

Light Berries and Extraneous Matter in Black and White Pepper

Purpose: To determine the amount of light berries and extraneous matter in black or white pepper.

A. Apparatus:

1. A standard pepper sieve, (No. 9 1/2 round screen with a frame 18 to 22 inches in diameter and 2 3/4 inches in height. The bottom is a metal sheet perforated with round holes of 7/64 inch in diameter, with an average of 5 1/2 holes per linear inch. Screen only with standard pepper sieve obtainable from: McNichols Company, 5501 Gray Street, Tampa, Florida 33609 (813) 876-4100 or (800) 237-3820. U.S. Standard No. 8 sieve (0.0937 in. or 2.38 square mm opening) provide equivalent sieve opening.
2. Balance -- sensitivity 0.01 g.
3. Beaker, 600 ml. Griffin, Low form, pyrex approximately 85 mm. in diameter and 120mm. in height is recommended. (Note 1).
4. Blotting paper or other similar absorbent material.
5. Tweezers.
6. Stereoscopic, Binocular, wide-field microscope (40-50x).

B. Reagents:

1. Alcohol-water solution of a specific gravity 0.80 - 0.82 at 25°/25°. The alcohol may be ethanol, denatural ethanol (Note 2) or isopropanol.

C. Preparation of Sample:

1. The number of samples drawn must be equal to the square root of the packages, bags, or containers in the lot, with maximum of 10 samples drawn.
2. The sample size shall be 3/4 to 1 pound (340 g to 454 g).
3. Each entire subsample must be analyzed.

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D. Procedure:

1. To Determine Excreta, Insects, Mites, Psocids, Mold, and Percent Black Pepper in White Pepper:
 - a. Weigh each sub-sample to the nearest gram. Sprinkle and examine a small portion at a time, with a good light and against a white background into a standard pepper sieve. Pick out any bird, rodent, or other animal excreta. Separate mammalian from non-mammalian excreta. Weigh to the nearest 0.1 mg and record. Do not remove other extraneous/foreign material at this time.
 - b. Shake pepper sieve moderately back and forth, examine siftings collected on white background for live and dead insects and for excreta.
 - c. Accumulate the siftings.
 - d. Mix sub-sample of pepper on sieve and weight 50 g of aliquot into a pan. Hand-pick moldy peppercorns and weigh. In case of white pepper, additionally hand pick for black peppercorns (with skin coat attached) and record. Each sub-sample is examined in sequence in a similar manner and the results are averaged.
2. Extraneous/Foreign Matter by Sifting:
 - a. Weigh to nearest 0.1 g the cumulated siftings and calculate the percentage by weight. Percent siftings must be determined after the removal of small berries that pass through the pepper sieve. (See Calculations)
3. Light Berry Determination for Black Pepper:
 - a. Combine sufficient material from each sub-sample to give a composite sample of approximately 5 lbs. Mix composite well.
 - b. Form the composite into a pile shaped like a cone. Quarter the cone designating each quarter as A, B, C, or D in a clockwise sequence.
 - c. Set aside two opposite quarters such as A and C.
 - d. Mix and reduce each quarter separately.
 - e. Remove a 50.0 g sample from each reduced quarter.
 - f. Place the weighed sample in the 600 mL Griffin, low-form pyrex beaker and add 300 mL of the alcohol-water solution.
 - g. Stir the material in the beaker with a spoon and allow to settle two minutes; then spoon off the berries which float.
 - h. Repeat the stirring, settling and removal of the floating berries until two successive additional stirrings raise no more berries to the surface. Remove only the berries that actually float (Note 3).
 - i. Blot the removed berries to free them from excess liquid and spread them out to dry on a piece of paper towel or other absorbent material.

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- j. Air dry for one hour and weigh the air dried light berries to the nearest 0.01 g and calculate and report the percent of light berries to the nearest 0.1%.
(See Calculations)
 - k. If the range of two determinations is not over 0.8%, the two determinations shall be averaged and reported as percent light berries. If the difference is greater than 0.8%, determine the light berries in a third sample obtained from either quarter B or D. Average all three values and report as percent light berries.
4. Extraneous/Foreign Matter by Hand Picking
- a. From the two opposite quarters set aside for light berries determination such as A & C, weigh 100 grams from each and hand pick for any sticks, stones, stems, foreign seeds, other extraneous matter and make note of its nature.
 - b. Weigh the pickings and calculate. (See Calculations)

E. Calculations:

$$\% \text{ Extraneous/Foreign Matter by Sifting} = \frac{\text{Wt. of combined sifting (g)}}{\text{Combined wt. of sub-samples (g)}} \times 100$$

$$\% \text{ Light Berries} = \frac{\text{weight of light berries (g)}}{\text{weight of samples (50 g)}} \times 100$$

$$\% \text{ Extraneous/Foreign Matter by Hand Picking} = \frac{\text{A (in grams) + C (in grams)}}{2}$$

F. Statistics:

TBD

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G. Notes:

1. Other transparent beakers may be used, but they should be between 75 and 100 mm. in diameter and between 100 and 140 mm in height.
2. Specially denatured alcohols no. 3A, 23A, or 30 are recommended.
 - a. SDA no. 3A: 5 gallons of methyl alcohol plus 100 gallons 95% ethyl alcohol.
 - b. SDA no. 23A: 10 gallons USP acetone plus 100 gallons 95% ethyl alcohol.
 - c. SDA no. 30: 10 gallons methyl alcohol plus 100 gallons 95% ethyl alcohol.
3. Some berries may remain suspended some distance below the surface of the liquid. These are not considered as floaters.

H. References:

Macroanalytical Procedures Manual 1984, Chapter 5.