

16.13.02

**AOAC Official Method 993.28
Light Filth in Bean Paste**

**Flotation Method
First Action 1993
Final Action 1996**

(Applicable to determination of light filth in bean paste.)

See Table 993.28 for results of the interlaboratory study supporting the acceptance of the method.

A. Principle

Bean paste sample is treated with surfactants and is wet-sieved with hot water to remove surfactants and emulsified fats and oils. Residue is defatted with isopropanol, wet-sieved, boiled in 40% isopropanol, cooled, and trapped 2× with flotation liquid in aqueous 40% isopropanol to recover light filth.

Light filth, such as insect fragments and hairs, is unaffected by emulsification and defatting steps. In contrast to bean paste, light filth is attracted to oil phase in trapping procedure. When oil phase is trapped off, light filth is assessed microscopically.

B. Apparatus

- (a) Sieve.—(1) No. 230 plain-weave, **945.75B(r)** (see 16.1.01); and (2) sieve handle, **945.75B(t)** (see 16.1.01).
- (b) Reflux apparatus.—**975.49A(e)** (see 16.14.05).
- (c) Wildman trap flask.—2 L, **945.75B(h)(4)** (see 16.1.01).
- (d) Filter paper.—Ruled, **945.75B(i)** (see 16.1.01).

C. Reagents

- (a) Surfactant mixture.—Igepal CO-730, **945.75C(j)(1)** (see 16.1.01), and Igepal DM-710, **945.75C(j)(2)** (see 16.1.01). Prepare surfactant mixture, DM-710 and CO-730 (1 + 2).
- (b) Isopropanol solutions.—(1) 100%. (2) 40% aqueous solution.
- (c) Flotation liquid.—**945.75C(k)** (see 16.1.01).

D. Isolation

Accurately weigh 100 g bean paste to nearest 0.5 g into 1.5 L beaker. Add ca 400 mL hot tap water and hand-stir 1 min. Bring volume in beaker to 600 mL with hot (55–70°C) tap water. Add 15 mL

surfactant mixture, **C(a)**, cover beaker with watch glass, and bring to boil with magnetic stirring, **945.75B(n)** (see 16.1.01). Remove watchglass and boil solution for 10 min.

Transfer beaker contents portionwise to sieve, **B(a)(1)**. Wet-sieve, **970.66B(a)** (see 16.1.02), between portions. Rinse beaker and transfer washings to sieve. Retain beaker. Wet-sieve, using forceful stream of hot tap water until rinse is clear. Use of sieve handle, **B(a)(2)**, is recommended. (Note: If sieve becomes clogged during sieving, gently tap on side of sieve to drain excess water.) Wet residue on sieve with 100% isopropanol, **C(b)(1)**, to reduce foam.

Quantitatively transfer residue to retained beaker using 100% isopropanol. Wash sides of beaker with 100% isopropanol from wash bottle; and then fill beaker to 400 mL with 100% isopropanol. Defat contents by boiling 5 min, using reflux apparatus, **B(b)**. Quantitatively transfer contents from reflux apparatus to sieve.

Wet-sieve until rinse is clear. Wash residue on sieve with 40% isopropanol, **C(b)(2)**, and quantitatively transfer to trap flask, **B(c)**. Wash sides of flask with 40% isopropanol from wash bottle; then fill to 600 mL with 40% isopropanol. Bring to boil and boil gently 10 min with magnetic stirring. Remove from heat and loosen any adhering materials from sides of flask using stopper (wafer) and by washing sides of flask with 40% isopropanol. Cool flask contents to 28–30°C in water bath.

Remove from water bath and add 60 mL flotation liquid, **C(c)**. Stir magnetically 10 min, **970.66B(c)** (see 16.1.02). Fill flask with 40% isopropanol by pouring slowly down stirring rod. Stir intermittently 10 min and let stand undisturbed 20 min. Gently spin stopper to remove sediment and trap off, **970.66B(b)** (see 16.1.02), into 400–600 mL beaker, using 40% isopropanol. Add 35 mL flotation liquid to flask and hand-stir with gentle up-and-down motion 30 s. Fill flask to top with 40% isopropanol and let stand undisturbed 20 min. Trap off as before, using 100% isopropanol, and filter entire beaker contents through filter, **B(d)**, using 2 filters if necessary.

Examine microscopically, **945.75B(o)(2)** (see 16.1.01), at ca 30×

Reference: *J. AOAC Int.* **77**, 1143(1994).

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Table 993.28 Interlaboratory study results for determination of light filth in bean paste by flotation method^a

Spike added	\bar{x}	s_r	s_R	RSD _r	RSD _R
Rat hairs					
5	4.55 (4.50)	1.63 (1.00)	1.63 (1.05)	35.8 (22.2)	35.8 (23.4)
10	8.85 (8.65)	2.04 (1.56)	2.09 (1.56)	23.0 (18.1)	23.6 (18.1)
15	12.15 (11.70)	2.20 (1.67)	2.28 (1.85)	18.1 (14.3)	18.8 (15.8)
Insect fragments					
5	4.35 (4.10)	0.87 (1.00)	1.05 (1.02)	19.9 (24.4)	24.1 (24.9)
15	14.20 (14.25)	1.22 (1.32)	1.45 (1.53)	8.6 (9.3)	10.2 (10.7)
30	28.00 (29.11)	1.87 (1.05)	2.81 (1.05)	6.7 (3.6)	10.1 (3.6)

^a Third-party counts in parentheses.