

SAMPLING OF FIBER RAW MATERIAL

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ABSTRACT

A mill quality control program that includes fiber raw material in addition to other raw materials, in-process pulp and the finished product must pay particular attention to the sampling of the chips of all types, sawdust, shavings and wood waste fuel. With the possible exception of digester blow-line samples, the sampling of fiber raw material is the most difficult in the mill and if samples are not representative, testing should not even be attempted. Biased test results due to poor sampling can lead to wrong operating and economic decisions. The most common sources of sampling error are: a) incorrect sampling location; b) wrong sampling frequency; c) wrong sample size; d) lack of operator training in manual sampling techniques; e) poorly designed automatic sampling devices; f) sample handling techniques that allow drying or material loss, and; g) inappropriate use of compositing. This paper discusses statistical design of sample collection, sampling technique for specific mill equipment situations (chippers, screens, storage areas, conveyor belts, etc.), use of automated sampling devices, sample handling and interpretation of test results for day-to-day woodyard operation.

Almost every pulp mill tests the chips that are received from sawmills, veneer plants, other wood products mills satellite chip mills and on-site woodyards and woodrooms. The extent, intensity and rigor of this testing depends on how convinced the mill management is that the pulping operation, economics and product quality are influenced by chip quality. No matter what amount of testing and data analysis is done at a mill, there must be careful attention paid to the sampling process and preparation of samples for testing. If samples are not representative, there is no point in testing them and risking wrong decisions about chip sourcing, processing and mill operations on incorrect data. As obvious as this might seem, observations of chip sampling at a majority of mills would indicate much room for improvement. One major source of this problem is that sampling is not an integral part of the overall quality control program, but often a separate activity done by relatively untrained operators in the woodyard or at worst by truck drivers who are only told to fill a bag with chips when they dump the truck. To correct this, a well-designed sampling program will include the following elements:

- Statistical analysis to determine sampling frequency.
- Assessment or reassessment of quantities of chips needed to meet all test needs.
- Selection and design of sampling locations to allow representative and safe sampling.
- Design, installation, operator training and start-up of manual and, where justified, automatic sampling devices.

- Determination of advisability of compositing on a source-by-source and stream-by-stream basis.
- Follow-up of sampling results with the testing program on a periodic basis and, if needed, revision of methods and retraining of personnel.

These components of a sampling program will be covered in the rest of this paper, drawing much from the extensive experience in the ore, coal and mining industry.

SAMPLING FREQUENCY

Determining the number of samples that need to be taken from a population of chips is a statistical problem for which there are numerous techniques. The key issues that must be addressed are the variability of the properties to be measured (more variability means more samples) and the level of precision required (higher precision requires more samples).

An estimate of the variability can either be determined experimentally or implied from the experience of other mills. The most commonly used measures of variability are the standard deviation and the confidence interval. Figure 1 shows a plot of the results of sampling a chip belt for chip size with the confidence intervals indicated. This type of data is then analyzed by well-established statistical analysis methods to determine sampling frequency. Discussion of these methods can be found in numerous practical articles (1-13). Hatton and Hejas applied these methods to purchased chip evaluation and from their work, Figure 2 shows the interaction of sample frequency and confidence intervals at the 95% confidence level, a level commonly used in quality control programs (13). Note that tests which are detecting relatively small amounts of contaminants or particle types require larger sampling frequency, especially bark. Also, although relatively few samples are needed to assess the amount of accepts in a chip size distribution, the sampling frequency for chip size tests is determined by the oversize and small chips which require more samples to reach guarantee 95% confidence interval.

A common error in chip quality programs is the use of single railcar or truckload data to evaluate suppliers and to correct quality problems. Hatton and Hejas also looked at this problem and concluded that while hundreds of samples would have to be taken from a single railcar or truckload to be within 5-10 percent of the mean for many key chip quality parameters (see Figure 2), a mill can devise an acceptable sampling plan that spot checks chip quality on a number of cars delivered to the mill from a supplier and still provides data with a confidence interval reasonably close to the desired values for the key chip quality factors. Table 1 shows the tolerable errors of estimate for a comprehensive list of chip quality factors (13) and Tables 2 and 3 show how Weyerhaeuser has applied this type of data at both the 90 and 95% confidence intervals.

Using the above approach, a sampling frequency plan was developed for all Weyerhaeuser pulp mills using guidelines shown in Table 4. For a large mill that receives chips from outside suppliers and from an internal woodroom, the sampling plan in Table 5 was developed. The decision on the number of samples was not simply a statistical decision. The economics of testing also entered into the decision as did the use of the data. It is not necessary to have information on each railcar or truck load, so the

samples are distributed randomly across the deliveries. Note that all the loads are sampled for vendors who send only a few loads to the mill.

Not every property that will be tested in a sample has the same variability, so after each has been analyzed, some judgment will have to be exercised to arrive at a compromise frequency. Also, the sample frequency determined by statistical analysis may have to be altered due to the current economics of sampling. If people must do all the sampling, then automatic sampling equipment may remedy the problem. The equipment alternatives will be discussed in a later section. Compositing of samples leads to fewer tests, but statistical information will be lost, as will be also discussed later. However, the basis for the determination of sampling frequency must always be statistical analysis of source variability and an agreement on the precision required in the test data to make quality control and mill operation decisions.

SAMPLING LOCATION

Selection of the sampling location is one of the most important factors in obtaining representative samples. It is not possible to remove representative samples from the back of a chip truck after the door has been opened, from the top of a moving conveyor belt, from the chips just blown onto the top of a chip pile by a pneumatic conveyor or from a railcar as it is being dumped into a roll-over dump pit. In each of these cases, stratification of particle sizes during movement or being representative of a small fraction of the load prevents the use of the data from these samples.

The results of a set of experiments on sampling from conveyor belts illustrates the bias that can occur. It was found that chip samples taken from the top of a conveyor belt contained 12 to 33% less fines than the total cross-section (14). Also, it was found that the fines shifted to the lower part of the belt as the chips fell onto the belt and that once the chip mass comes to rest on the belt, conveying does not appreciably shift the fines further. There was also considerable bias in chip sizes from one side of the belt to the other. In another experiment, it was found that samples from a railcar rollover dump caught by a sampling device were biased compared to two sets of samples taken from the same load at separate belt transfer points. As shown in Table 6, small fractions were overestimated and large fractions underestimated (15).

Based upon mill experience, the following recommendations are made for sampling chip streams or delivery units:

Belt Conveyors - Transfer points between conveyors allow the removal of representative samples if the sampling device can pass through the flow and collect a complete cross-section. This applies to hand and automatic sampling. It is also possible to install mixing vanes in a tall (at least 10 feet) transfer point and then design a sample point at the bottom of the transfer point. The design of such a system must be checked by comparison testing and adjustments in the vanes and sampling point will be needed before the samples are considered representative.

Pneumatic Conveyors - It is not recommended that samples be removed from the blowline, but rather should be taken as the chips fall into the blower feeder before conveying.

Chippers - A method has been patented for removing chips from the discharge side of a chipper allowing chipper performance to be assessed (19). Devices have been constructed to catch chips produced by a portable chipper since due to stratification, it is not possible to obtain a sample from the loaded van as it is delivered. However, if it is not necessary to remove the sample immediately after chipping, going to a conveyor transfer point or other sampling point after the chipper may be more practical and cheaper.

Chip Screens - There are usually several screens operating in parallel in a woodyard and unless the performance of individual screen units is being assessed, a sample of screened chips should be taken at a belt transfer point after the screens so that the performance of all the screens contributes to the test data.

Chip Silos and Bins - The same principle applies to these storage vessels as did with screens.

If samples are taken by hand, the safety of the technician taking the samples must be an important consideration in the location and design of sampling locations. Moving conveyor belts, conveyor drives, moving mobile equipment and other hazards must be examined, protective devices installed where possible and the technicians trained to work in and around these inherently hazardous areas.

SAMPLING DEVICES

A sampling device must be capable of removing a representative sample from a chip flow without creating biased results. This criteria is one of the most difficult to meet in developing a sampling program since the number of variables that must be considered is large. The following must be considered:

1. Range of particle sizes.
2. Shape of the sampling device.
3. Dimensions of the sampling device in relation to largest particle size and amount of sample required.
4. Speed and direction of chip flow in relation to the sampling device.
5. Location of the chip flow - free falling, on a belt, in a silo or pile, etc.

Scoops for manual sampling should not be made from convenient materials such as a cut-up bleach bottle or a sauce pan wired onto a broom handle. The ore and coal industry has studied scoop design and developed a recommendation depending on the maximum particle size to be sampled (3). Figure 3 shows the scoop shape and dimensions in relation to the maximum particle size in the material flow. For chips, the larger scoop sizes are needed to be sure the oversize material are captured and depending on chip quality, even larger sizes than shown may be needed. Smaller scoops could be used for sawdust, shavings or fine hogged fuel, but it is not necessary to build a smaller scoop just for this material if one for chips is already available.

The same sizing principle applies to the design of scoops, and flow diversion devices used in automatic samplers. The results of an experiment in which several sizes of horizontal flow diverters were placed into a chip flow at a

transfer point between conveyors are shown in Figure 4. While the 127 mm wide diverter plugged immediately, the 152 mm plugged after the chip flow was approximately doubled. If put into the flow at an angle, a 100 mm diverter could be used without plugging, but the angle is also critical. Such experiments should be conducted whenever an automatic sampler is being designed. Figure 5 shows their performance. It must be again emphasized that after an automatic sampler has been designed and installed, cross-checking with manual sampling and statistical analysis of the results must be done to be sure the device is not creating biased results. Again, the ore, coal and grain industries have provided a sound basis to begin the design of samplers specifically for chips (17, 18). There are unique sampler designs for special situations. LaPointe has patented a device for removing chip samples from a chipper discharge by deflecting a portion of the flow (19). This is useful for evaluating individual chipper performance. White developed an auger sampler for removing fuel samples from the interior of a hogged fuel pile (20). This allows assessment of deterioration inside the pile without extensive digging.

SAMPLE SIZE

Sample size is one of the last decisions that is made in the design of the sampling system. Since some tests require dry material (particle size, bark content and grit content, for example) and some, wet chips (rot content, moisture content and bulk density, for example), enough chips must be collected to meet the needs of all the tests. This can be done by either taking large enough initial samples or by compositing smaller samples where compositing makes practical and statistical sense. Table 7 lists example sample sizes required for various tests using Weyerhaeuser chip testing methods. Using these data, a complete analysis would require at least 20-25 dry pounds of chips.

COMPOSITING

Compositing can be defined as mechanical averaging. If a group of primary samples were thoroughly blended and a representative test or secondary sample drawn, the resulting test value should be equal to the average of all the samples tested individually. However, this process does not yield any statistical information about variability in the samples. In spite of this significant drawback, there are some good reasons for compositing depending on the use of the data. If the source being sampled has known or predictable variability, compositing may be a useful way to reduce the testing load and still monitor the general trend of the test data. If decisions on the data will be based over fairly long time periods and variability is not an issue, compositing may be a useful tool. Some tests are very expensive (such as extractive analysis and grit content) and compositing may allow development of longer term trends in situations where some data is better than no data at all.

If compositing is done, the methods must be carefully developed and technicians thoroughly trained. Done properly, it is possible to composite up to 200 pounds of chips using a broad, flat shovel to blend and divide the chips on a smooth concrete floor. Figure 6 shows such a technique (3). Mechanical blenders have also been developed to composite samples. Modified cement mixers are used in some labs in addition to specifically designed blenders such as the one shown in Figure 7. Because of

the tendency for fines to settle to the lower portion of a mass of chips when agitated, compositing must be done carefully to avoid erroneous particle size data. Once blended, care must be taken in secondary sampling not to disturb the chip mass and cause migration of the fine particles.

SAMPLING OF WOOD WASTE FUEL

Most mills are now using wood waste fuel to meet the mill's energy demands. The quality of fuel has a significant impact on boiler performance and emissions. The same criteria for sampling chips apply to fuel, but because of greater variability, a greater sampling frequency is needed. Table 8 shows the range of variability for various wood waste fuel characteristics and estimates of precision at the 95% confidence level for a range of sample sizes. Compared to chips, from 2 to 100 times the samples are required to obtain comparable precision in testing wood waste fuel. The highest number is needed for bulk density which is greatly influenced by particle size, one of the most variable characteristics of wood waste fuel. Fortunately, bulk density is not as critical in boiler operation as it is in digesters.

FOLLOW-UP OF RESULTS

All the components of quality monitoring programs must be periodically audited to be sure they are performing as originally designed. The areas of a sampling program that need to be routinely checked are:

- Sample frequency of outside suppliers compared to the number of deliveries being made.
- The variability of supplier quality vs. the frequency of sampling.
- The opportunity to composite samples where variability is low and the statistical data lost in compositing is not critical.
- Performance of technicians in taking manual samples to be sure samples are being removed across the full width and depth of the chip flow, the number of sub-samples is understood for each flow and supplier, and that they understand the importance of the sampling process in the overall quality testing program.

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	Tolerable Errors of Estimate (13)
Oven Dry Solids Content	±2%
Packing Density	±0.2 lb/ft ³
Overlarge Chips	±0.5%
Overthick Chips	±0.5%
Accept Chips	±1.0%
Pin Chips	±0.6%
Fines	±0.3%
True Overs	±0.5%
Acceptable Chips	±1.0%
Bark	±0.5%
Rot	±0.3%
Knots	±0.3%

Table 1
Tolerable Errors of Estimate for Chip Quality Variables

Characteristics	Error of Estimate*										Tolerable Error of Estimate (13)
	3	5	8	10	12	15	20	40	50	100	
% OD Solids	6	3	2	1.5	1.5	1.5	1	0.8	0.7	0.5	2
Packing Density OD lb/ft ³	0.6	0.3	0.2	0.2	0.2	0.1	0.1	0.1	0.1	0.1	0.2
Overlarge Chips % (45 mm)	2	1	0.7	0.6	0.5	0.5	0.4	0.3	0.2	0.2	0.5
Overthick Chips % (10 or 8 mm)	4	2	1.5	1	1	0.9	0.8	0.5	0.5	0.3	0.5
Accepts %	8	4	2.5	2.5	1	1	1.5	1	1	0.7	1.0
Pin Chips % (-7, +3 mm)	4	2	1.5	1	1	0.9	0.8	0.5	0.5	0.3	0.6
Fines % (-3 mm)	1	0.5	0.3	0.3	0.3	0.2	0.2	0.1	0.1	0.1	0.3
Bark %	1	0.5	0.3	0.3	0.3	0.2	0.2	0.1	0.1	0.1	0.5

*There is only one chance in 10 that the calculated mean value is in error more than + or - the amount shown.

Table 2
Errors of Estimate for Given Sample Sizes
90% Confidence Level

Characteristics	Error of Estimate*											Tolerable Error of Estimate (13)
	Number of Samples, Individual or in Composites											
	3	5	8	10	12	15	20	40	50	100	Estimate	(13)
% OD Solids	7	4	2.5	2	2	1.5	1.5	1	0.8	0.5		2
Packing Density OD lb/ft ³	0.7	0.4	0.3	0.2	0.2	0.2	0.1	0.1	0.1	0.1		0.2
Overlarge Chips % (45 mm)	2.5	1	0.8	0.7	0.6	0.6	0.5	0.3	0.3	0.2		0.5
Overtick Chips % (10 or 8 mm)	5	2.5	1.5	1.5	1.5	1	1	0.6	0.6	0.3		0.5
Accepts %	10	5	3.5	3	2.5	2	2	1.5	1	0.7		1.0
Pin Chips % (-7, +3 mm)	5	2.5	2	1.5	1	1	1	0.6	0.6	0.3		0.6
Fines % (-3 mm)	1	0.6	0.4	0.4	0.3	0.3	0.2	0.2	0.1	0.1		0.3
Bark %	1	0.6	0.4	0.4	0.3	0.3	0.2	0.2	0.1	0.1		0.5

* There is only one chance in 20 that the calculated mean value is in error more than + or - the amount shown.

Table 3
Errors of Estimate for Given Sample Sizes
95% Confidence Level

Location	Reporting Frequency	Quality Criteria	Target Range	Accuracy of* Average Range	Sampling Frequency	Testing Frequency
Digester Feed	Daily	Moisture	—	±1.5**	½ Hours	1/Day
	Weekly	Bark	0.5 – 2	±0.3**	½ Hours	1/Day
	Monthly	Overthick Pin Chips Fines	4 – 10 7 – 13 1 – 3	±1.0** ±1.0** ±0.3**	½ Hours ½ Hours ½ Hours	1/Day 1/Day 1/Day
Sources	Weekly or Bi-Weekly	Moisture	—	±3.0	5/Report	5/Report
		Bark	0.5 – 2	±0.3	10/Report	10/Report
		Overthick Pin Chips Fines	4 – 10 7 – 13 1 – 3	±2.0 ±2.0 ±0.5	5/Report 5/Report 5/Report	5/Report 5/Report 5/Report
Monthly	Moisture	—	±1.5	10/Report	10/Report	
	Bark	1.0 – 2.0	±0.2	20/Report	20/Report	
	Overthick Pin Chips Fines	7 – 10 7 – 13 2 – 3	±1.0 ±1.0 ±0.3	10/Report 10/Report 10/Report	10/Report 10/Report 10/Report	
Monthly	Moisture	—	±1.0	20/Report	20/Report	
	Bark	0.5 – 1.0	±0.1	40/Report	40/Report	
	Overthick Pin Chips Fines	4 – 7 7 – 13 1 – 2	±0.8 ±0.8 ±0.2	20/Report 20/Report 20/Report	20/Report 20/Report 20/Report	

*i.e., Error of estimate for given sample frequencies with 90% chance that true average is within indicated range

** Assuming the test result on the composite would be the same as the averaged result of testing all 12 samples

Table 4
Fiber Raw Material Quality Monitoring Reporting, Sampling and Testing Frequency

Flow	Transportation Mode	# Deliveries per Week	# Samples/Week	
			% OD Testing	Full Testing ¹
Kamyr	Belt	10.5 MBDT	21	21
Bath	Belt	6.0 MBDT	21	21
Springfield Sawmill	Belt	1.3 MBDT	10	2
Springfield Plywood	Belt	1.0 MBDT	10	2
Springfield Dia Lil	Belt	11.3 MBDT	10	2
Springfield B/C	Pipe	2.1 MBDT	2	2
Cottage Grove Mill A	Truck	20	7	2
Cottage Grove Mill B	Truck	75	15	3
Klamath Falls Sawmill #1	Rail	15	10	3
Klamath Falls Sawmill #2	Rail	7	7	2
Klamath Falls Sawmill Fiber	Rail	18	10	3
Klamath Falls Sawmill Ply.	Rail	7	7	2
Bly Mill A	Rail	5	5	2
Bly Mill B	Rail	25	10	3
Outside Purchase #1	Truck	75	15	2
#2	Truck	10	7	2
#3	Truck	30	10	3
#4	Truck	15	7	2
#5	Truck	25	7	2
#6	Rail	15	10	3
#7	Rail	5	5	2
#8	Rail	10	10	3
#9	Rail	10	10	3
#10	Rail	5	5	2
#11	Rail	12	10	3
#12	Rail	15	10	3
#13	Truck	35	10	2
#14	Truck	35	10	2
#15	Truck	20	7	2
#16	Truck	7	7	2
#17	Truck	5	5	2
#18	Truck	20	7	2
#19	Truck	20	7	2
#20	Truck	50	10	0
Other Small Suppliers	Truck/Rail	40	40	10

1. Except for 1% Sodium Hydroxide Solubility. This should be:
- | | |
|---------------------|----------------|
| 1/day/Kamyr & Batch | -14/week |
| 1/DF Supplier/week | -14/week |
| | <u>28/week</u> |

Table 5
Sampling Requirements at Springfield

Chip Size Distributions of:

Chip Size Classes	Manual Multiple-Sample Composite Taken at Transfer Point		
	Automatic Railcar Sampling Device, Single Grab Sample	In Railcar Dump Pit by WSF	At Chip Stacker by RAB
True Overs	(3)	(1)	(2)
+ 1 1/8"	4% 4	7% 11	7% 8
+ 7/8"	10	19	20
+ 5/8"	45	42	43
+ 3/8"	26	17	18
+ 3/16"	10	3	4
Pan			

Table 6

Performance of a Poorly Designed Automatic Sampler, Compared to Careful Sub-Sampling and Compositing by Hand (20)

<u>Test</u>	<u>Oven-Dry Weight (pounds) of Chips Required for a Single Test</u>
Moisture Content**	5
Size Classification** (EMP Classifier)	2
Bark Content**	4
Bulk Density**	13
Specific Gravity	4
pH and Acid Number	1
Grit (Acid Insoluble Ash)	4
Extractives and Chemical Analysis	2
Decay Content**	4

**Indicates sample can be used for other tests

Table 7
Sample Requirements for Weyerhaeuser Chip Test Methods

Characteristics	Approximate Range of Values in Characteristics	No. of Samples or "Scoops" per Composite										
		2	3	5	7	9	12	15	20	25	100	
% OD Solids	40 – 80%	55	15	8	6	5	4	3	3	3	3	
% Wood	20 – 100%	64	18	9	7	6	5	4	3	3	1	
Bulk Density (kg/m ³)												
Uncompacted	120 – 160%	85	23	12	9	7	6	5	4	4	2	
Compacted	140 – 180%	93	26	13	10	8	7	6	5	4	2	
True Oversize (+100 mm)	0 – 10%	25	7	4	3	2	2	2	1	1	1	
+64 mm	0 – 20%	20	6	3	2	2	1	1	1	1	0.5	
-2 mm	30 – 80%	59	16	8	6	5	4	4	3	3	1	

1. Tabulated values estimate the average precision interval (i.e., ± tabulated value around the mean) that will contain the true population mean 19 out of 20 times. A "large" interval indicates a variable characteristic, and hence, we are less sure what the true population mean really is.

2. Individual fuel flows can deviate substantially from these average values.

Table 8
Estimate of Attainable Precision ^{1/2} in Characteristics of Hogged Fuel Feed to Boilers

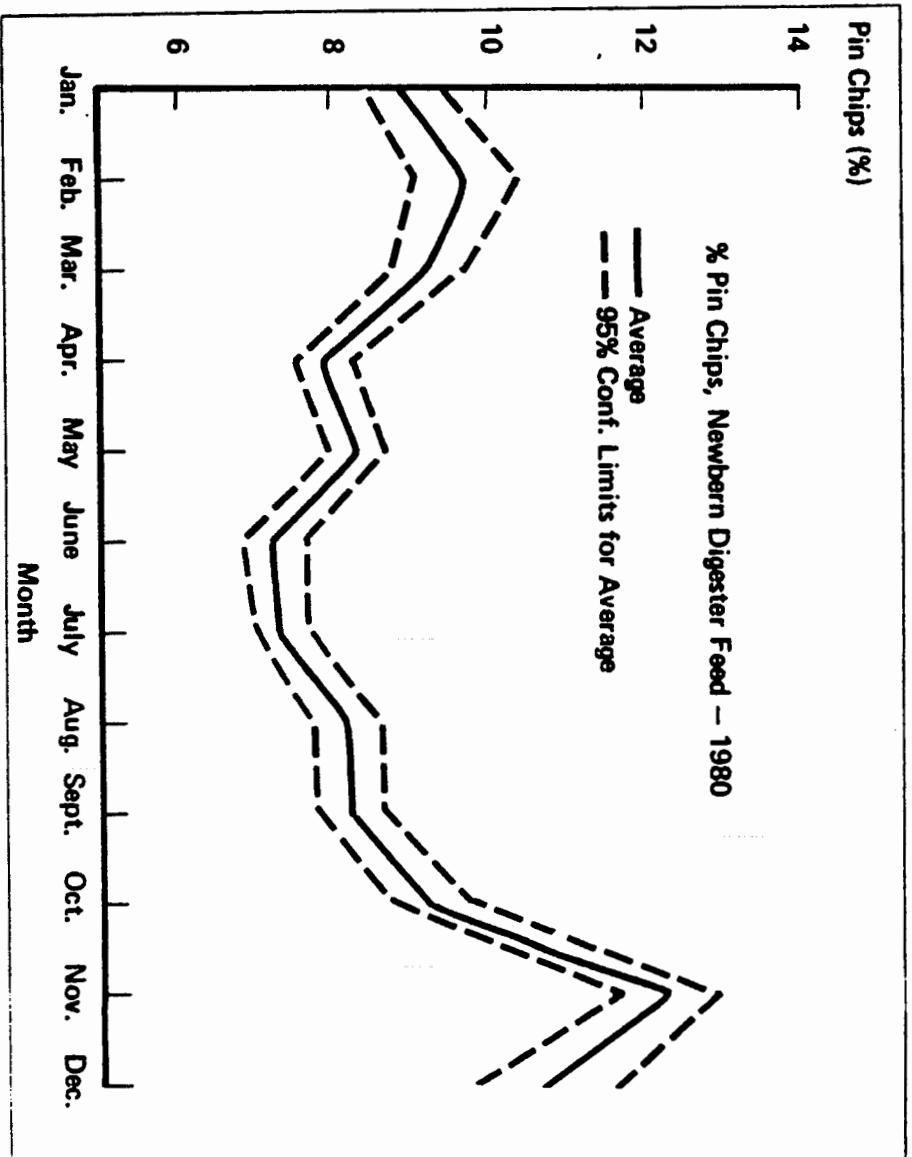


Figure 1
 Variability in Chip Quality

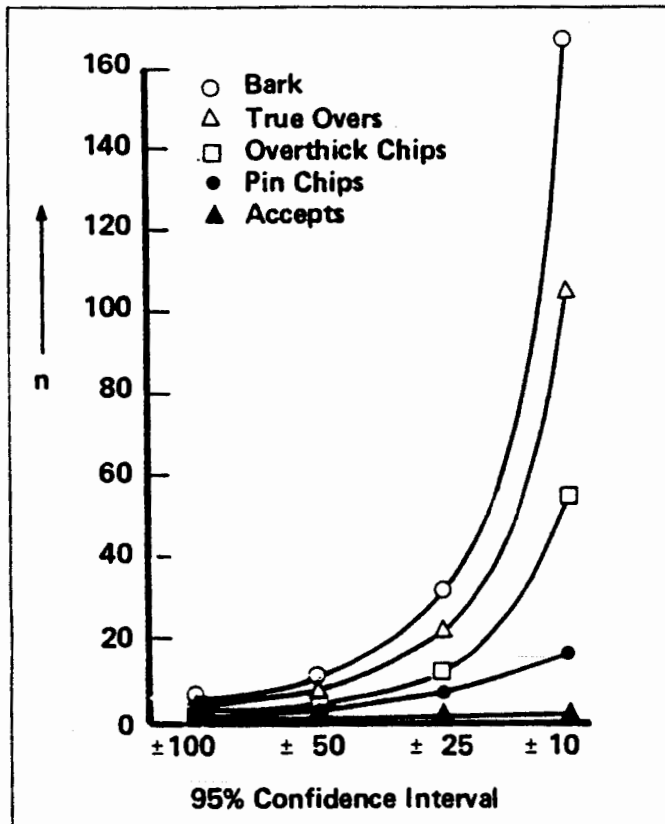


Figure 2
Rail Car Sampling. Sample sizes (n) required to guarantee the specified 95% confidence intervals 95% of the time (13)

Scoop No.	Maximum Particle Size (mm)	Dimensions (mm)					Capacity (mL)
		a	b	c	d	e	
150	150	350	140	350	300	140	16,000
125	125	300	120	300	250	120	10,000
100	100	250	110	250	220	100	7,000
75	75	200	100	200	170	80	4,000
50	50	150	75	150	130	65	1,700
40	40	110	63	110	95	50	790
30	30	90	50	90	80	40	400
20	20	80	45	80	70	35	300
15	15	70	40	70	60	30	200
10	10	60	35	60	50	25	125
5	5	50	30	50	40	20	75
3	3	40	25	40	30	15	40
1	1	30	15	30	25	12	15

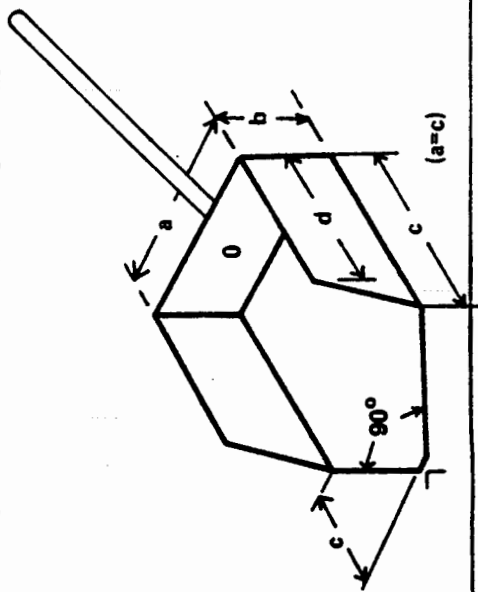


Figure 3
Design of Sampling Scoops (3)



Figure 4a. In an experiment to design chip flow diversion devices for automatic sampling, three widths were tested – 100 mm (not shown), 127 mm and 152 mm (foreground)



Figure 4b. When held perpendicular to the chip flow, even the largest diverter (152 mm) plugs quickly.

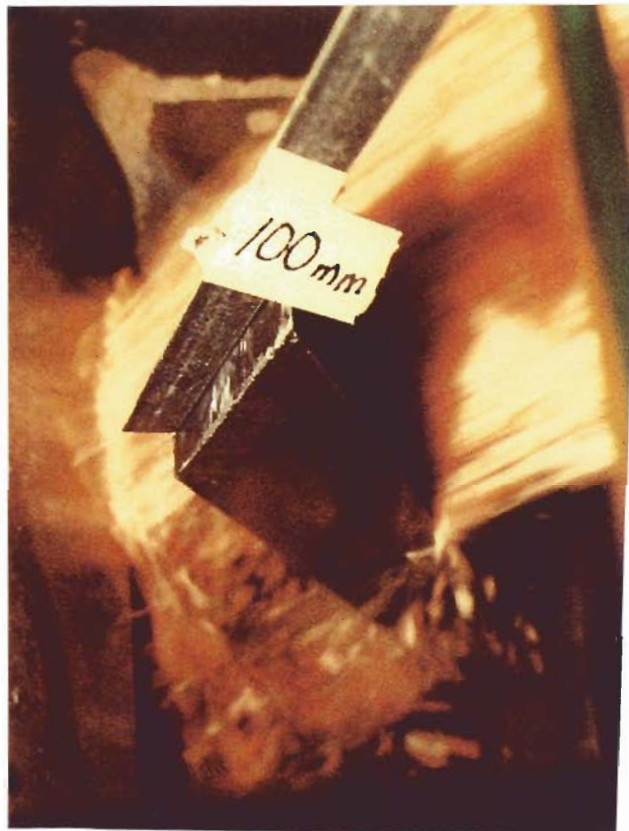
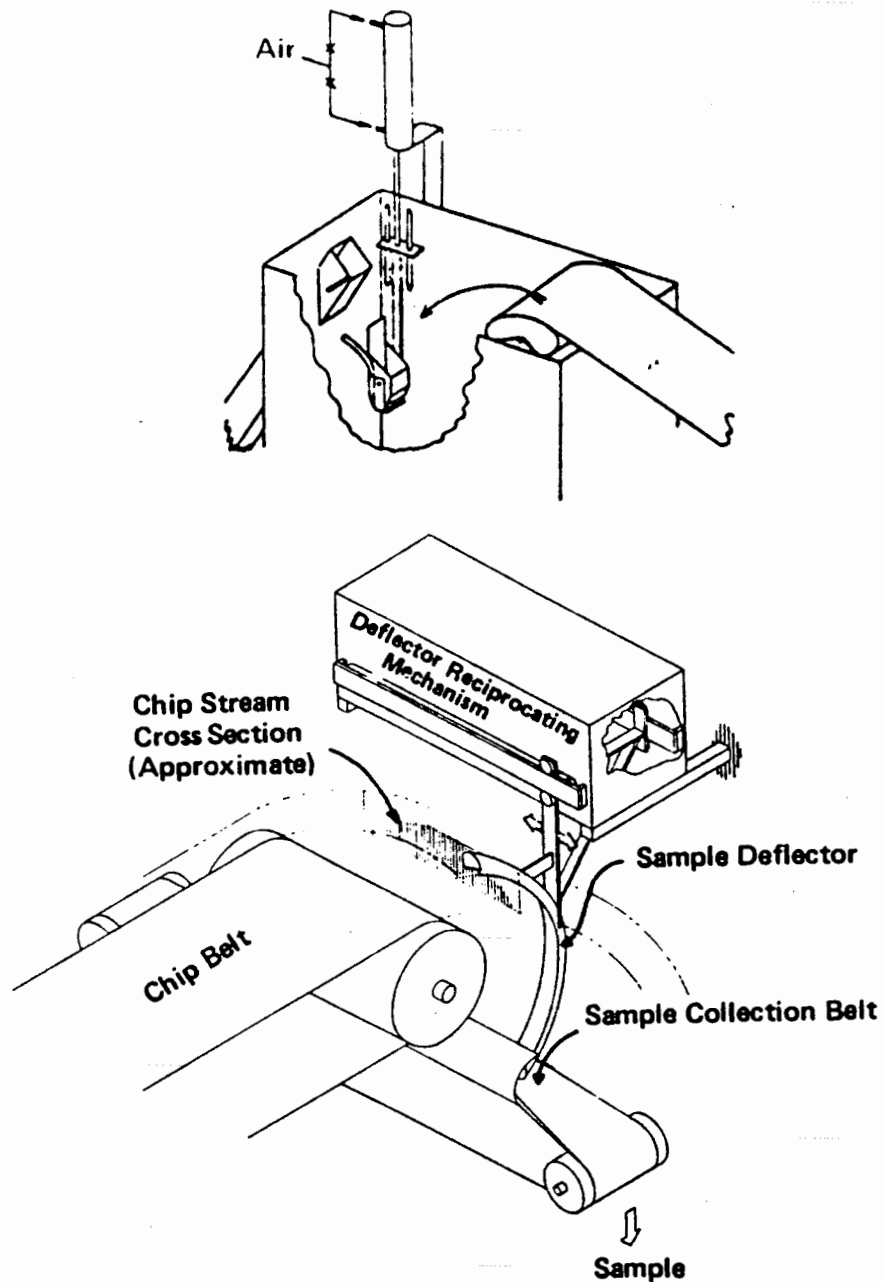


Figure 4c. An optimum angle can be determined where even the smallest (100 mm) would not plug.



Figure 4d. At greater angles, however, the 100 mm diverter plugged within seconds.



- Figure 5**
- a) **Pneumatic Actuated Grab Sampler (15)**. This type sampler can only be used at transfer points where good mixing occurs after the chips leave the belt and may require installation of mixing vanes in the free-fall zone.
- b) **Reciprocating Deflector Sampler**. Adapted from the ore and coal industry, this is a highly reliable design that can be fit into existing transfer points with moderate rebuild.

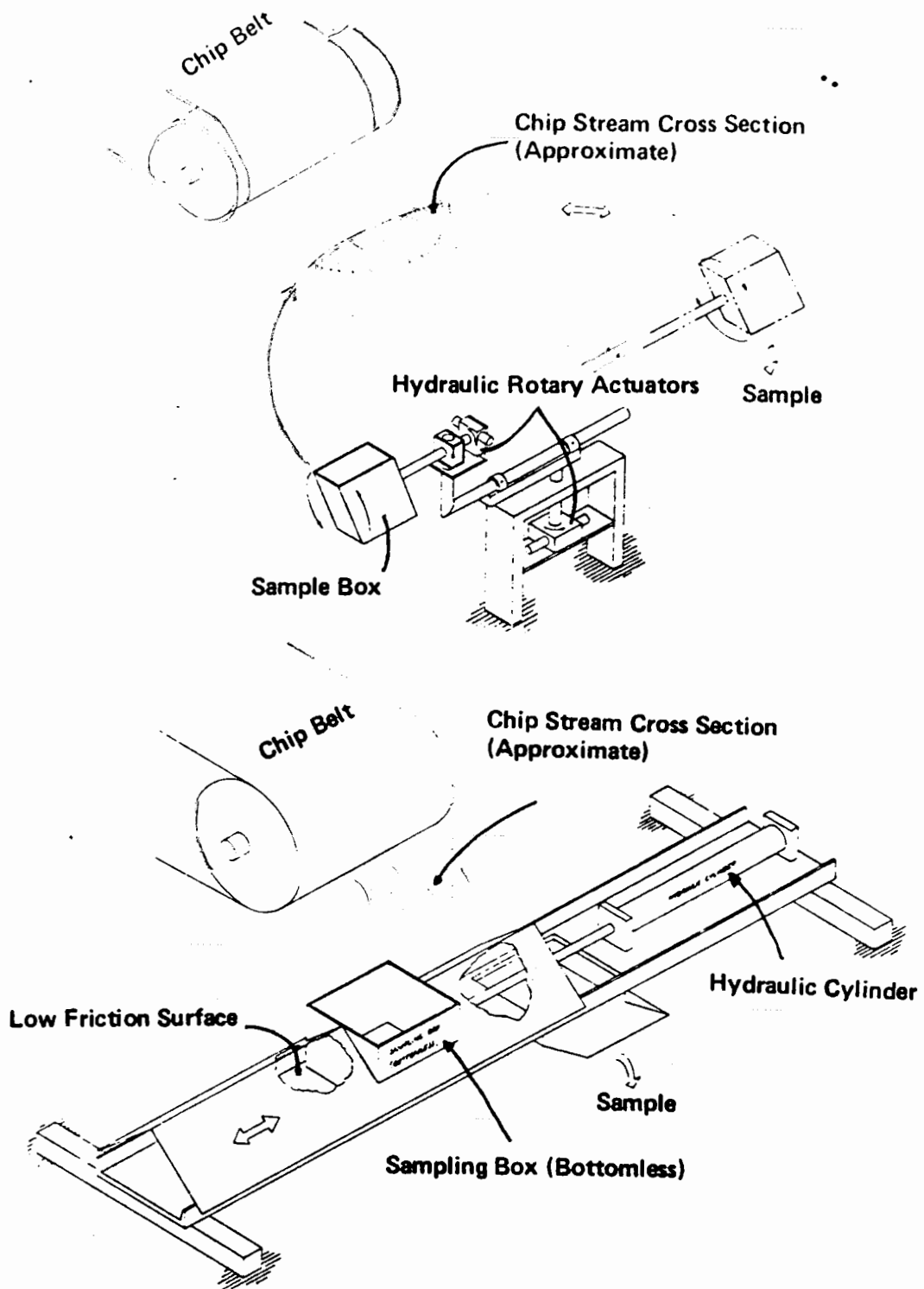


Figure 5 c) **Rotary Sampler.** A good design in multiple grab samples are needed, but works best on small particles (sawdust, hogged fuel) due to limits of sample box size vs. maximum particle size.

d) **Linear Sampler.** This removed grab samples, but is best used on well-mixed, low flow streams. The box should be half full when it makes first pass and fill completely as it returns to hole for dumping or two holes for dumping can be built.

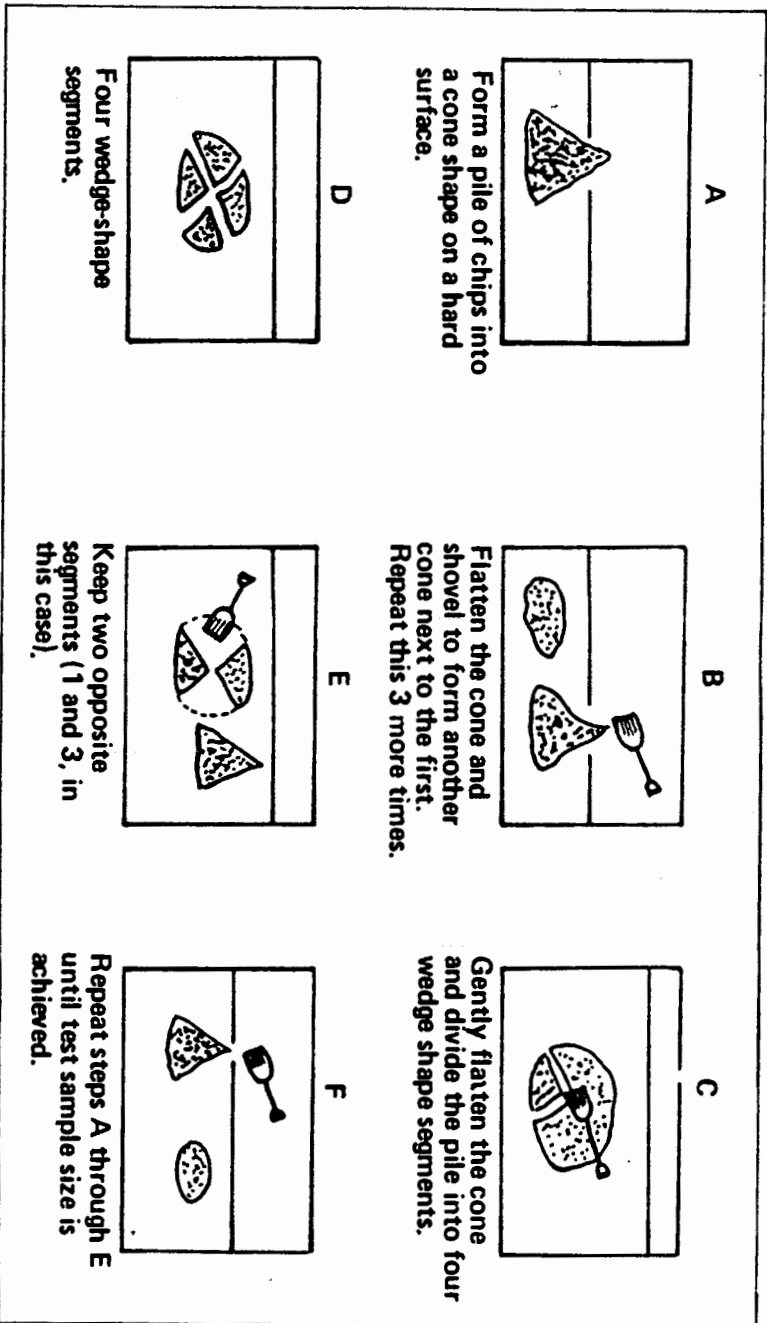


Figure 6
Suggested Method for Manually Blending, Compositing and Sub-Sampling Chips and Wood-Waste Fuel (Adapted from 3)

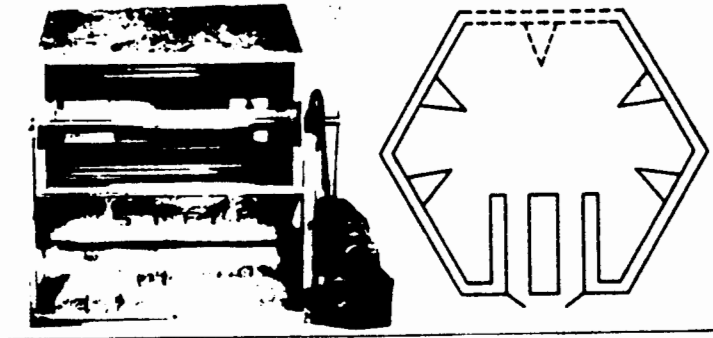


Figure 7. Photo (left) and cross-section (right) of a rotating drum device made at STPI for removal of test sample from larger chip samples with volume-adjusted boxes to be emptied by means of shutters (Hartler).