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WI 110809.05

T 257

DRAFT NO. 3

DATE November 1, 2012

WORKING GROUP
CHAIRMAN John Walkinshaw

SUBJECT
CATEGORY Pulp Properties

RELATED
METHODS See "Additional Information"

CAUTION:
This Test Method may include safety precautions which are believed to be appropriate at the time of publication of the method. The intent of these is to alert the user of the method to safety issues related to such use. The user is responsible for determining that the safety precautions are complete and are appropriate to their use of the method, and for ensuring that suitable safety practices have not changed since publication of the method. This method may require the use, disposal, or both, of chemicals which may present serious health hazards to humans. Procedures for the handling of such substances are set forth on Material Safety Data Sheets which must be developed by all manufacturers and importers of potentially hazardous chemicals and maintained by all distributors of potentially hazardous chemicals. Prior to the use of this method, the user must determine whether any of the chemicals to be used or disposed of are potentially hazardous and, if so, must follow strictly the procedures specified by both the manufacturer, as well as local, state, and federal authorities for safe use and disposal of these chemicals.

Sampling and preparing wood for analysis (Proposed revision of T 257 cm-02 as a Standard Practice)

(underscores and strikeouts indicate changes from Draft 2)

1. Scope and summary

1.1 This practice ~~method~~ is applicable to the sampling of wood for all chemical tests.

1.2 The procedures ~~given~~ describe the sampling of wood in all forms, i.e., logs, chips, or sawdust.

1.3 Two sampling plans are described: A probability sampling plan which provides test units from which some property of the wood may be determined within known and controlled limits at a minimum total cost; an economic or engineered sampling plan which minimizes errors due to variations in the raw material or the quality of the lot.

Approved by the Standard Specific Interest Group for this Test Method
TAPPI

2. Significance

2.1 The physical procedures for obtaining the sample vary because of differences in form and in the condition of the wood (in bulk, in piles on the ground or in bins, in transit, in flumes, on conveyor belts, or separated into sublots in trucks, railroad cars, or containers). The various sampling devices used also affect the sampling procedure. The governing principle for obtaining an economical sampling is the use of a random selection procedure for the shipment or lot as a whole or for obtaining subsamples proportionally for subdivisions of the lot.

2.2 A probability sampling plan generally requires an estimate of the standard deviation of the lot, determination of the maximum allowable difference between the estimate of the average quality and the actual value, and selection of a probability factor to give a selected level of confidence to the decision regarding the lot.

2.3 In many commercial situations, there are significant economic consequences depending on the quality of the lot; for example, proper payment may depend upon the precise determination of some property; or in subsequent use of the wood, the acceptability of the final product may be sensitive to variation in the raw material. If the economic penalties of a wrong decision regarding the lot are considered to be significantly large, care should be exercised in selection of the sample according to a probability-based sampling plan. In some circumstances, however, where the economic differences are of minor consequence, and thus the penalty for making an erroneous decision is small, by mutual agreement an empirical or engineered sampling plan may be adopted.

3. Definitions

3.1 The term *random* carries the statistical implication of planned equality in the probability of selecting appropriate samples, not haphazard or subjective selections.

3.2 An *engineered sampling plan* is one using procedures based on a study of the physical aspects of obtaining samples in an unbiased manner and on experimentation to determine how to exercise maximum control over errors in sampling.

3.3 A *probability sampling plan* is one giving procedures for use of the theory of probability to combine a suitable method of selecting the sample with an appropriate method for summarizing the test results so that inferences may be drawn and risks calculated.

3.4 A *homogeneous lot* is one in which all the items are from the same statistical population or from populations with the same mean, standard deviation, and skewness.

4. Apparatus

4.1 *Saw*, power driven, for cutting disks from logs.

NOTE 1: The use of an electric handsaw or band saw is quite appropriate for cutting discs from small diameter logs, i.e., less than 150 to 200 mm. Diameters greater than 200 mm; however, dictate the use of an electric or gas-powered chain saw. These saws are now frequently equipped with an automatic chain lubrication system which must be cleaned up with a suitable solvent before use. The chain should be resharpened frequently during use to minimize heating.

4. 2 *Screen,* 40 mesh (0.40 mm), for sifting finer material.

NOTE 2: A power-driven screen is preferable.

4.3 *Mill*, hand-driven or Wiley type (see 5.4.1)

4.4 *Mason jar*, or similar tight sealing container for keeping sample air-tight.

4 5 *Shovel or scoop*, for use in taking samples of chips.

5. Procedures

CAUTION: If chemical analysis is to be performed on test samples, do not use tools, or grinders, with metal surfaces to prepare the final test specimen for analysis as this could bias the test results.

5.1 *Engineered sampling plan*

5.1.1 Divide the lot into at least two subdivisions (on a rational basis considering its form and condition) and take at least two subsamples from each subdivision.

5.1.2 For logs, a practical way to attain randomness is to subdivide the shipment into approximately equal quantities by carloads, truckloads, or cords, identify each subdivision by number, and select the subdivisions to be included in the sample by the use of a random number table. From each equal subdivision so selected an equal number of logs are to be taken at random during unloading or stacking. The number of logs comprising the complete sample should be sufficient to produce at least twice the amount of sawdust or raspings required for the moisture content test and the chemical analyses desired. The amount of material obtained by sampling should be reduced to the amount necessary for analysis by quartering (see TAPPI T 605 “Reducing a Gross Sample of Granular or Aggregate Material to Testing Size”).

5.1.3 Subsamples of chips may be taken using a shovel or scoop after identifying subdivisions of the lot in the same way as for logs.

5.1.4 An engineered sampling plan requires accepting broad assumptions regarding the relative unimportance of, or the lack of, differences in variation between and within subdivisions of the lot. It avoids questions regarding the limits of uncertainty in the decision made based on the sample, and it provides no real control over that uncertainty. It does not ensure a least-cost balance of sampling against the risk of economic loss due to a bad determination.

5.2 *Probability sampling plan*

5.2.1 If the lot can be assumed to be homogeneous and a prior estimate of the standard deviation of the lot (derived from subsampling) is available, the number of subsamples (n) may be calculated by

$$n = \left[\frac{t\sigma}{E}\right]^2 \qquad (1)$$

where

σ = the prior estimate of the standard deviation of the lot.

E = the maximum allowable difference between the estimate to be made from the sample and the actual value

t = a probability factor to give a selected level of confidence that the difference is greater than E

See any standard statistical text for values of *t*. The value for *t* should be based on the number of degrees of freedom used to calculate the standard deviation. If the standard deviation is based on calculations made on previous samplings of similar materials and the number of degrees of freedom is unknown, for 95/5 confidence use t = 1.96, for 99/5, t = 2.58.

5.2.2 *Example:* Assume that repeated sampling in the past had resulted in a standard deviation of 0.187 in measurements of the property of interest. The number of subsamples required to assure with 95% confidence that the average quality of a shipment lies within limits of ± 0.15 of the mean of the determinations is, from Eq. 1,

$$n = \left[\frac{1.96 \times 0.187}{0.15}\right]^2 = 5.97 \; or \; 6 \; subsamples$$

5.2.3 Within the physical limitations of handling equipment, the subsamples may be selected with the entire lot considered as a whole. An alternative practical way is to make a random selection of subdivisions and, from them, a random selection of subsamples, using always a random number table.

5.2.4 In a lot assumed to be homogeneous, the balance between number of subdivisions to be sampled and the equal number of subsamples from each may be adjusted to the relative ease, or cost, of selecting the subdivision and of selection of the subsamples therefrom. It is essential, however, that selections made at each stage be random.

5.2.5 If it is known that the lot is not homogeneous, it should be subdivided into rational subdivisions such as opposite quarters of a pile, carloads, or cords. The number of subdivisions to be selected for the sample and the equal number of subsamples to be selected from each may be determined according to the following formulas:

Number of subsamples (k) per subdivision:

$$k = \frac{\sigma_W}{\sigma_b}\left[\frac{c_1}{c_2}\right]^{1/2} \qquad (2)$$

Number of subdivisions (n) in sample:

$$n = \frac{N\left(\sigma_W^2 + k\sigma_b^2\right)}{Nk\left(E/t\right)^2 + k\sigma_b^2} \qquad (3)$$

where:

$\sigma_w{}^2$ = variance between subsamples averaged over all subdivision units

$\sigma_b{}^2$ = variance between subdivisions

c_1 = cost of identifying and preparing a subdivision (or selecting and acquiring the unit to sample)

$\cancel{c^2}$ $\underline{c_2}$ = cost of taking a subsample (cost of selecting and measuring)

N = number of subdivisions in lot

E = allowable uncertainty in the sample result

T = probability factor

5.2.6 The total cost of the sample can be represented by

$$c = nc_1 + nkc_2$$

Accordingly, sampling schedules can be set up for any set of conditions for which variances and costs can be determined, to make possible the selection of samples with predetermined precision at minimum cost.

5.2.7 If the standard deviation of a lot is not known, or if the variances between and within subdivisions have not been determined previously, sampling experiments must be performed to provide data for use in subsequent calculations of sample size.

5.3 *Preparation of laboratory sample.* Obtain a composite sample of wood from the selected logs by either of the following procedures:

5.3.1 Use a power-driven, sharp saw having a guide to permit cuts being made across the end of each log just the width of the saw teeth, and fitted with a clean box or other device for collecting all the sawdust without contaminating the sample. With the guide removed from the saw, cut about one-third of the length off one or both ends of each sample log across a portion free from knots or decayed wood.

NOTE 3: If the shipment is to be analyzed for its quantity of decay, for example by TAPPI T 265 "Natural Dirt in Wood Chips," take special care in selecting the sample logs. Take double the number specified above and take cuts across five cross sections of each log at intervals of one-sixth of its length.

Remove any bark from the sawed ends of the logs. Replace the guide. Completely clean the box or collecting device. Take one or more cuts across each of the sawed ends of all the logs and collect all the sawdust.

5.3.2 Using the power-driven saw, cut a sample disk 6.4 to 25 mm (1/4 to 1 in.) thick from each log. Take the disk from a point not nearer the end than one-third the total length of the log. If only a single log is provided for analysis, cut three such disks from the log. Take one from approximately the center of the log and the other two about 152 mm (6 in.) from the ends. Cut all the disks into two semicircles or into four sectors by two cuts at an angle depending upon the amount of sample required. Include opposite equal sectors in the final sample. Separate and discard all bark and knots, decayed portions, compression wood and other abnormalties. From each semicircle reduce equal sectors situated opposite in the log to sawdust by means of the specially equipped power-driven saw described above. Use the portion to be discarded for feeding the selected sector to the saw. Alternatively use a hand rasp to produce a satisfactorily divided sample from the sectors. In this case, take care to keep the teeth of the rasp clean and not to heat the wood unduly by vigorous rasping. In each case reduce a complete sector to sawdust or raspings to ensure maintaining unchanged the correct proportions of sapwood and heartwood.

5.4 *Grinding and screening*

5.4.1 If moist, let the composite sample air-dry thoroughly. Separate the finer material by sifting on a 40-mesh (0.40-mm) screen. Grind the coarser material in a mill of the Wiley type or in a hand-driven grinding mill. In any case, however, avoid using a mill which heats the material appreciably during grinding or which produces many fines.

NOTE 4: The use of a laboratory refiner equipped with a “pyramid” plate is a convenient means of reducing chip samples to a suitable size for Wiley milling.

NOTE 5: The Wiley-type mill is not satisfactory for all “green” hardwoods and it is sometimes necessary to use a hammer-type mill or to prepare shavings of the wood using a rotary-knife cutter. Green hardwood can become contaminated if the mill is mild steel *(1)*, and stainless steel construction is recommended for such wood.

5.4.2 Preferably use a power-driven screen because the fine material may then be more effectively separated. Again separate the finer material by sifting. Discard the portion which does not pass through the screen. Do not regrind any material. Continue grinding and sifting until sufficient material has been prepared or until all the available material has been ground.

5.4.3 Place the entire sample so prepared in an airtight container, e.g., a Mason jar, from which portions may be withdrawn for analysis as desired. It is good practice to expose the prepared sample to average atmospheric conditions (see TAPPI T 402 “Standard Conditioning and Testing Atmospheres for Paper, Board, Pulp Handsheets, and Related Products”) for a period before closure. This is in order to minimize changes in the moisture content of the material during subsequent handling and weighing operations.

6. Precision

Precision is not applicable for Standard Practices

7. Keywords

Wood, Analysis, Sampling, Sample preparation, Chips, Logs, Sawdust

8. Additional information

8.1 Effective date of issue: to be assigned.

8.2 The particle size to which wood should be reduced for the purpose of certain analyses has been the subject of differences of opinion and complete agreement with respect to the most suitable size has not yet been reached.

8.3 Moisture content. If the percentage moisture-free wood in the sample is required, proceed according to TAPPI T 264 “Preparation of Wood for Chemical Analysis.”

8.4 The 2012 ~~This~~ revision differs from the previous version in that equations have been corrected. The 1974 revision recommended not regrinding coarse material. Regrinding can significantly alter the chemical composition of the wood material. This method was first issued as a tentative standard in 1942; an official standard in 1945; was corrected in 1959 and revised in 1974.

8.5 This former Classical Method was reissued in 2012 as a Standard Practice to permit a better fit with the definitions established in the present version of the Test Method Guidelines. ~~This method had been placed in the Classical Method category due to lack of timely review. In 1995-96, the Test Methods Subcommittee of the Quality and Standards Management Committee required all committees to review such methods and either confirm as Classical or revise as Official or Provisional. The responsible committee for this method confirmed that this version is properly classified as a Classical Method, in accordance with the current test method regulations.~~

Literature cited

1. Stewart, C. M., *et al.*, “The Selection and Preparation of a Reproducible Wood Sample for Chemical Studies,” *Australian Pulp and Paper Ind., Tech. Assoc. Proc.* **5**:267, 312 (1951); also abridged as “Species Sampling on a Reproducible Basis,” *Tappi* **35** (4): 129 (1952).

Your comments and suggestions on this procedure are earnestly requested and should be sent to the TAPPI Standards Department. ■

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WI 110809.05

T 257

DRAFT NO. 2

DATE December 9, 2011

WORKING GROUP
CHAIRMAN John Walkinshaw

SUBJECT
CATEGORY Pulp Properties

RELATED
METHODS See "Additional Information"

CAUTION:
This Test Method may include safety precautions which are believed to be appropriate at the time of publication of the method. The intent of these is to alert the user of the method to safety issues related to such use. The user is responsible for determining that the safety precautions are complete and are appropriate to their use of the method, and for ensuring that suitable safety practices have not changed since publication of the method. This method may require the use, disposal, or both, of chemicals which may present serious health hazards to humans. Procedures for the handling of such substances are set forth on Material Safety Data Sheets which must be developed by all manufacturers and importers of potentially hazardous chemicals and maintained by all distributors of potentially hazardous chemicals. Prior to the use of this method, the user must determine whether any of the chemicals to be used or disposed of are potentially hazardous and, if so, must follow strictly the procedures specified by both the manufacturer, as well as local, state, and federal authorities for safe use and disposal of these chemicals.

Sampling and preparing wood for analysis (Proposed revision of T 257 cm-02 as a Standard Practice)

(underscores and strikeouts indicate changes from Draft 1)

1. Scope and summary

1.1 This method is applicable to the sampling of wood for all chemical tests.

1.2 The procedures given describe the sampling of wood in all forms, i.e., logs, chips, or sawdust.

1.3 Two sampling plans are described: A probability sampling plan which provides test units from which some property of the wood may be determined within known and controlled limits at a minimum total cost; an economic or engineered sampling plan which minimizes errors due to variations in the raw material or the quality of the lot.

Approved by the Standard Specific Interest Group for this Test Method
TAPPI

2. Significance

2.1 The physical procedures for obtaining the sample vary because of differences in form and in the condition of the wood (in bulk, in piles on the ground or in bins, in transit, in flumes, on conveyor belts, or separated into sublots in trucks, railroad cars, or containers). The various sampling devices used also affect the sampling procedure. The governing principle for obtaining an economical sampling is the use of a random selection procedure for the shipment or lot as a whole or for obtaining subsamples proportionally for subdivisions of the lot.

2.2 A probability sampling plan generally requires an estimate of the standard deviation of the lot, determination of the maximum allowable difference between the estimate of the average quality and the actual value, and selection of a probability factor to give a selected level of confidence to the decision regarding the lot.

2.3 In many commercial situations, there are significant economic consequences depending on the quality of the lot; for example, proper payment may depend upon the precise determination of some property; or in subsequent use of the wood, the acceptability of the final product may be sensitive to variation in the raw material. If the economic penalties of a wrong decision regarding the lot are considered to be significantly large, care should be exercised in selection of the sample according to a probability-based sampling plan. In some circumstances, however, where the economic differences are of minor consequence, and thus the penalty for making an erroneous decision is small, by mutual agreement an empirical or engineered sampling plan may be adopted.

3. Definitions

3.1 The term *random* carries the statistical implication of planned equality in the probability of selecting appropriate samples, not haphazard or subjective selections.

3.2 An *engineered sampling plan* is one using procedures based on a study of the physical aspects of obtaining samples in an unbiased manner and on experimentation to determine how to exercise maximum control over errors in sampling.

3.3 A *probability sampling plan* is one giving procedures for use of the theory of probability to combine a suitable method of selecting the sample with an appropriate method for summarizing the test results so that inferences may be drawn and risks calculated.

3.4 A *homogeneous lot* is one in which all the items are from the same statistical population or from populations with the same mean, standard deviation, and skewness.

4. Apparatus

4.1 *Saw*, power driven, for cutting disks from logs.

NOTE 1: The use of an electric handsaw or band saw is quite appropriate for cutting discs from small diameter logs, i.e., less than 150 to 200 mm. Diameters greater than 200 mm; however, dictate the use of an electric or gas-powered chain saw. These saws are now frequently equipped with an automatic chain lubrication system which must be cleaned up with a suitable solvent before use. The chain should be resharpened frequently during use to minimize heating.

4. 2 *Screen,* 40 mesh (0.40 mm), for sifting finer material.

NOTE 2: A power-driven screen is preferable.

4.3 *Mill*, hand-driven or Wiley type (see 5.4.1)

4.4 *Mason jar*, or similar tight sealing container, for keeping sample air-tight.

4 5 *Shovel or scoop*, for use in taking samples of chips.

5. Procedures

5.1 *Engineered sampling plan*

5.1.1 Divide the lot into at least two subdivisions (on a rational basis considering its form and condition) and take at least two subsamples from each subdivision.

5.1.2 For logs, a practical way to attain randomness is to subdivide the shipment into approximately equal quantities by carloads, truckloads, or cords, identify each subdivision by number, and select the subdivisions to be included in the sample by the use of a random number table. From each equal subdivision so selected an equal number of logs are to be taken at random during unloading or stacking. The number of logs comprising the complete sample should be sufficient to produce at least twice the amount of sawdust or raspings required for the moisture content test and the chemical analyses desired. The amount of material obtained by sampling should be reduced to the amount necessary for analysis by quartering (see TAPPI T 605 “Reducing a Gross Sample of Granular or Aggregate Material to Testing Size”).

5.1.3 Subsamples of chips may be taken using a shovel or scoop after identifying subdivisions of the lot in the same way as for logs.

5.1.4 An engineered sampling plan requires accepting broad assumptions regarding the relative unimportance of, or the lack of, differences in variation between and within subdivisions of the lot. It avoids questions regarding the limits of uncertainty in the decision made based on the sample, and it provides no real control over that uncertainty. It does not ensure a least-cost balance of sampling against the risk of economic loss due to a bad determination.

5.2 *Probability sampling plan*

5.2.1 If the lot can be assumed to be homogeneous and a prior estimate of the standard deviation of the lot (derived from subsampling) is available, the number of subsamples (n) may be calculated by

$$n = \left[\frac{t\sigma}{E}\right]^2 \qquad (1)$$

where

σ = the prior estimate of the standard deviation of the lot.

E = the maximum allowable difference between the estimate to be made from the sample and the actual value

t = a probability factor to give a selected level of confidence that the difference is greater than E

See any standard statistical text for values of *t*. The value for *t* should be based on the number of degrees of freedom used to calculate the standard deviation. If the standard deviation is based on calculations made on previous samplings of similar materials and the number of degrees of freedom is unknown, for 95/5 confidence use t = 1.96, for 99/5, t = 2.58.

5.2.2 *Example:* Assume that repeated sampling in the past had resulted in a standard deviation of 0.187 in measurements of the property of interest. The number of subsamples required to assure with 95% confidence that the average quality of a shipment lies within limits of ± 0.15 of the mean of the determinations is, from Eq. 1,

$$n = \left[\frac{1.96 \times 0.187}{0.15}\right]^2 = 5.97 \textit{ or } 6 \textit{ subsamples}$$

5.2.3 Within the physical limitations of handling equipment, the subsamples may be selected with the entire lot considered as a whole. An alternative practical way is to make a random selection of subdivisions and, from them, a random selection of subsamples, using always a random number table.

5.2.4 In a lot assumed to be homogeneous, the balance between number of subdivisions to be sampled and the equal number of subsamples from each may be adjusted to the relative ease, or cost, of selecting the subdivision and of selection of the subsamples therefrom. It is essential, however, that selections made at each stage be random.

5.2.5 If it is known that the lot is not homogeneous, it should be subdivided into rational subdivisions such as opposite quarters of a pile, carloads, or cords. The number of subdivisions to be selected for the sample and the equal number of subsamples to be selected from each may be determined according to the following formulas:

Number of subsamples (*k*) per subdivision:

$$k = \frac{\sigma_W}{\sigma_b}\left[\frac{c_1}{c_2}\right]^{1/2} \tag{2}$$

Number of subdivisions (*n*) in sample:

$$n = \frac{N\left(\sigma_W^2 + k\sigma_b^2\right)}{Nk\left(E/t\right)^2 + k\sigma_b^2} \tag{3}$$

where:

${\sigma_w}^2$ = variance between subsamples averaged over all subdivision units

${\sigma_b}^2$ = variance between subdivisions

c_1 = cost of identifying and preparing a subdivision (or selecting and acquiring the unit to sample)

~~c^2~~ $\underline{c_2}$ = cost of taking a subsample (cost of selecting and measuring)

N = number of subdivisions in lot

E = allowable uncertainty in the sample result

T = probability factor

5.2.6 The total cost of the sample can be represented by

$$c = nc_1 + nkc_2$$

Accordingly, sampling schedules can be set up for any set of conditions for which variances and costs can be determined, to make possible the selection of samples with predetermined precision at minimum cost.

5.2.7 If the standard deviation of a lot is not known, or if the variances between and within subdivisions have not been determined previously, sampling experiments must be performed to provide data for use in subsequent calculations of sample size.

5.3 *Preparation of laboratory sample.* Obtain a composite sample of wood from the selected logs by either of the following procedures:

5.3.1 Use a power-driven, sharp saw having a guide to permit cuts being made across the end of each log just the width of the saw teeth, and fitted with a clean box or other device for collecting all the sawdust without contaminating the sample. With the guide removed from the saw, cut about one-third of the length off one or both ends of each sample log across a portion free from knots or decayed wood.

NOTE 3: If the shipment is to be analyzed for its quantity of decay, for example by TAPPI T 265 “Natural Dirt in Wood Chips,” take special care in selecting the sample logs. Take double the number specified above and take cuts across five cross sections of each log at intervals of one-sixth of its length.

Remove any bark from the sawed ends of the logs. Replace the guide. Completely clean the box or collecting device. Take one or more cuts across each of the sawed ends of all the logs and collect all the sawdust.

5.3.2 Using the power-driven saw, cut a sample disk 6.4 to 25 mm (1/4 to 1 in.) thick from each log. Take the disk from a point not nearer the end than one-third the total length of the log. If only a single log is provided for analysis, cut three such disks from the log. Take one from approximately the center of the log and the other two about 152 mm (6 in.) from the ends. Cut all the disks into two semicircles or into four sectors by two cuts at an angle depending upon the amount of sample required. Include opposite equal sectors in the final sample. Separate and discard all bark and knots, decayed portions, compression wood and other abnormalties. From each semicircle reduce equal sectors situated opposite in the log to sawdust by means of the specially equipped power-driven saw described above. Use the portion to be discarded for feeding the selected sector to the saw. Alternatively use a hand rasp to produce a satisfactorily divided sample from the sectors. In this case, take care to keep the teeth of the rasp clean and not to heat the wood unduly by vigorous rasping. In each case reduce a complete sector to sawdust or raspings to ensure maintaining unchanged the correct proportions of sapwood and heartwood.

5.4 *Grinding and screening*

5.4.1 If moist, let the composite sample air-dry thoroughly. Separate the finer material by sifting on a 40-mesh (0.40-mm) screen. Grind the coarser material in a mill of the Wiley type or in a hand-driven grinding mill. In any case, however, avoid using a mill which heats the material appreciably during grinding or which produces many fines.

NOTE 4: The use of a laboratory refiner equipped with a “pyramid” plate is a convenient means of reducing chip samples to a suitable size for Wiley milling.

NOTE 5: The Wiley-type mill is not satisfactory for all “green'“ hardwoods and it is sometimes necessary to use a hammer-type mill or to prepare shavings of the wood using a rotary-knife cutter. Green hardwood can become contaminated if the mill is mild steel *(1)*, and stainless steel construction is recommended for such wood.

5.4.2 Preferably use a power-driven screen because the fine material may then be more effectively separated. Again separate the finer material by sifting. Discard the portion which does not pass through the screen. Do not regrind any material. Continue grinding and sifting until sufficient material has been prepared or until all the available material has been ground.

5.4.3 Place the entire sample so prepared in an airtight container, e.g., a Mason jar, from which portions may be withdrawn for analysis as desired. It is good practice to expose the prepared sample to average atmospheric conditions (see TAPPI T 402 “Standard Conditioning and Testing Atmospheres for Paper, Board, Pulp Handsheets, and Related Products”) for a period before closure. This is in order to minimize changes in the moisture content of the material during subsequent handling and weighing operations.

6. Precision

Precision is not applicable for Standard Practices

7. Keywords

Wood, Analysis, Sampling, Sample preparation, Chips, Logs, Sawdust

~~7.~~ 8. Additional information

8.1 ~~7.1~~ Effective date of issue: to be assigned.

8.2 ~~7.2~~ The particle size to which wood should be reduced for the purpose of certain analyses has been the subject of differences of opinion and complete agreement with respect to the most suitable size has not yet been reached.

8.3 ~~7.3~~ Moisture content. If the percentage moisture-free wood in the sample is required, proceed according to TAPPI T 264 "Preparation of Wood for Chemical Analysis."

8.4 ~~7.4~~ This revision differs from the previous version in that equations have been corrected. The 1974 revision recommended not regrinding coarse material. Regrinding can significantly alter the chemical composition of the wood material. This method was first issued as a tentative standard in 1942; an official standard in 1945; was corrected in 1959 and revised in 1974.

8.5 ~~7.5~~ This method had been placed in the Classical Method category due to lack of timely review. In 1995-96, the Test Methods Subcommittee of the Quality and Standards Management Committee required all committees to review such methods and either confirm as Classical or revise as Official or Provisional. The responsible committee for this method confirmed that this version is properly classified as a Classical Method, in accordance with the current test method regulations.

Literature cited

1. Stewart, C. M., *et al.*, "The Selection and Preparation of a Reproducible Wood Sample for Chemical Studies," *Australian Pulp and Paper Ind., Tech. Assoc. Proc.* **5**:267, 312 (1951); also abridged as "Species Sampling on a Reproducible Basis," *Tappi* **35** (4): 129 (1952).

Your comments and suggestions on this procedure are earnestly requested and should be sent to the TAPPI Standards Department. ■

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WI 110809.05

T 257

DRAFT NO. 1

DATE August 26, 2011

WORKING GROUP
CHAIRMAN to be determined

SUBJECT
CATEGORY Pulp Properties

RELATED
METHODS See "Additional Information"

CAUTION:
This Test Method may include safety precautions which are believed to be appropriate at the time of publication of the method. The intent of these is to alert the user of the method to safety issues related to such use. The user is responsible for determining that the safety precautions are complete and are appropriate to their use of the method, and for ensuring that suitable safety practices have not changed since publication of the method. This method may require the use, disposal, or both, of chemicals which may present serious health hazards to humans. Procedures for the handling of such substances are set forth on Material Safety Data Sheets which must be developed by all manufacturers and importers of potentially hazardous chemicals and maintained by all distributors of potentially hazardous chemicals. Prior to the use of this method, the user must determine whether any of the chemicals to be used or disposed of are potentially hazardous and, if so, must follow strictly the procedures specified by both the manufacturer, as well as local, state, and federal authorities for safe use and disposal of these chemicals.

Sampling and preparing wood for analysis
(Ten-year review of T 257 cm-02)

1. Scope and summary

1.1 This method is applicable to the sampling of wood for all chemical tests.

1.2 The procedures given describe the sampling of wood in all forms, i.e., logs, chips, or sawdust.

1.3 Two sampling plans are described: A probability sampling plan which provides test units from which some property of the wood may be determined within known and controlled limits at a minimum total cost; an economic or engineered sampling plan which minimizes errors due to variations in the raw material or the quality of the lot.

2. Significance

2.1 The physical procedures for obtaining the sample vary because of differences in form and in the condition of the wood (in bulk, in piles on the ground or in bins, in transit, in flumes, on conveyor belts, or separated into sublots in

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trucks, railroad cars, or containers). The various sampling devices used also affect the sampling procedure. The governing principle for obtaining an economical sampling is the use of a random selection procedure for the shipment or lot as a whole or for obtaining subsamples proportionally for subdivisions of the lot.

2.2 A probability sampling plan generally requires an estimate of the standard deviation of the lot, determination of the maximum allowable difference between the estimate of the average quality and the actual value, and selection of a probability factor to give a selected level of confidence to the decision regarding the lot.

2.3 In many commercial situations, there are significant economic consequences depending on the quality of the lot; for example, proper payment may depend upon the precise determination of some property; or in subsequent use of the wood, the acceptability of the final product may be sensitive to variation in the raw material. If the economic penalties of a wrong decision regarding the lot are considered to be significantly large, care should be exercised in selection of the sample according to a probability-based sampling plan. In some circumstances, however, where the economic differences are of minor consequence, and thus the penalty for making an erroneous decision is small, by mutual agreement an empirical or engineered sampling plan may be adopted.

3. Definitions

3.1 The term *random* carries the statistical implication of planned equality in the probability of selecting appropriate samples, not haphazard or subjective selections.

3.2 An *engineered sampling plan* is one using procedures based on a study of the physical aspects of obtaining samples in an unbiased manner and on experimentation to determine how to exercise maximum control over errors in sampling.

3.3 A *probability sampling plan* is one giving procedures for use of the theory of probability to combine a suitable method of selecting the sample with an appropriate method for summarizing the test results so that inferences may be drawn and risks calculated.

3.4 A *homogeneous lot* is one in which all the items are from the same statistical population or from populations with the same mean, standard deviation, and skewness.

4. Apparatus

4.1 *Saw*, power driven, for cutting disks from logs.

NOTE 1: The use of an electric handsaw or band saw is quite appropriate for cutting discs from small diameter logs, i.e., less than 150 to 200 mm. Diameters greater than 200 mm; however, dictate the use of an electric or gas-powered chain saw. These saws are now frequently equipped with an automatic chain lubrication system which must be cleaned up with a suitable solvent before use. The chain should be resharpened frequently during use to minimize heating.

4. 2 *Screen,* 40 mesh (0.40 mm), for sifting finer material.

NOTE 2: A power-driven screen is preferable.

4.3 *Mill*, hand-driven or Wiley type (see 5.4.1)

4.4 Mason jar, for keeping sample air-tight.

4 5 *Shovel or scoop*, for use in taking samples of chips.

5. Procedures

5.1 *Engineered sampling plan*

5.1.1 Divide the lot into at least two subdivisions (on a rational basis considering its form and condition) and take at least two subsamples from each subdivision.

5.1.2 For logs, a practical way to attain randomness is to subdivide the shipment into approximately equal quantities by carloads, truckloads, or cords, identify each subdivision by number, and select the subdivisions to be included in the sample by the use of a random number table. From each equal subdivision so selected an equal number of logs are to be taken at random during unloading or stacking. The number of logs comprising the complete sample should be sufficient to produce at least twice the amount of sawdust or raspings required for the moisture content test and the chemical analyses desired. The amount of material obtained by sampling should be reduced to the amount necessary for analysis by quartering (see TAPPI T 605 “Reducing a Gross Sample of Granular or Aggregate Material to Testing Size”).

5.1.3 Subsamples of chips may be taken using a shovel or scoop after identifying subdivisions of the lot in the same way as for logs.

5.1.4 An engineered sampling plan requires accepting broad assumptions regarding the relative unimportance of, or the lack of, differences in variation between and within subdivisions of the lot. It avoids questions regarding the limits of uncertainty in the decision made based on the sample, and it provides no real control over that uncertainty. It does not ensure a least-cost balance of sampling against the risk of economic loss due to a bad determination.

5.2 *Probability sampling plan*

5.2.1 If the lot can be assumed to be homogeneous and a prior estimate of the standard deviation of the lot (derived from subsampling) is available, the number of subsamples (*n*) may be calculated by

$$n = \left[\frac{t\sigma}{E}\right]^2 \qquad (1)$$

where

σ = the prior estimate of the standard deviation of the lot.

E = the maximum allowable difference between the estimate to be made from the sample and the actual value

t = a probability factor to give a selected level of confidence that the difference is greater than E

See any standard statistical text for values of t. The value for t should be based on the number of degrees of freedom used to calculate the standard deviation. If the standard deviation is based on calculations made on previous samplings of similar materials and the number of degrees of freedom is unknown, for 95/5 confidence use $t = 1.96$, for 99/5, $t = 2.58$.

5.2.2 *Example:* Assume that repeated sampling in the past had resulted in a standard deviation of 0.187 in measurements of the property of interest. The number of subsamples required to assure with 95% confidence that the average quality of a shipment lies within limits of ± 0.15 of the mean of the determinations is, from Eq. 1,

$$n = \left[\frac{1.96 \times 0.187}{0.15}\right]^2 = 5.97 \ or\ 6 \ subsamples$$

5.2.3 Within the physical limitations of handling equipment, the subsamples may be selected with the entire lot considered as a whole. An alternative practical way is to make a random selection of subdivisions and, from them, a random selection of subsamples, using always a random number table.

5.2.4 In a lot assumed to be homogeneous, the balance between number of subdivisions to be sampled and the equal number of subsamples from each may be adjusted to the relative ease, or cost, of selecting the subdivision and of selection of the subsamples therefrom. It is essential, however, that selections made at each stage be random.

5.2.5 If it is known that the lot is not homogeneous, it should be subdivided into rational subdivisions such as opposite quarters of a pile, carloads, or cords. The number of subdivisions to be selected for the sample and the equal number of subsamples to be selected from each may be determined according to the following formulas:

Number of subsamples (k) per subdivision:

$$k = \frac{\sigma_W}{\sigma_b}\left[\frac{c_1}{c_2}\right]^{1/2} \tag{2}$$

Number of subdivisions (n) in sample:

$$n = \frac{N\left(\sigma_W^2 + k\sigma_b^2\right)}{Nk\left(E/t\right)^2 + k\sigma_b^2} \tag{3}$$

where:

σ_w^2 = variance between subsamples averaged over all subdivision units

σ_b^2 = variance between subdivisions

c_1 = cost of identifying and preparing a subdivision (or selecting and acquiring the unit to sample)

c^2 = cost of taking a subsample (cost of selecting and measuring)

N = number of subdivisions in lot

E = allowable uncertainty in the sample result

T = probability factor

5.2.6 The total cost of the sample can be represented by

$$c = nc_1 + nkc_2$$

Accordingly, sampling schedules can be set up for any set of conditions for which variances and costs can be determined, to make possible the selection of samples with predetermined precision at minimum cost.

5.2.7 If the standard deviation of a lot is not known, or if the variances between and within subdivisions have not been determined previously, sampling experiments must be performed to provide data for use in subsequent calculations of sample size.

5.3 *Preparation of laboratory sample.* Obtain a composite sample of wood from the selected logs by either of the following procedures:

5.3.1 Use a power-driven, sharp saw having a guide to permit cuts being made across the end of each log just the width of the saw teeth, and fitted with a clean box or other device for collecting all the sawdust without contaminating the sample. With the guide removed from the saw, cut about one-third of the length off one or both ends of each sample log across a portion free from knots or decayed wood.

NOTE 3: If the shipment is to be analyzed for its quantity of decay, for example by TAPPI T 265 “Natural Dirt in Wood Chips,” take special care in selecting the sample logs. Take double the number specified above and take cuts across five cross sections of each log at intervals of one-sixth of its length.

Remove any bark from the sawed ends of the logs. Replace the guide. Completely clean the box or collecting device. Take one or more cuts across each of the sawed ends of all the logs and collect all the sawdust.

5.3.2 Using the power-driven saw, cut a sample disk 6.4 to 25 mm (1/4 to 1 in.) thick from each log. Take the disk from a point not nearer the end than one-third the total length of the log. If only a single log is provided for analysis, cut three such disks from the log. Take one from approximately the center of the log and the other two about 152 mm (6 in.) from the ends. Cut all the disks into two semicircles or into four sectors by two cuts at an angle depending upon the amount of sample required. Include opposite equal sectors in the final sample. Separate and discard all bark and knots, decayed portions, compression wood and other abnormalties. From each semicircle reduce equal sectors situated opposite in the log to sawdust by means of the specially equipped power-driven saw described above. Use the portion to be discarded for feeding the selected sector to the saw. Alternatively use a hand rasp to produce a satisfactorily divided sample from the sectors. In this case, take care to keep the teeth of the rasp clean and not to heat the wood unduly by vigorous rasping. In each case reduce a complete sector to sawdust or raspings to ensure maintaining unchanged the correct proportions of sapwood and heartwood.

5.4 *Grinding and screening*

5.4.1 If moist, let the composite sample air-dry thoroughly. Separate the finer material by sifting on a 40-mesh (0.40-mm) screen. Grind the coarser material in a mill of the Wiley type or in a hand-driven grinding mill. In any case, however, avoid using a mill which heats the material appreciably during grinding or which produces many fines.

NOTE 4: The use of a laboratory refiner equipped with a "pyramid" plate is a convenient means of reducing chip samples to a suitable size for Wiley milling.

NOTE 5: The Wiley-type mill is not satisfactory for all "green'" hardwoods and it is sometimes necessary to use a hammer-type mill or to prepare shavings of the wood using a rotary-knife cutter. Green hardwood can become contaminated if the mill is mild steel *(1)*, and stainless steel construction is recommended for such wood.

5.4.2 Preferably use a power-driven screen because the fine material may then be more effectively separated. Again separate the finer material by sifting. Discard the portion which does not pass through the screen. Do not regrind any material. Continue grinding and sifting until sufficient material has been prepared or until all the available material has been ground.

5.4.3 Place the entire sample so prepared in an airtight container, e.g., a Mason jar, from which portions may be withdrawn for analysis as desired. It is good practice to expose the prepared sample to average atmospheric conditions (see TAPPI T 402 "Standard Conditioning and Testing Atmospheres for Paper, Board, Pulp Handsheets, and Related Products") for a period before closure. This is in order to minimize changes in the moisture content of the material during subsequent handling and weighing operations.

6. Keywords

Wood, Analysis, Sampling, Sample preparation, Chips, Logs, Sawdust

7. Additional information

7.1 Effective date of issue: to be assigned.

7.2 The particle size to which wood should be reduced for the purpose of certain analyses has been the subject of differences of opinion and complete agreement with respect to the most suitable size has not yet been reached.

7.3 Moisture content. If the percentage moisture-free wood in the sample is required, proceed according to TAPPI T 264 "Preparation of Wood for Chemical Analysis."

7.4 This revision differs from the previous version in that equations have been corrected. The 1974 revision recommended not regrinding coarse material. Regrinding can significantly alter the chemical composition of the wood material. This method was first issued as a tentative standard in 1942; an official standard in 1945; was corrected in 1959 and revised in 1974.

7.5 This method had been placed in the Classical Method category due to lack of timely review. In 1995-96, the Test Methods Subcommittee of the Quality and Standards Management Committee required all committees to review such methods and either confirm as Classical or revise as Official or Provisional. The responsible committee for this method confirmed that this version is properly classified as a Classical Method, in accordance with the current test method regulations.

Literature cited

1. Stewart, C. M., *et al.*, "The Selection and Preparation of a Reproducible Wood Sample for Chemical Studies," *Australian Pulp and Paper Ind., Tech. Assoc. Proc.* **5**:267, 312 (1951); also abridged as "Species Sampling on a Reproducible Basis," *Tappi* **35** (4): 129 (1952).

Your comments and suggestions on this procedure are earnestly requested and should be sent to the TAPPI Standards Department. ■