

Macroanalytical Procedures Manual (MPM)

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

B. Supplemental Method for Black, White, Green, and Red Peppercorns, (V- 39)

Version 2 — March 2026

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B. Supplemental Method for Black, White, Green and Red Peppercorns, *Piper nigrum* L. (V-39)

(1) Scope

This method supplements and/or replaces those procedures in **Section 8.A.**, by describing procedures specific to black, white, green and red peppercorns, *Piper nigrum* L.. Pepper was the first spice traded worldwide (Lim, 2012). When dried, the fruit is referred to as peppercorns (Lim, 2012). Peppercorns come in different colors based on harvest and processing conditions. Green peppercorns are unripe and dried or brined to retain their green color. Black peppercorns are mature green peppercorns, which after picking are allowed to ferment, then dried to produce the familiar black coloration. White peppercorns are just a hullless black peppercorn. When peppercorns are ripe, they turn from their green coloration to red (Katzer, 2015). Tellicherry peppercorns are also *P. nigrum* L. Tellicherry peppercorns have gone through a sorting process and are larger than the 'regular' black peppercorns (Katzer, 2015) (Figure V-8-B-1). The term "siftings and pickings" is used to describe certain objectionable matter which may be present in peppercorns. When examining peppercorns, the procedure for "siftings and pickings" described below may be used in lieu of **Section 8.A(4)**.

Note: pink peppercorns (Spiegel, 2022) should not be confused with true red peppercorns, *P. nigrum* L. Pink peppercorns can come from three different types of plants: corktree, *Euonymus phellomanus* Loes, Peruvian peppertree, *Schinus molle* L. and Brazilian peppertree, *S. terebinthifolius* Raddi. Other types of pepper include Indian long pepper, *Piper longum* L., and cubeb pepper (tailed pepper), *P. cubeba* L.f. (Figure V-8-B-2).

(2) Applicable Documents

- [CPG Sec. 525.625 Whole and Ground Pepper - Adulteration with Insect & Rodent Filth; Mold; Mammalian Excreta; Foreign Matter](#)

(3) Defects

Storage pest arthropods often infest dried peppercorns. Two of the more common storage pest insects found in peppercorns are drug store beetles, *Stegobium paniceum* (Linnaeus) and cigarette beetles, *Lasioderma serricorne* (Fabricius). Psocodea and Acari are regularly found in peppercorns as well. Various molds have been found in peppercorns including *Aspergillus* sp. Micheli ex Haller (White, 1957).

(4) Procedure: Determination of "Siftings and Pickings" and Animal Contamination in Peppercorns

a. Special Apparatus -- This procedure calls for use of a standard pepper sieve. The sieve consists of a No. 9-1/2 round screen with a frame 45.72 cm to 55.88 cm diameter and 6.985 cm in height. The bottom is a metal sheet perforated with round holes 7/64 in. in diameter with an average of 5-1/2 holes per linear inch (small or "office" size 20.32 cm to 22.86 cm in diameter). U.S. Standard No. 8 sieves (0.0937 in. or 2.38 square mm opening) provide equivalent sieve opening. Note: if the peppercorns appear shiny and leave an oily stain on paper, they may be coated with an oil to mask any appearance of surface mold. This oil layer should be removed before the peppercorns are examined for mold. This oil layer can be removed by soaking 50 g of peppercorns in 100 ml petroleum ether. After soaking for a few minutes, pour off the solvent and allow the peppercorns to thoroughly dry (ASTA 2014). Then, continue with the examination.

b. Siftings – Examine six subsamples containing at least 700 g each. Combine equal aliquots from the six subsamples to give a composite sample of approximately 4 kg, unless product lot variability indicates analysis of individual subsamples. Weigh composite sample and divide into approximately two equal portions. Screen each portion separately, using the standard pepper sieve (when using the small or "office" sieve, screen only 400 to 500 g at a time) or the U.S. No. 8 equivalent. Obtain the siftings by tilting the sieve from side to side so that the pepper passes from one side of the sieve to the opposite side 10 times. Sieving the material over a large sheet of white paper can be helpful. Weigh siftings after removal of pinheads/broken pieces/light and immature peppercorns, which are not considered 'foreign matter. Classify the foreign material as in [Section 8.A\(4\)c](#).

To facilitate the removal of pinheads and immature peppercorns from the white paper, separation may be performed using one of two techniques after sieving. **Method 1:** Gently tilt the white paper so the peppercorns roll to one side, allowing them to be easily separated from the lighter material. **Method 2:** Attach an aspirator to a vacuum pump and *carefully* aspirate the peppercorns into a collection jar, leaving the foreign material behind. Limit the aspirator in-tube diameter to the size of the peppercorns being removed. Other techniques can be used, if they do not interfere with the siftings. Examine siftings; identify, describe, and quantify, as necessary. Siftings include insect excreta which passed through the screen. Peppercorns with attached stems should be treated like the pinheads, while stems lacking peppercorns are to be treated as foreign

material. Note: Cubeb pepper (tailed pepper), *P. cubeba* L.f, has attached stems to the peppercorns (Figure V-8-B-2(A)).

c. Pickings -- After sifting, hand-pick sample ("overs" on the sieve) for foreign material such as sticks, stones, stems, clay, foreign seeds, insect excreta and other extraneous matter. Weigh pickings other than animal contamination. Classify this material as in [Section 8.A\(4\)c](#).

d. Animal Contamination -- Examine siftings and pickings for animal contamination, such as mammalian excreta, bird excreta, and whole insects or equivalent. Classify, weigh, and report as in [Section 8.A\(4\)c and d](#).

e. Report -- Tabulate results, adding additional categories as necessary ([Table V-8-A-2](#)).

(5) Procedure: Determination of Arthropod Damaged, Moldy, and Otherwise Reject Peppercorns

a. Sample Preparation – Using the remaining portion from each of the six subsamples in [Section 8.B\(4\)b](#), examine a representative sample by counting out 200 peppercorns from the first subsample and weighing it. From the remaining five subsamples, examine a representative portion from each, based on the weight of the 200 peppercorns counted and weighed in subsample one. Cut open any suspect peppercorn as necessary.

b. Visual Examination -- Examine each analytical unit for reject material visible to the naked eye up to 10x assisted magnification. If the magnification exceeds 10x for the initial examination, this should be stated in the report of the results. Higher magnification may be used for confirmation of findings after the initial examination. Figure V-8-B-3 displays examples of peppercorns affected by arthropod damage. Meanwhile, Figure V-8-B-4 demonstrates both internal and external mold in peppercorns.

c. Classification of Reject Product Material – Classify reject material as in [Section 8.A\(5\)c](#). Report findings by weight and percentage in ([Table V-8-A-3](#)).

d. Report - Tabulate results as follows ([Table V-8-A-3](#)):

Figures

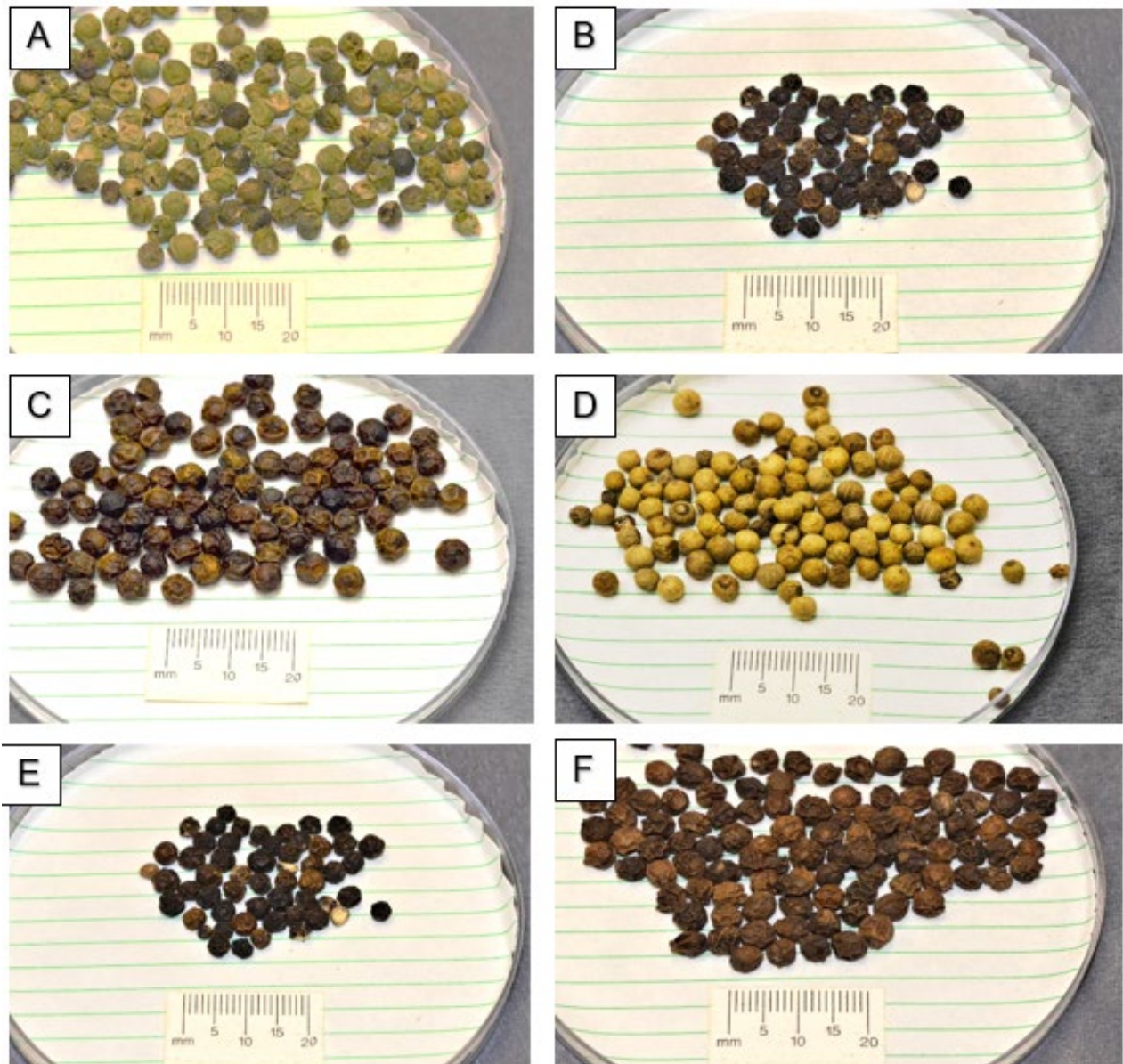


Figure V-8-B-1. Black peppercorns, *Piper nigrum* L. **A.** Unripe green peppercorns. **B.** Black peppercorns. **C.** Ripe red peppercorns. **D.** White peppercorns, black hulls removed. **E.** 'Regular' black peppercorns. **F.** Tellicherry black peppercorns. (Source: Photos courtesy of H. Loechelt-Yoshioka, FDA).

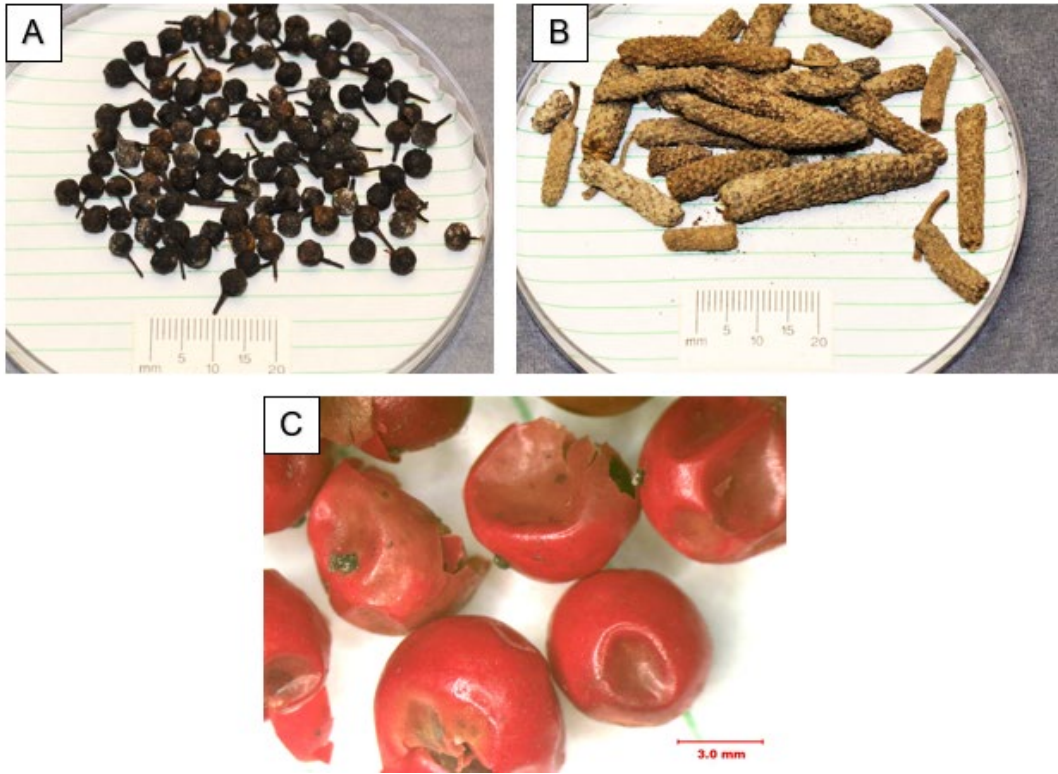


Figure V-8-B-2. A. Cubeb pepper (tailed pepper), *P. cubeba* L.f.. B. Indian long pepper, *P. longum* L. C. Peruvian peppertree, *Schinus molle* L. (scale bar: 3.0mm). (Source: Photos courtesy of H. Loechelt-Yoshioka, FDA).

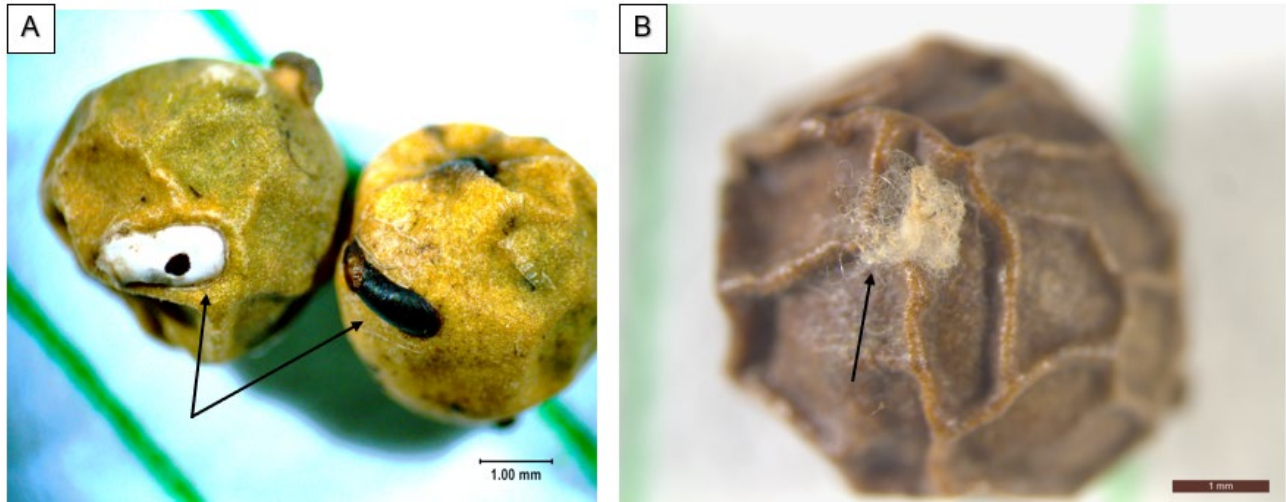


Figure V-8-B-3. A. Peppercorns with attached scale insects, Diaspididae, indicated by arrows. (scale bar: 1.00mm). B. Arrow pointing to external insect webbing material. (scale bar: 1mm). (Source: Photos courtesy of H. Loechelt-Yoshioka, FDA).

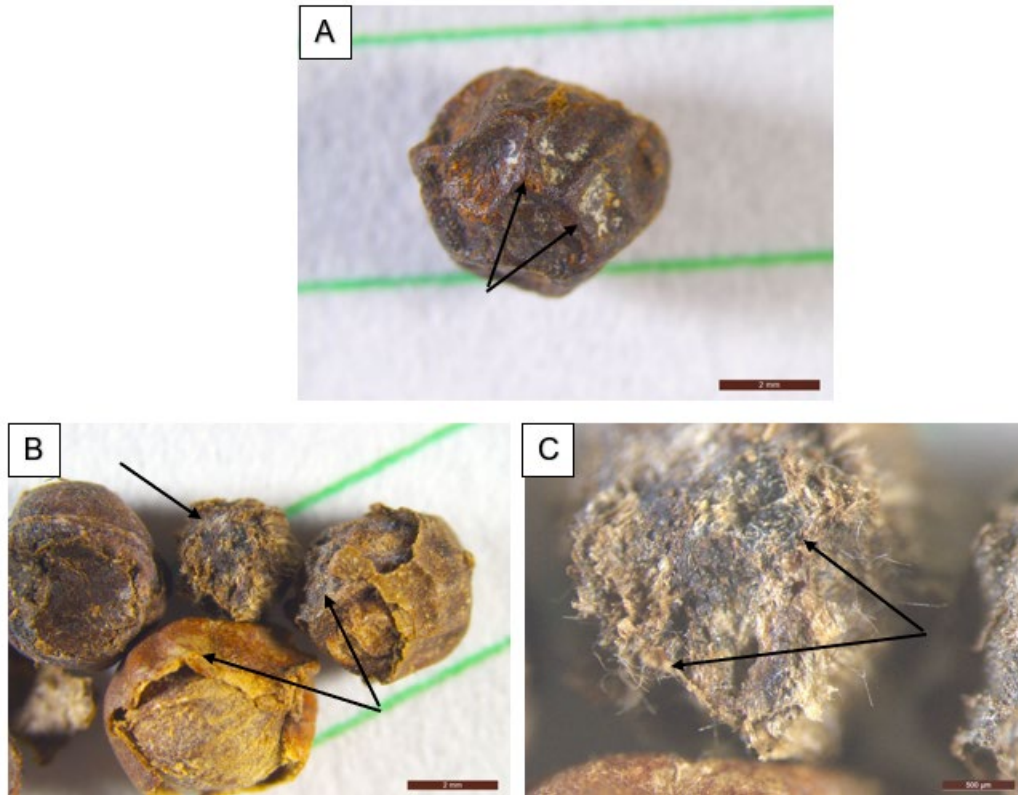


Figure V-8-B-4. A. Peppercorn with external mold indicated by arrows. (scale bar: 2mm). **B.** Peppercorn with internal mold indicated by arrows. (scale bar: 2mm). **C.** Close-up of peppercorn with internal mold indicated by arrow. (scale bar: 500μm).
(Source: Photos courtesy of H. Loechelt-Yoshioka, FDA)

Acknowledgements

The editors are grateful for the following USFDA staff members for their input and review of this document: Roger Burks, jr., Heather Hawk, Karen Jackson, James Madenjian, Amy Miller, and Monica Pava-Ripoll.

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Additional Information

Informational articles not cited in the above section, but still useful:

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Revision History

Version No.	Purpose of change	Date
V0	New process	1984
V1	Electronic Version	1998
V2	Added defects, images and references. Updated scope, procedures, and tables.	2026