

16.1.02

AOAC Official Method 970.66 Light and Heavy Filth

General
First Action 1970

A. Definition of Terms

(a) *Filth*.—Any objectionable matter contributed by animal contamination of product such as rodent, insect, or bird matter; or any other objectionable matter contributed by insanitary conditions.

(b) *Heavy filth*.—Filth material separated from product by sedimentation based on different densities of filth, food particles, and immersion liquids such as CHCl_3 , etc. Examples of such filth are insect and rodent excreta pellets and pellet fragments, sand, and soil.

(c) *Light filth*.—Particles that are oleophilic and are separated from product by floating them in an oil-aqueous liquid mixture. Examples are insect fragments, whole insects, rodent hairs, and feather barbules.

(d) *Sieved filth*.—Filth particles of specific size ranges separated quantitatively from product by use of selected sieve mesh sizes.

B. Special Techniques

(a) *Wet sieving technique*.—Use clean sieve of correct diameter (8 in. (20 cm) minimum), mesh type (plain *not* twill weave) and mesh number (100, 140, 230, etc). Hold sieve under aerator, **945.75B(a)** (see 16.1.01), spray of specified temperature water at approximately 30° angle. Use of sieve handle, **945.75B(s)** (see 16.1.01), or similar device helps maintain proper angle of sieve. Pour well-mixed test sample, portionwise (not so much that clogging or excessive foaming results) onto sieve so that moderate pressure spray of water contacts material on sieve. Increase water pressure to achieve maximum spray action on sieve, but not so violent that product froths over lip of sieve. Keep product washed to lower inside edge of sieve (while held at 30° angle) and direct water spray onto product until majority of detergent foaming subsides and through water is essentially clear. Repeat portionwise addition of product and wash container thoroughly on final addition. Continue washing material on sieve until all detergent foaming subsides and through water is clear. Quantitatively transfer sieve retainings as specified in method. Clean sieve inside walls using rubber policeman and direct water spray on screen, held at angle, to collect all product residues at lower edge of sieve. Repeat sidewall and screen washing, as necessary, to ensure quantitative transfer of sieve retainings.

(b) *Operation of Wildman trap flask*.—Unless otherwise directed in specific method, cool mixture in flask to room temperature. Bring volume of liquid to ca 900 mL in 2 L flask and to ca 600 mL in 1 L flask. Add volume of flotation liquid as stated in method by pouring down stirring rod. Stir magnetically, **B(c)**. Add enough liquid to bring flotation liquid well into neck of flask. (*Note:* Deaerate all flotation liquids before use.)

Unless otherwise stated, let mixture stand 30 min, intermittently stirring bottom layer every 3–6 min during first 20 min of standing. Spin stopper (wafer) to remove sediment and trap off by raising stopper (wafer) as far as possible into neck of flask, being sure that oil layer and 1 cm of liquid below interface are above stopper (wafer). Hold stopper (wafer) in place and pour off liquid into beaker. Rinse out material on rod and in neck of flask with liquid extraction medium in which floating was performed and add to beaker.

Do not wash out neck of flask with alcohol or other liquid which may interfere with surface relationships of the 2 phases; this will cause loss in recovery in subsequent trappings.

Filter trapped material and rinsings with suction through rapid paper in Hirsch funnel. Add flotation liquid as specified to trap flask and stir vigorously. Add enough liquid extraction medium to bring flotation liquid into neck of flask. Trap off again, rinse, and filter as above.

(c) *Operation of magnetic stirrer*.—To disperse flotation liquid through product, dilute liquid extraction medium to volume specified in method and bring to proper temperature. Add magnetic stirring bar, **945.75B(n)** (see 16.1.01), and proper volume of flotation liquid. Slowly bring unit to maximum speed that does not produce visible or audible splashing (central portion of stirring bar is usually just visible at bottom of Vortex) and stir for time stated in method. Time stirring interval after achieving proper speed and Vortex.

(d) *Filtration technique*.—(Treatment of trapped-off material.) If material trapped off in beaker contains appreciable starchy debris, add enough HCl to make solution 1 (1 + 99)–2 (1 + 49)%, bring to boil, and filter while hot. If fats or colloidal material retard filtration, hasten by playing stream of hot water over paper during filtration.

(e) *Clearing of plant materials*.—With sedimentation or flotation procedures, some food material may be trapped off with filth particles. By proper clearing, filth may be made to stand out in contrast with white background of filter paper by one of following techniques: (1) For heavy filth, moisten paper with H_2O or 50% alcohol. (This method does not clear material completely, but it leaves rodent pellets and other filth soft and pliable.); (2) For light filth examination, wet paper with glycerol–alcohol (1 + 1) immediately after filtering. Place enough liquid on paper to fill fibers but not enough to cause flowing of extracted materials. This clearing agent does not harden filth material on paper, as do many oils which might be used as clearing agents; (3) Clove oil can be used for clearing plant materials. This oil has high refractive index and clears more completely than does alcohol–glycerol solution.

(f) *Illumination for the widefield stereoscopic microscope*.—By *direct light*.—Focus and adjust light to strike paper from above at ca 70° angle from horizontal. Light may come from right or left.

(g) *Microscopic examination of filter papers*.—Make examination at 30 (unless otherwise specified), using widefield stereoscopic microscope, on properly cleared paper on opaque white background. Continually tease and probe particles while observing through microscope. Turn over all large pieces of material, such as bran, which might obscure filth elements. Examine all doubtful pieces of material at 60–75 . At least twice magnification used in original examination is necessary to show new details not observable at lower power. If doubt still remains, mount piece, clear thoroughly, and examine under compound microscope. Thorough knowledge of appearance of authentic materials is assumed.

(h) *Counting insect and other animal filth*.—*Diagnostic characteristics of insect fragments*.—Count as of insect origin any fragment showing one or more of the following characters: (1) characteristic shape of whole or portion of specific appendage or body part; (2) articulation point (various types of joints); (3) one or more body hairs or setae; (4) one or more setal scars; (5) surface pattern (sculpturing) characteristic of a specific insect; (6) one or more sutures present (various types separating body plates or sclerites). *Diagnostic characteristics of animal hairs*.—See

Vazquez, A.W., Structure and Identification of Common Food-Contaminating Hairs, *JAOAC* **44**, 754(1961), and Vazquez, A.W., "Hairs" in *Principles of Food Analysis for Filth, Decomposition, and Foreign Matter*, AOAC INTERNATIONAL, 481 N. Frederick Ave, Suite 500, Gaithersburg, MD 20877-2417, USA.

(i) *Format for reporting filth.*—*Container.*—Describe size, type, and closure(s) of immediate container and note condition if not intact. *Product.*—Common name, if identity is known, or simple description. *Code(s).*—Manufacturer's or distributor's name and identification marks. *Method(s).*—Cite AOAC paragraph number(s) and note any modifications made. *Amount examined.*—Number subsamples analyzed and amount per subsample. If amount is variable, report for each subsample under Findings. *Findings.*—Report findings on analyst's worksheet by subsample number. Use only categories that apply and report any filth element that is found under no more than one category. Within categories, group filth elements by identity, when known, and then by size or other appropriate descriptive feature. If amount filth present makes exact count impractical, report either approximate or minimal figure rather than term "too numerous to count." Summarize product results by category totals and averages. Note whether or not product was fumigated before shipment or on receipt at laboratory, if there are whole insects, mites, or other arthropods.

(1) Number whole insects or equivalents (i.e., separate heads or body portions with head attached). Distinguish whole insects and equivalents in subtotals. Give identity, stage of life cycle, and size (mm). State whether whole insects are alive or dead.

(2) Number insect cast skins. Give identity (if known), size (mm), and state whether nymphal, larval, or pupal. Distinguish whole cast skins (with head portion) and cast skin fragments.

(3) Number insect eggs. Give identity, if known.

(4) Number insect fragments, other than separate setae. Give identity (if known), dimensions or size range (mm), and name of part. State whether identified fragments are from adult or immature insects.

(5) Number setae (if fly, state).

(6) Insect excreta. Report weight (mg) and/or count of excreta pellets with dimensions or size range (mm). Give identity, if known.

(7) Insect penetration of container. Report number, size (mm), and direction. Note container integrity and completeness of closures and seams.

(8) Number mites. Give identity, if known. State whether alive or dead. Report mite fragments here as subcategory.

(9) Number arthropods other than insects and mites. State whether alive or dead. Give identity (e.g., spiders, pseudoscorpions). Report fragments here as subcategory.

(10) Number rat or mouse fecal pellets (state which or give length and diameter in mm). Give weight (mg) if from condimental seeds, spices, cocoa beans, coffee, or grains.

(11) Number rat or mouse fecal pellet fragments. Give basis for identification. Give dimensions or size range (mm). Give weight (mg) if from condimental seeds, spices, cocoa beans, coffee, or grains.

(12) Other mammalian feces. Report size (mm) and weight (mg). Give identity (e.g., cat, cow), if known, and basis for identification.

(13) Number rat or mouse hairs or hair fragments. Report length (mm) of each hair and hair fragment or, if numerous, separately group intact hairs and hair fragments by the following size categories: (a) <5 mm long, (b) 5–10 mm long, and (c) >10 mm long.

(14) Number other hairs and hair fragments. Report length (mm), grouping as in (13). If unidentified, state whether striated or nonstriated.

(15) Number feathers, feather fragments, and barbules. Give dimensions or size range (mm) of feathers and fragments.

(16) Urine on container or food (state which). Report odor of urine, if detected. Give number and dimensions or size range (in.) of stains and note if penetration is to product. Report component(s) detected by AOAC test.

(17) Bird excreta on container or food beneath (state which). Report amount as weight (mg) or number and dimensions of droppings, as appropriate.

(18) Hard or sharp foreign objects. Give identity and length (mm) for each element. Group findings by the following size categories: (a) <2 mm long, (b) 2–7 mm long, and (c) >7 mm long.

(19) Other extraneous materials (describe and report each type by appropriate quantitative figure).