UNIT 11 SAMPLING TECHNIQUES OF FOOD PRODUCTS

Structure

11.0 OBJECTIVES

After reading this unit, we shall be able to:

- state the importance of sampling in analysis of food products;
- enlist standards and guides on sampling;
- prepare sampling plans;
- describe different sampling techniques; and
- devise ways to draw a representative sample from lots.

11.1 INTRODUCTION

To control food quality and acceptance within satisfactory limits, it is important to monitor the vital characteristics of raw materials, ingredients, and processed foods. This could be done by evaluating all foods or ingredients from a particular lot, which is feasible if the analytical technique is rapid and non-destructive. However, it is usually more practical to select a portion of the total product volume and assume the quality of the selected portion is typical of the whole lot.

Obtaining a portion, or sample, that is representative of the whole is referred to as sampling, and the total quantity from which a sample is obtained is called the population. Adequate sampling technique helps to ensure that sample quality measurements are an accurate and precise estimate of the quantity of the population. By sampling only a fraction of the population, a quality estimate can be obtained more quickly and with less expense and personnel time than if the total population were measured. The sample is only an estimate of the value of the population, but with proper sampling technique, it can be a very accurate estimate.

11.2 SAMPLE COLLECTION

It is important to clearly define the population that is to be sampled. The population may vary in size from a production lot, a day's production, to the contents of a warehouse. Extrapolating information obtained from a sample of a production lot to the population of the lot can be done accurately, but conclusions cannot be drawn from data describing larger populations, such as the whole warehouse.

Populations may be finite, such as the size of a lot, or infinite, such as in the number of temperature observations made of a lot over time. For finite populations, sampling provides an estimate of lot quality. In contrast, sampling from infinite populations provides information about a process. Fig.11.1 represents the sampling for physical and chemical characteristics of food. Regardless of the population type, that is, finite or infinite, the data obtained from sampling are compared to a range of acceptable values to ensure the population sampled is within specifications.

11.2.1 Homogeneous Versus Heterogeneous Populations

The ideal population would be uniform throughout and identical at all locations. Such a population would be homogeneous. Sampling from such a population is simple, as a sample can be taken from any location and the analytical data obtained will be representative of the whole. However, this occurs rarely, as even in an apparently uniform product, such as sugar syrup, suspended particles and sediments in a few places may render the population heterogeneous. In fact, most populations that are sampled are heterogeneous. Therefore, the location within a population where a sample is taken will affect the subsequent data obtained. However, sampling plans and sample preparation can make the sample representative of the population or take heterogeneity into account in some other way.

11.2.2 Manual Versus Continuous Sampling

To obtain a manual sample the person taking the sample must attempt to take a "random sample" to avoid human bias in the sampling method. Thus, the

sample must be taken from a number of locations within the population to ensure it is representative of the whole population. For liquids in small containers, this can be done by shaking prior to sampling. When sampling from a large volume of liquid, such as that stored in silos, aeration ensures a homogeneous unit. Liquids may be sampled by pipetting, pumping, or dipping. However, when sampling grain from a rail car, mixing is impossible and samples are obtained by probing from several points at random within the rail car. Such manual sampling of granular or powdered material is usually achieved with triers or probes that are inserted into the population at several locations. Errors may occur in sampling, as rounded particles may flow into the sampling compartments more easily than angular ones. Similarly, hygroscopic materials flow more readily into the sampling devices than does non hygroscopic material. Horizontal core samples have been found to contain a larger proportion of smaller-sized particles than vertical ones. Continuous sampling is performed mechanically.

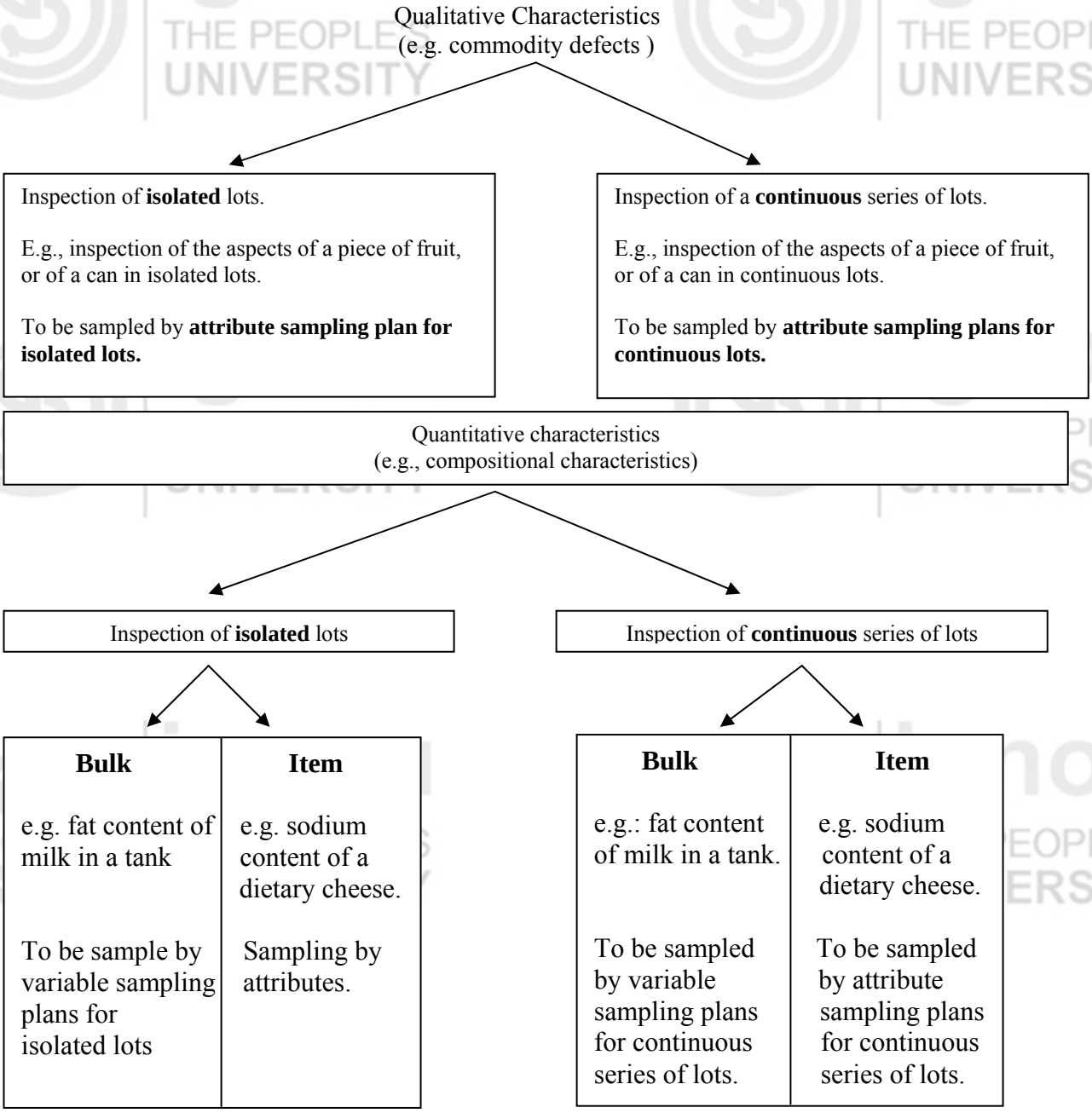

Fig. 11.1: Sampling Flowchart for Chemical and Physical Characteristics

11.2.3 Importance of Sample Collection

The reliability of analytical data thus obtained depends on several factors, sampling being the major factor. Current analytical methods require only few grams of food sample to analyze. Thus, it is necessary that a sample be as representative of the population as possible.

There are three basic activities involved in analysis of food products:

- Collection of representative sample.
- Sample preparation.
- Analysis using appropriate methods and instruments.

These activities, although independent in nature, yet can have decisive influence on each other. Furthermore, each of these activities have their own potential sources of variations that contribute to the uncertainty level associated with any analytical result. Thus, care must be taken to identify the sources of variation and minimize or avoid them while accomplishing any activity. On the part of the laboratories, it is therefore necessary to develop a plan for the proper performance of each activity, and then establish quality standards and written procedures in compliance with the standards. Many times, the activity of sampling falls outside the purview of a laboratory's mandate or control. This is especially true in commercial testing laboratories where the "first contact" is the arrival of samples. To improve the overall quality of the analytical process, a laboratory must do all it can to receive appropriate, applicable, defensible samples. The development of appropriate plans will depend upon an understanding of the problems involved in each activity, and then the application of reasonable judgements in seeking solutions.

It should be noted that sampling terminology and procedures used may vary between companies and between specific applications. However, the principles described in this Unit are intended to provide a basis for understanding, developing, and evaluating sampling plans and sample handling procedures for specific applications encountered.

A sample should represent a population as adequately as possible. To ensure proper sampling, the analysts need to be consulted time to time concerning proper sample size, suitable containers for sampling or the use of appropriate preservatives to prevent any spoilage or transformation in a sample before analysis. One common cause of lack of precision or lab-to-lab variation in analytical results for a particular population can be traced back to erroneous sampling. For example, in case of grapes, a laboratory sample size of meager 3 kg berries represents the whole population of > 10000 kg in 1 hectare vineyard area. Thus, if the sample collected is not representative, then there will be sample-to-sample variation in results. When significant difference in results occurs among laboratories which have supposedly analyzed the same sample, a serious conflict may arise questioning the competence and credibility of the laboratories. Many of these situations can be avoided if samples are collected according to a rational plan that gives some assurance that the sample delivered to the laboratory represents the composition of the parent lot.

There are at least two ways to measure a given lot of goods: one, that we often assume to be the "proper" way, is to find its "true value", by which we mean its average value. The other way, often discovered accidentally as a result of

"poor" sampling, is to measure its variability. So called proper sampling of drug dosage forms, for example, may involve compositing 20 tablets, by which the majority of the tablets could be used to dilute and conceal the fact that several of them are severely sub- or super- potent. Similarly, two lots of grain may have been purposely, but ineffectively, mixed in an attempt to reduce the average level of a contaminant. Sampling that led to the laboratory finding inconsistent results would reveal the attempt to dilute an illegal product.

11.2.4 Errors in Analytical Results due to Improper Sampling

Few studies have been conducted on the distribution of error among the three activities: sampling, sample preparation, and analysis. In one such study, which involved analysis of 20 nanogram/gram concentration of aflatoxin in a lot of peanuts, the error contributed by the sampling step was as high as 67% of the total variance, in comparison to 20% and 13% errors contributed by the analyst and the analytical procedure, respectively. In a field study conducted at the National Research Centre for Grapes, Pune, the pesticide residues in grape samples analyzed in 15 individual grape bunches collected out of 1 acre area showed above 50% sampling-induced variations. The results of such experimentations are not unusual and it illustrates the proportion of error that can be attributed to sampling. For peanuts, the distribution of aflatoxin can vary widely, with a few peanuts accounting for most of the contamination. Similarly, in case of the field sampling of grapes, the pesticide residues might have deposited in variable concentrations in different grape bunches and thus when they were analyzed separately, showed variable results. The important point in these examples is to show that sampling error can play a very significant part in the overall error in the analytical system.

11.2.5 Risks Associated with Sampling

There are two types of risks associated with sampling. Both should be considered when developing a sampling plan. The consumer risk describes the probability of accepting a poor quality population. This should happen rarely (<5% of the lots) but the actual acceptable probability of a consumer risk depends on the consequences associated with accepting an unacceptable lot. These may vary from major health hazards and subsequent fatalities to a lot being of slightly lower quality than standard lots. Obviously, the former demands a low or no probability of occurring whereas the latter would be allowed to occur more frequently. The second risk i.e., vendor risk is the probability of rejecting an acceptable product. As with consumer risk, the consequences of an error determine the acceptable probability of the risk. An acceptable probability of vendor risk is usually 5-10%.

11.3 SAMPLING STANDARDS

Data obtained from an analytical technique are the result of a stepwise procedure from sampling, to sample preparation, laboratory analysis, data processing, and data interpretation. There is a potential for error at each step and the uncertainty, or reliability, of the final result depends on the cumulative errors at each stage. Variance is an estimate of the uncertainty. The total variance of the whole testing procedure is equal to the sum of the variances associated with each step of the sampling procedure and represents the precision of the process. Precision is a measure of the reproducibility of the data. In contrast, accuracy is a measure of how close the data are to the true value. The most efficient way to improve accuracy is to improve the reliability

of the step with the greatest variance. Frequently, this is the initial sampling step. The reliability of sampling is dependent more on the sample size than on the population size. The larger the sample size, the more reliable the sampling. However, sample size is limited by time, cost, sampling methods, and the logistics of sample handling, analysis, and data processing.

It should be noted that sampling terminology and procedures used may vary between companies and between specific applications. Several standards and recommendations provide the ways and means to sample a particular lot.

ISO specifies various standards and guidelines for drawing samples and data interpretation for samples. Few such standards related to food are mentioned in Table 11.1.

Table 11.1: Standards and Guides of Sampling

ID of Standard/Guide	Title of Standard/Guide
ISO 2854 : 1976	Statistical Interpretation of data- techniques of estimation and tests relating to means and variances.
ISO 2859-0 : 1995	Sampling procedures for inspection by attributes-Part 0 ; Introduction to the ISO 2859 Attribute sampling system
ISO 2859-1 : 1999 / IS 2500 (Part I) : 1989	Sampling procedures for inspection by attributes-Part 1; Sampling plans indexed by Acceptable Quality Level (AQL) for lot-by-lot inspection.
IS 2500 (Part II) : 1965	Sampling inspection procedures.
ISO 2859-2 : 1985	Sampling procedures for inspection by attributes- Part 2; Sampling plans indexed by Limiting Quality (LQ) for isolated lot inspection.
ISO 3494 : 1976	Statistical interpretation of data – Power of tests relating to mans and variances.
ISO 3951 : 1989	Sampling procedures and charts for inspection by variables for per cent non-conforming.
ISO 5725-1 : 1994	Application of statistics- Accuracy (trueness and precision) of measurement methods and results- Part 1; General principles and definitions.
ISO 7002 : 1986	Agricultural food products-Layout for a standard method of sampling a lot.
ISO 8423 : 1991	Sequential sampling plans for inspection by variables for per cent non-conforming (known standard deviation).
ISO 8422 : 1991	Sequential sampling plans for inspection by attributes.
ISO/ TR 8550 : 1994	Guide for the selection of an acceptance sampling system, scheme or plan for inspection of discrete items in lots.
ISO 10725 : 2000	Acceptance sampling plans and procedures for the inspection of bulk material.

ISO/ FDIS 11648/1	Statistical aspects of sampling from bulk materials – Part 1; General principles.
ISO/ DIS 14560	Acceptance sampling procedures by attributes – specified quality levels in non-conforming items per million.
Other IS specifications	Specifications published by BIS pertaining to individual products contain the details of lot size and corresponding number and quantity of samples to be drawn and tested for conformance.

Since all standards are subject to revision, parties to agreements based up on these guidelines should ensure that the most recent editions of the standards are always applied.

An example for flour sampling as per AOAC method is presented here. The AOAC Method 925.08 (6) describes the method for sampling flour from sacks. The number of sacks to be sampled is determined by the square root of the number of sacks in the lot. The sacks to be sampled are chosen according to their exposure. The samples that are more frequently exposed are sampled more often than samples that are exposed less. Sampling is done by drawing a core from a corner at the top of the sack diagonally to the center. The sampling instrument is a cylindrical, polished trier with a pointed end. It is 13 mm in diameter with a slit at least one third of the circumference of the trier. A second sample is taken from the opposite corner in a similar manner. The cores are stored for analysis in a clean, dry, airtight container that has been opened near the lot to be sampled. The container should be sealed immediately after the sample is added. A separate container is used for each sack. Additional details regarding the container and the procedure also are described below.

Title 21 CFR specifies the sampling procedures required to ensure that specific foods conform to the standard of identity. In the case of canned fruits, 21 CFR 145.3 defines a sample unit as "container, a portion of the contents of the container, or a composite mixture of product from small containers that is sufficient for the testing of a single unit". Furthermore, a sampling plan is specified for containers of specific net weights. The container size is determined by the size of the lot. A specific number of containers must be filled for sampling of each lot size. The lot is rejected if the number of defective units exceeds the acceptable limit. For example, out of a lot containing 48,001 to 84,000 units, each weighing 1 kg or less, 48 samples should be selected. If six or more of these units fail to conform to the attribute of interest the lot will be rejected. Based on statistical confidence intervals, this sampling plan will reject 95% of the defective lots examined, that is, 5% consumer risk.

Table 11.2: Minimum Number of Primary Samples to be taken from a Lot

(Ref: CODEX doc. CAC/GL 33)

Name of Commodity	**Minimum Number of Primary Samples to be taken from a Lot**
1. Meat and Poultry	
a) Non-suspect lot	1
b) Suspect lot	Determined according to Table 11.3

2. Other products	
a) Products packaged in bulk, which can be assumed to be well mixed or homogeneous.	1 (A lot may be mixed by grading or manufacturing process)
b) Products packaged in bulk, which may not be well mixed or homogeneous.	For products comprised of large units, the minimum number of primary samples should comply with the minimum number of units required for the laboratory sample.
Either Weight of lot, kg	
<50	3
50-500	5
>500	10
or, Number of cans, cartons, containers in the lot	
1-25	1
26-100	5
> 100	10

Table 11.3: Number of randomly selected primary samples required for a given probability of finding at least one non-compliant sample in a lot of meat or poultry for a given incidence of non-compliant residue in the lot

(Ref: CODEX doc. CAC/GL 33)

Incidence of Non-compliant Residues in the Lot	**Minimum number of samples (n_o) required to detect a non- compliant residue with a probability of**		
%	**90%**	**95%**	**99%**
90	1	-	2
80	-	2	3
70	2	3	4
60	3	4	5
50	4	5	7
40	5	6	9
35	6	7	11
30	7	9	13
25	9	11	17
20	11	14	21
15	15	19	29
10	22	29	44
5	45	59	90
1	231	299	459
0.5	460	598	919
0.1	2302	2995	4603

Notes:

a) The table assumes random sampling.

b) Where the number of primary samples indicated in Table 11.2 is more than about 10% of units in the total lot, the number of primary samples taken may be fewer and should be calculated as follows:

$$n = \frac{n_o}{1 + (n_o - 1) / N}$$

Where, n = minimum number of primary samples to be taken

n_o = number of primary samples given in Table 11.2

N = number of units, capable of yielding a primary sample in the lot.

c) Where a single primary sample is taken, the probability of detecting a non-compliance is similar to the incidence of non-compliant residues.

d) For exact or alternative probabilities, or for a different incidence of non-compliance, the number of samples to be taken may be calculated from:

$$1 - p = (1 - i)^n$$

Where, p is the probability and i is the incidence of non-compliant residue in the lot (both expressed as functions, not percentages) and n is the number of samples.

Table 11.4: Sampling Procedure and Minimum Amount (composite/bulk) to be Sampled from lots

Crop Type	Sampling Procedure	Example	Minimum Quantity
Root, tuber and bulb vegetables	Take samples from all areas of the crop. Remove as much adhering soil as possible from samples but do not wash.	Beet (red, sugar, fodder), onions, parsnips, potatoes, sweet potatoes, turnips	5 kg (and not less than 5 items)
	(**Note:** In some cases, where leaf parts are used as stock feed, they may need to be sampled separately).	Carrots, radish, spring onions.	2 kg
	Take the sample from all areas of the crop. Sample parts of the crop exposed to the spray and also those apparently protected by foliage.	Brassica (cabbage, cauliflower, broccoli, kohlrabi, curly kale).	5 kg (and not less than 5 items)
Leafy, stem, fruiting and legume vegetables	Remove as much soil as possible from crops such as celery, but do not wash.	Asparagus, brussel sprouts, celery, chicory, lettuce, spinach, turnip tops	2 kg
		Cucumber, melon, squashes, eggplant	5 kg (and not less than 5 items)
		Peppers, tomatoes, gherkins	2 kg
		Beans, peas, etc. (with pods)	2 kg
All tree and bush fruit, including vines, small fruits and berries	Select fruit from all parts of the tree/bush, high and low, and from both sides of the row, and select fruits according to abundance whether in each segment or	Apples, citrus, peaches, pears	5 kg

	the whole tree/bush. More fruit should therefore be selected from the more densely laden parts of the crop.		
	Sample parts of the crop exposed to the spray and also those apparently protected by foliage.	Cherries, nuts, olives, plums.	2 kg
	Take large and small fruits, perfect or slightly blemished, but not so small or blemished that they would not normally be saleable.	Bush fruit (all types), grapes, strawberries	2 kg
Cereal Grains	Cut not less than ten small areas (approximately 0.1 m^2) chosen randomly from all areas of the crop. Cut stalks about 10 cm above the ground. Remove grain from the straw.	Maize (grain and cobs)	2 kg
Oil Seeds	Collect the heads when they have reached the stage of maturity at which they are normally harvested and if convenient thresh to remove the seeds.	Sesame, canola, soybeans, sunflower	1 kg of seeds

Table 11.5: Description of Primary samples and min. size of laboratory sample

(Ref: CODEX doc. CAC/GL 33)

S. No	Commodity Classification	Examples	Nature of Primary Samples to be taken	Minimum Size of Lab Sample
1. Primary food commodities of animal origin				
1.1	Large mammals *Whole or half carcass* *Usually 10 kg or more*	Cattle Sheep Pigs	Whole or part of diaphragm, supplemented by cervical muscles, if Necessary	0.5 kg
1.2	Small mammals *Whole carcass*	Rabbits	Whole carcass or hind quarters	0.5 kg, after removal of skin and bone
1.3	Mammal meat parts, loose fresh/chilled/frozen Packaged or other wise	Quarters Chops Steaks Shoulders	Whole unit(s),or a portion of a large unit	0.5 kg, after removal of bone
1.4	Mammal meat parts, bulk frozen	Quarters Chops	Either a frozen cross-section of a container or the whole (or portions of individual) meat parts	0.5 kg, after removal of bone
2. Poultry fats				
2.1	Birds, at slaughter whole or part-carcass	Chickens Turkeys	Units abdominal fat from at least three birds	0.5 kg

2.2	Bird meat parts	Legs Breast – muscle	Either visible fat, trimmed from units	0.5 kg
			Whole units or portions where fat is not trimmable	2 kg
2.3	Bird fat tissue in bulk		Units taken with a sampling device from at least three portions	0.5 kg
2.4	Poultry eggs		Whole eggs	12 whole eggs
2.5	Liquid, frozen or dried egg products		Whole units	0.5 kg
3. Processed foods of animal origin				
3.1	Mammal or bird comminuted, cooked, canned, dried, rendered or otherwise processed products including multi ingredient products	Ham sausage / Minced beef / Chicken paste	Packaged units or a representative cross section from a container or units (including juices, if any) taken with a sampling device	0.5 kg or 2 kg if fat content < 5%
3.2	Liquid milks, milk powders, ice creams etc.		Packaged units	0.5 L or 0.5 kg
3.3	Cheese	Units > 0.3 kg	Whole unit	0.5 kg
		Units < 0.3 kg	Whole unit	0.3 kg
4. Herbs		Fresh	Whole units	0.5 kg
		Dried	Whole units	0.1 kg
5. Feed commodities of plant origin				
5.1	Legume animal feeds and other forages and fodders		Whole units	1 kg (at least 10 units)
5.2	Straw, hay and other dry products		Units taken with a sampling device	0.5 kg (at least 10 units)
6. Processed foods of plant origin				
6.1	Products of high unit value		Packages or units taken with a sampling device	0.1 kg
6.2	Solid products of low bulk density	Hops Tea	Packages or units taken with a sampling device	0.2 kg
6.3	Other solid products	Bread Flour Dried fruits Vegetable	Packages or units taken with a sampling device	0.5 kg
6.4	Liquid products	Oils Juices	Packages or units taken with a sampling device	0.5 L or 0.5 kg

Check Your Progress Exercise 1

Note: a) Use the space below for your answers.
b) Check your answers with those given at the end of the unit.

1) What is the importance of sample collection in analysis of food products?

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2) What are homogenous and heterogeneous populations?

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11.4 THE SAMPLING PLAN

11.4.1 Understanding a Sample Plan

Sampling is generally done for a specific purpose and the purpose may indeed suggest or dictate the nature of any sampling plan. The International Union of Pure and Applied Chemistry (IUPAC) defines a sampling plan as "a predetermined procedure for the selection, withdrawal, preservation, transportation, and preparation of the portions to be removed from a lot as samples". A sampling plan should be a well-organized document that establishes the required procedures for accomplishing the program's objectives. It should address the issues of who, what, where, why, and how. The primary aim of sampling is to obtain a sample, subject to constraints on size, that will satisfy the sampling plan specifications. A sampling plan should be selected on the basis of the sampling objective, the study population, the statistical unit, the sample selection criteria, and the analysis procedures. Factors determing the choice of a sampling plan are enlisted in Table 11.6. The two primary objectives of sampling are often to estimate the average value of a characteristic and determine if the average value meets the specifications defined in the sampling plan. The presence of a well designed plan is important because it provides a consistent model to guide people performing the sampling activity, and it serves as a reminder of the important elements in this part of the overall sample analysis program.

Table 11.6: Factors Affecting Choice of Sampling Plans

Factors to be considered		Questions
Purpose of inspection	a	Is it to accept or reject the lot?
	b	Is it to measure the average quality of the lot?
	c	Is it to determine the variability of the product?
Nature of Product	a	Is it homogeneous or heterogeneous?
	b	What is the unit size?
	c	How consistently have past populations met specifications?
	d	What is the cost of the material being sampled?
Nature of the test method	a	Is the test critical or minor?
	b	Will someone become sick or die if the population fails to

		pass the test?
	c	Is the test destructive or non-destructive?
	d	How much does the test cost to complete?
Nature of the population being investigated	a	Is the lot large but uniform?
	b	Does the lot consist of smaller, easily identifiable sublots?
	c	What is the distribution of the units within the population?

11.4.2 Statistical Approaches

In many sampling programs, statistical approaches are not given the requisite attention. Percentage sampling systems that specify a fixed percentage of a lot, say 5 or 10%, do not provide the quality protection that is often assumed. Statistical sampling theory furnishes the means to analyze the relationship between a lot of goods and the samples that are drawn from it. It can be used to estimate population measure, or "parameters," such as variance and correlation, from knowledge of corresponding samples quantities. The importance of sampling is recognized in ISO 17025, (this standard will be discussed in detail in Course 5, Block 3) which requires that test reports make reference to the sampling procedure used by the laboratory or the submitting body.

Check Your Progress Exercise 2

Note: a) Use the space below for your answers.
b) Check your answers with those given at the end of the unit.

1) Sampling plan is very important in sampling. Why?

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2) Mention the purposes that are served by a sampling plan.

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11.5 SAMPLING TECHNIQUES/METHODS

There are several sampling methods/techniques in common use. These are probability sampling, non-probability sampling, bulk sampling, and acceptance sampling. These are described in brief below:

11.5.1 Probability Sampling

Probability sampling is used when a representative sample is desired, and uses principles of statistical sampling and probability i.e. elimination of human bias.

It is a random selection approach that tends to give each unit an equal chance of being selected.

Simple random sampling requires that the number of units in the population be known and each unit is assigned a number. A specific quantity of random numbers between one and total number of population units is selected. Sample size is determined by lot size and potential impact of a consumer or vendor error. Units corresponding to the random numbers are then analyzed as an estimate of the population.

Systematic sampling is used when a complete list of sample units is not available, but when samples are distributed evenly over time or space, such as on a production line. The first sample is selected at random and then every *n*th unit after that.

Stratified sampling involves dividing the population into overlapping subgroups so that each subgroup is as homogenous as possible. Group means, therefore, differ from each other as much as possible. Random samples are then taken from each subgroup. The procedure provides a representative sample because no part of the population is excluded and it is less expensive than simple random sampling.

Cluster sampling entails dividing the population into clusters or subgroups so that cluster's characteristics are as identical as possible, that is, the means are very similar to each other. Any heterogeneity occurs within each cluster. Clusters should be small and having a similar number of units in each cluster. The clusters are sampled randomly and may be either totally inspected or sub-sampled for analysis. This sampling method is more efficient and less expensive than simple random sampling, if populations can be divided into homogenous groups.

Composite sampling is used to obtain samples from bagged products such as flour, seeds, and larger items in bulk. Two or more samples are combined to obtain one sample for analysis that reduces differences between samples. For example, FDA composite 12 and at least six subsamples, respectively, for the sample to be analyzed for compliance with nutrition labeling regulations.

11.5.2 Non-probability Sampling

Non-probability sampling is used when it is not possible to collect a representative sample, or a representative sample is not desired. For example, in case of adulteration such as rodent contamination, the objective of the sampling plan may be to highlight the adulteration rather than collect a representative sample of the population. The sample collector uses judgement rather than statistical considerations in the selection of the sample. The unusual or unexpected characteristics in a population could be selected to be identified. This type of sampling is done in many ways, but in each case the probability of including any specific portion of the population is not equal because the investigator selects the samples without estimating sampling error.

Judgement sampling is solely at the discretion of the sampler and therefore is highly dependent on the person taking the sample. This method is used when it is the only practical way of obtaining the sample. This method may present a better estimate of the population than random sampling if sampling is done by an experienced individual and limitations of extrapolations from the results are understood.

Convenience sampling is performed when ease of sampling is the key factor. The first pallet in a lot or the sample that is most accessible is selected. This type of sampling will not be representative of the population, and therefore is not recommended.

Restricted sampling may be unavoidable when the entire population is not accessible. For example, if sample is to be taken from a loaded truck, but the sample is not a representative of the entire population.

Quota sampling is the division of a lot into groups representing various categories, and samples are then taken from each group. This method is less expensive than random sampling but also is less reliable.

11.5.3 Types of Sampling

A) Bulk sampling

Bulk sampling involves the selection of a sample from a lot of material that does not consist of discrete, identifiable or constant units. Sampling may be performed in static or dynamic situations. Bulk sampling poses special problems requiring certain decisions to be made: the number of increments to be taken, the size of the increments, from where in the pile or stream they should be drawn, the sampling device to be used, and how to reduce the increments taken to a reasonable size of sample for delivery to the laboratory.

B) Acceptance sampling

Acceptance sampling differs from the previous types and involves the application of a predetermined plan to decide whether a lot of goods meet defined criteria for acceptance. The risks of accepting "bad" or rejecting "good" lots are stated in conjunction with one or more parameters, for example, quality indices of the plan. Statistical plans can be designed to regulate the probabilities of rejecting good lots or accepting bad lots.

Refer Tables 11.2 and 11.3 in the annexure.

There are two broad categories of acceptance sampling: sampling by attributes and sampling by variables.

Sampling by attributes

In sampling by attributes, the unit of product is classified as defective or non-defective, or the number of defects in a unit of product is counted with respect to a given requirement. Or, the sampling is performed to decide on the acceptability of a population based on whether the sample possesses a certain characteristic, for example, *Clostridium botulinum* contamination in canned goods. An example of net weight determination may serve to explain the differences between the two categories. In attribute sampling, each unit that weighs 1 pound or more is accepted, and each unit that weighs less than 1 pound is rejected. If the number of rejects exceeds a predetermined number, the lot is rejected. If the number of rejects is less than the predetermined number, the lot is accepted.

Sampling by variables

In variable sampling, sampling is performed to estimate quantitatively the amount of a substance (e.g., salt) or a characteristic (e.g., color) on a continuous scale. The estimate obtained from the sample is compared with an

acceptable value (i.e., previously determined) and the deviation measured. This type of sampling usually produces data that have a normal distribution such as in the per cent fill of a container and total solids of a food sample. In general, variable sampling requires smaller sample size than attribute sampling and each characteristic should be sampled for separately when possible.

11.5.4 Operating Characteristic (OC) Curves

Operating Characteristic (OC) curves are used extensively in acceptance sampling. The OC curve shows the relationship between the quality and the per cent of lots expected to be acceptable for the quality characteristic inspected. In other words, the OC curve is a graph of lot defectives against the probability that the sampling plan will accept the lot. Fig. 11.2 depicts OC curves for an ideal sampling plan.

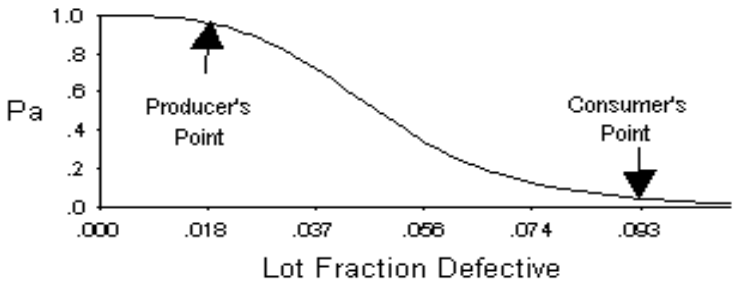

Fig. 11.2: Operating Characteristic Curve

The Operating Characteristic (OC) curve shows the probability of acceptance, Pa, for any level of lot quality. On the horizontal axis is the quality characteristic. This OC curve enables you to evaluate the probability of acceptance for any true lot quality level-on a what-if basis. This way, you can design sampling plans that **perform** the way you want.

We can interpret the curve according to this example:

1) If the lot quality is 0.093 fraction defective, then the probability of acceptance, Pa, is 0.05.

2) If the lot quality is 0.018 fraction defective, then the probability of acceptance, Pa, is 0.95.

11.5.5 Requirements of Good Sampling Methods

Samples are useful for their intended purpose when they are taken in a manner consistent with generally recognized good sampling techniques and good sampling practices. This requires the following:

- Inspection of the lot before sampling.
- Use of suitable sampling devices for the particular commodity and type of sample desired.
- Use of suitable containers to hold the sample.
- Maintenance of the integrity of the sample and associated records.
- Use of adequate precautions in preserving, packing and delivery of the sample to the lab in a timely manner.

- Provision of appropriate storage conditions for the sample both prior to and following analysis.

All of these factors, along with others such as cost versus benefits analysis, and a review of program objectives and regularity requirements, are to be assessed and brought together in a sampling plan that serves as a guide to management, as well as to operating personnel as a firm plan to achieve quality in sampling.

11.5.6 Cost of Sampling

The attention of users is drawn upon relation between the efficiency and size of sample. For a given Acceptable Quality Level (AQL), the smaller the sample size, the smaller the cost of sampling, but the worse the efficiency, that is the risk to wrongly accepting a lot increases and worsens the damage in trade.

11.5.7 Problems in Sampling

Analytical data never are more reliable than the sampling technique. Sampling bias, due to non-statistically viable convenience, may compromise reliability. Errors also may be introduced by not understanding the population distribution and subsequent selection of an inappropriate sampling plan.

Unreliable data also can be obtained by non-statistical factors such as poor sample storage resulting in sample degradation. Samples should be stored in a container that protects the sample from moisture and other environmental factors that may affect the sample (e.g., heat, light, air). To protect against changes in moisture content, samples should be stored in an airtight container. Light sensitive samples should be stored in containers made of opaque glass, or the container wrapped in aluminum foil. Oxygen sensitive samples should be stored under nitrogen or an inert gas. Refrigeration or freezing may be necessary to protect chemically unstable samples. However, freezing should be avoided when storing unstable emulsions. Preservatives (e.g., mercuric chloride, potassium dichromate, and chloroform) can be used to stabilize certain food substances during storage.

Mislabeling of samples causes mistaken sample identification. Samples should be clearly identified by markings on the sample container in a manner such that markings will not be removed or damaged during storage and transport. For example, plastic bags that are to be stored in ice water should be marked with water-insoluble ink.

If the sample is an official or legal sample the container must be sealed to protect against tampering and the seal mark easily identified. Official samples also must include the date of sampling with the name and signature of the sampling agent. The chain of custody of such samples must be identified clearly.

11.6 THREE CLASS SAMPLING PLAN

Another type of sampling used frequently by regulatory agencies to determine acceptance or rejection of a lot (often defined as the quality of product produced under essentially the same conditions but representing no more than one day's production) is the three-class sampling plan. This approach is often used when assessing microbiological contamination of foods. In this case, "n" is the number of samples, usually selected at random from the lot, the

numerical value "m" represents acceptable concentrations, the numerical value "M" represents un-acceptable concentrations, and "c" is the maximum allowable number of marginally acceptable sample units such that if this number is exceeded, the lot is considered as un-acceptable. While "m" separates sample units of acceptable quality from those of marginally acceptable quality, "M" separates sample units of marginally acceptable quality from those of defective quality.

For enforcement purposes, the sampling technique used should be the same as the sampling technique used to set the standard. For example, minimum reportable limits for particles are based on composite samples and not on individual lots.

11.7 PREPARATION OF SAMPLING PLANS

The development of quality sampling plans is a science in itself and has been given consideration by a number of organizations. One plan format that deserves serious consideration, developed by the International Organization for Standardization, is shown with comments in ISO/TC 34, ISO/DIS 7002.2, "Agricultural food products- Layout for a standard method of sampling from a lot "(1988).

It can serve as a starting point or check list for developing a sampling plan for most commodities. The title and headings from sections in the monograph are as below:

11.7.1 Model Sampling Plan

Agricultural food products- Layout for a standard method of sampling from a lot.

1) Title (short but appropriate for index identification)
2) Introduction (describing the purpose of the plan)
3) Scope (describing the breadth of coverage of the plan)
4) Field of application (products to be covered; where sampling will be done)
5) References (documents, the validity of the plan with reference to other requirements)
6) Definitions (specific terms associate with a particular matrix)
7) Principle (statistical basis of the method of sampling)
8) Administrative arrangements
   a) Sampling personnel
   b) Representation of parties concerned
   c) Health, safety and security precautions
   d) Preparation of the sampling report
9) Identification and inspection of the lot prior to sampling (important in survey sampling for identification, condition of the lot and selection of method of sampling).
10) Sampling equipment and ambient conditions (proper tools such as use of sterile equipment for aseptic sampling).

11) Sample containers and packing (essential to prevent contamination and damage during shipment or storage).

12) Sampling procedures (as dictated by the plan objectives).

   a) Sampling size (adequate for all analytical testing to be done. Refer Tables 11.2 to 11.4 in Annexure)

   b) Taking the sample.

   c) Preparation of bulk samples (Table 11.3) and reduced samples (Table 11.4).

   d) Selection of samples of pre-packaged products.

13) Packing, sealing and marking of samples and sample containers (identification of units and to establish chain-of-custody).

   a) Filling and sealing sample containers.

   b) Labeling or marking (including signature of sampling personnel).

   c) Packing samples for storage or transportation.

14) Precautions during storage and transportation of samples.

15) Sampling report

   a) Administrative details.

   b) Details of unit packs or enclosure containing the lot.

   c) Material samples.

   d) Marking and sealing of samples.

16) Annexure (supplemental information, if necessary).

Check Your Progress Exercise 3

Note: a) Use the space below for your answers.
b) Check your answers with those given at the end of the unit.

1) Mention some sampling methods/techniques related food products?

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2) What is probability sampling?

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3) Describe advantages and disadvantages of the plans- Sampling by attributes and sampling by variables?

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4) Write down the application flow of three- class sampling plan?

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5) Mention some basic requirements of good sampling methods?

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6) How much quantity of sample is required for the following items?

Root and bulb vegetables, Cereal grains, Poultry eggs, Liquid milk, Liquid products.

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11.8 SUB SAMPLING FOR ANALYSIS AND TAKING THE TEST PORTION

If the test portion analyzed does not represent the sample or the lot from which it was taken, in that case, even the best analysis could give misleading information. Distortions introduced at this point will carry through the path of analysis and adversely affect the final results and the conclusions drawn from them. There are generally two choices in analytical sub sampling:

- Preparation of a composite laboratory sample (if multiple units are submitted for analysis).
- Examination of individual units.

11.8.1 Composite Lab Sample Preparation

A composite lab sample is one in which the individual units or representative portions of units are mixed to form a uniform mixture. Portions are then taken

from the composite for analysis. Compositing saves analytical time and in some types of contract testing it may be the procedure specified. If the results indicate that there may be a problem, it will likely be necessary to go back and analyze individual samples. Compositing is not the procedure of choice when there is a chance that an individual unit that constitutes a public health or safety threats will not be detected (there are some exceptions) or where a unit at or outside of tolerance level will not be detected because of matrix dilutions. Multiple unit lab sampling is indicated when the possible range of values among individual units is considered significant or it is desirable to establish the variability of the lot.

Refer to Tables 11.4 and 11.5 for the details commodities and quantity of sample required.

11.8.2 Opinions of Experts

You den and Steiner (Statistical Manual of the AOAC, AOAC International, Arlington, VA, p 41) observed that, "Many materials are notoriously difficult to sample. Often the variability among samples is the controlling factor in the confidence placed in the analytical result." They note further that

A mistake sometimes made is to composite several samples and then to run repeat determinations on this composite sample. The analyst may be happy with several results that are in close agreement because only the analytical error is involved in the results. And some may put their faith in the result admittedly, if the individual samples were of the same weight and properly mixed, the same average will result whether the samples are analyzed individually or repeats are made on the composition. Using the composite sample effectively conceals the between-sample variation. It should be mandatory to run the samples individually, for only by doing so will anybody be in a position to make any statistical statements about the results, no matter how good the analytical procedure.

Somewhat similar view of sub sampling for analysis is expressed in an article published in Chemical and Engineering news by an ad hoc sub committee of The American Chemical society for "Dealing with the Scientific Aspects of Regularity Measurements."

This report observes that *the number of samples to be analyzed in a given situation usually is limited by the resources available for collection of samples or for their analysis. However, the reliability of the result generally increases with the square root of the number of samples analyzed. For this reason, analysis of multiple samples are preferred over single samples since, single samples give no information on the homogeneity of the lot that was sampled. In addition, for single samples, the sampling error is also confounded with the analytical error. As a result, if the total number of determinations must be fixed, multiple independent single samples are preferred over replicate aliquots per a single random sample. In any case, the sampling decisions should be a priori decision and should be based on the question at issue.*

In addition to the number of sub samples taken for analysis, it is essential that each be prepared in a way that achieves homogeneity and is handled in a manner that prevents alteration from the original composition. Obviously, failure to prepare a homogeneous sub sample at this point will affect the results of the analysis regardless of the method used.

11.9 SAMPLE PREPARATION FOR ANALYSIS

Every type of material that is to be prepared for analysis presents its own practical difficulties. The requirements for suitable sample preparation are dictated by the consistency and the chemical characteristics of the analyte and the matrix, and by the distribution of the analyte in the sample. Even seemingly homogeneous materials such as liquids may be subject to sedimentation or stratification. Thus, vigilance and care are the watch words to ensure homogenity.

11.9.1 Precautions to be followed while Preparing a Sample for Analysis

Mixing: Single phase liquids can generally be mixed, stirred, shaken or blended. Dry particulate materials can be reduced in the volume by coning and quartering, by rolling and quartering, or by the use of a splitter, such as a refill. A variety of implements and machines are available for sample disintegration, such as mills, grinders and cutters. Care in their use is necessary to prevent loss of dust or change in composition through partial separation of components. Screening can be used to improve the efficiency of particle size reduction, followed by mixing to attain homogeneity. Sampling errors can occur even in well mixed particulate mixtures especially in trace analysis if the particles differ appreciably in size or physical properties.

Cleanliness of equipment used in process

Every piece of equipment used in the preparation of a sample must be examined critically to ensure their cleanliness, so that they do not contaminate or decompose or cause any physical loss of the sample while processing. Grinders were mentioned above as contributing to the loss of finer particles as dust. They have been known to segregate materials with in the mix by size as well, with the finer material, collecting beneath the blade e.g., Metal screens can pass fine particles, but retain powder that adheres to the screen materials. Glass containers and laboratory apparatus can adsorb certain materials and may require surface treatment. Plastic containers can retain contaminants, such as animal hairs, while the rest of the sample is transferred with apparent ease. In the other words, validation of a method of analysis, includes, most certainly validation of the method of sample preparation and storage.

Changes in physical characteristics

Loss or gain of moisture during processing can be a problem. Loss can be minimized by keeping samples covered with plastic or aluminum foil. A cold product can be protected from gaining moisture by allowing the sample to come to room temperature before preparation begins. High fat samples such as nuts may be difficult to grind without clogging up the grinder; one technique that is used is to freeze the samples prior to grinding.

Changes in chemical characteristics

When volatile organic constituents are present in any sample, processing may be difficult and needs special care, e.g. maintaining chilled condition to prevent any loss of volatile constituents. Similarly, in case of photo-sensitive chemicals (e.g. natural product pesticides), it is required to process a sample under darkness to prevent degradation on exposure to light.

Portions for sampling

As a general guide, food samples are analyzed in the form they are commonly consumed. Inedible portions, such as stones (e.g. for mango), nutshells, or fish bones are removed and discarded prior to analysis, and suitable note made of how the sample was prepared. The technique used for setting the standard should be used to ensure comparability.

Sampling for Trace metals

Trace metals analysis can present significant problems, For example, the trace metals can be distributed unequally between liquid and solid phases in pickles, canned vegetables and canned fruits. Obviously, such irregular distribution of metals can pose problems for the analyst in establishing the level of metal residues in the product, as well as for those concerned with setting tolerances. Thus, it becomes necessary to analyze both the solid and liquid phases.

Check Your Progress Exercise 4

Note: a) Use the space below for your answers.
b) Check your answers with those given at the end of the unit.

1) Write down some precautions while preparing a sample for analysis?

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11.10 DIFFICULTIES IN SAMPLING

As mentioned earlier, one of the most difficult problem in sampling from a lot, and in subsequent lab subsamples, is trying to obtain a representative sample for the analysis of aflatoxins in raw agricultural commodities. Aflatoxin contamination exhibits a highly erratic distribution, with a reduction in heterogeneity as the food or feed is reduced in particle size. After it was recognized that there was a high rate of variability and within same samples from the same lot, there was a moment towards the collection of larger and larger samples. Sample sizes started, for peanuts with 1 kg, and the size increased as more reliable results were required by food procedures (increasing sample size reduces the number of good lots that are likely to be rejected and the number of bad lots accepted).

11.10.1 Example for Effect of Sampling on Analytical Result

At the present time in the United States, the sample taken from a lot of shelled peanuts of 144 pounds; three 48 pounds samples with portions taken at random from the lot. Examination in the lab is by sequential analysis with first 48 pounds sample ground in a subsampling mill and test portions examined in duplicate. If the average of the test portions is below the established tolerance (set by US Food and Drug Administration), the lot is passed. If the average is above the acceptance level, the lot is rejected. If the findings fall between the two figures the second 48 pound sample is comminuted and the analysis

repeated. If a decision cannot be made to accept or reject the lot, the third 48 pounds sample is prepared, assayed, and the cumulative results considered. The foregoing example point out dramatically the need for attention to lot sampling, lab subsampling, and sample preparation for analysis. While this is a rather extreme case, it illustrates that sampling problems cannot be ignored or treated indifferently. In Canada, while the specified sample sizes are smaller, ranging from 12 to 20 kg. depending on the commodity and the lot size, and minimum number of sampling sites are also stipulated to address the erratic distribution of aflatoxin contamination.

11.11 SAMPLE ACCOUNTABILITY

11.11.1 Documentation

A laboratory sample is generally the starting point for analytical work. The sample may be delivered by mail, courier, flight, or directly by the collector. It may arrive in any of various containers and conditions: frozen, packed in ice, or at room temperature. The package may be sealed or unsealed, and the sample itself may be spoiled or broken. The sample may or may not be accompanied by appropriate documentation to advise the laboratory regarding purpose, test parameters and the conditions of storage, etc.

Once a sample is received, all the circumstances and conditions must be documented as they could have bearing upon the quality or the significance of the test results. It is important for appropriate quality analysis that sample arrives in proper condition with meaningful documents. Procedures for these must be established, continually reviewed, and enforced, to keep poor sample handling and delivery to a minimum level. To avoid any future legal complications, the laboratories are advised to protect themselves with the cautionary statement in the test report indicating that the results relate only to the sample that was tested.

11.11.2 Chain of Custody Form

In most organizations specific sampling procedures are written and the sample collectors are trained regarding their responsibilities. The first important activity is the documentation to ensure product traceability. The sample should be easily identifiable and placed under seal and packing. Shipping and delivery instructions are followed to effect delivery to the laboratory. The documentation consists of a chain-of-custody that accompanies the sample as it moves through the laboratory and subsequent administrative handling. This form is usually prepared in multiple copies for distribution to various units in the organization, may be supplemented with affidavits, dealer's statements, bills or other relevant information that concerns the sample, its origin, the transfers from one custodian to the next and the sample's significance or importance. Information such as sample number, product name and identification, reason for collection, description of the sample and of the method of collection, size of the lot from which the sample was taken, codes, shipment information, collection date, name of the collector, means of transportation, and whether or not sealed are supplied with the sample. If the sample is sealed, the seal includes the sample number, date the seal was affixed, and the collector's signature. The seal is attached to the package in such a way that it must be broken before the sample can be obtained.

11.11.3 Sample Receipt and Handling

The next step in the sample accountability system is receipt of the sample in the laboratory. A dependable record of sample handling is important so that the sample is accepted by a sample custodian who documents the action by completing a sample accountability record. This document should contain the sample number, the name of the product and date received, indicate who received it, describe the method of shipment or delivery, describe the packages received and their condition, and provide space for recording various storage locations before and after analyses. Deliveries of the sample or portions of the sample to the analyst, and its return, will also be recorded on this form. There will be a signed statement concerning the final disposition of the reserve sample. A two-part form can be used for this purpose; one copy remains with the sample custodian and the other moves with the sample through the laboratory and is used by a supervisor for sample management purposes. Some laboratories use a sample receiving log book for sample control. The information entered in the log book is essentially the same as that described for the two-part form.

11.11.4 Monitoring of Samples

The sample accountability in a laboratory can be monitored by a simple computer program; a unique label should be generated and affixed to the sample container, and all the pertinent sample information should be entered into the computer database. The information entered at log–in becomes part of the data base, which is then built up through the manual or automatic addition of sample handling information and analytical data. Worksheet pages or reports can be calculated and printed, and the data base itself latter queried and manipulated for various information and reporting purposes. Regardless of the recording system used, the analytical information generally reported includes a description of the sample, subsampling procedure sample preparation methods used, deviations from methods, validation and recovery experiments (if performed), standards used, source of reference materials, raw data, calculations and description of the reserve sample and how it was prepared for storage after the completion of the analysis. In addition, pertinent supporting documents such as chromatograms, spectra, and other charts are suitably identified with instruments identification, operating conditions, analyst name, sample number and date. If the reserve sample is sealed, the information placed on the seal is shown in the report. The sample is then returned to the sample custodian to be stored for whatever future action may be necessary, or until the sample is destroyed.

11.12 RETENTION OF SAMPLES AND RECORDS

After an analysis is complete and the results reported, the laboratory needs a written policy for guidance on the retention of the samples and the associated records. For samples and records that may be involved in litigation, the storage period can extend for years. For the majority of samples, fortunately, this is not usually the case. The objective should be to destroy samples as soon as it can be analyzed, with certainty that they will no longer be required for further testing or as evidence. The records may be disposed after they are no longer legally or administratively important.

11.12.1 Identify the Properties of Retained Samples

It is very important for the laboratory management to determine whether or not the materials being discarded are hazardous in nature. Although samples themselves may not be hazardous, acid digestions and organic solvent extractions certainly can be hazardous. Sample management includes the proper disposal of samples and laboratory preparations. Standard operating procedures for samples for sample disposal are essential.

11.12.2 Retention Period

Storage periods, obviously, must be determined by each facility depending on its obligations, but clear policy must be in place to prevent both the destruction of important items, and the accumulation of what is essentially junk. From a quality assurance point of view, the improper destruction of active samples or records is low quality performance in violation of policy, and the Quality Assurance (QA) program must provide a means to detect such actions in an effort to prevent their recurrence.

11.13 CASE STUDY

In a 500 T consignment of imported frozen animal carcasses, 300 T labeled as produced by A and 200 T labeled as produced by B is to be checked for residues.

Assumed facts:

i) The carcasses are from an exporter whose products have recently been associated with excessive residues of Permethrin (fat soluble) and Diflubezuron (non-fat soluble).

ii) Carcasses in lot A have trimmable fat, where as those in lot B do not.

iii) The sampling plan is to provide a 95% probability of detection if 10% of the carcasses contain excessive residues.

iv) There is no legal requirement to prepare replicate lab samples.

v) Sampling records are in hard copy form.

vi) Rendering of fat tissue for extraction of lipid acceptable under national law.

Consequent actions and discussions:

i) The consignment is sampled as two separate, suspect lots, A and B

ii) Table 11.3 shows that 29 lab samples should be taken and therefore, as far as practicable, 29 carcasses are selected at random from each lot.

iii) From each selected carcass in lot A, a minimum of 0.5 kg of adhering fat tissue is taken as a (primary) lab sample and a minimum of 0.5 kg of meat (meat does not include bone) is taken as a separate (primary) lab sample.

iv) The carcasses in lot B have no trimmable fat and 29 samples of 2 kg meat are taken.

v) As each lab sample is taken, it is placed in a new polythene bag, securely labeled and sealed, and the sample record completed. The samples are sent to the lab, ensuring that they do not thaw.

Copies of the sample records are given to the owner/custodian of the consignment. Copies are sent with the samples and also retained by the sampling officer.

vi) Fat tissue lab samples from lot a are rendered, the lipid collected and aliquots (analytical portions) analyzed for Permethrin residues. The results are expressed on a whole fat tissue basis.

vii) Bones, if any, are removed from the meat lab samples, which are minced before the determination of Diflubenzuron residues in analytical portions. The results are expressed on the basis of whole meat without bones.

viii) If meat samples from both lots contain Diflubenzuron ≤ 0.05 mg/kg and all samples from lot A contain < 1 mg/ kg Permethrin, lot B is acceptable and lot a is acceptable with respect to Diflubenzuron residues.

ix) If 3 of the 29 fat samples of lot A contain Permethrin > 1 mg/kg, replicate analytical portions of fat from these 3 lab samples are analyzed. Taking into account the analytical uncertainty, if the results conform that the MRL is exceeded, if the 3 carcasses do not comply with the MRL, where as the other 26 do comply with the MRL.

x) If the entire lot is not to be rejected on this basis, lab samples of fat tissue from the remaining carcasses in lot A may be taken for analysis, in order to separate the acceptable carcasses for those that are unacceptable.

11.14 LET US SUM UP

Actions for the management and control of sampling, sample preparation, and sample analysis are summarized below:

1) Work with appropriate persons to develop sampling plans for the various types of products delivered to the laboratory for analysis.

2) Establish sub sampling procedures for various products, giving consideration to the use of composites or individual unit examinations, based on the variability to be expected among sample units and the resources available for their analysis.

3) Prepare guidelines for sample preparation for analysis that will minimize composition change.

4) Choose the appropriate sampling selection method to achieve the intended purpose of taking samples.

5) Describe subsampling within the lab in written procedures which analysts have the responsibility to follow.

6) Manipulation and preparation must be done with care to avoid losses of material.

7) Develop a system to ensure sample accountability.

8) Provide policy for the management of samples when they are no longer needed.

9) Maintain written procedures for sample disposal taking into consideration hazardous waste regulations.

11.15 KEY WORDS

The definitions of sampling terms used in this document are mostly those specified in ISO 7002. Some of the more commonly used terms in acceptance sampling are described in this section.

Lot : A definite quantity of some commodity manufactured or produced under conditions, which are presumed uniform for the purpose of this document.

Consignment : A consignment is a quantity of some commodity delivered at one time. It may consist in either a portion of a lot, either a set of several lots.

Sample (Representative sample) : Set composed of one or several items (or a portion of matter) selected by different means in a population (or in an important quantity of matter). It is intended to provide information on a given characteristic of the studied population (or matter), and to form a basis for a decision concerning the population or the matter or the process, which has produced it.

A representative sample is a sample in which the characteristics of the lot from which it is drawn are maintained. It is in particular the case of a simple random sample where each of the items or increments of the lot has been given the same probability of entering the sample.

Sampling : Procedure used to draw or constitute a sample. Empirical or punctual sampling procedures are sampling procedures, which are not statistical-based procedures that are used to make a decision on the inspected lot.

Total Estimation Error : In the estimation of a parameter, the total estimation error is the difference between the calculated value and the true value of the parameter. The total estimation error is due to:

i) Sampling error
ii) Measurement error
iii) Rounding-of-values or sub-division in to the classes
vi) Bias of the estimator

Sampling Error : Part of the total estimation error due to one or several of the following parameters:

- The heterogeneity of the inspected characteristics.
- The random nature of a sampling.
- The known and acceptable characteristics of the sampling plans.

Acceptable Quality Level (AQL) : The inspection of a lot using either an attributes or variables sampling plan will allow a decision to be made on the quality of the lot.

AQL for a given sampling plan is the rate of the non-conforming items at which a lot will be rejected with a low probability, usually 5%.

Limiting Quality (LQ) : For a given sampling plan is the rate of non-conforming items at which a lot will be accepted with a low probability, usually 10%.

Sampling Plan : A pre-determined procedure for the selection, withdrawal, preservation, transportation and preparation of the portions to be removed from a lot as samples.

11.16 ANSWERS TO CHECK YOUR PROGRESS EXERCISES

Check Your Progress Exercise 1

Your answer should include following points:

1) The quality of a small sample analyzed is attributed to the lot, which the sample represents. If the sample does not represent the population adequately and efficiently, then the results obtained may not truly represent the quality of the lot.

2) Homogenous population would be uniform and identical at all locations. These populations/samples in which the composition would vary at different locations is heterogenous population.

Check Your Progress Exercise 2

Your answer should include following points:

1) It is very important to arrive at a well designed sampling plan because it is a guide to the people who are going to perform sampling. It also serves as a reminder of the important elements in the over all analytical program.

2) a) Sampling plan is a guide for the whole analytical program.
   b) It is a means for operating on a planned basis which reduces variation.
   c) It serves as a reference document for similar activities in the future.
   d) A document for comparison of performances against objectives.
   e) It also serves as a source for imparting training.

Check Your Progress Exercise 3

Your answer should include following points:

1) i) Probability sampling,
   ii) It is a means for operating on a planned basis which reduces variation,
   iii) Non-Probability sampling,

iv) Bulk sampling,

v) Acceptance sampling,

vi) Sampling by attributes, and

vii) Sampling by variables.

2) Probability sampling is used when a representative sample is desired, and uses principles of statistical sampling and probability, a random selection approach that tends to give each unit an equal chance of being selected.

3) Sampling by attributes:

Advantages: 1. No condition on the mathematical law of distribution of the variable inspected. 2. Greater simplicity of the processing the results on the sample.

Disadvantages: 1. Less effective than variables plans for a same sample size of n increments (Least Quality, LQ is higher). 2. More costly than variables plans because the collected sample requires more increments than those required, for the same efficacy, by a variables plan.

Sampling by variables:

Advantages: 1. More effective than attributes plans for the same sample size of *n* increments (LQ is lower) for the same AQL, they are less expensive than attributes plans because the sample collected requires fewer increments than those required for a same efficacy, by attributes plans.

Disadvantages: They cannot be used in all cases because to validate the calculation formulas of the inspected variable must necessarily or approximately follow a normal law.

4) Set the values of m, M, n and c → Collect the sample with n items → Inspect each item in the sample → Accept the lot if: number of marginally defective items (i.e. a concentration of micro-organisms between m and M) ≤ c. Immediately reject the lot if the concentration of the micro-organisms in any item > M and / or the number of marginally defective items > c.

Where, m: accptable concentration; M: unacceptable concentration; c: maximum allowable number of marginally acceptable sample units; and n is the number of sample units selected randomly from the lot.

5) a) The lots shall be thoroughly inspected before sampling to design a good sampling plan.

b) Suitable sampling devices shall be identified and used while sampling.

c) Compatible containers shall be identified and used. The material of the container shall not cause any undue contamination to the quality of sample collected. For example, Non-sterile containers shall not be used while the sample has to undergo microbiological tests.

d) Suitable packing and delivery method.

e) Provision of appropriate environmental conditions. For example, When there is a need to determine Volatile Organic Constituents in a sample of water, it has to preserved/transported at a temperature 4 to 8^{o}C.

6) Root and bulb vegetable- 5 kg, cereal grains- 2 kg, poultry eggs- 12 eggs, liquid milk- 0.5 L, liquid products- 0.5 L

Check Your Progress Exercise 4

Your answer should include following points:

1) a) Use a suitable method for homogenization. For example, Liquids can be homogenized by stirring, shaking or by blending and take an aliquot. Solids can be homogenized by grinding, pulverizing and volume reduced by coning and quartering.

   b) Use clean and suitable sampling devices and containers. For example, A scoop used for sampling a food shall be sterile.

   c) A glass container is not compatible for collecting water sample intended for the determination of metals, since metals like sodium are absorbed by glass.

   d) Moisture content of sample changes with surrounding temperature. Suitable precautions shall be taken to retain the originality.

   e) Maintain suitable environmental conditions to minimize expected chemical changes if any. For example, A food sample for microbiological enumeration shall be collected in a sterile container and be stored/transported in chilled condition.

   f) If the sample is presented in both liquid and solid phases, homogenize both phases before a test portion is taken.

11.17 SOME USEFUL BOOKS

Nielsen, S. Suzanne (2003). *Food Analysis Laboratory Manual*, 3rd Edition, CHIPS Publishers, U.K.

International Organization for Standardization (ISO): www.iso.org

Codex Alimentarius (CODEX), www.codexalimentarius.net

Agriculture and Processed Food Products Export Development Authority (APEDA), Ministry of Commerce and Industry: www.apeda.com

Bureau of Indian Standards (BIS), Ministry of Consumer Affairs, Food and Public Distribution: www.bis.org.in / www.fcamin.nic.in

Export Inspection Council of India (EIC), Ministry of Commerce and Industry: www.eicindia.org

Prevention of Food Adulteration Act (1954), 24th Edition, 2003, Ministry of Health and Family welfare: www.mohfw.nic.in