Reducing a gross sample of granular or aggregate material to testing size

1. Scope

1.1 This method describes the selection and preparation of a representative laboratory sample from a gross sample of granular or aggregate material.

1.2 Generally, the procedure for the analysis of the material covers both the selection of the gross sample from the lot or shipment and the preparation of individual test specimens from the laboratory sample. For use when the analysis method does not cover the selection of the gross sample, this document provides general principles for the selection.

2. Significance

The analysis of granular or aggregate material requires the use of a representative sample of the material. Other factors being equal, larger samples will tend to be more representative of the total lot or shipment. Thus a large gross sample may be necessary, especially when the shipment may contain an uneven distribution of impurities, particle sizes, or densities. This method provides for reducing this large sample to a convenient size for conducting the laboratory tests, yet retain the representatives of the large sample.

3. Summary

The particles in the gross sample are reduced, if necessary, and the sample mixed and divided in successive stages by mechanical devices or hand methods until a laboratory sample of the size required for all of the tests is obtained.

4. Apparatus

4.1 For obtaining gross sample if necessary.

4.2 Sample cutter, wooden scraper, or other device for removing a portion of the material from a stopped or moving conveyor belt, and so designed as to avoid separation of various densities and particle sizes.

4.1.2 Bulk sampler¹, commonly known as a grain sampler or thief, consisting of a probe having two close-fitting concentric tubes with open slots along the sides and ending in a cone, with the tubes and openings large enough to accommodate the largest lumps in the material to be sampled. Tube diameter and openings of about 35 mm (1 3/8 in) should be satisfactory for material containing lumps no larger than 19 mm (3/4 in). Samplers of 1.67 m (66 in.),

¹Names of suppliers of testing equipment and materials for this method may be found on the Test Equipment Suppliers list in the bound set of TAPPI Test Methods, or may be available from the TAPPI Technical Services Department.

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1.83 m (72 in.) and 3.05 m (120 in.) in length of the type recommended by the Association of Official Agricultural Chemists are available if desired.

4.1.3 Thief, for removing representative portions from a bag, drum, or other package, such as a smaller version of the sampler described in 4.1.2 or a thin-walled rigid plastic or metal tube, about 150 mm (6 in.) longer than the container to be sampled, and about 38 mm (1.5 in.) in diameter. The tube is cut in half lengthwise from one end to about 150 mm from the other and cut perpendicular at this position to remove one of the long sections. The other long section is ground or filed to a gentle point to permit easy insertion through the container filling valve.

4.1.4 Shovel, scoop, or other device for removing representative portions from a pile or car load of the material. The size of the shovel must be large compared with the lumps in the material being sampled in order to assure the representativeness of the gross sample.

4.2 For reducing gross samples

4.2.1 Rotating or reciprocating sample divider, a mechanical device for automatically separating a portion of a stream of granular material (see Fig. 1).

4.2.2 Riffle sample divider, a manual sample divider (sometimes with motor-driven shaker) for splitting a stream of granular material, usually into two approximately equal portions. Depending on how the material to be sampled flows, riffle divisions should be 1.5 to 3 times the size of the largest particles of the material (Table 1). The slope of the feed shoots and riffles must be at least 60°. An enclosed riffle is preferred (Fig. 2).
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Table 1. Weights of samples with corresponding crushing sizes.

<table>
<thead>
<tr>
<th>Weight of sample to be divided</th>
<th>Minimum size of riffle division</th>
<th>Largest size of particles allowable in sample before division</th>
</tr>
</thead>
<tbody>
<tr>
<td>kg</td>
<td>lb (approx.)</td>
<td>mm</td>
</tr>
<tr>
<td>250 and up 500 and up</td>
<td>50</td>
<td>2</td>
</tr>
<tr>
<td>150-250</td>
<td>250-500</td>
<td>38</td>
</tr>
<tr>
<td>60-120</td>
<td>125-250</td>
<td>25</td>
</tr>
<tr>
<td>30-60</td>
<td>60-125</td>
<td>19</td>
</tr>
<tr>
<td>15-30</td>
<td>30-60</td>
<td>16</td>
</tr>
<tr>
<td>5-15</td>
<td>12-30</td>
<td>12.5</td>
</tr>
<tr>
<td>2-5</td>
<td>5-12</td>
<td>9.5</td>
</tr>
<tr>
<td>g 500-2000</td>
<td>6.3</td>
<td>1/4</td>
</tr>
<tr>
<td>200-500</td>
<td>6.3</td>
<td>1/4</td>
</tr>
<tr>
<td>100-200</td>
<td>6.3</td>
<td>1/4</td>
</tr>
</tbody>
</table>

*Sieve sizes smaller than 0.25 mm (No. 60) are not normally required.

4.2.3 Straight-edge scoop, shovel, or trowel and a hard, clean surface, for halving or quartering a pile of granular material. For some material, or if only a rough surface is available, a canvas or heavy plastic blanket may need to be used in place of or in addition to the hard surface.

4.2.4 Small sampling thief, scoop, or spoon, for sampling from a miniature stockpile of damp fine particles.

4.2.5 Hammer, for breaking large lumps so as to pass a 26.5-mm (1.06-in.) sieve.
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4.2.6 Jaw, cone, or rotary crusher, or other equipment for reducing the approximately 26.5-mm lumps to pass a No. 20 sieve (850 µm).
4.2.7 Pulverizer, hammer mill, or porcelain-jar mill, for reduction of laboratory sample to pass No. 60 (250 µm) sieve.
4.2.7.1 Hammer mill, completely enclosed to avoid loss of dust or moisture.
4.2.7.2 Porcelain-jar ball mill, approximately 230 mm (9.0 in.) in diameter and 250 mm (10.0 in.) in height with smooth, hard, well rounded flint pebbles or equivalent that do not appreciably increase the ash content of the sample.
4.2.8 Mechanical grinder, bucking board, or agate mortar and pestle, for reducing the No. 60 sieve material to pass a No. 100 sieve, or to reduce the small fraction of sample not passing a No. 60 sieve after pulverizing.
4.2.9 Sieves, a set of sieves, with covers and receivers, whose dimensions are in accordance with the current ASTM Specification E11, for the sizes given in Table 1. (The old size sieves may be used until normal replacement).
4.3 Sample containers, non-interactive with sample, for sealing against loss of contents during storage and transport, and against contamination from the air (e.g., dust, carbon dioxide) or moisture pick-up or loss.
4.3.1 Sealable, heavy vapor-impervious bags.
4.3.2 Noncorroding cans, such as those with air-tight friction top or screw top, sealed with a rubber gasket and pressure-sensitive tape.
4.3.3 Glass container, e.g., 175-mL (6-oz) wide-mouthed glass bottle, sealed with rubber gasket or stopper. Requires special care to avoid breakage in transport.

5. Procedure for obtaining gross sample

5.1 Normally, the procedure for obtaining the gross sample to represent the lot or shipment is covered in the analysis method for the specific method being tested. When this is not the case, the following general principles (5.2 and 5.3) and procedures (5.4, 5.5, and 5.6) should be followed in obtaining the gross sample.
5.2 Special handling problems. Determine from the nature of the material whether any special handling problems may be encountered.
5.2.1 Determine needed safety precautions for personnel and equipment in handling caustic or dusty materials during sampling, e.g., protective clothing and breathing devices.
5.2.2 Determine protection requirements for materials that may absorb moisture or other contaminants from the atmosphere or from sampling devices and containers. See 5.6 for special procedure for obtaining a gross sample for moisture content.
5.3 Uneven distribution. Determine from the method of shipment and the nature of the material the likelihood of uneven distribution of impurities, particle sizes, or densities, and plan the time and place of sampling, the total size of the gross sample, and the details of drawing increments of the material to assure that the gross sample will be representative of the lot or shipment; that is, the gross sample will contain the same portions of lumps, fines, and impurities as in the sampled material.
5.3.1 Plan to sample the shipment as soon as possible after receipt (within 24 h) in order to avoid undue exposure to moisture and other contaminants.
5.3.2 Plan to take a gross sample of approximately 200 kg for each 500 metric tons (or 500 lb for each 500 U.S. tons) or fraction thereof, or in the case of larger tonnages, for such tonnage as may be agreed upon. If the original material contains very large lumps (200 mm or 8 in.) or an irregular distribution of impurities, then the gross sample should be two or three times the above size.
5.3.3 Wherever possible, plan to collect the gross sample while the material is being loaded into or unloaded from cars, boats, wagons, or other conveyors, or collect from a conveyor belt or as the material comes from a crusher, but as close to the point of interest as possible. Increments collected from the surface of material in piles, bins, cars, ships, or barges are generally not representative. However, a representative sample may be obtained by discarding the outer surface and securing as nearly as possible the same amount from the top, middle, and bottom (see 5.4.2).
5.3.4 Plan to collect increments regularly, systematically, and with such frequency so that the entire quantity of material sampled will be represented proportionally in the gross sample, and a gross sample of the required amount will be collected (but see 5.4.1).
5.3.5 Nonuniform distribution of particle sizes and impurities may be introduced into a shipment in several ways. Some examples are:
5.3.5.1 A car or truckload of material in bags, drums, or other packages usually contains a number of different manufactured batches or lots.
5.3.5.2 Normal transit vibration may cause particles of different sizes or densities to segregate.
5.3.5.3 Absorbed moisture or other contaminants may cause variations between outer and inner layers or packages of the material.

5.3.5.4 When molten material is packaged in drums and cooled to a solid, impurities may concentrate near bottom or top.

5.3.5.5 The initial discharge from a conveyor may not be representative.

5.4 Use of sampling devices. Use noncontaminating sampling devices and containers (4.3) to select and store a representative gross sample of the material.

5.4.1 If the material passes along a conveyor belt, use the sample cutter or scraper specified in 4.1.1 to periodically cut (scrape) an increment of the material into the container. If the possibility exists that the increment collection may get “in phase” with the sequence of material variability, use random rather than periodic spacing of the increment collection. Close the container between cuts to prevent moisture pickup or loss and to reduce the possibility of contamination. Cut approximately equal increments from 15 to 40 locations, depending on the size of lot. Use a width of cut at least three times the size of the largest particle and verify that all material without regard to particle size or density is scraped into the container.

5.4.2 If the material is in bins or piles, use the bulk sampler specified in 4.1.2 or the device specified in 4.1.4 to cut increments from representative locations of the material.

5.4.2.1 Rotate the inner tube of the bulk sampler in the outer tube until the openings are misaligned, thus forming a closed hollow tube. Insert vertically in the dry bulk material being sampled. When in place, rotate so that openings are aligned, thus permitting the inner tube to be filled. Tap or shake to facilitate filling. Rotate the inner tube to close the openings and then withdraw the sampler. Quickly empty it into the sample container and reclose the container.

5.4.2.2 Alternatively, use a shovel, scoop, or similar device of sufficient size that large particles are not lost in sampling.

5.4.2.3 Withdraw 9 to 40 increments depending on size and anticipate variability of the lot. To sample a carload of material, withdraw 9 increments at the locations shown in Fig. 3.

5.5 Sealing containers. Seal, identify, and protect the container or containers containing the gross sample until ready for preparing the laboratory sample.

5.6 Gross sample for moisture content.

5.6.1 When the moisture content of a shipment or lot is important, any sample taken must be protected against moisture gain or loss and must be representative of the moisture distribution in the shipment. Since much of the moisture tends to be distributed uniformly across the surfaces of granular material, moisture bias will be present if the size distribution of the sample is not the same as the size distribution of the lot sampled.

5.6.2 Special gross sample for moisture content.

5.6.2.1 When an accurate determination of total moisture is required, take a special moisture sample weighing approximately 50 kg (100 lb). Take the moisture sample, when possible, while the material is being loaded or unloaded.
and accumulate, by placing in a water and moisture vapor proof receptacle with a tight fitting lid, small equal parts of the freshly taken increments of the standard gross sample (5.3).

5.6.2.2 Rapidly crush the entire moisture sample, if necessary to pass a No. 4 sieve. Do not sieve the portion to be retained for the moisture sample. Reduce to about 2.5 kg (5 lb), using an enclosed riffle (6.6) or other method that will assure preservation of the moisture content of the sample.

5.6.2.3 Place in a container that is moisture vapor proof, seal, and forward to the laboratory without delay.

5.6.3 Sample for moisture content from entire gross sample.

5.6.3.1 When no special sample for moisture has been taken simultaneously with the gross sample, any sample for moisture content must be taken from the gross sample.

5.6.3.2 Rapidly crush the entire gross sample, if necessary to pass No. 4 sieve. Do not sieve the portion to be retained for the moisture sample, and take any other steps necessary to preserve the moisture content of the gross sample while crushing. Divide out a moisture sample of about 2.5 kg (5 lb) using an enclosed riffle (6.6) or other method that will preserve the moisture content.

5.6.3.3 Place in a container that is moisture vapor proof, seal, and forward to the laboratory without delay.

6. Procedure for reducing gross sample to obtain laboratory sample

6.1 Laboratory sample for moisture content.

6.1.1 The special gross sample for moisture content determination, whenever possible, should be obtained directly at the same time as the gross sample is taken (5.6.2), but, if necessary, from the gross sample (5.6.3), and reduced to approximately 2 kg (5 lb), sealed, and shipped to the laboratory.

6.1.2 Open this sample in the laboratory where further reduction will occur. However, before any further sample reduction operation is performed, weigh the entire laboratory moisture sample on tared pans, spread on the pans and air dry at room temperature, and reweigh, thus determining the present air-dry loss (A) from the “as received” condition to the laboratory condition.

6.1.3 Crush if necessary to pass a No. 20 sieve by passing through rollers or an enclosed grinder. Without sieving, thoroughly mix, repile, and use a spoon, small scoop, or small thief to take at random from the pile a 50-g sample.

6.1.4 In accordance with the analysis method for the material, determine the percent oven-dry loss (B).

6.1.5 Calculate the total moisture in the “as received” sample as follows:

Percent total moisture as received = \( A + B \times \left( \frac{100-A}{100} \right) \)

6.2 Examination of entire gross sample.

6.2.1 Under certain circumstances reduction of the gross sample prior to examination is not recommended, e.g., when chance exclusion or inclusion of only a few relatively large size or “potent” particles, especially of contaminations, may importantly influence interpretation of the characteristics of the gross sample. In such cases, the entire gross sample must be examined.

6.3 Initial steps for reducing gross sample.

6.3.1 Determine from the analysis method(s) for the specific material being tested the total quantity required for the laboratory sample from which the individual test specimens will be drawn.

6.3.2 Place the gross sample (for general testing other than moisture) on a hard, clean surface, free of cracks, or alternatively, when required or permissible by the nature of the material, on a canvas or heavy plastic blanket. Protect from rain, snow, wind, beating sun, and dark of night. Do not let cinders, sand, chippings from the floor, or any other foreign matter get into the sample. Protect the sample from loss or gain of moisture.

6.3.3 Depending on the total weight of the gross sample, break down the larger lumps of the material to the size required by Table 1. Use the hammer (4.2.5) for lumps above 26.5 mm (1.06 in.) or suitable crusher, mill, or other equipment (4.2.6, 4.2.7, 4.2.8) for smaller particles. Thoroughly mix, then divide the sample in accordance with 6.4 to the next weight specified by Table 1 and crush to specified size. Repeat until the desired quantity and fineness have been achieved.

6.4 Nature of material. The method of dividing or splitting the sample depends on the quantity, particle size, and nature of the material:

6.4.1 For coarse material, particle size 26.5 mm (1.06 in.) to 4.75 mm (No. 4 sieve), use (a) a larger riffle (6.6), (b) a rotating or reciprocating sample divider (6.5), (c) the alternate-shovel method above 13.2 mm and the
quartering method 13.2 to 2.36 mm (6.7), or (d) a combination of these methods, depending on the sizes and capacities
of available equipment.

6.4.2 For dry fine material, particle size 2.36 mm (No. 8 sieve) or smaller, use (a) a smaller riffle (6.6), (b) a
rotating or reciprocating sample divider (6.5), (c) the miniature stockpile method (6.9), or (d) a combination of these
methods, depending on the sizes and capacities of available equipment.

6.4.3 For damp fine material, use (a) the miniature stockpile method (6.9), or (b) oven or air-dry, if this will
not affect results, and follow 6.4.2.

6.5 Rotating or reciprocating sample division.
6.5.1 Crush the gross sample in stages and divide to quantities not less than those shown in Table 1.
6.5.2 Using one of the devices shown in Fig. 1, automatically collect at least 60 increments equally distributed
throughout the entire discharge of the sample from the sampler crusher. The collection must be equally distributed
throughout the discharge since crushers can introduce appreciable segregation.

6.5.3 Crush (4.2.6) or pulverize (4.2.7) and divide for each stage. At the final stage before dividing, if any
particles are retained on the screen (No. 60 sieve), use the bucking board or mortar and pestle (4.2.8) to reduce them until
they pass the sieve.

6.6 Riffle sample division.
6.6.1 Determine the number of passes required in the riffling operation from the total weight of the gross
sample and the minimum permissible weight for each size given in Table 1.
6.6.2 Reduce the gross sample to a top size of No. 4 or No. 8 sieve. Crush (4.2.6) or pulverize (4.2.7) and
riffle for each smaller size. For some materials, the No. 20 sieve size may be passed over.
6.6.3 Pass the gross sample through a riffle of appropriate size (4.2.2 and Table 1). Feed from a filling
container consisting of a scoop, bucket, or riffle pan having a lip or opening the full length of the riffle. Spread the
material evenly in the container, raise the container and hold it with its front edge resting on top of the feed chute, then
tilt it so that the material flows in a uniform stream through the chute straight down over the center of the riffle into all
the slots, then into the riffle pans, one half being collected in each pan. Under no circumstances shovel the material
directly into the riffle or dribble it into the riffle from a small-mouthed container. Do not allow the material to build up
in above the riffle slots. If the material does not flow freely through the slots, shake or vibrate the riffle to facilitate even
flow.

6.6.4 Same as 6.5.3
6.7 Alternate-shovel method of sample division.
6.7.1 Carry out the division of the quantity of material on hand as described, even though the initial particle
size is less than indicated in Table 1.
6.7.2 At each stage:
6.7.2.1 Use a straight-edge shovel, scoop, or trowel (4.2.3) to form a conical pile of the crushed material (Fig.
4A) by depositing each shovelful on top of the preceding one.
6.7.2.2 Form into a long pile (Fig. 4B) by taking a shovel from the conical pile and spreading it out in a straight
pile having the width of the shovel and a length of 1.5 to 3 m (5 to 10 ft). Spread the next shovelful directly over the top
of the first but in the opposite direction, and so on back and forth, occasionally flattening the pile, until all of the material
has been formed into one long pile.
6.7.2.3 Beginning on one side of the pile, at either end, and shoveling from the bottom of the pile, take one
shovelful and set it aside (Fig. 4C). Advancing along the side of the pile, take a second shovelful and discard it.
6.7.2.4 In like manner, advancing always in the same direction around the pile, alternatively set aside or discard
shovelfuls, until the long pile is removed and two nearly equal conical piles have been constructed (Fig. 4D), one of
which is to be retained and the other discarded.
6.7.2.5 Crush the retained sample (Fig. 4E) to the next size required by Table 1, and reform into a conical pile
(Fig. 4A).
6.7.3 Continue until an approximately 60-kg (125-lb) sample of 13.2-mm (0.53-in.) particles has been obtained.
Then proceed by the rotating or reciprocating sample divider method of 6.5, the riffling method of 6.6, or the quartering
method of 6.8.
6.8 *Quartering method of sample division.*

6.8.1 *Protection of sample.* For most materials, quartering may be done on a hard, clean surface (6.7.3) or on a canvas or heavy plastic blanket (6.7.4). The latter must be used when the available floor surface is uneven. The main concern is that there be neither loss of material nor the accidental addition of foreign material.

6.8.2 *Hard surface method:*

6.8.2.1 Start with the conical pile formed in the last step of the alternate-shovel method, or if another method has been used for initial reduction of the sample, form a conical pile of the retained material (Fig. 5A).

6.8.2.2 Use a straight-edged shovel, scoop, or trowel (4.2.3) to mix the material thoroughly by turning the entire pile over three times. With the last turning, form a new conical pile (Fig. 5B) by depositing each shovelful on top of the preceding one.

6.8.2.3 Carefully flatten the conical pile to a uniform thickness and diameter by pressing down the apex with a shovel so that each quarter sector of the resulting pile will contain the material originally in it. The diameter should be five to ten times the thickness (Fig. 5C).

6.8.2.4 Divide the flattened mass into four equal quarters with a shovel or trowel (Fig. 5D).

6.8.2.5 Remove two diagonally opposite quarters, including all fine material, and brush the cleared spaces clean (Fig. 5E).

6.8.2.6 Crush the retained sample to the next size required by Table 1, and reform into a conical pile (Fig. 5A).
6.8.2.7 At each stage repeat steps 6.8.2.2 through 6.8.2.6 until the sample is reduced to approximately 5 kg (10 lb) and will pass a No. 4 sieve.

6.8.3 **Canvas or heavy plastic blanket method.**

6.8.3.1 Mix with a shovel as in 6.8.2.2 or by alternately lifting each corner of the blanket and pulling it over the sample toward the diagonally opposite corner causing the material to be rolled (Fig. 6A).

6.8.3.2 Form a cone after mixing (Fig. 6B).

6.8.3.3 Flatten the pile as in 6.8.2.3 (Fig. 6C).

6.8.3.4 Divide the sample as described in 6.8.2.4 (Fig. 6D), or if the surface beneath the blanket is uneven, insert a stick or pipe beneath the blanket and under the center of the pile, then lift both ends of the stick, dividing the sample into two equal parts. Remove the stick leaving a fold of the blanket between the divided portions. Insert the stick under the center of the pile at right angles to the first division and again lift both ends of the stick, dividing the sample into four equal parts.

6.8.3.5 Remove two diagonally opposite piles, being careful to clean the fines from the blanket (Fig. 6E).

6.8.3.6 Crush the retained sample to the next size required by Table 1.

6.8.3.7 At each stage repeat steps 6.8.3.1 through 6.8.3.6 until the sample is reduced to approximately 5 kg (10 lb) and will pass a No. 4 sieve.

6.8.4 If further reduction of the quartered sample is desired, use a small riffle (6.6) or the miniature stockpile method (6.9).

6.9 **Miniature stockpile method of sample division.**

6.9.1 Place the sample on a hard, clean, level surface where there will be neither loss of material nor the accidental addition of foreign material. Mix the material thoroughly by turning the entire sample over three times with a straight-edged scoop, shovel, or trowel (4.2.3).

6.9.2 With the last turning, shovel the entire sample into a conical pile by depositing each shovelful on top of the preceding one. If desired, the conical pile may be flattened to a uniform thickness and diameter by pressing down the apex with a shovel so that each sector of the resulting pile will contain the material originally in it.

6.9.3 Obtain the laboratory sample by selecting at least five increments of material at random locations from the stockpile, using the small sampling thief, scoop, or spoon (4.2.4).

7. **Obtaining test specimens from laboratory sample**

7.1 Thoroughly mix the laboratory sample (for example, following the procedure of 6.9.1 and 6.9.2).

7.2 For each test, withdraw the material for each test specimen at random from the pile, unless the test method specifies another procedure.

8. **Report**

8.1 Report size and source of gross sample.

8.2 Report method or methods of dividing gross sample to obtain laboratory sample.
9. **Accuracy and precision**

9.1 Failure to carefully follow the procedures described herein could result in providing a sample that is biased or excessively variable or both, with subsequent bias and variability in test results.

9.2 A precision statement is not appropriate for this qualitative procedure.

10. **Additional information**

10.1 Effective date of issue: August 10, 1992.


*Your comments and suggestions on this procedure are earnestly requested and should be sent to the TAPPI Technical Divisions Administrator.*