.9
26028	2.5	105.3	52.8	44.4	8.4	84.1	15.9
33279	3	106.1	57.6	45.0	12.6	78.1	21.9
38686	3.5	106.7	61.2	49.2	12.0	80.4	19.6
43146	4	108.1	63.6	50.4	13.2	79.2	20.8
Distillery No. 10*a*; Serial No. 1434							
93692	25 days	101.3	31.2	24.0	7.2	76.9	23.1
2789	0.5	102	48.0	37.8	10.2	78.8	21.2
8206	1	102.1	50.4	37.2	13.2	73.8	26.2
14826	1.5	103.6	56.4	43.2	13.2	76.6	23.4
23615	2	103.8	60.0	44.4	15.6	74.0	26.0
31309	2.5	105.5	66.0	47.4	18.6	71.8	28.2
36866	3	105.7	67.2	49.2	18.0	73.2	26.8
41720	3.5	106.9	72.0	51.0	21.0	70.8	29.2
45884	4	107.1	73.2	54.6	18.6	74.6	25.4
Distillery No. 11*a*; Serial No. 649							
83220	4 days	100.5	8.4	6.6	1.8	78.6	21.4
93685	0.5	99.7	33.6	28.2	5.4	83.9	16.1
2069	1	100.5	50.4	39.0	11.4	77.4	22.6
10203	1.5	100.7	51.6	42.6	9.0	82.6	17.4
18416	2	101.3	58.8	46.2	12.6	78.6	21.4
26024	2.5	101.9	60.0	48.6	11.4	81.0	19.0
33275	3	102.4	64.8	52.2	12.6	80.6	19.4
38688	3.5	102.9	68.4	52.2	13.2	80.7	19.3
43142	4	104.0	73.2	55.8	17.4	76.2	23.8
Distillery No. 8*b*; Serial No. 424154							
90000	New	100.0	6.0	4.8	1.2	80.0	20.0
168	0.5	100.3	42.0	33.0	9.0	78.6	21.4
7868	1	100.8	40.2	33.0	7.2	82.1	17.9
16895	1.5	102.5	55.2	43.2	12.0	78.3	21.7
24285	2	103.5	57.6	42.0	15.6	72.9	27.1
31396	2.5	105.2	62.4	45.6	16.8	73.1	26.9
36952	3	105.9	64.8	49.2	15.6	75.9	24.1
41869	3.5	107.3	68.4	51.0	17.4	74.6	25.4
46038	4	108.9	72.0	51.6	20.4	71.7	28.3
Distillery No. 9*b*; Serial No. 663							
83495	5 days	102.5	8.4	6.0	2.4	71.4	28.6
93688	0.5	102.8	33.6	27.6	6.0	82.1	17.9
2076	1	103.2	43.2	35.4	7.8	81.9	18.1
9910	1.5	103.4	46.8	37.8	9.0	80.8	19.2
18514	2	104.2	51.6	40.2	11.4	77.9	22.1
26029	2.5	105.1	52.8	43.2	9.6	81.8	18.2
33280	3	106.1	56.4	48.6	7.8	86.2	13.8
38687	3.5	106.7	58.8	49.2	9.6	83.7	16.3
43147	4	107.9	62.4	49.8	12.6	79.8	20.2
Distillery No. 10*b*; Serial No. 1435							
93691	25 days	101.1	31.2	26.4	4.8	84.6	15.4
2790	0.5	102.1	43.2	31.8	11.4	73.6	26.4
8207	1	103	45.6	36.0	9.6	78.9	21.1
14827	1.5	103.8	54.0	41.4	12.6	76.7	23.3
23616	2	104.8	55.2	41.4	13.8	75.0	25.0
31310	2.5	105.4	61.2	45.6	15.6	74.5	25.5
36867	3	105.7	63.6	46.8	16.8	73.6	26.4
41721	3.5	107.2	70.8	50.4	20.4	71.2	28.8
45885	4	107.3	70.8	50.4	20.4	71.2	28.8
Distillery No. 11*b*; Serial No. 650							
83221	4 days	100.6	7.2	7.2	0	100	0
93686	0.5	100.3	28.8	23.4	5.4	81.3	18.7
2070	1	101.0	42.0	34.8	7.2	82.9	17.1
10204	1.5	102.0	45.6	36.6	9.0	80.3	19.7
18417	2	102.9	51.6	39.6	12.0	76.7	23.3
26025	2.5	103.3	52.8	39.0	13.8	73.9	26.1
33276	3	104.1	56.4	46.8	9.6	83.0	17.0
38689	3.5	104.3	60.0	49.2	10.8	82.0	18.0
43143	4	106.1	62.4	51.6	10.8	82.7	17.3

tories, particularly in England and France. The usual European method is as follows:

Fifty cubic centimeters are evaporated in a beaker to near dryness over a steam bath, about 25 cc. of distilled water are added, and the solution is again evaporated. The residue is then dissolved in about 25 cc. of cold, recently boiled, distilled water, and titrated with 0.1 *N* soda or baryta. The difference between the value so ascertained and that obtained in the determination of total acids is a measure of the volatile acid, which, as well as the total acid, is expressed in terms of acetic acid as grams per 100 cc. of the sample.

The method used for the determination of volatile acids in samples shown in Table X was made in exactly the same manner as the A. O. A. C. for volatile acids in wine (Official Method 1), except that 50 cc. of whisky are used in place of wine.

In comparing the total acids with the volatile and fixed, the amount of total acids was that found in the whisky after it had stood 4 years, or nearly 4 years, in glass; these acids have not been calculated to proof. Table X shows the total fixed and volatile acids and the percentage of volatile and fixed acids in whiskies when first introduced into the barrel, or a few days thereafter, and every 6 months up to and including the 4-year aging period. The increased acidity is due to both volatile and fixed acids. The volatile acids develop during storage and the fixed acids are extracted from the wood.

Changes in Glass Containers

Schidrowitz and Kaye (*4*) remarked that "the alkalinity of glass bottles used for spirits may have a considerable influence on the latter." Crampton and Tolman (*2*) believed that no changes took place during the 8 years in which their whisky was stored in glass. Determinations were made here, therefore, to see if there were actually any changes taking place in glass and, if so, to what extent. All of the samples were reanalyzed for acid content and it was found that, while a few, particularly the new whiskies, showed little or no difference in acid content, practically all of the others showed a decrease in acid content. In some instances this decrease was as much as 12 parts per 100,000; the average was approximately 5 parts per 100,000. This change—namely, loss in acid content—may have been due to free alkali extracted from the glass; it may have been due to equilibrium changes or possibly oxidization changes. The results of this investigation clearly indicate that changes do take place in glass bottles; whether this is due to the glass container is not known. The depth of color of the whisky was also reread, and it was found that in practically all cases there was an increase. A thorough investigation should be made to determine more fully what changes take place in glass containers and the cause of such changes.

Table XI shows the change of color resulting from storage in glass.

Some explanation is due concerning the first samples taken from each barrel. While these first samplings are called "new," some of the whiskies were in these barrels as much as 24 days. The length of time each whisky was actually in contact with the barrel is shown in Table II. The ideal factor would have been to take the first sample just before it entered the barrel so that its character could be ascertained before it was in the least affected by the charred inner surface of the barrel. In most instances this was impossible; however, some of the samples were taken from the barrel the same day the barrel entered the warehouse. With the exception of the four barrels that were quick-aged, the color found in the other first samples was due to the brief time they were in wood until the samples were taken by the U. S. storekeeper-gager.

TABLE XI. CHANGE OF COLOR IN GLASS (NOT CALCULATED TO PROOF)

Distillery No. 1			Distillery No. 2			Distiller No. 3			Distillery No. 4			Distillery No. 5			Distillery No. 6		
	Color in 0.5-in. cell			Color in 0.5-in. cell			Color in 0.5-in. cell			Color in 0.5-in. cell			Color in 0.5-in. cell			Color in 0.5-in. cell	
Lab. No.	Original	July, 1935	Lab. No.	Original	July, 1935	Lab. No.	Original	July, 1935	Lab. No.	Original	July, 1935	Lab. No.	Original	July, 1935	Lab. No.	Original	July, 1935
83492	0.5	1	88291	0.5	1	89997	0.5	0.5	84639	0.5	1.0	91673	3.5	4.0	83218	7.0	7.5
83493	0.5	1	88292	0.5	1	89998	0.4	0.5	84640	0.5	1.0	91675	3.5	4.5	83219	6.5	7.5
93689	4.5	5	99308	7.0	..	170	7.8	9.0	94289	6.5	8.5	98607	10.0	10.0	93681	9.0	11.0
93690	5.0	6.5	99307	7.5	10.5	171	7.5	7.5	94290	6.5	8.0	98608	9.5	11.5	93682	9.5	11.0
2793	6.5	7.0	8208	8.0	10.0	7869	10.0	10.5	2792	8.0	9.5	7211	12.5	14.5	2073	10.5	12.0
2794	7.5	10.0	8209	9.0	..	7870	9.0	9.5	2791	8.0	10.0	7212	12.0	14.0	2074	10.5	13.0
10205	7.5	9.0	14824	9.5	13.0	16986	10.5	13.0	10207	9.5	11.0	16785	14.0	16.5	9907	12.0	14.0
10206	9.0	11.0	14825	10.5	14.0	16897	9.5	10.0	10208	9.5	11.0	15174	12.5	14.5	9908	12.0	14.0
17869	8.5	9.5	22467	9.5	11.0	24286	12.2	..	19462	11.0	13.0	23787	14.5	17.0	17442	14.0	14.5
17870	11.0	13.0	22468	10.5	13.5	24287	10.5	11.0	19463	11.0	12.0	23788	13.5	15.5	17443	14.0	15.0
25780	9.5	11.0	30136	11.0	14.5	31397	13.0	14.5	27356	12.0	13.0	31307	16.0	19.0	25354	14.5	16.0
25781	12.5	14.0	30137	12.5	16.0	31398	11.0	12.0	27357	12.5	14.0	31308	14.5	17.5	25355	14.5	15.5
32745	10.0	10.5	36252	11.0	13.0	36953	14.5	17.0	34020	12.0	14.0	36603	16.5	20.0	32585	16.5	17.5
32746	13.0	14.0	36253	12.5	14.0	36954	12.5	13.0	34021	12.5	14.5	36604	15.5	18.5	32586	15.5	16.5
38345	13.5	15.5	40880	13.0	13.5	41870	16.5	17.0	39272	13.0	14.0	41655	17.0	19.5	38034	17.0	18.5
38344	10.5	11.0	40881	15.0	15.0	41871	14.0	14.5	39273	13.7	14.5	41656	16.5	19.0	38035	16.0	18.5
43140	12.0	12.0	45320	14.0	14.5	46039	19.0	19.0	43820	15.0	16.5	45984	18.2	21.0	42615	18.5	19.0
43141	14.5	16.5	45321	15.5	16.5	46040	16.3	16.5	43821	15.0	16.5	45985	17.5	19.0	42614	17.0	18.0

Distillery No. 7			Distillery No. 8			Distillery No. 9			Distillery No. 10			Distillery No. 11		
	Color in 0.5-in. cell			Color in 0.5-in. cell			Color in 0.5-in. cell			Color in 0.5-in. cell			Color in 0.5-in. cell	
Lab. No.	Original	July, 1935	Lab. No.	Original	July, 1935	Lab. No.	Original	July, 1935	Lab. No.	Original	July, 1935	Lab. No.	Original	July, 1935
83217	3.5	5.0	89999	0	0	83494	0.5	1.0	93692	6.0	7.0	83220	1.0	1.0
83216	0	0	90000	0	0	83495	0.5	1.0	93691	7.0	8.0	83221	1.0	1.0
93683	7.5	10.5	169	8.0	9.0	93687	4.5	5.0	2789	9.5	11.0	93685	5.5	5.5
93684	6.0	7.5	168	7.5	8.0	93688	5.0	6.0	2790	8.5	10.0	93686	5.0	5.0
2071	10.0	12.5	7867	8.5	9.5	2075	6.5	8.0	8206	11.0	12.5	2069	7.5	9.0
2072	6.5	8.0	7868	8.5	9.0	2076	7.0	8.5	8207	9.5	11.0	2070	7.0	8.0
9905	11.0	14.0	16894	9.5	10.0	9909	7.5	8.0	14826	14.0	14.5	10203	8.5	10.0
9906	7.5	9.5	16895	10.0	11.0	9910	8.0	9.0	14827	11.5	13.0	10204	8.0	8.5
18512	12.0	14.0	24284	11.0	13.0	18513	8.5	8.5	23615	15.0	16.5	18416	10.0	12.0
18511	8.0	10.0	24285	11.2	13.0	18514	9.0	9.0	23616	13.0	14.0	18417	9.0	10.0
26027	13.5	16.0	31395	13.0	13.0	26028	10.0	10.5	31309	16.5	17.5	26024	11.0	12.0
26026	9.0	..	31396	12.5	13.0	26029	10.5	11.0	31310	14.0	15.0	26025	10.0	10.0
33277	14.0	15.5	36951	13.5	13.5	33279	10.5	11.0	36866	16.5	17.5	33275	12.0	13.0
33278	10.2	11.0	36952	12.5	13.0	33280	10.5	11.0	36867	14.0	15.0	33276	10.5	11.0
38690	14.0	16.5	41868	14.5	14.5	38686	10.5	12.0	41720	17.5	19.0	38688	13.7	14.0
38691	10.5	11.0	41869	14.0	14.5	38687	11.0	13.0	41721	15.0	16.0	38689	11.0	12.0
43144	14.5	17.5	46037	15.0	15.0	43146	12.0	14.0	45884	19.0	19.0	43142	14.7	14.7
43145	11.0	12.0	46038	14.0	16.0	43147	12.5	14.5	45885	16.5	16.5	43143	13.0	13.0

TABLE XII. CHANGES IN AGED WHISKY STORED IN GLASS (NOT CALCULATED TO PROOF)[a]

	Acids		Esters		Aldehydes		Furfural		Analysis Date		Color in 0.5-In. Cell	
Lab. No.	When received	After standing in glass	When received	After standing in glass	When received	After 1 yr. in glass	When received	After 1 yr. in glass	First	Second	When received	After standing in glass
10206	58.8	51.6	31.7	32.4	5.6	5.6	2.0	1.0	7/14/31	7/9/35	9.0	10.0
38345	75.6	66.0	41.4	44.3	6.4	6.4	2.0	0.8	6/14/33	7/9/35	13.5	14.5
32746	72.6	63.6	37.8	50.9	6.4	6.4	2.0	1.0	12/27/32	7/9/35	13.0	15.0
31397	69.6	60.0	39.2	54.9	24.0	19.2	2.2	0.7	11/3/32	7/9/35	13.0	15.0
17870	63.6	55.2	35.2	44.4	5.6	5.6	2.0	1.0	12/26/31	7/9/35	11.0	12.0
14826	63.6	56.4	37.8	39.6	10.4	8.8	3.2	1.0	10/16/31	7/9/35	14.0	14.0
23616	61.2	55.2	39.6	41.4	13.6	13.6	3.0	0.7	5/2/32	7/9/35	13.0	13.0
2789	50.4	48.0	33.4	37.8	9.6	8.0	3.0	0.7	1/24/31	7/9/35	9.5	11.0
10203	55.2	51.6	30.8	36.9	11.2	11.2	2.2	0.7	7/14/31	7/9/35	8.5	10.0
26024	66.0	60.0	37.8	44.0	12.0	12.0	2.4	0.8	7/6/32	7/9/35	11.0	13.0
33275	69.6	64.8	39.2	45.8	12.4	12.0	2.6	0.8	1/6/32	7/9/35	12.0	14.0

[a] Solids were redetermined, and it was found that there was practically no increase in standing in glass over the length of time indicated.

TABLE XIII. EFFECT OF STORING IN GLASS ON NEWLY DISTILLED WHISKY[a]

			Acids		Esters		Aldehydes		Furfural		Color in 0.5-In. Cell	
Sample No.	Age	Serial No.	When received	After 5 yr. in glass	When received	After 5 yr. in glass	When received	After 1 yr. in glass	When received	After 1 yr. in glass	When received	After 5 yr. in glass
	Days		*Grams per 100 liters not calculated to proof*									
83216	1	5968	6.0	6.0	15.8	10.6	2.4	1.6	0	0	0	0.5
83220	4	649	6.6	6.6	16.7	16.7	5.6	3.7	Trace	0	1.0	1.0
83221	4	650	6.6	7.2	16.7	14.9	6.0	3.6	Trace	0	1.0	1.0
83492	17	105	5.4	4.8	14.1	9.7	4.0	4.0	Trace	0	0.5	1.0
83493	14	251	18.0	18.0	15.0	14.1	4.0	4.0	Trace	0	0.5	1.0
83495	5	663	9.6	8.4	14.1	11.4	5.0	3.2	Trace	0	0.5	0.5
84639	3	1490	9.0	9.6	21.6	19.4	10.4	8.0	0	0	0.5	0.5
89997	3	2691	9.6	9.6	15.8	15.8	20.8	16.0	0.6	0	0.5	0.5
89998	3	2751	9.0	8.4	15.0	15.8	20.8	16.0	0.6	0	0.4	0.5
91673 (Q. A.)	3	3570	15.6	15.6	21.1	20.2	4.0	4.0	1.4	0.7	3.5	4.0

[a] The solids were redetermined on these samples; there were practically no changes in the amounts of solids when first analyzed and after more than 5-year storage in glass.

Picking more or less at random among the samples which showed the larger loss of acids from the time they were originally analyzed to the present, while stored in glass, a convenient number of these samples were analyzed again for ester, aldehyde, and furfural content. In practically all cases the esters were slightly higher than before, and in every case the furfural was reduced from the amount which they originally showed.

Table XII shows the changes which actually took place in the clear-glass quart bottles from the time the samples were received and analyzed in the laboratory and after they stood in glass, up to the present time.

TABLE XIV. MINIMUM, AVERAGE, AND MAXIMUM DATA ON ALL WHISKY SAMPLES

Age	Range	Proof	Total Acids	Esters	Fusel Oil	Solids	Aldehydes	Furfural[a]	Color in 0.5-In. Cell
Years			*Grams per 100 liters calculated to proof*						
New[b]	Min.	100.0	5.3	13.7	78.7	2.9	2.4	0	..
	Av.	101.2	7.7	17.0	161.1	10.5	7.6		
	Max.	102.6	9.6[c]	21.5[c]	230.7	20.1	20.8		
0.5	Min.	100.0	31.7	18.2	87.0	61.6	3.9	0.6	4.4
	Av.	101.3	40.3	26.5	166.8	92.5	9.6	1.7	7.1
	Max.	102.9	52.4	32.8	244.3	121.9	22.7	2.2	9.9
1	Min.	100.5	38.5[b]	21.9	92.0	89.8	4.3	0.6	6.3
	Av.	101.9	50.1	29.9	166.5	114.4	10.2	1.9	8.6
	Max.	103.3	53.4[b]	35.3	245.4	135.1	20.5	2.2	12.2
1.5	Min.	100.7	44.7	22.4	96.7	98.4	4.7	0.8	7.2
	Av.	102.8	55.9	32.4	167.0	131.3	10.4	1.9	9.8
	Max.	104.0	62.8	38.2	240.4	160.7	21.8	2.2	13.6
2	Min.	101.2	48.2	25.2	97.6[c]	113.7	4.6	0.8	7.7
	Av.	103.8	59.7	34.7	168.3	143.0	11.0	1.9	10.8
	Max.	105.3	65.6	38.7	232.1	166.7	24.5	2.2	14.5
2.5	Min.	101.9	51.4	28.3	96.6	125.0	4.6	0.8	8.6
	Av.	104.8	62.8	37.0	168.6	155.8	11.0	2.1	11.9
	Max.	106.1	72.6	43.1[c]	237.7	183.1	22.7	2.4	15.6
3	Min.	102.4	54.1	28.5	98.4[c]	129.8	4.6	0.8	9.4
	Av.	105.6	65.2	38.9	172.0	163.0	11.1	2.1	12.3
	Max.	107.3	73.6	43.9[d]	249.8	197.9	22.6	2.7	15.7
3.5	Min.	102.8	58.9	29.1	95.6	132.5	5.2	0.8	9.8
	Av.	106.6	67.9	40.0	171.2	172.8	11.2	2.1	13.1
	Max.	109.0	74.8	46.2	241.3	207.4	22.1	2.9	16.1
4	Min.	104	59.8[b]	37.6	96.0	141.8	6.0	0.8	10.2
	Av.	107.7	70.6	45.0	178.5	178.7	11.6	2.2	14.1
	Max.	110.7	78.6	48.8[c]	260.8	213.8	21.7	3.0	17.3

[a] In the maximum furfural the quick-age samples and those from one distillery which seemed abnormal were omitted.
[b] Does not include quick-aged samples.
[c] Highest or lowest was omitted as being slightly abnormal, and the next highest was taken instead.
[d] Highest and next highest were omitted as being slightly abnormal.

In order to determine whether the new whisky —that is, the original samples taken as first samples from the barrels set aside—had made any change during standing in glass, the samples were reëxamined for acids, esters, and furfural. The actual change in the bottle is given in Table XIII. The changes that have taken place have been discussed elsewhere in this paper. The aldehydes and furfural were determined near the end of the 4-year aging period, which leaves only the short period between the first analysis for these substances and the analysis made in July, 1935, for the changes to take place.

Acknowledgment

Credit should be given Albert C. Blaisdell and James L. Young for the fusel oil determinations; George E. Mallory for the determinations of color insoluble in water; Joseph Levine for the volatile acid determinations; George F. Beyer and John W. Williams for assistance in preparing the manuscript.

Practically all of the eleven distilleries, which set aside twenty-two barrels of whisky for this investigation, have consented to allow them to remain for experimental purposes, and samples will be taken throughout the 8-year storage period.

TABLE XV. GAIN OR LOSS OVER 4-YEAR AGING PERIOD FROM CALCULATED OR ORIGINAL VOLUME DATA

	Acids	Esters	Fusel Oil
	Grams/100 liters calcd. to proof		
1*a*, No. 105, 4-yr. analysis calcd. to orig. vol. (28.67% loss)	41.88	26.80	186.03
New analysis	5.28	13.79	230.68
Gain or loss	+36.60	+13.01	−54.65
1*b*, No. 251, 4-yr. analysis calcd. to orig. vol. (29.52% loss)	51.52	30.67	104.12
New analysis	17.61	14.68	145.5
Gain or loss	+24.91	+15.99	−41.38
2*a*, No. 3630, 4-yr. analysis calcd. to orig. vol. (24.1% loss)	51.01	33.67	133.54
New analysis	8.33	17.45	158.82
Gain or loss	+42.68	+16.22	−25.28
2*b*, No. 3642, 4-yr. analysis calcd. to orig. vol. (25.59% loss)	52.27	35.27	145.25
New analysis	9.55	17.50	155.80
Gain or loss	+42.72	+17.77	− 9.55
3*a*, No. 2691, 4-yr. analysis calcd. to orig. vol. (33.55% loss)	48.24	36.44	66.01
New analysis	9.5	15.81	83.19
Gain or loss	+38.74	+20.63	−17.18
3*b*, No. 2751, 4-yr. analysis calcd. to orig. vol. (25.15% loss)	53.53	35.63	71.86
New analysis	8.84	14.70	78.74
Gain or loss	+44.69	+20.93	− 6.88
4*a*, No. 1490, 4-yr. analysis calcd. to orig. vol. (26.08% loss)	52.72	28.84	162.01
New analysis	8.95	21.44	188.18
Gain or loss	+43.77	+ 7.40	−26.17
4*b*, No. 1491, 4-yr. analysis calcd. to orig. vol. (27.25% loss)	52.80	30.18	161.57
New analysis	8.99	21.55	194.40
Gain or loss	+43.81	+ 8.63	−32.83
5*a*, No. 3570, 4-yr. analysis calcd. to orig. vol. (23.48% loss)	64.25	44.63	98.57
New analysis (quick-aged)	8.27	23.33	124.70
Gain or loss	+55.98	+21.30	−26.13
5*b*, No. 3831, 4-yr. analysis calcd. to orig. vol. (26.02% loss)	56.61	36.10	99.88
New analysis	5.91	20.79	119.60
Gain or loss	+50.70	+15.31	−19.72
6*a*, No. 2797, 4-yr. analysis calcd. to orig. vol. using regage of 4.5 yr. (26.03% loss)	58.11	35.70	96.05
New analysis (quick-aged)	28.26	15.54	126.18
Gain or loss	+29.85	+20.16	−30.13
6*b*, No. 2798, 4-yr. analysis calcd. to orig. vol. using regage of 4.5 yr. (25.69% loss)	55.50	35.83	96.49
New analysis (quick-aged)	28.32	17.21	131.89
Gain or loss	+27.18	+18.62	−35.40
7*a*, No. 5967, 4-yr. analysis calcd. to orig. vol. (20.37% loss)	47.62	31.36	115.09
New analysis (quick-aged)	11.86	14.35	124.35
Gain or loss	+35.76	+17.01	− 9.26
7*b*, No. 5968, 4-yr. analysis calcd. to orig. vol. (24.83% loss)	47.79	32.58	109.43
New analysis (not quick-aged)	5.92	15.63	127.65
Gain or loss	+41.87	+16.85	−18.22
8*a*, No. 424153, 4-yr. analysis calcd. to orig. vol. (27.40% loss)	50.41	35.23	162.32
New analysis	6.00	16.73	205.12
Gain or loss	+44.41	+18.50	−42.80
8*b*, No. 424154, 4-yr. analysis calcd. to orig. vol. (26.96% loss)	49.1	34.83	164.32
New analysis	6.6	16.73	222.73
Gain or loss	+42.5	+18.10	−58.41
9*a*, No. 662, 4-yr. analysis calcd. to orig. vol. (23.48% loss)	50.10	30.51	143.84
New analysis	9.35	15.43	176.67
Gain or loss	+50.75	+15.08	−32.83
9*b*, No. 663, 4-yr. analysis calcd. to orig. vol. (22.65% loss)	49.47	30.29	145.14
New analysis	9.36	13.74	185.36
Gain or loss	+40.11	+16.55	−40.22
10*a*, No. 1434, 4-yr. analysis calcd. to orig. vol. (22.75% loss)	57.88	34.89	160.92
New analysis (quick-aged)	33.16	26.92	171.97
Gain or loss	+24.72	+ 7.97	−11.05
10*b*, No. 1435, 4-yr. analysis calcd. to orig. vol. (22.70% loss)	57.05	30.43	164.80
New analysis (quick-aged)	31.24	28.09	179.80
Gain or loss	+25.81	+ 2.34	−15.00
11*a*, No. 649, 4-yr. analysis calcd. to orig. vol. 23.04% loss)	57.27	33.86	164.13
New analysis	6.56	16.63	199.56
Gain or loss	+50.71	+17.23	−35.43
11*b*, No. 650, 4-yr. analysis calcd. to orig. vol. (22.24% loss)	50.57	33.53	171.57
New analysis	6.56	16.63	213.50
Gain or loss	+44.01	+16.90	−41.93
Average	+40.10	+15.57	−28.66

Literature Cited

(1) Assoc. Official Agr. Chem., Official and Tentative Methods, 3rd ed., p. 148 (1930).
(2) Crampton, C. A., and Tolman, L. M., *J. Am. Chem. Soc.*, 30, 97–136 (1908).
(2A) *Ibid.*, 30, 129 (1908).
(3) Mallory, G. E., and Valaer, Peter, IND. ENG. CHEM., Anal. Ed., 6, 475 (1934).
(4) Schidrowitz, P., and Kaye, F., *Analyst*, 30, 149–52 (1905).
(5) Williams, J. F., IND. ENG. CHEM., 18, 841 (1926).

RECEIVED September 21, 1935.