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**43.1.05 - Spices and Other Condiments**

**AOAC Official Method 941.12**  
**Ash of Spices**

**Gravimetric Methods**  
**First Action 1941**  
**Final Action**

***A. Determination***

(a) *Most spices*.— Weigh ca 2.000 g test portion in flat-bottom dish, preferably Pt. Place dish in entrance of open furnace so that sample fumes off without catching fire. Then ash in furnace 30 min at 550°C, break up ash with several drops H<sub>2</sub>O, evaporate carefully to dryness, and heat in furnace 30 min. If previous wetting showed ash to be C-free, remove dish to desiccator containing fresh, efficient desiccant [H<sub>2</sub>SO<sub>4</sub> or anhydrous Mg(ClO<sub>4</sub>)<sub>2</sub> is satisfactory], let cool to room temperature, and weigh soon. If first wetting showed C, repeat wetting and heating until no specks of C are visible; then heat 30 min after disappearance of C. If C persists, leach ash with hot H<sub>2</sub>O, filter through quantitative paper, wash paper thoroughly, transfer paper and contents to ashing dish, dry, and ignite at 550°C until ash is white. Cool dish, add filtrate, evaporate to dryness on steam bath, and heat in furnace 30 min. Cool, and weigh as previously.

(b) *Nutmeg, mace, ginger, and cloves*.—Proceed as in (a), but heat at 600°C.

(c) *Ground mustard or mustard flour*.—Ignite as in (a) and heat 30 min at 550°C. Leach ash with hot H<sub>2</sub>O, filter, and wash thoroughly. Transfer paper and contents to ashing dish, dry, and heat in furnace 30 min. Remove dish, let cool, add 5-10 drops HNO<sub>3</sub>, evaporate to dryness, and heat in furnace 30 min. Repeat HNO<sub>3</sub> and heating treatment until residue is white. Add filtrate, evaporate to dryness, and heat in furnace 30 min. Cool, and weigh as in (a).

**Reference:**

*JAOAC* **24**, 667(1941).

***B. Ash Insoluble in Acid***

Boil residue from **A** in 25 mL HCl (2 + 5) 5 min, covering dish with watch glass to prevent spattering; collect insoluble matter on Gooch or ashless filter, wash with hot H<sub>2</sub>O until washings are acid-free, ignite until C-free, cool, and weigh.

### ***C. Calcium in Ash***

Ignite 2-4 g test portion as in **A**, digest with hot HCl (2 + 5), evaporate to dryness, moisten dry residue with dilute HCl, and again evaporate to dryness to make SiO<sub>2</sub> insoluble. Treat residue with 5-10 mL HCl, add ca 50 mL H<sub>2</sub>O, let stand on H<sub>2</sub>O bath few minutes, filter, and wash insoluble residue with hot H<sub>2</sub>O. Determine CaO in combined filtrate and washings as in [910.01](#) (see 3.3.02).