

16.6.02

AOAC Official Method 972.37 Light Filth in Baked Goods with Fruit and Nut Tissues First Action 1972

A. Pancreatin Digestion Method

Post-milling contamination.—Weigh 225 g test portion into 2 L beaker, add enough hot water to soften and saturate material, and proceed as in (a). If lumps persist or if H₂O is not immediately absorbed uniformly through entire mass, proceed as in (b).

(a) Adjust mixture to pH 7–8 with ca 5% Na₃PO₄ solution. Stir and break up material as much as possible. Cool to 40°C and add 100 mL pancreatin solution, **945.75C(q)** (see 16.1.01). Stir thoroughly and readjust to pH 7–8. Let stand 30 min, stir, and readjust pH.

(b) Estimate volume of mixture and add HCl to ca 1 + 49 concentration. Boil until solids become finely divided and so digested that mixture will not froth over when covered during boiling. Neutralize to ca pH 6 with NaOH solution; then add Na₃PO₄ solution to pH 8 and continue as in (a).

For white flour products, add 0.2 mL or 4 drops HCHO and digest overnight. For products made from whole wheat and rye flours and from similar materials of high bran content, digest only 2–3 h.

Pour digested material through 5 or 8 in. No. 140 sieve, **945.75B(r)** (see 16.1.01). While pouring, play forcible stream of hot water from tap on this material. Wash well with large stream of hot water. After complete washing (no starchy material visible unattached to bran), wash twice alternately with alcohol and CHCl₃ in that order, and then rinse thoroughly with alcohol and finally with H₂O.

Transfer material to filter paper if little residue remains or to 1 or 2 L trap flask, **945.75B(h)(4)** (see 16.1.01), if large amount remains. Transfer bulk of material with spoon. Rinse residue from screen with 60% alcohol from wash bottle. Wash screen with forcible stream of hot water, collecting final residue at one edge of screen and transferring to trap flask with stream of 60% alcohol as above. Add 400 or 900 mL 60% alcohol, depending on size of trap flask.

Boil 20 min. Cool to <20°C and add 20 or 40 mL heptane, **945.75C(l)** (see 16.1.01); fill flask with 60% alcohol, and trap off twice. Use care in stirring and adding alcohol to prevent emulsions or inclusion of air. If residue in flask tends to rise, stir material down 2 or 3 times. Filter trapped-off material and examine microscopically.

B. Acid Hydrolysis Method

(Rapid method; also applicable to flours.)

Post-milling contamination.—Add 225 g test portion to 2 L beaker containing ca 1 L H₂O and 30 mL HCl. Wet product completely and, for flour, stir until slurry is practically lump-free. Add antifoam solution, **945.75C(e)** (see 16.1.01), cover with watch glass, and heat 15–20 min in autoclave at 121°C. Let pressure fall to 0 before opening vent valve. Transfer digest in small portions to 5 or 8 in. (12.7 or 20.3 cm) No. 140 sieve, **945.75B(r)** (see 16.1.01), washing thoroughly between additions with needle spray from aerator. After all solids have been transferred, continue washing until there is no further reduction in amount of residue. Proceed as in A, paragraph 5, beginning “After complete washing ”.

Reference: *JAOAC* **45**, 660(1962).