

dered alumina. After several rinsings with 25-ml. portions of diethyl ether, the solvent is evaporated over a water bath and the oil is weighed again. In all chromatographic determinations a solution of 975 cc. of anhydrous diethyl ether and 25 cc. of anhydrous (absolute) ethyl alcohol was used.

To say that this loss is refining loss in the ordinary sense is not entirely true. It is not the loss that would be expected in caustic refining. Instead it is the removal of all non-lipid fractions in the oil. What remains is neutral oil. The chromatographic method simply removes the free fatty acids, pigments, and other non-lipid bodies. In this respect it is similar to the Wesson loss (Table III).

TABLE III  
Chromatographic Loss Method  
% Loss (Duplicates)

	1	2
Degummed.....	2.04	2.02
Degummed.....	2.48	2.42
Crude.....	4.28	4.28
% Free fatty acid (av.).....		.08
% Total gossypol (av.).....		.06
Color (av. Lovibond Red).....		5.5

Here we see a comparison of the chromatographic loss with the regular cup method, using the same oil in each case. In every case the chromatographic loss is lower than the cup loss and in most cases considerably so. Judging from the very close checks with duplicate samples, shown in Table IV and the lower

TABLE IV  
A Comparison of Refining Methods  
(% Loss)

Crude		Degummed	
Official Method	Chromatographic	Official Method	Chromatographic
8.3.....	4.7	5.2	2.6
9.3.....	4.6	5.7	3.0
8.7.....	4.8	6.3	2.8
8.3.....	3.8	5.2	2.3
10.3.....	4.1	4.9	2.3
7.9.....	4.8	3.6	2.1
9.7.....	4.2	4.1	2.5

loss figures, it would seem that in this method practically 100% of the neutral oil is recovered. The oil is in every respect a refined oil. The color is very low, as is the free fatty acid and the gossypol content. The colors approach the bleached oil figures in some cases. Since very small samples are used, usually 2-3 g., reading of individual colors, using the Lovibond glasses, is impractical. Colors can only be read by diluting with 25 ml. of anhydrous diethyl ether and placing the mixture in a Beckman DU or other suitable spectrophotometer, using selected wavelengths in much the same manner as is done in the A.O.C.S.

spectrophotometric method to convert wavelength readings into comparable Lovibond Red figures (2).

Since the chromatographic method is a true measure of what is actually oil and what is not, it would seem that it would be a more accurate basis for accounting for losses and gains in the refinery. Heretofore the refiner has been able to realize savings over the laboratory cup loss of from 25% to 50%. In other words, he has always, since the advent of continuous centrifuge refining, been able to beat what the laboratory says he should do with a given oil. This seems to be a rather archaic way of doing business. The laboratory should be the one to set the pace and let the refiner try to shoot for the mark it sets.

If this were to come about, a re-evaluation of our laboratory-refinery relationship would necessarily ensue. Instead of the refiner being able to beat what the laboratory says, he would then try to approach the figure that the laboratory says is perfection. Then instead of savings over cup loss of 25 to 50%, he would perhaps do only 95-98% as well as the laboratory does.<sup>2</sup> A revamping of present accounting systems would be a natural by-product if this came about.

### Summary

In the foregoing we have set forth our views and findings on possible ways of handling degummed cottonseed oil in the laboratory. Foreseeing the day when degummed cottonseed oil will come into prominence, we offer these suggestions and findings as an incentive to spur collaborative efforts in a search for a better laboratory method. Two possibilities have been explored. One is a centrifuge method of refining, approximating actual refining conditions in present-day refineries. The other is a chromatographic determination of total neutral oil. It is indeed heartening to know that the A.O.C.S. total neutral oil subcommittee is actively engaged in a collaborative program of work on such methods as the chromatographic loss method. This is good evidence that we are making a realistic approach to the problem of better laboratory methods.

### REFERENCES

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<sup>2</sup>The only actual figure is from one refinery showing an average figure of 90%.

### Erratum

ACCORDING to Thomas F. Boyd of the Industrial Test Laboratory, Philadelphia Naval Shipyard, Philadelphia, Pa., a correction should be made as follows: "in the article, 'An Investigation of the Adsorption of Detergent and Builders onto Cotton and Soil by Radio-tracer Methods,' 33, 614-619 (1956), under Table I, lines 15 20, 27 58, and 30 38 should read >15 20, >27 58, and >30 38."