IAFMM FISH OIL BULLETIN
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RECOMMENDED METHOD OF ANALYSIS FOR DETERMINATION OF MOISTURE IN FISH OIL

1. Principle

This method determines the moisture in fish oil by distillation with an immiscible solvent.

2. Reagents

All reagents shall be of analytical reagent grade.
Toluene, A.C.S. grade or equivalent.

3. Apparatus

Glass distillation apparatus with ground glass connections, constructed and assembled in accordance with the specifications shown in the illustration and described below:

a. Flask, short neck, round-bottom type, 500 to 1000 ml.

b. Water-cooled condenser, the lower tip of which should be about 7 mm above the surface of the liquid in the trap after distillation conditions have been established.

c. Trap, constructed of well-annealed glass and graduated to contain 5 ml at 20°C. It shall be subdivided into 0.1 ml divisions with each ml line numbered and the 5 ml line at the top. The error at any indicated capacity shall not exceed 0.05 ml.

d. Distillation may be better controlled by wrapping the tube which leads from the flask to the receiver with a thermal insulator. It is advantageous to wrap the flask also, especially if there are draughts in the vicinity of the distillation apparatus.

e. Calibrate the receiver by the following procedure: Add an accurately measured portion, 1 ml $\pm$ 0.01 ml, of distilled water to 100 ml of toluene in the distillation flask. Conduct the distillation as described in the Procedure section and calculate as directed. Cool the apparatus, add another ml of water and repeat the distillation. Continue in this manner up to the capacity of the receiving tube.

Pipette capacity 1.0 ml, graduated in 0.01 ml.

Heat source, either an oil bath, or an electric heater provided with a suitable means of control.

Copper or nichrome wire, long enough to extend through the condenser with one end twisted into a spiral. The diameter of the spiral is such that it will fit snugly within the receiver and yet can be moved up and down.

4. Method

Since water tends to settle in samples which are liquid, care must be taken to mix samples thoroughly so as to distribute the water uniformly.

The entire apparatus should be cleaned with potassium dichromate sulphuric acid cleaning solution to minimize the adherence of water droplets to the sides of the condenser and receiver. Rinse thoroughly and dry completely before using.

Take about 200g of the analytical sample and weigh accurately to nearest 1 mg into a distillation flask, add at least an equal volume of solvent (never less than 100 ml) and swirl to mix. Assemble the apparatus and fill the receiver with solvent by pouring through the condenser until the solvent begins to overflow into the distillation flask. Insert a loose cotton plug in the top of the condenser to prevent condensation of atmospheric moisture within the tube.

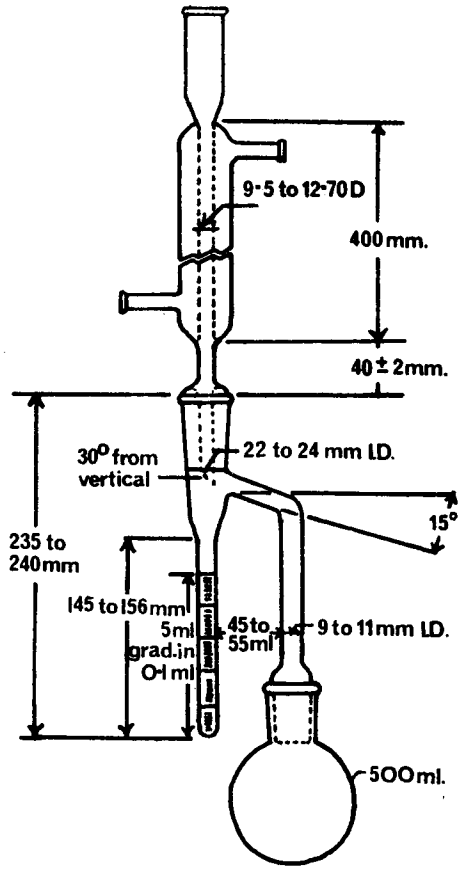
Heat the flask so that the distillation rate will be about 100 drops per minute. When the greater part of the water has distilled increase the distillation rate to about 200 drops per minute and continue until the water level in the receiver remains unchanged for 30 minutes.

Discontinue heating, dislodge any drops of water which may be adhering to the inside of the condenser tube or receiver with a wire spiral and wash down with about 5 ml of toluene.

Immerse the receiver in water at 25°C for at least 15 minutes or until the toluene layer is clear and then read the volume of water.

5. Calculation

$$\text{Moisture \%} = \frac{\text{Volume of water X 99.7}}{\text{Weight of sample}}$$



Moisture Distillation Apparatus

6. Repeatability

The difference between the results of two parallel determinations carried out simultaneously or in rapid succession should not exceed 0.2% moisture.