DETERMINATION OF OIL IN FISH MEAL

The method for the determination of oil in fish meal which is given by the Association of Official Agricultural Chemists (AOAC) (1950) entails considerable manipulation, since two extractions of the meal with solvent are required. That is, the meal is first given an acetone extraction, and the extracted meal is digested with hydrochloric acid and then re-extracted with acetone. Such a procedure is much more time-consuming than are most of the methods for the determination of oil in other feedstuffs. Consequently, a number of feed-testing laboratories have been using a simple one-step ethyl-ether extraction for fish meal in spite of the fact that this method may give oil-content values that are more than 75 percent too low.

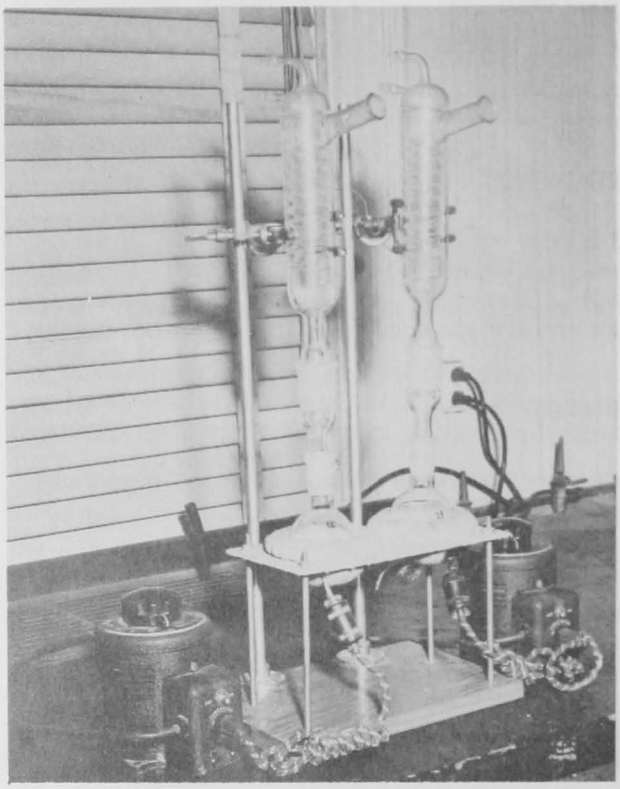

Fig. 1 - Apparatus for refluxing experiments with fish meal. The meal and solvent are heated by means of heating mantles the temperature of which is controlled by continuously adjustable transformers. The flasks, provided with standard taper glass joints, are connected to water-cooled condensers.

Although the AOAC acetone determination yields far more accurate results than does the one-step ethyl-ether extraction, the AOAC method does tend to give values that are a little low if the samples of fish meal have been in storage for more than a year.

The objective of the present project is to develop a modified procedure for oil in fish meal which will be simpler and less time-consuming than the AOAC method. Efforts are also being directed towards improving the accuracy of results when the procedures are used on meals which have been in storage for protracted periods of time.

In the AOAC method, two extractions are required because the first extraction of the meal with acetone removes only that portion of the oil which is loosely bound to the meal. A second portion of the oil is held to the meal in such a way that it can be solvent-extracted only after some sort of hydrolysis has taken place, such as is brought about by the refluxing of the meal with acid. This portion of "bound" oil in the meal is greater, the longer the meal has been held in storage. The amount of bound oil ranges from practically nothing in meals just as they emerge from the dryer of the reduction plant to 75 percent or more of the oil in certain meals which have been stored for periods of a year or more.

The increase in the proportion of bound oil with storage time takes place at a greater rate, the more unsaturated is the oil in the meal. Thus, in the case of meals made from cod or flounders (the oil of which species is, for fish oil, relatively low in unsaturation), the increase in binding of the oil to the meal occurs at a very slow rate. With herring meal, the increase in the rate of binding is considerable, and with pilchard meal (which contains oil that is highly unsaturated), the rate is very high.

In previous experiments (Stansby 1953), it was shown that when pilchard meal is refluxed (rather than extracted) with acetone containing a little (8 percent) water, the solvent not only extracts the loosely-held oil but also hydrolyzes that which is bound to the meal, releasing the oil and at the same time dissolving it. In such a procedure, it is possible to extract all of the oil in a single step. There is no certainty, however, whether this procedure might not also extract considerable quantities of substances other than oils. The experiments at present under way are set up to determine whether the refluxing of fish meals with 92-percent acetone plus 8 percent water extracts any significant amount of material which is of a non-lipid nature.

In previous work on this problem, the solubility of the extractive in ethyl ether has been taken as a criterion of whether the extractive was oil. Recent experiments have shown that, for freshly-prepared meals, all of the material extracted by acetone is completely soluble in ethyl ether. This is true, however, only if the meals have really been freshly prepared. Meals which have aged for as short a time as 12 hours may have undergone some change in the oil such that the extractives removed by acetone are no longer completely soluble in ethyl ether.

EXPERIMENTAL PROCEDURE

Regular commercial meal is quite coarse and contains large particles of bone. Consequently it is so nonhomogeneous as to make a very unsatisfactory sample for analysis. Accordingly, the samples taken at the fish-meal plants were immediately ground in a laboratory mill[1/] adjusted to grind the meal to a fineness such that at least 99 percent of the meal passed through a 30-mesh screen and at least 85 percent through a 35-mesh screen.

Wherever possible the meal was obtained immediately as it emerged from the dryer of the fish-meal plant. The ground (but not sieved) meal was mixed thoroughly. Immediately, thereafter, samples of about 3 to 5 grams were accurately weighed into glass-stoppered refluxing flasks, and other samples of about 4 to 5 grams were accurately weighed into thimbles held in small bottles fitted with leak-proof covers. The samples in the refluxing flasks were then covered with 92-percent acetone, and the samples in the thimbles were covered with 100-percent acetone. Coverage of the samples with solvent prevented any oxidative changes from taking place while the samples were being conveyed to the laboratory for analysis.

The samples in the flasks were refluxed (see equipment in fig. 1) for 18 hours with a total of 80 ml. of the 92-percent acetone. The mixture of the meal and solvent was then filtered through a sintered-glass funnel; the meal residue was washed several times with the solvent; and the total filtrate was evaporated just to dryness and then placed for one hour in a vacuum oven at 80° C. and 24-25 inches of vacuum. After being cooled for 45 minutes in a desiccator, the samples of oil were finally weighed.

In the analysis of the thimble samples, which had been transported to the laboratory in small bottles with leak-proof covers, the thimbles were transferred from the bottles to Sohxlet-extraction apparatus, and the samples were extracted for 16 hours with 100-percent acetone. The acetone used to protect the sample was a part of that used for the extraction. The residue was then hydrolyzed with acid and again given a 100-percent acetone extraction. A complete description of this procedure can be found in the methods of the AOAC (1950).

1/ Laboratory Construction Co.

Excess samples of the meals were stored in glass-stoppered bottles. At intervals of time these samples are being analyzed by the two procedures described above.

Experiments have been started in this way on two types of meals. One is a meal prepared from cod-fillet waste by commercial dry rendering; the other is a meal prepared from whole herring by commercial wet rendering (Butler 1947) and obtained from two different plants, which in this report are called plant 1 and plant 2.

EXPERIMENTAL RESULTS

The values in table 1 under the column headed "AOAC procedure (initial value)" represents the "true" oil content of the meals. In previous work it has been shown that all extractives obtained from strictly fresh meal by the AOAC procedure are soluble in ethyl ether. In order for the reflux procedure, using 92 percent acetone plus 8 percent water as solvent, to be practical, values obtained by it should not be greater (or much greater) than those obtained by the AOAC method. As can be seen

Table 1 - Comparison of Analytical Data Obtained on the Oil Content of Various Fish Meals by the AOAC Procedure and by the Reflux Procedure

Species of Fish from Which Meal Was Made	Plant Producing Meal	Oil Content of Meal			
		AOAC Procedure (Initial Value)	Reflux Procedure Using 92-Percent Acetone		
			Initial Value	Value after Meal Was Stored ½ Month	Value after Meal Was Stored 1 Month
	Number	Percent	Percent	Percent	Percent
Cod	1	8.5	8.3	-	-
Herring	1	13.1	13.3	13.0	13.3
Herring	2	12.1	13.5	12.8	12.1

from table 1, both procedures gave similar values with the cod meal and with the herring meal from plant 1. Herring meal from plant 2 had a somewhat higher oil content as determined by the reflux procedure (13.5 percent) than by the AOAC procedure (12.1 percent). The value by the reflux method for this meal, however, declined quite rapidly with storage so that after the meal had been stored for one month no difference existed between the value obtained by the AOAC procedure on the fresh meal and by the reflux procedure on the one-month-old meal. Values on oil content of herring meal from plant 1 as determined by the reflux procedure showed no change with storage over the one-month period.

The two plants from which the herring meal was obtained were quite similar, both being of the wet-rendering type. Furthermore, the samples were obtained on the same day, and the herring used as the source of the meals were presumably identical, since all were taken from the same fishing ground. Hence, no explanation can be given for the difference in analytical results obtained with the two herring meals.

Had all the results turned out like that for the cod and for the herring meal from plant 1, there would be good reason to believe that the reflux method employing 92-percent acetone extracted only oil and that it could therefore be used in place of the AOAC method. Because of the rather peculiar results obtained on herring meal from plant 2, however, it will be necessary to repeat the analyses with other herring meals. If most of the analyses by the reflux method deviate but little from those of the AOAC method and if, in the analyses that do deviate, the deviation is not greater than occurred with the herring meal from plant 2, it may still be possible to use the reflux method with fish meals. Because of the small discrepancy occurring with herring meal from plant 2, however, it will be necessary to obtain considerable data on other meal samples before this procedure can be adopted.

It is now planned to make comparative analyses on pilchard meal, which contains oil of a considerably greater degree of unsaturation than does herring oil. In addition to such new experiments, work will, of course, continue in following the change, with storage time, in the extractives obtained on the three meals reported here.

SUMMARY

A rapid simple extraction method for the determination of oil in fish meal is being tried on different types of meals. This procedure involves a single extraction in which the meal is refluxed with a solvent composed of 92-percent acetone and 8-percent water. Experiments are under way to determine whether this procedure can replace the more complicated two-stage extraction method of the Association of Official Agricultural Chemists (1950). Preliminary experiments give considerable promise that this simple reflux method may be developed into a practical procedure, but additional work remains to be done before it can be recommended.

LITERATURE CITED

Association of Official Agricultural Chemists
1950. Official and Tentative Methods of Analysis. Seventh Edition, Association of Official Agricultural Chemists, P. O. Box 540, Benjamin Franklin Station, Washington 4, D. C., p. 346.

Bulter, C.
1947. Fish Reduction Processes. U. S. Fish and Wildlife Service, Fishery Leaflet 126 (May 1947).

Stansby, M. E.
1953. Report on Fat in Fish Meal, Journal of the Association of Official Agricultural Chemists 36, #2, pp. 202-8 (May 1953).

--M. E. Stansby, Chief,
Pacific Coast and Alaska Technological Research,

and Wm. Clegg, Chemist,
Fishery Technological Laboratory,
Branch of Commercial Fisheries,
U. S. Fish and Wildlife Service, Seattle, Wash.

FRENCH-FRIED SHRIMP INCREASING IN POPULARITY

With the advent of the electric deep-fat fryer, many hostesses can now fry shrimp right in the dining room, terrace, patio, or wherever they are entertaining. The shrimp may be crumb-coated or batter-coated; the coating may be lightly seasoned or highly seasoned. But, regardless of coating or seasoning, fish cookery experts say that French-Fried Shrimp are here to stay. Like the popcorn habit, you can't stop eating them as long as the supply holds out.

The home economists of the Fish and Wildlife Service offer the following recipes as popular ways of preparing this appetizing, nutritious, and plentiful shellfish.

FRENCH-FRIED SHRIMP

1½ POUNDS SHRIMP, FRESH OR FROZEN	½ CUP FLOUR
2 EGGS, BEATEN	½ CUP DRY BREAD CRUMBS
1 TEASPOON SALT	

Peel shrimp, leaving the last section of the shell on if desired. Cut almost through lengthwise and remove sand veins. Wash. Combine egg and salt. Dip each shrimp in egg, and roll in flour-and-crumb mixture. Fry in a basket in deep fat, 350° F., for two to three minutes or until golden brown. Drain on absorbent paper. Serve plain or with a sauce. Serves 6.