REPORT OF THE FREE FATTY ACID COMMITTEE

A. O. C. S. 1935

WHEN President Hutchins asked me to act as chairman of the Free Fatty Acid Committee, he also asked me to prove or disprove the correctness of several points where the present method has been questioned in the past.

Since there have been no new ideas brought forward in regard to the present method as a whole, it seemed the most important work was to prove whether by following it as at present written we are extracting all of the oil, evaporating all the solvent, and correctly titrating the fatty acids. If this is being done there can be no question of the accuracy of the results.

In the following work then, we wish to state at the outset that the present official method was followed except where differences are noted.

The first point obviously is whether or not we are getting all the meats. In the first two samples worked on by the committee the percentage of meats and hulls was determined by cutting and picking. The actual meats were shown to be 55.45 per cent and 51.56 per cent by this method.

The average percentages of meats obtained by the members of the committee who reported were 55.9 per cent and 51.6 per cent, determined by grinding and sieving as in the present method. This close agreement with the theoretical percentage proves that the meats can be more completely separated than is done in the oil mill.

The separated hulls when picked over carefully by hand proved to contain only 0.25 per cent and 0.17 per cent whole seed and uncut meats showing the separation to be almost perfect. In view of this, it seems entirely unnecessary to worry about the effect of hulls in the meats to be extracted.

The two questions as to whether the grinding and extracting of the meats is sufficient to remove about all of the oil can be answered by the determination of the percentage of oil remaining in the extracted meats.

The committee members were asked to regrind and re-extract the residual meats following the same procedure as for total oil in seed. The following results were submitted:

Jamieson	Seed A	3.45%	Seed B	3.01%
Rettger	Seed A	1.91%	Seed B	1.16%
Robertson	Seed A	2.22%	Seed B	1.88%
Byers	Seed A	3.63%	Seed B	2.17%
Evans	Seed A	10.96%	Seed B	6.97%
Cox	Seed A	4.87%	Seed B	3.57%
Average, excluding high results		3.22%		2.16%

Inasmuch as these meats are purer than oil mill meats, we are extracting a much larger percentage of the total oil in the seed than can be expressed in any oil mill.

The point as to whether the oil first extracted differs from the last portion does not seem to enter greatly into any discussion of the correctness of the method, when we have shown that we are leaving only an extremely small percentage unextracted.

A simple calculation will show that even if this oil contained 50 per cent more fatty acids than the oil extracted, the percentage of free fatty acid to be reported would not be affected, being 1.13 per cent rather than 1.1 per cent and 6.0 per cent rather than 5.9 per cent on the particular samples.

However, our experiments do not show excess high fatty acid to be present in the residual oil and it is interesting to note that the laboratory reporting the highest oil left in meats was exactly on the average fatty acid reported by all the committee.

The removal of the solvent is another point studied by the committee. The present method calls for leaving the extraction flask on the steam bath for ninety minutes. In none of the other methods for the removal of the solvent is a time specified. Probably in the majority of cases the time given in the method is sufficient, but we think it would be better to make the method uniform with the other methods of the society.

There seems to be no doubt, however, about the fact that prolonged heating, even on a steam bath, darkens the oil and renders the titration more difficult.

The present method calls for the addition of gasoline and alcohol to the weighed oil before titration. A review of the cooperative work of this year on this point shows that in all but a few cases identical results are obtained whether the gasoline is added or not. The use of the solvent dates back to the days when we were trying to titrate the small amount of oil obtained in the extraction of the seed to determine the oil content. Its antiquity is about its only recommendation.

Samples of high fatty acid seed were sent out this year to determine the effect of allowing the ground meats to stand for some time in the laboratory. A large quantity of meats from these seed were ground and mixed thoroughly.

Duplicate samples were then extracted immediately. Another set of duplicates were allowed to stand for four hours before being extracted. The results in practically every case showed from 0.1 per cent to 0.3 per cent increased fatty acid in the samples allowed to stand. These seed, too, were rather dry and it is the opinion of the committee that the difference would be even greater with damp, early-season seed.

It seems essential then that the meats be extracted as soon as prepared as called for in our present method.

The question of the indicator to be used is one which has been discussed several times. Some analysts prefer alkali blue to the official Phenolphthalein. Probably on extremely dark oils it gives a more distinct end point. However, as our method for crude oil gives us no choice of the indicator to be used in that determination, we do not think it would be advisable to add another possibility of variance by the addition or substitution of another indicator. When the present method was first adopted several years ago alkali blue was named as the indicator, but the change to Phenolphthalein was made for uniformity by a vote of the society in 1929.

There is one point in the present method that requires revision. The procedure requiring the ground meats to be passed through a 15-mesh screen must be deleted or at least changed.

In the first place, there is no 15-mesh screen on the market. In the second place, it would be impossible to pass the meats of a damp or high oil seed through such a screen if one were obtainable. The committee feels that this provision should be omitted and the method changed to read "Grind the meats in a Russwin No. 1 Food Chopper equipped with a 16-tooth blade, returning the first few grams that pass the knives to the hopper to insure proper grinding."

We also suggest that in the place of specifying one and one-half hours on the steam bath, the method be changed to "allow to remain on the steam bath until no trace of solvent remains."

We further suggest that the addition of 10 cc. of petroleum ether be omitted or made optional.

We believe that with the above changes, a close adherence to the method as written will give results as accurate at least as the sample submitted.

C. H. COX, Chairman.

ABSTRACTS

Oils and Fats

Edited by
W. F. BOLLENS nd M. M. PISKUR

Refining castor oil. R. Heublyum. *Mat. grasses* 27, 10463, 10482-3 (1935).—A brief review of work carried out during the past few yrs. on the refining of pharmaceutical and lubricating castor oil, and more particularly of H.'s process. (C. A. 27, 4432.)
A. PAPINEAU-COUTURE.

Varieties of fish oils and their uses. Louis J. Reizenstein. *Paint Varnish Production Mgr.* 12, 18, 20, 22-3 (June, 1935); cf. C. A. 29, 4191.[2]—A paper dealing with the chem. nature of fish oils, their outstanding characteristics and merits, the types of oils available, the particular application of these oils in their different fields, and the different type fish oils which are used in the various types of protective coatings. Twenty-two formulas of fish-oil applications are given.
W. H. BOYNTON.

Selective absorption in the examination of the unsaponifiable matter of marine oils. T. Thorbjarnarson, A. Santos Ruiz and J. C. Drummond. *Analyst* 60, 382-8 (1935).—In the study of the unsaponifiable matter from a variety of oils and fats in connection with investigations on vitamins A and E, selective absorption on Al_2O_3 proved of value. A tube evenly packed with Merck's Al_2O_3 was prepared by pouring a thick suspension of the oxide in "light petrol" (b. 40-60°). A thin wad of cotton wool was placed on top of the long column to prevent superficial disturbance when the liquid was added. The column of oxide should not be allowed to drain dry and should be kept covered with the "petrol" when not in use. Usually the passage of the soln. through the tube results in the formation of clearly marked zones indicated by coloration of the oxide. As washing with the solvent progresses the position of the zones may change and, as a rule, washing is continued until no further change is noted. The filtrate is evapd. *in vacuo*. The column of oxide is divided on a visual basis and the different portions of the oxide are eluted before the adhering petrol has evapd. The best eluting agent is usually a 1:1 or 3:1 MeOH-ether mixt. Sometimes, when pigments of the astacene type are present, the absorbent retains its color and MeOH or $CHCl_3$ with a little acid is best to dissolve it. The method was applied to the examn. of shark-liver oil, ratfish-liver oil and herring oil concerning which considerable analytical data are given.
W. T. H.

Courtesy "Chemical Abstracts"

Development of odor and flavor in mustard oil. J. A. H. Dale, D. Y. Athawale and P. N. Mathur. *J. Sci. Tech. India* 1, 103-5 (1935).—Mustard oil produced by the use of hydraulic presses or expellers has not heretofore been equal in pungency to the oil produced by the use of the native wooden rotary "ghanni" mill. This difficulty has been removed by the invention of a cooking kettle contg. an upper compartment where the milled seed is first treated with 3-4% of H_2O; it is then passed to a lower compartment and heated at 40° for 45 min. to develop the required odor by enzymic action. The meal is pressed twice. O. W. WILCOX.

Mustard seeds and mustard oils of the United Provinces. J. A. H. Dale, D. Y. Athawale and P. N. Mathur. *J. Sci. Tech. India* 1, 98-102 (1935).—Out of 50 samples produced from 7 varieties only 28 fall within the Bengal specifications, which require sapon. nos. 169-175 and I nos. 94-104. O. W. WILCOX.

Verifying the Ban method of extracting raw fat (raw vegetable oils). P. M. Ivanov. *J. Applied Chem.* (U. S. S. R.) 7, 1523-6 (1934).—Extn. of seeds with ether by the Ban method gives 0.5-0.9% less than extn. in a Soxhlet app. Since the amt. of oil is detd. by the residue left, ether once used can be used over again; about 40-50 cc. of ether is required. The entire detn. requires 6-7 hrs. in comparison with 15 hrs. for the Soxhlet method.

The significance of the individual constants of oils in comparative analyses. E. V. Dodonova and Z. P. Prizemina. *Bull. Applied Botany, Genetics, Plant Breeding* (U. S. S. R.) Ser. 3, No. 5, 223-53 (in English 253-4) (1934).—In detg. the quality of the common vegetable oils it is not important to det. all the consts. The acid and I no. are sufficient for the common oil plants. The Richert-Meissl no. is important in detg. the freshness of the oil. In the oils of the castor bean, sesame, perilla, cotton, mustard, rape and poppy the I no. varies within narrow limits. The Ac no. is characteristic only for oils of the series of ricinolic acids. No definite conclusions were drawn as to the interrelation of the I no. and drying capacity of the oil.
J. S. JOFFE.

A new steroisomer of eleostearic acid in pomegranate-seed oil. Yoshiyuki Toyama and Tomotaro Tsuchiya. *J. Soc. Chem. Ind., Japan* 38, Suppl. binding 182-5 (1935).—*Punicic acid,* a new stereoisomer of