

Macroanalytical Procedures Manual (MPM)

MPM: V-8. Spices, Condiments, Flavors, and Crude Drugs

A. General Method for Spices, Herbs, and Botanicals (V-32)

Version 1 - May 1998

Contents

A. General Method for Spices, Herbs, and Botanicals (V-32)	2
(1) Scope	2
(2) Applicable Documents	3
(3) Defects	3
(4) Procedure: Determination of Contamination in Spices, Herbs, and Botanicals Caused by Insects, Animal Excreta, and Extraneous Material	4
(5) Procedure: Determination of Insect-Damaged, Moldy, and Otherwise Reject Product Material in Spices, Herbs, and Botanicals	5
References	7
Revision History	7

A. General Method for Spices, Herbs, and Botanicals (V-32)

(1) Scope

This method covers several procedures applicable to spices, herbs, and botanicals that consist of dried plant parts which have been processed and marketed in various sizes and forms. Included in this group are parts of plants such as dried leaves, seeds, fruits, bark, roots, stems, buds, and flowers. Examples of plant part(s) products include but are not limited to:

- Leaves
 - Sage (*Salvia officinalis* L.)
 - Bay leaves (*Lauris nobilis* L.)
- Seeds
 - Flax (*Linum usitatissimum* L.)
 - Nutmeg (*Myristica fragrans* Houttuyn)
 - Fenugreek (*Trigonella foenum-graecum* L.)
- Fruits
 - Cardamom (*Elettaria cardamomum* (L.) Matonâ)
 - Pepper (*Piper nigrum* L.)
 - Paprika (*Capsicum annuum* L.)
 - Cassia buds (*Cinnamomum* spp.Schaeffer)
- Bark
 - Cinnamon (*Cinnamomum* spp.Schaeffer)
 - Sassafras (*Sassafras albidum* (Nuttall) Ness)
 - Soapbark (*Quillaja saponaria* Molina)
- Roots
 - Licorice (*Glycyrrhiza glabra* L.)
 - Chicory (*Cichorium intybus* L.)
 - Comfrey (*Symphytum officinale* L.)
- Rhizomes
 - Ginger (*Zingiber officinale* Roscoe)
 - Calamus (*Acorus calamus* L.)
 - Turmeric (*Curcuma domestica* Valetton)
- Flower Buds -- Clove (*Syzygium aromaticum* (L.) Merrill & Perry)
- Flower parts -- Saffron (*Crocus sativus* L.).
- Flowers -- German chamomile (*Matricaria chamomilla* L.)

a. General Method -- Procedures applicable to macroscopic examination of most of these products fall into two categories:

(i) A procedure for separating, identifying, and recording amounts of contaminants such as insects, animal excrement, and extraneous matter which have been removed by pick-out or mechanical means from the product material itself.

(ii) A procedure for separating and classifying defective product materials (such as insect-damaged or moldy material); these are recorded as percentages of reject material by weight or count of each type of reject.

b. Specific methods -- Procedures for certain types of products (e.g., black pepper, nutmegs, etc.) are specialized for dealing with specific problems. Special techniques related to these products are covered by supplemental methods described in Sections 8.B and C.

(2) Applicable Documents

- [CPG 7109.17 Defect Action Levels](#)

(3) Defects

Defects in these products may be categorized as due to insect infestation, contamination by animals, mold development, or contamination by extraneous material.

a. Insect Infestation and Damage -- Products may be contaminated and damaged by insects in the growing area during production and harvest. Typical field insect pests include Lepidoptera, Coleoptera, Hymenoptera, Homoptera and Hemiptera. A wide variety of stored-product insects, principally Lepidoptera and Coleoptera, also attack these products. Other arthropod pests, such as mites, ticks, and spiders, may be present. Examples of insect infestation and damage range from excreta, webbing and frass, insect tunnels or evidence of surface feeding, to whole insects in the product. Although numerous insect species are associated with these products, serious damage to particular products is usually due to a few species, and it may be helpful, in some cases, to define such damage as part of a specific method.

b. Animal Contamination -- Contamination of these products by animals usually results from either gnawing or defilement by excreta. Whole rodent pellets, bird droppings, and other pieces of animal dung are typically found.

c. Moldiness and Fungal Deterioration -- Products may be attacked by fungi in the field or while in storage. Field fungi capable of attacking and infecting the growing product may cause varying degrees of decomposition and damage. The damage from invading fungi and molds may be manifested as leaf spot diseases, dry rot, decomposed and discolored tissue of stems and roots, or decay in seeds and fruits. Storage fungi (which can grow under limited moisture conditions) may cause moldiness in some products stored under conditions of temperature and relative humidity favorable to their growth. Pockets of moist product can arise in a dried and otherwise normal product through roof leaks, insect activity, and moisture translocation when temperature gradients develop within the product mass. These pockets can promote the rapid growth of molds in the stored product. Moldiness can range in appearance from

mycelium-matted leafy spices and surface mold on cassia bark, to internal molds in nutmegs and capsicum pods.

d. Contamination by Extraneous Material -- Objectionable matter such as sticks, stones, burlap bagging, or cigarette butts may enter the product at various points during its production, transit, and storage because of improper preparation or handling. Also, valueless parts of the raw plant material and other foreign plant material may contaminate the product and require special attention for removal. This general category is intended to include all other miscellaneous objectionable matter not reported in the other specific categories.

(4) Procedure: Determination of Contamination in Spices, Herbs, and Botanicals Caused by Insects, Animal Excreta, and Extraneous Material

a. Sample Preparation -- Sample a representative or selective number of analytical units of the lot sample, depending on the history of the lot.

b. Visual Examination -- Analyze 300-500g subsamples of high-density products (cassia, ginger, cloves, allspice, turmeric, condimental seeds) and 200-300g subsamples of light, bulky products (capsicum pods, mace, leafy spices). Examine the product in small amounts with good light and against a white or other suitable contrasting background. A moving belt or other mechanical device may be used if all the material can be seen easily. Sifting may facilitate separation and concentration of certain types of objectionable matter. Examine macroscopically for rodent excreta, manure, insects, and insect debris, mold clumps, miscellaneous objectionable matter, and other evidence of contamination. If sifting is performed, size of screens used and method of use should be stated in the report of results.

c. Classification of Contaminants -- Separate contaminants into suitable groupings relative to defect action levels, regulatory guidelines, or other applicable requirements. Add categories to tabulation of results depending on type(s) of contaminants found. Classify as follows:

(i) *Insects* -- Count number of whole insects and equivalent visible to the naked eye (corrected as necessary for abnormal vision) with such magnification as may be necessary. If the magnification exceeds 10X, this should be stated in the report of results. Identify insects, using appropriate insect identification keys. Classify insects as "Field" or "Storage," making a special notation when they are found alive. Note the size of any unidentified insects and larvae found.

(ii) *Rodent (Rat or Mouse) Excreta* -- Rodent excreta pellets are normally black or dark colored, roughly cylindrical, blunt at one end and pointed at the other. They range in length from 1.5 to 15 mm. They usually contain rat or mouse hairs, partially digested plant material, and sometimes insect parts. When wet with water, rodent pellets form a characteristic gray mucous coating. Weigh suspect pellets and report as such *only* if rat or mouse hairs are present. Confirm identification by removing a hair from the pellet and identifying it microscopically. When none are present, proceed with AOAC 44.B08-44.B11, alkaline phosphatase method for mammalian excreta.

(iii) *Animal Dung* -- Animal dung consists of an amorphous, usually dark colored material pressed into a matrix. Incorporated plant material usually consists of ligneous, fibrous material which is either pale-yellow or green. Parts of insects and small amounts of inorganic, earthy material may also be present. Weigh suspect material and report as animal dung or excreta, *only* when matricized plant material predominates. Confirm as excreta, using 44.B08-44.B11, alkaline phosphatase method for mammalian excreta.

(iv) *Bird Excreta* -- Bird excreta will appear as rounded droppings, sometimes coiled with a white residue. Weigh droppings and test a portion of the white, amorphous particles for uric acid by AOAC 44.177 or 44.183 - 44.185.

d. Report -- Tabulate results as follows, adding additional categories as necessary:

	Subsample No.			
	1	2	3	etc.
Weight Examined (g)				
Excreta (mg/lb) ^a				
Whole Insects or Equivalent ^b				
Extraneous Material (% by wt) ^c				
Other ^d				
Remarks:				

Notes:

- a. Excluding insect excreta
- b. Describe ((4)c(i))
- c. Describe ((3)d)
- d. Substitute appropriate heading(s)

(5) Procedure: Determination of Insect-Damaged, Moldy, and Otherwise Reject Product Material in Spices, Herbs, and Botanicals

a. Sample Preparation -- Weigh or count out representative analytical units from sieved material remaining after completing procedure (4). Alternatively, draw analytical unit directly from sample. Prepare a composite analytical unit, if appropriate, taking an approximately equal weight of product from each subsample. State how analytical unit is taken. AOAC methods are available for light filth in leafy products and excreta in condimental seeds.

b. Visual Examination -- Examine each product piece in the analytical unit for reject material visible to the naked eye. Magnification may be used for confirmation, as necessary. If the magnification exceeds 10X, this should be stated in the report of results. Classify, weigh, or count each category according to (5)c. below.

c. Classification of Reject Product Material -- Classify reject product material (leaf, seed, fruit, root, bark, etc.) as follows:

(i) *Insect-Damaged* -- Any product material exhibiting definite evidence of insect feeding or containing one or more whole insects or equivalent, webbing, or excreta. Determine, if possible, whether infestation is "Field" or "Storage," making special notation for live insects. Determine average length and note range of lengths for any larvae and/or unidentified insects present. Examine 50 g subsamples of bay leaves for field insect damage according to instructions given in Figure V-12.

(ii) *Moldy* -- Any product material bearing mold on more than 1/4 of its surface area or any material where the aggregate moldy area is greater than 1 cm². Confirm presence of mold with magnification as necessary, but determine the area affected without magnification. Describe general appearance of the moldy areas.

(iii) *Animal-Contaminated* -- Any product material showing animal excreta, animal chewing, or gnawing.

(iv) *Otherwise Reject Material* -- Any product material that is not classified as above, but is otherwise decomposed, discolored, abnormal in appearance or otherwise unfit for food.

d. Report - Tabulate results as follows:

	Subsample No.			
	1	2	3	etc.
Amount Examined (wt or no. of units)				
Insect-Damaged ^a Wt or No. %				
Moldy ^b Wt or No. %				
Animal Contamination Wt or No. %				
Total Percent of Reject				
Remarks:				

Notes:

- a. Describe ((5)c.(i)); report under Remarks
- b. Describe ((5)c.(ii)); report under Remarks

References

- (1) American Spice Trade Assn. Inc., *Cleanliness Specifications for Unprocessed Spices, Seeds and Herbs*, ASTA, 580 Sylvan Ave., Englewood Cliffs, NJ 07632, Jan. 1983.
- (2) ASTA, *Official Analytical Methods of ASTA*, 2nd Ed., 1968.
- (3) Parry, J. W., *Spices - Their Morphology, Histology, and Chemistry*, Chemical Publishing Co., Inc., New York, NY, 1962.
- (4) White, Ralph T., "Studies on the Storage and Shipment of Whole Black Pepper Grown in the Orient," *J. Econ. Entomol.* 50: 423-428, 1957.

Revision History

Version No.	Purpose of change	Date
V0	New process	1984
V1	Electronic Version	1998