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# A Volumetric and a Weighing Method for Measuring Semi-Micro Oil Samples*

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DURING several years of spectrophotometric vitamin A assay and plant testing of the liver reduction process two methods for measuring semi-micro oil samples have been developed which have served almost all laboratory and plant requirements. The volumetric procedure described below enables the analyst rapidly to test vitamin A bearing oils for routine control work. The volumetric method also has been of value in conducting vitamin A stability tests on oils during storage. The weighing procedure described has been used as an accurate and rapid method in the quantitative estimation of vitamin A in fish liver oils and concentrates.

*Volumetric Method:* This procedure is suitable for routine use in vitamin A liver processing plants. Blood pipettes commonly in use for red and white cell counts are employed. The red cell type holds approximately 9 mg. of oil and the white cell type approximately 26 mg. of oil. The pipettes are standardized by weighing them filled to the mark below the bulb with the oil commonly being analyzed. In the assaying procedure the pipette is filled to the mark with the oil, and the tip is wiped carefully with a cleansing tissue or towel. The filled pipette is then attached to a siphon containing the desired solvent, and the sample is washed thoroughly into a volumetric flask. The pipette may then be cleaned with petroleum ether and dried with suction. Acetone should not be used since it may dissolve the mixing bead.

*Weighing Method:* In vitamin A assay, a simple method of weighing small oil samples is that of using a micro cover glass. The square cover slip, which has been cut into halves or thirds by using the edge of a carborundum pencil or stone, is suitable as long as it will easily slip through the neck of the volumetric flask. The cut cover slip will weigh approximately 100 mg. With the aid of a small glass rod, samples of oil from 10 to 30 mgs. may be transferred to the slip and weighed. With forceps the slip with oil is dropped into a volumetric flask, which contains a small amount of suitable solvent. The flask is swirled for a few seconds until all visible oil is dissolved then made up to volume and mixed. This technique eliminates the washing error that may be inherent in some oil weighing methods since fish liver oils frequently vary as regards their solubility, e.g. in isopropanol. The error involved in using the cover slip procedure is roughly plus 0.04% in 100 ml. and is of little concern in the present vitamin A assay.

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THE foregoing note by Sinnhuber and Ruggles was shown before publication to the members of the Vitamin Committee of the American Oil Chemists' Society, and two of them made further suggestions.

For field use, when samples of 100 to 500 mg. are needed, we have satisfactorily employed a calibrated 0.5 cc. tuberculin syringe. The conventional weighing of a sample of a vitamin A oil in a volumetric flask can be somewhat simplified by adjusting the length of brass jack chain (used to secure the stopper to the volumetric flask used for the primary dilution) so that the tare is always within + 100 mg. of an even gram. This permits the tare to be obtained on a chainomatic balance without the use of the weight on the notched beam and simplifies somewhat the manipulation and arithmetic involved. A small card in the balance case giving flask numbers and tare weights also facilitates the weighing of the sample.

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A useful and accurate method for weighing semi-micro samples is the following, which was suggested by Dr. K. C. D. Hickman in 1935, and has since been used for rapid tests as well as exact control work:

The carrier for the oil is a light gauge nichrome wire about three inches long bearing a loop on one end and on the other a helix consisting of five or six turns $\frac{1}{8}$" in diameter and spaced about $\frac{1}{64}$" apart. The coil is dipped into the oil sample to pick up a 20 to 40 mg. drop. To be weighed, the carrier is then hung by the loop on a balance. The drop of oil is rinsed off into the volumetric flask, or the whole carrier may be dropped in before the flask is filled with solvent. Very rapid weighings can be made with the use of a Roller-Smith 125 mg. torsion balance with counterweight.

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