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FIELD METHOD FOR DETERMINATION OF THE OIL CONTENT OF FISH LIVERS

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The lack of sufficiently rapid and simple field methods for the determination of the oil and vitamin A contents of fish livers has prevented the universal adoption of the preferred practice of selling livers on the basis of actual analysis. A complete determination of the vitamin value of a shipment of livers involves three separate operations; efficient procedures are already available for two of these operations. The procurement of a homogeneous, representative sample is adequately handled by use of the electrically-operated sampler developed by the Seattle Technological Laboratory;1/ and rapid field methods for the determination of vitamin A in an oil sample have been devised;2/ but no practical field procedure for extracting and estimating the quantity of oil in the livers has been available. A procedure which has been developed in the Seattle Technological Laboratory, and is described briefly here, may fill this gap between liver sampling and oil analysis.

The method is based upon extraction of the oil by means of methylene chloride, the use of which has three advantages:

(1) It is non-inflammable (therefore it can be safely evaporated over a camp stove or the like);

(2) As shown by Tompkins and Bolomey 3/ it does not interfere with the Carr-Price reaction for the colorimetric determination of vitamin A; and

(3) It is easily volatile, and therefore requires less time for evaporation than would a higher boiling point liquid (such as chloroform).

1/ Charles F. Shockey and F. Bruce Sanford, "Preliminary Report on Drill Sampling Device for Fish Livers," Fishery Market News, May 1944, pp. 9-10.

2/ At least one scientific equipment manufacturer has available a field kit for determining colorimetrically the vitamin A content of fish liver oil.

3/ Paul C. Tompkins and Rene A. Bolomey, "Methylene Chloride," Industrial and Engineering Chemistry, Anal. Ed., 15, 437 (1943).

Note: This leaflet supersedes Sep. 98, a reprint from Fishery Market News, April 1945, pages 2-3.

Equipment and supplies required for running simultaneous duplicate samples are:

1. One hand-operated grinder. This should be of the attrition-type having two grooved plates between which the material is macerated.
2. Suitable containers and paddles for mixing ground liver material.
3. Four 250 ml. beakers.
4. One triple-beam, laboratory balance of 500-gram capacity, graduated to 0.1 g.
5. Four 100 ml. graduated cylinders.
6. Two 4 oz. funnels.
7. One teaspoon.
8. Two glass stirring rods with flattened ends.
9. Suitable heat source.
10. Sodium sulfate (anhydrous).
11. Methylene chloride (technical grade).
12. Cotton.

The equipment listed here is fairly rugged and if properly packed will withstand considerable abuse.

Procedure--The sample of livers is first reduced to a pulp by means of the grinder and then stirred vigorously to homogeneity by the use of any suitable available containers and paddles. Since the analysis can be no more reliable than the sample, considerable care is advisable to assure the procurement of a homogeneous mixture. A 20-gram portion of the homogenized sample is transferred into a 250 ml. beaker and weighed accurately; a rounded teaspoon of anhydrous sodium sulfate is added and thoroughly stirred into the liver material with a stirring rod. With a graduated cylinder approximately 30 ml. of methylene chloride are then added. After thorough agitation, the solids are allowed to settle and the solution is decanted--through a funnel having a cotton plug--into a graduated cylinder. (If the cotton plug is made too tight, filtration will be slow.) A second portion of 15 ml. of the solvent is then added to the liver material, the mixture stirred and the liquid decanted into the graduate as before. The process is repeated with additional 10 ml. portions of the solvent until the volume in the graduated cylinder is made up to approximately 100 ml.

If it is desired only to determine the oil content, the solution is then poured into a second 250 ml. beaker which previously has been weighed (tared), washing out the graduated cylinder with two 5 ml. portions of the solvent; the solution is placed over almost any suitable source of heat and the solvent evaporated until only oil remains. (Care must be taken to prevent spattering. A hot plate, if available, is most convenient, but any portable device such as an alcohol lamp or camp stove may be used.) CAUTION: Although methylene

chloride fumes are not inflammable, they are poisonous. As soon as the solvent has been removed, the beaker is weighed, reheated (being careful not to heat the oil so strongly as to cause it to decompose) and reweighed until two successive weighings give the same result.

$$\% \text{ Oil} = \frac{(\text{weight of oil})(100)}{(\text{weight of sample})}$$

If a portion of the oil solution is to be taken for vitamin A analysis, the graduate is filled to exactly the 100 ml. mark. A completely uniform solution is made by placing the palm of the hand over the end of the graduate and carefully inverting it two or three times. A small amount of solution will be lost in this process, but the error will be negligible. After the portion needed for the vitamin A estimation has been taken, the volume of the solution remaining is read, and the solution is poured into a 250 ml. beaker. The graduate is washed out once or twice with solvent and the washings added to the beaker; the solvent is evaporated and the weight of oil determined as before.

$$\% \text{ Oil} = \frac{(\text{weight of oil})(10{,}000)}{(\text{weight of sample})(\text{number of ml. of solution evaporated})}$$

Accuracy--With livers of high oil content, the method is found to be quite accurate; it regularly gives results only one to three percent (relative) lower than the results which are obtainable by the careful laboratory method described by Stansby and Lemon.4/ On low oil content livers, the results are likely to be somewhat less satisfactory. Following are examples of results obtained by this method.

Sample No.	Species	OIL CONTENT — Method		Relative Error
		Accurate Laboratory	Rapid Field	
		Percent	Percent	Percent
KY-11		70.9	69.0	2.7
KY-12	Grayfish.....	69.3	68.3	1.4
KY-13		74.6	73.4	1.6
KY-9		20.2	19.2	5.0
KY-10	Lingcod.....	19.4	18.3	5.7

For most purposes, errors of these magnitudes are not critical, and will probably be within the error of the method used to estimate vitamin A.

4/ M. E. Stansby and J. M. Lemon, "Quantitative Determination of Oil in Fish Flesh," Industrial and Engineering Chemistry, Anal. Ed., 9, 341 (1937).

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