A Comparison of Wesson Loss and Cup Refining Loss Analyses of Crude Cottonseed and Soybean Oils

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A HISTORY of the development of the present official A.O.C.S. cup method for determining laboratory refining loss of crude vegetable oils is given by Bailey, Feuge, and Bickford (1). They stress the need for a new test which will give results more in line with actual plant losses sustained in refining with modern centrifugal procedures. Prior to the advent of centrifugal refining, losses determined by the laboratory cup method were in rather close agreement with those sustained in kettle refining in the plant. At present, however, the cup method yields much higher losses than does centrifugal refining in the plant. Improvements are continually being made in the plant scale refining methods which are bringing about reductions in refining loss to values close to the theoretical loss. This makes it desirable to base efficiency of plant operation on some laboratory test which gives results comparable to plant results rather than on the relatively high results of a cup refining test.

The Refining Committee of the A.O.C.S. has been gradually developing a laboratory centrifugal refining loss test which gives results some 20 to 40% lower than the standard cup method and therefore approximates plant refining losses by centrifugal processes. Another and much older applicable method is the determination of the actual neutral oil content of the crude from which can be determined what is known as the "Wesson Loss" or theoretical loss.

In 1922, Wesson (2) published an article describing a method for determining the percentage of total neutral oil in crude oils. In 1926 he (3) reported data showing the percentage of total neutral oil, as determined by this method, recovered in commercial refining of crude cottonseed oils of various quality characteristics. In prime quality crude oils an average of 3.5% of neutral oil was lost in the soapstock, and only a relatively slight variation was found for different oils. The ratio of Wesson loss to refining loss varied considerably on off-quality crudes. In 1942 Mattikow (4) discussed the importance of the Wesson loss in evaluating refining procedures and presented data comparing Wesson loss with the cup refining loss and loss sustained in the soda ash method of centrifugal refining of several crude oils. The ratio of cup loss to Wesson loss averaged approximately 2:1 on three samples of crude cottonseed oil with an average cup loss of about 6%.

The present work was undertaken to compare the Wesson loss with the cup loss on crude oils obtained from different localities and to determine whether there is a useful correlation between results of the two methods. A sufficiently large number of Wesson loss determinations were made in duplicate and in triplicate to estimate reproducibility of results. Also, a limited amount of data were obtained to indicate how closely the Wesson loss is indicative of the actual neutral oil content of crude.

Experimental

Total neutral oil was determined by a modification of the Wesson method as described by Jamieson (5). This value subtracted from 100 was reported as the Wesson loss. Wesson loss was determined on 60 laboratory retained samples of crude soybean oil and 40 samples of crude cottonseed oil picked at random from tank car shipments received from the middle of December, 1946, through March, 1947; 30% of the determinations were made in triplicate and the rest in duplicate.

Refining loss was determined on identical samples, using the A.O.C.S. official method Ca 9a-41, which, for the sake of brevity, is herein referred to as the cup method, and results as cup loss. Cup loss as recorded represents the "settlement" loss as defined in trading rules of the N.C.P.A. or N.S.P.A. In almost all cases the settlement loss is the lowest of the several results on the same sample, using different strengths and amounts of lye.

In order to determine whether or not all of the neutral oil is recovered in the Wesson method, Wesson loss was determined on a sample of refined and bleached oil. Since soap is a catalyst for hydrolysis of neutral oil, it was thought advisable to determine the effect of various soap concentrations on neutral

TABLE 1—CSO

Sample	% FFA	Cup Loss	Wesson Loss (1)	Wesson Loss (2)	Wesson Loss (3)	Wesson Loss Average	Wesson Loss Deviation
1	0.9	5.8	2.18	2.19	2.28	2.22	.10
2	1.5	7.2	3.15	2.95	3.26	3.12	.31
3	1.9	9.1	3.79	3.73	3.80	3.77	.07
4	1.1	6.6	2.69	2.67	2.60	2.65	.09
5	2.8	9.3	3.92	4.05	3.87	3.95	.18
6	2.4	8.8	3.50	3.66	3.53	3.56	.16
7	1.7	8.0	4.39	4.31	4.33	4.34	.08
8	1.1	7.7	2.91	2.92	2.81	2.88	.10
9	1.1	7.9	3.10	3.14	3.26	3.17	.16
10	1.3	7.4	3.01	3.12	2.98	3.04	.14
11	2.0	7.3	3.54	3.53	3.49	3.52	.05
12	1.4	8.0	3.32	3.19	3.07	3.19	.25
13	1.3	3.4	2.02	2.03	1.84	1.97	.19
14	1.4	7.4	3.38	3.22	3.29	3.30	.16
15	2.0	8.3	4.71	4.64	4.69	4.68	.07
16	1.4	7.8	3.32	3.28	3.29	3.30	.04
17	2.1	7.3	3.57	3.51	3.56	3.54	.06
18*	1.7	17.0	10.51	10.05	10.03		
18					10.31	10.22	.48
19	1.5	8.1	3.47	3.42		3.45	.05
20	1.1	6.9	2.30	2.25		2.27	.05
21	1.3	7.5	3.18	3.11		3.15	.07
22	1.9	7.0	2.56	2.50		2.53	.06
23	1.4	7.4	2.87	2.79		2.83	.08
24	1.7	6.1	3.29	3.31		3.30	.02
25	2.2	8.1	3.59	3.53		3.56	.06
26	1.9	7.4	3.45	3.50		3.48	.05
27	1.0	8.3	3.12	3.28		3.20	.16
28	2.5	9.4	3.92	3.80		3.86	.12
29	1.1	7.1	3.08	3.09		3.08	.01
30	1.5	8.2	3.48	3.47		3.48	.01
31	1.5	8.5	3.43	3.32		3.38	.11
32	1.6	6.7	3.15	3.05		3.10	.1
33	1.5	9.0	3.83	3.88		3.86	.05
34	1.3	8.4	3.78	3.83		3.80	.05
35	1.3	6.5	3.03	3.10		3.07	.07
36	1.6	7.5	3.26	3.29		3.28	.03
37	1.6	7.1	3.03	3.11		3.07	.08
38	1.3	7.0	2.94	2.91		2.93	.03
39	1.3	6.8	3.18	3.10		3.14	.08
40	1.7	9.2	3.51	3.69		3.60	.18

* Tank bottoms.

oil recovery. This was done by determining Wesson loss of refined and bleached oil to which different percentages of free fatty acids, prepared from a separate portion of the oil, had previously been added. The fatty acids are, in the course of the test, converted to soap in the saponification procedure prior to the extraction of the neutral oil. It was assumed that the per cent free fatty acids plus per cent volatile matter, plus per cent insoluble impurities should equal the Wesson loss if the latter is indicative of the total neutral oil content. The effects of mucilaginous materials and other non-oil constituents of crude on recovery of neutral oil were not investigated. Attempts were made to determine total fatty acid content of the aqueous phase separated from the neutral oil, but results were not reproducible.

Reproducibility of Results

Standard deviation for Wesson loss results was calculated from the sum of the squares of half the differences in individual samples tested in duplicate. For samples tested in triplicate this calculation was based on the two results that were at the greatest variance. For the 40 samples of cottonseed oils the standard deviation was .068. For the 40 samples of soybean oils, excluding one extremely erratic result, the standard deviation was .073. Considering only the two determinations that checked closest of the samples tested in triplicate, the standard deviation was .033.

Cup loss determinations were not made in duplicate on the present set of samples. However, the standard deviation for the cup test has been previously determined in this laboratory. With the same method of calculation on duplicate tests as herein used for the Wesson loss results, the standard deviation was 0.270 for both cottonseed and soybean oils.

These data indicate that the Wesson method is about four times more accurate than the cup method in terms of deviation of results. However, since the cup loss is approximately twice as large as the Wesson loss, the latter can practically be considered as only twice as accurate in reproducibility as the cup method based on these data. Complete results obtained on cottonseed oil are given in Table I and on soybean oil in Table II.

TABLE II—SBO

Sample	Type of Oil	% FFA	Cup Loss	Wesson Loss (1)	Wesson Loss (2)	Wesson Loss (3)	Wesson Loss Average	Wesson Loss Deviation
1	Exp.	0.7	6.6	3.48	3.50	3.53	3.50	.05
2	Hyd.	1.0	6.4	3.42	3.51	3.43	3.45	.09
3	Exp.	1.2	9.3	5.21	5.21	5.33	5.25	.12
4	Hyd.	1.1	8.3	4.85	4.79	4.69	4.74	.16
5	Hyd.	1.4	11.3	6.09	6.15	6.07	6.10	.08
6	Hyd.	1.2	7.9	4.72	4.85	4.89	4.82	.17
7	*Exp.	0.7	7.6	4.7	5.03	6.67		
7				4.09	4.74	5.96		
7				5.31	6.03	7.07	5.62	2.98
8	Exp.	1.3	7.1	3.90	3.86	3.85	3.87	.05
9	Exp.	1.3	5.8	2.98	2.87	2.93	2.93	.11
10	Exp.	0.6	5.8	2.75	2.84	2.84	2.81	.09
11	Exp.	0.7	6.7	3.05	3.17	3.05	3.09	.12
12	Exp.	1.4	13.5	7.49	7.20	6.91	7.20	.58
13	Hyd.	0.6	5.2	2.54	2.58	2.51	2.54	.07
14	Hyd.	1.2	10.1	6.31	6.30	6.28	6.30	.03
15	Hyd.	1.0	8.1	4.43	4.28	4.30	4.34	.15
16	Hyd.	0.8	3.0	1.59	1.45		1.52	.14
17	Hyd.	1.8	9.4	6.57	6.51		6.54	.06
18	Hyd.	1.1	8.4	4.53	4.71		4.62	.18
19	Hyd.	1.3	8.5	4.98	4.95		4.97	.03
20	Hyd.	1.1	8.2	4.03	4.27		4.15	.24
21	Hyd.	1.3	9.8	5.48	5.58		5.53	.1
22	Exp.	0.6	5.5	3.74	3.80		3.77	.06
23	Exp.	1.5	5.4	2.89	2.89		2.89	0
24	Exp.	0.7	6.0	3.36	3.43		3.40	.07
25	Exp.	0.5	4.9	3.29	3.33		3.31	.04
26	Exp.	0.4	4.3	3.53	3.56		3.55	.03
27	Exp.	0.7	7.1	3.98	3.88		3.93	.10
28	Exp.	0.6	7.3	5.51	5.46		5.49	.05
29	Exp.	0.7	8.1	4.44	4.49		4.47	.05
30	Hyd.	1.3	7.1	2.90	2.99		2.95	.09
31	Hyd.	0.9	6.6	4.30	4.15		4.23	.15
32	Hyd.	0.6	4.8	3.04	3.06		3.05	.02
33	Exp.	0.8	7.5	4.14	4.17		4.16	.03
34	Hyd.	0.7	5.9	4.48	4.28		4.38	.2
35	Hyd.	1.1	7.9	4.62	4.81		4.70	.19
36	Exp.	0.4	5.1	3.21	3.17		3.19	.04
37	Exp.	0.6	5.5	2.98	3.15		3.07	.17
38	Hyd.	0.9	7.4	3.99	3.95		3.97	.04
39	Hyd.	1.0	7.9	4.31	4.44		4.38	.13
40	Exp.	0.9	7.5	3.88	3.99		3.93	.11
41	Hyd.	1.0	7.9	4.65	4.93		4.79	.28
42	Hyd.	0.9	7.7	4.50	4.35		4.43	.15
43	Hyd.	1.1	8.4	5.27	5.55		5.41	.28
44	Hyd.	1.1	8.3	5.06	5.21		5.14	.15
45	Exp.	0.8	7.0	3.87	3.94		3.91	.07
46	Hyd.	1.0	8.0	4.28	4.27		4.28	.01
47	Hyd.	1.0	8.8	5.17	5.13		5.15	.04
48	Exp.	1.1	12.3	6.76	6.77		6.77	.01
49	Exp.	1.3	11.2	6.79	6.99		6.89	.20
50	Hyd.	1.0	7.4	4.58	4.51		4.55	.07
51	Exp.	0.9	4.5	2.48	2.64		2.56	.16
52	Hyd.	1.0	8.2	4.27	4.35		4.31	.08
53	Exp.	1.0	8.6	4.95	5.17		5.06	.22
54	Exp.	0.7	7.2	3.64	3.59		3.62	.05
55	**Ext.	0.4	2.0	2.01	2.15		2.08	.14
56	Exp.	0.9	12.2	7.27	7.24		7.26	.03
57	Exp.	0.7	6.1	3.42	3.55		3.49	.13
58	Exp.	0.9	5.8	4.15	3.92		4.04	.23
59	Hyd.	1.2	9.1	5.17	5.14		5.15	.03
60	Hyd.	1.2	8.7	4.31	4.39		4.35	.08

* Oil contained hard lumps of suspended matter that could not be evenly dispersed.
** Extracted—Degummed oil.

Significance of Wesson Results

Wesson losses of refined and bleached cottonseed oil with and without added free fatty acids are shown in Table III. The original oil contained 0.1% free fatty acids, 0.07% volatile matter, and an insignificant quantity of insoluble impurities. Therefore, 0.17 plus the percentage of free fatty acids added was subtracted from the Wesson loss to account for these impurities. Results indicate that a slight quantity of neutral oil is saponified in the presence of relatively large amounts of soap. Assuming that non-fat constituents other than free fatty acids have no effect on hydrolysis or entrainment of neutral oil when the Wesson method is followed, the quantity of neutral oil as determined should be correct within 0.2% for crude oils containing up to 2% free fatty acids.

TABLE III

Sample	% FFA Added	Wesson Loss			% Loss of Neutral Oil		
		Det. 1	Det. 2	Average	Det. 1	Det. 2	Average
1	0	.17	.16	.165	0	—.1	—.005
2	.54	.80	.83	.815	+.09	+.12	+.105
3	1.03	1.22	1.29	1.255	+.02	+.09	+.055
4	2.04	2.38	2.43	2.405	+.17	+.22	+.195

Cup Loss vs. Wesson Loss

A comparison of cup loss and Wesson loss of crude cottonseed and soybean oils produced in different sections of the country is shown in Table IV. The soybean crudes were fairly representative of the entire soybean producing area, but most of the cottonseed crudes were produced in Texas.

Hydraulic pressed soybean oil averaged somewhat higher in free fatty acid content and refining loss than expeller pressed, but the average ratio between the refining loss and Wesson loss was practically the same. The ratio varied as much as 40% on individual cottonseed oil samples and 50% on soybean oil samples.

Only one sample of extracted soybean oil was tested, and it was also a degummed oil. Refining loss and Wesson loss were the same on this oil. A sample of cottonseed oil tank bottoms with 17% refining loss showed a Wesson loss of 10.2%. Results obtained on these two samples were excluded from averages.

Since the data organized in this manner (Table IV) show a very poor correlation between Wesson loss and cup loss on the oils as a whole, they were grouped according to percentage levels of cup loss and also percentage free fatty acids. These results, given in Tables V and VI, show that the ratio of cup loss to Wesson loss varied about as much in each of these groupings as it did in the general grouping of Table IV. Therefore, it appears that the total neutral oil content of crudes cannot accurately be predicted from the results of the cup refining test.

TABLE V

Kind of Oil	Loss Limits—%	Avg.% Refining Loss	Avg.% Wesson Loss	Ratio of Cup Loss to Wesson Loss		
				High	Low	Average
CSO	7 & below	6.28	2.72	3.04	1.73	2.31
CSO	7-8	7.50	3.27	2.67	1.85	2.30
CSO	8-10	8.62	3.68	2.59	1.77	2.34
SBO	5 & below	4.30	2.80	1.98	1.21	1.54
SBO	5-6	5.60	3.31	2.04	1.35	1.69
SBO	6-7	6.57	3.62	2.17	1.56	1.81
SBO	7-8	7.56	4.28	2.4	1.33	1.79
SBO	8-9	8.33	4.72	2.0	1.55	1.77
SBO	Over 9	10.82	6.30	1.87	1.44	1.72

Summary

Wesson loss and cup refining loss were determined on 100 samples of crude cottonseed and soybean oils produced in different sections of the country. The Wesson method gave more reproducible results than the cup method. Wesson loss results were found to be closely indicative of the total neutral oil content of crude oils. The average ratio of cup loss to Wesson loss was 2.3 for crude cottonseed oil and 1.7 for crude soybean oil. The ratio varied considerably for individual crude oils of a given kind, or type within each kind, or a local producing section.

Therefore, no real useful correlation between the results of the two methods was found to exist.

TABLE VI

Kind of oil	Limits—%	Avg.% FFA	Avg.% Refining Loss	Avg.% Wesson Loss	Ratio of Cup Loss to Wesson Loss		
					High	Low	Average
CSO........	Below 1.4	1.17	6.7	2.87	3.04	1.73	2.33
CSO........	Over 1.8	2.08	8.18	3.76	2.56	1.77	2.18
SBO.......	Below 0.8	0.62	6.0	3.51	2.17	1.21	1.71
SBO.......	Over 1.1	1.29	8.6	5.02	2.40	1.44	1.79

REFERENCES

1. "Oil and Soap," June, 1942—page 97.
2. "Cotton Oil Press, 6, No. 4—33, 1922.
3. "Oil and Fat Industries," 3, 307, 1926.
4. "Oil and Soap," May, 1942, page 83.
5. "Vegetable Fats and Oils," George S. Jamieson, 2nd edition, 1943, page 454.

TABLE IV

Kind of Oil	Source	No. of Samples Tested	Average % FFA	Average Refining Loss	Average Wesson Loss	Ration of Refining Loss to Wesson Loss		
						High	Low	Average
CSO	Average—All	39	1.55	7.53	3.25	3.04	1.73	2.32
SBO	Average—All	58	0.95	7.47	4.33	2.40	1.21	1.72
SBO Expeller	Average—All	29	0.82	7.04	4.13	2.17	1.21	1.71
SBO Hydraulic	Average—All	31	1.07	7.87	4.53	2.40	1.35	1.73
CSO	E. Texas	16	1.59	7.87	3.34	2.67	2.20	2.45
CSO	W. Texas	15	1.30	7.47	3.11	3.04	1.77	2.23
CSO	Okla., La., Miss.	8	1.75	7.21	3.45	2.56	1.73	2.09
SBO	Texas	13	1.11	9.52	4.63	2.00	1.35	2.10
SBO	Oklahoma	12	1.04	7.61	4.31	2.40	1.56	1.75
SBO	Ark. & La.	6	1.12	8.30	5.01	1.85	1.55	1.66
SBO	Midwest	28	0.80	7.61	4.04	2.17	1.21	1.88

NOTE: Only 4 of the 40 cottonseed oil crudes were known to be expeller oils, remainder presumed to be hydraulic.