the butterfat triglycerides represent pooled contributions of triglycerides from two or more distinct triglyceride populations, each of which may possess a random distribution for its fatty acids. It has already been postulated that both blood and the mammary glands contribute triglycerides to butterfat (4). In the lactating woman, milk fat triglycerides have been shown (14) to be derived from dietary and depot fat, as well as from fat synthesized in the mammary glands.

Although the fatty acids from the individual triglyceride peaks have not yet been isolated and identified, mathematical evaluations of the peak composition based on the peak proportions indicate that such short chain fatty acids as butyric and caproic, which occur to a significant extent in this fat, are found exclusively in combination with medium and long chain fatty acids, as there are only traces of triglycerides found of carbon number lower than 26. Also, this would mean that these fatty acids occur rarely in combinations of two per given triglyceride molecule. These observations are supported by the analyses of the molecular distillates of butter oil, all of which have been shown to contain about the same fatty acids despite considerable differences in the carbon number of the constituent triglycerides. Even with short chain triglyceride enrichments approximating 20–25 fold, there were no indications (8) obtained of the occurrence of any tributyrin, tricaproin, or even any significant amounts of the dibutyro- or dihexano-glycerides of medium chain length fatty acids Support for such a distribution for butyric acid residues is also suggested by the observation that pancreatic lipase is capable of releasing practically all of the butyric acid by hydrolyzing the alpha-, alpha'-linkages of the glycerides (15).

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# A Comparison of the Cup Refining Loss and Neutral Oil Determinations for Evaluating Crude Soybean Oil[1]

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## Abstract

Data from 833 non-degummed and degummed soybean oil samples, which were analyzed by both the neutral oil loss and cup loss methods, were examined, and it was found that the total premiums paid under the cup loss method and the proposed National Soybean Processors Association Technical Committee's neutral oil analysis were the same. However, better quality oils would have received a higher premium, while poorer oils would have been penalized more heavily under the new procedure.

## Introduction

APPROXIMATELY 42 years ago a group of cottonseed crushers and oil refiners operating through an association of the Interstate Cottonseed Crushers set a series of specifications for crude cottonseed oil. At that time the average kettle refining loss was 9.0%. Oils having a loss of 9% or less were considered prime. Other specifications were included such as odor, taste, and color. Penalties were assessed for oil having more than 9.0% loss at the rate of three-quarters of 1% of the purchase price for each percent in excess of 9.0%. This led the Chemists' Committee of the Association (1) to develop what is commonly referred to as the cup loss determination (2) for trading.

It has been reported (1) that some mill managers established the practice of adding cottonseed meal to oil containing less than 9.0% cup loss because the crusher could then sell his meal at oil prices. The practice spread and in 1927 the refiners agreed to pay a premium for oils having a settlement loss under 9.0%, at the same rate as the penalty. The Interstate Cottonseed Crushers Association was succeeded by the National Cottonseed Products Association and since that time the cup loss has served very well for controlling the quality of crude oil. About 1936 soybean oil began to appear on the vegetable oil market in appreciable quantity. However, no means of trading on quality existed. In World War II the OPA froze the vegetable oil prices and the premium system for cottonseed oil put soybean oil at a definite disadvantage. This led to the establishment of the National Soybean Processors Association. It was not until after the war when price controls were removed that the premium system using the cup loss method was organized. Prime oil was set at 7.0% cup loss and the same premium rate as cottonseed oil.

For many years the cup loss test has served the refineries as a means of measuring plant efficiency, and the method worked very well during the period when open kettle refining was paramount. Later technical and mechanical improvements in refining methods reached the point where plant losses were generally lower than the laboratory estimates. For

[1] Presented at the AOCS meeting in Toronto, Canada, 1962.

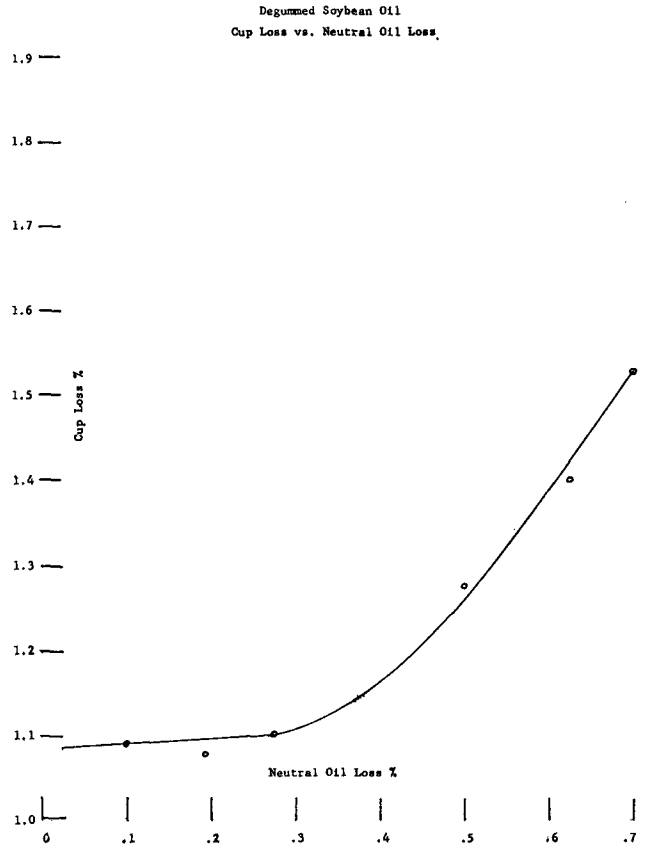


FIG. 1.

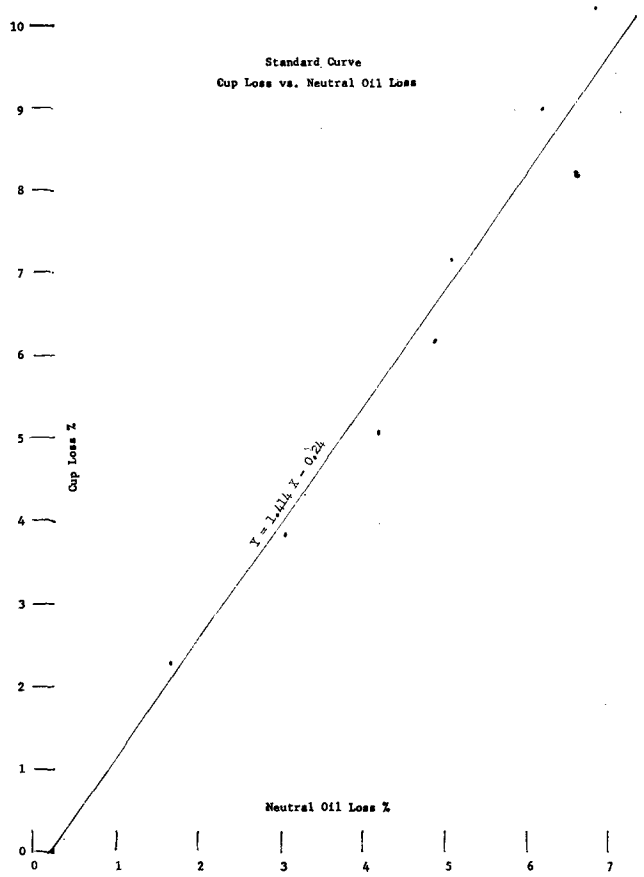


FIG. 2.

many years our refining committee has worked to improve the existing methods. It also became apparent that it was unsound to confuse crude oil evaluations with a "trial test" rather than an analysis. In 1950, when Linteris and Handschumaker (3) published a new chromatographic technique (neutral oil determination) some refineries began to use it as a yardstick of plant efficiency.

The Technical Committee of the National Soybean Processors' Association has been studying the neutral oil determination as a means of trading in place of the present cup loss. The value 100 minus the neutral oil has been suggested as a method of trading. Some people have referred to this method as chromatographic loss, however, the former is a better description of the assay. It has been suggested that crude oil be sold as 100% neutral oil. The base price would need to be raised to permit penalties on all crude oils such that a normal oil would bring the current market price.

Sipos (4) reported a detailed mathematical evaluation of the relationship of cup loss and neutral oil loss in soybean oil. He concluded that most of the discrepancy in correlation between the two is inherent in the cup loss assay and he states, "The neutral oil loss is an accurate, easily obtainable index for unavoidable losses resulting from the non-neutral oil fraction of oil."

## Discussion

The purpose of this paper is to present a means of arriving at an equitable procedure for trading for the transition from cup loss to neutral oil loss. The cup loss has served its purpose as a pilot test for estimating alkali refining loss but it falls short of being a precise chemical determination. Neutral oil loss on the other hand has been shown by Linteris and Handschumaker (3) to quantitatively separate the free fatty acids, phospholipids, and moisture. It has been observed that alkali refined oil will yield values very close to zero by this method. Research in this laboratory has indicated, as shown in Figure 1, that as the efficiency of the degumming operation increases a graduated decrease in neutral oil loss is obtained as shown by the graph. The corresponding cup loss values, however, reached a plateau at about 1.1%.

On the other side of the picture, soybean oil containing 6% and more cup loss is penalized by the fact that the cup method does not remove all the entrained oil. In this study an additional 0.8–1.6% oil was recovered in a laboratory tube centrifuge after completing the cup loss assay with oils ranging from 6–10% cup loss.

To establish the relationship between neutral oil loss (N.O.L.) and cup loss, graded quantities of tank bottoms were added to one sample of laboratory refined oil. The tank bottoms were essentially free of meal fines and assayed 33.2% cup loss. A linear relationship was obtained (standard curve) which had the following equation: (Figure 2)

$$\text{Cup Loss} = 1.414\ \text{N.O.L.} - 0.24 \qquad [1]$$

Quadratic curvature was not observed. As a further check a number of high cup loss oils from different sources were analyzed by both methods, as is and diluted with equal parts of alkali refined oil. These data yielded the following linear regression: (Figure 3)

$$\text{Cup Loss} = 1.194\ \text{N.O.L.} + 0.58 \qquad [2]$$

The above two equations show that there is not a 1:1 correspondence between the two methods, but

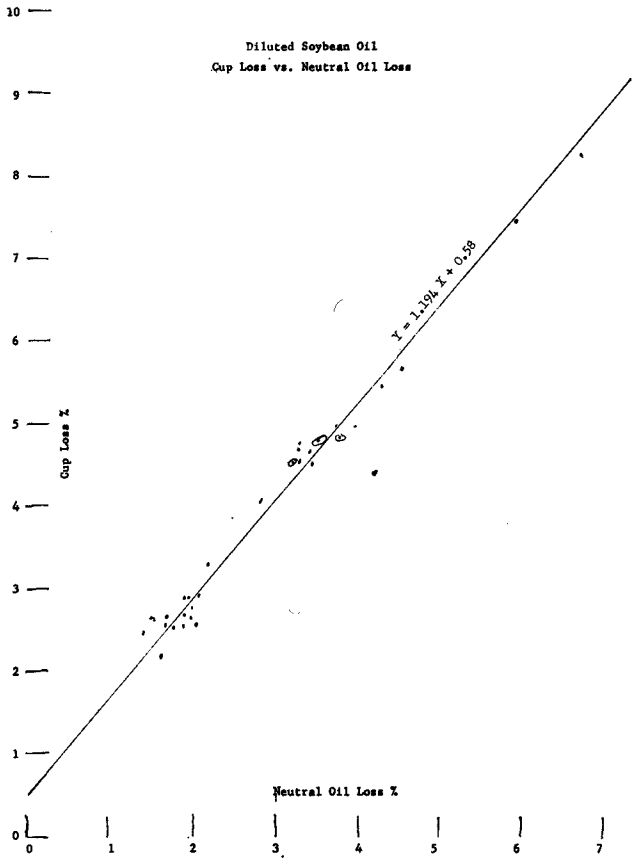


FIG. 3.

such would not have been anticipated. Since there are certain losses in the cup loss method such as saponification of neutral oils due to excess alkali and possible entrainment of triglycerides in the refining loss material, one would *a priori* expect a slope of greater than one, as was the case in the above two regression equations. Similar data have been obtained by Sanders (5) in a survey of non-degummed and degummed oils which yielded the following regression equation:

$$\text{Cup Loss} = 1.35\ \text{N.O.L.} + 0.29 \qquad [3]$$

However, Smith (5) obtained the following equation for only degummed oils:

$$\text{Cup Loss} = 0.60\ \text{N.O.L.} + 0.50 \qquad [4]$$

Thus it would appear that the above regression coefficient does not fit the theory discussed above. This was most likely due to a rather narrow range of cup loss values even though the total number of samples were very large, since only degummed oils were involved.

The present trading rules of the National Soybean Processors Association employ the following formula for calculating oil premiums by the cup loss method:

$$\text{Premium} = 7.0 - \text{Cup Loss}\ (0.0075)\ (\text{Price}) \qquad [5]$$

If we substitute [1] into [5] we obtain

$$\text{Premium} = (5.43 - 1.0602\ \text{N.O.L.})\ 0.01\ (\text{Price}) \qquad [6]$$

This formula is approximately the same as one under consideration by the National Soybean Processors Association's Technical Committee which is of the following form:

$$\text{Premium} = (5.00 - \text{N.O.L.})\ 0.01\ (\text{Price}) \qquad (7)$$

In order to determine the effect of several trading

TABLE I

Oil Premiums

| Number samples | Cup loss, % | Neutral oil loss, % | Formula used | | |
|---|---|---|---|---|---|
| | | | Current [5] | Standard curve [6] | Formula [7] |
| 1 | 1.50 | 0.4 | 0.412 | .500 | .460 |
| 1 | 1.50 | 0.5 | 0.412 | .490 | .450 |
| 9 | 1.43 | 0.6 | 0.418 | .479 | .440 |
| 4 | 1.45 | 0.7 | 0.416 | .469 | .430 |
| 7 | 1.47 | 0.8 | 0.415 | .458 | .420 |
| 3 | 1.47 | 0.9 | 0.415 | .448 | .410 |
| 4 | 1.52 | 1.0 | 0.411 | .437 | .400 |
| 2 | 2.50 | 1.4 | 0.338 | .394 | .360 |
| 3 | 3.03 | 1.5 | 0.292 | .384 | .350 |
| 5 | 2.90 | 1.6 | 0.308 | .373 | .340 |
| 16 | 3.28 | 1.7 | 0.278 | .363 | .330 |
| 6 | 3.23 | 1.8 | 0.282 | .352 | .320 |
| 16 | 3.38 | 1.9 | 0.271 | .342 | .310 |
| 26 | 3.59 | 2.0 | 0.256 | .331 | .300 |
| 37 | 3.43 | 2.1 | 0.263 | .320 | .290 |
| 39 | 3.69 | 2.2 | 0.248 | .310 | .280 |
| 39 | 3.69 | 2.3 | 0.248 | .299 | .270 |
| 62 | 3.79 | 2.4 | 0.241 | .288 | .260 |
| 69 | 3.62 | 2.5 | 0.253 | .278 | .250 |
| 87 | 3.76 | 2.6 | 0.243 | .267 | .240 |
| 72 | 3.76 | 2.7 | 0.243 | .257 | .230 |
| 56 | 3.90 | 2.8 | 0.233 | .246 | .220 |
| 67 | 3.98 | 2.9 | 0.226 | .236 | .210 |
| 55 | 4.04 | 3.0 | 0.222 | .225 | .200 |
| 44 | 4.00 | 3.1 | 0.225 | .214 | .190 |
| 34 | 4.22 | 3.2 | 0.208 | .204 | .180 |
| 14 | 4.21 | 3.3 | 0.209 | .193 | .170 |
| 19 | 4.28 | 3.4 | 0.204 | .182 | .160 |
| 12 | 4.37 | 3.5 | 0.197 | .172 | .150 |
| 11 | 4.58 | 3.6 | 0.182 | .161 | .140 |
| 3 | 4.23 | 3.7 | 0.208 | .151 | .130 |
| 3 | 4.50 | 3.8 | 0.188 | .140 | .120 |
| 1 | 4.30 | 3.9 | 0.202 | .130 | .110 |
| 3 | 4.40 | 4.0 | 0.195 | .119 | .100 |
| 1 | 5.00 | 4.1 | 0.150 | .108 | .090 |
| 1 | 6.20 | 4.4 | 0.060 | .076 | .060 |
| 1 | 5.30 | 4.6 | 0.128 | .055 | .040 |
| Weighted average premium | ..... | ..... | 0.244 | .268 | .241 |

[5] Prem. = (7.0 — C.L.) .0075 × $10.00.
[6] Prem. = (5.43 — 1.0602 N.O.L.).01 × $10.00.
[7] Prem. = (5.00 — 1.00 N.O.L.).01 × $10.00.
Oil Value = 100 # price $.10/lb.

formulas on the premiums which would have been paid under varying trading rules, data from 833 soybean oil samples, collected during 1961 and 1962, were analyzed by both cup loss and neutral oil methods (Table I). Those included non-degummed and some degummed. Premiums were calculated, based on 100 lb of oil at a market price of 10¢/lb. The fourth column of Table I lists the premiums paid under the condition of using the cup loss assay procedure. Column 5 shows the premiums using the formula derived in [1] based on the standard curve assay of the neutral oil method. The final column in this table shows the premiums which would have been paid using [7] as proposed by the National Soybean Processors Association's Technical Committee. These data show that when we compare the cup loss method with [7] that the average premium paid, i.e., total, is the same for both methods of calculation. However, the higher refining loss oils are penalized more heavily when using the neutral oil method and, conversely, rewarded more heavily when of high quality. This appears to be a desirable situation. The premiums based on [6] are, on the average, somewhat higher than those using [7] which may be due to the fact that this regression equation was based on only limited data. However, the same trend, as discussed above, existed for this assay.

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