

Director's Digest



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Polyethylene Analysis Method Accepted by American Oil Chemists Society

A procedure for the analysis of polyethylene in fats and oils has been published upon recommendation of the Polyethylene in Fats Subcommittee of the American Oil Chemists' Society as an Information Method. The analytical method, which was developed largely by Mr. Gene Rosendahl of National By-Products, Inc., appeared in the Journal of the American Oil Chemists' Society 52, 522A (September, 1975).

Based upon a simple gravimetric procedure in which polyethylene in fat is separated from other insolubles, extracted with hot tetrachlorethylene and subsequently reprecipitated with methanol, the analysis can be carried out in less than one hour. Accuracy is said to be in the order of 20 ppm in samples of fats containing from 50-500 ppm polyethylene.

Additional collaborative tests among participating analytical laboratories are now being conducted in order to develop statistical data for eventual inclusion in "Official and Tentative Methods", published by the American Oil Chemists' Society.

Polyethylene in Fats and Oils

Laboratory General Information Method Revision 4, Proposed Procedure

Definition: This method determines polyethylene present in Fats and Oils by a gravimetric procedure. A fat-chloroform solution is filtered to remove polyethylene and other chloroform insoluble matter. Polyethylene then is dissolved in hot tetrachloroethylene, filtered, and precipitated by addition of cold methanol. The resulting precipitate is isolated and weighed as polyethylene.

Scope: Applicable to fats and oils.

A. Apparatus:

- Two (2) beakers; 600 ml and 400 ml.
- One (1) California Modified Buchner funnel, size 7.0 cm internal diameter (ID) (Labconco, 8811 Prospect Road, Kansas City, MO); 1 porcelain Buchner funnel, size #1, 56 mm ID.
- Two (2) vacuum filtering flasks, 1 liter and 500 ml.
- Metal retaining rings: Size #1—7.0 cm outer diameter (OD) x 5 cm length x 3.0 mm wall thickness; and Size #2—5.4 cm OD x 4.0 cm length x 2.0 mm wall thickness (G & G Mfg. Co., 7006 SW 14th Ave., Des Moines, IA 50315).
- Filter paper: Glass Fibre, 7.0 cm and 5.5 cm; Reeve Angel #934-AH or Whatman Type GFA, predried at 150 C, Store Indessic; Whatman #4—5.5 cm or equivalent.
- Three (3) wash bottles, Guth Universal, 500 ml, hand operated with Kool-Grip neck; or equivalent.
- Filter aid, official diatomaceous earth of AOCS.
- Hot plate
- Drying oven, 105 C.
- Cold water bath, 10 C maximum.

B. Reagents:

Chloroform, reagent grade
Tetrachloroethylene, reagent grade
Methanol, reagent grade

C. Procedure:

- Warm, or cool sample to 40 C \pm 2 C and agitate thoroughly until a uniform homogeneous mixture is obtained. This should be followed for each individual sampling.
- Immediately weigh 100 \pm 0.5 gm of sample to be analyzed into a 600 ml beaker. (See note #1 for weighing instruction.)
- Add 300 ml chloroform. Stir until fat is completely dissolved. Add 2.0 \pm 0.1 gm filter aid; mix until completely dispersed. DO NOT HEAT.
- Attach the California Modified Buchner funnel to a 1 liter filtering flask. Place 2 pieces of 7.0 cm fiber glass paper and size 1 retainer ring in the funnel. Pre-wet filter paper with chloroform prior to filtration. Apply gentle suction and pour the mixture rapidly into the funnel, taking care to keep it from going dry.
- Wash beaker, retainer ring, and paper thoroughly with ca. 50 ml chloroform from the wash bottle. Pull air through filter for 2 min (minimum) to dry. Discard filtrate.
- Bring 75 ml tetrachloroethylene to a brisk boil on a hot plate in a 400 ml beaker. Using gloves or tongs, remove from hot plate and transfer filter paper from step #4 to beaker; stir for 2 min with a glass rod.
- Attach porcelain Buchner funnel, to which a 2 in. length of rubber tubing has been attached to the stem to prevent filtrate from being drawn into the side delivery tube on a clean 500 ml filtering flask (see note #2). Place a disk on 5.5 cm Whatman #4 filter paper, 2 discs of 5.5 cm glass fibre paper, and size #2 retainer ring in the funnel, in that order.
- Replace the 400 ml beaker from Step #5 onto hot plate and bring to a gentle boil.

9. Pre-wet with Tetrachloroethylene filter paper prior to filtration and apply gentle suction to the flask; then rapidly pour the hot tetrachloroethylene solution thru the filter. Using a wash bottle, rinse beaker, retainer ring, and paper 4 times with 10-15 ml portions of near boiling tetrachloroethylene (see note #3). Disconnect funnel from the filtering flask.

10. To contents of the filtering flask add 175 ml pre-cooled methanol (maximum 10 C). Swirl to disperse thoroughly; let stand for 10 min in cold water bath to completely precipitate the polyethylene.

11. Weigh accurately 2 pieces of 7.0 cm moisture free fiber glass paper; and place them, along with the size #1 retainer ring, in the California Modified Buchner funnel. Place funnel on a 1 liter filtering flask, and with gentle suction, filter the solution from Step #9. Using a wash bottle, wash flask, retainer ring, and paper 4 times with 20 ml portions of methanol.

12. Pull air through the fiber glass paper for 2 min. Carefully remove paper from the funnel; dry in an oven at 150 C to constant wt. Ten (10) min drying time should be sufficient. Cool in a dessicator; re-weigh paper and contents.

D. Calculation:

Calculate the ppm polyethylene in the sample as follows:

PPM Polyethylene analyzed =

$$1. \frac{\text{Wt of precipitate in gm} \times 10^6}{\text{Wt of sample in gm}}$$

or

$$2. \text{Wt of precipitate in gm} \times 10,000 \times \text{dilution factor.}$$

E. Accuracy:

Probable accuracy is \pm 20 ppm from 50-500 ppm (see note 7).

F. Notes:

- When samples high in polyethylene (PE) are anticipated or found experimentally by prior analysis, the sample should be diluted with a polyethylene free vegetable salad oil according to the following schedule:

PPM PE expected	Sample wt/gm		Dilution factor
	Fat	Salad oil	
0-750	100	0	1.0
751-1500	50	50	2.0
>1501	25	75	4.0

- The filtering flask (500 ml) used in steps 7 thru 10 should be cleaned thoroughly of any residual polyethylene film after each completed analysis. A strong, hot, caustic solution is adequate for this purpose.
- Step 9 requires complete and thorough washing. Tetrachloroethylene must be kept near the boiling point at all times.
- Methanol used in steps 10 and 11 should be pre-cooled below 10 C by refrigeration or cold water bath to insure complete and rapid precipitation.
- Fiber glass paper is very rapid and extremely retentive, but it is also very delicate and must be handled with great care at all times.
- Adequate ventilation should be used at all times to avoid exposure to solvent vapors.
- The probable accuracy is thought to be due to the inherently non-uniform nature of materials samples. It represents approximate agreement that is attainable. This method is not intended to replace "house" methods now in use; it is presented for general information only. It is recommended that the analyst familiarize self with procedure on trial samples to be able to recognize or anticipate difficulties. ■