Macroanalytical Procedures Manual (MPM)

II. Apparatus for Macroanalytical Methods

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Some pieces of apparatus and their application are very closely associated with macroanalytical methods. This chapter describes some of the apparatus of special significance to macroanalytical procedures contained in this manual. Unless otherwise noted, these items of apparatus are available through most scientific supply houses.

When following the procedures outlined in this Manual, the analyst should consider the general points noted below:

- The use of polyethylene or spun-metal non-polished beakers, funnels, and containers should be avoided, as insect fragments and rodent hairs may adhere to apparatus made of these materials.
- Glassware should be heat-resistant and, where possible, of heavy wall construction.
- Glass slides should be ordered "pre-cleaned." Despite the label, they should be cleaned prior to first use.
- Glass cover slips should be ordered to meet specifications built into the microscope optical system. Also, cover slips of various sizes/diameters (round, square, rectangular) should be available to select according to the intended use.

(1) Blender, ***a. High Speed Blender***

A blender/mixer with 4 canted, sharp-edged, stainless-steel blades rotating at the bottom of a 4-lobed jar. Waring blenders, or equivalent, meet these requirements (Figure II-1).

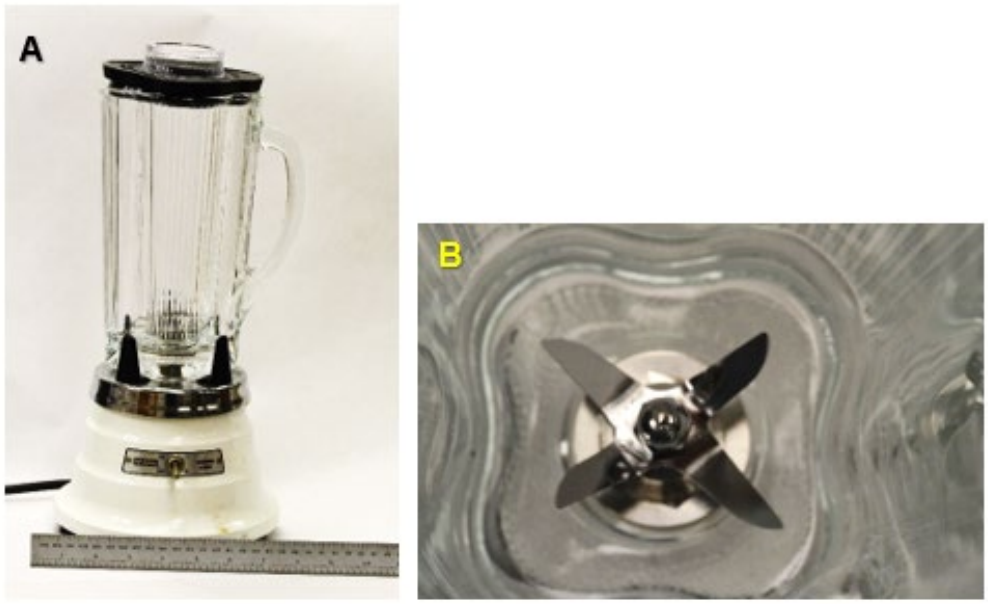

Figure II-1. A. High speed blender, Waring blender **B.** Close-up view of the canted stainless-steel blades. (Source: Photos courtesy of H. Loechelt-Yoshioka, FDA).

b. Variable Speed Transformer

Used with the blender to control the speed. Output voltage 0-1.40; max amperes 7.5 (Figure II-2).

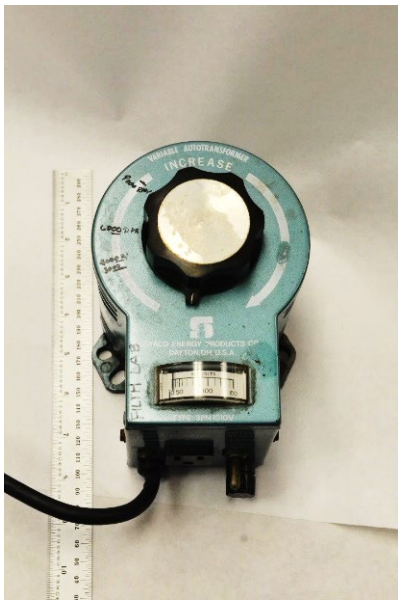

Figure II-2. Variable Speed Transformer. (Source: Photo courtesy of H. Loechelt-Yoshioka, FDA).

(2) Bolting Cloth

A silk cloth used to filter extraneous material. The cloth is woven to provide a mesh of standard sized openings and thickness, which is approximately similar to that used in flour mills. The number of the silk specifies the number of openings per linear inch in the mesh. The addition of "X", "XX", or "XXX" after the number refers to the thickness of thread from which the cloth is woven. Since both the number and the "X" rating affect the size of the openings in the bolting cloth, recommendations should be followed exactly.

a. Preparation of silk bolting cloth

Discs are best prepared by boiling large squares of silk before cutting them into circles. Circles cut from un-boiled silk will shrink and become misshapen. Cut circles of ca 85 mm diameter or larger (Figure II-3). To aid in analysis, rulings should be made with India ink or other permanent marking material, using a crow-quill pen, on the boiled and pressed cloth. The inked lines should be set 5-7 mm apart, or temporarily with a line-marking ring as described in (10) below.

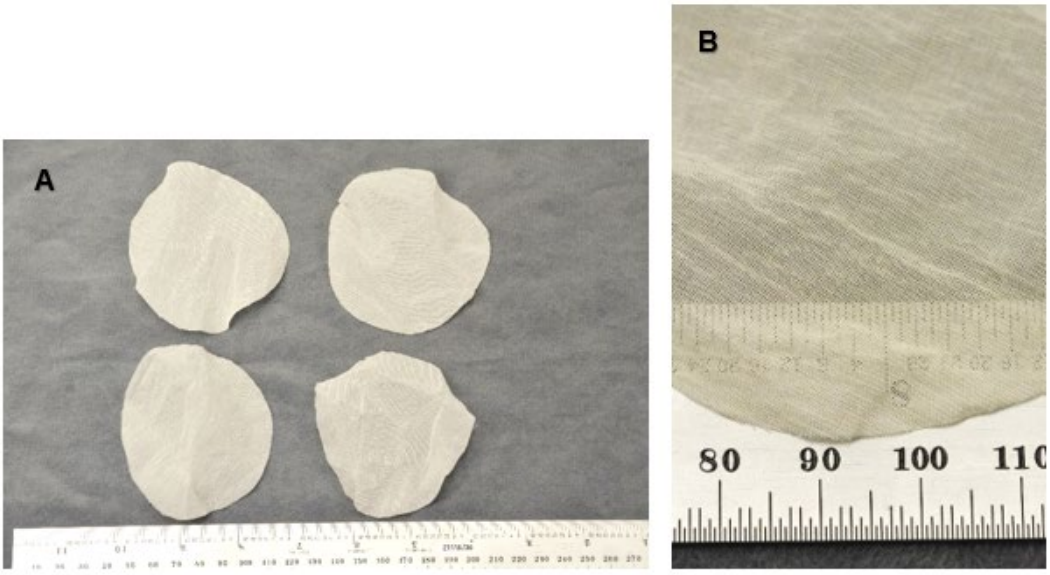

Figure II-3. A. Bolting cloth discs. **B.** Close-up view of the bolting cloth. (Source: Photos courtesy H. Loechelt-Yoshioka, FDA).

b. Preparation of dyed bolting cloth discs:

Prepare silk bolting cloth as described above, then follow the dying procedure: in an1.5 L beaker dissolve 0.5g of FD&C Blue No. 1 (also known as Brilliant Blue FCF) in 1 L tap water. Add 2.5 ml concentrated acetic acid and warm to about 80° C. Add up to 6 ruled cloth discs to the beaker and keep at 80°-85° C for 15 min, with occasional stirring.

Rinse cloths in running tap H_2O and dry. Store cloths in darkness to retain color (USFDA, 1971).

c. Polyethylene cloth:

Polyethylene cloth is another option to use, it is much cheaper than silk bolting cloth, does not have to be boiled for sizing, and can be dyed with certain dyes suitable for plastic fabrics.

(**3) Cracking Board**

A cracking board is made from at least 38.10 cm square sheet of 0.635 cm aluminum or plywood drilled with one hundred, 25.0 mm holes in diameter, equally spaced in 10 rows of 10 holes each (Figure II-4). When examining the product, place the board on a large sheet of paper on a hard surface. Scatter the product on the board to fill the holes. Sweep the excess product off with the hand and adjust any empty or double-filled holes so that each of the 100 holes contains a single item. For examining cocoa beans, open each bean by placing an iron bolt or similar object on the bean and gently tapping the head of the bolt with a hammer. When examining other products, use appropriate devices to break open the product

Figure II-4. An aluminum cracking board being used for examining whole nutmegs. (Source: Photo courtesy of FDA).

(4) Crystalizing Dish

Consists of a large diameter beaker with a flat bottom and short height. By having a large surface area, solutions tend to evaporate faster, causing the compounds to recrystallize. The crystallizing dish can be thick walled or thinned walled (Figure II-5).

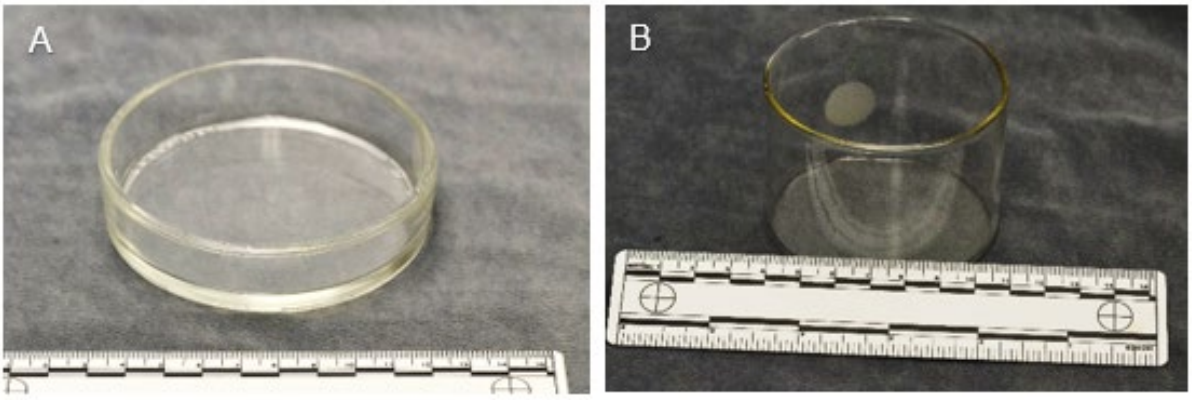

Figure II-5. A. Thick-walled crystallizing dish. **B.** Thin-walled crystallizing dish. (Source: Photos courtesy of H. Loechelt-Yoshioka, FDA).

(5) Dividers/Splitters

Dividers/Splitters are used in representative sampling and sample reduction of free-flowing products such as grains. The dividers described here stationary mechanical dividers, which are used to divide samples into two equal representative portions, rotary splitter, and fractional shoveling.

a. Jones Divider

The Jones divider consists of a hopper that opens to numerous chutes which discharge alternately on opposite sides of the apparatus (Figure II-6). The chutes should be an even number, so the two sub samples formed have the same mass. Material poured into the hopper is thus directed in approximately equal portions to the two trays positioned below the chutes. The two resulting subsamples are each representative of the original sample. This sampler has been used with good results for many years in FDA field sampling applications. However, there are two disadvantages in using the Jones divider: 1. The analyst using the divider needs to be trained on the proper use of the equipment. 2. When sending product through the divider, some of the dust/fines of the product remain in the channels. This can be reduced by sending the product through the divider 6 times.

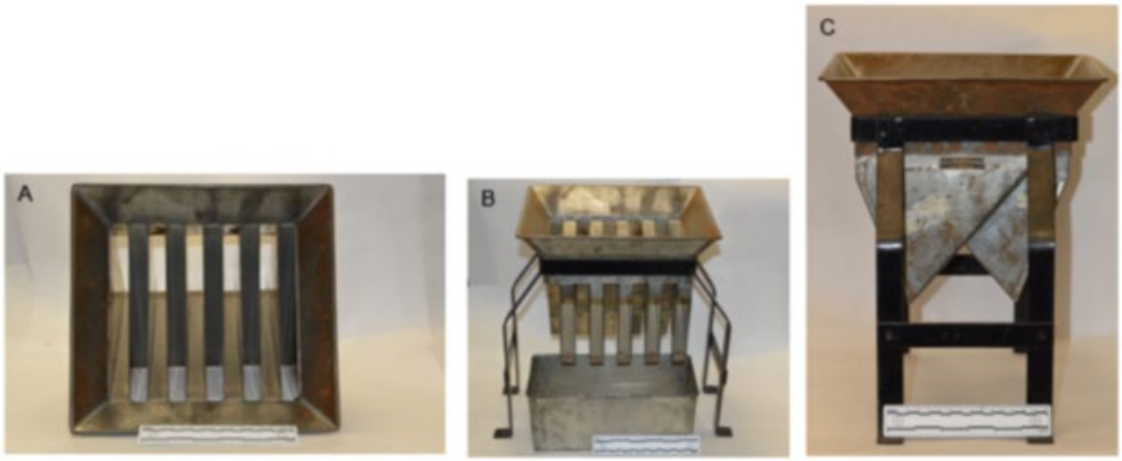

Figure II-6. Jones divider **A.** Top View. **B.** Side view with collector pan. **C.** End view. (Source: Photos courtesy of H. Loechelt-Yoshioka, FDA)**.**

Below is a list of recommendations when using the Jones divider, (Figure II-7) shows a Jones divider being used and (Figure II-8) shows a flow diagram of the recommended process for using the Jones divider.

- If the sample is less than 280 g it does not need to go through a divider.
- Pass the product through the divider 6 times. On the sixth time retain half the product in a separate container and the remaining half of product is sent through the divider 6 more times. Reduce product by half after each sixth pass of the product through the divider. Continue this reduction process until the amount of product needed for analysis is reached.
- Have one collecting pan on each side of the divider.
- The divider must have an even number of riffles, so the two-sub samples have the same mass.
- The chute widths should be 2.5× the maximum particle size so as to preclude blocking of the chutes by groups larger fragments in the lot (i.e.,1.90 cm for dent corn.)
- The material has to be dry.
- The divider should be fed by a pan, bucket or hopper that is the same width as the set of riffles in the device (Figure II-7). The product should be spread in the pan, bucket or hopper evenly and so it is level in the container. When splitting, the material should be fed slowly and evenly to the center of the device (over the chutes), thus it is important to use a tray that is the width of the chutes, and the material is level and spread evenly over the tray before dumping. Dumping material from bags, buckets, scoops or shovels is incorrect and may lead to unacceptable sub sampling biases.
- Importantly, the divider needs to be cleaned after each use, using a brush or vacuum. If wet washed, it has to be dried prior to use.
- Analytical error is small, typically less than 2%, when properly used.

Figure II-7. A. Correct use of the Jones divider. The pouring container is the same width as the divider. **B.** Incorrect use of the Jones divider. The pouring container needs to be the width of the divider. (Source: Photos courtesy of H. Loechelt-Yoshioka, FDA)

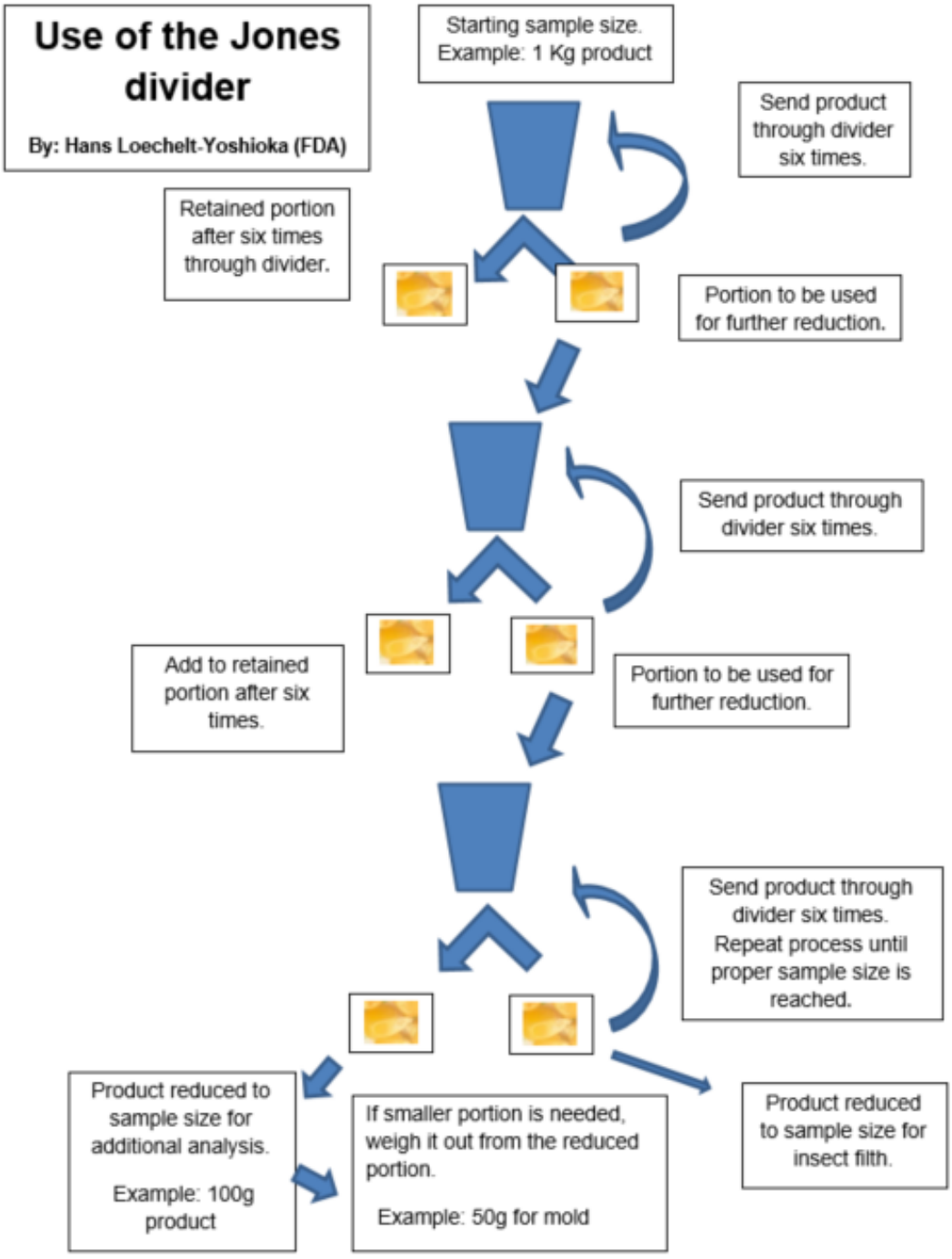

Figure II-8. Flow diagram of the process of using a Jones divider.

b. Boerner Divider

The Boerner divider employs a conical design to perform the same function as the Jones divider (Figure II-9). The material flows from a funnel-like hopper down the sides of a cone, the tip of which is directly below the center of the hopper opening. A series of channels around the periphery of the cone direct the material into one of two collecting bins. The direction of flow of the channels alternates around the edge of the cone so that every other channel directs the flow into the same collecting bin. The Boerner divider is a highly accurate device for sample division and is standard equipment in Federal, state and local grain inspection offices.

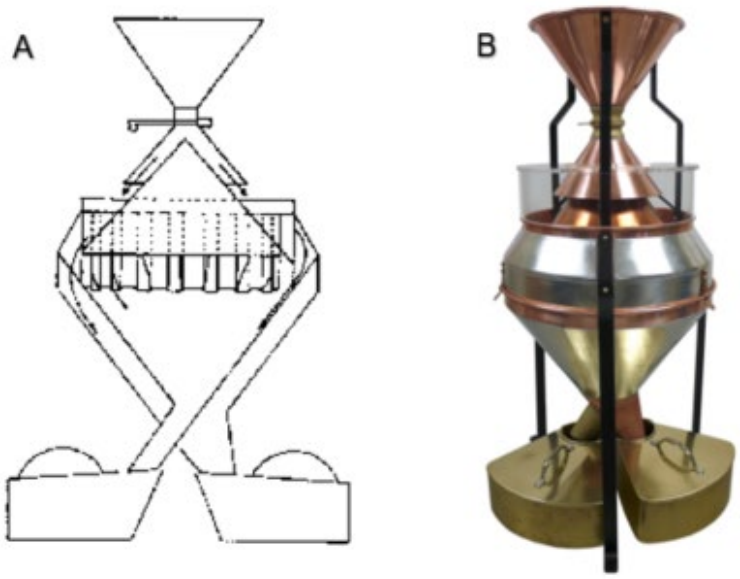

Figure II-9. **A.** Boerner Divider. (Source: MPM 1984, electronic version 1998). **B.** Boerner Divider. (Source: Photo courtesy of the USDA-AMS-FGIS Equipment Handbook, 2016).

c. Rotary Splitter

The rotary splitter is a motorized splitter, consisting of a hopper, chute, and rotating receptacles. A certain amount of product is placed into each receptacle after each rotation. Some of the requirements for the rotary splitter: cutting edge should be radial from center, splitter needs to split the sample into equal portions, should maintain a constant speed, and the drop from the feeding chute to the receptacles should be minimal to reduce dust formation. The best results are obtained when operating the rotating receptacle with a constant rotational velocity and feeding from the hopper at a constant rate.

d. Fractional Shoveling

The sample is divided and placed in individual receptacles to form even splits (Figure II-10). This technique can produce any number of splits from (2 to n). The increment mass needs to be divided so all receptacles have the same mass of material. The created increments need to be sequential across all the receptacles and repeated in the same sequence until all the sample is divided evenly into all receptacles. This technique does not require expensive equipment, but it is time consuming. All splits need to be equal.

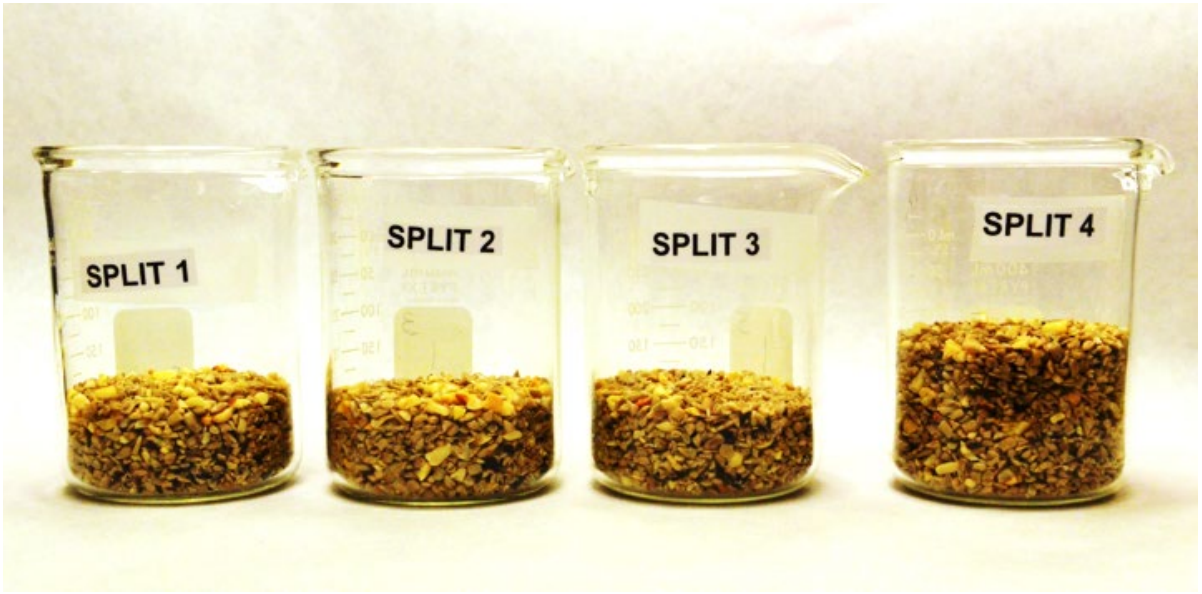

Figure II-10. Fractional Shoveling. Splits 1-3 are correct and split 4 is incorrect. (Source: Photo courtesy of H. Loechelt-Yoshioka, FDA)

(6) Filter Paper

For macro or micro examination, use rapid acting paper, ruled with permanent inks, 5 mm apart #8 ruled or equivalent) (Figure II-11). For heavy filth or ashing, use fast, ashless filter paper (41 Ashless or equivalent). Black filter paper can be useful when checking for arthropod eggs, glass, mites, or other light colored filth elements (Microanalytical Items, 1952). Black filter paper can be ruled. Gridded filter paper can be useful as well.

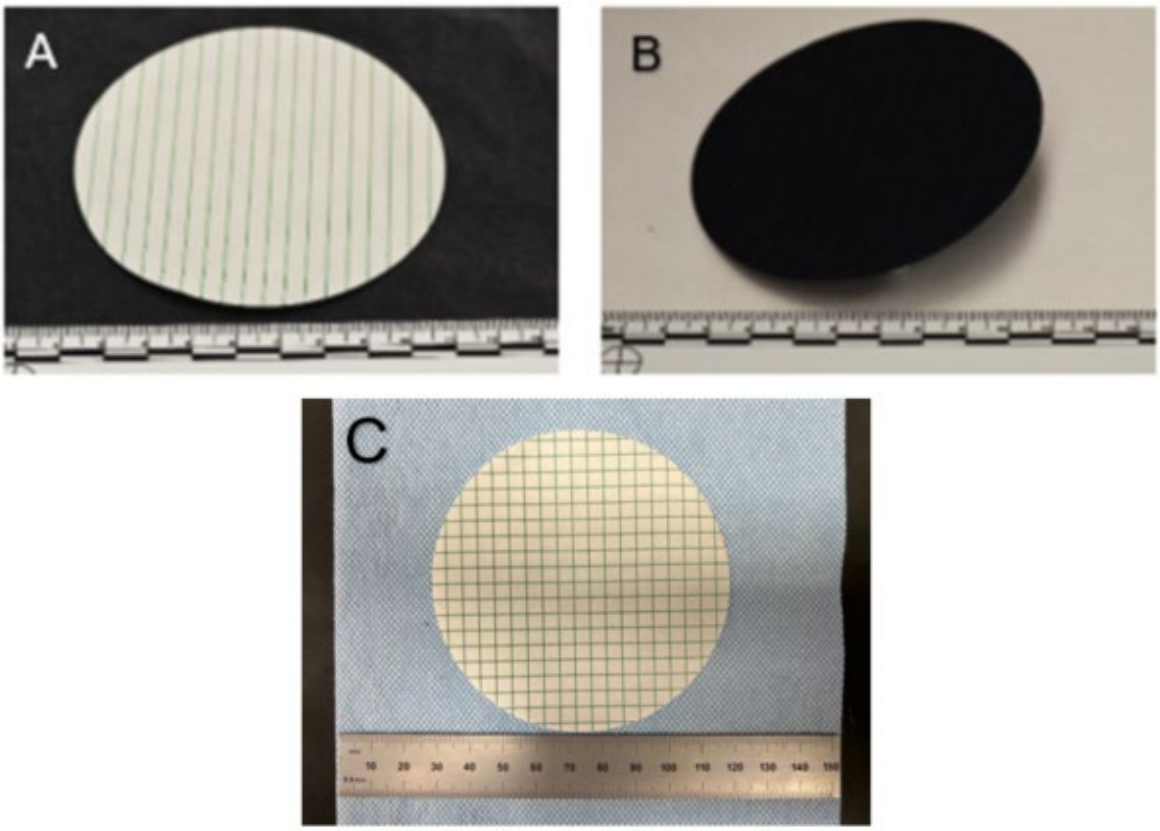

Figure II-11. A. #8 white ruled filter paper. B. #8 black ruled filter paper. C. 9 cm gridded filter paper. (Source: Photos courtesy of H. Loechelt-Yoshioka & J. Nickelsen, FDA).

(7) Funnels

Several types are useful in macroanalytical procedures.

The Hirsch funnel and the Büchner funnel are used for filtration with suction (Figure II-12). Use either funnel with filter papers or bolting cloth cupped up on the sides to eliminate loss of solids. Use rapid filter paper for filtration. Bolting cloth or screen (wire, fiberglass, or plastic) should be placed between the perforated funnel plate and filter paper to accelerate filtration and more uniformly distribute the solids. Changing the vacuum, breaking up clumps, and washing down the sides of the paper will also facilitate more uniform distribution of the filtrate. For the Hirsch porcelain funnel, which has an approximate top diameter of 94 mm and plate diameter of 56 mm, use rapid paper, 9 cm in diameter or #8, 7.5 cm. Note: a Büchner funnel has a wider base and straight sides (Figure II-12B).

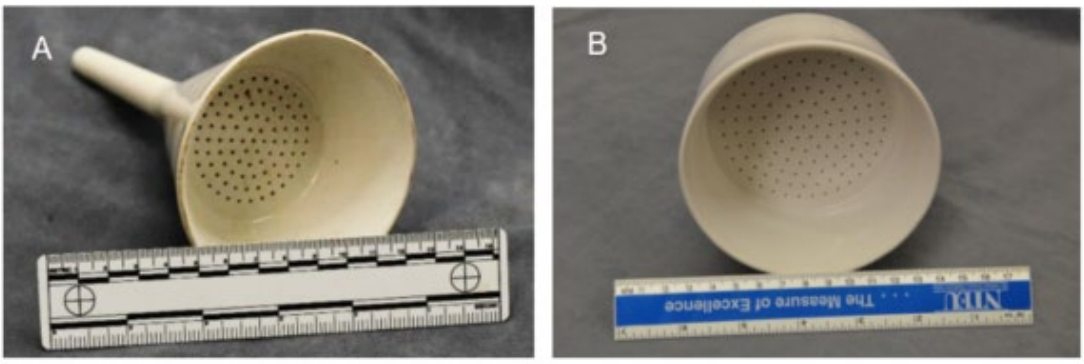

Figure II-12. A. Hirsch funnel. **B.** Büchner funnel. (Source: Photos courtesy of H. Loechelt-Yoshioka, FDA).

For transferring the product to a trap flask, a 6-10 in. stainless steel or glass funnel with an opening or stem of. 15.8 mm to 25.4 mm is required (Figure II-13).

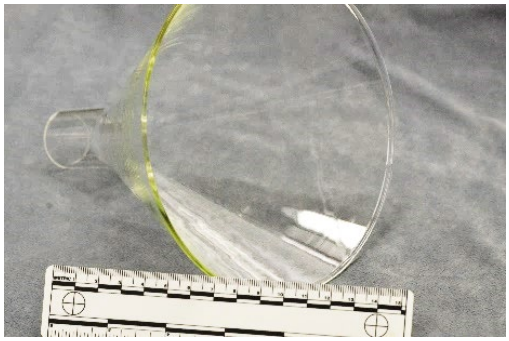

Figure II-13. Glass transfer funnel. (Source: Photo courtesy of H. Loechelt-Yoshioka, FDA).

For other purposes, standard glass or stainless-steel powder funnels are available (Figure II-14)

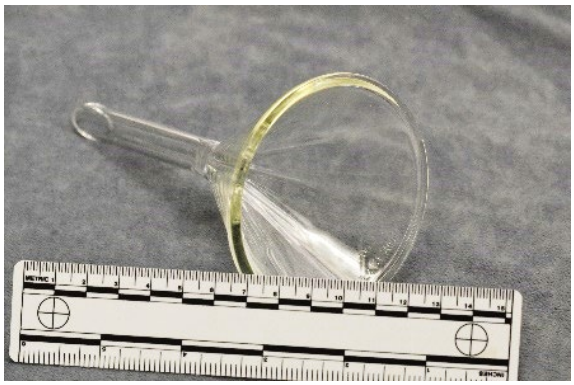

Figure II-14. Glass powder funnel. (Source: Photo courtesy of H. Loechelt-Yoshioka, FDA).

(9) Jeweler's Forceps

Used to manipulate delicate specimens (Figure II-15).

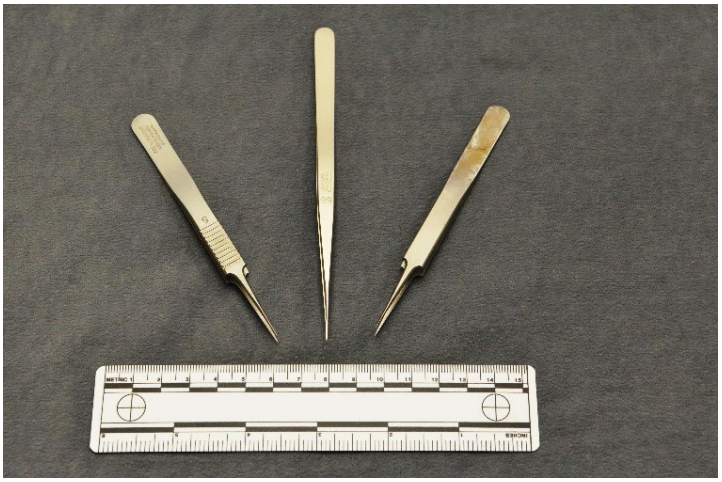

Figure II-15. Various jeweler's forceps. (Source: Photo courtesy of H. Loechelt-Yoshioka, FDA).

(10) Line-Marking Ring

Used to make a counting grid on prepared filter papers or bolting cloth. Consists of a steel or plastic ring (made to fit inside petri dishes and over filter papers) strung with nylon line (Figure II-16). Lines are set 5 or 7 mm apart with small holes or slits in the ring to hold lines in place.

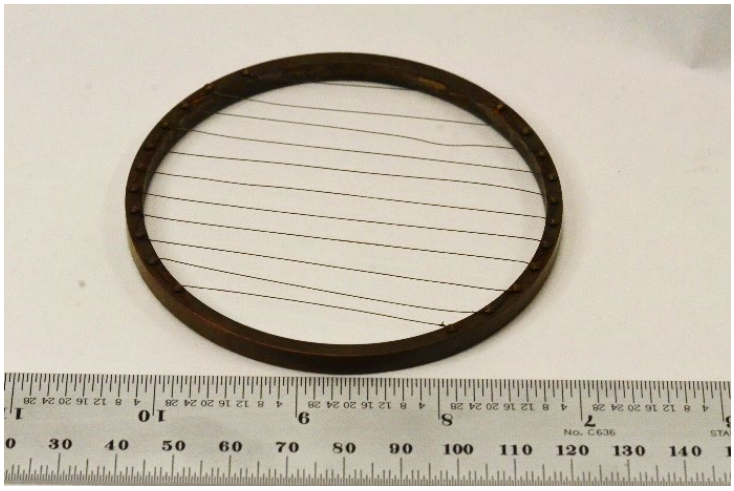

Figure II-16. Line-marking ring. (Source: Photo courtesy of H. Loechelt-Yoshioka, FDA).

(11) Laboratory Cyclone

The laboratory cyclone consists essentially of a cylindrical perforated metal screen in which revolves a paddle which forces the soft material from the food product out through the openings in the screen (Figure II-17). Tough materials such as seeds, skins, and stems are removed through a second opening. The screen is of 22-gauge material having 400 holes per square inch, each 0.027” in (0.6858 mm) diameter.

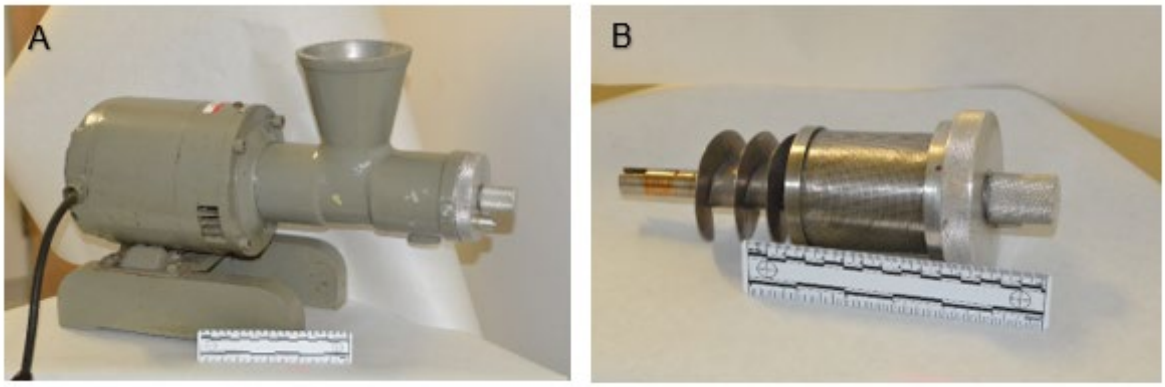

Figure II-17. A. Laboratory cyclone. **B.** Internal paddle with screen removed from cyclone. (Source: Photos courtesy of H. Loechelt-Yoshioka, FDA).

(12) Light Table

Used in the examination of fish fillets for parasites. A light table or sometimes called a light box contains a bright light source covered by a translucent white plastic cover (Figure II-18). Parasites will often appear as darker shadows in the translucent white flesh of the fish. The Bacteriological Analytical Manual (BAM) also gives some additional information on specifications of a light table (USFDA, 1998).

Figure II-18. Example of a light table. (Source: Photo courtesy of H. Loechelt-Yoshioka, FDA).

(13) Magnifying Desk Lamp

The device consists of a low power (3-5X) single lens magnifier encased by a fluorescent ring lamp on a flexible arm (Figure II-19). It permits both hands to be free to manipulate sample material.

Figure II-19. Magnifying desk lamp. (Source: Photo courtesy of FDA)

(14) Microscopes

Used to magnify samples to enhance classification of defects. Two basic units are required:

a. Stereomicroscope

Minimum specifications call for: inclined binocular body, with adjustable interpupillary distance, over sliding or revolving, parfocal, achromatic objectives, with a geared prism housing, mounted on a base and capable of illumination by transmitted or reflected light. Eyepieces of 10, 15, or 20X magnification may be paired with objectives ranging from 0.6 to 7.5X. The microscope should be flexible for adaptation to other applications and stable for possible photomicrography (Figure II-20). Protective cover is required. Eyepiece micrometer and hand rests are recommended. Stereomicroscopes can also be fitted with a lightfield/darkfield light base, where either a white background (fully illuminated field of view) or a dark background (with incident light coming in at an angle) is used.

Figure II-20. An example of a stereomicroscope with attached camera indicated by arrow. (Source: Photo courtesy of H. Loechelt-Yoshioka, FDA).

b. Compound Microscope

Minimum specifications: inclined binocular body, with adjustable interpupillary distance, over revolving planachromatic objectives, with a geared prism housing, mounted on a base with built-in illuminator and a centerable iris diaphragm/condenser assembly (Achromatic-aplanatic, N.A. 1.40 or equivalent). Eyepieces of 10, 15, or 20X magnification may be paired with lower objectives of 10X, 40X, or 100X (Figure II-21). A mechanical stage is required, and a color-correcting blue filter is needed. The microscope should be flexible for adaptation to other applications and stable for possible photomicrography. Protective cover is required. Other types of compound microscopes which can be helpful include comparison, polarization and phase-contrast.

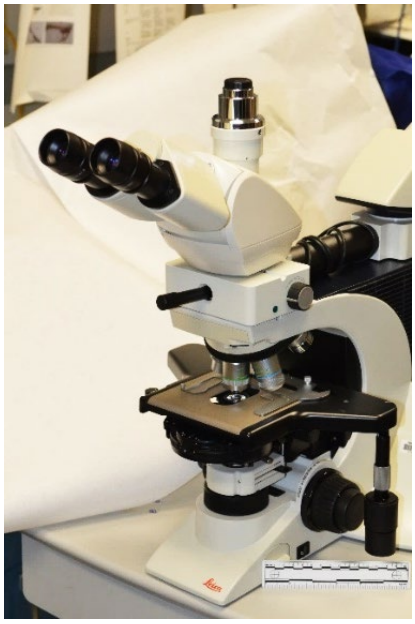

Figure II-21. An example of a compound microscope. (Source: Photo courtesy of H. Loechelt-Yoshioka, FDA).

c. Bench Top Scanning Electron Microscope

Scanning electron microscopy (SEM) (Figure II-22) is used to visualize samples at higher magnifications (10X – 500,000X) and can provide morphological information about a sample. SEMs have applications in particle analysis and detailed micro-structural analysis. Several publications describe the use of electron microscopes in the examination of mites and stored product beetle mandibles, antennae, animal hair and related structures (Ahamad, 2011, Arbogast, 1981, Gabrasanders, 1984, & Zohry, 2017). Tabletop/Benchtop SEMs can be configured to easily image an object *in situ* or without many modifications. This may include using a low vacuum or atmospheric mode to allow for the imaging of biological samples. An Energy Dispersive X-ray Spectrometer (EDS) can be added to an SEM and is used to obtain qualitative elemental information about the sample. In the area of particle analysis, SEM/EDS can help to identify particles using not only their structure and morphological form, but also their elemental composition (USFDA, 2022).

Figure II-22. Bench Top Scanning Electron Microscope (SEM). (Source: Photo courtesy of H. Loechelt-Yoshioka, FDA).

(15) Nutcracker

A tool commonly used to crack in-shell nuts. Nutcrackers can come in many different styles and designs. Even a hammer could be used in place of a nutcracker. (Figure II-23).

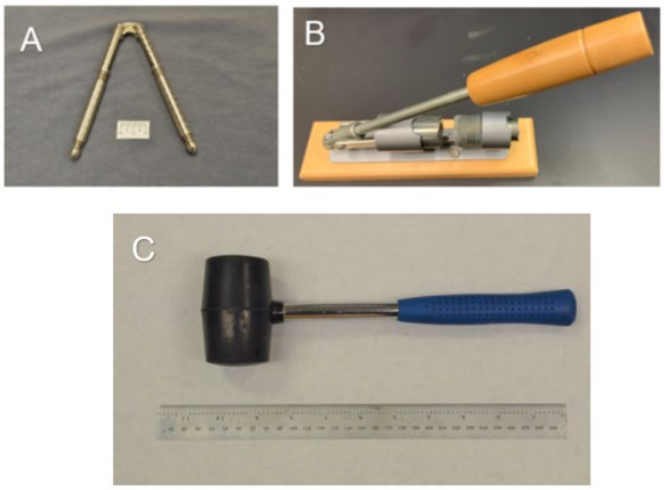

Figure II-23. A. Hand squeeze nutcracker. **B.** Piston action nutcracker. **C**. A hammer. (Source: Photos courtesy of H. Loechelt-Yoshioka & Ibrahim Ali, FDA).

(16) Ocular Loupe

Used to provide low-power magnification. Also called a jeweler's eyepiece. They can come in many different styles (Figure II-24).

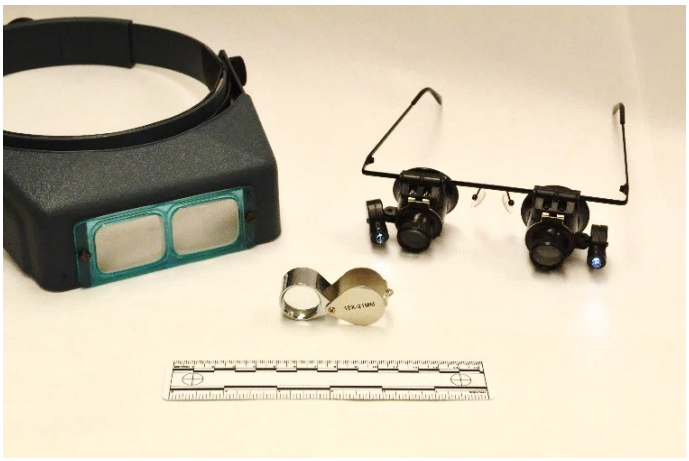

Figure II-24. Various types of ocular loupes. (Source: Photo courtesy of H. Loechelt-Yoshioka, FDA).

(17) Petri dishes

Used to hold filter papers, bolting cloths, etc., for microscopic examination (Figure II-25). Petri dishes 90 mm x 10mm (low-edge type) or 100 mm x 15 mm plastic (disposable) or glass may be used for most applications. Additionally, the smaller 50 mm x 9 mm disposable petri dishes with a tight fit lid are also very useful for holding specimens. Note: glass petri dishes will reduce static electricity issues on dry powdery products.

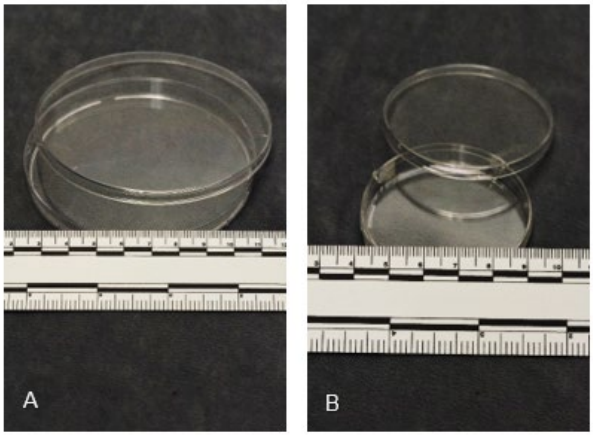

Figure II-25. A. Disposable low edge 90 mm x 10 mm petri dish. **B.** Disposable tight fit lid 50 mm x 9 mm petri dish. (Source: Photos courtesy of H. Loechelt-Yoshioka, FDA).

(18). Picking Tray

The Seedburo picking tray is a useful tool for the examination of grains, pseudograins, peas, beans and similar types of products (Figures II-26, 27). It can speed up the examination process when both sides of the product need to be examined

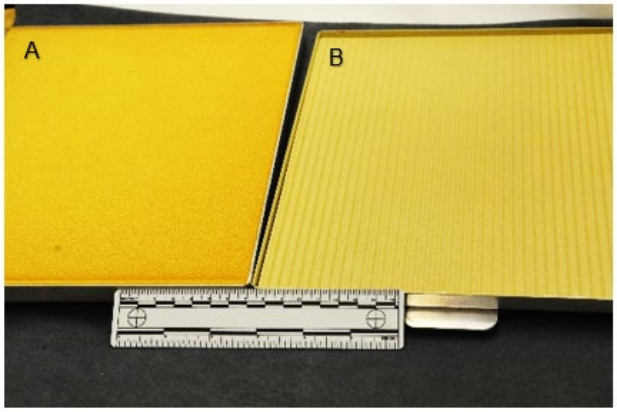

Figure II-26. A. Foam side of a picking tray. **B.** Corrugated side of a picking tray. (Source: Photo courtesy of H. Loechelt-Yoshioka, FDA).

Using a picking tray:

1. Place product on corrugated half of the tray. Shake tray lightly to spread product evenly for easy of examination.
2. Remove damaged and suspect product with forceps.
3. The foam side of the second tray is carefully placed over the product on the corrugated side. Hold both trays tightly and turn them over together, so the corrugated half is now on top.
4. Carefully remove the corrugated half of the tray. Examine product as in step 2.

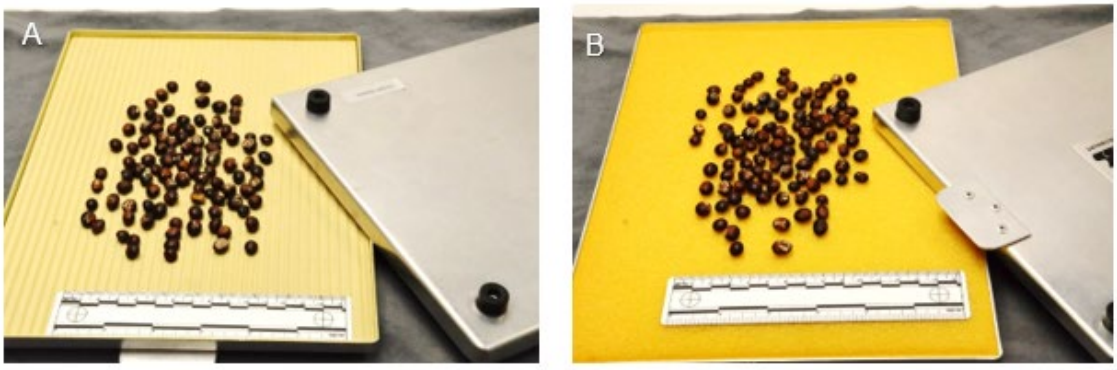

Figure II-27. A. Beans on the corrugated side of a picking tray. **B.** The same beans on the foam side of the picking tray after being inverted. (Source: Photos courtesy of H. Loechelt-Yoshioka, FDA).

(19) Sieves

a. Sieve

Sieve designations, unless otherwise specified, are those described in ASTM E-11 and E-323 (ASTM, 2016). (EverySpec 2021). Sieves of No. 100 or finer should be "plain-weave" stainless steel, since twill weave allows more filaments to pass through. The wires for “plain-weave” have a typical over-under pattern, going over one wire and then under one wire (Figure II-28). Twill weave will go over two wires then under two wires.

The International Standard sieve designations and their corresponding U.S. Standard sieve sizes are given in (Table II-1). Stainless steel or brass frames in standard heights and diameter are available.

Figure II-28. A. Examples of various sieves. **B.** Close-up view of a plain weave sieve (55x). (Source: Photo’s courtesy of H. Loechelt-Yoshioka, FDA).

TABLE II-1 NOMINAL DIMENSIONS OF STANDARD TEST SIEVES (U.S.A. STANDARD SERIES)

Sieve Designation		Nominal Sieve Opening, Inches	Typical Wire Diameter, mm
International Standard[a] (ISO)	U.S.A. Standard		
12.50 mm[b]	1/2 in.[b]	0.5000	2.500
11.20 mm	7/16 in.	0.4380	2.500
9.50 mm	3/8 in.	0.3750	2.240
8.00 mm	5/16 in.	0.3120	2.000
6.70 mm	.265 in.	0.2650	1.800
6.30 mm[b]	1/4 in.[b]	0.2500	1.800
5.60 mm	No. 3 1/2	0.2230	1.600
-	No. 2	0.4460	1.372
4.75 mm	No. 4	0.1870	1.600
4.00 mm	No. 5	0.1570	1.400
3.35 mm	No. 6	0.1320	1.250
2.80 mm	No. 7	0.1100	1.120
2.36 mm	No. 8	0.0937	1.000
2.00 mm	No. 10	0.0787	0.900
1.70 mm	No. 12	0.0661	0.800
1.40 mm	No. 14	0.0555	0.710
1.18 mm	No. 16	0.0469	0.630
1.00 mm	No. 18	0.0394	0.560
850 µm[c]	No. 20	0.0331	0.500
710 µm	No. 25	0.0278	0.450
600 µm	No. 30	0.0234	0.400
500 µm	No. 35	0.0197	0.315
425 µm	No. 40	0.0165	0.280
355 µm	No. 45	0.0139	0.224
300 µm	No. 50	0.0117	0.200
250 µm	No. 60	0.0098	0.160
212 µm	No. 70	0.0083	0.140
180 µm	No. 80	0.0070	0.125
150 µm	No. 100	0.0059	0.100
125 µm	No. 120	0.0049	0.090
106 µm	No. 140	0.0041	0.071
90 µm	No. 170	0.0035	0.063
75 µm	No. 200	0.0029	0.050
63 µm	No. 230	0.0025	0.045
53 µm	No. 270	0.0021	0.036

a. These standard designations correspond to the values for test sieve apertures recommended by the International Organization for Standardization, Geneva, Switzerland.
b. These sieves are not in the standard series, but they have been included because they are in common usage.
c. 1000 µm=1mm

b. Pepper Sieve

The pepper sieve consists of a No. 9-1/2 round screen with a frame 45.72 cm to 55.88 cm in diameter and 7.0 cm in height (Figure II-29). The bottom is a metal sheet perforated with round holes 2.78 mm in diameter with an average of 5-1/2 holes per linear 2.54 cm (small or 'office' size 20.3 cm to 23.0 cm in diameter). U.S. Standard No. 8 sieve (0.0937 in. or 2.38 square mm opening) provide equivalent sieve opening.

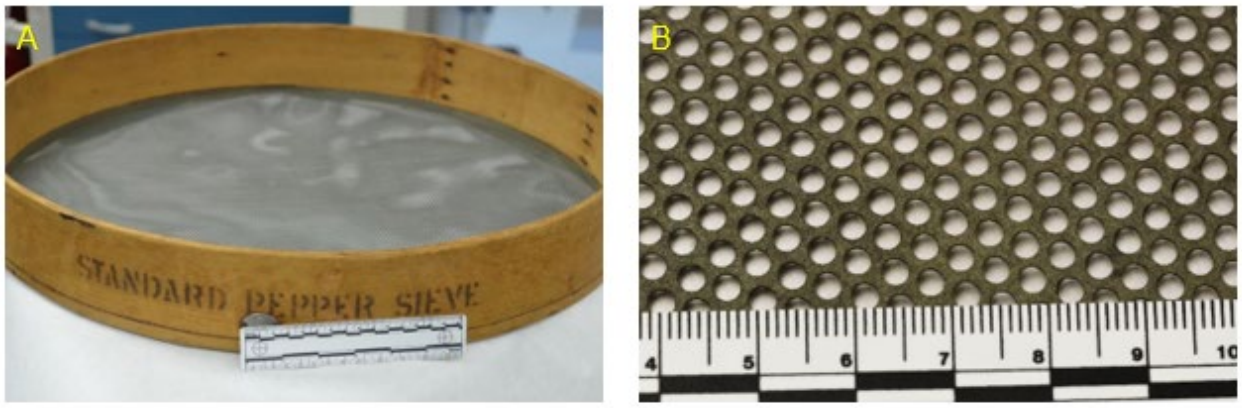

Figure II-29. A. Pepper sieve. **B.** Close-up view of the mesh. (Source: Photos courtesy of H. Loechelt-Yoshioka, FDA).

(20) Tally Counters

Tally counters are a useful apparatus to aid in counting insects and or damage in samples (Figure II-30). They can consist of single key operation to multiple key operation. The two key counter is especially useful in performing Howard mold counts. One key can be used to count fields examined, while the second key can be used to count positive fields. (Microanalytical Items, 1952)

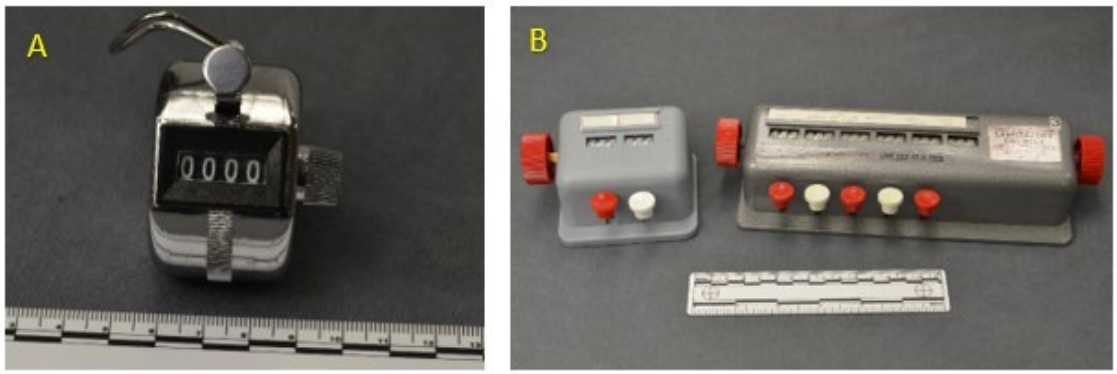

Figure II-30. A. A single key tally counter. **B.** Multiple key tally counters. (Source: Photos courtesy of H. Loechelt-Yoshioka, FDA).

(21) UV Light

The UV light can come in many different shapes and styles (Figure II-31). The UV light is an important tool in the macroanalytical examinations of urine stains, parasites in dark colored fish flesh, or even for bone material. It is important the light source can produce a wavelength of 366 nm.

Figure II-31. Examples of different UV light sources. (Source: Photo courtesy of H. Loechelt-Yoshioka, FDA).

(22) X-Ray Machine

X-ray radiography can be used for rapid examination of grains, coffee beans, spices and legumes for internal insect damage (Boniecki, 2014, Melo, 2010). It can also be used for other types of contamination like metal and bone particles in processed foods. Modern bench top x-ray machines are rapid and relatively easy to use. They do not require radiographic film and developing, like their predecessors (USFDA, 2022).

Figure II-32. Example of a bench top X-ray machine. (Source: Photo courtesy of H. Loechelt-Yoshioka, FDA).

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Revision History

Version No.	Purpose of change	Date
V0	New process	1984
V1	Electronic version	1998
V2	Added images for apparatuses, instructions for Jones Divider and fractional shoveling, additional apparatuses added: high speed blender, cracking board, crystalizing dish, black filter paper, fractional shoveling, lab cyclone, light table, nutcracker, picking tray, pepper sieve, rotary splitter, tally counters, UV light, SEM & X-ray. MPM Council voted to remove the *Patterson-Kelley "Intensifier" Twin-shell V Blender.*	2023