

31.4.02

AOAC Official Method 963.15

Fat in Cacao Products

Soxhlet Extraction Method

First Action 1963

Final Action 1973

Office International du Cacao et du Chocolat—AOAC Method

(Applicable to cacao products with or without milk ingredients or to products prepared by cooking with sugar and H₂O, and drying.)

A. Apparatus and Reagents

(a) *Soxhlet apparatus*.—With standard taper joints, siphon capacity ca 100 mL (33–80 mm thimble), 250 mL Erlenmeyer, and regulated heating mantle.

(b) *Petroleum ether*.—Distilled in glass, bp 30–60°C.

B. Determination

Prepare test sample as in 970.20(b)(1) (see 31.1.01). Accurately weigh 3–4 g chocolate liquor, 4–5 g cocoa, 4–5 g sweet chocolate, or 9–10 g milk chocolate into 300–500 mL beaker. Add slowly, while stirring, 45 mL boiling water to give homogeneous suspension. Add 55 mL ca 8M HCl (2 + 1) and few defatted SiC chips or other antibumping agent, and stir. Cover with watch glass, bring slowly to boil, and boil gently 15 min. Rinse watch glass with 100 mL H₂O. Filter digest through 15 cm S&S 589 medium fluted paper, or

equivalent, rinsing beaker 3 times with H₂O. Continue washing until last portion of filtrate is Cl-free as determined by addition of 0.1M AgNO₃. Transfer wet paper and residue to defatted extraction thimble and dry 6–18 h in small beaker at 100°C. Place glass wool plug over paper.

Add few defatted antibumping chips to 250 mL Erlenmeyer and dry 1 h at 100°C. Cool to room temperature in desiccator and weigh. Place thimble containing dried residue in Soxhlet, supporting it with spiral or glass beads. Rinse digestion beaker, drying beaker, and watch glass with three 50 mL portions petroleum ether, and add washings to thimble. Reflux digested residue 4 h adjusting heat so that extractor siphons 30 times/h or condensation rate of 5–6 drops/s.

Remove flask, and evaporate solvent on steam bath. Dry flask at 100–101°C to constant weight (1.5–2 h). Cool in desiccator to room temperature and weigh. Constant weight is attained when successive 1 h drying periods show additional loss of <0.05% fat. Fat, % = g fat / 100/g test sample. Duplicate determinations should agree within 0.1% fat.

References: *JAOAC* **28**, 482(1945); **33**, 342(1950);

34, 442(1951); **53**, 490(1970).

Analytical Methods of the OICC (1963) Ref. 2,
p. 8a-E.