Method 26.1

Screening Method for the Determination of Defatted Meal in Paprika and Black Pepper

Purpose: *To screen for the presence of defatted paprika and black pepper meal in **ground** products. Because the addition of spent paprika and black pepper meal will be in high concentrations (usually more than 20%), this rapid screening method will differentiate between pure paprika/black pepper and products that have been blended with the extracted meal depending on the color reactions displayed.*

A. Apparatus

1. Stereoscopic binocular microscope - wide-field capability with 30X - 70X capabilities, mounted on a base and capable of illumination by reflected light (transmitted light source also desirable for this method). Lights should be adjustable halogen sources corrected with daylight filters.

2. Microscope slides, glass – 3” x 1” x 1mm

3. Microspatula, stainless steel with 1/8” wide blade – any brand

4. Dissecting needle or stainless steel probe – any brand

5. Absorbent lab tissue – Kimwipes

6. Stirring rods, glass – any brand

7. Beaker, 200 mL, glass

8. Graduated cylinder, 100 mL, glass

9. Stopper/Dispenser Bottle, 1 - 2 oz., glass

10. Screw cap storage bottle, 2 – 4 oz., glass

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B. Reagents

1. Sulfuric Acid (H_2SO_4), 28.8N with boric acid. (See Note 1)

   a. In a glass beaker, carefully add 80.0 mL concentrated H_2SO_4 (reagent grade–any brand) to 20.0 mL distilled water.

   b. Allow mixture to stand and cool. Add 1.0 g boric acid (reagent grade – any brand).

   c. Stir solution with glass rod to dissolve boric acid.

   d. Dispense in a glass stopper/dispenser bottle. Mixed reagent is stable for months at room temperature. Extra reagent may be stored at 4^{o}C in glass bottle. (See Note 2)

C. Preparation of Sample

No preparation of sample is necessary.

D. Procedure

1. Take four (4) small aliquots of a ground paprika or ground black pepper sample (approx. 1/8" x 1/8" x 1/8" cubed) and place on 2 glass microscope slides (2 aliquots per slide).

2. Working with one slide at a time, add 4 – 6 drops of reagent to each aliquot. (See Note 3)

3. Thoroughly mix the reagent and first sample aliquot with a stainless steel needle or probe making sure the sample is broken up and evenly distributed on the slide.

4. Clean and dry needle (probe) thoroughly before proceeding to next sample aliquot.

5. View sample at 7X – 10X with incidental light. Switch to the transmitted base light within a few seconds for optimum viewing of color reactions in and surrounding the various types of particles in each sample**. Reactions must be observed within one minute from time of preparation.**

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6. A greenish olive to blue/black coloration indicates true paprika. The absence of the blue – greenish color and the presence of yellowish color to light brown bleeding reaction indicates the presence of defatted paprika meal. In a ground black pepper sample, a red bleeding color reaction indicates true black pepper. The absence of the red color and the presence of dull brown particles that **do not bleed** indicate the presence of defatted black pepper meal.

7. The presence of defatted material in a sample may be confirmed by using ASTA Method 27.0.

8. Discard reacted sample after one minute (from the time of adding reagent and mixing it with the sample aliquot). (See Note 4)

E. Interpretations

1. Paprika particles that rapidly bleed yellow to brown with the addition of the screening reagent are most likely **fully defatted spent paprika meal particles**.

2. Some potential paprika adulterants react with the screening reagent to form other colored chromophores that do not confound the screening test or they may not react at all. These suspected adulterant particles (ground pecan shells, etc.) are highlighted against the normally reacted particles. Identification of the adulterants or contaminants should be made via structural characteristics (see example above) at 200X – 400X magnifications with the compound light microscope (polarization may, at times, be a helpful aid toward final identification).

F. Statistics

1. Three analysts trained to perform and interpret this method analyzed sixty-four samples covering a variety of paprika and/or black pepper products with varying levels of added spent paprika/black pepper meal from 0 to 100%. Statistical evaluation of the data (conducted by way of Chi Square Analysis of Categorical Data – at level of 90% confidence) showed no significant difference between the analysts. At ranges of 20 – 80%, the defatted meal could be detected.

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G. Notes

1. **Do not** use stainless steel or plastic containers to prepare reagent.

2. Reagent will slowly degrade plastic dropper bottles. Discard any reagent that develops black specks or exhibits any discoloration. Reagent should be water-clear.

3. Four (4) to six (6) drops of reagent provides enough liquid to insure that all paprika and black pepper particles are coated and the reaction is complete (provided sample is adequately stirred with probe).

4. The blue/greenish/black reaction in paprika and the red reaction in black pepper fade rapidly and browning reactions intensify; each color on a sample slide merges with the others so that interpretation of the reaction becomes unreliable and therefore invalid.

H. References

1. Lynn Bates, Alteca Ltd., Manhattan, Kansas, developer of method, 3/2000.