Chapter 19

Determining the Quality of Raw Peanuts and Manufactured Products

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A. Introduction

The rise of the consumerist movements have intensified the significance of quality and quality control for consumer products of all kinds including peanuts and peanut products. The influence of this movement has reached all segments of the industry so that no phase is exempt or can claim immunity from it. The peanut industry has taken an enthusiastic and responsible approach to matters of public health and safety as well as to providing a higher quality nutritive food source. This is particularly exemplified by the formation of the Peanut Administrative Committee (PAC) under the auspices of the U. S. Department of Agriculture in the control of aflatoxin. The industry has also been productive in promoting quality through the American Peanut Research and Education Association (APREA) and its predecessor, the Peanut Improvement Working Group (PIWG).

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A broad concept of quality will be taken in this chapter since the subject encompasses so much of the technology and production techniques of peanuts and their products. The further importance the consumerists movement adds to this subject highlights the necessity that each area and field of specialization in the peanut industry give more than lip service to this subject. There are three phases to qualify planning and control that must be considered for a total quality philosophy, namely, 1) defining the quality level needed or desired, 2) measuring the desired quality and 3) the decision or evaluation phase.

The first phase, defining quality, is possibly the most difficult of all three. Top management has to be concerned. Too often directives come in terms of negatives after the damage has been done. Thus a good quality engineer or researcher has to read the intent of top managers and marketing departments and anticipate problems in order to accomplish the desired ends. This chapter makes no attempt to define quality although a number of suggestions will undoubtedly come to the reader as he reviews the methods available. On the other hand, there may be quality criteria for which no answer is provided herein. In such a case, the reader is referred to the references given at the end of this chapter. If this is insufficient, he will have to rely on his own ingenuity in developing the needed technology. Another good way to obtain definitions for quality criteria is from ones customers. For industrial products this may come in the form of requests or specifications. Producers of consumer products have a greater problem in that the number of customers is so much greater and their quality demands are ill defined and much wider in scope. Many of the consumer questionaires and panels discussed later can be helpful in this area.

Interpretation of consumer needs should be a joint effort between the marketing, technological and manufacturing segments of the operation to insure reasonable and attainable demands that will meet the criteria of the consuming segment that is being courted. Probably, the most prolific number of quality criteria in a consumer marketing operation will be negative ones dealing mostly with public health and safety, i.e., no aflatoxin, no pesticides, no foreign material, no packaging defects, etc. Then too, different segments of the industry will require different quality criteria. In an attempt to generalize quality criteria, Sexton and the Quality Committee of the PIWG (89) have listed a number of quality factors which they felt were important to the industry.

Most of this chapter is concerned with the second phase of the quality philosophy, i.e., measuring quality. This is done in a review manner since there is insufficient room to describe the methods and statistical techniques in detail. If the reader is not familar with the methodology, the details can be obtained from the references. Much stress has been given to statistical techniques in this chapter. This was done intentionally since quality is usually a relative quantity. Thus, it is important to know the precision as well as the accuracy of a method. In some cases there is poor precision but through proper experimental design and data interpretation, differences can be detected. Sampling is also discussed since in most cases the quality of a lot or similar distinctive group of material is measured on a sample representing the lot. Qualitative methods often require a nonchanging standard and a subjective comparison and, thus, have limited application. The choice of a method will be determined by the type of information required, the cost of the method, the available amount of time, and the need for accuracy and precision. The simplest method is usually the best. For instance, Siggia (91) makes some interesting observations on fads in analytical testing, which would indicate that intricate apparatus can often become a fetish. On the other hand, some characteristics and quality informa-

tion cannot be adequately obtained without the use of expensive and elaborate instrumentation.

Special attention has been given to experimentation and particularly statistical techniques for experimentors in this chapter. Since improved quality is often the goal of laboratory or peanut research, the results of experimentation is most important to the subject material.

The third phase of the quality philosophy i.e. the evaluation and decision phase, should be relatively simple if the first two phases have been adequately followed. If the problem is properly defined and the quality characteristic is quantitatively measured, then the indicated action should be evident. However, there are several considerations which will make this difficult. First, not every quality goal may have been attained or competing materials may have superior qualities in one aspect and not in another. Thus, a system of priorities or weights have to be established. This leads to the possibility of trade offs which is often necessary particularly when one characteristic can be improved only at the cost of another. A third consideration has to be given to practicality. What if a certain quality level cannot be attained or what if a substandard lot has been delivered and no other material is immediately available? Most people concerned solely with quality do not like to compromise in such situations yet it is sometimes necessary to do so. A fourth consideration often overlooked or maybe more accurately ignored is risk considerations. It has been mentioned that statistical criteria are necessary to obtain significance from analytical tests and research experimentation yet there is always the chance for a wrong interpretation. This is particularly true when testing for low levels of contaminants. It seems methodology can usually be developed that will detect minute quantities in a sample but the sampling problems usually are forgotten until trouble arises. Aflatoxin and salmonella are two instances where this has been true in late years. Even at that, there is a risk factor which good management will recognize and weigh with other alternatives.

Finally, *quality is value.* It costs something to attain it and it brings greater returns in income to an operation when it is present. Therefore, the cost of any quality program must be weighed in relation to the enhancement of the quality of the finished product. Sometimes, this is defensive since competition or government regulations require action. In such cases, the product may not bring a higher price than the competitors but will insure a continuance of the business.

B. Statistical Techniques

The ultimate goal of performing tests on peanuts or peanut products is to obtain a measure of some particular quality characteristic of the material. Thus it is necessary to know how to interpret the data and the significance of the data. Since this is basic to sampling and methodology it will be discussed first.

1. *Mean and Standard Deviation*

The most common and familiar statistic is the average or mean. There is a good likelihood that two measurements on a single lot or sample will not be exactly identical. The average of the two is considered a better measurement of the true condition of the lot as a whole than either one of the determinations by itself. In most cases, the distribution of the value of individual determinations will fall equally around the average. However, there are few isolated instances where there is a skewed distribution, i.e., where there are more values on one side of the average than on the other. In these cases, it is often enlightening to study the median which is the value of the test where half the values are below and half are above it.

The spread of the distribution about the mean is commonly described by the standard deviation. The standard deviation, σ, is calculated by the formula: where x_i

$$\sigma = \sqrt{\Sigma(x_i - \bar{x})^2/(n-1)}$$

is the value of any individual determination, $\bar{x}$ is the average of all the results, and n is the number of observations made. The sign Σ signifies the addition of all the individual terms behind it. Another form of this equation which is easier to handle on desk computers is as follows:

$$\sigma = \sqrt{(\Sigma x^2 - (\Sigma x_i)^2/n)/(n-1)}$$

The standard deviation informs the experimentor of the width of uncertainty that he has in any individual determination. Two thirds of the results of a given population will be within plus or minus one standard deviation. 95% of the values will lie between plus or minus two standard deviations. 99% of the values will lie between plus or minus 3 standard deviations. The experimentor should measure or know the standard deviation of any test which he is using. This can be done by running multiple tests on a given sample and obtaining the standard deviation as given above. It is also advisable to use more than one sample for doing this to include sampling variation which is part of the total uncertainty in characterizing a material.

To obtain the best estimate of the standard deviation of a method from two or more independent sets of results, the variance has to be averaged not the standard deviation. The variance is the square of the standard deviation and is averaged by adding the independent values and dividing by the number of values in the set. The square root of the average variance is the pooled standard deviation. In some cases, the number of determinations in each set may be different in which case the weighted average of the variance is used. Thus the pooled standard deviation is calculated as follows: where σ_i is the

$$\sigma_t = \sqrt{\Sigma(n_i \sigma_i^2)/\Sigma n_i}$$

standard deviation of the samples and n_i the number of tests on each of the samples to determine the overall standard deviation in a method from several sources, a fresh sample taken for each new test will include the contribution to the variability from the sampling procedure and calculated operator and interlaboratory variability can also be found.

A similar approach is used to find the variability from each of the components of a test procedure. Again, the overall standard deviation is not the sum of the individual standard deviation, but rather the square root of the sums of the variances. Mathematically this is: where σ_t is the standard deviation of the total procedure, σ_s is the stan-

$$\sigma_t = \sqrt{\sigma_s^2 + \sigma_m^2 + \sigma_a^2}$$

dard deviation of the sample, σ_m is the standard deviation of the method and σ_a is the standard deviation of the analyst. Other causes of variability can be calculated by using this principle. For instance, if there is some suspicion that a given step in a method

might be causing more variability than the rest, an experiment can be designed where this step can be modified and the variability before and after the modification noted.

2. *Test of Significance*

The standard deviation is useful in determining whether a lot is substandard or if two lots are different from each other with respect to a particular quality aspect or not. A common way of doing this is by use of the t statistic. To use this statistic the following

$$t = (\bar{x}_1 - \bar{x}_2)/(\sigma/\sqrt{n})$$

equation is applied: where $\bar{x}_1$ is the average on lot 1, $\bar{x}_2$ is the average on lot 2. The numerical value of t is compared to the given values of this statistic as found in most texts on statistics for the number of degrees of freedom used in the tests. If the value found is greater than that in the table, the difference is significant. The tables give t at various probability levels. The experimentor must select the level of the risk he assumes to be safe. If he wishes to be certain that there is a small risk of the two lots actually being the same when he says there is a difference, he will assign a high probability level (β risk) when taking his t value. As an example, two lots of peanuts measured in duplicate had a difference in moisture content of .5% when examined by the Steinlite Moisture Tester. The standard deviation of this method is 0.10% and equation (5) becomes:

$$t = 0.5/(0.1/\sqrt{2}) = 7.1$$

The t value for 95% confidence and 1 degree of freedom is 6.3. Since the above t is greater than this, it is safe to assume that the lots are different, since there is less than a 5% chance that the lots are the same. It should be noted that the value of t becomes larger with more replicas because of the term $\sqrt{n}$ while the tabular value of t becomes smaller, therefore, the distinctiveness of a treatment can be determined more accurately when small differences exist by running many tests. Thus, if one is not satisfied with the first conclusion, more tests can be made, recalculate equation (5) and compare to the tabular value of t to see if there is then a significant difference.

3. *Precision and Accuracy*

The discussion so far has been concerned with precision or the ability of a method to reproduce itself. Accuracy is the important criteria, although good precision is usually an indication of good work which in turn gives good accuracy. However, an analyst obtaining consistently small differences between results on a given lot of product is not necessarily obtaining the correct value or the one that best describes the lot. Precision is easy to measure as pointed out above. Accuracy is usually determined by running analysts on material of known composition or comparing the results of a given method with another method the accuracy of which is known. However, the absolute condition of any quality factor is rarely that well defined or known. Furthermore, some methods are empirical in nature and require strict adherence to a set of exact experimental conditions in order to obtain reproduceable and accurate results. As an example, the Penetrometer method of consistency on peanut butter can be cited. The shape of the needle or cone that pierces the surface of the material and the weight of the plunger are critical factors in the reading obtained. One set of conditions might suit one manufacturer better than another and each must set his own standards and paramaters of the test

to be used. Other methods use reference material to standardize the test procedures. For instance, the Steinlite Moisture Tester requires standardizing the instrument by making readings on different lots and comparing the results as found by a standard procedure, such as the oven procedure. Standard materials are now available for standardizing the Steinlite which simplifies the process.

TABLE I

Errors and Risks Associated with the Measurement of Quality.

Condition of Lot is	*Test Indicates Lot is*	
	ACCEPTABLE	UNACCEPTABLE
ACCEPTABLE	NO ERROR	PRODUCERS ERROR
UNACCEPTABLE	CONSUMER'S ERROR	NO ERROR

4. *Error and Risk*

It is possible to distinguish between two types of error that can be made in measuring quality characteristics. It will be useful to the analyst and experimentor to recognize the possibility of these errors as depicted in table I. If the product is acceptable and the test indicated it so, there is no error. Likewise if the product quality is unacceptable and the test so indicates it, there is no error. However, if the quality of the lot is acceptable and the test indicates that it is unsatisfactory, there is what is referred to as a type I error. On the other hand, if the test indicates the product is acceptable when it really is not, then there is a type II error. The type II risk is also referred to as the consumers risk and the type I risk as the producers risk. The reason for this is obvious since the possibility of accepting a lot which in reality is over the specifications is detrimental to the buyer or consumer. Likewise, the probability of rejecting a lot which is actually acceptable hurts the producer. Therefore, when a specification is set and a procedure established to test for a given quality level, the corresponding risks should be ascertained and the procedure designed so that these risks will be reasonable. Note that the risk can be reduced by taking more samples and more tests to get a better average value which will come closer to the true characterization of the lot. More sophisticated control procedures will have dual or multiples sampling plans. If the first sample shows the lot to be very good or very bad, it can be accepted or rejected immediately. However, if the analysis falls in a mid range where the possibility of taking an unacceptable producers or consumers risk is possible, then a second and possibly a third or fourth analysis is made, the results averaged and the average used to determine the disposition.

5. *Operating Characteristic Curves*

Operating characteristic (OC) curves are means of depicting the relationship between the specification and the probability of accepting a given lot of known quality level. The ordinate gives the probability of accepting a lot while the true condition is given on the abscissa. Although the true condition of the lot may never be known, it is possible to determine the probability of accepting a bad lot or rejecting a good lot from the standard deviation.

An illustration of an OC curve can best demonstrate its usefulness. The sampling risks can be calculated of an attribute sampling plan associated with determining grade

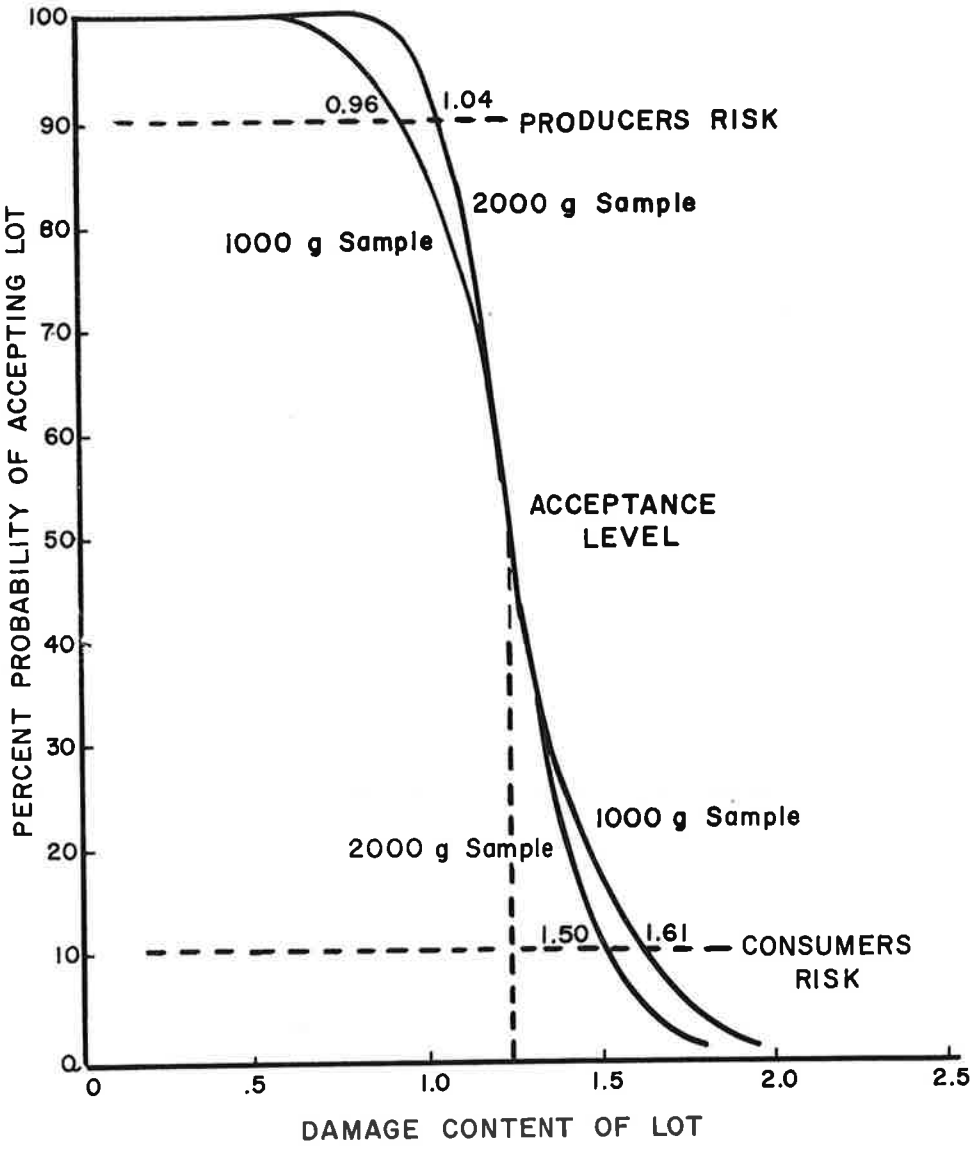

Figure 1. Operating Characteristic Curves for Sampling of Shelled at an Acceptance Level of 1.25%.

factors. The variability from the sampling only will be used since it probably is the greatest portion of the error that exists. All grades of peanuts to meet U. S. grade qualification must have less than 1.25% major damage. Two sample sizes are given, 1000 and 2000 grams. Using the theory of binomial probability, the variability to be expected when a lot has 0.8, or 0.9, . . . or 1.8% damage can be determined (see equation 10). From this, the area under the normal distribution curve lying below 1.25% can be calculated. This will be the probability of lots with 0.8, 0.9, . . . 1.8% damage being accepted. These, in turn, can be plotted to give the OC curves shown in Figure 1. From this curve the probability of accepting a lot with 1.5% major defects is 10% with a sample size of 2000 grams and 17% with a sample size of 1000 grams. If the experimen-

tor decided that this level of major damage was particularly dangerous for his use, he might wish to hold the probability of accepting such a lot at 5% which would necessitate a larger sample than either of the two. The OC curve passes through the acceptance level at .50 probability level. A lot with a true value exactly at the acceptance value will have a probability of being accepted half the time. This is not true for all OC curves; those with skewed distribution will vary in this regard. (Tiemstra 105). The OC curves illustrate the value of multiple sampling plans as they are being applied to quality control situations. If the original test on a lot is below or over the area of uncertainty, the disposition of the material can be made immediately basis the test. In the area of uncertainty around the acceptance value, the experimentor may wish to know more about the material before he accepts it or rejects it. Therefore, he can resample and reanalyze the material to gain a second value. Such sampling plans decrease the consumer and producers risk without appreciably increasing the number of samples and tests that have to be made. Such economy of effort is rewarding to those who have to cope with high laboratory expense.

Several statistical techniques have been sketchily described. These have been included to remind the reader that there is uncertainty in making objective measurements and to acquaint him with the jargon and techniques which can be used to handle such variation. Too often a numerical value of a test result is taken as absolute. This will lead eventually to wrong conclusions and shake the experimentors confidence in the method or himself. Statistics will help the experimentor in

. . . evaluating the methods
. . . comparing lots
. . . determining the goodness of a lot as regards a specification
. . . setting meaningful specifications
. . . determining optimum sampling conditions

Therefore, statistics will give more confidence in the results of a test. To master these techniques, the reader is referred to one of several excellent texts given in the bibliography (44, 53, 57, 67). Particular emphasis is placed on Youdens (123) and Hinchen's (46) booklets since they are extremely practical, short and inexpensive.

C. Experimentation

The measurement of quality in a lot is, in reality a very small part of quality measurement or control. Quality is the result of processing by machines or man. Therefore, it is often necessary to measure the efficiency of such factors and detail the conditions under which a process is to be conducted in order to obtain optimum results. The science of process capability and evolutionary operation are two techniques which have found their way into modern industry to improve these factors.

An illustration of process capability study can best demonstrate its value. Blanchability is a difficult, if not impossible, factor to measure in the laboratory on a sample basis. This does not mean that it cannot be evaluated. First, a method such as will be described later must be developed to determine the "degree" of blanching that has been accomplished. Then the "laboratory" can be moved into the plant and the effect of given plant operations studied on different lots of peanuts. Or it may be desired to know what roasting or blanching conditions are optimum to good blanching. Again, the plant is the best place to study this. Unfortunately, such experimentation usually takes

time since the experimentor will have to collect data on several lots and evaluate each lot before he can make a judgment as to the effectiveness of the variable or the condition under which it was done.

In the few paragraphs that follow, the experimental approach to determine the significant factors and the quantitative effect these factors have on a given response will be discussed. In this chapter, a variable is an input or experimental condition. Usually a variable can be controlled if it is recognized as an important factor in the experiment, but in certain cases they are uncontrolled because their effect is not anticipated or it is too expensive to control them. Their effect can usually be measured if the variable can be measured. The response is the quality factor being sought. The evolutionary operation (EVOP) process will be briefly described which has been found very useful when optimizing conditions in manufacturing processes which ultimately result in improved quality. Regression analysis will also be described briefly since this is a very useful tool in gathering data from many diverse points with many variables which can then be put together and give a quantitative estimate of the importance of the variables. Such techniques might seem sophisticated for many applications, however, once they are learned, they will be found to be an invaluable tool for the serious researcher and experimentor who wants to obtain the most out of the data that he collects. Even if one rarely uses the techniques, the reader will develop understanding and confidence in reading technical reports where they are used.

1. *Experimental Design*

A properly designed experiment is essential for evaluating a method or a quality characteristic of a lot when no existing methodology is available. Many methods are simple and straightforward and can be readily evaluated. Even at that, such things as the adequacy of the sample to represent the lot, as well as the actual test procedure, can be examined independently if the experiment is properly designed.

a) *Block Design*

One of the better designs for a single factor is the block design. Block design minimizes the effect from variables often associated with biological systems. This is particularly true in agronomic studies where soil conditions, rainfall, climatic conditions, can affect the result. To minimize the variation from these sources of error, a block design would be commonly used. If four varieties of peanuts were to be evaluated and four fields are available, each field could be sub-sectioned into quardrants. One variety would be assigned to each quardrant in the field but they would be varied in position so that each variety would be placed in a different relationship in each field to randomize the position error as to wind direction, sunlight, drainage, etc. Another example of such a design would be the study of a given treatment on a particular variety of peanut. Several fields, with different soil types, could be chosen. The treatment would be added at one or more levels in each of several plots in each field. All other conditions would be kept as constant as possible. Thus, the cumulative results from all fields would give an excellent indication of the effect of the treatment.

b) *Factorial Design*

A second type of experimental design is the factorial design. All combinations of several variable factors at several levels of use are tested. The number of tests necessary is given by the number of levels of each variable raised to the nth power, where n is the number of variables. For instance, if a variety of peanuts were to be grown under conditions to test the effect of fertilizer and herbicide, each at two levels, the herbicide could be tested at one pound and two pounds per plot, and the fertilizer could be used

at ten pounds and twenty pounds per plot. n in this case, would be 2 and it would be a factorial experiment of the 2^2 type and four tests would have to be run. It will take four plots to have all combinations for the two variables being studied. If it was desirable to test the variables at three levels, it would be necessary to have nine fields for all the possible combinations. This is a 3^2 factorial experiment.

Three levels are frequently used to test for non-linearity. In most systems, such as fertilizer utilization, there is an optimum amount of fertilizer to use in order to get the maximum yield at the lowest cost. Therefore, only two experimental points would indicate a straight line relationship when actually a curvilinear relationship might be present. Another way to find non-linearity is to run the 2^n factorial experiment and add one additional test at the mid-point between the two extreme levels. An advantage of a factorial experiment is that is tests for interactions between variables. For instance, if there is a definite increase in yield going from ten to twenty pounds of fertilizer per plot and there is an increase of yield in going from one to two pounds of herbicide per plot, it normally would be expected that there would be an additive effect of both, i.e., the use of more herbicides and fertilizer would cause an increase of yield of the sum of the two individual factors put together. In many instances, this is not the case. The increase that might be found in yield with the fertilizer might only be slightly improved by adding herbicides. On the other hand, the opposite effect might be true also,

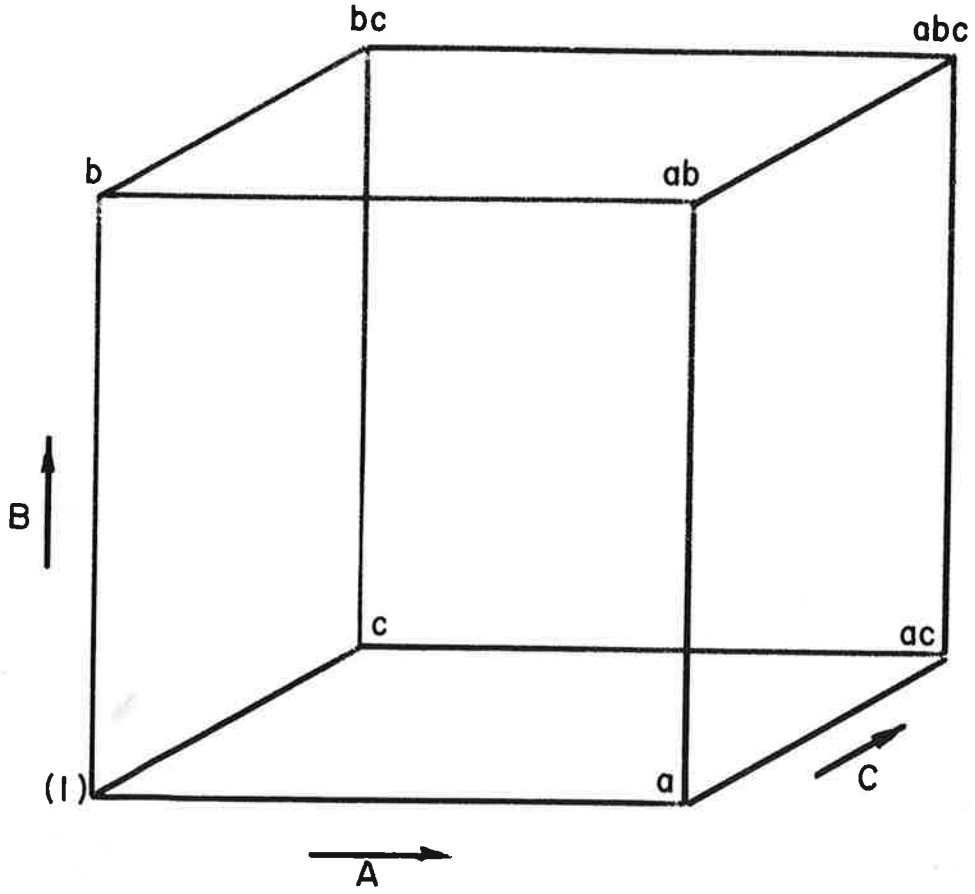

Figure 2. Three Dimensional Representation of a 2^3 Factorial Experimental Design.

i.e., the increase in yield using both fertilizer and herbicide might result in a greater yield than what would be expected from just the additive effect of the two.

As the number of variables that are to be studies become greater, the number of experiments that have to be run grows exponentially. If five variables are to be tested, it will take 25 experiments in order to conduct all the possible combinations. Obviously, the designs become so intricate and involved that they become impractical or there is not enough room to make all the possible combinations. In such cases, it is possible to run a partial factorial experiment. To visualize this, the three dimensional space diagrams of a 2^3 factorial experiment is shown in Figure 2. This is a cube where the levels of each of the three variables are described in space along one of the three axis. Thus, the lower front left-hand corner of the cube would be the experiment where all the factors are present at the low level. Going to the right from this reference point, increases variable A. Going up from this reference point, increases variable B. Going to the rear from this reference, increases variable C. The usual notation indicates the presence of a variable at the high level when its lower case letter is written. Wherever the letter is absent, that variable that is in the design will be present at the lower level. Studying Figure 2 will indicate that each factor will be present at the high level four times and at the low level four times in all of the eight experiments. Thus, the main effect of any one variable can be estimated by adding the four experiments where it is present at the high value together and subtracting the sum of the four experiments where it is present at the low level and dividing by four. Likewise, all the two factor interactions can be estimated in this design. It would appear that the three factor interaction is also available, however, it must be remembered that the experimental error is not known, and so, it is impossible to know whether the three factor interaction is significant or if it is just experimental error. This is known as confounding the three factor interaction with the error estimate. Duplicating several of the experimental points will give an estimate of the error and then the three factor interaction can be tested for significance.

If eight experiments are too many for the space or time available, half the experiments can be run and the main effects estimated. For instance, (1), ab, bc, and ac would be a one-half replica of the 2^3 factorial experiment. Each factor must be present in half the experiments at the high level and in the other half at the low level. Thus, the main effects can be estimated as indicated above. However, now it is impossible to obtain an estimate of any of the interactions since they are confounded with the error estimate. Partial factorial experiments of higher power can be run and some of the interaction effects preserved. For instance, in a 2^5 factorial experiment, the complete design would require 32 tests. A half replica of 16 tests could be run which would give a measurement of all the two and three level interactions. The four level interaction would be confounded with the error estimate.

Partial factorial experiments are very common when a large number of variables are to be investigated. This can be done conveniently if the experimentor has some prior knowledge or some intuitive reason to believe that there are no interactions of particular factors or interactions of large numbers of variables. By carefully selecting the interactions which can be confounded without losing valuable information, economy of time and effort can be made. Since it is rare that there are high levels of interaction between a number of variables, it is usually desirable to confound the high order interactions and try and maintain as much distinction between the individual variables or two factor interactions. A factorial experiment does not normally give estimates of error. Therefore, it is often desirable to duplicate several points in order to obtain an error estimate and

TABLE II

Nested Design and Analytical Results of Moisture Analysis on Shelled Peanuts.

Lot	I				II			
Samples	1		2		1		2	
Methods	A	B	A	B	A	B	A	B
Duplicates	8.2	7.9	8.4	8.3	6.9	7.4	7.6	6.9
	8.1	7.9	8.1	8.4	7.1	7.0	7.5	7.5

to test the significance of the various factors being investigated. Naturally, the more repetition that is done, the better estimate of the standard deviation will be obtained and a smaller difference can be estimated with a greater degree of significance. Often an estimate of the error is made from the higher level interactions assuming them to be non-existent which they often are.

A special case of the factorial experiment is the nested design. Nested designs are very convenient for determining the variability of steps in a method or sampling plan. A simple illustration of a nested design is given in Table II. Two lots of peanuts were evaluated for moisture by two methods, the Steinlite and vacuum oven. Each lot was sampled twice so that a sample variation could be estimated from the results. Duplication of the analysis of each gives a measure of experimental error. The difference between the two methods can be ascertained because each method was tried on each sample. It will be noted that this is a 2^4 power experiment with sixteen results. In this case, the variables are not quantitative levels of a variable but are sample differences, methods and duplicates. It is nested because each variable is represented in each other variable and yet individual effects can be determined.

2. *Analysis of Variance*

As important as designing the experiment is the analysis of the results obtained from the experiment. The calculations will be briefly described but the main intent is to stress the importance of the analysis of variance (ANOVA) tables and its significance to the experimentor. The example just cited on the moisture test will be used to illustrate the procedure.

TABLE III

Analysis of Variance (ANOVA) Table of Data from Nested Experimental Design Given in Table II.

Source	*df*	*ss*	*m.s.*	*components*	*F*
Lots	1	3.4225	3.4225	$\sigma^2_D + 2\sigma^2_M + 4\sigma^2_S + 8\sigma^2_L$	22.64**
Samples	2	.3025	.1512	$\sigma^2_D + 2\sigma^2_M + 4\sigma^2_S$	3.16N.S.
Methods	4	.2350	.0589	$\sigma^2_D + 2\sigma^2_M$	1.39N.S.
Duplicates	8	.3400	.0425	σ^2_D	
Total	15	4.3000			

**Significant at the 99% level.

The results from Table II are used in calculating the ANOVA table shown in Table III. An expanded form is used here to help the reader understand the full scope

and value of this method of analyzing the data. In the first column, the source of variation is indicated. The second column headed by "df" is the degrees of freedom associated with each of the variables tested. In the third column (ss) is the sum of squares associated with each of the variables. The fourth column (m.s.) is the ss column divided by the degrees of freedom or the mean square. The mean square is the total variance of the experiment to that point as described by the components of variance which is shown in the fifth column. The fifth column is often omitted (ss) since these are actually considered extraneous to the important features of the ANOVA table. However, the components of variance is important in understanding the F statistic. The F statistic is used to determine which variables are significant and which are not. To obtain the F statistic, the ratios of the mean squares are taken starting with the mean squares for duplicates which is compared to the mean squares between the methods. If there was no difference between the two methods, the variance of methods (σ_m^2) will be near zero and the ratio of the mean squares will approach one. Thus an F value of one indicates no significant difference in the methodology and thus that particular variable would be non-significant. This is indicated by "ns" shown after the F ratio. However, if this ratio should be greater than one, the value would have to be compared with those in the F statistic tables to see if it was large enough to show that the variable was truly significant. Two values for the F statistic are usually given, one for a 1% significant level and the other for a 5% significant level. The F ratio will have to be larger to show a 1% significance level than for a 5% level. Basically the significance level of the F statistic refers to the probability of having a type I error or assuming there is a difference when there really is none. Thus at the 1% level, there is only a 1% chance that the variable is not significant when the table says it is. Likewise, at the 5% level there is a 5% chance that the variable is not significant when the ratio is of that magnitude. Table III indicates that there is no significance between methods or samples but there is a highly significant difference between the lots.

A factorial experiment is analyzed in the same way with ANOVA tables. The reader is referred to Hicks (45) and Davies (29) for a more detailed description of experimental designs and ANOVA tables.

3. *Response Surface Methodology*

The experimentor not only desires to know what factors are important but often wishes to know the optimum conditions for running a process. What is said here of testing for quality can be said equally well for many phenomena of cause and effect relationships either in industrial or agronomic application. Since we are interested primarily in testing for quality, our discussion will be aimed at that goal.

One of the most useful tools for finding optimum conditions is the response surface methodology (RSM). This can be visualized most readily by a two dimensional or two variable system. Each variable is plotted along one of the axises and the response is shown as contours of isobars much the same as elevation can be shown on a map by contour lines. Many types of optimum conditions either maxima or minima, can be found such as a ridge, saddle, peak, etc. To find the optimum conditions, the experimentor has to "creep" over the surface of this "terrain" to find the crest of a hill. All he knows is what he can feel. First, he will find the steepest slope in his immediate area since this will usually lead him directly towards the peak and he will climb this making adjustments as he goes along. So too, the experimentor can investigate a relatively small area and find the direction in which the greatest improvement takes place. He can then follow this line until he finds the maximum. When he finds the maximum he once

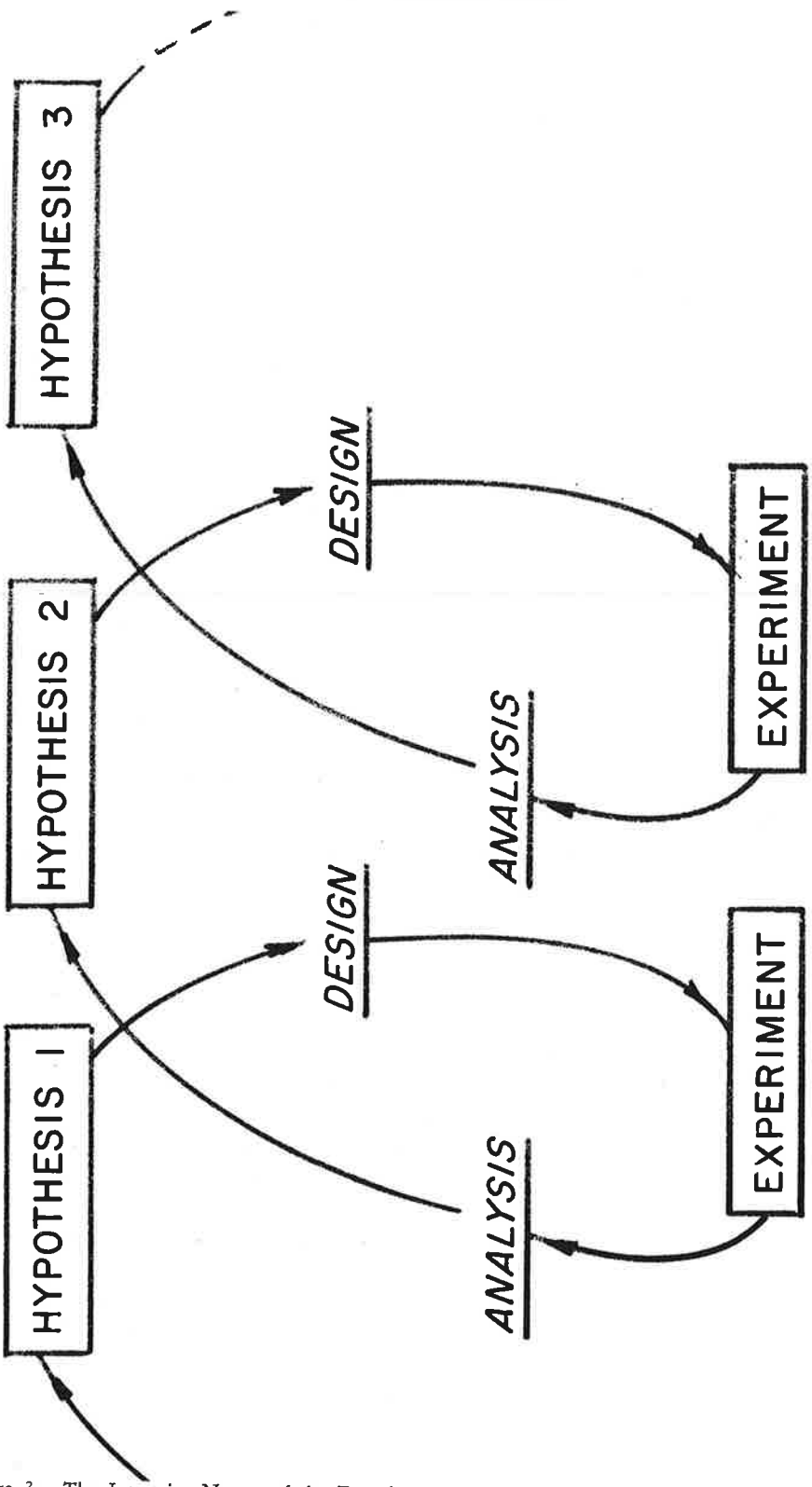

Figure 3. The Interative Nature of the Experimental Process.

more will search the area to see if he is at the top or if there is still another direction in which he should travel to find the top.

Such an approach is most economical in experimental effort since usually it is very time consuming and inefficient to test the whole surface with adequately small increments to indicate the true maxima. It is more difficult to visualize optimum conditions of a higher order factorial experiment, however, this can be handled mathematically and the reader is referred to Davies (29) and Hunter (52) for the mathematical treatment associated with this type of experiment.

The Response Surface Methodology is a representation of what Box (22) has termed the iterative nature of experimentation. Figure 3 graphically depicts the iterative process. The experimentor starts with a hypothesis which he wants to examine. In order to do so, he must design his experiment to test his hypothesis. He then runs the experiment, collects the data, and analyzes his data to see if he has confirmed or not confirmed his hypothesis. His experiment yields new evidence, upon which he bases a new hypothesis and designs a new experiment to gain further information. Eventually he will reach a hypothesis or theorem which he can use profitably in his operation.

4. *Evolutionary Operation*

Box (21) has used the iterative process along with the response surface methodology in developing a practice which is very useful in optimizing the industrial processes. He observed that the conditions of an industrial process can be controlled quite closely and that in many cases the control specifications of an industrial process are great enough to allow him to operate at two measurably different levels without materially affecting the usual operation of the process. Thus factorial experiments can be run with small increments of the variables as long as the variables are not outside the limits specified in the processing instructions. The test has to be repeated a number of times in order to obtain enough data to show a significant trend.

Usually the mid-point of such an experiment is also run. When it is found that there is a significant improvement in a given direction, the processing parameters are redefined and the whole experimental design is moved one step in that direction. At the new set of conditions, the process is repeated until a further directional improvement can be indicated. Eventually, there will be some place where the mid-point will be the optimum and all other points will be less desirable. This method has been termed evolutionary operation (EVOP) since it is a slow but sure method of arriving at the optimum conditions for operating the process. More often than not, cost and yield factors are optimized by this method but equally important are the quality factors that can be improved by such a technique. Often quality and the economics are mutually optimized. Since it is also desirable to operate methods at optimum conditions for better precision and accuracy, it is not impossible to believe that this technique could not be equally useful in developing laboratory procedures.

5. *Regression Analysis*

One of the techniques that the experimentor can use in the correlation of data with a given quality response is regression analysis. Frequently the experimentor does not have control of the variables and so cannot run a factorial or other designed experiment. However, he does not need to despair of obtaining a quantitative relationship since regression analysis makes it possible for him to handle such non-controllable data. Large quantities of data with many variables are particularly suitable for this technique. The end product of this technique is an algebraic expression relating the variables to the response.

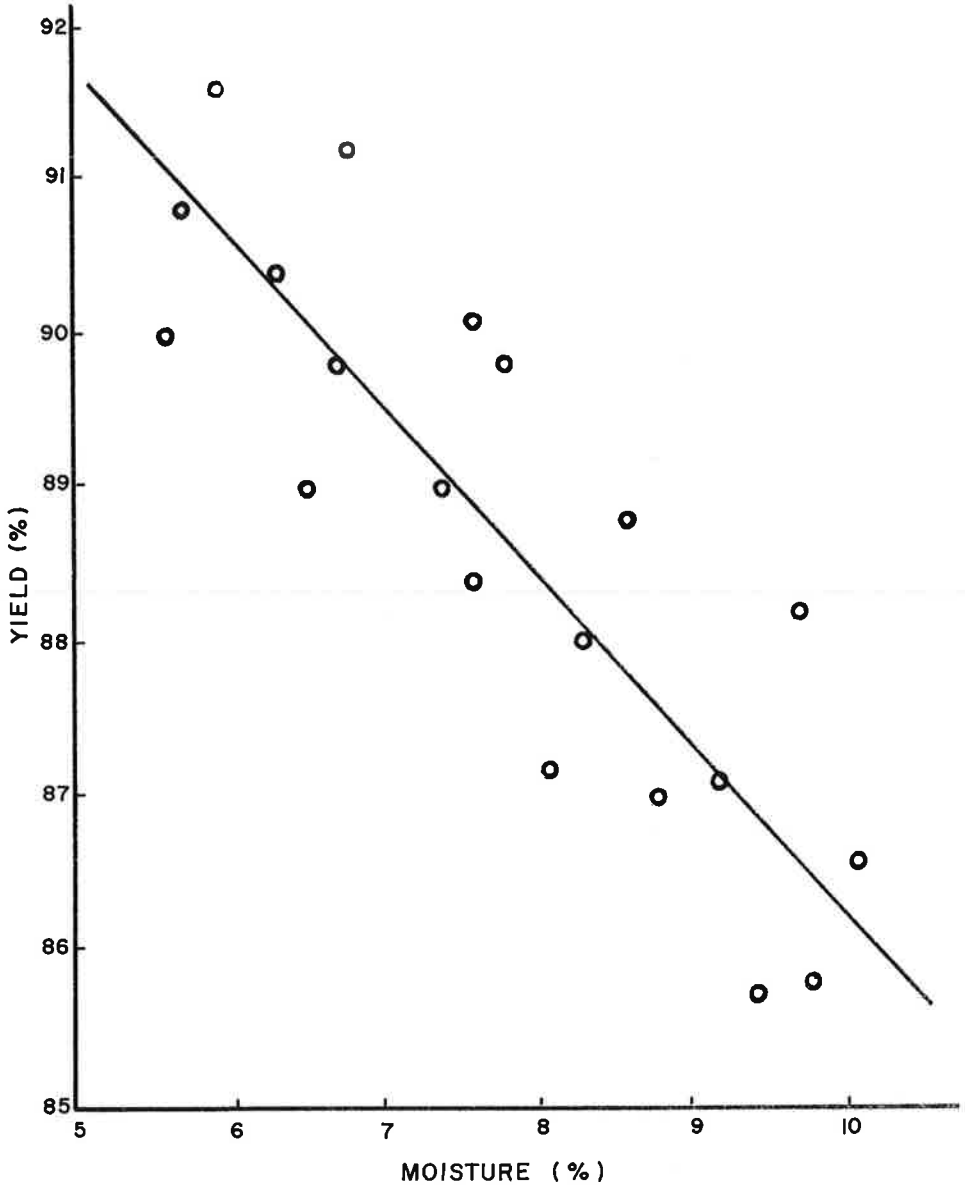

Figure 4. Peanut Butter Yield as a Function of Raw Peanut Moisture Content by Regression Analysis.

This method is most readily described and understood looking at one variable on a single response. Assume we wish to know how the variables moisture effects the yield in making peanut butter. As the moisture increases, the yield is expected to decrease. This should be an inverse linear relationship. Figure 4 shows a hypothetical case where moisture measurements have been made on a number of lots of peanuts and corresponding yield obtained. If all the points laid on a perfect line, there would be no need for analyzing the data. However, it can be seen that there is variation on the results and the question is what line best describes the relationship.

For a simple linear relationship such as this, the method of least squares can be applied. The theory behind the method of least squares is to find the best line so that the square of the perpendiculars from each point to the line is a minimum. To find the slope the following equation is used:

$$b_1 = (\Sigma xy - (\Sigma x)(\Sigma y)/n)/(\Sigma x^2 - (\Sigma x)^2/n)$$

The equation for the intercept is as follows:

$$b_0 = \bar{y} - b_1\bar{x} = \Sigma y/n - b_1 \Sigma x/n$$

Solving these equations will give the equation of the line which will look like this:

$$\hat{y} = b_o + b_1 x_1$$

where $\hat{y}$ is the expected yield, x_1 is the moisture, b_1 is the slope and b_o is the intercept. Thus, the experimentor can calculate the expected yield from the moisture using the relationship given above.

The suitability of the algebraic relation for predictive purposes can be determined. The percentage variation explained by the above relationship is given by the statistic r-square which is calculated from the following equation:

$$r^2 = (\Sigma xy - (\Sigma x)(\Sigma y)/n)^2/(\Sigma x^2 - (\Sigma x)^2/n)(\Sigma y^2 - (\Sigma y)^2/n)$$

r-square will vary between O and 1. At O, there is no correlation between the variable and the response and the points will be a perfect scattering such as might be expected from a shot gun blast. If r-square is 1, there is a perfect correlation between the variable and the response and all the points will lie on the line. Rarely are these extremes encountered in practice. However, the magnitude of this statistic, particularly as it approaches 1, is an index of the ability of the variable to predict the response. The data described in Figure 4 indicates that the equation does not fit the data perefectly. ($r^2 = .77$). This is expected since it is recognized that other variables such as the amount of foreign material, the major damage, the minor defects, the unblanchable pickouts, etc., all contribute to yield as well. The square root of this statistic, R, is defined as the correlation coefficient.

It is possible to put other variables in the equation. The general form of such an equation is:

$$y = b_o + b_1 x_1 + b_2 x_2 + \ldots b_n x_n$$

where y is the response, $x_1, x_2, \ldots, x_n$ are the variables and $b_o, b_1 \ldots, b_n$ are the coefficients for the variables. Interactions can be introduced as follows:

$$y = b_o + b_1 x_1 + b_2 x_2 + b_{12} x_1 x_2 + \ldots$$

To employ this technique, the experimentor would collect data on the variables and the yield associated with a number of lots. The algebra of solving the regression model is very complex and a computer is almost mandatory in solving complex regression models with more than two or three variables. Understanding of Matrix Algebra is

also useful in running the program. However, "canned" programs are available from most computer manufacturers to obtain regression analysis. A stepwise regression analysis is often employed where there are a large number of variables since this will weed out the less significant ones and give the experimentor the simplest and most meaningful mathematical model with the least number of terms. For instance, in the above example moisture would undoubtedly show up as the most significant variable explaining 77% of the variation. The next variable might add 12% to this, thus, with two variables, 89% of the total variation can be explained. One of the other variables might increase R^2/by 0.06 and, thus, with three variables 95% of the variation might be explained. It would be senseless to try any other variables although the computer program would go on until it had completed all variables that the experimentor had proposed in the model. Thus, with three terms and a constant a very effective equation to predict yield could be obtained.

Interactions or other forms of the variables such as inverse forms (1/x) or higher powers (x^2) can be investigated with this procedure as well. For instance, the moisture content and temperature of peanuts in storage has an effect upon the formation of aflatoxin in a lot. These variables will have an optimum and so a strictly linear relationship will not hold. Therefore, a second order and possibly a third order model equation would be proposed and the program run to obtain the best coefficient and powers of the variables to describe the relationship.

Regression analysis has proved to be a very powerful tool. For instance, large quantities of field data are collected yearly on climatic conditions, disease occurrence, pest prevalence, fertilizer usage, yield, etc., that could be analyzed by regression analysis. State Universities and Experimental Stations have access to computers and experts who can be consulted about the models and mathematics. These services can also be obtained in most industrial centers and the sheller and smaller manufacturers can make good use of them in many cases.

D. Sampling

1. *Lot Identification*

It is obvious that a measurement of quality must represent some uniform mass of material. For purposes of this discussion, the term lot will be used to describe a uniform quantity of material that is designed or expected to be homogenous in nature. Peanut lots generally are kernels from one given variety. In commercial, edible trade, no distinction is made between sub-varieties within the group. Thus, Early or Dixie Runners are sold intermixed as one variety. On the other hand, Virginia peanuts are sized to meet certain screen requirements and thus, a lot contains specific characteristics dictated by the intended usage. The varietal differences are more rigidly maintained in seed promulgation and agronomic research. The size of the lot should be defined at the time the sample is taken. It can be small as the peanuts from one bush or it may be as large as a truck or rail carload. Usually the size of the sample is related to the size of the lot, i.e., the larger the lot, the larger the sample. Lots should be adequately identified until they are used. This is true of both raw or processed goods. Part of the reason for this is to satisfy regulatory demands. But more important, a processor should be able to check back on a quality characteristic of a given lot to know its suitability for a given usage or to answer a complaint.

2. *Types of Quality Characterization*

Most test procedures are destructive, i.e., the sample is ground or destroyed chemically in the test and the sample is irretrievably lost. Therefore, it is often necessary to

sub-divide a sample into many small portions, one for each test to be made. Since peanuts are particulate in nature, the sub-sample can be made more homogenous by pre-grinding and blending. Care must be taken in grinding since peanuts have a tendency to oil off which will change the compositional values of the test.

There are a number of tests which are non-destructive. For instance, grade factors can be determined on any size sample and the sample reused to determine another quality factor. This has been useful in correlating damage extent with aflatoxin level. In a test we made, a sizeable sample (5000 grams) was visually inspected for damage. The two portions, damaged and sound kernels, were subsequently ground and assayed for aflatoxin.

There is a difference in the approach to sampling depending upon the nature of the material and the quality characteristics. There are two types of sampling approaches, variable and attribute sampling techniques. When a record is made of an actual measured quality characteristic, such as moisture, the quality is said to be expressed by variables. When a record shows only the number of articles failing to conform to a specific requirement, it is said to be a record by attribute. An example of attribute characteristics is major damage; a given peanut is either damaged or sound.

a. *Variable Sampling*

The accuracy of the measurement of a variable characteristic depends upon the uniformity of the characteristic from kernel to kernel. Variable characteristics such as protein and fat content are usually highly uniform. Low levels of off-quality peanuts will have very little effect upon the measurement of such a quality characteristic. Moisture is a variable attribute which might show some stratification from one part of the lot to another. Uneven drying in a wagon of farmers' shelled peanuts will produce stratification, i.e., kernels with different moisture levels from one end of the lot to the other. Therefore, it is mandatory that a good cross section of the lot be obtained in the sample. It is also desirable to pre-grind a sizeable sample and take sub-samples from the ground portion for the chemical tests to be made. This will assure an even distribution of the characteristics being sought and a better estimation of their value. However, the size of the sample for variable quality characteristics is generally not as critical as it is for attribute quality characteristics and a sample size of one half to one pound will usually suffice, regardless of lot size.

b. *Attribute Sampling*

The variability associated with an attribute, sampling program is directly related to the level of the characteristic being measured. If the expected number of "bad" kernels in a given sample is small, the variability of the number of those kernels appearing in the sample will also be small. However, the ratio of the variance of this number to the sample size will be great as compared to a quality characteristic where a high proportion of expected "bad" kernels is to be found. The standard deviation (σ) is expressed by the following equation:

$$\sigma = \sqrt{np'(1-p')}$$

n is the number of pieces in the sample and p′ is the proportion of the quality factor in the lot.

This equation indicates that as the percent-defective or the size of the sample increases, the standard deviation also increases. However, the percentage error will de-

crease as the sample size increases. For instance, the percentage of the quality characteristic is the number of defectives divided by the sample size times one hundred (n p'/n x 100 or p'100). The standard deviation as a percentage is:

$$\sigma = \sqrt{p'(1-p')/n}$$

From this, it is evident that an increase in the sample size n will result in a lower percentage standard deviation.

This principle is more readily seen by the use of an example. In a sample of 1,000 kernels for a characteristic which is present at 1%, 10 defective kernels will be expected in the sample. Using equation 10) the standard deviation is found to be 3.15 kernels or 0.315%. Nineteen times out of twenty or 95% of the time, the actual sample of this lot will contain 4 to 16 bad kernels (10 ± 6) or 0.4 to 1.6% (1.00 ± 0.63%). If a 10,000 kernel sample were taken instead of a 1,000 kernel sample, the standard deviation according to equation (10) would be almost 10 kernels or 0.10%. The 95% confidence interval would then be 0.8 to 1.2%. Thus, by increasing the sample size tenfold, the analyst has decreased his standard deviation threefold. The significance of this is that the tester can control the degree of accuracy that he needs by the size of the sample he chooses.

Figure 5 will assist the analyst in determining the size of a sample and its relationship to the standard deviation. The sample size is found on the abscissa (horizontal axis) and where the value intercepts the line at the expected level of the quality characteristic, the standard deviation in percent can be found on the ordinate (vertical axis).

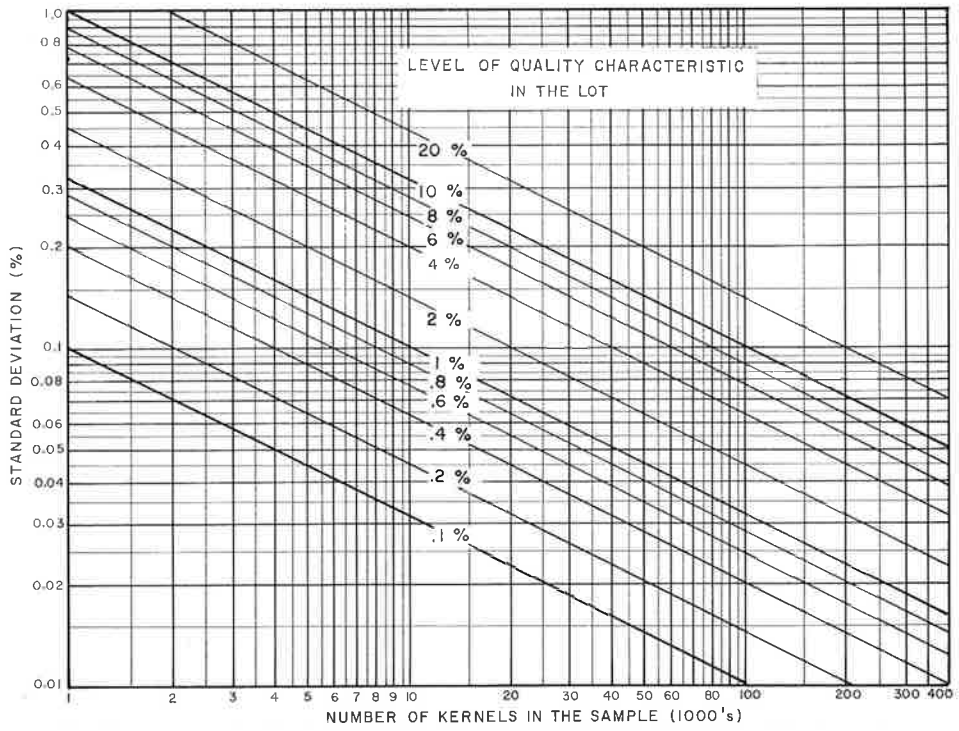

Figure 5. The Effect of the Sample Size on the Standard Deviation.

To illustrate the use of this figure, consider the example given in the previous paragraph. The 1% line crosses the abscissa value for the sample size of 1,000 kernels at the value of 0.32% on the ordinate which is the standard deviation. The same line at a sample size of 10,000 gives a value of 0.10%.

There are several instances where the quality characteristic is a combination of an attribute and variable characteristic. A notable example of this is the aflatoxin content of a lot where the aflatoxin is concentrated in the rancid and moldy fraction of the peanuts. However, not all such peanuts have aflatoxin or if they do, they do not have the same amount. Thus, the variation is very large as discussed by Tiemstra (105) and Whitaker (118). Flavor testing by the CLER procedure is another case where judgment factors are required on individual kernels which introduces two sources of variability, i.e., that of the number of off flavor kernels per sample and the degree of off flavor in such kernels.

A good cross section of the lot should be represented in the sample for attribute sampling as well as for variable sampling to minimize the effect of stratification. The variability that has been discussed under attribute sampling will only apply if random sampling conditions are adhered to. Thus, every peanut should have an equal probability of being in the sample.

3. *Sampling Devices*

Peanuts handled in large quantities such as truck loads pose a problem in obtaining samples particularly from the center or bottom of the lot. A pneumatic sampler is used at practically all shelling plants and buying points for farmers stock peanuts. This device is described in the U. S. Department of Agricultural's circular 1967A (110). The sampler is supported on a framework under which a truck or wagon can be driven. The sampler is a tube about two inches in diameter which can be lowered through the load while rotating. Suction is applied to the tube to bring the peanuts up into the sample bag at the top. The tube can be positioned horizontally anywhere over the load so that several borings can be made of a single load thus, obtaining a good cross section.

Shelled goods are normally shipped in burlap bags. The sampling of such shipments is described in the U. S. Department of Agricultural Bulletin 1967B (111). A pointed trier is used which is inserted between the weaving of the burlap bag and gently prodded to remove two or three handfuls into the sample container. After the trier is removed, the point of the trier is drawn over the weave of the burlap to reseal the hole. One out of every four bags in a lot is sampled in order to get a good cross section of the lot. All the kernels thus taken are composited for the final sample.

With more interest in bulk handling of peanuts, there is increased need for better sampling techniques. The best way to handle this problem is to take a sample of peanuts while they are being processed or moved from one place to another. There are several commercial sampling devices available and other's are being built, all of which are being studied for an optimum solution of this problem. Generally, the manufacturers of peanut products have less problem in sampling. The products are usually packed in small units, a certain number of cases of such units to a lot. Thus, a cross section of the lot can be obtained readily by removing a number of these smaller units and compositing the contents. Peanut butter is even less annoying to sample because a lot can be designed as a single batch. The mixing and formulation intimately blend the material so that a single grab sample or single packed off unit represents the material very adequately.

E. Sample Preparation

1. *Grinding*

The problem of obtaining better precision on peanut samples for aflatoxin have led some experimentors to evaluate grinding methods to prepare the sample for chemical assay tests. Since the principle will hold for any variable testing, it can serve as an example of the overall problem of grinding and subsampling for all chemical and variable assay procedures.

Stoloff, *et al.,* (98) studied the effect of several mills on reducing peanut kernels to a homogeneous small particle mixture. His results indicated that the Hobart vertical cutter-mixer provided the most uniform material. However, in order to obtain the uniformity he desired, it was necessary to add a dilutent such as clam shells to the material. The other mills investigated were the Hobart food cutter, the Bauer mill, the Thomas nut cutter and Dickens hammer mill. Most of these had the disadvantage of becoming dull rather rapidly and not reducing the sample to as uniform a size. In addition, most of them required blending and mixing after grinding in order to provide a homogeneous mass. Care must be taken in grinding peanut samples since the finer the sample is ground, the more oil is released from the cellular structure and the composition of the mass can be altered. Our preference for several applications is to grind the material rather coarsely in a food cutter and take a sample four or five times greater than what is necessary to use in the test. This sample is diluted with an equal portion of chloroform and ground for three minutes at high speed in a Waring blender. The resultant slurry is easy to handle and very uniform in composition since the specific gravity of the solvent is about the same as the peanut particles. One of the most useful means of reducing a large sample to a representative ground sample suitable for sub-sampling is the Dickens Mill (33). This mill has a convenient feature in that it reduces the sample and simultaneously takes a sub-sample which is a twentieth portion of the original. Basically, it is a hammer mill placed on a vertical axis and as the peanuts are fed through the top, it throws them to the side. As they are broken up, the peanut particles pass through the screen which surrounds the blades. One twentieth of the circumference of the mill is open to the sample chute and the remainder of the material falls into the discard pile.

2. *Roasting*

Most laboratories have developed their own technique for roasting peanut samples. The most common method is to construct a basket on the spit of a rotisserie to hold the sample. The rotisserie can be heated electrically or by gas burners. Thomas *et al.,* (104) described such a device to roast 200g/ quantities. The basket of his roaster was 5½ inches in diameter and 15 inches long. The sample is roasted for 5 minutes at a temperature which would give him the desired degree of roast. The temperature range was 300° to 350°F. Willich *et al.,* (120) indicated that the time of roasting was most critical on the effect of the end product under the conditions he describes. No difference on blanchability was noted as to the degree of roast given to the samples. Morris and Freeman (69) also described a means of roasting peanuts and their effect on the palatability of peanut butter. Some experimentors have installed a fan in the heating chamber to obtain a more uniform temperature in the basket. Figure 6 shows the basket which we have made for use in a household oven equipped with a rotisserie motor. The axis of the spit is placed on an angle from the axis of the cylinder. Thus, as the spit turns, the peanuts are mixed from end to end and a much more uniform roast can be obtained even though there might be temperature gradations within the oven. The basket

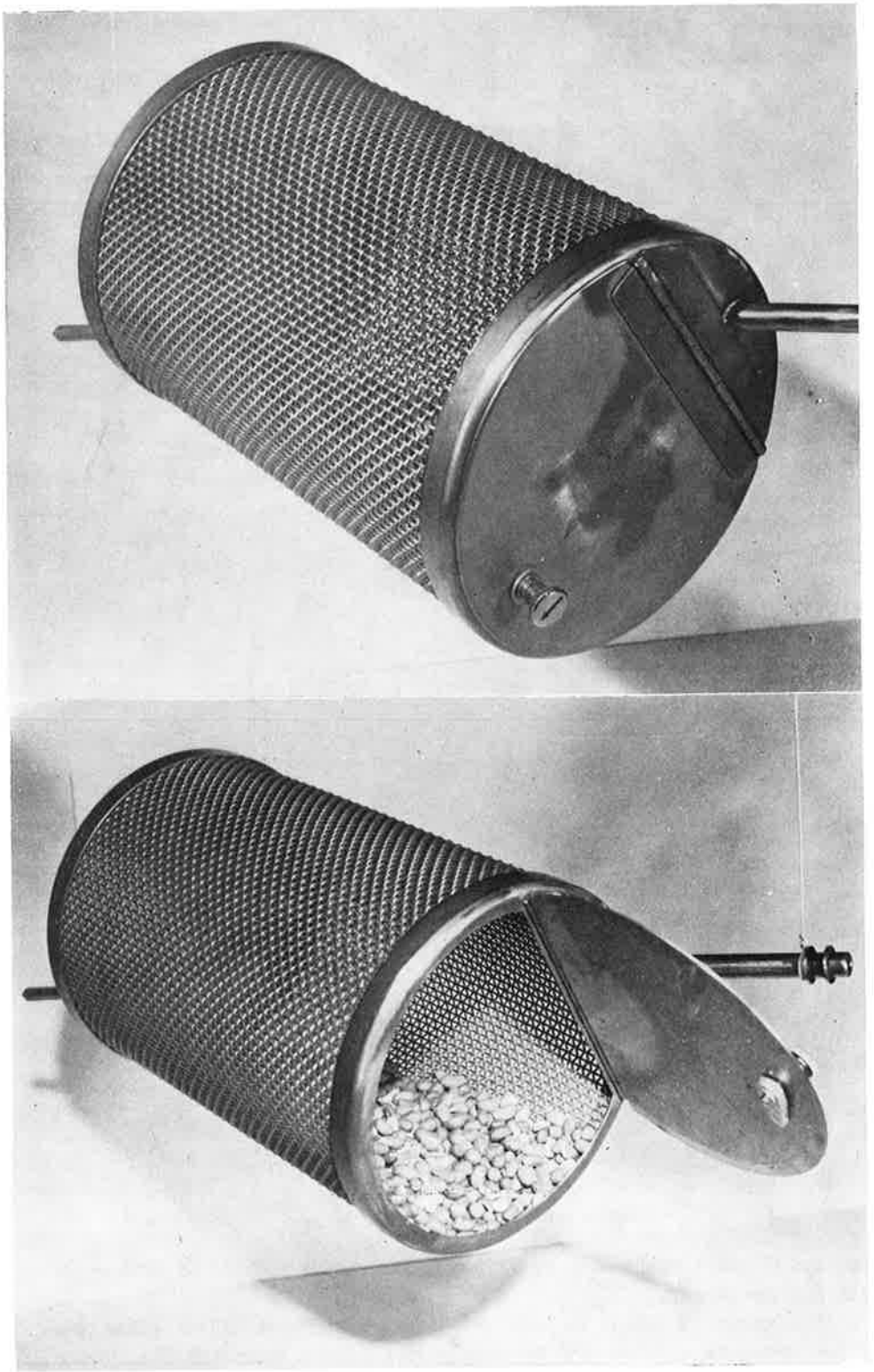

Figure 6. Rotating Basket Used in Oven for Roasting Peanuts.

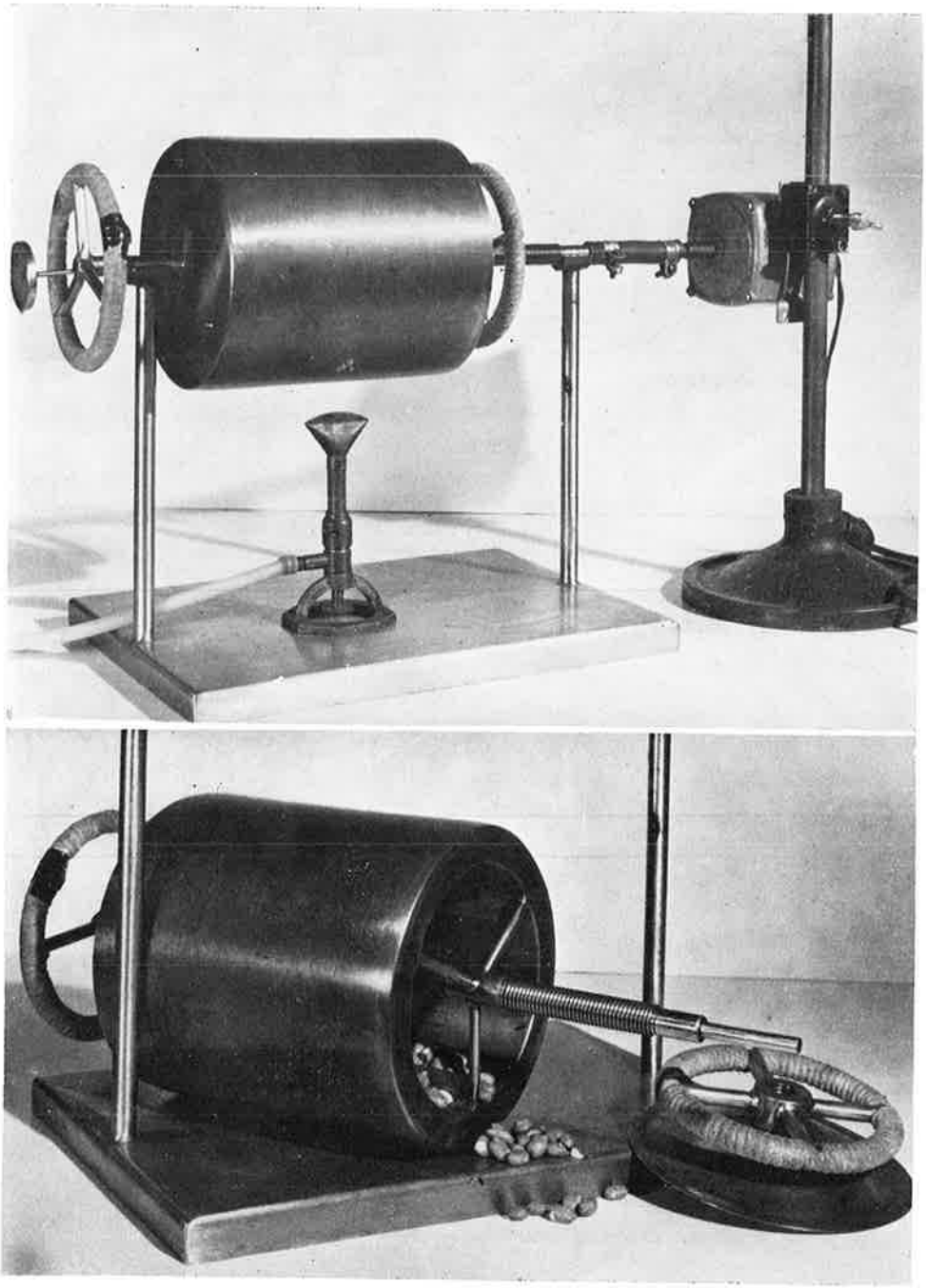

Figure 7. Double Wall Cylinder Roasting Chamber.

measures 8 inches in diameter by 12 inches long and is capable of holding up to 2000 grams of peanuts.

Another type of roaster we have built is shown in Figure 7. This roaster consists of two concentric stainless steel cylinders welded together with a dead air space of about ½ inch between the shells of the two cylinders. Bunsen burners are used to

apply heat to the outer shell. The axle away from the drive side is hollow so that a thermometer can be inserted and the inside temperature read. The temperature will rise to between 100 & 105°C and stay there until most of the moisture has been removed from the peanuts. Then it begins to rise rapidly. When the prescribed temperature is reached, the peanuts are removed. Thus, the degree of roast can be controlled by the temperature to which the peanuts are taken. This final temperature has to be determined experimentally but when once established, is good indefinitely for the variety and degree of roast required. Thus, variations from gas pressure, atmospheric conditions or the size of the sample are minimized.

Oil roasting is an acceptable way of preparing peanuts for flavor and some other tests. The oil used to roast the peanuts should be fresh and should not have a deleterious effect upon the final flavor. The temperature at which the roasting is done will also have an effect upon the amount of oil absorbed with corresponding differences noted in the final flavor or other analysis. Thomas *et al.,* (104) has described conditions to be used in a Sunbeam skillet. Temperatures were varied from 320 to 360°F to get the varying degree of roast that he studied. The time was held constant at 20 minutes.

F. Wholesomeness

One of the prime considerations for quality peanuts (when they are to be used for human consumption) is wholesomeness. In order to be wholesome, a product has to be free of foreign material, unadulterated with toxic or noxious substances such as insecticides and mycotoxins, not infested with insects or rodents and free of microorganisms that may cause spoilage or disease. Furthermore, the processing equipment used to handle and process peanuts must be clean so that the product never comes in contact with any surface which might be considered undesirable. This is true of personnel that are also handling the product and making finished goods from it.

1. *Sanitation*

The Food and Drug Administration (114) has recently issued guidelines for industries to follow in producing wholesome food items. These guidelines apply to shellers and manufacturers who produce shelled peanuts or finished goods for the market. Whereas sanitation is not the topic for discussion in this chapter, the evidence of poor sanitary practices are a form of quality determination and must be mentioned here.

The "Official Methods of the AOAC" (12) gives methods (36.020 to 36.024) for the determination of adulteration. Foreign material and evidence of insect or rodent contamination can be determined in shelled peanuts by running them over a screen with openings of about ¼ inch diameter. Usually 4 or 5 bags are so examined and the siftings are visually examined for insect parts, maggots and rodent dirt. The FDA* has recently published guideline limits for insects, insect parts, rodent hairs and water insoluble inorganic residue in peanuts and peanut products. Ultraviolet examination for rodent excrement is another inspection often carried out on lots of sacked, shelled peanuts. Peanuts are a favorite nesting environment for rodents since they have material from the burlap sacks to build their nests and a ready food supply. On running over the bags, the rodents will invariably leave a trail which is readily discernible under ultraviolet light. The urine stains fluoresce and can be easily identified.

Bacteria content of food items is often an index of the conditions under which a food product has been handled. High counts are undesirable because they usually

*Federal Register 37, No. 62, March 30, 1972.

indicate spoilage or poor processing conditions or contamination. Raw peanuts are traded under 8.5% moisture and most peanut product are usually sold in the final form at a moisture content of around 1%. At these moisture levels, there is not enough water activity to support bacterial growth. Therefore, peanut butter, roasted peanuts and peanut candy can be considered shelf stable from a microbiological point of view. However, organisms have been found to remain viable, although not active, in peanut products for extended periods of time. Therefore, sanitation practices are mandatory in order to prevent exposure to or the introduction of microorganisms during any part of the processing history. Usual procedures for determining the total bacteria count or the presumptive test for coliform can be applied to peanut products (11).

2. *Grade Standards*

The United States Department of Agriculture has established standards for all the varieties of domestically produced peanuts. These standards are issued to insure the lot as being of a given variety and containing a minimum of inedible, foreign or esthetically undesirable material. Therefore, the quality of individual kernels in the lot are important and have to be evaluated. The percentage of peanuts of other varieties, damaged peanuts, peanuts with minor defects and foreign material is determined and maximum limits set. With the exception of varietal differences, the kernel of this nature are not suitable for consumption. Besides limiting the amount of esthetically unsound material, the grade standards also provide the manufacturer who buys such material with the assurance that other quality criteria, such as the amount of splits or size of the kernel is also kept within bounds.

It is impossible to list all the grade factors here but they can be obtained in the documents supplied by the USDA (107, 108, 109). However, in table IV specifications for the number 1 grade of each variety for several characteristics are compared. The size of the sample used to determine these grade qualities is usually 1,000 grams or approximately 2,000 kernels. In the case of some large lots, 2,000 gram samples are often taken for this evaluation. Figure 1, page 609, shows the sampling OC curve of a 1,000 and 2,000 gram sample for an acceptance values of 1.25%. In addition, we have shown the 10% consumer and producers' risks of these same curves to give an indication of the spread of results to be expected and the improvement possible with a larger sample. These curves are for sampling variability only and do not include the error inherent in the human element in reading damage or other quality factors.

TABLE IV

Specifications of Raw Shelled Peanuts of No. 1 U. S. Grade.

		Runners	*Spanish*	*Virginia*
Other Varieties	Max.	1.0%	1.0%	1.0%
Splits	Max.	3.0%	2.0%	3.0%
Damaged	Max.	1.5%	1.5%	1.25%
Damaged Plus Minor Defects	Max.	2.0%	2.0%	2.0%
Foreign Material	Max.	0.1%	0.1%	0.1%
Pass Through Screen	Max.	3.0%	2.0%	3.0%
Screen Slot Width		16/64"	15/64"	15/64"

Farmers stock is sampled and tested by government graders at all shelling and buying points. This grading insures a sheller that he is obtaining materials from which he can make satisfactory shelled goods. The value the grower receives is based on contain quality factors and levels of this analysis. Moisture is a very important consideration in both the farmers stock and the shelled goods since high levels will support bacteria and mold growth. The measurement of moisture will be discussed later (page 631).

3. *Maturity*

The evaluation of peanuts as to their maturity logically belongs in this section since mature peanuts are considered esthetically more desirable for edible use, so a brief discussion of the methodology developed for this purpose will be given. The experienced grower or agronomist can note certain physical characteristics such as size and color (particularly of the inside of the shell) which are evidence of maturity. Although this may be very adequate for most applications, it is not very objective. Therefore, several workers have sought methods that would detect chemical changes which occur during the maturation process and can be used as an objective index of maturation. Emery *et al.,* (37) determined that the pigment of the oil at 444 mμ appears to be a more satisfactory index than the internal pod color, seed coat smoothness or average kernel weight. Sharon (90) looked at moisture, crude lipid, total sugars, protein, fatty acid composition, free fatty acid and peroxide as a means of measuring maturity as well. He also indicated that the color of the solvent oil extract was the most significant as far as measuring maturity. Holley and Young (51) first described a method for determining the maturity of peanuts by measuring the color of the oil. Pattee and Purcell (76) have further characterized the pigments which are responsible for the coloration of the oil from immature peanuts. Kramer *et al.,* (58) gave some actual readings on peanuts of known maturity. They found a direct correlation between color and degree of immaturity. However, as the color value increased, the standard deviation also increased quite appreciably. For instance, between a value of 45 and 65, the standard deviation was about 18. Thus, a lot with an average reading of 50 would have a 95% confidence interval of 24 to 86. The lower part of this range is in the acceptable levels for mature peanuts. Therefore, in order to obtain satisfactory values and assurance of true maturity levels, it is necessary to take several independent readings on a lot. Although this method of evaluating peanut maturity appears to be promising, it has not found wide acceptance as a quality control procedure. Possibly the time necessary to make the determination is not worth the results. Certainly, the sizing of the peanuts, removing of the shrivels and runts, is satisfactory for most commercial purposes. The color method is well suited for varietal development, and undoubtedly will interest researchers. It may also find use in evaluating curing conditions.

4. *Aflatoxin*

Although a bacterial spoilage or contamination of peanuts and peanut products is not too hard to detect, the formation of the metabolite, aflatoxin, by molds in peanut products has posed a problem to the industry. One of the problems has been the successful identification of the mycotoxin when it is present. This subject is of sufficient interest to the industry that Chapter 16 has been largely devoted to it. However, the methods used to determine the presence of aflatoxin will be mentioned here.

The AOAC (13, 15, 16) has adopted three first action methods for determining the amount of mycotoxin in peanuts and peanut products. Originally reported by

Nesheim *et al.,* (72), one is commonly referred to as the Celite procedure because it uses celite in the column chromatographic step to remove extraneous interferring materials from the final extract, which is then used for the thin layer chromatographic (TLC) separation and determination. The CB procedure proposed by Epply (38) uses a silicate gel column chromatograph for clean up but its basic principle is very similar to the Celite procedure.

The following principles apply to both of these procedures. The aflatoxin is extracted by a suitable solvent from a ground portion of the original peanuts. The solvent containing the extract is separated from the bulk of the peanut material by filtration or centrifugation and an aliquot of the extract is column chromatographed to remove interferring substances. The eluent from the chromatographic step is evaporated to near dryness and the residue taken up in a small volume of benezene. This solution is spotted on a TLC plate consisting of a thin film of silicate gel. The chromatograph is developed with a solvent mixture consisting of nine parts of chloroform to one part of acetone. A standard is spotted along with the unknown to indicate the Rf values for the spots and give a reference as to the quantity of the various toxins present. The plates are read under UV light where the aflatoxin spots fluoresce and the quantity can be estimated by comparing the intensity of the spot with that of the standard.

The above procedures are both lengthy and detailed requiring up to three to four hours in order to obtain a determination. The need for a much faster method has led Walking and his co-workers (116) to develop one. They have been able to speed up the extraction process and to eliminate the column chromatographic step which is very time consuming. Although there appears to be more interference from other fluorescing materials than in the CB procedure, the plates are still clear enough to give readings as to the aflatoxin content. This method has also been adopted by the AOAC and all the AMS laboratories of the USDA use this procedure for the official determination on peanuts.

The foregoing methods are all chemical using thin layer chromatography to separate the aflatoxin. It is possible to have small amounts of other materials in some peanuts that have the same Rf value as aflatoxin. Therefore, other means have been developed to verify its presence, the derivative method and the bioassay tests. The aflatoxin is separated from a kilogram of the peanuts or peanut product in the same manner as is used in the official procedures providing there is enough material to work with. Since the chemical derivative procedure (14) is cheaper, faster and more reliable, it is used as an intermediate step to confirm the presence of aflatoxin before the more laborious procedures of the bioassay test are run. Stoloff (97) has reported on a collaborative study indicating the ability of the chemical method to confirm aflatoxin.

Bioassay tests, due to their inherent reliance on living organisms, are difficult to reproduce as well as costly and time consuming. Thus, a diligent search has been made for more susceptible organisms which would show results in a shorter time. The Duckling test (Armbrecht and Fitzhugh, 10) was the original bioassay test, however it has been replaced by the more sensitive chick embryo test (Verrett *et al.,* 115). There has not been complete satisfaction with this test and many other organisms have been investigated. The one that presently seems most promising is the bacillus megaterium method reported by Clements (26).

Sampling is one of the big problems in obtaining accurate aflatoxin results. The evidence indicates that aflatoxin is carried in a small proportion of the peanuts and

that even in this small proportion, there is a wide variation among the kernels as to aflatoxin content. It has been the practice of the industry to use a 10-pound sample for the aflatoxin assay, but it has been shown that a larger sample would produce a smaller degree of uncertainty (105, 118, 119). Therefore, sequential sampling plans are being considered in controlling the level of aflatoxin in raw shelled peanuts as well as finished salted nuts. The PAC has adopted such a plan for the 1971 crop. It is basically a double sampling plan where two 24-pound samples are taken. If the first result is 15 ppb or less, the lot is passed. If the reading is over 15 ppb, the second sample is analysed. The average of the two tests has to be 25 ppb or less in order for the lot to pass. Undoubtedly this sampling plan will be revised as more stringent requirements for aflatoxin content are imposed on the industry.

There has been a concerted effort to eliminate aflatoxin from passing into commercial trade from the growers by a more adequate screening at the shelling plants and buying stations. Dickens and Welty (34) have developed a method for visual examination of farmers stock to determine whether Aspergillus Flavus is present in farmers stock or not. Positive evidence of this mold automatically places the lot into Segregation 3. The average level of aflatoxin found in shelled goods has been materially reduced by this procedure, although the incidence has been about the same. Holaday (49) has developed a rapid procedure for detecting aflatoxin in farmers stock or shelled goods which shows promise. Unfortunately, neither of these methods have wholly coped with the sampling problem. It appears that the best solution is to eliminate it at the field level if at all possible.

There are some quality factors which have a concurrent incidence with aflatoxin and so more workers have tried to show a correlation between the two. Parker (unpublished data) and Pattee and Dickens (75) have studied the relationship of aflatoxin to the amount of damage present in certain lots of peanuts. Whereas the aflatoxin resides primarily in the damage fraction, there appears to be no direct correlation between the amount of damaged peanuts and the level of aflatoxin in the lot. Pattee and Sessions (77) have studied the relationship of the fat acidity to aflatoxin readings. When the acidity reached or exceeded 16 mg of KOH per 100 peanuts, they found positive aflatoxin results. It is assumed that the biological effect of the mold growing on the kernel causes the acidity to develop in much the same way that Baker *et al.*, (19) describe it.

G. Compositional Methods

The composition of peanuts by varieties can be found in the literature (Woodroof 122). Nutritional information is also available and adequate for normal commercial practices. However, the determination of the compositional properties of peanuts or peanut products is necessary for many agronomic problems and for the control of formulation in many end use applications.

1. *Moisture*

Of all the compositional properties that can vary in peanuts, moisture is the most variable of all. It changes from the time it is harvested to the time it is shelled. It can further change in storage or en route. It undergoes further change when it is roasted, ending up at 1% or less. Therefore, the accurate determination of this ingredient is most desirable.

The usual method of determining moisture is by evaporation loss on heating. The official method of the American Oil Chemists Society (AOCS) Ab-2 – 49 (1)

specifies the drying of a two gram sample at 130°C for three hours. This is practically identical to the Association of Official Agricultural Chemists (AOAC) (12) method 13.004. The AOAC gives an alternative method which dries the sample in a vacuum oven at 25mm of vacuum at 98 to 100°C for five hours.

The oven methods are necessarily long and, therefore, are not suitable for many applications where time is essential. The Steinlite method * is much faster and has been universally accepted in the industry. The Steinlite instrument measures the capacitance of a given weight of peanuts. The newer models use a 250g size, although we have found the 100g size of the older instrument to give good precision. The instruments have to be calibrated for each variety of peanuts by taking readings on samples and determining the moisture on the same samples by an accepted alternative procedure such as one of the oven method. The percentage of moisture in unknown samples are read from this calibration curve. Readings on the same samples as much as a week apart have given a standard deviation of 0.10%.

There are a number of other electronic methods and moisture meters which could undoubtedly be applied to peanuts and peanut products. Another instrument that deserves notice here is one manufactured by Microwave Instrument Company (9). It gauges moisture electronically by measuring the absorption of a microwave beam passing through the material. A similar apparatus is produced by Moisture Register ** which measures the conductance of the product. Both of these instruments can measure the moisture of the product in process lines and, therefore, may be very useful in process control.

2. *Fat*

The fat content of peanuts and its properties has a dual role in the palatability of finished products. First, it is the lubricating constituent that keeps the other ingredients from becoming pasty and too dry to swallow. Because of its fluid character, it is the major controlling factor in the consistency of peanut butter. Second, the chemical composition of the fat, i.e., the unsaturation which is present, causes this component to be readily oxidized to form off flavor components. Thus, it is the determining factor in the shelf life of the product. Recent interest in polyunsaturated and saturated fatty acids and their relationship to blood cholesterol formation and arteriosclerosis have further heightened the interest of researchers in the chemical composition.

a) *Quantitative Measurement*

Solvent extraction procedures are the most common for determining the oil content of peanuts and peanut products. The AOCS method Ab 3-49 pre-dries a 50 gram sample which is sliced or ground, mixed and a two gram portion put into a Butt Extraction Tube. The fat is extracted with petroleum ether for four hours in a butt extraction apparatus. The solvent is evaporated off of the extracted solution and the residue weighed and calculated as the fat in the sample.

Several methods exist which use a common approach. The pre-weighed sample is ground in the presence of a given volume of solvent. The slurry is filtered and some physical property of the solution measured to determine the concentration of fat in the solvent. One such method measures the capacitance of such a solution where the solvent is a chlorinated hydrocarbon with a very closely controlled dielectric value. Basically, the method is as follows: The sample is ground in a special mill with the solvent filtered through a Buckner Funnel with vacuum. A measured amount of the

*Steinlite 500-RCT, Seedboro Equipment Co., Chicago, Ill.
**Moisture Register Co., 1510 W. Chestnut St., Alhambra, Cal. 91802.

filtrate is placed into the cell of the Steinlite Tester* and the conductance read. The fat content of the sample is read from a prepared calibration curve. The instrument reading is sensitive to temperature, but a correction can be applied to correct it to the standard temperature.

The U. S. Department of Agriculture evaluated this procedure on soybeans (113). Thirty-five laboratories participated in this study and a pooled standard deviation of 0.25% was obtained. Since peanuts are very similar to soybeans, it is not unreasonable to expect similar precision on them.

A variation of this approach is the refractometric method, or Halowax procedure. A solvent is used whose refractive index is a good deal different than the oil so that the concentration effect of the oil on the refractive index of the mix will be pronounced enough to obtain good differentiation. The recommended solvents for this procedure are chloronapthalene, (RI about 1.6335 at 25°C) and bromonaphehalene (RI about 1.6564 at 25°C). The two are mixed to give a refractive index for the solvent of 1.6450. As in the Steinlite or Capacitance procedure, oil seeds are ground with the solvent and the extracted fat solvent mixture is filtered. The filtrate is measured in a suitable refractometer (reading to 5 decimal points) and the concentration of oil in the solvent read from a graph which has been prepared by calibrating with known solvent-fat solutions. One advantage of this method is that the temperature control is inherent in making the RI reading. Further details can be found in Mehlenbacher (66).

Still another variation of this approach is the measurement of the specific gravity of the solvent oil mixture after extraction. Again, it is desirable to have a solvent whose specific gravity is as different from the oil as possible to get good differentiation with respect to concentration.

Broadline Nuclear Magnetic Resonance (NMR) has been used to determine the fat content of individual peanuts or samples of ground product or finished product. The induced field can be varied in a pre-programmed method. The hydrogen molecules resonate with the induced field depending on the radicals of which they are a part. At a given field strength, they process and absorb energy which can be detected by an Rf signal. The instrument is calibrated with the specific oil being sought. Proctor (86) gives a descriptive introduction to this technique. Conway and Earle (28) describe this method for determining the oil content of a number of grain seeds including peanuts. Since the method is non-destructive, the peanuts used in the analyses can be peanuts to raise new generations. Thus, it is possible to determine the oil content of seeds prior to planting and permit selection for this factor.

The cost of NMR equipment has, till recently, been prohibitive for most quality control applications. However, an instrument** has been introduced into the market under $8,000 which makes this technique available to product control laboratories and smaller operators. Certainly the rapidity with which tests can be made make it a desirable method to pursue. The peanut butter manufacturers can also use this equipment to determine the solid fat index, as will be discussed later. In addition, it is possible to control the fat content of peanut butter by measuring the incoming product and formulating accordingly. Salters, too, can determine the amount of fat picked up in the frying operations. It is even possible to control the fat of the finished product if the incoming fat content of the peanuts is known and the conditions of frying adjusted to give the desired fat content in the finished product.

*Steinlite Model 300 LOS.
**Mansfield, 62.

b. *Type of Fat*

Edible fats are mixture of triglycerides. A triglyceride is an ester of glycerine and three fatty acids. The fatty acids vary in length and in the degree of unsaturation, i.e., the number of double bonds in the chain. The melting point of the fat is directly proportional to the chain length and inversely proportional to the number of double bonds. The double bonds are also sites where oxygen can react and cause off flavor compounds. Therefore, the degree of unsaturation is also inversely proportional to the shelf life of the oil.

The usual index of unsaturation is the iodine value which is described in methods Cd 1-25 of the AOCS (1). A sample of the extracted oil is placed in a given volume of Wijs solution. This is an acetic acid solution of iodine monochloride which is an element consisting of one atom each of iodine and chlorine. This compound adds readily to the double bond and the excess amount can easily be titrated by converting it all to iodine and titrating with sodium thiosulfate. The iodine value of triolein is theoretically 98, which is a good reference for the degree of saturation of any oil. The iodine value of peanut oil is between 88 and 98. The determination of iodine values by the standard procedure is time consuming and, therefore, Avera *et al.,* (17) developed a method whereby the iodine number could be determined from the refractive index of the expressed oil. To make this determination, it is necessary to have a five place precision refractometer in order to obtain the sensitivity which is normally desired. This method has been adopted by the Quality Committee of APREA as an official method (50).

The Foregoing methods are indicative of the total unsaturation but do not give the relative proportions of saturated, mono and polyunsaturated present. Gas liquid chromatography can be used to obtain the amount of each individual fatty acid present in the fat. Method Ce 1-62 of the AOCS (1) describes this technique. The methyl esters of the fatty acids are prepared. The methyl esters are introduced into a GLC apparatus where they are volatilized and passed through an alumina column. Both length and unsaturation of the fatty acid radical has an effect on the Rf value, i.e., retention time on the column. Thus as the gases are swept through the column, the fatty acids are separated and come out at different time intervals. They are detected by a suitable detection device at the outlet and the quantity of each determined. French (42) has investigated this method on peanut oil and substantiated fractions in the effluent with ultraviolet spectrophotometry. He found good agreement between the iodine values calculated from the GLC and those obtained by the Wijs method. Although these techniques are rarely used in quality control, it is imperative to be able to determine the unsaturation in fats in both developing new varieties of peanuts and in formulating for better shelf life and nutrition. GLC is the most useful tool for this purpose.

There still is need for a procedure to determine the triglyceride composition of fats. A GLC technique has been described by Kukus and McCarthy (59). Very high temperatures are necessary to volatilize the triglycerides which cause some complications. Thin layer chromatography on silver nitrate impregnated silica gel plate has been described by DeVries and Jurriens (32) and Iurriensand and Krolsen (54). The identification of the fatty acid radicals of the triglyceride in each spot on the plate can be verified by GLC.

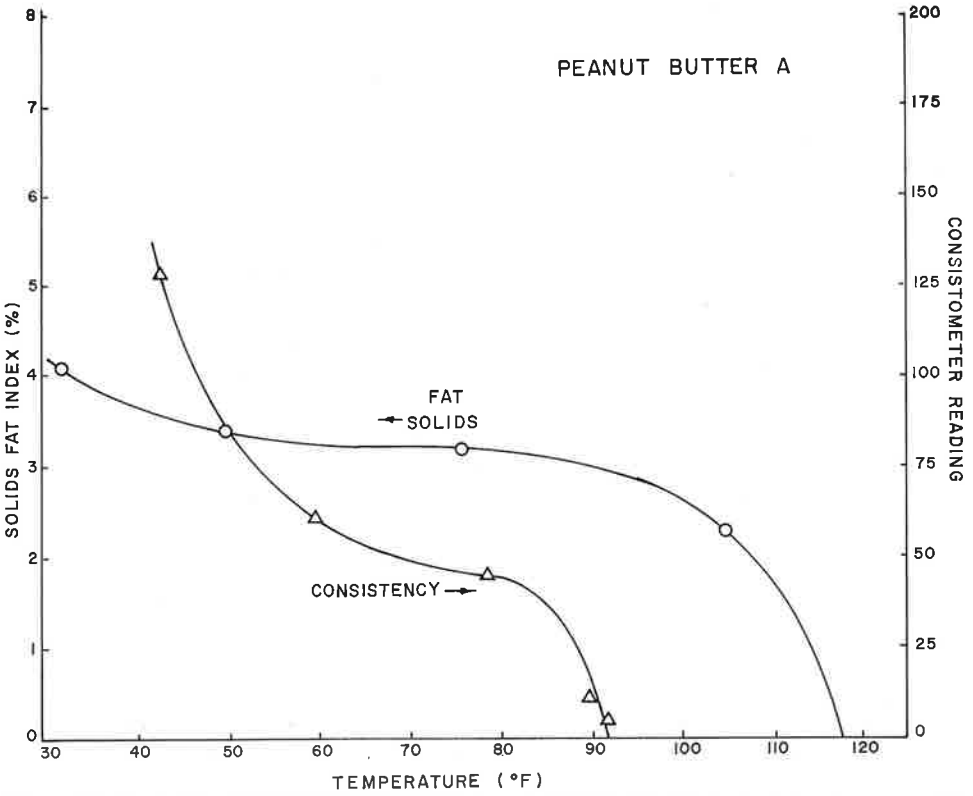

Figure 8. Consistency and Solid Fat Index on Peanut Butter A.

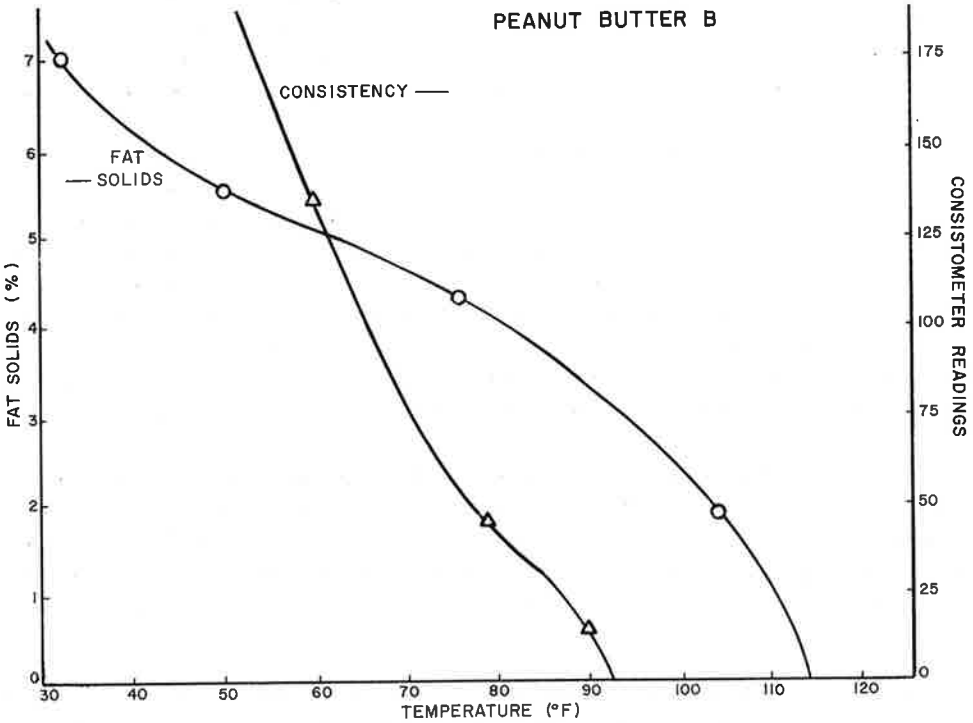

Figure 9. Consistency and Solid Fat Index on Peanut Butter B.

C. *Physical Characteristics*

The physical properties of the oil play an important part in the manufacture of peanut butter. The oil in the peanuts act as a lubricant in the palatability of peanuts as well as peanut butter. In peanut butter the crystal lattice structure set up in the fat prevents oil separation and produces a plastic, spreadable material. To accomplish this, hydrogenated oils or monoglycerides of various hardness are used to form the lattice structure.

There are several techniques used to measure and control these important qualities. The solids fat index (SFI) is given in method Cb 10-57 of the AOCS (1). This is the classical dilatometric method pioneered by Bailey (18). The specific volume of the fat sample is measured and compared to the extrapolated value of the specific volume as a liquid at the same temperature. This difference is proportional to the percentage of solids present as compared to the difference between the extrapolated values of the liquid and solid specific volumes at the same temperature. This method is long and cumbersome requiring at least five hours to complete a test.

A method which may in time supplant the SFI method is the broadline NMR procedure as outlined by Chapman, *et al.,* (24). NMR only reads the hydrogen associated with fat in the liquid state and not crystallized fat. Thus it is possible to temper the sample at various temperatures and read the amount of liquid fat. The difference between this value and the total fats will be the solid phase. Pohle, *et al.,* (84) compared an NMR procedure with the dilatometric procedures. Walker and Bosin (117) have made a similar comparison including calorimetry in the comparison. In a paper by Taylor and his co-workers (103) the relationship of the solids fat index by NMR was found to correlate very well with consistometer readings. We have also found that the SFI by NMR on peanut butter parallels the consistometer or pentrometer readings of finished peanut butter very well as is shown in figure 8 and 9. Thus, there is direct correlation between the solids content of the fat and the final consistency of the product. Only the general shape of the curves can be compared not the actual relative values since the solids phase of peanut butter also has a bearing on the consistency.

One advantage of using NMR Broadline Spectroscophy is the ability to make measurements directly on finished peanut butter. Since bound water, solid fat and non-fat solids give a very broad and low signal level, the liquid fat portion can be detected by the method outlined by Chapman. We have been able to correlate the NMR readings made on the whole butter with the NMR readings on the extracted oil. There are two practical applications of this technique, 1) it is possible to detect whether the proper level of emulsifier has been added and 2) whether the proper processing or tempering conditions have been followed in order to ensure the correct finished consistency. A review of the application of Broadline NMR has been given by Pohle and Gregory (81).

There are other physical characteristics of fats and oils which are of particular value to the processors and extractors of oil. Color is one of the primary considerations to indicate the quality of vegetable oils. The Lovibond method Cc 13a-43 of the AOCS (1) is the standard procedure for this determination. However, we prefer the spectrophotometric method of Pohle and Tierney (85) since it is more objective.

D. *Chemical Changes*

Aging and other forms of deterioration have an adverse effect and cause chemical changes in the constituents of peanuts and peanut products. Since many of these

changes effect the fat in particular, is is appropriate to discuss these changes in relationship to the fat phase of the peanut and its products.

As has been stated, fat is an ester where three fatty acid radicals are esterified to a glycerine molecule. Under cretain conditions and in the presence of moisture, the ester linkage can be broken and a free fatty acid formed. Baker and his co-workers (19) have used the fat acidity as an index of grain deterioration. Usually it is necessary to extract the oil from the product in order to determine the acidity. They modified the methodology of the AOCS (1) method Ab 5-49 by grinding 40 grams of product with 100 ml of benzene in a Stein Mill Model M1. The mixture was milled for a total of 5 minutes. The benzene fat solution is decanted off and filtered. 25 ml of the filtrate is titrated to obtain the amount of fatty acid present. They found this method to give slightly lower values than the official method. One of the problems they ran into was the amount of moisture present in the grain at the time of extraction which has an adverse effect on quantitative recovery.

Another chemical change that the fat phase of peanuts can undergo is oxidation by contact with air. The first step in air oxidation is thought to be the formation of peroxide at the site of the double bond. These ultimately oxidize further to a host of other products, including aldehydes, ketones, acids and some polymerized products. These compounds are the cause of rancidity development and the accompanying off-flavor associated with it. Schultz *et al.,* (88) give a review of the chemistry of liquid oxidation.

The usual method of determining the degree of oxidation or rancidity in fat is the peroxide procedure (AOCS Cd 8-53). This method is particularly suitable for shelf life studies and packaging studies where air permeability of packaging films might be a problem. In our laboratory we weigh 20 grams of peanut butter, add 40 ml of chloroform and stir until a uniform slurry is made. To this, 60 ml of glacial acetic acid is added. The slurry is filtered and washed with small amounts of chloroform in a Buckner funnel under a vacuum. To the filtrate, 5 ml of saturated potassium iodide is added. The solution is titrated with 0.01N Sodium Thiosulfate solution to the disappearance of the starch iodine blue color.

One of the by-products of oxidation is malonaldehyde. This substance can be determined colorimetrically with thiobarbituric acid. The malonaldehyde has to be obtained from the peanut product by steam distillation. Therefore, it is a longer and more involved method than the peroxide procedure. However, peroxides are an intermediary compound in the oxidation reaction and will reach a maximum and then begin to disappear. Therefore, the malonaldehyde content is more nearly related to the extent of oxidation and will be a better index of rancidity as indicated by Kwon and Watts (60), and Sinnhaber and Yu (93).

Scholz and Ptak (87) have reported on a GLC procedure to determine the n-hexonal in oils which is also a by-product of oxidative rancidity. It, too, is directly related to rancidity like the malonaldehyde content. However, it requires more sophisticated instrumentation than the Malonaldehyde method without being any shorter in time or effort.

3. *Protein*

One of the most desirable constituents of peanuts from a nutritional point of view is the protein. Therefore, not only the protein content of the peanut is of interest, but the amino acid composition of the protein has also been investigated. Fontaine *et al.,* (40) and Karon, *et al.,* (55) examined the protein of peanuts and peanut meals by

an electrophoretic procedure. Hirsch and his co-workers (47) investigated the use of a microbiological assay procedure.

Proteins are long chain molecules consisting of amino acids chemically bound together by peptide linkages. These peptide linkages can be hydrolized as the fat ester linkages can be. During roasting, some of these peptide linkages are broken and other linkages with the carbohydrate fraction of the peanut molecule are formed to give the typical brown color (Maillard reaction). If all the peptide linkages are broken, the result would be a mixture of amino acids. Of the eighteen or so amino acids usually found in normal food stuffs, only eight are considered essential for human nutrition. The determination of the amino acids composition is a highly specialized technique and one which is not met very often and particularly not in most routine control work or even much of the more detailed research. Therefore, the reader is referred to the work of Hoffpauir and Guthrie (48) as well as the above references, for information on methodology.

The amount of protein in peanuts or peanut products is often desirable to know. The classical Kjeldahl method is usually employed and remains a very satisfactory technique. This method determines all nitrogen, inorganic as well as organic. However, in peanuts this poses no problem since the amount of inorganic nitrogen is negligible. A two gram sample is digested in concentrated sulphuric acid. This converts all the nitrogen to ammonium salts. This solution is made alkaline and the released ammonia is distilled off and determined by suitable titrametric procedures. The amount of nitrogen found by this procedure is multiplied by 6.25 to obtain the amount of protein present.

Smith, *et al.,* (95) used the AOAC version of this method along with several other methods to determine the constituents in peanut butter. The primary object of his work was to obtain a means of controlling the formulation for the peanut content of peanut butter. Since there are no other nitrogen bearing raw materials used in the production of peanut butter, it was proposed that the ratio of protein found in peanut butter divided by the percentage of protein on a solids basis found in raw peanuts would give the percent of peanuts in the peanut butter. The 95% confidence interval for the final result was about $\pm$ 1.2%. For example, a product having a level of peanuts of 90%, would analyze between 88.8 to 91.2% 19 times out of 20.

There is little choice in methodology for protein. Olson and Heiges (73) have reported on a dye binding technique for use on barley protein (Udy, 106). Whereas they appear to get good results on barley where the level of protein is around 12%, the technique does not look suitable for peanuts where protein is considerably higher. Colorimetric procedures (which this is) are limited by the reproducibility of the instrument reading which, in most cases, approaches 3% of the actual value. Therefore, for a protein content of 30%, the standard deviation would be expected to be around one percent.

4. *Carbohydrates*

Peanuts are not normally consumed or processed for their carbohydrates value since they are only about 18% of the total. Also, there is no commercial value for peanut carbohydrates. Therefore, there has been little interest in determining either the carbohydrate structure or the amount of carbohydrate present.

Woodruff (122) reports the sucrose content of peanuts to be 4 to 7%. It is this constituent that is also the principal carbohydrate in the Maillard reaction (reaction with amino acid), which is the source of the brown color of the roasted products.

In the production of peanut butter and peanut candies, sugar is added as a sweetening agent. Smith, *et al.,* (95) used the AOAC (12) method (22.042) for determining the sugar content of peanut butter. This is the lane-eyon procedure using the reduction of cupric ion by reducing sugars. If the procedure is carried out on the product before inversion, only the reducing sugars such as dextrose and glucose will be determined. The product can be inverted with hydrochloric acid solutions and the determination repeated. The total reading is the result of the reducing sugars present before inversion as well as those formed by the inversion. The difference is proportional to the amount of sucrose or other polysaccharides present in the product.

Starch, a larger polysaccharide molecule, has been reported on by Steiner and Guthrie (96). This is a polarimetric method and is particularly suited for quantities in excess of 10%. It is reported that there is a minimum of interference from protein.

5. *Salt*

The flavor of roasted peanuts is greatly enhanced by the addition of a small amount of salt. To the manufacturer of peanut products, the ability to accurately determine the salt level in his product is important. The usual titration procedure (AOCS 31.009, 12) is time consuming and, therefore, not as suitable as some of the newer, faster methods. The methods will be illustrated on peanut butter, although they apply equally well to salted nuts and candy.

In the titration procedure, the salt is extracted from peanut butter with warm water and filtered to get rid of the insolubles. A pre-determined quantity of silver nitrate solution is added and the excess silver ion titrated with ammonium thiocyanogen solution.

A method which has been highly publicized and is very good for such determinations is the Quantab* procedure. A known amount of hot water is added to the peanut butter or other peanut products and the resulting slurry is filtered. The concentration of the salt in the solution is measured on a Quantab and related to the amount of the original material. Quantabs are graduated plastic tubes containing an absorbent. When dipped into the solution, they show the osmotic pressure by the height of the darkening in the stripe. The height of the discoloration within the stripe is read on a scale and the percent of salt determined from this value.

Recently, electrodes have been made available for reading sodium ion concentration in aqueous solutions on an expanded scale pH meter. Again, the salt has to be extracted from the product by means of warm water. Adding a small amount of chloroform helps to remove the fat which appears to interfere somewhat with the readings. Furthermore, we find that the protein appears to foul the electrode with time so that it is best to store the electrodes in dilute acid and wash them frequently with a mild solution of nitric acid.

The method for peanut butter is as follows:

One gram of peanut butter is dissolved in 25 ml of chloroform. 100 ml of warm water is added to this solution and the mixture intimately stirred. A portion of the aqueous solution is decanted off the top and filtered. The sodium ion concentration is read on the pH meter with the sodium electrode on the resultant filtrate. The concentration of this solution will read between 8 and 9 pH units. The salt concentration can be read directly on the inverse logarithmic scale if the meter is equipped with one. The instrument is standardized by reading a known solution and adjusting it to

*Quantab chloride titrator #1176, Ames Co. Div. of Miles Lab., Inc. Elkhart, Ind.

an appropriate reading. For instance, the meter can be standardized by adjusting a 1% solution to 2.0 on the scale. A reading of 3.0 will then be 1½% and a reading of 1.0 will be .5%. We found the precision of the sodium ion electrode procedure to be good at a salt content of 1%.

The Quantab procedure can be improved by removing the fat with chloroform as was done in the sodium ion electrode procedure. On comparing the two methods, we found the Quantabs tend to give a higher result than the sodium electrode or titration procedure.

6. *Minor Constituents*

There are a number of minor constituents in peanuts which can effect the nutritional and flavor qualities of the product. Like other seed crops, peanuts contain minerals (3% ash), vitamins and cellulose. Often it is necessary to be able to determine minor constituents for specific purposes particularly when studying varietal differences. Willich *et al.,* (121) reported on the variation in thiamine content of peanut butter due to roasting and blanching as measured by a modified thiamine method. Some of the constituents which effect the flavor characteristics will be listed under the section on flavor. The reader, with interest in these components, is referred to the literature for the methodology relating to these components. In most cases, classical procedures can be applied.

H. Physical Properties

Among the more important aspects of the quality characteristics of peanuts are the physical qualities which the various varieties display. Since most of these properties cannot be approached with theoretical applications, empirical technology has to be employed.

1. *Color*

The color of oil in relationship to the maturity of peanuts has been discussed previously. Although this may be an important consideration in raw peanut color, other aspects of this quality characteristic has general application over and above the maturity problem. One of the most important aspects of color and peanut quality is the effect of mold growth or aflatoxin upon the color of the individual kernels. There is a darkening of a kernel when mold has grown on it and this phenomena is used for the electronic sorting of individual kernels. Most shelling plants employ electronic machines that sight the peanuts individually and reject those that are darker than a certain pre-set standard. In this way, many peanuts that are suspect of being contaminated with aflatoxin are removed. Other esthetically unsound or bad flavored peanuts are removed in this process as well.

Peanuts held in storage have a noticeable darkening of the skin color. There is also a notable difference in appearance of skin color from variety to variety. There evidently has not been enough interest in this problem to develop objective colorimetric methods of measuring it. Therefore, methods to evaluate products in this category have been purely subjective.

The color of roasted peanuts or peanut products has possibly received the most intensive search for objectivity. The peanut butter or peanuts are judged as to the degree of roast by the color of the finished product. The U. S. Department of Agriculture (112) has issued a set of color chips to be used when government purchases of peanut butter are made. Many private concerns use these color chips for their own roasting control.

Morris, *et al.,* (70) have described a method of using a reflectance cell on a Cary Recording Ford Spectrophotometer. The data they obtained was converted to several scaler systems such as tristimulus values and Hunter values. They found that it is possible to make an objective evaluation of the redness factor by measuring the color at a given wave length or using one of the hue characteristics of the other systems.

Other devices have been described (92, 112) which undoubtedly can be adapted to this problem. We have investigated the use of a reflectance cell attachment on a Spectronic 20. Although the procedure was adequate to show differentiation, we found it rather cumbersome to use routinely in controlling the roast. Possibly the best use that can be made of an instrumental procedure for reading colors is to establish standards which can be duplicated when age has made them unsuitable for further comparative work. Color chips or standard color sheets, which can be used on the line, are more satisfactory for quality control inspectors to use. MacKinney and Little (61) have given a good review of the importance and methods of measuring color in foods.

2. *Texture*

Foods satisfy the appetite of men in many ways not the least of which is the texture or the mouth feel that the product provides (Matz, 65 and Szczesniak, 99, 100, 101). Two problems related to texture will be discussed here, the first is the kernel hardness and the second is the texture of peanut butter.

a. *Kernel Hardness*

Methods of objectivity measuring the texture of foods have been reviewed by Szczesniak (100) and Elder and Smith (36). One piece of equipment appears to be particularly suitable for measuring a wide range of textural quality in foods is the texturometer developed by Friedman and his co-workers (43). Although this method can undoubtedly be applied to either raw or roasted peanuts, the greater interest in roasted nuts gives preference to discussion on that form exclusive of the other. The ARS Peanut Laboratory in Dawson, Georgia is presently investigating this equipment on peanuts and peanut products. The parameters are in terms of hardness, brittleness, cohesiveness, tenderness, chewiness, etc. How these parameters relate to peanut texture remains to be seen.

Another piece of equipment that has been used on peanuts is the Lee Kramer Instrument. This is a device where the sample is placed in a cage with slots in the bottom about an ⅛ of an inch apart and an ⅛ of an inch in width. A plunger is inserted into the cage with bars that fit through the slots. A sensing device is attached to the plunger so that the force necessary to shear the sample placed in the cage can be measured.

Bourne, *et al.,* (20) describes the use of the Instron machine which works on the same principle as the Lee Kramer. Although it has much more versatility, it also is more expensive and may not be economical unless there are a number of applications in the laboratory for such a piece of equipment.

An alternative procedure would be to grind the peanuts in a mill and measure the power consumption, i.e., the amperage that the mill drew. One problem with such a method is the necessity of always maintaining the mill in a standard condition, i.e., the blades always sharpened to the same degree and placed exactly the same distance apart. An adaptation of this procedure is used as a control to obtain adequate fineness in preparing peanut butter. An ampere meter on a mill will tell if the mill is doing enough work for the volume of peanuts being fed to obtain the fineness desired.

b. *Peanut Butter*

There are three problems related to the texture of peanut butter. The first is the fineness of the material or smoothness that is noted in the mouth. The second is the consistency, which is a viscosity problem. The third, adhesiveness or pastiness, is an inherent characteristic that has not been investigated quantitatively and so will not be discussed here. The texturometer may prove to be a valuable tool in measuring at least the last two of these properties.

1. *Fineness*

One of the easiest ways to determine the fineness is to wash the butter through a screen with a suitable solvent which will dissolve out the oil and not lump the non-oil constituents. The finer the grind, the smoother and more flavorful the product will be. Therefore, a particular level of fineness can be set as a standard based on the smoothness desired and the capability of the grinding equipment. A standard 200 U. S. mesh screen is usually used as a standard for this method.

A quick method for checking fineness is one adapted from the paint industry. This method uses a draw bar procedure described by Doubleday and Parkman (35).* A block with a ½ inch wide, 9 inch long tapered slot is used. It tapers from 20 mil deep at one end to 0 mil depth at the other. A depth scale in mils is etched along this slot. The sample is placed in the deep end of the trough and a draw bar is used to smear it toward the shallow end. At some place along the block, those particles which are too large to pass underneath the bar in the trough will be dragged through the sample giving a score mark. Where this mark begins is an indication of the size of the largest particle in the sample. This method is limited in that it is not strictly quantitative.

A much more sophisticated method of determining particle size has been developed by Coulter. This is the method of electronic gating the principle of which is given by Matter, *et al.,* (64). Basically, it consists of two cells with a given size aperture between them. An electrical current is passed from one cell through the aperture to the other. A sample is introduced in one cell and passed to the other. As the particles go through the orifice, they interrupt the current causing an electrical pulse which is counted. The orifice size is changed to give counts on different size particles. Corrections are made for counts where two or more particles may go through the gate simultaneously. Also, the relationship of the size of the particle and the orifice will determine the magnitude of the voltage drop so the instrument is adjusted to read only certain size range particles. This method has been used on peanut butter. A dielectric fat solvent has to be used for the current carrier rather than water. The benefit of this method is that it provides a size distribution curve which will show the uniformity of the particle sizes present rather than just the number larger than some specified level. Because of the expense of the equipment and the time necessary to run a determination, this method serves primarily as a research tool.

2. *Consistency*

Peanut butter is a plastic material having a plastic viscosity characteristic governed by the complex interaction of the oil and solids phases. This differs from Newtonian flow shown by many materials where the flow is proportional to the force or stress exerted on it. The ratio of the rate divided by the stress is a constant in such materials and is defined as the viscosity. Plastic materials differ in that they do not

*The Peanut Butter Gauge
Precision Gauge and Tool Co., 320 E. Third St., Dayton, Ohio.

flow with the immediate application of a small force. It is necessary to exert a certain force before flow begins. This force is known as the yield value. After the yield force has been exceeded, flow will proceed. Sometimes the flow that results is linear with force similar to Newtonian liquids. However, more often it displays other characteristics. One such viscosity pattern is shown by peanut butter which is defined as thixotropic. A thixotropic material will appear to have lower viscosity the more it is stirred or worked. However, it will regain its higher apparent viscosity after it has had a time to rest.

Since consistency is related to viscosity, it is reasonable to assume that various viscosimeters have been used to determine this particular property. One such instrument

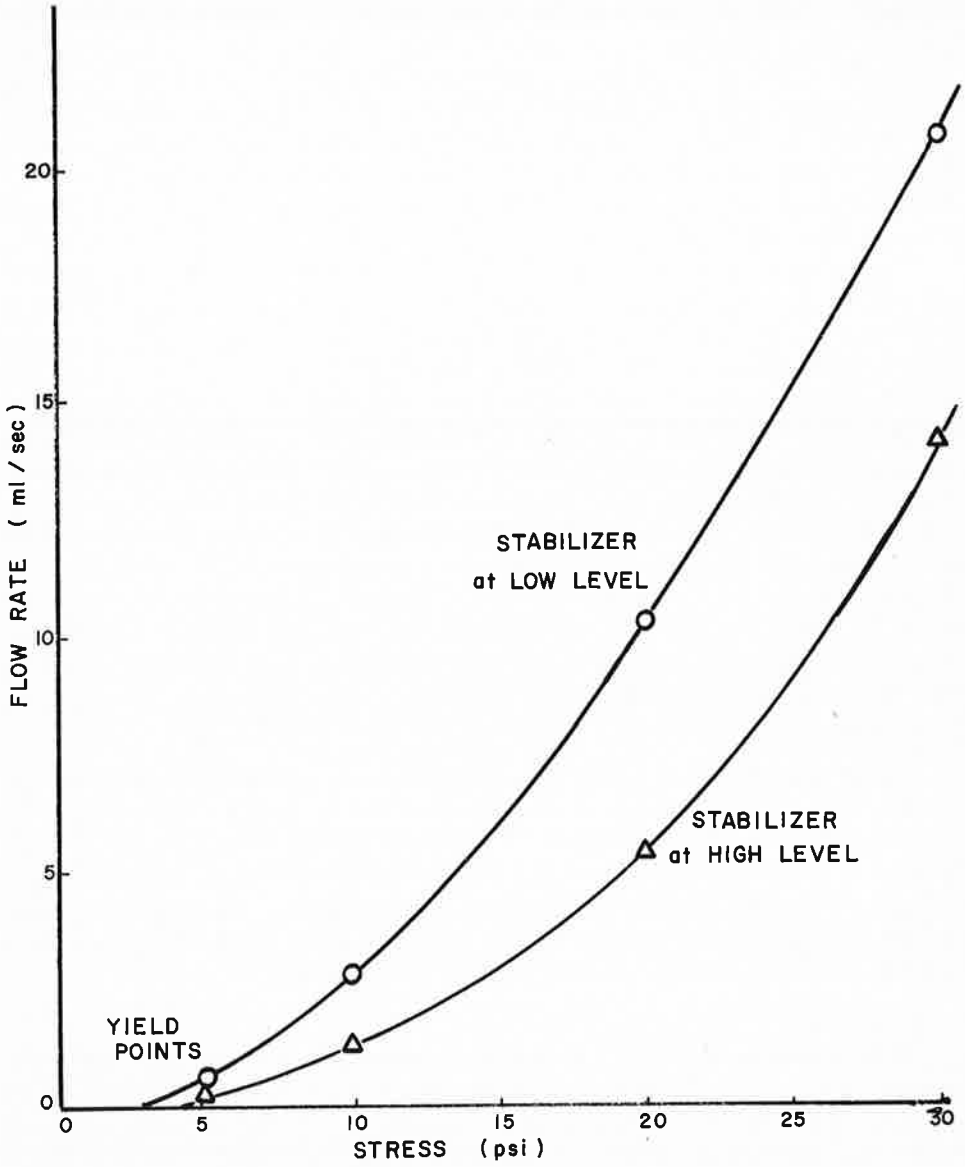

Figure 10. Stress-Rate Relationship of Peanut Butter.

is an Extrusion Rheometers*, which is a capillary tube instrument. Controlled air pressure is the stress exerted on the sample and the rate is measured as the material is extruded from a capillary tube. The stress (air pressure) can be changed and the resulting change in the rate of flow observed. Figure 10 gives the results of two peanut samples run in this apparatus. For plastic materials, it is meaningless to measure viscosity without giving the stress parameters that go with it. Therefore, such a piece of equipment is very useful for research investigation.

For control purposes and many experimental purposes as well, the consistency of peanut butter is much better described by some empirical procedure. One such method is the ASTM Penetrometer procedure (5). A needle plunger of given shape, dimensions and weight is allowed to fall freely from just above the surface into the sample. The depth to which it penetrates the sample is measured and related to the spreadability or consistency.

Clardy, *et al.,* (25) described a shortening consistometer which we have used on peanut butter with great success. A plunger is moved through the sample and the back pressure exerted on the plunger is read on a calibrated dial. A one inch diameter disc with 16 holes of 1/16″ diameter is used to make readings on peanut butter. We have found the relationship between the consistometer and the ASTM Penetrometer to be remarkably good.

These two latter methods measure the yield value primarily. A single value is obtained which is the resistance of the product to force. Since the peanut butter is thixotropic. it is necessary to allow it to set up and not be disturbed before the reading is made. If the product is stirred, anomalous readings will result.

Since consistency is a function of the crystal lattice structure of the fat component as well as the solids present, it is important that temperature be considered when taking consistency readings. Not only is it desirable to know the consistency at room temperature, it is also desirable to know the consistency over the entire range from ice box temperature through room temperature and on past the melting point of the fat phase. For a given level of non-fat solids in the product, the NMR solids fat index is a good index of the consistency. One of the important points is the melting points of the fat phase since products with temperatures over this will lose stability and oiling off will occur. On the other hand, high melting fats have a tendency to give a lardy feel and prevent flavor release in the mouth.

3. *Hull Hardness*

Shellers are desirous of having peanuts that are easy to shell and, therefore, hull hardness is of vital importance to them. I know of no objective measurement to evaluate this property except to observe it in actual plant operation. The Federal State Inspector service of the USDA has small shelling devices which were designed to shell farmers stock samples. Possibly such a device could be used to determine the length of time or the amount of power necessary to shell a given sample of peanuts. Hull thickness has also been proposed as a gauge of hull hardness.

4. *Blanchability*

A large portion of the peanut crop is consumed after the red skins have been removed. To salters, candy makers and peanut butter manufacturers, it is desirable to have peanuts that blanch readily. Shrivels and maturity are two factors which are known to affect the blanchability of peanuts, but there appear to be other unknown factors

*Burrell — Severs Rheometer, Model A-120 Burrell Corp., 2223 Fifth Ave., Pittsburgh 19, Pa.

as well. It would be desirable to have a standard procedure for blanching the roasted kernels and an objective method of measuring the success of the operation.

It has been indicated that samples of roasted peanuts can be hand blanched and the amount of skin left on the peanuts measured. To evaluate the amount of unblanched peanuts, the kernels are divided into five categories: no skin, one quarter, half, three quarters and full skin. The percentage in each pile is calculated, multiplied by the appropriate fraction of bare surface and the total amount of skin removed by summing the products. Others have advocated the use of a small blanching apparatus consisting of two rubber pads between which the roasted peanuts can be rubbed and the degree of blanch evaluated after the operation. Hand blanching by rubbing gently between the thumb and forefinger is possible, but gives a great deal of variability.

There is still more variability in the reading of the final amount of blanchability in the lot since the evaluation is very subjective. We evaluate blanching by counting the number of peanuts that have one fourth or more of the surface still covered with skin. In order to show any difference between lots of peanuts, it is necessary to run at least two 1,000 gram samples in order to show a difference between 80 and 90%. Others have used the number of "rednoses" as a measure of the degree of blanchability. This is effective for processes which are very efficient and leave few unblanched kernels.

I. Flavor and Shelf Life

Flavor and shelf life are two closely identified quality characteristics of peanut products. Because of their great import, they deserve a section of their own.

1. *Evaluating Flavor*

The evaluation of flavor is a rather subjective determination, particularly when the experimentors have to rely on biological judgment factors which not only can vary from person to person, but can vary with the time of day, season of year, appetite of the taster and many other physiological and psychological factors which might affect the results. Therefore, it is generally wise to rely upon a fairly sizable group of people in order to average out individual biases and sensitivities.

A second problem with flavor evaluation of peanuts is how they are to be prepared. Peanut products are normally consumed in the roasted form. Some experimentors have been proponents of grinding the sample and making a peanut butter of it before flavor evaluation. However, the Quality Committee of APREA (50) has adopted the CLER flavor score method which was developed by CPC, INC.

In this method the individually roasted kernels are tasted and an evaluation of the lot is made by this procedure. In the CLER procedure, only 20 peanuts are tasted and, thus, differences in individual kernels can greatly affect the results. We have found that the standard deviation between samples rated on a 100 basis using 5,4,2,0 rating scale for excellence to repulsion was 10. Thus, a significant difference exists between samples if 5 people were asked to run each one in duplicate and the average difference between two lots is 6 or greater. There is another problem in this procedure in that it requires twenty individual judgments on each sample. The saturation point is reached often before the first sample is finished. Thus, only a limited number of samples, certainly no more than two, can be tasted at a sitting. Furthermore, sampling becomes a very acute problem since the number of "bad" kernels in a lot may be fairly small and their incidence in the samples submitted to the judges quite variable. Then, too, the amount of off flavor one or two such kernels will contribute to a product such as peanut butter is questionable. The "peanut butter" method, where a sufficiently

Name_____ No._____

ACCEPTANCE EVALUATION

Please indicate your overall accepability of each sample by checking opposite the facial expression which best represents your acceptance of each product. Please add comments about each sample at the bottom of the sheet.

Sample #1		Sample #2
_____		_____
_____		_____
_____		_____
_____		_____
_____		_____
_____		_____
_____		_____

COMMENTS:

Figure 11. Hedonic Scale for Rating Flavor.

large sample is ground and submitted to a panel, appears to be more reliable. Usually a 10 point scale is used in such a case so that the judge has to make a more critical evaluation. On the other hand, the individual appearance of the kernels are lost and will not affect the judgment of the panelists.

The type of panel that is used depends on the information sought. If the experimentor is primarily interested in whether one sample is better than the other, a simple duo test is sufficient. Untrained panelists can be used for this, in fact, "persons off the street" are generally sought for this type of information. The samples are given to the panelists in pairs and they are asked to give their preference. They may also be asked to rate the samples as to how well they like them on a hedonic scale, such as one illustrated in Figure 11. This is useful in analyzing the data statistically and also will give some reference point for comparisons between tests. Although it is dangerous to draw conclusions between scores from two different tests, such a number will give the experimentor some guidance, particularly if large differences are found. Sometimes the panelists are asked to note any specific observations they may have, such as saltiness, color, appearance, etc. Usually it is desirable to have a broad group of tasters encompassing as many different socio-economic backgrounds as possible. However, Dethmers (31) has shown that panels can be formed from groups with restricted background such as employees of a given concern to obtain meaningful results.

Another instance for using flavor evaluation would be the case where it is desirable to know whether one formulation or one sample differs from another. Again, the duo test is often used, but the Triad test is considered to have greater statistical powers of differentiation and is often used for this purpose. It is desirable to use experienced panelists for these tests. Difference tests are used when substitutions are made in a formulation such as one variety of peanut for another. It can be used in a limited way for semi-routine checking of raw materials or processing conditions, i.e., testing a lot against a standard to see if it is as good or better. One benefit of such a test is that a few experienced panelists can give a significant information in a relatively short period of time.

There are other purposes where trained panelists are preferred to non-trained. For instance, experimentation on shelf life can be evaluated better by a panel which has been trained to distinguish aged (rancid) flavor. It has been our experience that up to four or five samples can be tasted without fatigue. Usually it is wise to repeat the flavor evaluation on the same sample at least once, in order to obtain precision data and more confidence in the results. The same can be done for any of the other specific flavor components such as salt, peanut flavor, off flavors etc. Such panels are very useful in developing formulas and following changes under specific sets of conditions which are known to produce certain types of off flavors.

There is one further step in expertise of the panelists which can be used beneficially by the researcher. Often it is desired to have a panel available that can taste a product and describe it with reference to flavor notes in such descriptive terms that components or flavor characteristics can be defined. Sjostrom and Caul (94) have reported on a procedure known as the flavor profile method. Panelists are trained to distinguish certain characteristic flavors with reference to specific materials as well as intensity of the flavor note. The result is a profile of the flavor of the material. Figure 12 shows the flavor profile on three peanut butter samples to illustrate the type of results that can be obtained from such a technique.

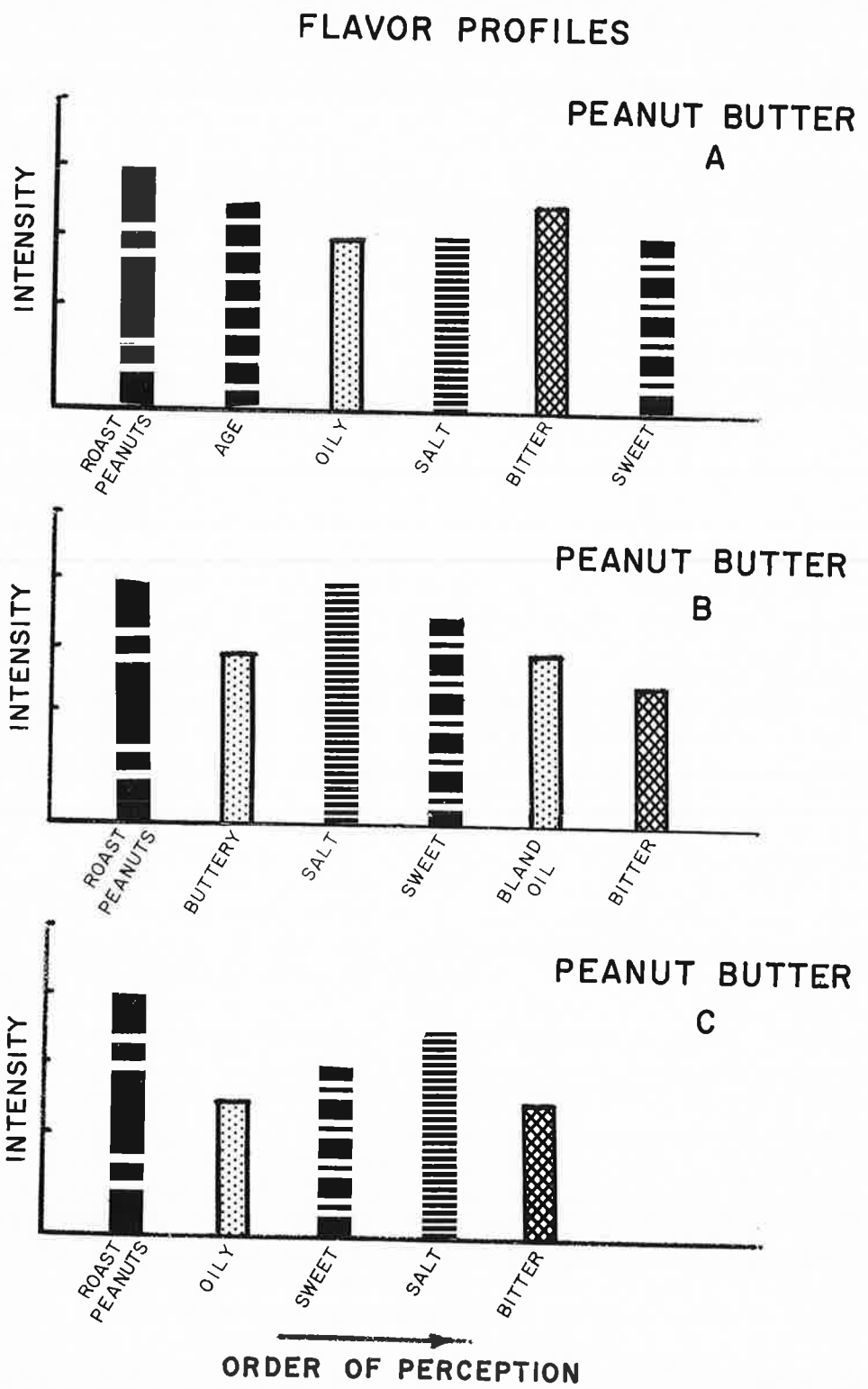

Figure 12. Flavor Profiles of Peanut Butter.

Since flavor testing is one that has many psychological factors, the method of preparing samples and placing them before the panelists has received a great deal of attention. If flavor alone is sought, the lighting of the room or the cubicle in which the panelist receives the sample must be controlled to remove any variability one may see in the sample such as color or texture. In preference or difference testing, it is desirable that the panelists be isolated from each other. This is usually accomplished by putting them into separate cubicles while judging. On the other hand, the profile procedure requires interplay between the various panelists in order to arrive at the best evaluation of the product and thus, such a panel is usually conducted in a room with a "round" table.

A comprehensive discussion of the techniques that can be used in the flavor testing and statistical evaluation of the data is given in the article by Pearson (79) and the books by Amerine, *et al.,* (6), Kramer and Twigg (56) and the Technical Publications by the ASTM (4).

2. *Determining Components Contributing to Flavor*

Several experimentors have worked out methods to determine the flavor components in peanuts and chemical changes and alterations that take place in processing and storage. Pickett and Holly (80) have described and characterized many of the changes that have occurred with roasting. Mason and Waller (63) have described their procedures for preparing the samples as well as the procedures for removing the flavor components by steam distillation and fractionating them on gas liquid chromatographic columns. The fractions received from the GLC columns were further analyzed by mass spectra analysis to determine the components present. Brown *et al.,* (23) has recently reported on a method for isolating large quantities of flavor components which will allow more extensive investigation of their properties. Pattee *et al.,* (74, 78) used a very similar procedure on raw peanuts. However, he was more interested in specific off flavors that might be present in raw peanuts and may carry through into final peanut products. Cobb (27) has developed a method for determining the flavorful carboniles in roasted peanuts using isotope dilution. He used vacuum distillation with radioactive compounds. The distillates were separated on thin layer chromotography and verified with UV Spectrophotography to identify the constituents in the various fractions. These determinations are not only time-consuming but require a great deal of sophistication in the type of equipment necessary to make the determinations. Undoubtedly, the evaluation and the results when properly interpreted can be very meaningful to the agronomist and to others interested in improving the quality of peanuts.

3. *Shelf Life*

Some of the chemical changes that take place in the oil phase upon aging of peanuts and peanut products were mentioned under the chemical changes when discussing the fat chemistry of peanuts. Since shelf life is a very important consideration of flavor change, it is desirable to treat this subject here. Shelf life studies are long and cumbersome although, in many cases, they cannot be entirely dispensed with. Ultimately, it is necessary to go through the job of setting up samples and holding them under given conditions to determine whether they will last the required time. However, accelerated methods have been used which give indications as to the expected shelf life of the product in very short time. Morris and Freeman (68) have demonstrated the use of the active oxidation method (AOM) on crude peanut oils. The oil

has to be extracted and air is passed through it while it is held at elevated temperatures and the degree of rancidity developed is measured by the peroxide value.

Another method for the rapid determination of shelf life of the oils is a modified ASTM method reported by Pohle *et al.,* (82). They found this method to be more reliable with actual shelf life data than the AOM procedure. The method consists of placing oil samples in an atmosphere of oxygen and noting the rate the oxygen is used up or reacts with the oil by the rate at which the pressure drops. Initially, the oxygen uptake is slow. At some place in the time a break in the curve takes place and the oxygen uptake becomes much more rapid. The shelf life is the time interval to the break. We have tried this method on peanut butter without extracting the oil. Although the method worked, the break was not as pronounced as it is in the extracted oil and it was difficult to interpret the end point. However, this method looks promising in that natural antioxidants or catalysts are not removed by the oil extraction process and, therefore, the results may have more commercial significance. Temperature is used in most accelerated methods in determining the stability of peanut products to oxidation. Undoubtedly, moderate temperatures can be used in studying shelf life on salted peanuts, but this is not desirable for peanut butter since stabilized (homogeneous) products will separate and oil out at temperatures over its melting point. It is doubtful whether the shelf life of a separated product is as meaningful as it would be on the completely homogenized product. A comparison of some of the methods for determining the stability of fats and oils is given by Pohle *et al.,* (83). As was mentioned earlier, there are several indices which show the degree of development of rancidity in oils. The peroxide method is frequently used, but the other two methods are preferred by some experimentors, i.e., the malonaldehyde determination and the n-hexonal determination. These techniques can be used in conjunction with flavor analysis in determining the extent of rancidity in peanuts and peanut products.

It has been found that lipids in the presence of proteins can interact creating the possibility of off flavor compounds. Desai and Tappel (30) have studied the reaction between linolenic acid, cystachrome C and oxygen and have described some of the side reactions that have taken place in the amino acid structure of the protein. Narayan and Kummerow (71) have studied the effects of oxidation on lipids in the presence of gelatin, egg albumin, sodium caseinate and a number of other peptones. Andrews *et al.,* (7) have also studied the reaction of fats and proteins. Thus, it is important to evaluate shelf life when all the components of the peanut or peanut products are present. It would be further helpful to evaluate the flavor components contributing to aged flavor by the GLC methods cited previously. What is more important is to determine the components of peanuts to find those which deter oxidation or the number and types of ill-flavored components caused by oxidation. This will assist in the development of varieties or types that will give better shelf life. Fore *et al.,* (41) have a start in their evaluation of sixteen varieties of peanuts.

J. Measurement and Control

A short discussion of the decision or evaluation phase of quality measurement was made in the introduction. A few comments regarding this phase is appropriate in ending this chapter. The measurement of quality can be viewed in two ways. First, it can be the evaluation of the inherent characteristic of a given lot or variety of peanuts. Objective means of measuring this have been described. Second, the upgrading of the product through processing by either machine or man can be measured and means

for this, too, have been described. Thus, many individuals will be interested in measuring quality depending on their use of the information and responsibility. Thus, purchasing agents, operating personnel, marketing and sales managers should be equally concerned about quality and what it means to their operations and give them guidance on the decisions they make.

The reader will recognize the lack of a universal solution to any one problem in measuring the quality of peanuts or peanut products. Box has been the proponent of a fairly rigid statistical approach, and has been cited frequently.

To present the other side of the picture, Feller (39) has decried the fact that too many scientists in the life sciences are relying too strongly on statistical techniques and are not looking for the unexpected. There is no substitute for observation and intuition. For instance, the role of unsaturation in fats and their effect on cholesterol development and arteriosclerosis has been largely established through statistics. The casual relationship has not been proven nor a completely satisfactory mechanism described, so there exist many doubts about the true inter-relationship of these factors. Then, too, many biological tests are long and expensive and require several generations and growing seasons to develop an answer. If after one generation, it is obvious that the test will not work, it is best to stop and try another approach. Likewise, many experimentors advocate trying one or two points in an experiment before going through a whole series to see if the approach is feasible. For example, if a person were interested in trying a liquid sweetener in peanut butter, one or two tests will tell him if the addition of the moisture with the sugar will have too much of an adverse effect to continue with the rest of the tests.

Finally, reference should be made to the control of quality, particularly in the industrial atmosphere. Like many other sciences, the depth and breadth of quality control has grown since World War II. I'd like to distinguish five activities of quality control:

1. Screening finished products.
2. Screening raw materials and supplies.
3. Study process capabilities to assist in running them efficiently for production of quality product.
4. Measuring quality of workmanship to assist in motivating personnel to product quality.
5. Analysis of quality cost to optimize performance.

It is not sufficient to screen finished products if the quality of the raw materials or supplies are not controlled. The quality control department can assist in setting specifications and rating vendors for better purchasing practices (Andrews, 8). However, the only way to properly control your operation is through process control, which requires studying the process and the workmanship to know what it is capable of and then making sure it is done (ASQC, 3).

Most of the emphasis in this chapter has been placed on the technical or physical nature of quality. However, quality control, like most phases of industrial practices, has to cope with human elements, i.e., communication (up, down and sideways and outside the company with vendors and customers), motivation, promotion and errors. In the final analysis, a quality control program has to pay (ASQC, 2). It must satisfy customers and establish the reputation of the manufacturer. Quality is Value!

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