

16.4.04

AOAC Official Method 968.35 Filth and Extraneous Material in Peanut Butter

Sedimentation/Flotation Methods

First Action 1968

Final Action 1988

A. Preparation of Sample

Examine individually at least 3, and preferably 6, jars. If jars contain <1 lb each, make 3–6 composites of 2 jars each so that composite sample will be ca 1 lb. Remove contents of each jar and mix thoroughly, preferably in evaporating dishes of convenient size, using heavy table fork or spatula. Peanut butter after warming may also be mixed in the jar by means of mixer, **945.75B(e)** (see 16.1.01), equipped with stiff paddles. If large number of jars is to be examined, make composite samples by thoroughly mixing contents of 3–6 jars of equal size.

B. Water-Insoluble Inorganic Residue (“WIIR”) and Excreta

(Caution: See Appendix B, Distillation, Flammable Solvents, Toxic Solvents, Chloroform, Petroleum Ether.)

Weigh 100 g sample into 250 mL beaker (hooked-lip type), add ca 10 mL petroleum ether, and mix thoroughly. Continue to add petroleum ether, mixing thoroughly until ca 150 mL has been added. Cover, let settle 25 min, and decant 100 mL petroleum ether layer and floating light tissue without losing any coarse peanut tissue. Add ca 125 mL petroleum ether to residue and mix, let settle 15 min, and decant 100 mL as before. Repeat with third ca 125 mL addition petroleum ether, stir, wash down sides of beaker with stream of petroleum ether, let settle 10 min, and decant 100 mL. Discard all decanted portions of petroleum ether.

Evaporate remainder of petroleum ether from residue in beaker; gentle heat may be used. Add 150 mL CHCl_3 to residue and mix thoroughly; cover beaker and let settle 20 min. Stir top layer several times during this period. Carefully decant CHCl_3 and floating peanut tissue onto 15 cm paper in Büchner without disturbing heavy residue in bottom of beaker.

Repeat extraction with small amounts of CHCl_3 , rinsing all particles from sides of beaker. At this point, watch for fragments of rodent excreta pellets on top of NaCl in bottom of beaker; do not decant them. (If sample contains considerable peanut skin, it may be necessary to use mixture of CHCl_3 and just enough CCl_4 to float skin particles away from heavy residue of NaCl, sand, etc.) Dry residue in air.

Add 50 mL HCl (1 + 35) to residue in beaker; then add 90 mL boiling water and let stand 30 min with occasional stirring to dissolve any phosphate, carbonate, or anhydrite (CaSO_4) included with the NaCl. Decant liquid through ashless filter in 60 glass funnel and finally transfer residue with hot water. Test filtrate for sulfate by adding 5 mL saturated BaCl_2 solution. Wash residue on filter several times with hot water. If test for sulfate in filtrate was positive, test residue on filter by placing clean beaker or test tube under funnel and treating residue with 25 mL HCl (1 + 35), adding little at time. Test filtrate with 20 drops saturated BaCl_2 solution (fine white ppt of BaSO_4 indicates presence of anhydrite in residue on filter; allow 5 min for ppt to appear). Wash residue on filter with hot water until all HCl is removed.

Examine residue microscopically for fragments of rodent excreta pellets (identified by presence of rodent hair fragments in mass), insect excreta pellets, and other filth. Ignite paper in weighed

crucible over medium Bunsen flame or in furnace at ca 500 C. Cool, and weigh crucible and contents to nearest 0.5 mg. If “WIIR” is excessive and application of above test indicates that all CaSO_4 has not been removed, make quantitative determination of either Ca or sulfate in “WIIR” in crucible, as in **925.55(F)** or **(G)** (see 11.2.01). Calculate this weight to CaSO_4 and correct weight of “WIIR.”

C. Rocks and Decomposed Peanuts

(Caution: See Appendix B, Toxic Solvents, Carbon Tetrachloride.)

Remove entire contents of jar to 1.5 L beaker or other suitable container. Add ca 700 mL CCl_4 and mix thoroughly, using mixer, **945.75B(e)** (see 16.1.01), if convenient. Rinse jar with CCl_4 and add rinsings to beaker. Let mixture stand 15 min with occasional stirring. Decant ca 2/3 of mixture and add ca 200 mL CCl_4 . Let stand 5 min and decant. Wash down sides of beaker with CCl_4 and repeat decantation until residue is free from peanut tissue. Save all decanted material.

Dry residue in beaker and wash out salt with hot water. If large amount of sand is present, wash residue to remove, silt, phosphate, carbonate, and anhydrite as in **B**, 4th paragraph. Transfer residue to ashless filter paper and examine under low-power microscope. Report number and approximate size of rocks and other extraneous material. If much sand is present, ignite filter and weigh residue, including rocks, reporting result in mg/100 g peanut butter.

Pour decanted CCl_4 -peanut mixture through No. 14 sieve and examine residue for gross filth, stems, other extraneous material, and decomposed peanut tissue.

D. Glass (Procedure)

Take precautions to avoid picking up glass particles when removing contents from glass container and during analysis. Use only stainless steel, Al, plastic, or other nonglass laboratory ware. Filter reagents.

Loosen jar tops and warm jars of peanut butter several hours at ca 50 C in oven.

Add 10 g household detergent powder, such as alkyl aryl sulfonate, to 2 L ca 70 C water. Mix thoroughly and filter through folded filter into 3–4 L stainless steel beaker. Place beaker in ca 70 C water bath and set beaker with bath under paddle-type stirrer, **945.75B(e)** (see 16.1.01). While stirring detergent solution, add entire contents of 12 oz jars, or composite contents of two 6-oz jars, or subdivided 12 oz sample portions of larger containers. Rinse jar and top portionwise with ca 100 mL kerosene and finally with detergent water, and washings to mixture. Continue stirring ca 3 min. Stop stirrer, scrape bottom and sides of beaker with metal spatula, and manually mix any residual peanut butter. Replace paddle and stir again ca 3 min until peanut material is completely dispersed. Stop stirrer, rinse paddle blades over beaker with water, and promptly begin decantation procedure.

In decanting, let mixture settle full 15 s each time after beaker is refilled with 55–70 C water, with beaker in upright position, or leaning up to 45° . Pour off *smoothly*, after indicated standing period,

80% of beaker contents. Repeat as necessary until coarser particulate matter in small amount of clear water is left. Carefully pour off as much of this clear water as possible; still leaving particulate matter in beaker. Thoroughly rinse down sides of beaker with alcohol from plastic wash bottle, and continue decantations after standing 15 s as above, finally pouring off all possible alcohol without losing particles. Rinse sides of beaker with ca 250 mL CHCl_3 . If plant particles tend to aggregate, add more alcohol down

sides of beaker until particles are freely mobile. After 20 s standing, decant, agitating plant particles to free any mech. entrapped glass. Be careful that glass from beaker pocket is not picked up by plant material as it sweeps forward from “back” side of beaker during decantation. Remove as much plant material as practical by CHCl_3 decantations, after 20 s standing with preceding precautions. Transfer beaker contents to filter paper (preferably black) on Büchner. Invert beaker over filter and scrupulously rinse sides and bottom of beaker with jet streams of alcohol and warm water alternately from plastic wash bottles.

Examine microscopically at 30X for glass particles, using only water as moistening agent.

Light Filth
First Action 1968

E. Reagent

Detergent solution.—Dissolve separately 20 g USP sodium lauryl sulfate and 10 g technical $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$ in H_2O , combine, and dilute to 1 L.

F. Determination

Weigh 100 g test portion into 1.5 L beaker and heat on steam bath until softened. Add 1 L filtered hot detergent solution, and stir well. Heat 10 min in steam bath. Stir well, decant onto No. 230 sieve, **945.75B(r)** (see 16.1.01), and wash with forcible stream of 55–70 °C tap water, using aerator, **945.75B(a)** (see 16.1.01). When foam is gone, transfer material on sieve to 2 L trap flask, **945.75B(h)(4)** (see 16.1.01), with 55% alcohol (or 40% isopropanol) and bring volume to 1 L. Add 50 mL HCl. Lower magnetic stirring bar into flask on stirring rod stopper. Heat to bp and boil 10 min while slowly stirring on magnetic stirring hot plate, **945.75B(n)** (see 16.1.01).

Transfer flask to unheated stirring unit and immediately add 40 mL mineral oil, **945.75C(p)** (see 16.1.01), by pouring down stirring rod. Stir magnetically 2 min. Fill with deaerated 55% alcohol (or 40% isopropanol) and gently stir 5–10 s with stoppered rod. Let stand 5 min. Trap off. Add 25 mL mineral oil, stir by hand gently 30 s, and let stand 5 min. Repeat trapping. Wash flask neck thoroughly with isopropanol. Filter onto ruled paper and examine microscopically.

Reference: *JAOAC* **51**, 531 (1968)