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Forest Products Laboratory  
Madison, Wisconsin

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Office of the Commissioner of Internal Revenue

July 25, 1950

A STUDY OF WHISKY STORED FOR FOUR YEARS IN PLYWOOD BARRELS

Four years ago, the Alcohol Tax Unit began a study of the aging of whisky stored in plywood barrels. This study included whisky produced at thirteen distilleries and stored in plywood barrels manufactured by three companies. It is believed that the study is of interest to the distilling and cooperage industries and for that reason it is being distributed to members of the industries. The results of the study are attached hereto.

*Carroll E. Mealey*

Carroll E. Mealey,  
Deputy Commissioner.

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A STUDY OF WHISKY STORED FOR FOUR YEARS IN  
PLYWOOD BARRELS

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In July 1945, a manufacturer informed this Unit that he had interested some distillers in using laminated barrels and requested cooperation in determining the effect on whisky. Consequently, letters were sent to several district supervisors to inform them that permission might be given distillers to set aside two or more charred laminated barrels and an equal number of standard charred barrels containing new whisky from the same distillate, and that quart samples should be taken every three months for analysis in the Washington laboratory. A separate series was used to determine soakage for 18 months.

Eventually, 13 distillers participated and have been herein identified by numbers. The first entry was made July 26, 1945, and the last on July 18, 1946. The laminated barrels were manufactured by three companies which are designated as A, B and C.

SCOPE OF EXPERIMENT  
Laminated Barrels Manufactured by A  
4 layers 3/16" Oak  
Bilge Shape

<u>Distiller</u>	<u>No. Bbls.</u>	<u>Kind</u>	<u>Entered</u>
1	2	Bourbon	6-7-46
2	1	Bourbon	7-18-46
	<u>3</u>		



Laminated Barrels Manufactured by B  
4 Layers 3/16" Oak  
1 Center Layer 1/20" Oak  
Drum Shape

<u>Distiller</u>	<u>No. Bbls.</u>	<u>Kind</u>	<u>Entered</u>
3	3	Bourbon	1-7-46
2	2	Rye	8-24-45
4	3	Rye	3-22-46
5	2	Bourbon	2-28-46
6	2	Bourbon	1-30-46
7	5	Bourbon	1-30-46
8	2	Bourbon	3-19-46
	<u>19</u>		

Laminated Barrels Manufactured by C  
Outer Layer 1/8" Oak  
Inner Layer 1/8" Oak  
4 Center Layers 1/8" Maple  
Bilge Shape

9	2	Bourbon	12-31-45
10	2	Rye	12-13-45
11	2	Bourbon	1-17-46
4	3	Rye	3-22-46
12	2	Rye	12-6-46
13	7	Bourbon	7-26-46
	<u>18</u>		

Standard barrels are made with solid white oak staves and are bilge shape. All barrels in this experiment contain 50-55 gallons. A total of 40 laminated and 40 standard were set aside.

The data obtained by analyses of the samples have been studied for the purpose of comparing the effect of laminated barrels on whisky with that of standard barrels, and this paper presents the relationship at the end of four years.

Proof, that is apparent proof, was determined by hydrometers, as prescribed by the Gauging Manual and was not corrected for obscuration due to solids. Distillation proof, entry proof and final proof are shown in Table No. 1.



Color was determined as degrees Lovibond by spectrophotometer according to the method published by G. F. Beyer in the Journal of the Association of Official Agricultural Chemists, February 1943, and was calculated at 100 proof.

All other congeners were determined by the official methods of the above Association and all results were calculated as grams per 100 liters at 100 proof.

Table No. 1 summarizes all the data in comparative form for 2, 3 and 4 years. The column titled Proof, L-S, shows for each distiller the difference between the average proof of the whisky in the laminated barrels and that in the standard barrels. The columns titled with the names of congeners show the average per cent of standard for that congener. Thus, opposite 1 and under solids, we find, as an average, that the laminated barrels are 93, 99 and 99 per cent of standard at 2, 3 and 4 years.

The next to last column in this table shows the preference by taste between samples taken from laminated barrels and those taken from standard barrels. To obtain this information, whisky from a laminated barrel and from a companion standard barrel was put into laboratory flasks numbered A-1 and A-2. The person tasting had no way of knowing which flask contained the whisky from the laminated barrel. Four sets, each representing a different distillery, were presented each time and the taster was asked to indicate on a provided form whether he preferred No. 1 or No. 2 in each set. About 20 persons from the ATU participated. When no preference was stated, credit was given to the laminated barrels.



The lower part of Table No. 1 shows total average change of proof and fusel oil after 4 years storage.

Table No. 2 shows the results of analyses for every sample and the intra-sample ratios of some congeners.

#### DISCUSSION

PROOF is 6.0 degrees (average) higher in laminated barrels manufactured by A than in the companion standard barrels. In B barrels it is 4.5 degrees higher.

The C barrels, excepting those stored by 13, average 0.5 higher proof than the standard.

The C barrels, stored by 13, are 4.8 degrees higher than the standards but both have lost proof. See Table No. 1.

It was noted that when the whisky was first entered, the proof generally dropped more in the standard than in the laminated barrels, and it is assumed that this is due to less moisture in the laminated staves. After about nine months, the proof, with some exceptions, began to rise but the rate for the laminated barrels was higher than that of the standard. Proof changes are influenced by many factors such as humidity, temperature, entry proof and physical character of the wood.

In this study, the difference in proof between standard and laminated barrels is not a significant feature but the fact that the proof does increase in laminated barrels shows that the staves are permeable to water. Experiments made by the Forest Products Laboratory, Madison, Wisconsin, show that thin layers of the synthetic resins, used as binders in laminated



barrels, form an almost perfect barrier to water; therefore, it appears that the porosity of such barrels must be due to faults and perforations in the binder layer.

The binders used are phenolic resins which are practically insoluble in whisky and probably do not present any threat of toxicity because they are used in other tight barrels in which liquid foods are stored. Producer A claims to have a special process for curing the resin which makes his barrels superior to others.

SOLIDS are extracted from the barrel and the amount affects the flavor of the whisky. No case was found where a laminated barrel exceeded its companion standard and in only one instance are they equal.

Figure No. 1 shows the range of solids from standard and laminated barrels. Each dot or other mark represents the amount of solids in a sample and thus the figure shows not only the range of solids for all samples but the frequency of occurrence of any concentration. Such charts are useful for detecting points which are abnormal to the universe being studied. In this case, the solids for laminated barrels by tabulation would be 122 to 248 but this high result appears abnormal and the more probable range is 122 to 196. There is some overlapping but laminated barrels, especially group C, are definitely below the average standard.

In per cent, the former are:

88%	of standard	A	bbls.
84%	"	B	"
78%	"	C	"
73%	"	C	" (stored by 13)



COLOR is derived largely from the charred layer and has a fairly constant relation to solids. (See ratios, Table 2 and Fig. 3). In the A and B laminated barrels, the color is often higher than standard although solids are lower. This means that the extract from laminated barrels has greater coloring power than that from standard barrels and consequently may also differ in flavoring properties.

Figure No. 1 shows considerable overlapping of range for standard and laminated barrels but this quantitative equality is not an indication of similar properties.

The per cent of standard for color is -

94	A	bbls.
103	B	"
86	C	"
74	C	" (stored by 13)

TOTAL ACIDS in the B barrels are, in a few cases, equal to standard but average less. The C barrels are notably low (see Fig. 2). The averages are -

78%	of standard for A bbls.
89%	" " " B "
56%	" " " C "
40%	" " " C " (stored by 13)

VOLATILE ACIDS show the following averages -

79%	of standard for A bbls.
89%	" " " B "
46%	" " " C "
31%	" " " C " (stored by 13)

Thus, the acids from laminated barrels are below standard but further information is obtained by observing what per cent of the total acid is volatile. Table No. 2 shows this to average -



79%	for	standard	bbls.	
79%	"	"	"	A
78%	"	"	"	B
63%	"	"	"	C
61%	"	"	"	C (stored by 13)

This shows that although the acids in A & B barrels are below standard, the proportion of volatile to total acid is the same. The C barrels differ so much that when plotted as in Fig. 2 and Fig. 3, they form a separate class.

ESTERS are by average -

50%	of	standard	in	A	bbls.
78%	"	"	"	B	"
63%	"	"	"	C	"
28%	"	"	"	C	" (stored by 13)

It is in this important class of congeners that we find the greatest divergence from standard, especially in C barrels. See Fig. 2.

Aldehydes appear to be slightly higher than standard in all laminated barrels but the accuracy of this determination does not permit close comparison. See Table No. 2.

Fusel oil was determined but did not yield any information of value for this report.

The progress of the changes in both types of barrels followed the usual familiar path as shown in Fig. 4 for acids. The first 18 months produce the most rapid change. This figure also shows the definite separation of C barrels.

#### CONCLUSIONS

The following is a summary of averages from Table No. 1 -

Proof:	A bbls.	3.2 to 8.8 higher in laminated	<u>Averages</u>
	B "	2.0 to 5.8 " " "	6.0
	C "	minus 0.8 to +3.4 " "	4.5
			1.9



				<u>Averages</u>
Solids:	A bbls.	77 to 99%	of standard	88
	B "	76 to 90%	" "	84
	C "	71 to 86%	" "	77
Color:	A bbls.	82 to 107%	of standard	94
	B "	87 to 104%	" "	103
	C "	72 to 96%	" "	84
Vol. Acids:	A bbls.	74 to 84%	of standard	79
	B "	74 to 95%	" "	89
	C "	28 to 52%	" "	42
Total Acids:	A bbls.	71 to 85%	of standard	78
	B "	74 to 94%	" "	89
	C "	38 to 60%	" "	52
Esters:	A bbls.	45 to 56%	of standard	50
	B "	65 to 88%	" "	78
	C "	25 to 70%	" "	53

If the idea of percentage comparison is carried further and the sum of the averages used, the final score would be -

A bbls.	78%	of standard
B "	89%	" "
C "	58%	" "

Taste preference at four years was -

A bbls.	50 to 63%	for laminated	Av. 56%
B "	40 to 56%	" "	Av. 44%
C "	41 to 60%	" "	Av. 50%

See Table No. 1

The C barrels stored by distiller 13 were so different, 14 per cent preference, that they were not included in the average.

It is evident by this summary that, by average, the laminated barrels furnish less solids, acids and esters than the standard. A study of Table No. 2 shows that for these congeners the laminated barrels rarely approach equality with the companion standard barrels. In the entire universe of this experiment, there is some overlapping of the ranges of laminated and



standard barrels but this should not be taken as denoting possible equality between barrels stored together. See Fig. 1.

The following summary of the intra-sample ratios shows that those for laminated barrels differ from their standards in several respects. This point is valuable because the relative amounts of congeners is as important to the character of a whisky as the quantity.

AVERAGE INTRA-SAMPLE RATIOS  
4 years  
See Table 2

<u>Solids</u> Color	<u>Acids</u> <u>Esters</u>	% Vol. Acids	% Water Insol. Color	<u>Solids</u> <u>Acids</u>	Barrels
13.8	1.82	79	63	2.5	Standard
11.4	2.69	79	49	3.2	A
11.3	2.12	78	64	2.3	B
12.1	1.61	63	58	3.6	C
13.5	2.92	61	65	4.3	C (stored by 13)

As a whole, it may be said that the results from laminated barrels are quantitatively and qualitatively below standard if taste preference is a measure of quality.

The 19 B barrels make the best showing but are definitely below standard.

The A barrels are next best but were represented by only 3 barrels from two warehouses.

A large number of these barrels are in storage and before any final decision is made as to their nature, enough samples should be taken to be representative.

The C barrels, represented by 18 samples, are by far the less effective than standards, but as their composition is largely maple they should be viewed as a special class.



The ratios shown in Table No. 1 appear to have become constant and it seems improbable that longer storage will make any significant change.

We now see that not only do laminated barrels differ from their standards but that those from different companies give different results. This means that the term laminated barrel must be preceded by the name of the manufacturer and that without this qualification a large undefined area would be included.

Soakage, determined on 14 A barrels and 27 B barrels at 18 months, was 18.9 and 18.5 lbs. The allowance given in the Gauging Manual is 14 lbs.

As to the physical properties of the laminated barrels, experiments on type A by the Forest Products Laboratory, Madison, Wisconsin, show that they are probably equal to standard barrels. A few barrels of the other two manufacturers were observed in warehouses and no serious defects noted.

July 20, 1950  
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TABLE NO. 1  
COMPARISON OF WHISKY IN LAMINATED  
AND STANDARD BARRELS

Distillery	Kind	Laminated Barrels by	Proof Difference				Solids				Color				Total Acids				Volatile Acids				Esters				Taste Preference % Laminated				
			L-S				2 Yr.				3 Yr.				3 Yr.				3 Yr.				3 Yr.				3 Yr.				
			2 Yr.	3 Yr.	4 Yr.	5 Yr.	2 Yr.	3 Yr.	4 Yr.	5 Yr.	2 Yr.	3 Yr.	4 Yr.	5 Yr.	2 Yr.	3 Yr.	4 Yr.	5 Yr.	2 Yr.	3 Yr.	4 Yr.	5 Yr.	2 Yr.	3 Yr.	4 Yr.	5 Yr.	2 Yr.	3 Yr.	4 Yr.	5 Yr.	
1	2 Bourbon	A	+2.0	+4.2	+8.8		93	89	83	80	94	97	101	104	90	90	92	91	90	87	87	86	54	57	54	56	61	72	63	12-14 Staves 4 Ply - 3/16" Oak	
2	1 Bourbon		+0.5	+2.1			83	80	77	75	84	82	89	94	65	68	71	74	70	70	74	74	51	51	45	45	50	50	50	Blige Type	
	3 Total	Av.	+1.2	+3.1			88	89	88	70	89	89	94	94	70	73	78	78	67	66	74	74	54	54	49	50	56	56	56		
3	3 Bourbons		+3.0	+4.4	+5.8		89	83	80	76	90	102	101	104	90	90	92	91	90	87	87	85	86	86	85	85	55	41	6-10 Staves 4 Ply 3/16" Oak		
4	2 Rye		+2.2	+3.9	+4.8		81	80	76	79	89	91	102	102	84	84	90	92	87	87	87	86	87	86	85	64	41	4 Ply 3/16" Oak			
5	2 Bourbon	B	+1.2	+1.6	+2.0		97	93	89	86	88	113	120	120	91	89	94	86	87	91	84	79	84	79	75	60	50	44	1 center ply 1/20"		
6	2 Bourbon		+2.3	+3.4	+3.9		84	87	80	78	89	87	87	87	69	74	74	67	74	74	74	70	70	63	65	35	32	55	Drum Type		
7	5 Bourbon		+2.8	+4.4	+4.8		93	75	78	83	99	105	104	104	91	82	88	91	83	83	88	84	73	74	74	59	41	44	50	Outer 1/8" Oak	
8	2 Bourbon		+2.1	+3.2	+4.4		90	83	80	78	83	108	105	103	85	91	90	85	85	94	90	83	83	83	83	50	40	40	50	Inner 3/16" Oak	
	19 Total	Av.	+2.8	+4.3	+5.0		93	97	90	84	90	102	102	104	88	86	92	88	86	86	86	83	83	83	83	42	42	42	50	Others 1/8" Maple Blige Type	
			+2.3	+3.6	+4.5		93	86	84	78	84	97	100	103	85	86	89	85	85	85	85	83	80	78	78	43	48	48			
9	2 Bourbon		+0.3	-0.8	-0.8		72	82	79	75	88	87	83	83	54	58	60	43	47	52	70	70	70	70	64	41	41	48	10-12 Staves		
10	2 Rye		+1.4	-2.2	-2.9		79	79	75	75	90	79	79	75	48	50	51	37	40	44	64	58	58	58	60	41	45	45	32	6 Ply	
11	2 Bourbon	C	+0.4	+0.3	+0.5		77	79	81	82	88	88	88	88	57	57	59	47	46	49	70	66	66	66	59	59	59	38	50	Outer 1/8" Oak	
12	3 Rye		+1.3	+1.6	+2.3		80	76	71	88	86	86	86	86	48	50	52	37	40	40	39	55	52	50	50	44	41	41	60	Inner 3/16" Oak	
	2 Rye		+2.1	+2.8	+3.7		86	85	86	86	94	95	95	96	53	54	58	34	39	47	47	70	64	64	70	41	41	41	50	Others 1/8" Maple Blige Type	
	11 Total	Av.	+1.1	+0.6	+0.5		80	80	78	78	90	85	85	85	52	54	56	40	42	45	56	52	52	52	63	45	45	45	50		
13	3 Bourbon		+1.1	+3.4	+4.8		74	72	75	75	76	71	72	72	35	36	38	25	30	28	31	31	31	25	44	37	14	5 Ply			
	4 Bourbon		+2.0	+3.3	+4.8		74	73	71	71	76	68	77	77	41	41	43	31	25	34	33	33	26	31	31	25	31	6 Ply			
	7 Total	Av.	+1.5	+3.3	+4.8		74	73	73	73	76	69	74	74	38	38	40	28	27	31	31	36	28	28	31	28	37	14	5 Ply		

Distillery	Entry	Distn. Proof	Laminated Proof	Laminated 4 Yrs.	Standard Proof	Standard 4 Yrs.	Laminated Change	Standard Change
1	124	105.2	118.5	+13.3	109.7	+4.5	191	184
2	141	109.5					183	- 7
3	155	103.0	111.8	+ 8.8	106.0	+3.0	97	114
4	160	108.8	118.1	+ 9.3	113.3	+4.5	177	203
5	127	110.0	110.7	+ 0.7	108.7	-1.3	145	165
6	119	101.1	107.0	+ 5.9	103.1	+2.0	211	251
7	160	103.1	112.5	+ 9.4	106.7	+3.6	172	186
8	122	102.8	110.1	+ 7.3	105.7	+2.9	96	106
	120	102.8	110.1	+ 7.3	105.1	+2.3	136	148
			Av.	+7.0		+2.4	159	+18
9	160	102.0	104.3	+ 2.3	105.1	+3.1	191	163
10	160	103.0	105.3	+ 2.3	108.2	+5.2	170	186
11	137	103.3	106.9	+ 3.6	106.4	+3.1	187	194
12	160	103.0	103.7	+ 0.7	100.0	-3.0	145	147
							120	137
13	157	109.0	108.6	- 0.4	103.0	+13	120	133
							110	-10

Treasury - Internal Revenue, Washington, D.C.

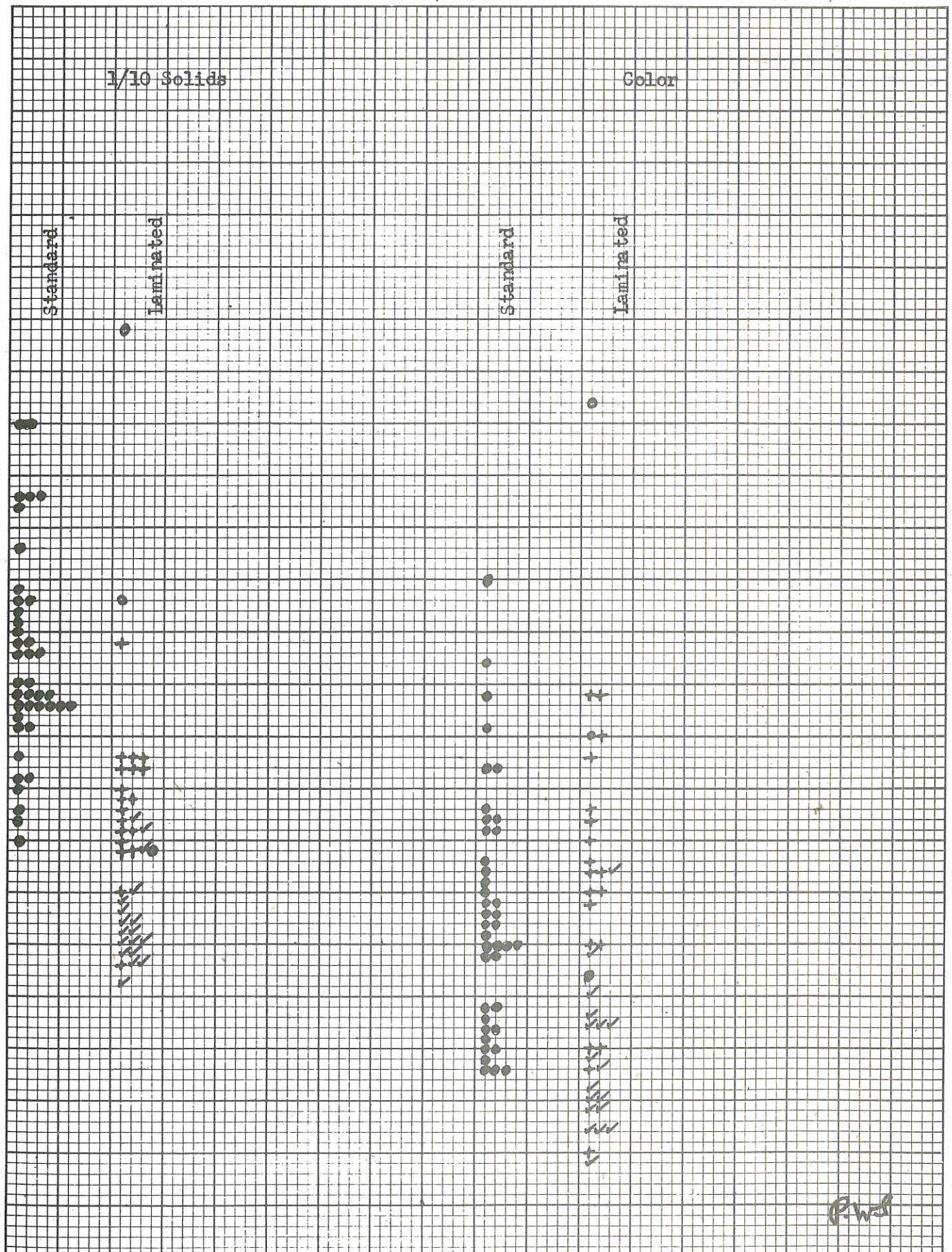






FIG. 1

4 Years

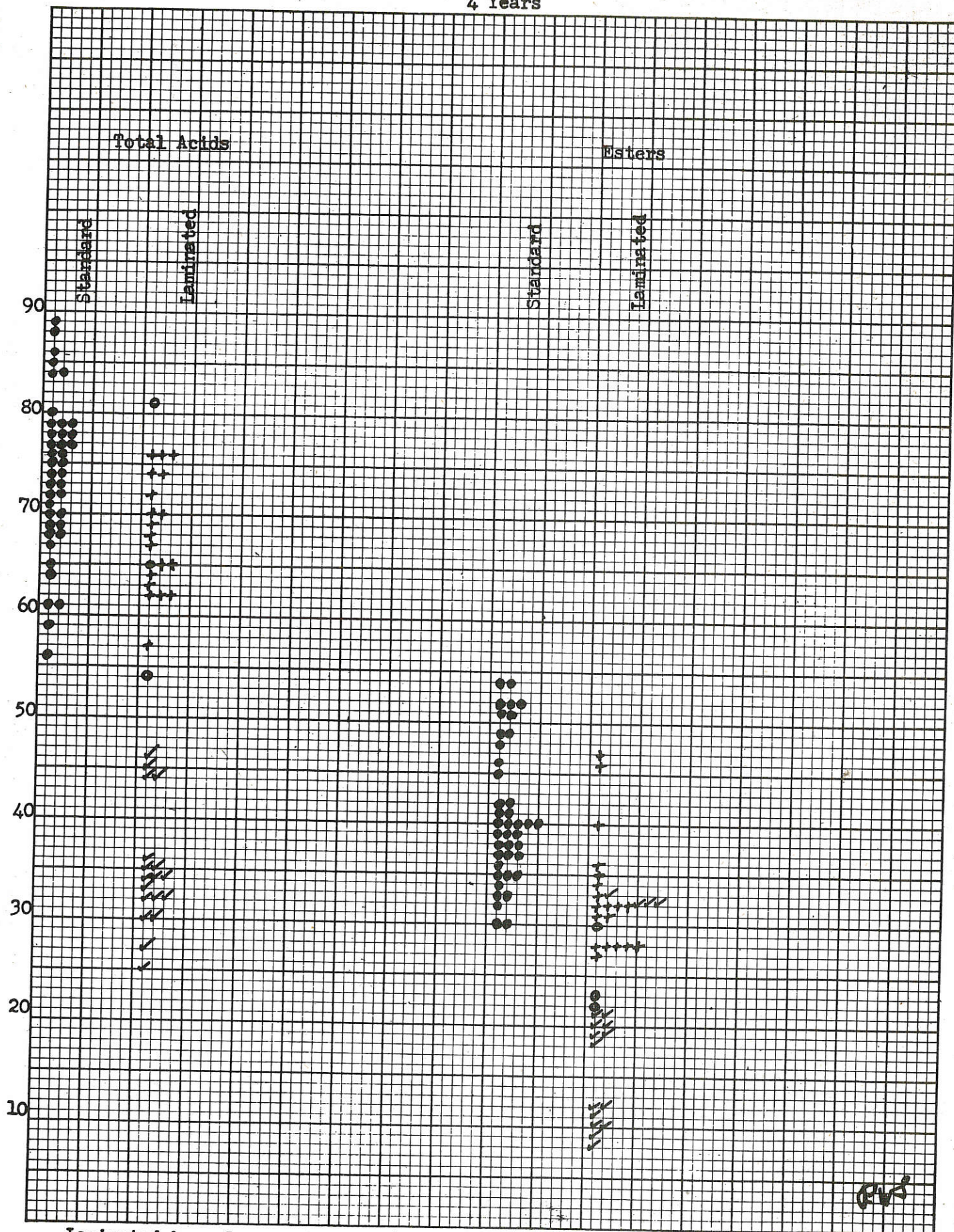


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FIG. 2

4 Years



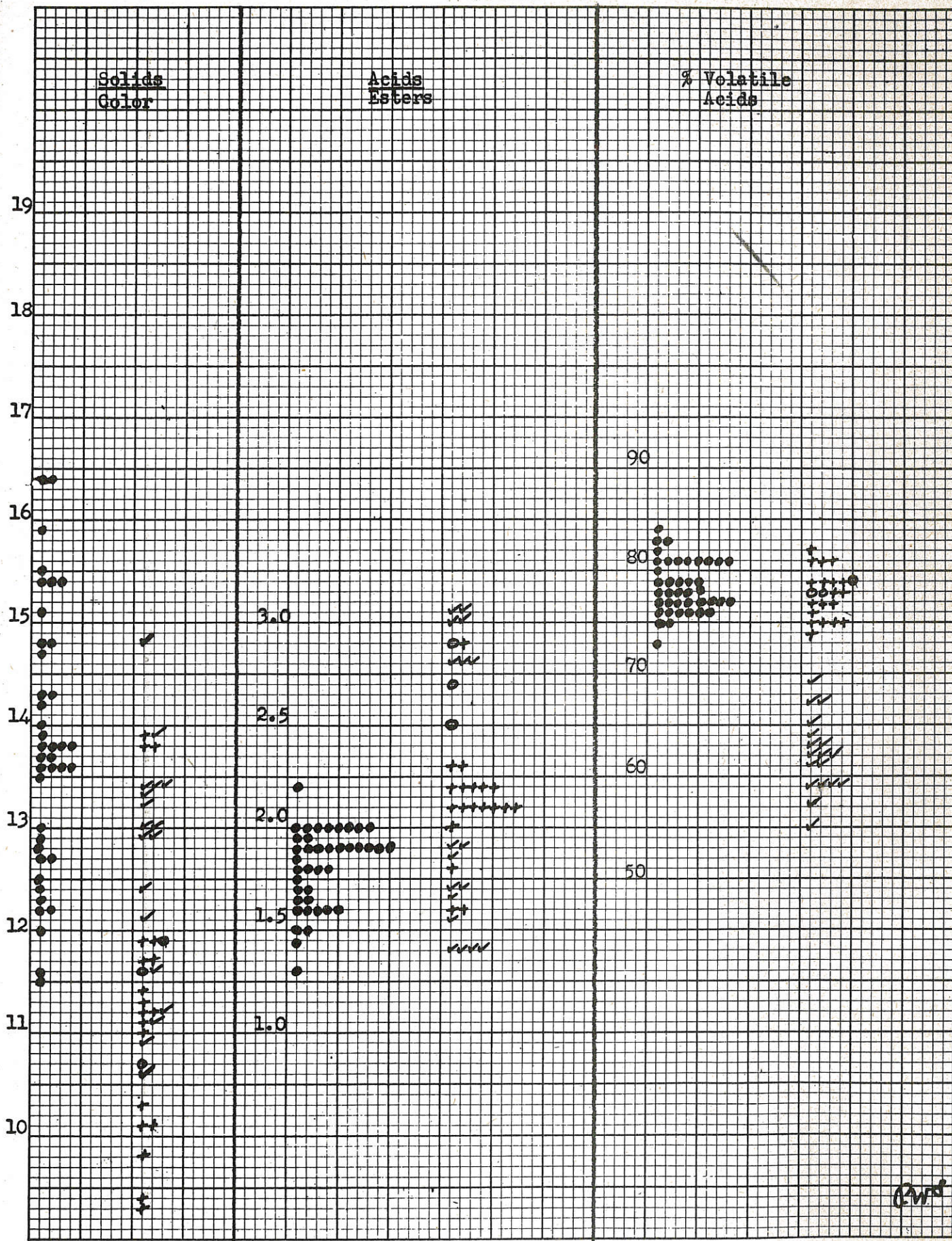
Laminated barrels manufactured by

○ A  
+ B  
✓ C



FIG. 3

4 Years



Treasury - Internal Revenue, Washington, D.C.

Laminated barrels manufactured by

0 A  
+ B  
✓ C

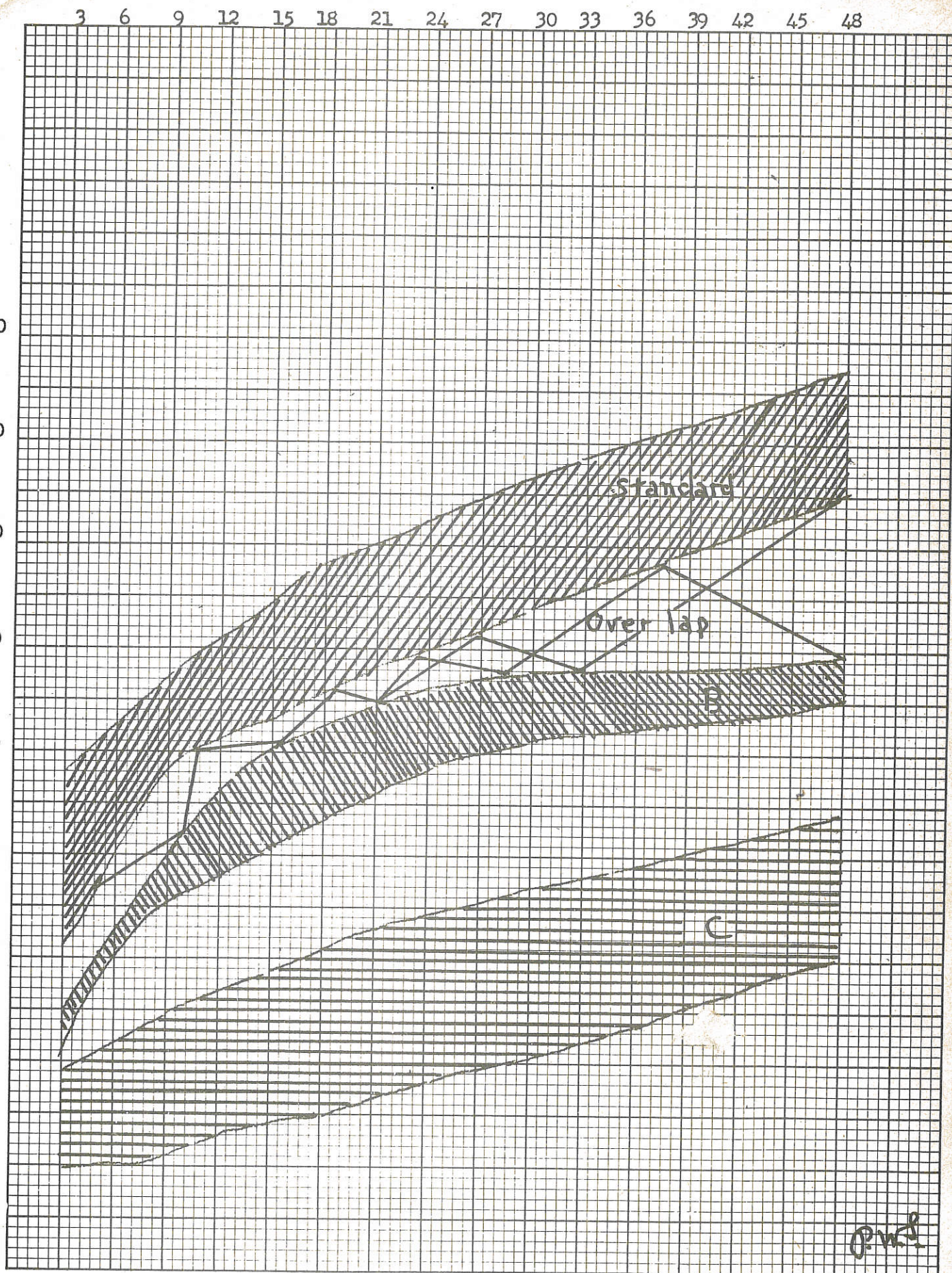


TOTAL ACIDS  
Months

FIG. 4

Grams per 100 liters

Treasury - Internal Revenue, Washington, D.C.



P.W.B.