

16.14.17

AOAC Official Method 985.38
Light Filth in Sage (Rubbed and Ground)

Brine Saturation Method
First Action 1985
Final Action 1996

A. Reagents

(a) *Brine*.—Prepare by dissolving ca 360 g NaCl/L H₂O. Less expensive sources of salt which were found by use and by comparative study to be equivalent to AR grade NaCl were: (1) Morton Rock Salt for making ice cream (Morton Salt Division of Rohm and Haas Co., Chicago, IL 60606, USA; www.mortonsalt.com); or, (2) sterling salt crystals for water softeners (AKZO Nobel Salt, Inc., Abington Executive Park, Clarks Summit, PA 18411, USA; www.aksonobelsalt.com). Filter brine before use.

(b) *Olive oil, NF*.—Less expensive sources of olive oil which are equivalent to NF grade oil are readily available commercially.

B. Extraction

Rubbed and ground sage.—Weigh 10 g test portion into 600 mL beaker. Add magnetic stirring bar, **945.75B(n)** (see 16.1.01), and 300 mL isopropanol. Cover with watch glass and boil ca 3 min with constant stirring on magnetic stirrer-hot plate. Transfer to 230 mesh sieve, **945.75B(r)** (see 16.1.01), with isopropanol, and wash with ca 100 mL isopropanol from wash bottle, then with hot tap water to remove isopropanol, and then with brine to remove tap water. Transfer to original 600 mL beaker and bring volume to ca 350 mL

with brine. Cover and bring to boil with constant stirring. (*Note*: Do not boil over. Product loss will occur from boil-over.)

Transfer beaker to cold water bath (use plastic or glass bowl filled with cold water on magnetic stirrer). Uncover, wash down insides of beaker, and cool to touch with constant stirring. Repeat heating and cooling to complete brine saturation, and then transfer to 2 L trap flask, **945.75B(h)(4)** (see 16.1.01), with ca 100 mL brine, fill flask ca $\frac{3}{4}$ full with 32 °C tap water, and insert trap rod. (*Note*: Temperature variation below that specified could result in loss of oil trapped due to adhesion to sides of flask.) Add 50 mL olive oil, and then fill flask to normal (neck) level with tap water. Place on magnetic stirrer with trap rod to one side of flask bottom to avoid interfering with stirring bar. Stir rapidly for 5 min so that no significant oil layer is visible in neck of flask but not so rapidly that vortex extends more than 1 in. into flask. Remove from stirrer and let oil layer rise. Stir bottom of flask to release any trapped oil; repeat after 5 min. Wait ca 20 min, and then briefly and gently stir entire flask contents, particularly at the neck, to release and sink any product that has accumulated at interface. After 10 min, stir interface only with pivoting rotary motion of trap rod, keeping plunger relatively stationary at bottom of flask. Wait 10 min and repeat previous stirring if product has accumulated at interface. Then wait 10 more min and trap oil layer into 250 mL beaker, rinsing neck of flask and trap thoroughly several times with isopropanol and finally with H₂O. Filter on ruled paper and examine microscopically, **970.66B(g)** (see 16.1.02).

Reference: *JAOAC* **68**, 894(1985).